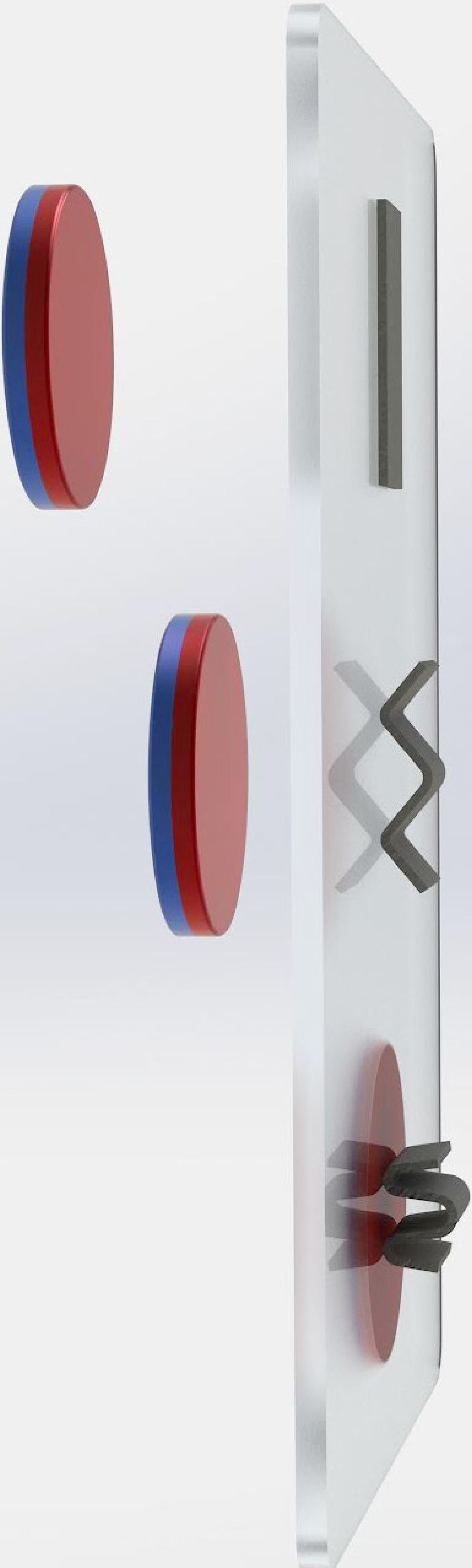

Designing Magnetic Soft Materials for 4D Printing

Sanne van Vilsteren
Master thesis
September 2021

Master thesis Integrated Product Design:
Designing Magnetic Soft Materials for 4D Printing
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Abstract

Magnetic soft materials are a newly developed material that can transform into programmed shapes when it is placed in an external magnetic field. It is made out of silicone with embedded magnetic particles that lay in magnetization patterns due to 3D printing them under a magnetic field. Its characteristics are fast, remotely controlled, reversible and heatless shape transformations. Because the material is new, not much is known about the parameters, how they are made or how they influence the performance of the material. The number of possible applications for this material is also very limited. This graduation project has addressed both these topics by using factorial designed experiments to understand different parameters of the magnetic soft material ink as well as starting the setup of the 4D printer system with which the material can be fabricated.

A design process was also done in which new idea directions have been identified. These were haptics, personalized fits, texture change, replacement of vulnerable mechanical parts, milli devices and scaffolding, and toys. A concept for a demonstrator was made to explain the material to designers and researchers and inspire them. It was a cleaning tool for hard to enter spaces. In the ink development, the influence of different ingredients was discovered as well as how they influence the viscosity of the ink, which is an important factor for the 3D printer system. The viscosity of the last measured ink, which was magnetized, was 500% higher than the viscosity limit of the 3D printer system. The 3D printer system should therefore be reconsidered for a new research project. In the end, also a proof of principle was made which showed the feasibility that magnetic soft material can be made.

This graduation project is the starting point of magnetic soft material at the faculty of Industrial Design Engineering. Even though the first parameters have been researched, much work remains to be done to fully understand magnetic soft materials.

Abbreviations

IDE	Industrial Design Engineering
IPD	Integrated Product Design
DUT	Delft University of Technology
MIT	Massachusetts Institute of Technology
Fe	Iron Carbonyl
SiO	Fumed Silica
NdFeB	Neodymium Ferrite Boron
SmCo	Samarium Cobalt
Fe3O4	Iron Oxide
nM	non-Magnetized
M	Magnetized

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1. Introduction

Magnetic soft material is a material that can change to its programmed shape when it is exposed to a magnetic field. It is composed of an elastomer with embedded magnetic particles. The magnetic fields of these magnetic particles lay in programmed directions so that the material has locally defined North and South poles. When the magnetic soft material is then placed into an external magnetic field, the magnetic fields of the particles will align with it and pull the elastomer with them into the programmed shape.

Magnetic soft materials have shape transformations that are characteristic of the material. The material responds fast, can be remotely activated and has reversible shape transformations. Since the shape change is activated by a DC magnetic field, magnetic soft material has heatless shape transformations which open up more domains for applications.

Magnetic soft materials are an innovative topic on a global scale as well as a new research line at the faculty of Industrial Design Engineering (IDE) at the Delft University of Technology. This report is about the first research done about magnetic soft materials at the faculty of IDE for a graduation project for the Integrated Product Design (IPD) master's degree.

Before this graduation project, I had done a research project and helped with writing a paper about magnetically activated shape transforming polymers or elastomers with incorporated magnetic particles. During that project, my chair and I decided to make magnetic soft material because of its unique characteristics as well as being achievable with the devices available at the IDE faculty.

Since magnetic soft materials are new on global, as well as on faculty level, many parameters of the material and the manufacturing process are unknown or not documented yet. For example, the ink composition, printing speed or applied magnetic field strength. Another gap can be found in the applications for which magnetic soft materials can be used. Right now there are many applications in the biomedical domain, but there is a lack of possible applications and domains for which magnetic soft materials can be used. This project will address both research gaps.

The research by Y. Kim, Yuk, Zhao, Chester, and Zhao (2018) of the Massachusetts Institute of Technology (MIT) will be used as starting point for this project. They have described the recipe for magnetic soft materials and the fabrication process with the most detail. They have also published supplementary video's in which their proof of principle is shown. For the fabrication of magnetic soft materials and magnetization patterns, they used a 3D printer with an electromagnet attached to the nozzle. The applied magnetic field during printing adds an extra dimension to the 3D printing process. That is why it is called 4D printing. The development of the 4D printing system will also be started during this project.

1.1 Assignment and project structure

The assignment of this graduation project exists out of two parts that are run simultaneously. One is making magnetic soft material ink and setting up a 3D printer system. The other is to make a concept for a demonstrator that can show all the magnetic soft material characteristics to designers and researchers. The starting point of both these parts is determined by a literature review about magnetic soft materials on their material composition, characteristics, current applications and manufacturing processes. (see figure 1.1)

The first part is about making ink for magnetic soft materials for the first time and setting up a 4D printer system for the fabrication of magnetic soft materials. Due to being a new material globally, but also because it is made for the first time at the faculty of IDE a lot of ink parameters and how they influence the ink and process are unknown. Therefore a parametric study will be conducted to identify and understand different parameters of the magnetic soft material. Later in the process, when there is more known about the ink, there will be started on the 3D printer set-up to be able to print magnetic soft materials and their magnetization patterns. During the 3D printer development, there will be iterative cycles in the ink development to start the process of optimizing the ink for 3D printing.

The second part of the assignment is focused on the development of a concept for a demonstrator made of magnetic soft material, that shows designers and researchers all the characteristics. The purpose of the demonstrator is to introduce them to the material and inspire them.

For this, a design process will be run that will for the biggest part exist out of the ideation face, in which ideas for magnetic soft material applications will be generated. One idea will be selected to develop into a concept based on including all the characteristics of the material to show the possibilities as well as fitting inside the boundaries of what is possible with the current knowledge. The remain ideas will be sorted into future idea directions for magnetic soft material applications, for which more research needs to be done.

This set-up has some deviations from the original assignment which can be found in Appendix A. There the project would have been finished with a demonstrator made out of magnetic soft materials. However, due to the fundamental and explorative nature of this research and it being the first project about magnetic soft materials at the faculty, some steps took longer than what was thought beforehand.

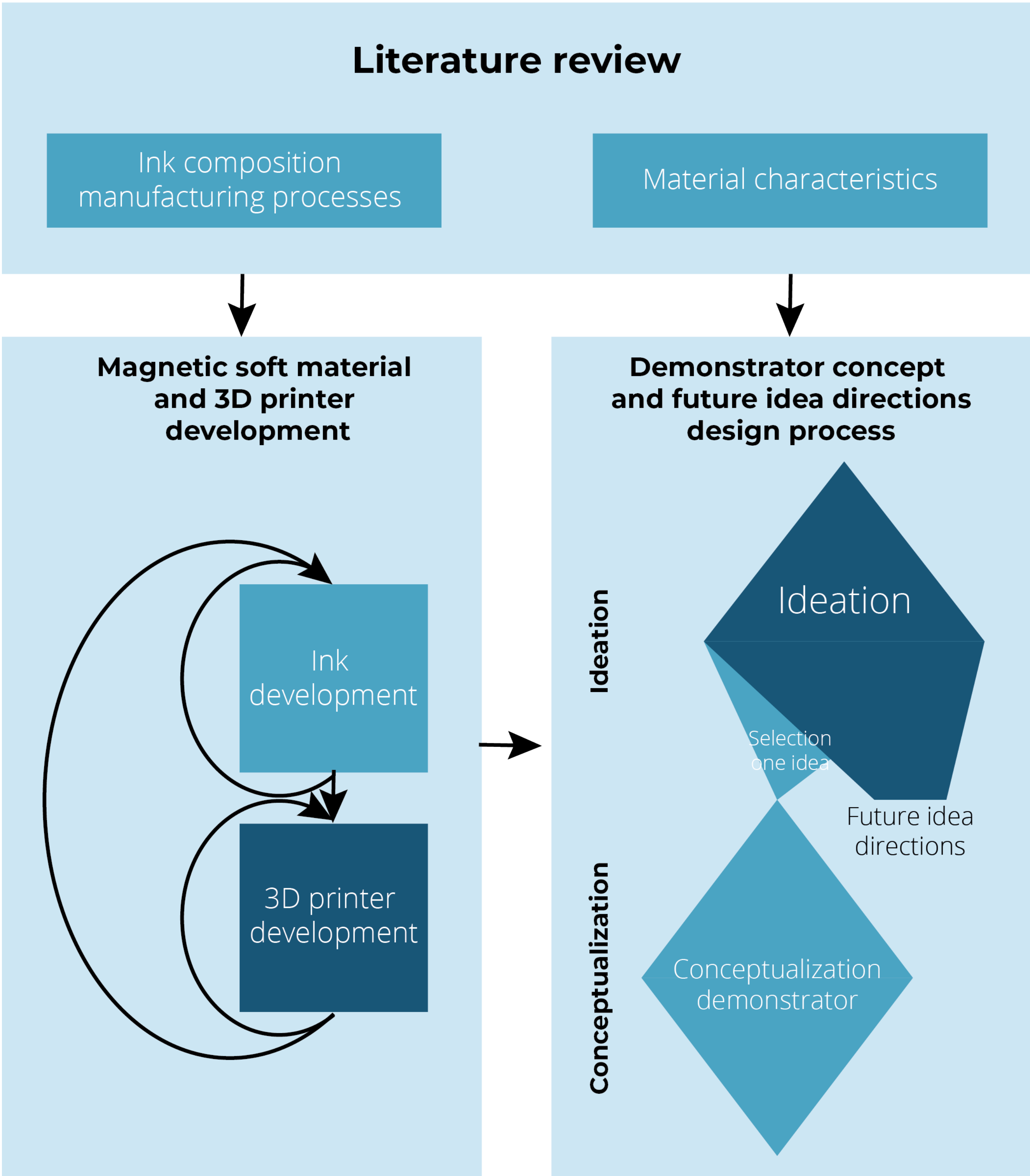

Figure 1.1: *Assignment and project structure*

2. Literature review

Magnetic soft materials are a new type of shape-changing materials that are currently being developed. They exist out of an elastomer with integrated magnetic particles that are embedded in programmed magnetization fields. Through these, the material will remember its shape to transform to. Their shape transformation is activated when they are placed in an external magnetic field.

This literature review will provide an overview of what research is done on magnetic soft materials and identify research gaps. The focus will be on the questions: What are magnetic soft materials? What are the advantages of magnetic soft materials? For what applications can magnetic soft material be used? What research is done on this material? How are magnetic soft materials made? The chapter will end with a conclusion addressing the research gap found in the literature.

2.1 Magnetic soft materials

Magnetic soft materials are elastomers with incorporated magnetic microparticles. Magnetic soft materials can transform into a programmed shape when they are placed in an external magnetic field. This shape change is programmed by creating a nonuniform magnetization pattern with the magnetic particles during the production process. This is done by specifying the North and South pole directions of their magnetic fields for different regions of the elastomer (see figure 2.1). When the magnetic soft material then is placed in an external magnetic field, the North and South poles of these magnetic microparticles will line up with the external magnetic field, like compass needles. This creates micro torques in the elastomer and those pull the elastomer matrix along with the movement of the magnetic microparticles. That is how the shape change is activated.

When the external magnetic field is removed, the magnetic microparticles will lose their forces caused by the alignment of the particles with the external magnetic field. When these forces are removed, the elastomer will return to its initial shape and pull the magnetic particles back in their original position in the elastomer. (Y. Kim, Yuk, Zhao, Chester, & Zhao, 2018; Ma et al., 2020)

Throughout multiple different studies, magnetic soft materials have had different names. For example shape-programmable magneto-active soft matter (Qi et al., 2020), magnetoactive soft material (Chen et al., 2020) or magnetically responsive soft materials (Y. Kim et al., 2018). However, in this thesis they will be called magnetic soft materials after the study by Ma et al. (2020) in which both magnetic soft material as well as magnetic shape memory polymers were developed and made.

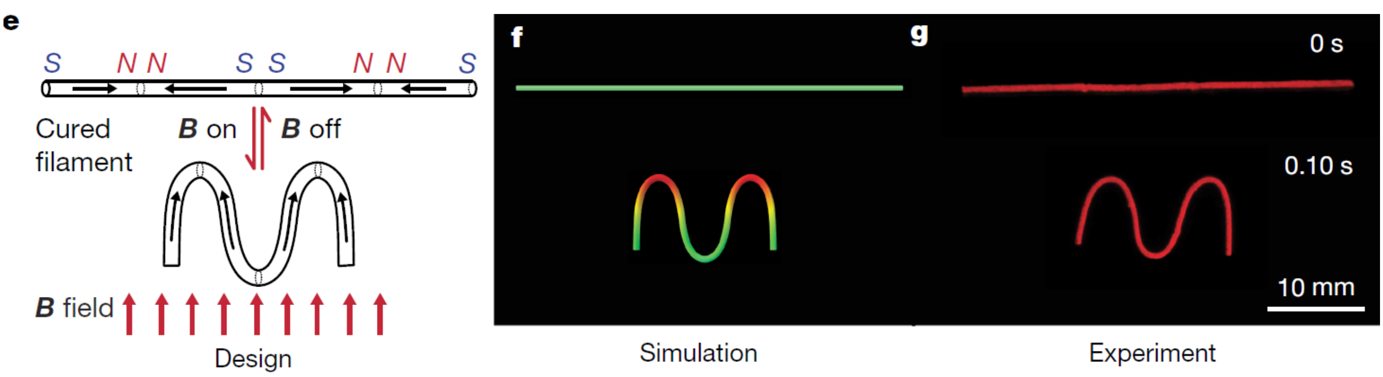

Figure 2.1: *Magnetic soft material with different magnetization patterns (e). When the strip is placed in a magnetic field the strip transforms to an "M" shape. (Y. Kim et al., 2018)*

2.2 Unique characteristics

Characteristics of magnetic soft materials are that they have remote, fast and reversible shape transformations (Y. Kim et al., 2018). They also do not require heat as a stimulus for their shape change. Which makes them applicable for more applications than other more common shape-memory materials.

The most common stimulus for other shape-changing materials like shape memory alloys is heat (Bengisu & Ferrara, 2018). This makes it harder to use them at places where heat is harmful. For example in the case of biomedical devices inside the human body. (F. Zhang, Wang, Zheng, Liu, & Leng, 2019) This makes magnetic soft materials more suitable to work in vulnerable environments.

The remote activation of magnetic soft materials is caused by the stimulus for the shape change being an external magnetic field. Due to this, the magnetic soft material does not have to be attached to wires or have to be in a specific warm environment (for example hot water (Özdemir, 2017)). Through this, they can also be used at multiple places where other material or products cannot come.

More common shape-memory materials do also mostly have a one-way shape activation (Bengisu & Ferrara, 2018), which needs to be counteracted by another force to bring it back to its initial shape. This is not the case with magnetic soft materials. They can be brought back to their initial shape by removing the external magnetic field. Due to the activation of magnetic soft materials by the direction and strength of an external magnetic field, it is also possible to make more shape changes with only magnetic soft material (see figure 2.2) by adjusting this external magnetic field in direction and strength.

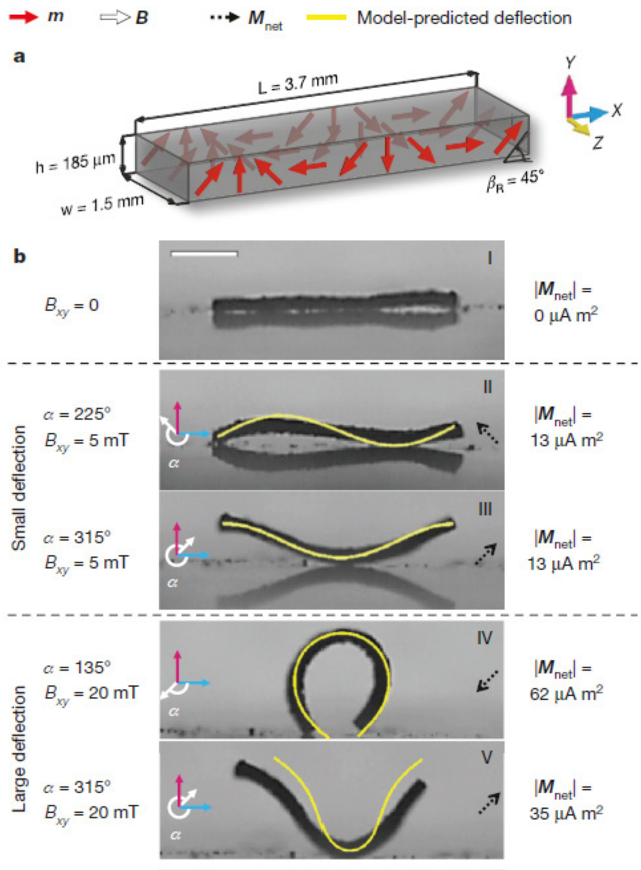

Figure 2.2: *Magnetic soft material strip that is activated by different directions and strengths of a magnetic field and therefore it creates different shape changes (Hu, Lum, Mastrangeli, & Sitti, 2018)*

2.3 Applications

In different studies, some first applications for magnetic soft materials have been made. These will be shown in this section together with how these materials are used in unique ways.

2.3.1 Micropumps

One of the first applications made with magnetic soft materials is a micropump developed by Khoo and Liu (2001) (see figure 2.3). The pump is made out of an elastomer with one incorporated solid permanent magnet. The pumping is activated by an external magnetic field pushing and pulling the magnet, through the constant flipping of the external magnetic field. A characteristic of the magnetic soft material that is used here is the reversible shape change and the fast response. Also, a unique design opportunity of this application is that the pumps could be made at a very small scale. This is partly due to the reduction of components needed to make the pumps.

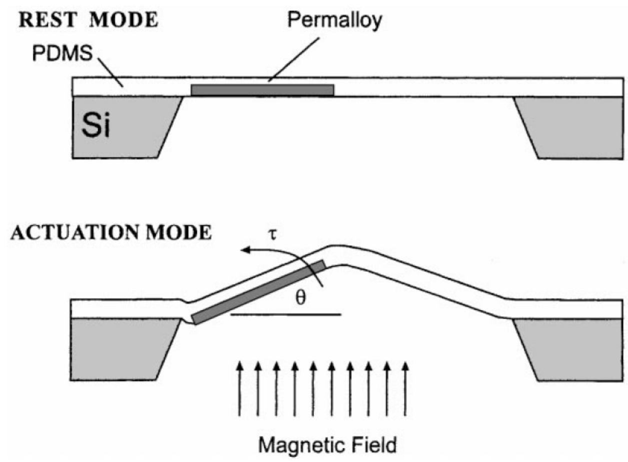

Figure 2.3: *Schematic representation of the micropump and its activation by an external magnetic field (Khoo & Liu, 2001)*

2.3.2 Autofocus and optical image stabilization system

Another application was a combined autofocus and optical image stabilization system for a camera (see figure 2.4). This system was made with a magnetic shape memory alloy, instead of magnetic soft material, but the material activation and working have some resemblance. Especially the fast response time. The magnetic shape memory alloy is moved to one direction by the activation of a magnetic field. It is moved back by a spring when the magnetic field is turned off. Another advantage of this application is also the reduced amount of components needed in comparison with the original actuators for the autofocus and optical image stabilization system. (Gabdullin, Ahmad, & Ro, 2020)

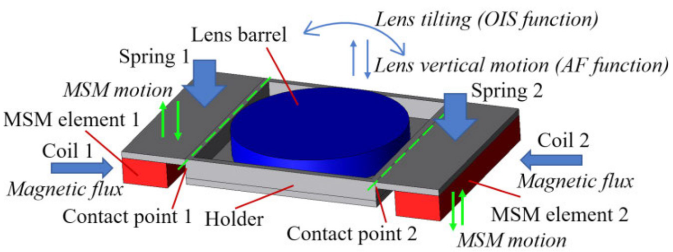

Figure 2.4: *The combined autofocus and optical image stabilization system for a camera. The red boxes are the magnetic shape memory alloy parts. (Gabdullin et al., 2020)*

2.3.3 Locomotion robots (milliswimmers)

One of the most populair applications for magnetic soft materials are robots that are capable of multiple ways of locomotion. These strips are made of magnetic soft material or a few magnetic particles connected with flexible parts (see figure 2.5) (Biswal & Gast, 2004; Hines, Petersen, Lum, & Sitti, 2017). These strips often have a sinusoidal magnetization pattern (see figures 2.2 and 2.6) (Diller, Zhuang, Lum, Edwards, & Sitti, 2014; Hu et al., 2018; J. Zhang, Jain, & Diller, 2016). To activate different modes of transportation, the external magnetic field has to flip or turn around at a certain frequency to keep the magnetic soft material strip moving. The direction and strength of the magnetic field determine the type of locomotion like swimming, crawling, walking or jumping (see figure 2.7) (Hu et al., 2018). The application context of these robots is mostly thought of in the biomedical context for target drug delivery. This application might show all the characteristics of magnetic soft materials. They make use of the remote, fast and reversible shape transformation in an environment where too high temperatures can cause harm. They also show that it is possible to make constantly moving applications with them like the micropumps.

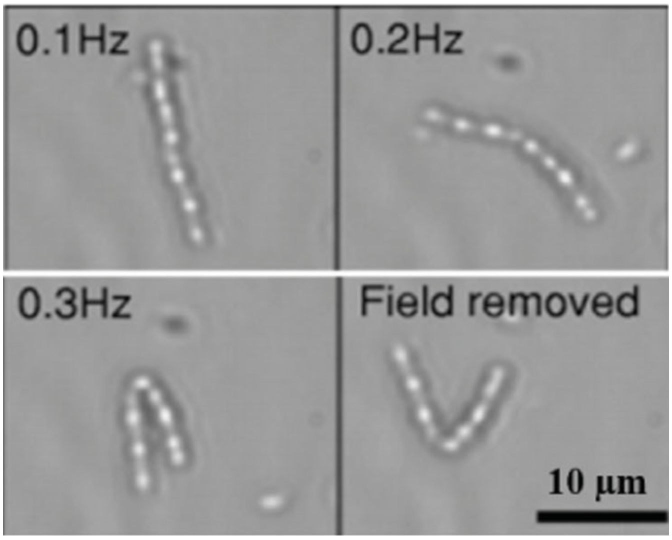

Figure 2.5: *A strip made out of a few magnetic particles that can change its shape by an external magnetic field (Biswal & Gast, 2004; Erb, Martin, Soheilian, Pan, & Barber, 2016)*

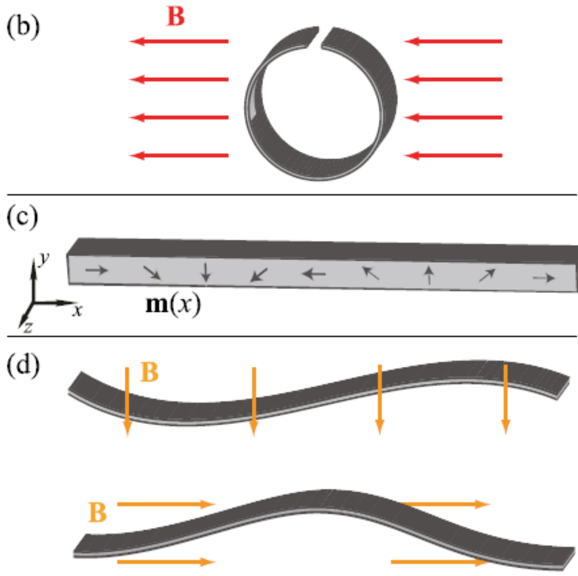

Figure 2.6: *A strip made of magnetic soft material with a sinusoidal magnetization pattern (c). b and d show different shapes caused by the different directions of the magnetic field. (Diller et al., 2014)*

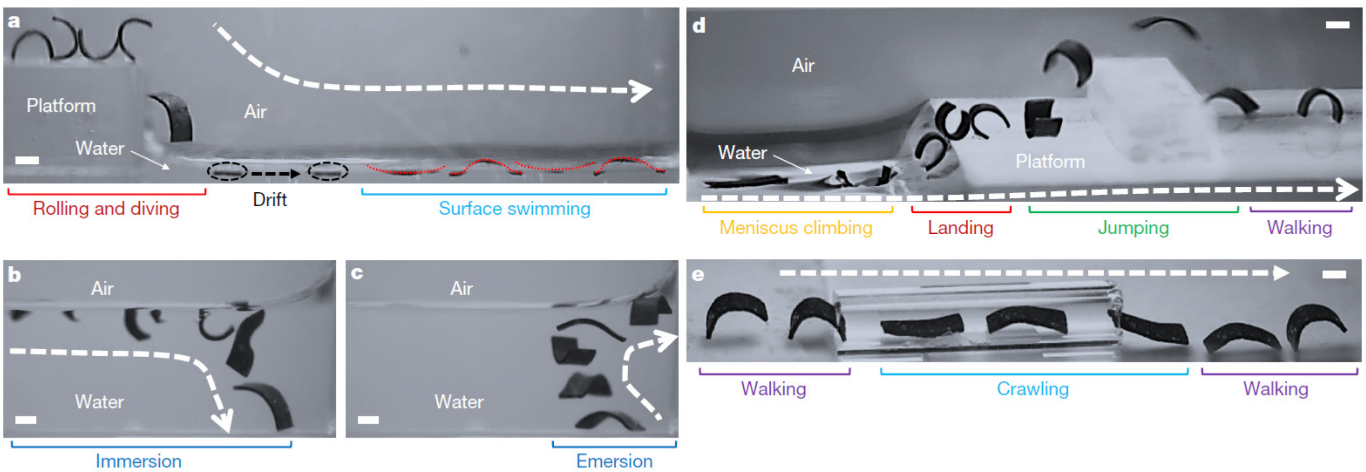

Figure 2.7: *The strip of figure 2.2 can have different ways of locomotion due to different orientations of the external magnetic field and the frequency in which it moves(Hu et al., 2018)*

2.3.4 Grippers

Next to the moving robots, another often recurring application for magnetic soft materials is a gripper. Those are made in studies by (Y. Kim et al., 2018; Xu, Zhang, Salehizadeh, Onaizah, & Diller, 2019). In both these studies, light objects were carried or moving balls were caught through a changing surface (see figure 2.9). Only in a study by Ze et al. (2020), a gripper was tested to lift a ball of a surface (see figure 2.8). Even though this was a magnetic shape memory polymer composite instead of a magnetic soft material, when the material was heated up, it had the same characteristics as magnetic soft material. The gripper was in this case not strong enough and the ball slipped through the teeth of the gripper. In this application the reversible, remote and fast shape change are used again, but also the lack in stiffness in that application is pointed out. Which could be a challenge while working with magnetic soft materials.

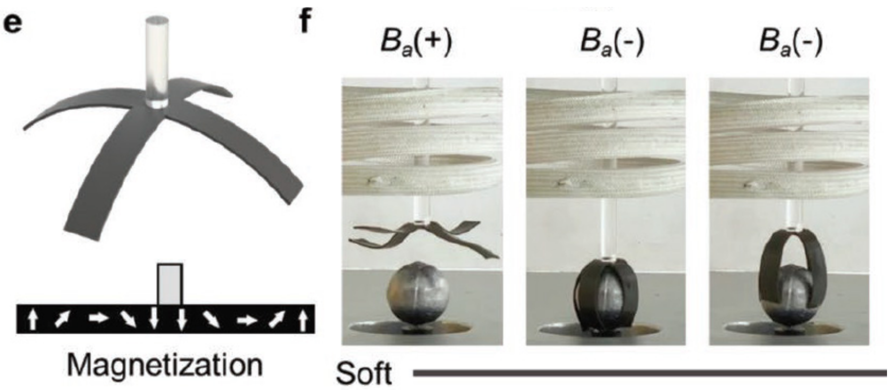

Figure 2.8: The gripper in its soft state (when it acts like magnetic soft material) is not able to lift the ball (Ze et al., 2020)

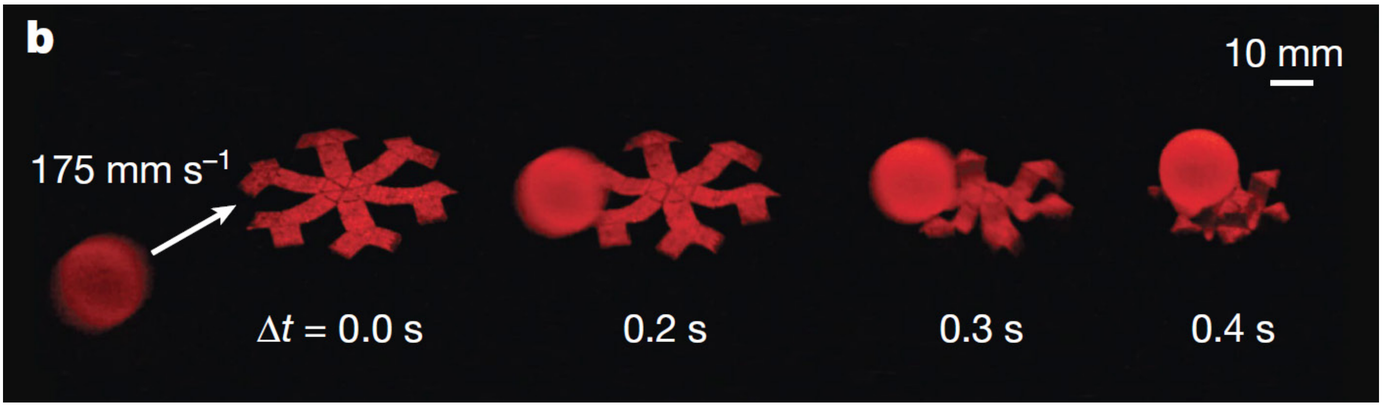

Figure 2.9: A gripper catches a ball when activated by a magnetic field (Y. Kim et al., 2018)

2.3.5 Jellyfish swimmers

Jellyfish inspired swimmers are a combination between the locomotion robots and the grippers (Goudu et al., 2020; Ren, Wang, Hu, & Sitti, 2019). These are designed to carry and deliver cargo grippers (see figure 2.10). The cargo used in the studies often is very small in comparison with the gripper from Ze et al. (2020). These are also controlled by moving magnetic fields.

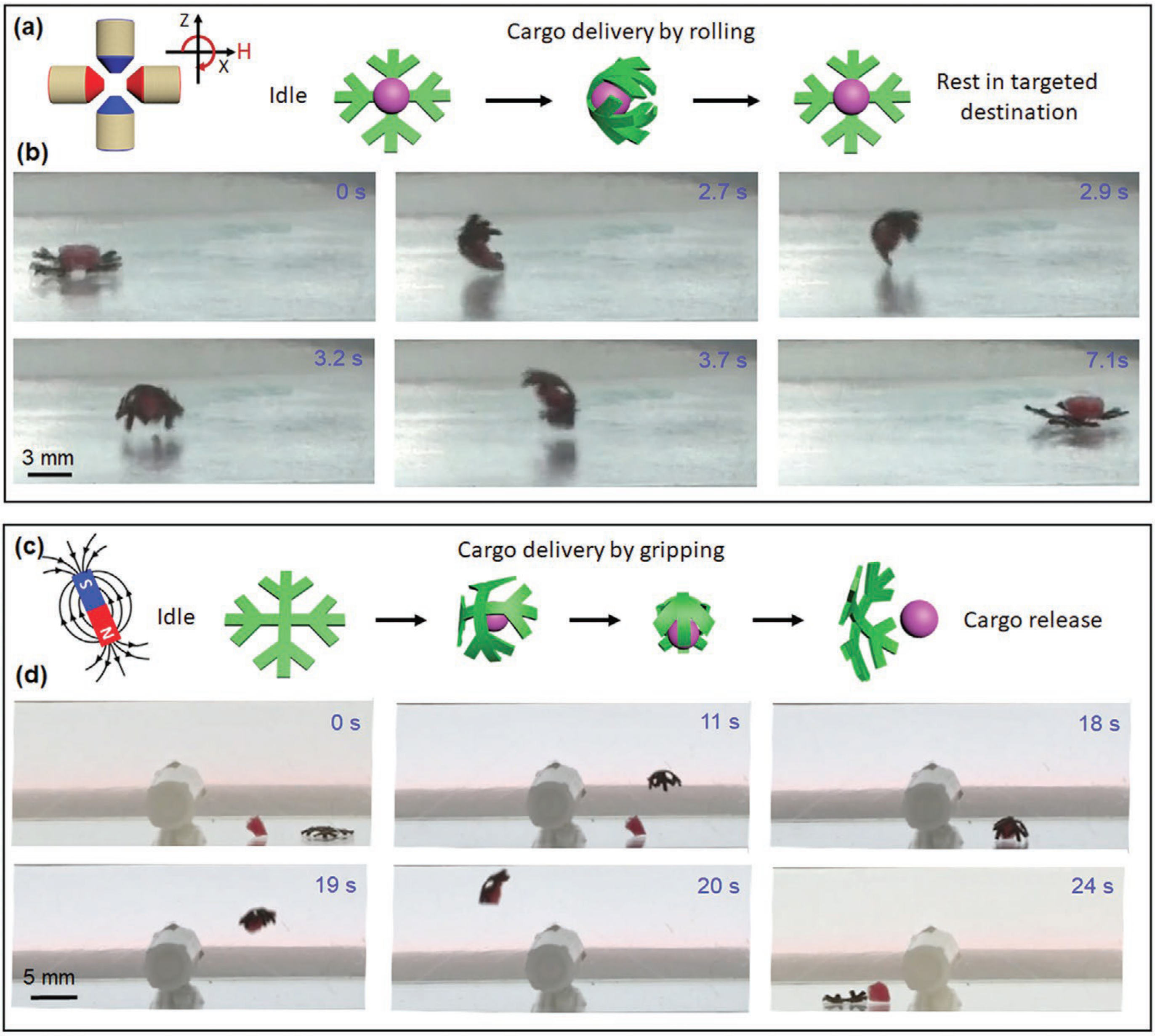

Figure 2.10: In picture (a)(b) the cargo is transported by a rolling jellyfish inspired swimmer. In picture (c)(d) the cargo is gripped moved and later released. (Goudu et al., 2020)

2.3.6 Dynamic material properties

Magnetic soft materials can be used to create a material with dynamic and controllable material properties. This is done by applying a magnetic field to the material, which makes the magnetic particles align and stiffen the material. This is used for dampers (Testa et al., 2019) (see figure 2.11) and noise-cancelling applications (Yu, Fang, Huang, & Wang, 2018). This application relies on the fast and reversible shape transformations.

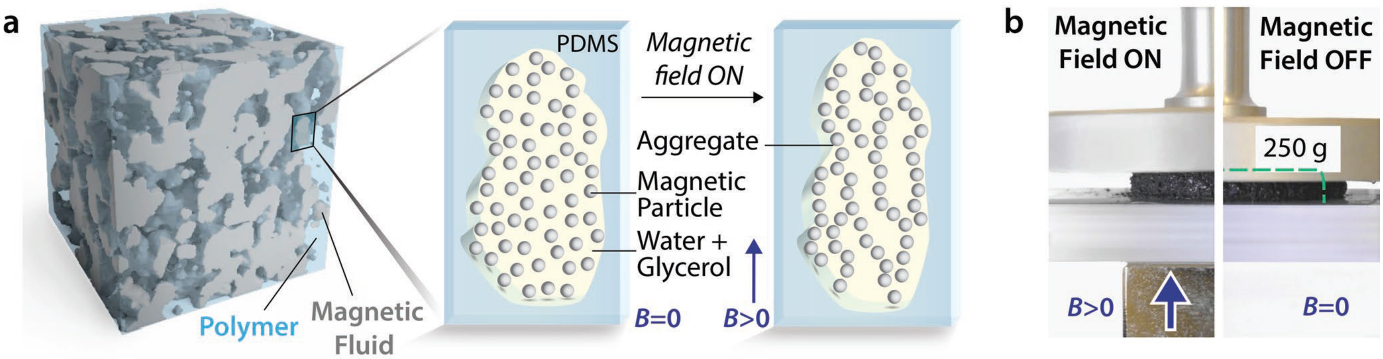

Figure 2.11: *(a) The magnetic particles align when the material is placed in a magnetic field. (b) On the left side, the material is placed in a magnetic field and lifts the weight put upon it, on the right the material with the same weight without the magnetic field. (Testa et al., 2019)*

2.4 Manufacturing processes

The last questions that remain are how are magnetic soft materials made and what research is done about them? These questions will be answered in this subchapter.

2.4.1 History magnetic soft materials

Magnetic soft material, as described at the beginning of this chapter, are composed of an elastomer with incorporated magnetic microparticles. In earlier studies, these materials were constructed by incorporating big magnets into an elastomer. Like for example the micropump application in the previous subchapter (see figure 2.3). These big magnets differ from one single magnet (Khoo & Liu, 2001) to disc formed magnets (Lagcore, Brand, & Allen, 1999) and platelets (Erb, Sander, Grisch, & Studart, 2013). There are also studies done that do not use elastomers, but still use the same type of magnetic responsive shape transformation. For example attaching magnets to textile (Alharbi, Ze, Zhao, & Kiourti, 2020).

2.4.2 Multiple fabrication techniques

For the fabrication of magnetic soft materials with their magnetization patterns, there are multiple fabrication techniques. Three main fabrication processes can be distinguished for magnetic soft materials based on when precisely the particles are magnetized and when the solution is cured. These are: magnetizing the material after curing, selective material curing and direct ink writing (a 3D printing technique) where the material is cured afterwards.

2.4.2.1 Magnetizing after curing

With magnetizing after curing, an elastomer with incorporated magnetic particles is cured in a specific shape (mostly strips). After the material is cured, the shape can first be cut out and is then placed in a specific shape that the material should transform into when it is activated by a magnetic field. This material in the specific shape then is magnetized in the direction from which the external magnetic field should come when the shape change is activated. After this, the fabrication process is done. A good example of his fabrication process can be seen in the study by Lum et al. (2016) (see figure 2.12) in which they placed the material in a mould before magnetizing the magnetic soft material. Another study using this method is the one of Diller et al, (2014) in which they made milli swimmers (see figure 2.6).

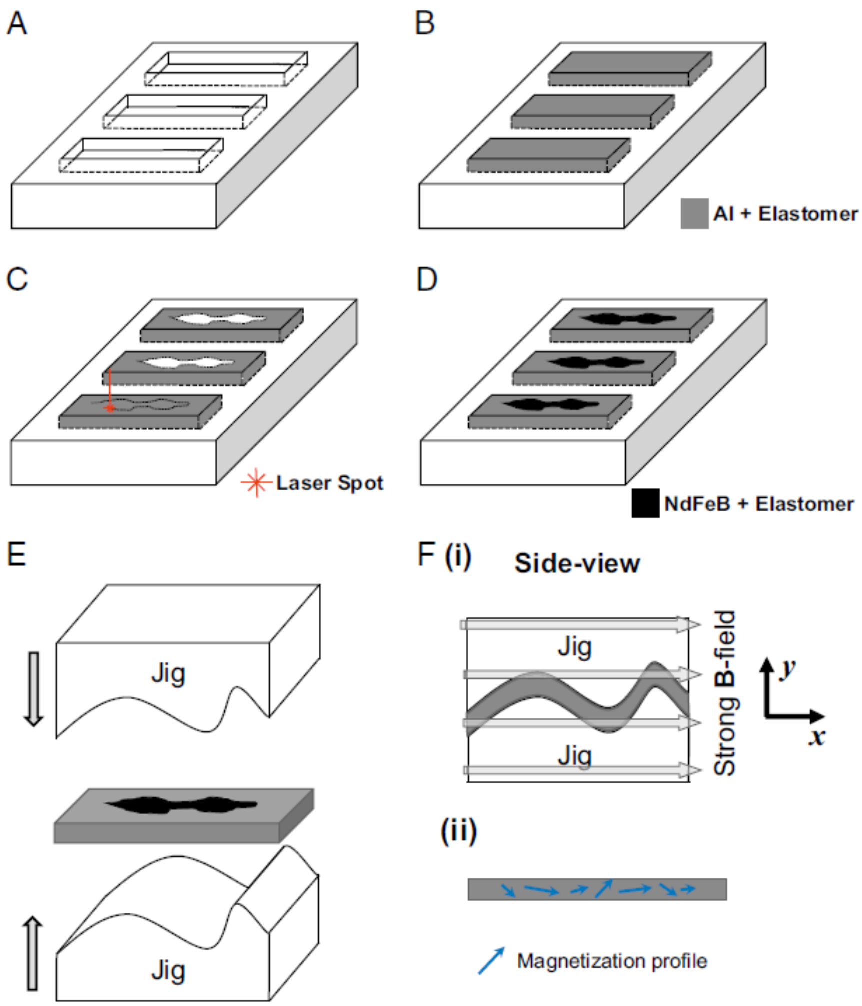

__Figure 2.12:__ B: the magnetic soft material cures in a mould. E: the material is placed in a mould before magnetization. F (i): the material gets magnetized. (Lum et al., 2016)

2.4.2.2 Selective material curing

In selective material curing, the uncured materials incorporated particles are magnetized before the material cures. During the process, an external magnetic field is placed on the uncured material. This makes all the particles turn so that their magnetic fields do align with the external magnetic field. Depending on the process there are two ways in which the shapes with a magnetization pattern can be build-up.

In one procedure, after the external magnetic field is applied, the entire material is cured. The material is then transported to a different mould. More material is poured into this mould to expand the shape and the external magnetic field is placed in the preferable direction, the material cures and the cycle repeats. See for example the study by Erb et al. (2013) (see figure 2.14).

The other option is to have a bigger mould at the start and only cure specific regions of the uncured elastomer. In the study by Xu et al. (2019), this is done by using a UV curable silicone ink and targeting the UV light to specific regions to cure (see figure 2.13). After the material was cured the direction of the external magnetic field was changed. The magnetic particles in the uncured elastomer aligned again with the external magnetic field and new regions were cured.

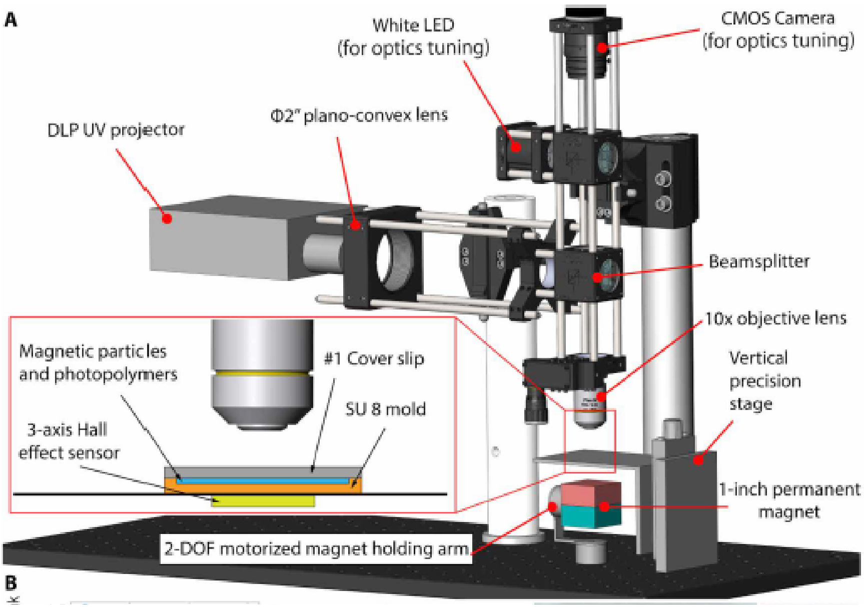

Figure 2.13: The manufacturing method from the study by Xu et al. (2019) where selective parts of the material are cured. The process is based on ultraviolet stereolithography. The 1-inch permanent magnet is the external magnetic field used to orient the magnetic particles. The lenses are used to cure specific regions of the material.

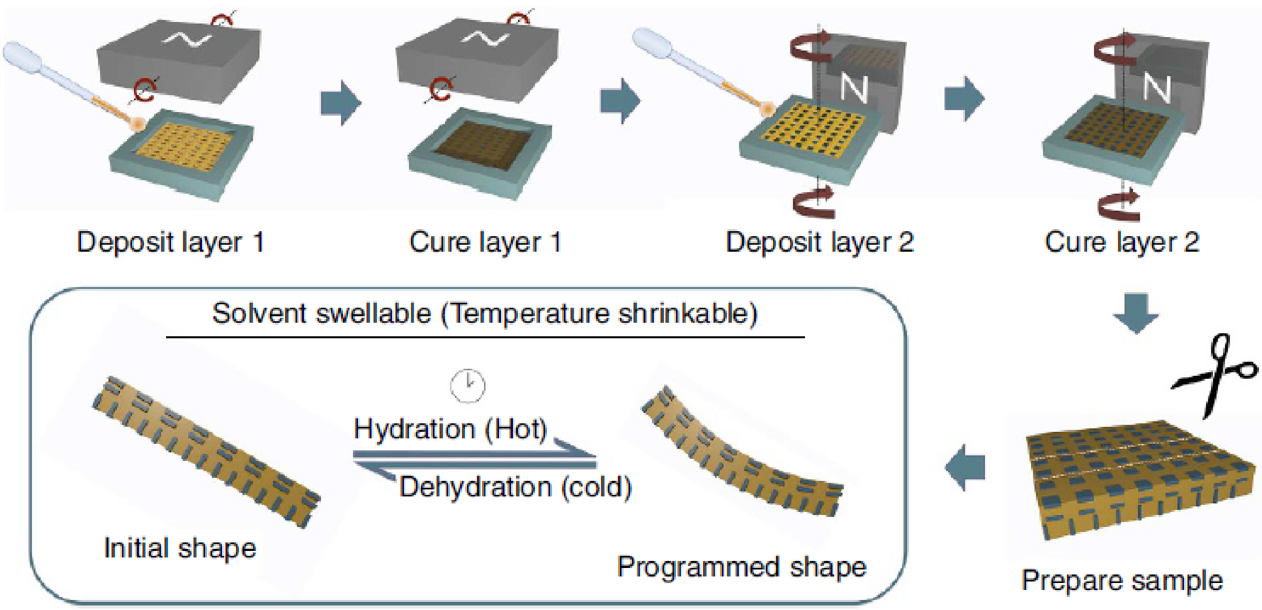

Figure 2.14: The production process in the study by Erb et al. (2013). First, the first layer is placed in the mould and cured. After that the second layer is placed on top of the external magnetic field is turned and the second layer is cured.

2.4.2.3 3D printing (direct ink writing)

The last fabrication method for magnetic soft materials is 3D printing or more specific direct ink writing. In this method, the elastomer is mixed with the magnetic particles. The magnetic particles are magnetized before the 3D printing. While the ink is being extruded through the nozzle in the programmed shape, the magnetized particles will align their magnetic fields with the one of the permanent magnet or the electromagnet attached to the nozzle of the 3D printer. This manufacturing technique was used in the study by Y. Kim et al. (2018) (see figure 2.16) and in a study by Ma et al. (2020) (see figure 2.15).

Where the previous two fabrication methods mostly create flat material structures, with this 3D printing technique it is possible to also make 3D and more complex structures of magnetic soft materials (see figure 2.17). This makes 3D printing a promising method for the fabrication of magnetic soft material applications.

The 3D printing of magnetic soft materials is a recent development. The research of Y. Kim et al. (2018) was one of the first that demonstrated 3D printing magnetic soft materials and their magnetization patterns. Before the study by Ma et al. (2020) other research on 3D printing magnetic soft materials has been reported. In those studies, the manufacturing technique only lacks the application of a magnetic field around the nozzle while printing and therefore the printing of magnetization patterns.

In the work of Roh et al. (2019) and Lantean et al. (2019) magnetization of the particles was mentioned. However, there is nothing mentioned about magnetization patterns made in their material. The reaction of the material to an external magnetic field does not seem to be influenced by a magnetization pattern, but more the general attraction of the magnetic particles to a magnetic field. It can be that they are based upon soft magnets instead of hard magnets (permanent magnets). Permanent magnets are required to create magnetiztion patterns (Xu et al., 2019).

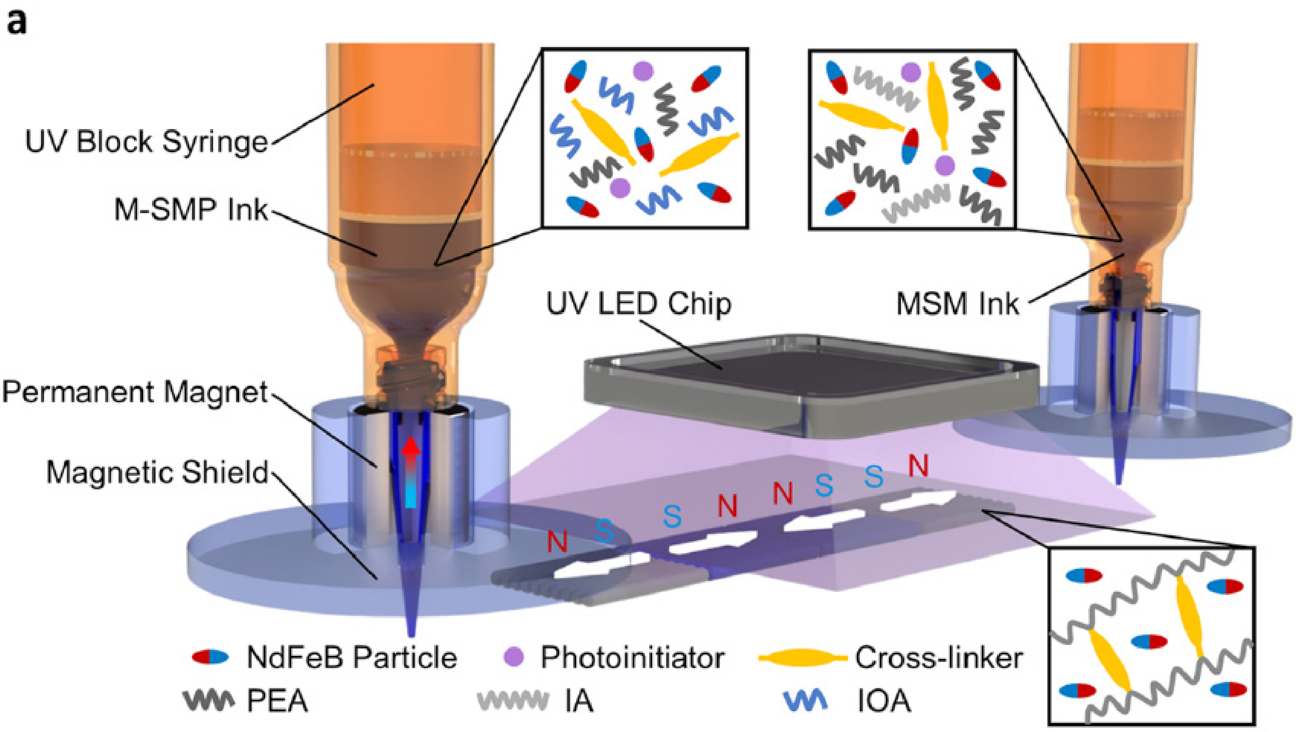

Figure 2.15: The 3D printing of magnetic soft materials in the study Ma et al. (2020) is part of a multimaterial printing system together with magnetic shape memory polymers. The magnetic fields of the magnetic particles are aligned with the field of the permanent magnet placed at the nozzle. A magnetic shield is placed between the permanent magnets and the printed material so that the printed magnetization patterns will not be distorted.

In the work of Chen et al. (2020), the magnetization of the particles is mentioned in their experimental section. They place the cured 3D printed structure of magnetic soft material in another 3D printed mould and magnetize the magnetic soft material in this shape. This is the same principle as described in the section magnetizing after curing.

So the studies by Y. Kim et al. (2018) and Ma et al. (2020) are a two of the more well-known works demonstrating this new fabrication technique for magnetic soft materials. Because there is almost no research done about this manufacturing technique for magnetic soft materials, a lot of information on preparing a setup for 3D printing magnetic soft materials, preparing the ink and the parameters influencing the magnetic soft material objects, are still unknown. Like for example how printing speed affects the magnetization pattern on how fast the particles do turn around in the elastomer matrix when the magnetic field is flipped for optimal results. Also, to make this material and fabrication technique available for designers, to use for new applications, this information will be useful and lower the threshold for working with it.

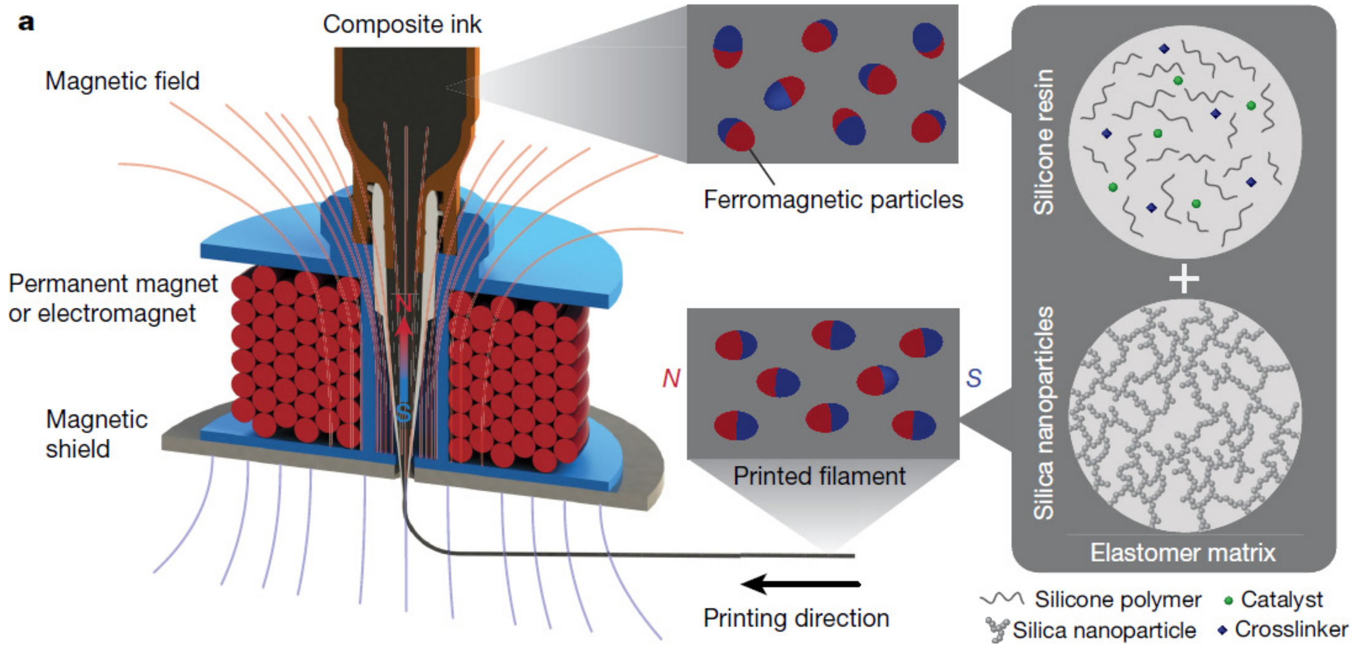

Figure 2.16: The 3D printing process of magnetic soft material developed by Y. Kim et al. (2018). The magnetic particles orient with the magnetic field that is applied through the electromagnet at the nozzle while printing. The shield is attached between the electromagnet and the printed material so that the printed magnetization patterns will not be distorted during the printing process.

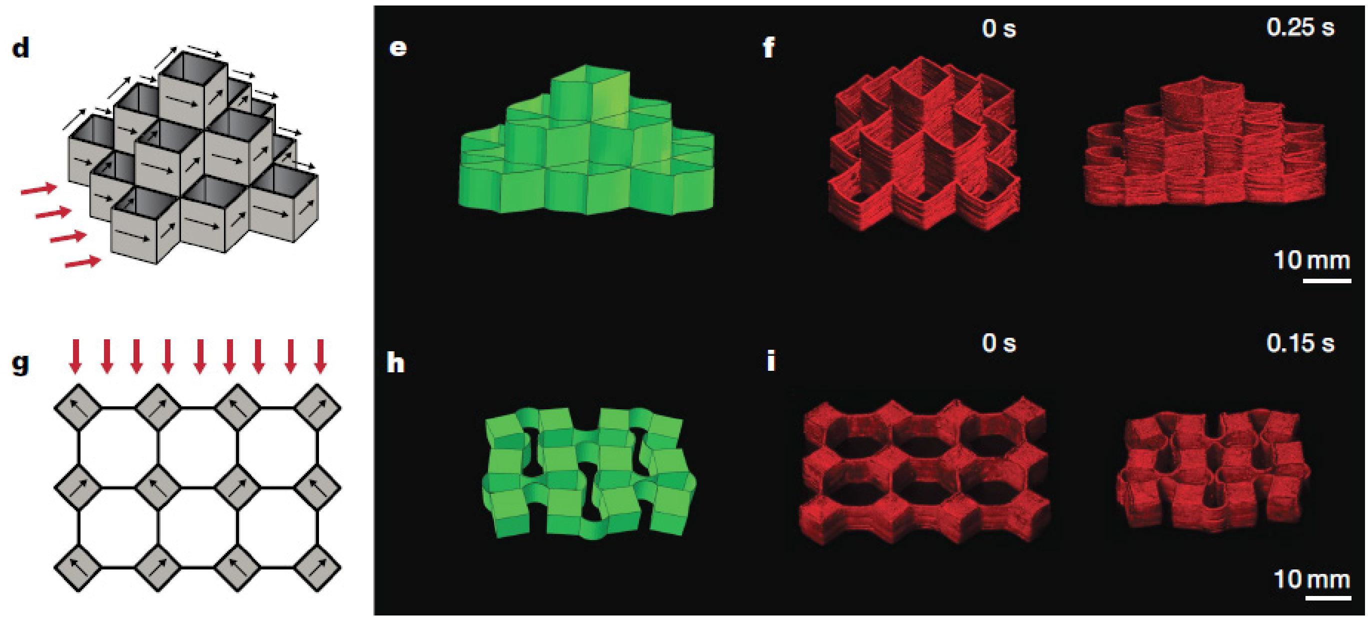

Figure 2.17: More complex shapes made from magnetic soft materials, made with 3D printing (Y. Kim et al., 2018)

Direct ink writing of silicones

Not only the magnetic properties of the magnetic soft materials influence the production process. Another important factor is the direct ink writing of the uncured silicone ink with incorporated particles. Direct ink writing is an extrusion-based 3D printing technique (Tagliaferri, Panagiotopoulos, & Mattevi, 2021). In this technique, the ink loaded in a syringe barrel is extruded.

The study by Zhou et al. (2019) points out that there are three difficulties for printing with silicones:

1. Materials: silicones are hard to print through their low viscosity and long curing time
2. Devices: the time to print with the ink is short because the ink starts curing after it is prepared when a two-component silicone is used. This makes the printing properties of the silicone unstable.
3. Process: silicones often have a slow printing process and the printing parameters are often determined by a trial-and-error process

This brings extra challenges while setting up a system to print magnetic soft materials. The first problem is addressed in both magnetic soft material printing papers as is in the study by Zhou et al. (2019). Fumed silica particles are added to the ink to increase the viscosity of the ink (Y. Kim et al., 2018; Kuang et al., 2018; Ma et al., 2020; Tagliaferri et al., 2021; Zhou et al., 2019). This gives the silicone shear-thinning properties, which means that under pressure the viscosity of the ink will decrease so that it is easier to extrude (see figure 2.18). When the ink is extruded and lays still again, the fumed silica rebuilds its internal bridges and the ink becomes more viscose again. That improves the stability of the printed structure. (Zhou et al., 2019)

Furthermore, Y. Kim et al. (2018) use a support ink to stabilize their printed structures and Ma et al. (2020) use UV curable silicone that is cured during the printing process after each layer.

The second and third challenges in printing silicones are not addressed in the studies by Y. Kim et al. (2018) and Ma et al. (2020).

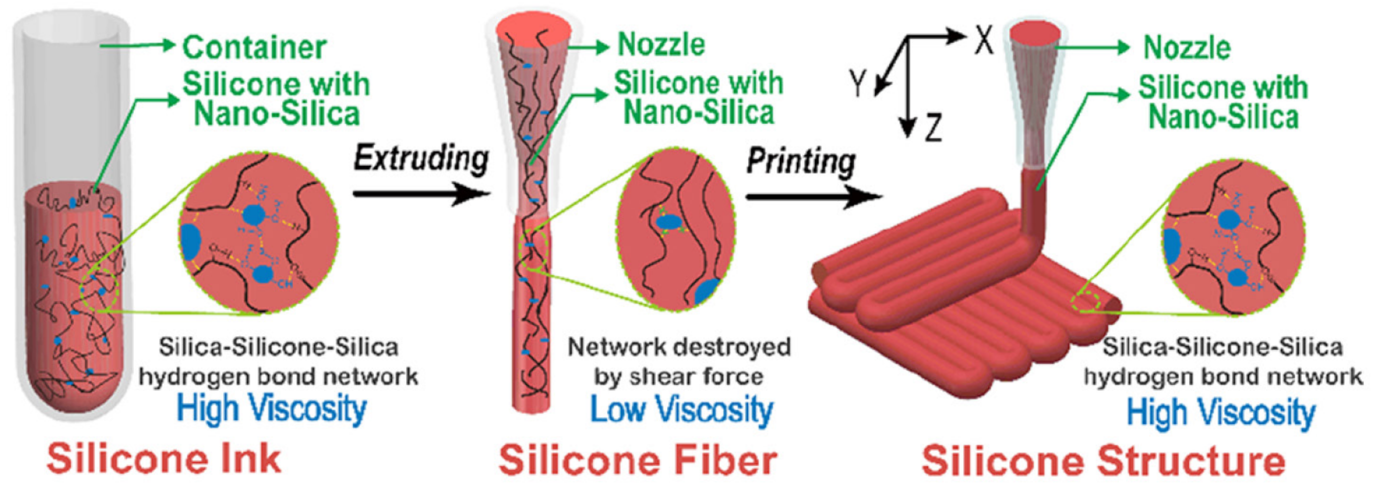

Figure 2.18: The viscosity of the silicone changes throughout the printing process through the addition of fumed silica particles (Zhou et al., 2019)

2.5 Conclusion and future perspective

2.5.1 Conclusion

In summary, magnetic soft materials are materials that can transform their shape when they are placed in an external magnetic field. They are made of an elastomer with incorporated magnetic particles that lay in designed magnetization patterns. The alignment of the magnetic fields of the particles with an external magnetic field will stimulate the shape change of the material and pull the elastomer along with the movement.

Unique characteristics of magnetic soft materials are their fast, reversible, remote and heatless shape transformations. These have been used in different applications and showed multiple advantages like a decreasing number of components needed for actuators and making actuators at a small scale. Another unique application of this material is that it can be used to create controllable and changeable material properties and move constantly while being influenced by a moving magnetic field. Currently, a lot of applications are being developed in the biomedical context because of the remote and heatless shape transformations.

The history of magnetic soft materials was discussed and three main fabrication principles. 3D printing mantic soft materials show the biggest potential because of the possibility to make complex and 3D structures. This field is quite new and a lot of research has to be done on fabrication magnetization of patterns and printing with silicones. Still the influence of multiple parameters, like for example the effect of speed on the magnetization patterns, are unknown.

2.5.2 Future perspective

So now, in the present research, I first start by making a proof of principle of magnetic soft material. For this, I use the study by Y. Kim et al. (2018) as a starting point, because it is a highly cited paper and in their supplementary information, they show videos with a proof of principle of their magnetic soft material. Also, their recipe is described in the most detail. For this, a magnetic soft material ink is made as well as a start on the 3D printer setup. During the process different parameters are discovered and tested systematically that are not always highlighted in previous studies, to broaden the understanding of magnetic soft materials and their production. There is also focussed on challenges about 3D printing silicone-based materials during the research.

The other focus of my graduation project is on designing a concept for a demonstrator that can be made with magnetic soft materials. To show the potential of the material for further implementations in products to other designers and researchers. Also, a list with possible idea directions, for further research or design is gathered to broaden the list of application domains.

3. Material and 4D printer development

To make magnetic soft materials, ink should be developed as well as a 4D printer system to print shapes and program the magnetization patterns.

This section of the report will show the procedure of MIT on which this research is based, and the used testing methods. After that multiple ink parameters and their influence, will be discussed. Also, a start is made on developing a 4D printer system. This section concludes with the proof of principle that was made of magnetic soft material.

3.1 Starting point research

As a starting point for this research and as the main guideline, the research by Y. Kim et al. (2018) will be used. Because this is the starting point, this paper will be referred to multiple times throughout this research. They have written their recipe and process with the most detail and have published video's that show their proof of principle.

In this chapter, an overview of the recipe and used devices of the MIT research will be shown. As well as some initial changes that were made from their recipe from the start of the project as a result of tot having access to all the devices and ingredients that they used.

3.1.1 MIT recipe

In the fabrication process of the magnetic soft material in the study of Y. Kim et al. (2018), they first make an ink which is later printed in specific shapes, under a magnetic field, with the use of a 3D printer. The ingredients and devices they have used can be found in figure 3.1.

The ink that Y. Kim et al. (2018) developed mainly exists out of three main ingredients: silicone, magnetic powder and fumed silica. The recipe of how MIT made their ink can be found in the blue box.

The silicone of their ink exists out of Ecoflex 00-30 Part B, SE 1700 base and SE 1700 catalyst. The SE 1700 parts together already form a silicone, but the Ecoflex 00-30 was added to change the material properties. The second component that was added, was the fumed silica. This was added to adjust the rheological properties of the ink so that it could be used for direct ink writing. At last, the NdFeB powder was added. These are the particles that will be magnetized and give the material its magnetic response. As an extra ingredient Ignite PMS 805C was added. This does not influence the performance of the material but was added for imaging purposes of the material.

Al the ingredients in the recipe were during the ink preparation mixed by a planetary mixer. Before the SE 1700 catalyst was added to cure the ink, the magnetic particles in the ink were magnetized by an impulse magnetizer. For this, a magnetic field with the strength of 2,7T was used.

In the study by Y. Kim et al. (2018), they also used a support ink to support printed structures that existed out of multiple layers. The microplate shaker was later used to remove the support ink from the magnetic soft material structure.

Finally, for the 3D printing of the magnetic soft material, a 3D printer is used with an electromagnet and magnetic shield attached to the nozzle. This electromagnet will create the magnetic field, under which the magnetic particles in the ink will align during the printing. The magnetic shield is used to protect the already printed structures of the ink from the electromagnet that is moving above them. Through the magnetic shield, the orientation of the printed magnetic particles will not be disoriented. (Y. Kim et al., 2018)

Preparation magnetic ink

- First, the SE 1700 base and the Ecoflex part B were blended together
- Then the fumed silica was added and together they were mixed in a planetary mixer for 2 min at 2.000 rpm
- The NdFeB particles were added and mixed in the planetary mixer for 3 min at 2.000 rpm
- The mixture was defoamed at 2.200 rpm for 1 min
- The composite ink was magnetized by impulse magnetic fields (about 2,7 T) generated by an impulse magnetizer
- Then the SE 1700 catalyst was added and mixed at 2.000 rpm 30 sec before printing
- For imaging purposes, fluorescent colourants can be added

Preparation support ink

- Mixing Elastosil with the fumed silica nanoparticles
- The mixture was defoamed at 2,200 rpm for 1 min

Printing procedure

- The inks were mounted on the custom-designed 3D printer based on a Cartesian gantry system (AGS1000, Aerotech)
- Conical nozzle inner diameter 410 µm (Smoothflow Tapered Tipp, Nordson EFD) were used to print both inks
- An electromagnet with a field strength of 50 mT was added at the tip of the nozzle

(Y. Kim et al., 2018)

	wt%	**Material**	**Supplier**	**Size**	**Extra information**
Magnetic ink	21,78	Ecoflex 00-30 Part B	Smooth-on Inc.		
	2,72	Fumed silica nanoparticles	US Research Nanomaterials Inc.	20-30 nm	
	11,71	SE 1700 base	Dow Corning Corp.		
	1,17	SE 1700 catalyst	Dow Corning Corp.		
	62,62	NdFeB microparticles	Magnequench	5 µm	MQFP-B-2007609-089
total	100				
Image colouring	2	Ignite PMS 805C	Smooth-on Inc.		
	mass ratio				
Support ink	5,45	Elastosil CAT PT-F	Wacker		
	1	Fumed silica nanoparticles	US Research Nanomaterials Inc.	20-30 nm	Amorphous
Support ink remover		Isopropyl alcohol			
Used devices		Planetary mixer	Thinky		AR-100
		Impulse magnetizer	ASC Scientific	About 2.7 T	IM-10-30
	2	Syringe barrels			
	2	Conical nozzles	Nordson EFD	Inner diameter 410 µm	Smoothflow Tapered Tip
		Permanent magnet or electro magnet		50 mT	
		Magnetic shield			
		Micro plate shaker	VWR		To remove the support ink

Figure 3.1: *The ingredients and devices used to make magnetic soft material in the study by Y. Kim et al. (2018)*

3.1.2 Alterations

From the start of this project, some alterations were made to the recipe and procedure, developed by Y. Kim et al. (2018). This was due to not having access to all the devices and ingredients that they used.

The support ink will not be a part of this research. This is due to the time restraints of this project, as well as the initial focus of the project being on developing the magnetic soft material ink and the 3D printer system.

For the silicone in this project Ecoflex 00-10 is used (part A and B) instead of the SE 1700. This is because the SE 1700 was not available in this region.

Furthermore, during the preparation, the ingredients will be mixed by hand. This is because there is no planetary mixer at the applied labs at IDE. A planetary mixer could also not be found at another faculty. For the degassing of the ink, a vacuum oven was used.

During many of the first ink development tests, Iron Carbonyl powder (Fe) was used instead of the NdFeB powder. This is because of the expense and delivery time of the NdFeB powder. In experiments where a lot of the first exploration was done or large amounts of ink were used for the first tests, in which the magnetization of the ink was not an important factor, the NdFeB powder was replaced by Iron Carbonyl powder.

3.2 Methods

This chapter will explain the main method that was used to set up the multiple tests for the ink development and the 3D printer set-up. As well as explaining the methodologies used for measuring material properties.

3.2.1 Parametric study

The main method behind the experimental design of the tests and experiments is factorial design. This means that at every experiment only one parameter is changed. This way the difference observed between the tests can be described to the one parameter that was changed.

Because this is the first time that magnetic soft material is made at the faculty of Industrial Design Engineering, there is not a lot known about the influence of the different parameters. Changing one parameter at a time is also a method that helps to understand the individual parameters influence on the performance of magnetic soft material.

During the reviewing of papers about magnetic soft materials and doing multiple experiments during the development of the magnetic soft material ink and the 3D printer setup, a designers taxonomy was made with all the found factors influencing the magnetic soft material. These factors have been sorted into four domains, material composition, process, material properties and design (see figure 3.2).

3.2.2 Measurement methods

To measure the influence of the changed factors during the experiments, multiple tests methods were used, of which the procedures will be explained in this section.

At the start of this project, unfortunately, not a lot of these methods were thought of or used yet. To compare the results, the samples were simply compared by the use of observation.

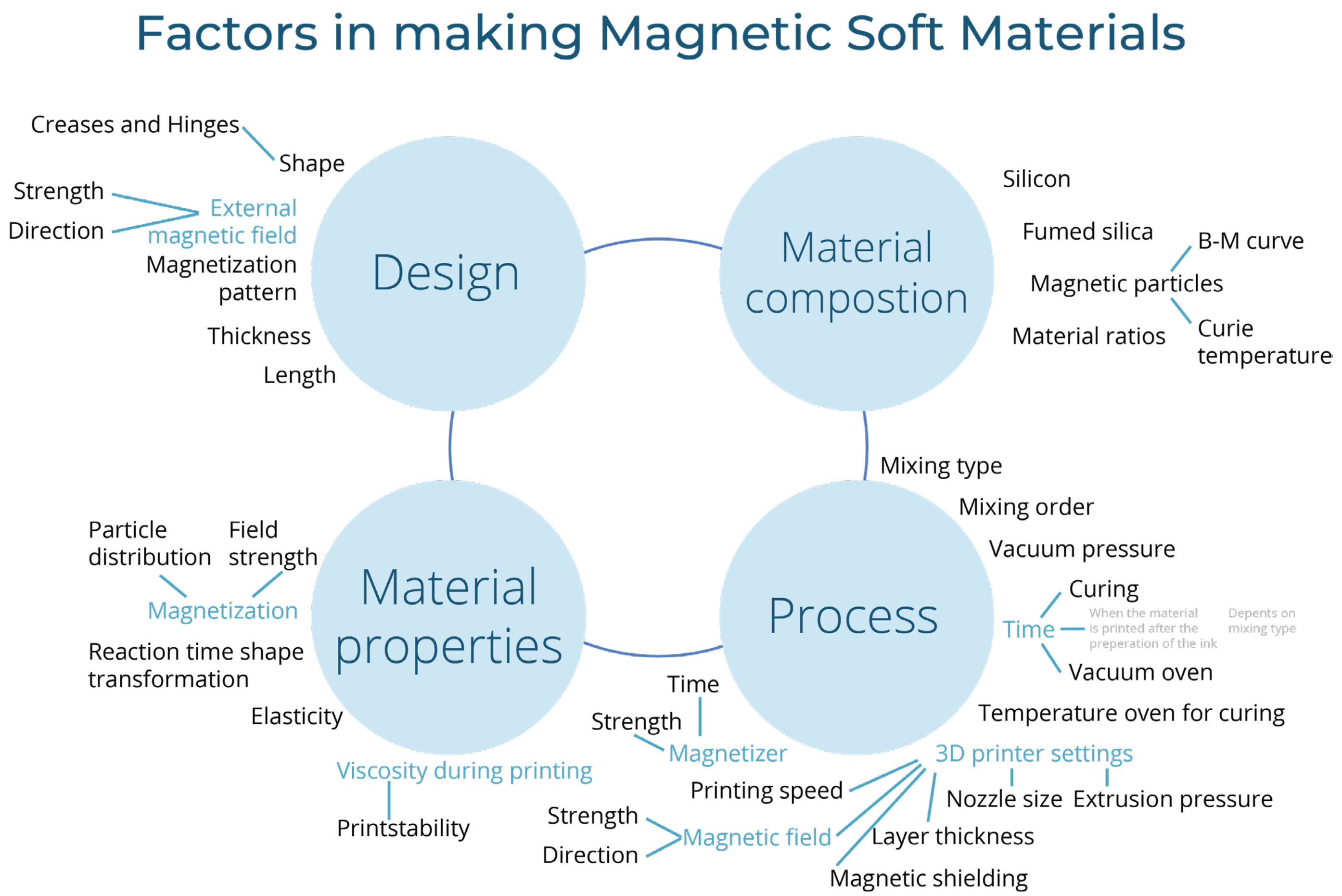

Figure 3.2: *The magnetic soft material designers taxonomy. Magnetic soft material factors that (can) influence the performance of the magnetic soft material.*

3.2.2.1 Viscosity

The viscosity measurement method was based on the method used by Y. Kim et al. (2018). By using the same method the results could be easily compared the those of Y. Kim et al. (2018). Therefore, the graphs are also plotted with a logarithmic scale.

The viscosity measurement procedure used in this graduation project (see Appendix C for the program settings):

- 40-mm-diameter steel plate geometry
- Steady-state flow experiments with a sweep of shear rates (0,0-100 s-1)
- The inks were brought to a temperature of 25°C for one minute before the experiment started
- The gap height between the geometry and the plate always was 0,5 mm

The only difference between this procedure and the procedure of the MIT research is the geometry of the steel plate that was used. MIT used a 20-mm-diameter steel plate geometry and during this research, a 40-mm-diameter steel plate geometry was used.

For the viscosity measurements, the Texas Instruments AR-G2 rheometer was used.

When the viscosity of a mixed ink was measured (all the ink ingredients mixed), the viscosity of the ink was measured directly after mixing, which took around 3 to 4 minutes. This was done to eliminate the curing effect of the measurement on the in and get all the mixed ink in the same curing condition when being measured.

3.2.2.2 Extrusion force through the static mixing nozzle

The force needed to extrude the magnetic soft material ink through the static mixing nozzle was measured. This was needed to get an indication of the force the 3D printer should be able to exert on the syringes. What a static mixing nozzle is and its purpose will be explained in chapter 3.4.1.

To measure the force that was needed to extrude the ink through the static mixing nozzle, a scale was used that was able to measure up to 24 kg.

For the measurement, two 10 mL syringes should be filled with the same amount of ink, each with a different ink component. The pistons will together stand flat on a surface and there can be pressed on the syringes with an even force when the syringes are filled with the same amount. This is also needed to extrude the ink at the right ratio which gives the best representation of the extrusion force because the viscosities of ink components A and B have a small difference. The two syringes should be inserted in the static mixing nozzle which has changed connections fit for the syringes.

Then, to perform the measurement, the pistons of the two syringes should be put on the scale. So that the static mixing nozzle is turned upwards (see figure 3.3). While one person is pressing on the static mixing nozzle to move the pistons in the syringes and extrude the ink through the static missing nozzle, another person should film the display of the scale. The pressing on the static mixing nozzle could be strenuous depending on the viscosity of the ink and it is normal if this person needs to take a break every now and then. The extrusion of the ink should be measured until the ink comes out of the static mixing nozzle.

The filmed values of the scale should be analysed so that the highest force reached could be taken out of the measurements. This value times 10 m/2 indicates the maximum force needed during the extrusion.

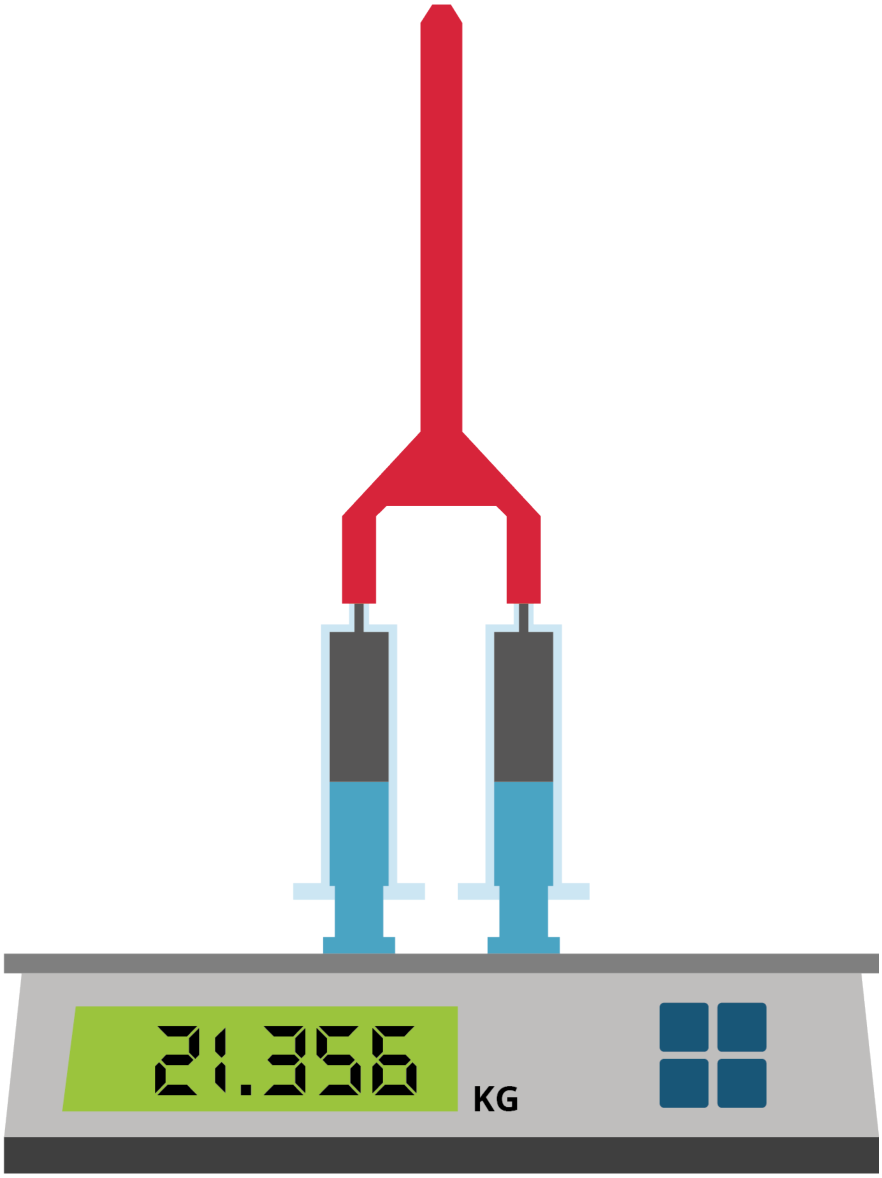

Figure 3.3: *The setup to measure the force needed to extrude the ink through the static mixing nozzle*

3.3 Ink development

This chapter will focus on the development of the ink. It will discuss the parameters that have been studied during the ink development. Separate documentation of all the tests can be read in Appendix B. The ink development is almost presented in every test.

3.3.1 Ink names and recipes

In figure 3.4 the names and compositions of the most mentioned and measured inks are presented. The numbers in the table are based upon the weight percentages of the MIT recipe. These are also placed in the table.

Throughout the project the amount of silicone and magnetic particles have been kept the same as MIT, to gain the same type of magnetic response when the particles are magnetized. The difference in weight percentage with the NdFeB particles depends on the difference in density with the Iron carbonyl powder. These are calculated in the experiments in Appendix B.

The names of "In Appendix B" are the names of the ink recipes as they are mentioned throughout Appendix B. Appendix B contains all the documentation of the tests that were done during this project. T3E1, for example, comes from Ink Development 3 experiment 1.

The names of the inks are based on their composition:

NdFeB12,5%SiO_nM

The type of magenetic particles used

The percentage of fumed silica based on the MIT value

If the ink is magnetized (M) or non magnetized (nM)

	MIT	NdFeB12,5%SiO_nM	NdFeB12,5%SiO_M	Fe25%SiO_nM
Particle type	NdFeB	NdFeB	NdFeB	Iron carbonyl
Total wt% Silicone (Ecoflex)	34,66	34,66	34,66	34,66
Wt% magnetic particles	62,62	62,62 --> 60,5	62,62 --> 60,5	62,62
Wt% fumed silica (SiO)	2,72	0,33	0,33	0,66
Total wt%	100	95,5	95,5	97,94
Magnetized		No	Yes	No
In Appendix B		T8E1 nM	T8E1 M	T6E3 IC

	NdFeB25%SiO_nM	Fe31,25%SiO_nM	Fe37,5%SiO_nM	Fe50%SiO_nM
Particle type	NdFeB	Iron carbonyl	Iron carbonyl	Iron carbonyl
Total wt% Silicone (Ecoflex)	34,66	34,66	34,66	34,66
Wt% magnetic particles	62,62 --> 60,5	62,62	62,62	62,62
Wt% fumed silica (SiO)	0,66	0,83	1	1,33
Total wt%	95,9	98,11	98,28	98,61
Magnetized	No	No	No	No
In Appendix B	T6E3 NdFeB	T7E1	T6E4	T3E1

Figure 3.4: *Ink names and their composition*

3.3.2 Mixing orders

The original mixing order of the MIT recipe has changed a lot in this project. This was mainly due to the different silicone ingredients and ratio's that were used (see chapter 3.3.3). In this project two different mixing orders were developed to fulfil different needs, depending on when the material preferably had to cure.

MIT's mixing order can be found in chapter 3.1.1. As a summary, they first mixed a large number of silicone parts together. After that, first, the fumed silica particles were mixed in and then the NdFeB particles. When the main ink was done, the ink got magnetized. Just before printing the catalyst for the silicone was mixed in so that the ink would cure during the printing. (Y. Kim et al., 2018)

In chapter 3.3.3 it is explained why Ecoflex parts A and B were used in a 1 to 1 ratio from ink development 2 forwards, in this project. There it was also explained that we started with an Ecoflex ratio of A:B = 1:1,7.

At MIT everything was mixed and defoamed with a planetary mixer. This type of mixer was not available at the faculty during this project. Therefore everything was mixed by hand. If this would be good enough was checked by looking at a cured ink sample under a microscope and to see if the particles were distributed evenly (see figure 3.6).

Mixing order for immediate curing
This mixing order (see blue box) for immediate curing was created first and was mostly based on MIT's mixing order. It was used for observing the cured material and experiments where no 3D printer was involved.

In ink development 1 (see Appendix B.a.1) there was first started according to MIT's recipe. The fumed silica was mixed in part B of the Ecoflex. Due to the different Ecoflex ratio that was used, there was less silicone to dissolve the fumed silica in. Which made it hard to mix the ink. The experiment was stopped.

During the second experiment in ink development 1, there first was started by mixing the magnetic particles with Ecoflex part B, because it had less volume than the fumed silica. This worked. However, the ink volume was not that large and the same as before. Then it did not work to mix the fumed silica in the volume of Ecoflex part B. Due to this Ecoflex part A was added to increase the silicone volume. After that, the fumed silica was added. This mixing order cured and worked well to evenly distribute the particles in the ink and was therefore used during the project.

Mixing order for 2 component ink used for the static mixing nozzle and able to store without curing
The previous mixing order was for when the ink had to cure immediately after mixing. To prepare ink for the 3D printing this was tot preferable. If the ink would cure over time during the printing, this might change the ink's properties and the print process would not be constant. This is also pointed out in the study of Zhou et al. (2019) (see literature review chapter 2.4.2.3). It also was not practical to get the ink magnetized. This happened in the reactor institute away from the labs where these experiments were conducted. Before the ink was magnetized the magnetic particles had to be mixed in the silicone because it was assumed that it would be hard to separate the magnetic particles from each other when they were magnetized and mix to be mixed into the silicone. The time it takes to prepare the ink, go to the reactor institute and preparing the printer would take too much time and already start the curing process of the magnetic soft material.

Mixing order to prepare ink that can cure immediately

- Add the Ecoflex part B and the magnetic particles together
- Mix by hand
- Add the Ecoflex part A
- Mix by hand
- Add the fumed silica
- Mix by hand

Mixing order to prepare the ink in two components mostly used for the static mixing nozzle

- Ecoflex part A and Ecoflex part B should be poured into different cups
- Measure the half amount of the iron carbonyl particles and add this to the cup of Ecoflex part A
- Measure the other half of the iron carbonyl particles and add these to a cup of Ecoflex part B
- Mix the Ecoflex part A and the iron carbonyl particles manually
- Mix the Ecoflex part B and the iron carbonyl particles manually
- Measure the half amount of the fumed silica and add this to the cup of Ecoflex part A
- Measure the other half of the fumed silica and add this to the cup of Ecoflex part B
- Mix the contents in the cup of Ecoflex part A manually
- Mix the contents in the cup of Ecoflex part B manually
- At the end of this step, there should be two cups with a mixture of Ecoflex part A or B with half of the amount of the fumed silica and the iron carbonyl particles.

To make the ink so that it could be prepared, magnetized and stored in the 3D printer without curing, was done by dividing the number of particles in two and mixing one half in Ecoflex part A and the other in Ecoflex part B (see blue box). Because the two components of the silicone were not mixed, the curing of the ink would not start and the ink could be stored. To cure the ink, the two ink components had to be mixed this was done with the use of a static mixing nozzle. More information about the static mixing nozzle can be found in chapter 3.4.1.

This mixing order was first used in static mixing nozzle test 1 (see Appendix B.b.1). In static mixing nozzle test 2 (see Appendix B.b.2), this mixing order was tested against a control group of the first developed mixing order (see figure 3.5). No significant difference was observed, so the mixing order was kept.

Figure 3.5: (left) cured ink that is mixed by the static mixing nozzle. (right) cured ink that is mixed by hand according to the first mixing order presented in this chapter.

Magnetized particles

Most of the experiments were done without magnetized particles due to not having a magnetizer in the Applied Labs. In magnetizer test 2 (see Appendix B.c.2), two magnetized ink components were mixed and checked under the microscope, if the magnetic particles had agglomerated (see figure 3.6). From this picture, it could not be concluded if the particles had agglomerated or not on a micro-level. However, it can be seen that the particles are still dispersed throughout the ink.

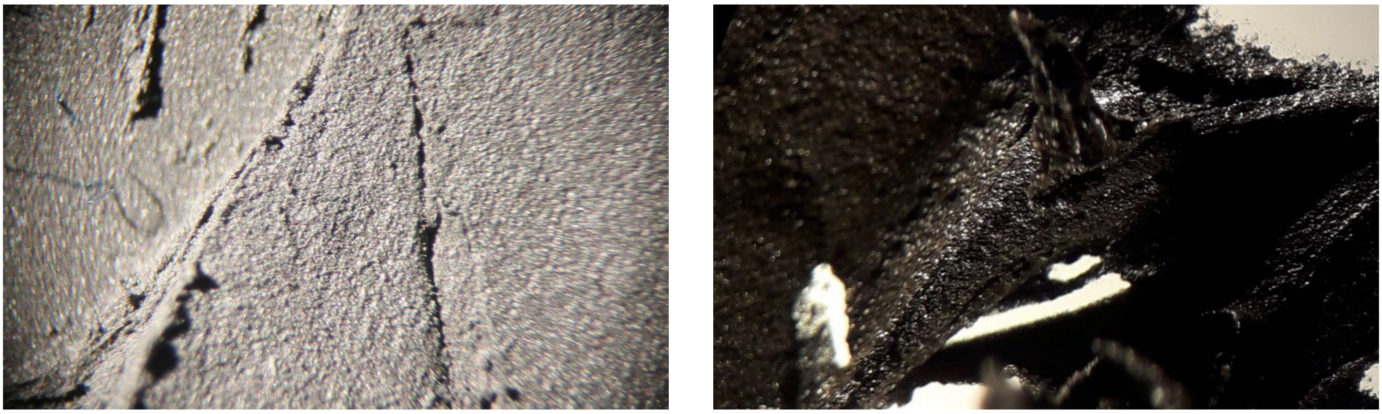

Figure 3.6: (left) Evenly distributed magnetic particles in Ecoflex. Mixed by hand. Non-magnetized ink. (right) Magnetized mixed ink. The picture is darker because another camera was used that was not compatible with the microscope.

3.3.3 Ecoflex ratio

For the Ecoflex ratio in the final recipes, the mixing ratio of 1:1 was used. This is the same ratio between the Ecoflex' A and B components as described by Ecoflex (Smooth-On, n.d.-a).

Ecoflex is the silicone of the magnetic soft material, that is used in the recipe. The silicone is the body of the material that keeps all the magnetic particles together and locks them in place. Other properties of silicone are that it gives flexibility and elasticity to the magnetic soft material. The elasticity is used to bring the magnetically transformed shape of the material back to its initial printed shape when the magnetic field is released, which is the shape of the silicone.

The 1:1 ratio was not used at the start of this project. The MIT research used three components for their silicone:

- SE 1700 base
- SE 1700 catalyst
- Ecoflex 00-30

MIT mixed different types of silicon to get the right rheological properties (Y. Kim et al., 2018). The SE 1700 could not be bought, so it was decided to only use Ecoflex in this project.

The initial weight percentages of the SE 1700 base and catalyst were added together and were replaced by Ecoflex part A in the first experiments. The Ecoflex was also changed from the 00-30 to the 00-10 type, which was available at the start of the experiments. The viscosity of the 00-10 (14000 cps) (Smooth-On, n.d.-a) is higher than the 00-30 (3000 cps) (Smooth-On, n.d.-b). The SE 1700's viscosity is much higher (542000 cp) (Dow, n.d.).

21,78 wt% Ecoflex 00-30 part B → **21,78 wt% Ecoflex 00-10 part B**

11,71 wt% SE 1700 base
1,17 wt% SE 1700 catalyst → **12,88 wt% Ecoflex 00-10 part A**

During this project, only Ecolex 00-10 was used. No other Ecoflex options or silicones were experimented with.

In ink experiment 1 (see Appendix B.a.1) this ratio from MIT was used B>A
In ink experiment 2 (see Appendix B.a.2) the A:B = 1:1 ratio was used
In ink experiment 3 (see Appendix B.a.3) the A>B ratio was tested

One of the reasons why there was chosen to continue with the 1:1 ratio, was that it was easier to mix and stir the recipe (see ink experiment two in Appendix B.a.2) than the converted MIT ratio and the texture of the ink was more smooth (see figures 3.7 and 3.8). This was due to a lower viscosity of the ink. When the Ecoflex ratio was altered to a higher percentage of Ecoflex part A in comparison to Ecoflex part B, the viscosity also became higher

Figure 3.7: *The grainy texture of the more viscous ink with the altered MIT ratio (B>A) from experiments 1 and 2*

Figure 3.8: *The smoother, less viscous ink with the 1:1 ratio from experiment 2*

and made the ink hard to mix. This was tested with two ratios 2A:1B (see figure 3.9) and 4A:1B. The latter, for some reason, mixed better than the 2A:1B ratio. This could have been due to an alteration in the mixing order. In the 2A:1B ratio, the iron carbonyl powder was dissolved in the Ecoflex part B and the 4A:1B in part A. This was done because in the 2A:1B there were almost too many particles to get dissolved in the Ecoflex component. From later in the research when the viscosity was measured, it was also found out that the viscosity of the Ecoflex part A was higher than Ecoflex part B (see figure 3.10). Which could also have increased the viscosity in these ratios. Due to the lower viscosity and the smoother, better to extrude inks from the 1:1 ratio there was chosen to use the 1A:1B ratio.

With all the mixing ratios that were made during these experiments, the mixed ratio's did cure.

Figure 3.9: *The ink with the 2A:1B ratio from experiment 3 which was too viscous to extrude through a 10 ml syringe*

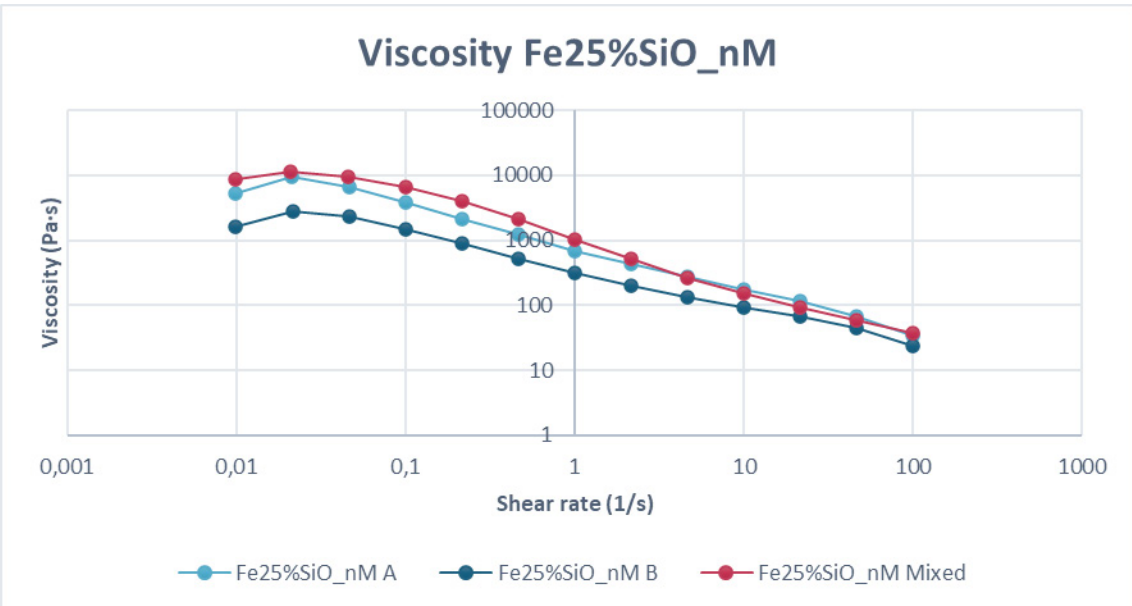

Figure 3.10: *The viscosity of the Fe25%SiO_nm ink from the sixth try of making ink recipes. There can be seen that the viscosity of component A is higher than that of component B. In this recipe, mixing order 2 is used.*

3.3.4 Fumed Silica

Fumed silica is the only ingredient in the recipe that in theory is not required to make a functional magnetic soft material. However, it is important to 3D print with silicones. In the recipes and experiments during this project, fumed silica was used to change the viscosity of the magnetic soft material and give it shear-thinning properties. That way the ink could be tuned to have a low enough viscosity to be extruded through the 3D printer, but also be viscous enough to have a good print stability. The optimal amount for fumed silica in the recipe has not been found yet.

Fumed silica was added to the MIT recipe to "achieve rheological properties for direct ink writing" (Y. Kim et al., 2018). These rheological properties are explained in the study by Zhou et al. (2019), as shear-thinning properties. This means that the viscosity of the ink is decreased when the ink is under pressure (during printing). However, after it is printed, the fumed silica will build up bonds so that the ink has enough print stability (see literature review chapter 2.4.2.3, direct ink writing of silicones figure 2.18).

Fumed silica is white and looks like snow (see figure 3.11). It is very lightweight and when it is brought from one cup to another, some of the fumed silica seemed to float. Due to this there was sometimes wondered if the scale was able to measure the exact amount of fumed silica that was added to the recipe.

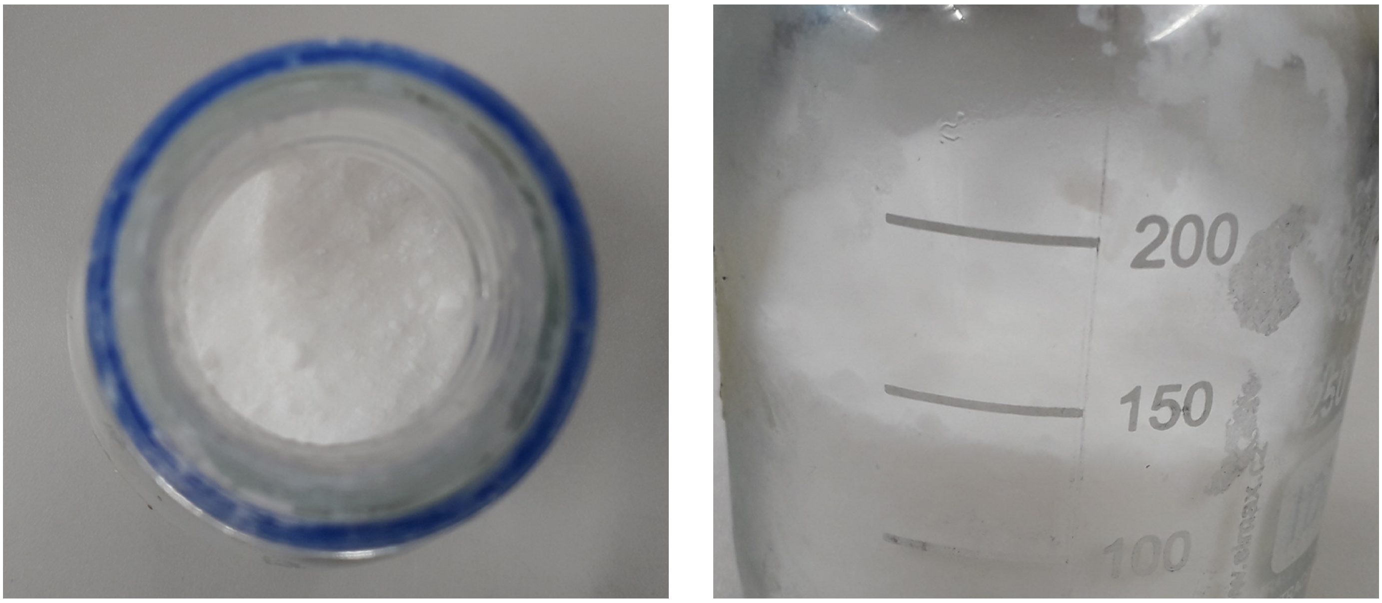

Figure 3.11: *The fumed silica that was used during this project*

The recipe of MIT had an amount of 2,72 wt% fumed silica. This decreased significantly during the experiments in this project.

In ink development 1 (see Appendix B.a.1) we used the full amount of fumed silica according to the MIT recipe and noticed that it was very lightweight. Due to that, a large volume of fumed silica had to be added. This was sometimes challenging for the volume of Ecoflex in which the fumed silica was to be dissolved.

In ink development 3 (see Appendix B.a.3) a 50 wt% and 200 wt% MIT amount fumed silica was tested in the recipe. It was observed that the more fumed silica was added the more viscous the ink became. The inks viscosity also affected the flexibility and elasticity of the cured material. These properties both increased when the amount of fumed silica was decreased.

In ink development 6 (see Appendix B.a.6) and in static mixing nozzle test 4 (see Appendix B.b.4), different amounts of fumed silica were added to the ink, to purposefully make inks with different viscosities. These viscosities have been measured with the rheometer and the amount of added fumed silica here really showed a difference in viscosity. The more fumed silica was added the more viscous the ink became (see figure 3.12 and 3.43). It was decided to only change the fumed silica amount to tune the viscosity so that the wt% of the magnetic particles stayed the same as in the MIT recipe. That way a similar magnetic soft material performance was expected.

From this experiment was also found that 50% to 37,5% of MIT's fumed silica amount gave a good print stability.

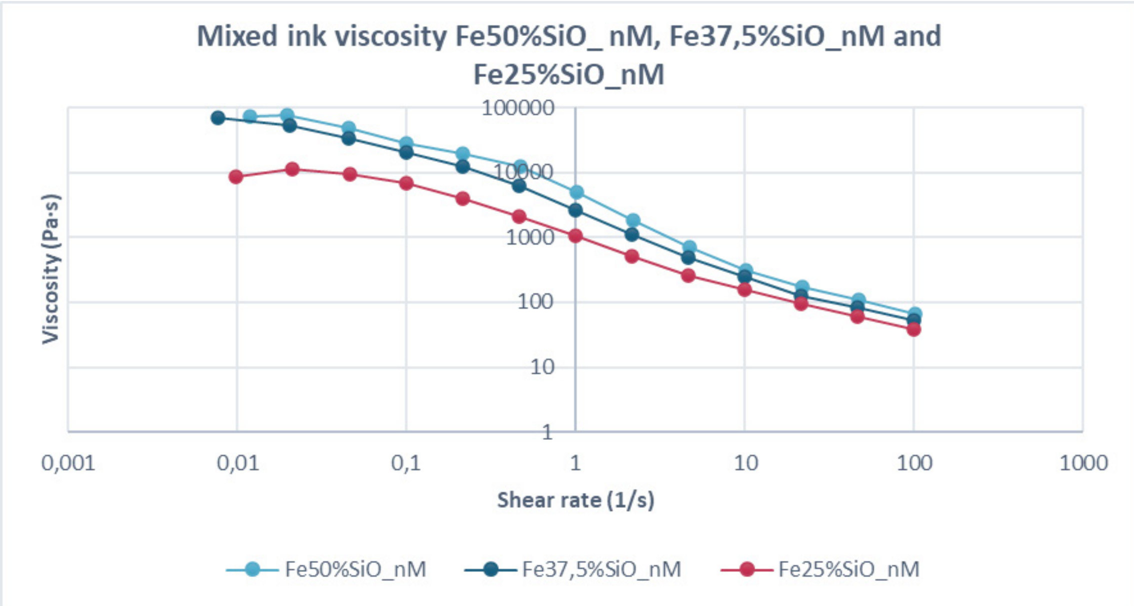

Figure 3.12: *The viscosity of inks Fe50%SiO_nM, Fe37,5%SiO_nM and Fe25%SiO_nM made in static mixing nozzle test 4 with different amounts of fumed silica relative to the MIT recipe. The viscosity of the ink increases if more fumed silica is added.*

3.3.5 Magnetic particles

Magnetic particles make the shape transformations of the magnetic soft material possible. NdFeB particles are the best to use to make magnetic soft materials, which are the same as in the MIT recipe (Y. Kim et al., 2018).

The magnetic particles in the magnetic soft material are the ingredient in the ink where the shape transformations are programmed. This is done by laying their magnetic poles in specific directions. When the magnetic particles are mixed in the ink, the particles are not magnetized. So the non-magnetized particles can be mixed easier, without them attracting each other by their magnetic fields.

Different tested particles

Before there was chosen to use NdFeB particles, different magnetic particles have been tested. These were iron carbonyl powder (Fe), Fe3O4, SmCo and NdFeB. NdFeB particles were tested because MIT used them in their recipe (Y. Kim et al., 2018). Iron carbonyl powder was available at the university and Fe3O4 and SmCo were tested because they were listed as strong magnets (FIRST4MAGNETS, n.d.). Iron carbonyl particles have been used the most throughout the tests. This was because the iron carbonyl powder was less expensive than the NdFeB particles and did not contain rare earth metals (Granta Design Limited, 2019). This could also be done because most of the experiments in this project have been done without magnetizing the magnetic particles.

Influence of non-magnetized particles on the ink

In ink development 4 (see Appendix B.a.4) the influence of the non-magnetized magnetic particles on the ink was tested. The more particles were added the more viscous the ink became. It was also found out that when the type of particles in a recipe should be changed to another particle type, this should be done by vol% instead of by wt%. More about this can be read in chapter 3.3.6 about the ink's viscosity. This also explains the changed weight percentage in the ink recipe tables in figure 3.4.

The particles also changed the inks' structure which could be observed while stirring the ink. NdFeB and iron carbonyl powder inks had a comparable structure. This also made Iron carbonyl a good replacement for NdFeB during the non-magnetized tests.

The Fe3O4 particles made the ink stickier than the NdFeB or iron carbonyl and were therefore sometimes hard to mix.

The SmCo particles always mixed in well with the Ecoflex and got dissolved very easily however when the ink was stirred by hand, the SmCo felt like grains of sand. SmCo is also a rare earth metal like NdFeB (Granta Design Limited, 2019).

Magnetization test 1

During magnetization test 1 (see Appendix B.c.1), there was chosen to use the NdFeB particles in the final recipe. For this experiment, four types of ink were made with the same vol% of magnetic particles. The cured ink samples have been magnetized at the Reactor Institute by the Versa Lab (see figure 3.13) which could create impulse magnetic fields up to 3 Tesla (Quantum Design, n.d.). The inks were magnetized one by one, by placing them in a tube that would go into the coil of the magnetizer that made the particles magnetic and give the cured ink a defined North and South pole (see figure 3.14).

Figure 3.13: *VersaLab used for magnetizing the ink*

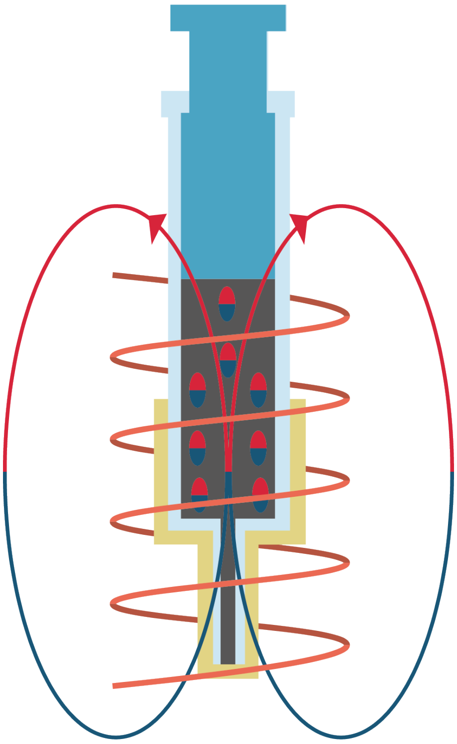

Figure 3.14: *The syringe is placed inside a coil in the magnetizer. The magnetic field of the coil magnetizes the magnetic particles and their poles become the same as that of the magnetic field.*

After their magnetization, the different inks were tested on if they had distinct North and South poles which are crucial for the working magnetic soft materials. For this, a North or South pole of a magnet was held close to an end of the cured magnetized ink to see if the cured ink would be attracted or repelled by the magnetic field.

Only the NdFeB and the SmCo had distinct North and South poles (see figures 3.15, 3.16, 3.17 and 3.18). Of which the NdFeB particles had the strongest magnetic fields. The Fe3O4 and iron carbonyl powder got attracted to the magnet, but on all sides and from a very small distance. They did not show distinct North and South poles, or magnetization.

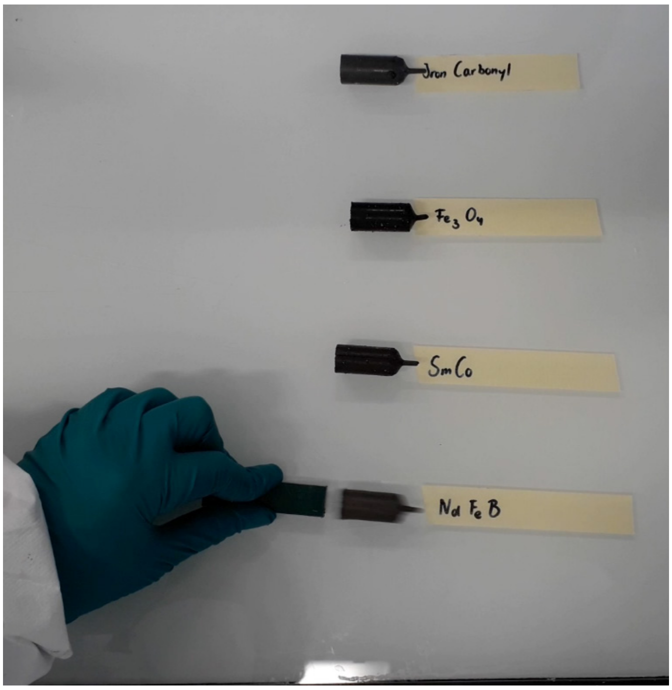

Figure 3.15: *Attracting the NdFeB sample*

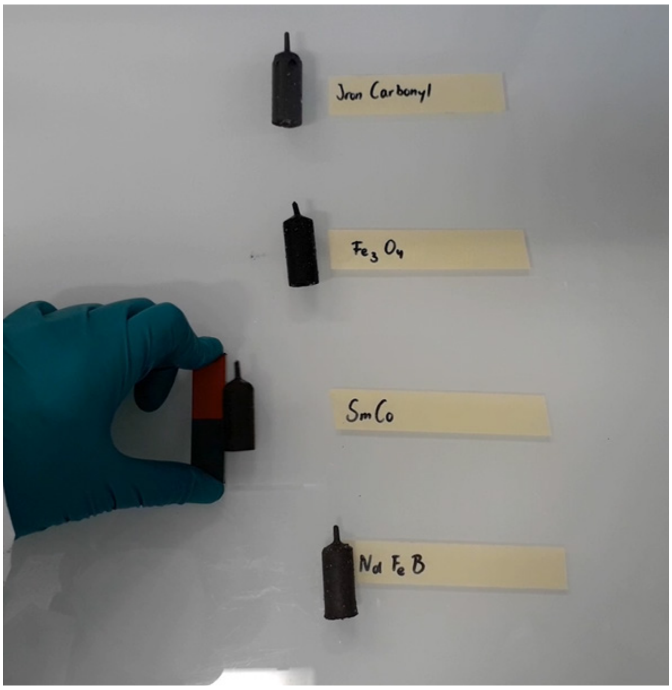

Figure 3.16: *Attracting the SmCo sample*

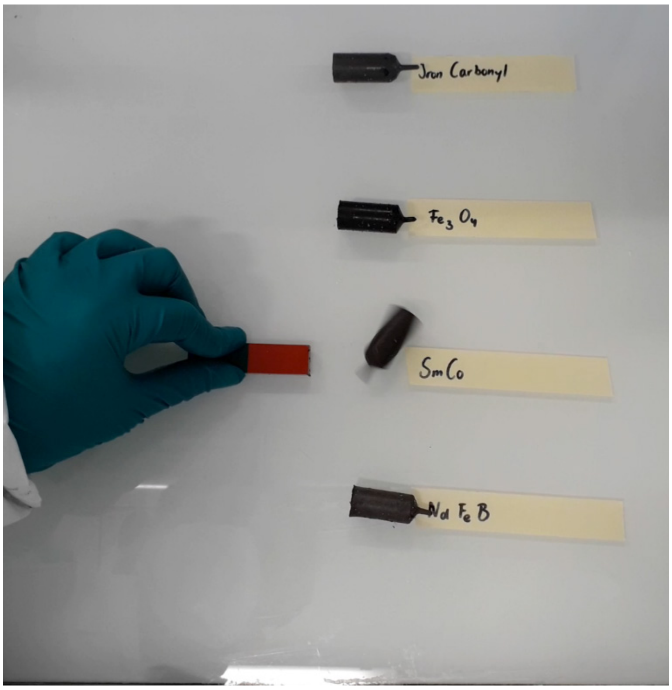

Figure 3.17: *Repelling the SmCo samples, makes them turn around so that two opposite poles can attract each other*

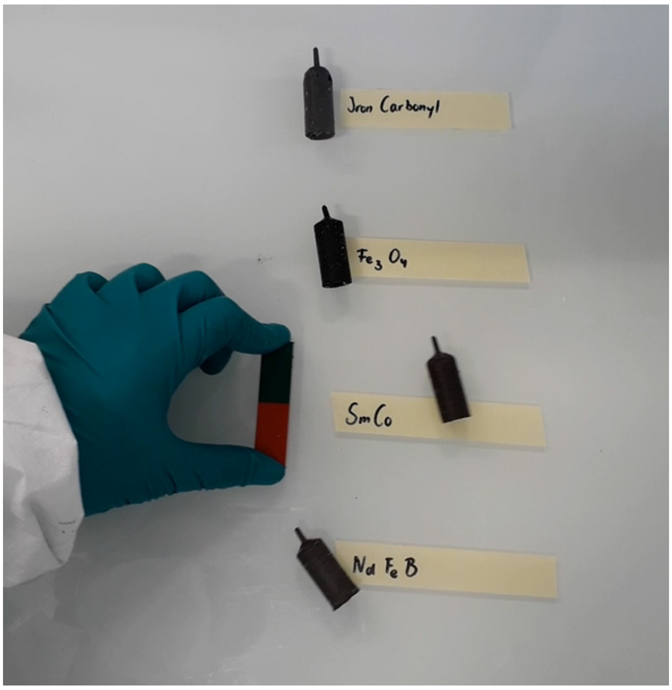

Figure 3.18: *Repelling SmCo the samples, makes them role away. The NdFeB particles have a big field strength and turn to the magnet.*

Hard and Soft Magnets

Why one type of particle can show distinct North and South poles and the other not, can be explained by hard and soft magnets. Hard magnets are permanent magnets because they keep their magnetization. Soft magnetic particles are easier to demagnetize than permanent magnets and therefore are often not permanent.

The ability for a magnet to stay magnetised is called coercivity. This is the resistance of a magnet to become demagnetized by an external magnetic field (Laughton & Warne, 2003). The coercivity is the value of the external magnetic field strength that is needed to demagnetize the magnet. On a B-H loop, this is the H-axis. The larger the coercivity of the magnet (bigger Hs gap in graphs see figure 3.19) the more resistance it has against demagnetization. Soft magnets have a low coercivity and therefore almost show no gap on a hysteresis graph between the two lines.

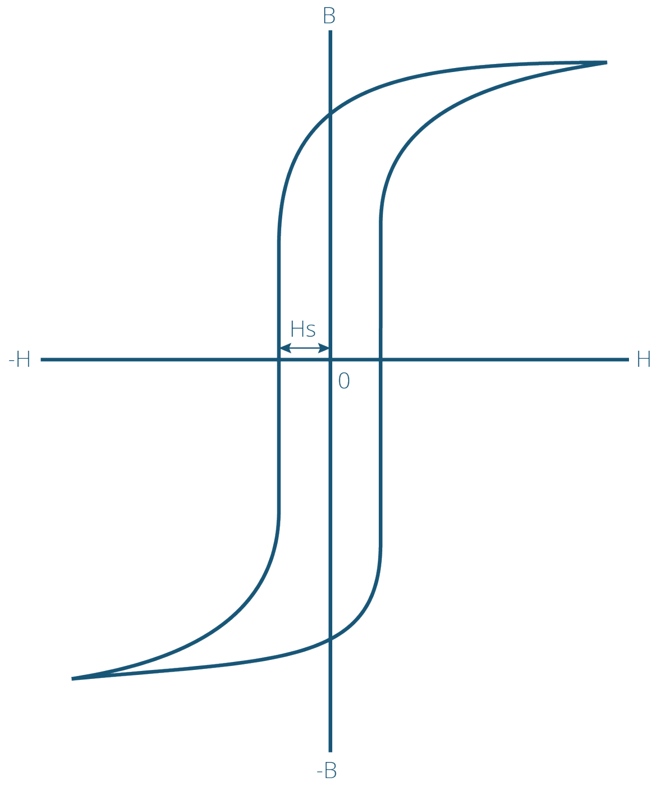

Figure 3.19: *Hysteresis curve of a magnet. The gap Hs shows the coercivity of the magnet.*

It is difficult to assign a general coercivity value to a magnet because they are dependent on their composition, grade and manufacturing technique. Of the NdFeB, SmCo and Fe3O4 different coercivity values have been found at multiple sources and are listed in figure 3.20.

The values differ a lot from each other, but the trend that can be seen is that the NdFeB particles always have the highest coercivity values and the iron oxide the lowest. The NdFeB particles are the hardest magnets because they have the highest coercivity value. Because Fe3O4 has the lowest values, these particles are the softest magnets. This could explain why the Fe3O4 and iron carbonyl powder were demagnetized after coming in contact with stronger magnets to attract them.

Reference	FIRST4MAGNETS, n.d.	Wikipedia, n.d.	Amazing Magnets, n.d.	BLS Magnet, n.d.)
Coercivity	Hc (Oesteds)	Hci (kA/m)	Hci (KOe)	HcJ (kA/m)
Particle				
NdFeB	115000	200-440	12-30	955-2387
SmCo	89700	120-240	5,5-25	800-1195
Fe_3O_4	2950	-	2,56-4,01	160-280

Figure 3.20: *Coercivity values of NdFeB, SmCo and Fe3O4 from different sources (AmazingMagnets, n.d.; BLSMagnet, n.d.; FIRST4MAGNETS, n.d.; Wikipedia, n.d.)*

Magnetized particles in uncured ink

The magnetized particles in uncured ink stayed dispersed throughout the ink. In figure 3.6 pictures can be seen after the mixing of ink with magnetized particles. It cannot be concluded if they agglomerated but they stayed divided throughout the ink.

The magnetized particles also increased the viscosity of the ink which can be read in chapter 3.3.6.

Magnetization curve

On September 2nd two cured and magnetized NdFeB37,5%SiO_M samples were measured in the Reactor Institute. One sample was made for, and magnetized during, the first magnetizer test and the other was magnetized on the same day as the measurement and made for magnetizer test 1B. The purpose was to see if the magnetic properties would change over time, but also to get an indication of the magnetic field strength. In the graph (see figure 3.21) can be seen that the most recent magnetized sample has the highest magnetization. The earlier magnetized sample has a magnetization that is 6,9% lower in comparison with the recent magnetized sample. This can indicate aging of the magnetic properties over time. However, they are two different samples that were measured, through which there might be slight differences in the number of particles or the distribution of them. For further research, it is suggested to use the same sample for the measurements.

The measurement files are transferred to the chair and mentor of this project.

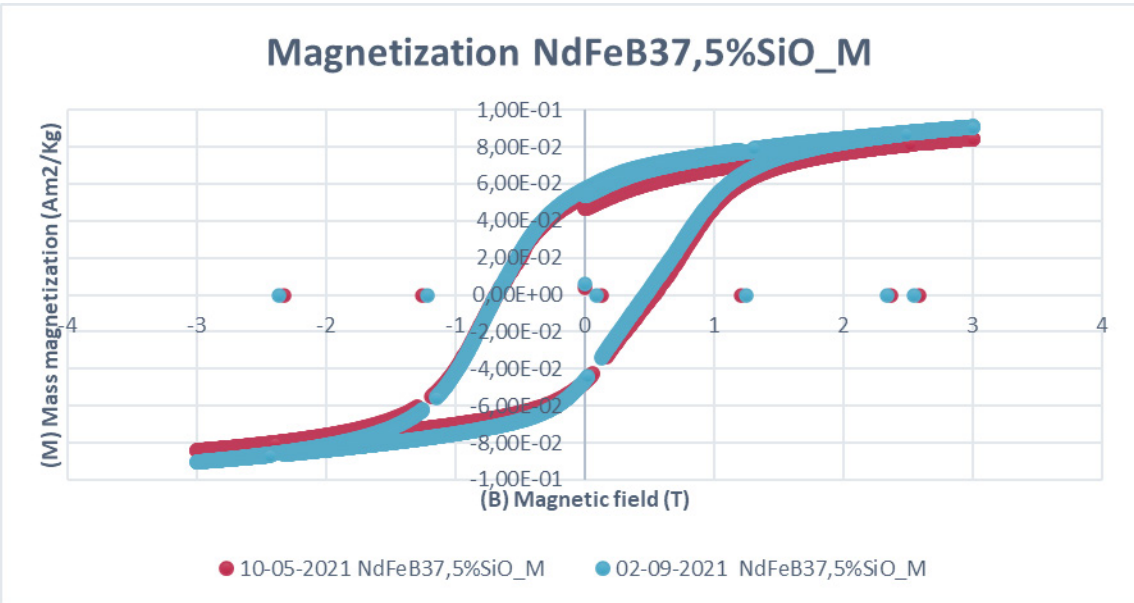

Figure 3.21: *Magnetization curve of NdFeB37,5%SiO_M. One sample was made during magnetizer test 1 and magnetized on 10-05-2021, the other sample was made during magnetizer test 1B and was magnetized on 02-09-2021. These measurements were both taken on 02-09-2021*

3.3.6 Ink viscosity

The inks viscosity is an important parameter. It influences both if the ink can be printed through the 3D printer system as well as the print stability of the printed shapes. During this project, the optimal viscosity has not been found yet. To tune the viscosity the amount of fumed silica in the recipe was changed.

The influence of viscosity on the printability of the ink

To make the ink easy to print for the 3D printer system, a low viscosity is preferred. The lower the viscosity of the ink, the less resistance is created in the system. The printer needs to have enough force to push the ink forward and through the static mixing nozzle (see chapter 3.4.1 static mixing nozzle for further explanations about the static mixing nozzle). When the ink is for example too viscous, it is not liquid enough to pass through the small chambers of the static mixing nozzle and a too high force is needed (see figure 3.22) (Static mixing nozzle tests 1 and 4 in Appendices B.b.1 to B.b.4).

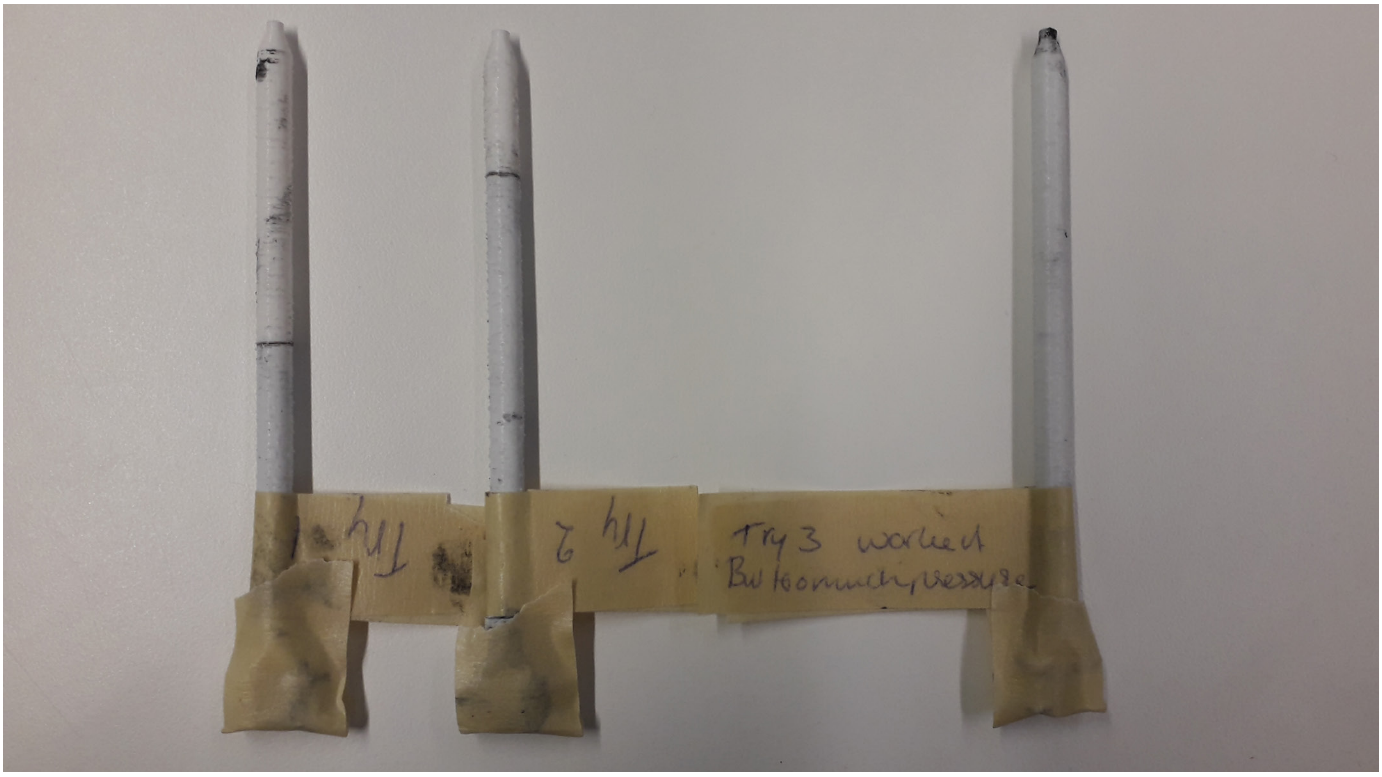

Figure 3.22: *The three static mixing nozzles from static mixing nozzle test 1 (Appendix B.b.1). The first two inks had a too high viscosity and the ink did not come completely through the static mixing nozzle. The stipes indicate to where in the nozzle the ink came. The third static mixing nozzle was used with a less viscous ink and it came through.*

A too viscous ink can also be too slow for extrusion at an end of a tube, so that it is easier for the ink to expand the tube it is in than to go forward in the tube (see figure 3.37) (3D printer experiments 5 and 7, Appendices B.d.5 and B.d.7). This was always a problem with ink component A which had a higher viscosity than ink component B. Therefore, lower viscosity inks work better for the 3D printer system. There was never a problem with ink component B.

The influence of viscosity on print stability

On the other side of the spectrum is print stability. The ability of the ink to hold the shape in which it is printed. For print stability, a high viscosity in is preferred. This is because it holds its shape without collapsing or melting in an undefinable shape of ink. A good example of this can be seen in the study by Zhou et al. (2019) (see figure 3.23). The more Nano-Silica is added, the more viscous the ink becomes and the better the ink becomes in holding its shape. It is important for the ink to holds its shape because it takes time for the silicone to cure (4 hours in the case of Ecoflex).

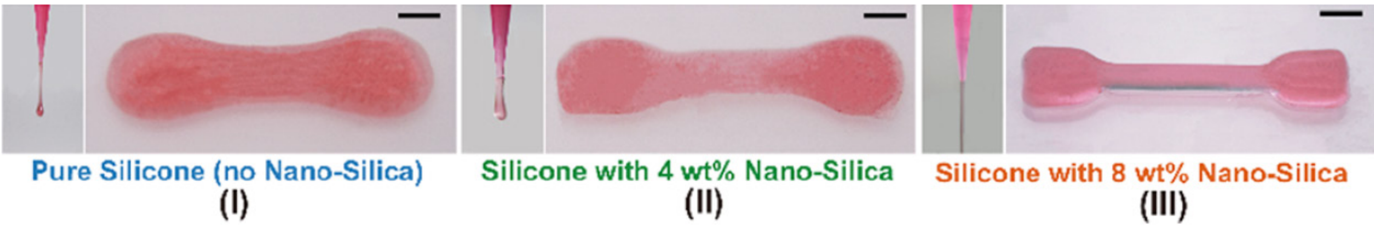

Figure 3.23: *Silicones with different amounts of Nano-Silica. The more Nano-Silica is added the better the silicone keeps its printed shape (III). In (I) the print stability is very low and the defined edges have deformed over time. (Zhou et al., 2019)*

In ink development 6 (Appendix B.a.6), the influence of the ink's viscosity, on the print stability can be seen. The higher viscous inks held their shape better (see figure 3.24). Their viscosities are measured in static mixing nozzle test 4 (Appendix B.b.4).

Figure 3.24: *Fe50%SiO_nM has a higher viscosity than Fe25%SiO_nM and is better at holding its shape. The holes present just after the ink was extruded were still present after more than 30 minutes of waiting. The Fe25%SiO_nM collapsed during the extrusion by hand (which could be a human error), and the extruded lines blended more into each other than the Fe50%SiO_nM.*

Two-component ink viscosity

The inks viscosity has been measured in multiple tests, for their freshly mixed ink, as well as the two ink components (the separate Ecoflex parts with mixed in particles) separately. In all measured viscosity data, part B has the lowest viscosity, part A is above that. This can be seen in picture 3.10, for example of if ink Fe25%SiO_ nM. T he viscosity difference between parts A and B is also observed during the experiments and is a quality of the Ecoflex itself.

Influence of the amount of fumed silica on ink viscosity

The influence of the amount of fumed silica added to the ink is that the more fumed silica is added, the more viscous the ink becomes. This can be seen in static mixing nozzle test 4 (Appendix B.b.4), where the ink with the highest amount of fumed silica has the highest viscosity and the one with the lowest amount of fumed silica has the lowest viscosity (see figure 3.12). Due to most of the experiments being done without a magnetizer and it was preferred to keep the magnetic response theoretically the same as the MIT paper, it was decided to use the fumed silica to change and adjust the viscosity of the ink. The fumed silica also gave shear-thinning properties to the ink, which can be seen in the viscosity measurements that decline under more shear stress. This is a property Ecoflex does not possess form itself (viscosity measurements Appendix C.a).

Influence of magnetic particles on ink viscosity (non-magnetized and magnetized)

The magnetic particles influence the ink's viscosity in two ways. In just adding particles to the silicone as well as internal forces created by magnetized particles that attract each other.

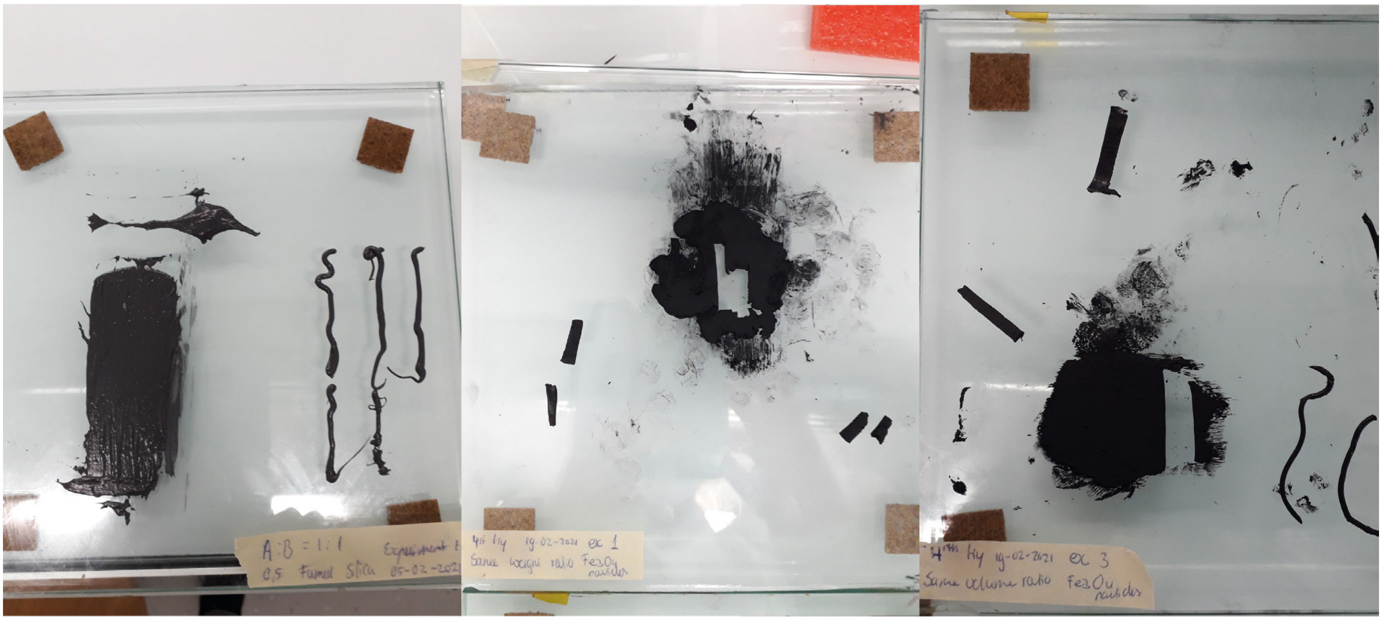

Figure 3.25: *On the left is the Fe50%SiO_nM ink with iron carbonyl particles on which the other two inks are based. In the middle is the Fe50%SiO_nM ink of which the iron carbonyl particles are converted to Fe3O4 particles by wt%. On the right is the Fe50%SiO_nM ink of which the iron carbonyl particles are converted to Fe3O4 particles by vol%.*

Non-magnetized particles

The non-magnetized particles influence the ink's viscosity in the same way as the fumed silica. When a larger number of particles is added to the silicone, the more viscous the ink becomes. It also adds shear thinning properties to the Ecoflex (viscosity measurements Appendix C.b).

In ink development 4 (Appendix B.a.4) the added amount of iron carbonyl powder was converted to other particles, Fe3O4, SmCo and NdFeB by wt% and vol%. Especially in the case of the Fe3O4 particles, a difference between the conversion in wt% and vol% was observed. This was due to the larger density difference between Fe3O4 and iron carbonyl powder. In picture 3.25 the Fe50%SiO_nM ink and the two inks that are Fe3O4 based can be seen. The figure in the middle has the magnetic particles that were converted by wt% and had too many particles to contain. The ink got a grainy structure and became too viscous to extrude through a 10 mL syringe. More added particles increased the ink viscosity.

Magnetized particles

When the ink of MIT was magnetized the viscosity increased by 65.000 Pa.s (see figure 3.26). This was due to the internal interaction of the magnetic fields of the magnetic particles. This also happened with the NdFeB12,5%SiO ink which was the only one that got magnetized. There the viscosity increased by around 54.000 Pa.s, which was in the same order of magnitude as the increase of MIT's ink (figure 3.27). This rise in viscosity is much larger than what is measured with the different fumed silica amounts. In further research, there should be seen if a different amount of fumed silica does influence the amount of viscosity increase.

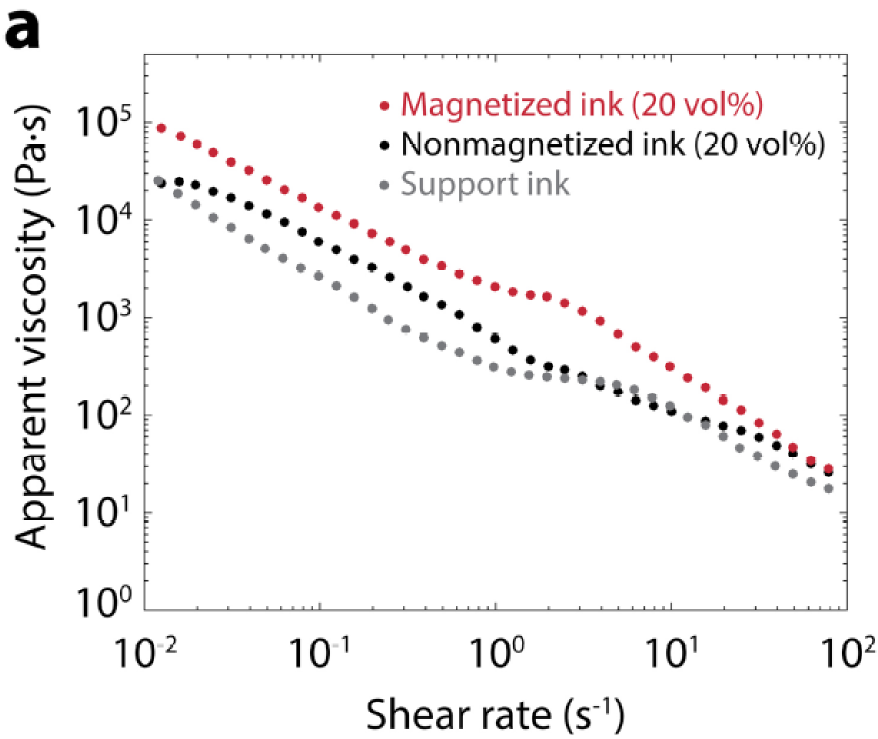

Figure 3.26: *The viscosity increase of the ink from the MIT paper (Y. Kim et al., 2018)*

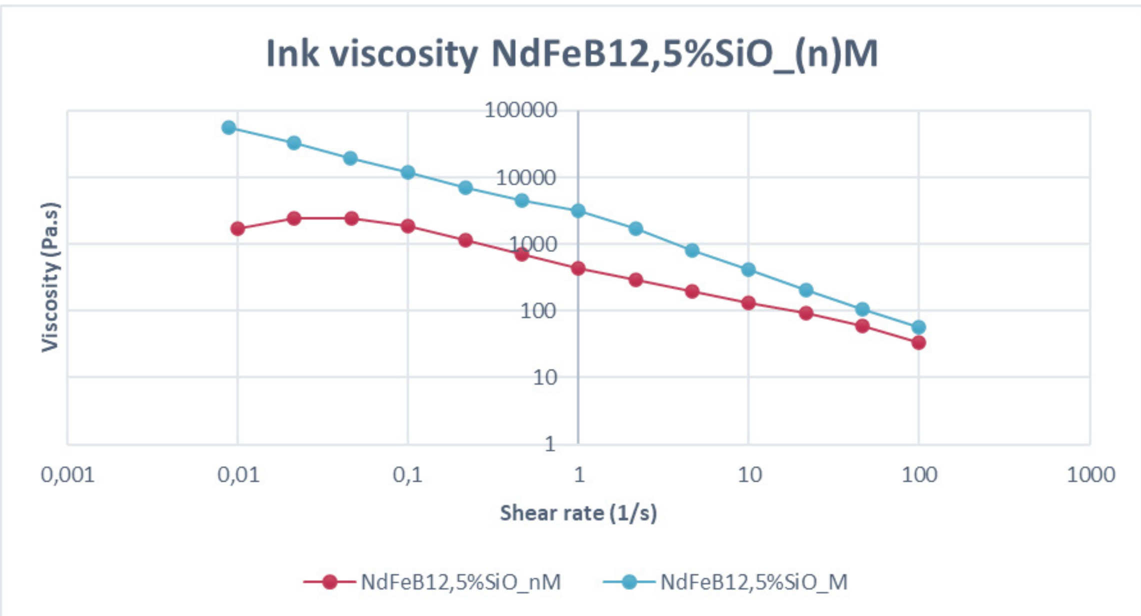

Figure 3.27: *The viscosity increase due to the magnetization of the mixed NdFeB12,5%SiO ink*

3.3.7 Curing time and heat

For the curing heat and time of the magnetic soft material, the oven settings of the MIT research were used, 120°C for 60 minutes (Y. Kim et al., 2018), to cure unmagnetized magnetic soft material. Magnetized magnetic soft material samples were cured for four hours without an oven according to the Ecoflex curing time (Smooth-On, n.d.-a).

At the start of the ink development, lower temperatures and shorter heating times were used (60°C for 20 minutes) (see ink development tests 1 and 2, appendix B.a.1 and B.a.2). In the second ink development test, there was tried to increase the curing time from 20 to 40 minutes (see appendix B.a.2). After 40 minutes the ink was not cured and was put back in the oven for an additional 30 minutes. Before the third ink development test, the MIT oven settings were found and they were used from that point forward. These settings were 120°C for 60 minutes (Y. Kim et al., 2018). The material was always cured when it came out of the oven with these settings.

What is not researched during this project, is the heat and curing time for magnetized samples, which would be able to speed up the curing process. This is because magnets could lose their magnetization when they are heated up over their Curie temperature. At that temperature, the atoms in the material get enough energy that the magnetic directions are destroyed and a magnet loses its magnetization (Morgan, 2018). The Curie temperature of the NdFeB particles that were used in this project is unknown. On the website from Supermagnete, they warn users to heat their neodymium magnets to temperatures between 80°C and 200°C (Supermagnete, n.d.-a). The 120°C of the MIT research is also within this range and therefore risky to use. The Curie temperature could not be tested within the time frame of this project but is recommended to find out and test in follow up research, to speed up the curing process of the magnetic soft material.

During this project, the curing time for the magnetized magnetic soft material ink was set to four hours without the use of an oven. Those four hours is the curing time of the Ecoflex (Smooth-On, n.d.-a).

3.4 4D printer set-up

After a basic understanding of the magnetic soft material ink and its parameters was established, the development of the 4D printer system started. This chapter goes into that development and the tests that were done. During this project, the 4D printer setup was not finished. Therefore the chapter will end with further work needed to complete the set-up.

What was needed for the 4D printer set-up were a paste printer and an electromagnet. The paste printer, because it can print with fluid inks. The electromagnet, to create a magnetic field at the tip of the nozzle, to guide the magnetic particles in the preferred magnetization patterns by aligning them before printing.

Other than the 3D printers used in the studies by Y. Kim et al. (2018) and Ma et al. (2020), there was chosen to use a dual syringe printer, instead of a single one (see figure 3.28). This was done so that the ink could have a constant quality over the print time, to address difficulty 2 from the study of Zhou et al. (2019) (also see chapter 2.4.2.3 literature review). The two ink components were put in different syringes so that the ink would not cure while it was in storage. From the syringes, during the print process, the components were guided through tubes to the static mixing nozzle. In the static mixing nozzle, the ink was mixed just before printing to always have recently mixed ink, in the same face of the silicone curing process.

This chapter will go into further detail about the static mixing nozzle, ink extrusion tests and physical optimization, and electromagnet development.

3.4.1 Static mixing nozzle

A static mixing nozzle can mix two ink components during the 3D printing process. One advantage of doing this is to keep the ink quality constant during the process. Before the decision was made to use a dual syringe printer, the ink had to be manually tested with the static mixing nozzle. This chapter will explain what a static mixing nozzle is, why it is used and important factors in combination with the ink. The experiments described in the chapter were all done manually.

Figure 3.28: Dual syringe printer extruder. The two syringes both contain another ink component based on Ecoflex parts A and B. The tubes will guide the ink to the white static mixing nozzle (hanging between the syringes) where the two components are mixed.

What is a static mixing nozzle

The static mixing nozzle is the nozzle through which the magnetic soft material ink will be printed. In figure 3.29 a section view of the static mixing nozzle can be seen. In the two openings on top, the two ink components can be loaded. These will be pushed through the entire nozzle and will come out at the bottom. The lower half of the static mixing nozzle has windings that will mix the ink's components A and B through each other. That way the ink that is printed will always be freshly mixed.

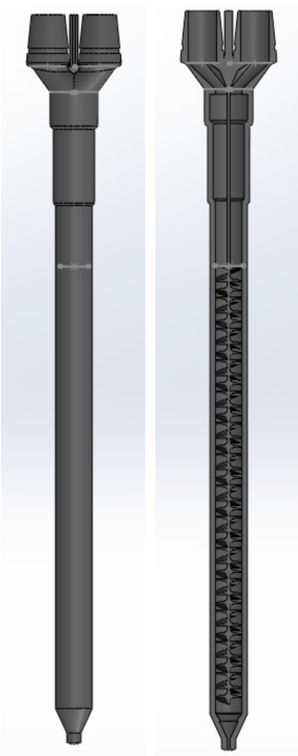

Figure 3.29: A closed and a section view of static mixing nozzle v2. In the two openings on top, the two ink components will be inserted. In the lower part of the section view of the static mixing nozzle, there is a mixing mechanism that will mix the ink by turning and dividing it.

Why using a static mixing nozzle

The advantage of using a static mixing nozzle is that the quality of the ink that comes out of the nozzle stays constant during the print process. This solves dilemma two that was presented in the study by Zhou et al. (2019). Because the ink is always just mixed when it comes out of the nozzle, the ink will not cure inside the nozzle and can be printed for a long time. If the ink is mixed separately before printing the ink will slowly cure in the printer during the print process and change the properties of the ink.

Static mixing nozzle and viscosity

One of the first important factors to make the ink able to extrude through the static mixing nozzle is the ink's viscosity. This was tested in the first static mixing nozzle test and came back when new inks were tested (see static mixing nozzle tests 1 and 4 in Appendices B.b.1 and B.b.4). The first test was started with ink Fe50%SiO_nM, which was the best ink at the moment. The ink, however, was too viscous to be extruded. During that experiment, the amount of iron carbonyl was altered to get a viscosity that could get through the static mixing nozzle and because that was not enough later the fumed silica amount was also changed (see figure 3.22). In later experiments, the viscosity was only changed by changing the amount of added fumed silica.

Static mixing nozzle and force

Another factor that was measured, was the force needed to extrude the ink through the static mixing nozzle (for the measuring procedure see chapter 3.2.2.2). This was important to know for the 3D printer which has a limited force. The more viscous the ink, the more force was needed to extrude the ink through the static mixing nozzle (see figure 3.30) (static mixing nozzle test 4 in Appendix B.b.4).

Another factor that influenced the extrusion force was the mixing mechanism of the static mixing nozzle. Two types of mixing mechanisms were tested of two different static mixing nozzles that were developed before and outside of this project. One stirred one time left, one time right (v2) and the other two times right and then two times left (v1) (see figure 3.31). The static mixing nozzle mechanism of v1 required the lowest force as was found in experiment 3 (Appendix B.b.3).

The last thing that might influence the force needed to extrude the ink through the static mixing might be the surface quality of the 3D printed static mixing nozzle. This was not measured during the tests, but after the first two static mixing nozzle experiments, the surface quality of the 3D printed static mixing nozzles was improved, by printing them one for one.

Ink	Fumed silica (MIT recipe %)	Measured extrusion (Kg)
Fe50%SiO_nM	50	above 24
Fe37,5%SiO_nM	37,5	22,566
Fe25%SiO_nM	25	19,069

Figure 3.30: *Three inks of static mixing nozzle 4 where the only difference in ink is the amount of fumed silica. The more fumed silica is added the more viscous the ink. The measured Kg increased when the amount of fumed silica increased (which did increase the viscosity).*

Figure 3.31: *The mixing mechanism inside static mixing nozzle v2 (1 left, 1 right) and v1 (2 left, 2 right)*

Curing ink

The last important factor of the static mixing nozzle was if the ink was mixed well enough so that it could cure. For this several lines of ink were extruded on a glass plate with the static mixing nozzle or the ink on the static mixing nozzle was just left to dry (see figure 3.32). Both the v2 and v1 could mix the ink components well, and the ink cured. Sometimes the ink was not cured after waiting (static mixing nozzle tests 1 and 4 Appendix B.b.1 and B.b.4) (see figure 3.33). This was always due to not having extruded enough ink through the nozzle or not extruding parts A and B evenly, through which only one component came out first and one component on its own cannot cure. This was tested in the following experiments to be sure (static mixing nozzle tests 2 and 5 Appendix B.b.2 and B.b.5).

Figure 3.32: Extruded static mixing nozzle ink and manually mixed ink, both cured. The ink that cured inside the nozzle.

Figure 3.33: Ink out of the static mixing nozzle that after the curing time had not cured

Decision dual syringe printer

Because the ink could go through the nozzle and was mixed well, there was decided to use the dual syringe printer. All the experiments in this chapter were done manually. The extrusion of the ink through the 3D printer system can be found in chapter 3.4.2.

3.4.2 Ink extrusion system

The ink extrusion system contains the ink and extrudes it through a system so that the ink is extruded out of the nozzle with which structures are printed. Several parts had to change to extrude the vicious magnetic soft material ink. This section will explain the system, the extrusion test and the changes that were made in the system.

The system
For the extrusion of the ink from two syringes, an extra device is used (see figure 3.28). This device was an extension connected to the Ultimaker 3, by which it was also controlled. The syringes with the ink were inserted into the device. Tubes were used to connect the syringes with the static mixing nozzle so that the ink could go there. The static mixing nozzle was then inserted into the printer head of the Ultimaker 3 so that it could be used to print (figure 3.24). For how to operate and connect the entire system see Appendix F.

Extrusion test
Multiple extrusion tests have been performed during this project. The goal was to see if the ink could be extruded through the system without the system failing. For these tests ink was made and put into the syringes. These syringes were then connected to the static mixing nozzle with tubes and put in the dual syringe extruder. Like in figure 3.28, for these tests the static mixing nozzle was not inserted in the printer head. For these tests the ink contained non-magnetized iron carbonyl particles, instead of NdFeB particles. The eight 3D printer tests can be found in Appendix B.d.

The line of G-code that that controlled the extrusion of the syringes was:

G1 F2 A89 B89

The A and B stand for the distance that the pistons of the syringes are pushed and the F for the speed with which this happens.

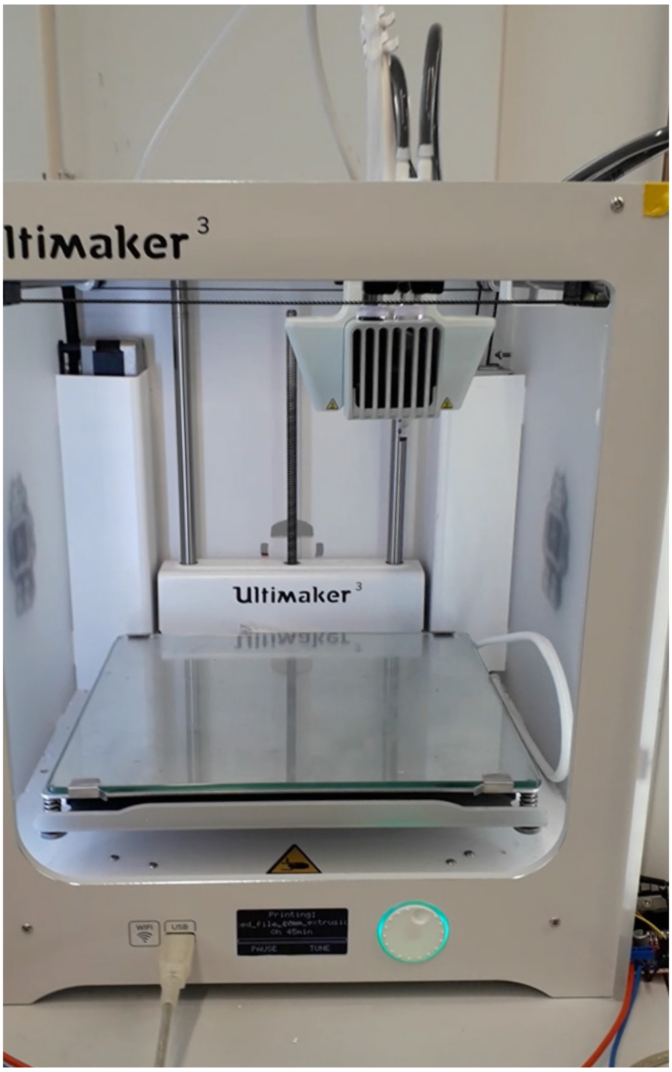

Figure 3.34: *The printer head with inserted static mixing nozzle. The grey tubes are the tubes from the syringes that are filled with ink.*

Alterations extrusion system

The first alteration that was made to the system was to increase the power of the motors that drove the pistons of the syringes to move the ink. At the first tests, this was too low and the motors stopped turning because they could not deliver the needed power. To solve this, the firmware on the extruder was changed, so that each piston could be pushed upon with a force of 180 N instead of 80 N. Also the 60 mL syringes were changed for 10 mL syringes. 10 mL syringes have a smaller area that pushes against the ink in comparison with the 60 mL syringes. According to the formula of pressure:

$$Pressure = \frac{Force}{Area}$$

The same fore with a smaller area makes that a higher pressure can be exerted on the ink. For this conversion, pieces were made so that the 10 mL syringes fitted in the 60 mL syringes slots of the dual syringe extruder (figure 3.35). For an overview of all the designed 3D printer parts see Appendix E.

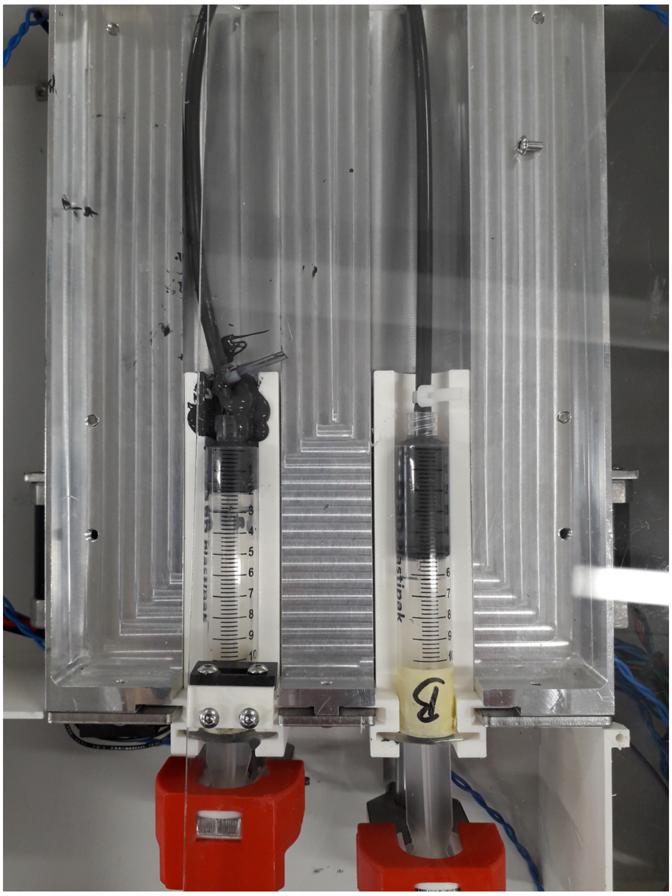

Figure 3.35: *The 60 mL syringe to 10 mL syringe conversion pieces, the white parts in which the syringes are clicked as well as the white part near the red clips*

Figure 3.36: *The new connections at the static mixing nozzle to secure the tubes*

The second alteration was the added Luer lock connectors and the changed connection of the static mixing nozzle (see figure 3.36 and the metal part in figure 3.37). These prevent the tubes from jumping off and spilling ink (see figure 3.35), due to the high pressure in the system. Sometimes a cable tie was needed to keep the tubes locked into place.

The last alteration was a replacement of the tubes. Due to the high pressure and the viscous A component of the ink, tubes with elastic walls tend to swell up when it takes too much force to push the ink forward in the tube. This caused the ink to delay and not come at the static mixing nozzle anymore. If it would come it would result in an uneven ink ratio. Therefore tubes were used with stiffer walls. (see figure 3.37)

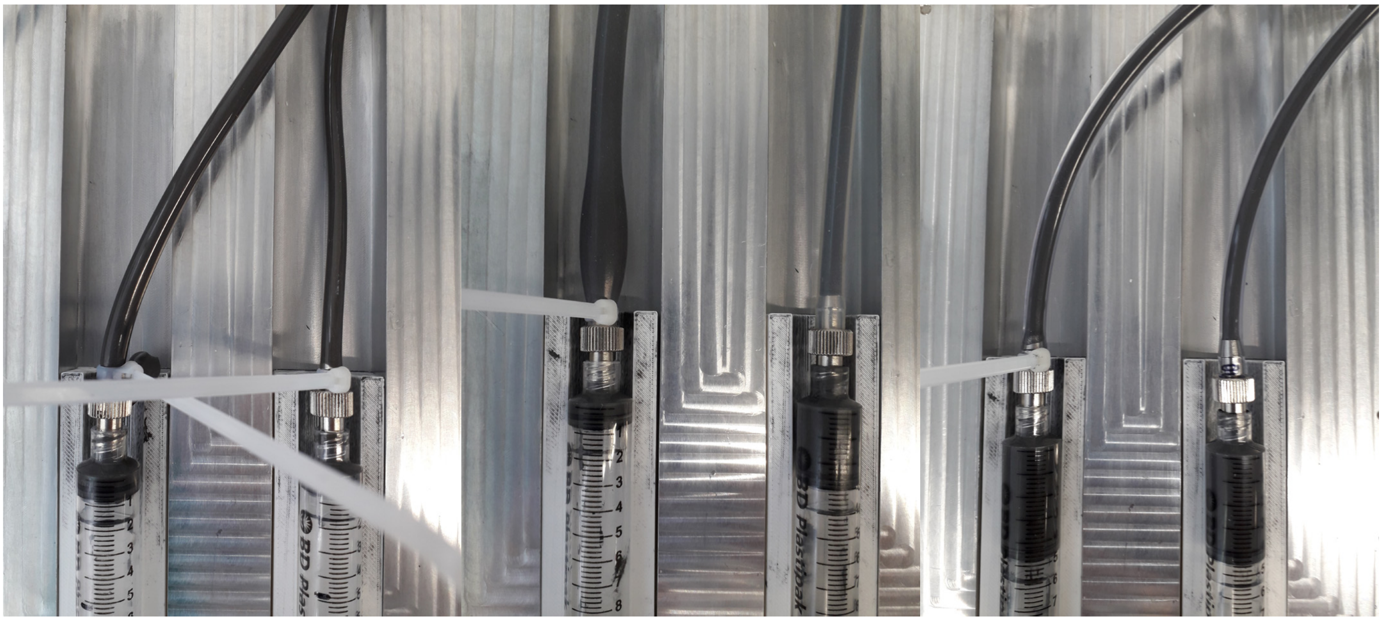

Figure 3.37: The three types of tubes tested during this project, the one on the right has the stiffest tubes and worked the best because they did not swell up

NdFeB particle ink extrusion
It was once tried to extrude non-magnetized NdFeB ink through the system. This was done to see if the ink would perform differently and if it was able to go through the system. The NdFeB particle-based ink was well extruded.

Measure volume and speed G-code
The A, B and F values of the G-code were also measured. More about this can be read in Appendix B.e. The value to extrude 1 mL of ink out of syringe A of V is 89 and for the graph of the measured speed in mL/s of command F, see figure 3.38.

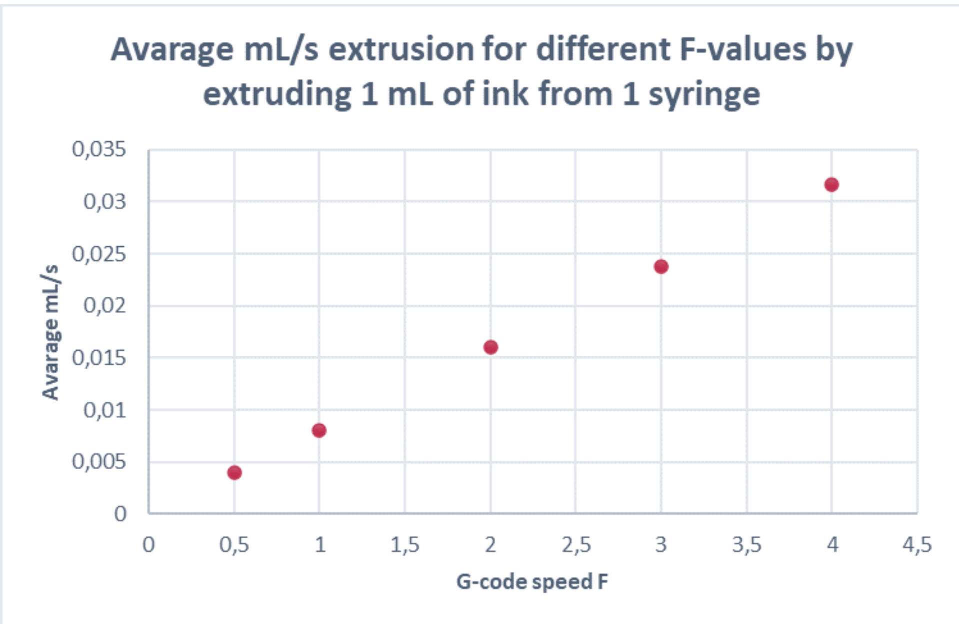

Figure 3.38: The value of F in the G-code and the corresponding extrusion speed. This was measured with the extrusion of 1 mL of ink; A89 or B89 in the G-code.

3.4.3 Electromagnet

The fourth dimension of the 4D printing system is the magnetic field at the nozzle under which the ink is printed. The magnetic field aligns the magnetic particles to create distinct North and South poles in the printed magnetic soft material structures.

Electromagnet or permanent magnet
For the 4D printer setup, there is chosen to work with an electromagnet instead of a permanent magnet. The advantage of an electromagnet is that the direction of the magnetic field can be turned around by changing the current flow through the coil. Due to that, the ink can be printed in a continuous line, which is easier for printing silicones. It also improves the surface quality of the ink. With a permanent magnet, the magnetic field direction cannot be switched and therefore the ink should stop and be brought to another spot to print. This can leave imperfections. (see figure 3.41)

Coil design
The magnetic field at the nozzle of the 3D printer of MIT had a strength of 50 mT (Y. Kim et al., 2018). This was therefore also the requirement for the electromagnet for this 4D printer because it is unknown if a weaker field will align the magnetic particles as well. The details of the electromagnet can be found in Appendix H.

Electromagnet attachment
To attach the electromagnet at the tip of the static mixing nozzle, a screw connection was designed with a thread at the static mixing nozzle and its negative in the centre of the coil (figure 3.40 top). This way there is no force needed to connect the electromagnet to the static mixing nozzle which is prone to breaking. The static mixing nozzle was also extended to be able to stick out under the printer head, with enough length to attach the electromagnet to (figure 3.40 bottom). This was done in the region outside the mixing mechanism which prevents extra needed force created by the mixing to push the ink through (figure 3.39).

__Figure 3.39:__ The original static mixing nozzle length and the extended static mixing nozzle. The extension was made in the part where both ink components are still separate to not add extra additional needed force in the system.

***Figure 3.40:** (top) The thread at the end of the static mixing nozzle. (bottom) The extended static mixing nozzle in the printer head with a prototype for the electromagnet spindle turned on.*

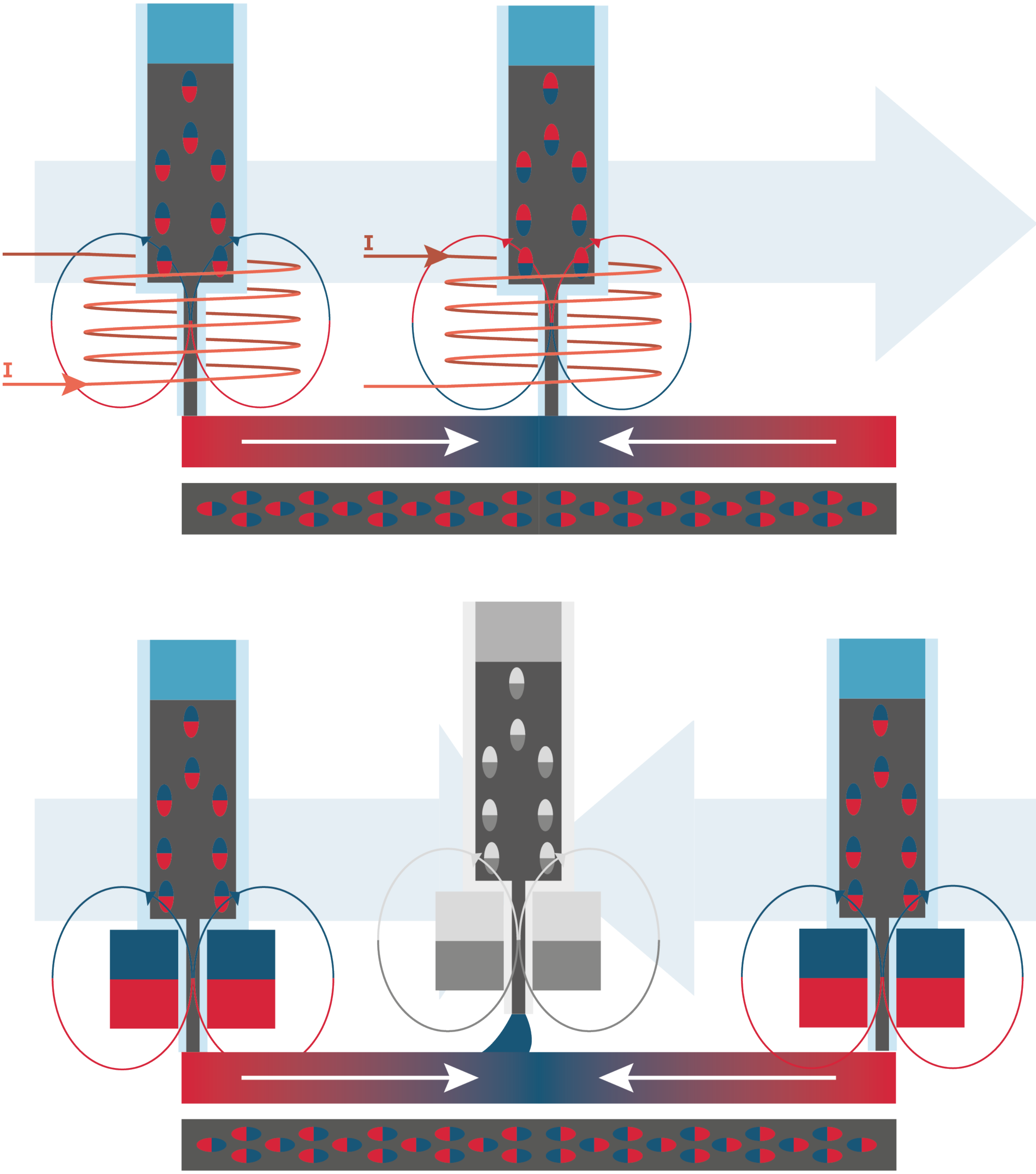

__Figure 3.41:__ The difference in printing one line with opposite magnetic field directions by an electromagnet (top) or a permanent magnet (bottom)

Magnetic shield

A magnetic shield was cut out at the same diameter as the electromagnet from first grade Non-Oriented Electrical Steel from Nippon Steel (Japan). This should be attached at the bottom of the electromagnet that is facing the print bed. The magnetic shield is supposed to weaken the magnetic field under it so that it will not influence the printed magnetization patterns (Y. Kim et al., 2018) (figure 2.16). This material did not work yet and in magnetizer test 2 (Appendix B.c.2) the ink still got attracted by the magnet on the other side of the magnetic shield. Therefore other materials should be selected and tested to work as a magnetic shield.

Electrical connection to the printer

The electromagnet was connected to an H-bridge to be both connected to a power supply for enough power (27 V and 0,3 A) as well as the 3D printer to control the direction of the magnetic field and turn it on or off. A connection to snap the H-bridge to the 3D printer cable clips was designed to keep the high power wires to the electromagnet as short as possible (figure 3.42). To control the electromagnet, the H-bridge was connected to two 3D printer pins. One was used to turn the electromagnet on or off and the was used to switch the direction of the current flow and therefore turn the magnetic field 180°. The pins were supposed to be controlled with the G-code (example Appendix G):

M42 P[number of the pin] S[0...255]

Unfortunately through a different firmware on the 3D printer, to probably control the dual syringe extruder, not one of the pins could be turned on (see Appendix B.e.2). The electromagnet was not fully connected and tested in this project.

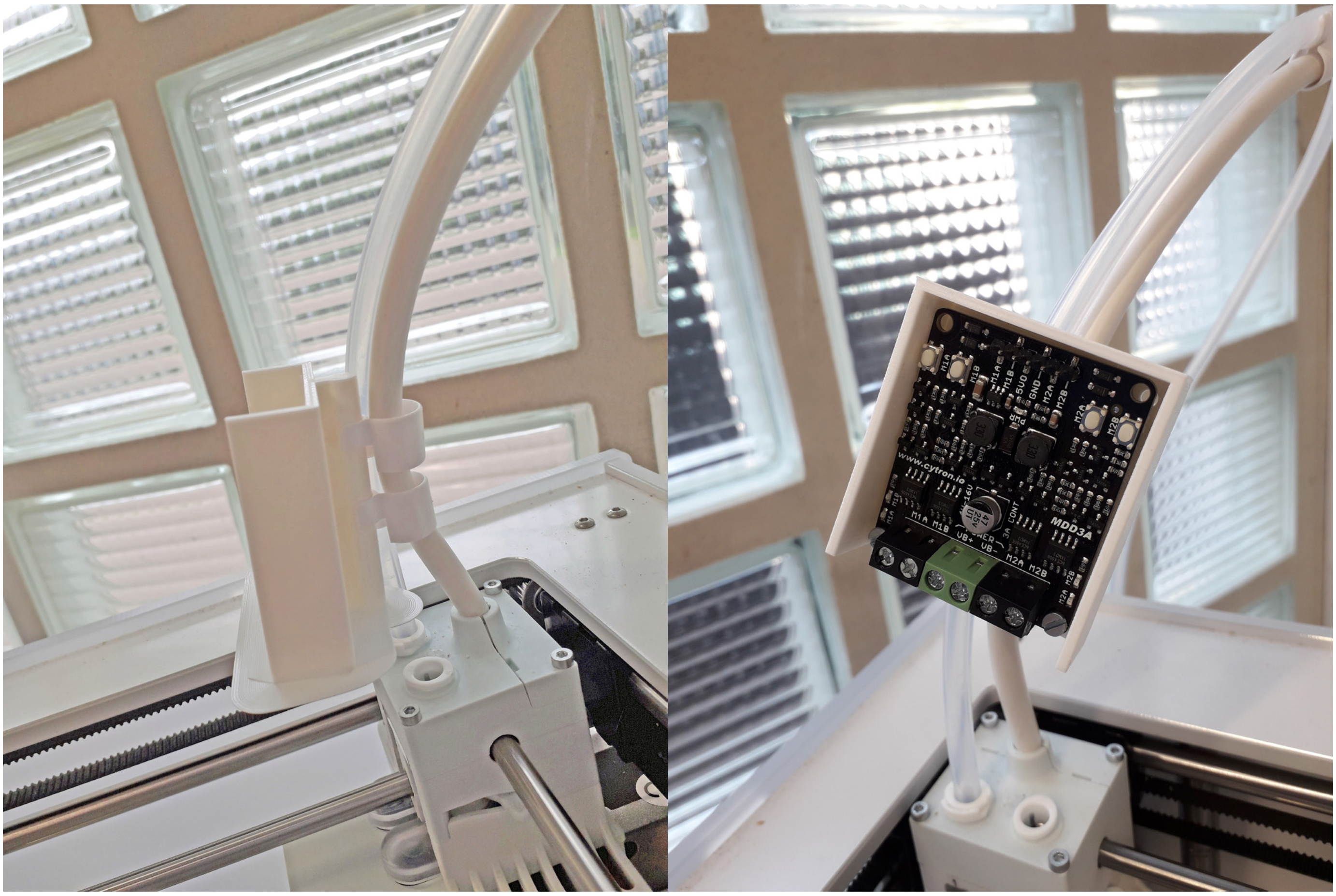

Figure 3.42: *The connection piece of the H-bridge that can snap on the cable clips of the 3D printer*

3.4.4 Remaining work

The 4D printer setup came far during this project but was not finished yet. This section will sum up some future work needed to finish the system.

Considering that the extrusion system worked after all the alterations, it was tried to print the first line with it. However, there is a delay of several minutes in extruding the ink at the syringes and when it comes out of the static mixing nozzle (Appendix B.d.8). Maybe the system has to be primed well enough, or the tubes do still expand a bit. For the latter, reinforced tubes could be tried and for the printing, extra tests for printing are needed.

For the electromagnet, the firmware of the 3D printer should be looked into, to enable controlling the pins by the G-code. The electromagnet itself should be tested on its magnetic field.

Another material should be selected and tested for the magnetic shield because the current one did not work.

Reconsidering the type of 3D printer
On the other side, before this project is continued by somebody else, this 3D printer system should be reconsidered. Until now, the highest viscosity ink that was extruded successfully through the system was the Fe31,25%SiO_nM, which started around 11.000 Pa.s (Appendix B.d.8). During the extrusion through the 3D printer system, this already seemed to be the limit of the system. The motors stopped turning sometimes for a fraction of a second (Appendix B.d.8). The 11.000 Pa.s is 45.000 Pa.s lower than the 56.000 Pa.s of the magnetized NdFeB12,5%SiO_M (see figure 3.43). The increase in viscosity of the magnetization of the NdFeB12,5%SiO already was 54.000 Pa.s, which is more than the viscosity of the ink that already seemed to be the limit of the printer.

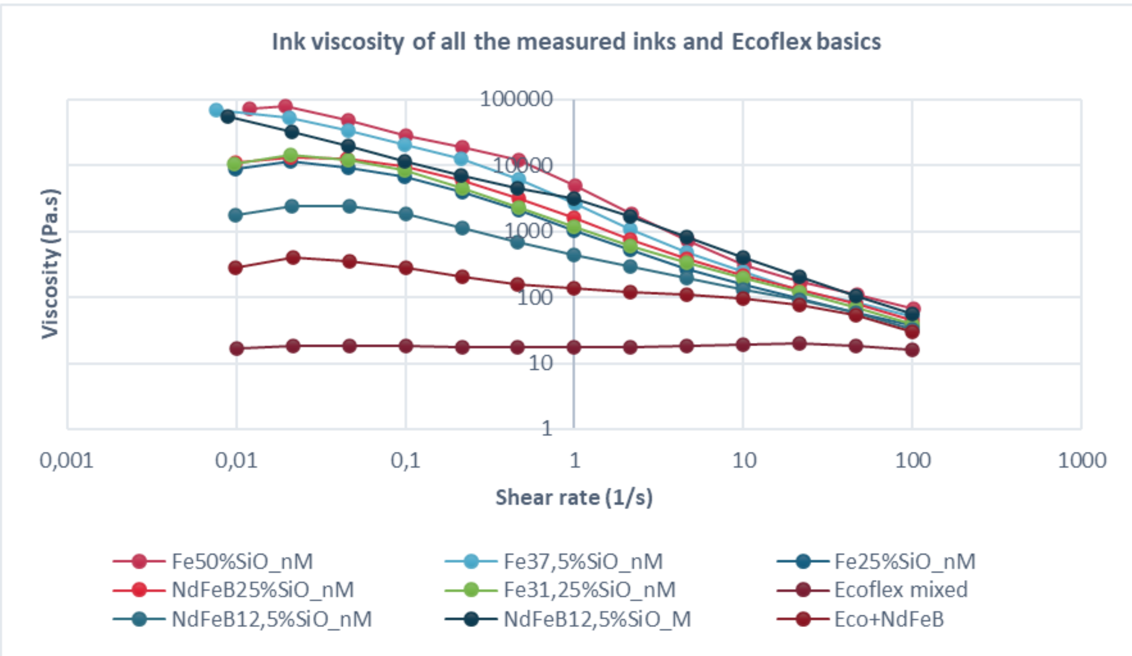

__Figure 3.43:__ The viscosity measurements of the inks that were measured throughout the project and two reference measurements of plain mixed Ecoflex and Ecoflex with only NdFeB particles which also shows shear-thinning properties

3.5 Proof of principle

At the end of the project, a proof of principle of the magnetic soft material was made. It was created from magnetized NdFeB12,5%SiO ink and extruded with a syringe with an attached ferrite ring magnet (figure 3.44). This was done because the 3D printer setup was not ready and tested for printing. Also, the viscosity of the ink would probably be too high for the 3D printer to extrude.

With the syringe, a strip was extruded with a magnetization pattern of North-South-North as was illustrated in the lower half of figure 3.41. The ring magnet was placed higher up the syringe because the magnetic shield did not work. Therefore the ink was pulled from the glass plate if the magnet was placed too close to the ink (neodymium ring magnet).

In figure 3.45 the magnetic soft material and its shape transformations can be seen. It is placed in water to reduce the friction of the silicone rubber with the glass. Depending on the direction of the magnetic field of the disk magnet under the glass, the middle of the strip or the ends of the strip stands up, because they are repelled by the magnetic field.

Even though the full setup is not finished and optimized yet, this result proofs that it is possible to make magnetic soft material.

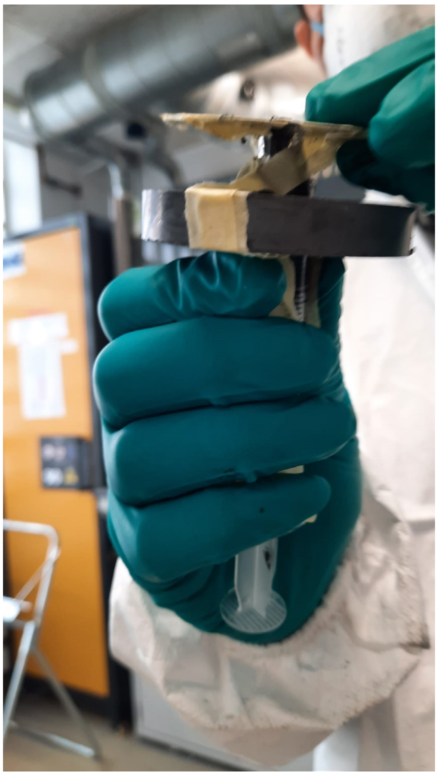

Figure 3.44: *Syringe with mixed ink and a ring magnet at the nozzle. The larger ferrite magnet is further up the syringe to prevent the magnetic field from pulling the extruded ink of the glass plate.*

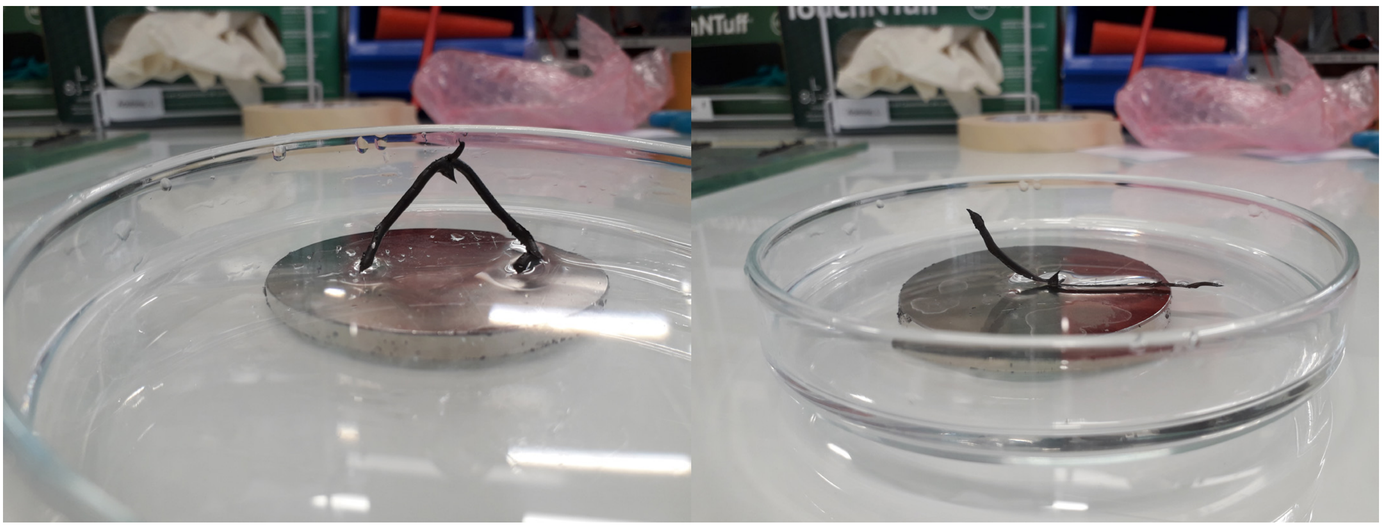

Figure 3.45: *Working magnetic soft material. The disk magnet is turned around in the other picture, therefore once the middle is repelled and stands up in the left picture and in the picture on the right an end of the strips stands up.*

4. Conceptualization

This section will focus on the development of a concept for a demonstrator to show designers and researchers the characteristics of magnetic soft materials and inspire them. Other generated ideas are sorted in idea directions that can be used for future research and to fill up the solution space in which magnetic soft material can be used

4.1 Design goal

This is the second part of the project that was run simultaneously with the ink development and 3D printer setup section. In this section of the report, two things will be developed. A concept for a demonstrator and ideas and idea directions for future applications of magnetic soft materials.

The concept for the demonstrator's purpose is to show other designers the unique characteristics of magnetic soft materials, and to inspire them to design with this new material and understand its properties. The concept for the demonstrator will be built upon the M-shape out of MIT's research (see figure 2.1). This shape transformation was proven at the end of the project in chapter 3.5 .

This section will be concluded with selected ideas and idea directions from the idea generation. To show the potential and use of the material for implementations in products. This can broaden the solution space for magnetic soft materials, which now is still narrow and can further inspire designers and researchers to look into magnetic soft materials.

4.2 Design criteria

The purpose of the demonstrator concept is to show designers the characteristics of the magnetic soft material and preferably show them all. This chapter will list the criteria and wishes for the demonstrator concept as well as present some boundaries for the concept to stay in, due to a lack of research and knowledge to make the demonstrator concept possible. The criteria in this chapter are used more for the demonstrator concept than the future design opportunities.

4.2.1 Criteria

1. The demonstrator should have remote shape transformations
2. The demonstrator should have fast shape transformations
3. The demonstrator should have reversible shape transformations
4. The demonstrator should incorporate the elasticity of the elastomer
5. The demonstrator should have constantly moving or dynamic shape transformations

The first three criteria are the characteristics that are often given to magnetic soft materials in research papers (see literature review chapter 2.2).

Criteria four comes from the early ink development in which the elasticity of the elastomer stood out as an interesting factor (ink development 3 Appendix B.a.3).

Criteria five is added to set magnetic soft materials more apart from shape memory alloys and polymers, which are more known at the faculty of IDE at Delft University of Technology, that are not designed for constant movement.

4.2.2 Wishes

1. The demonstrator could be used in an environment that may not heat up

This whish is also created to set magnetic soft materials more apart from shape memory alloys and polymers. But also to show opportunities. It is often a challenge to use shape memory alloys and polymers on or inside bodies because they often need to be heated up to harmful temperatures (above 40°) to activate their shape memory effect (F. Zhang et al., 2019).

4.2.3 Demonstrator concept boundaries

At last, there are some boundaries for the demonstrator concept. These come from a lack of time, knowledge and/or research and therefore the full potential of the magnetic soft material is not known. These boundaries or additional requirements make sure that the demonstrator could be made. They are also research gaps for follow up research.

1. The concept should not have a larger dimension than 68 mm

68 mm was the maximum dimension in the printed objects presented in the study of MIT (Y. Kim et al., 2018) and therefore can probably be activated. For those structures, a magnetic field of 200 mT was used. 200 mT can already endanger the functioning of some objects, like magnetic cards, pacemakers, mechanical watches and hearing aids. For these objects, magnetic fields start to be damaging between the 0,5 mT and 40 mT (Supermagnete, n.d.-b). Therefore it is important to handle larger magnetic fields with care so that they do not have a harmful influence on their environment.

It is also unclear to me if a long exposure to high magnetic fields is harmful to the body and/or the environment. The magnetic field of the earth has an average of 50μT with a maximum of 70μT. Small magnets that are worn close to the body, buttons, magnetic toys and jewellery mostly have a magnetic field of 0,5mT (Ziegelberger & ICNIRP, 2009). In the study of Ziegelberger and ICNIRP (2009), a guideline of no magnetic exposure to the general public above the 400 mT is suggested. However, more research is needed on this to know what limits should be used in designing with magnetic soft materials.

2. The concept should not carry or pull on high loads

This boundary is there because it is unknown what forces, activated magnetic soft materials can carry. In figure 4.1, a screenshot can be seen of an activated structure from a MIT video ((MIT), 2018), that is pressed flat to the surface by the force of a finger. That does suggest that the activated material can maybe not rest high forces. A lot of knowledge about this is unknown and could be found out in further research. Some lightweight objects could be carried as is seen in picture 2.10 by a gripper (Goudu et al., 2020; Ren et al., 2019).

3. The demonstrators' magnetization pattern should have the same magnetization pattern as MIT's M-shape

This should be done to prove that the concept can work. This simple bend was also proven by the proof of principle in chapter 3.5.

__Figure 4.1:__ The magnetic soft material that is transformed under a magnetic field, is easily pressed flat by the force of a finger

4.3 Ideation

4.3.1 Methods

This chapter will discuss the methods that were used during the ideation. Even though there were two design goals, the ideation was used to both find an idea to turn into a concept for a demonstrator, as well as to find interesting idea directions for the future, to broaden the solution space in which magnetic soft material can be used.

To find some promising ideas, many ideas should be generated, for which multiple methods have been used.

Creative session
The idea generation was kicked off by a small creative session of two hours with two other IDE students. The purpose was to gain inspiration and new perspectives for magnetic soft material applications. The session was designed around the first two diamonds of the revisited CPS model by Tassoul and Buijs (2007).

Brainstorming during conversations
Some ideas were generated by talking about my project with people during daily life. The purpose was again new perspectives, inspiration and different work backgrounds and interests.

Activation magnets
Also, some research was done about the magnetic fields used to induce the shape transformations. This was done to understand and see the possibilities of how products could be activated. There was looked into videos and papers about magnetic soft materials.

Magnetic soft material replacements for brainstorming
The magnetic soft material was not fully developed at the moment of ideation. Due to this there was looked into other materials to represent magnetic soft materials to come up with ideas. Like wire and a knitted square.

Inspiration search
Different topics were looked into, to serve for input in brainstorm sessions.

MIT shape analysis
A brainstorm was conducted by coming up with ideas to use MITs shape transformations shapes.

To get a better feeling for magnetic soft materials, without having a proof of principle or working material yet, the printed MIT shapes, from the paper (Y. Kim et al., 2018), were analysed by prototyping them with paper and analysing the shape transformations.

One idea selected by criteria

In the end, one idea was selected to develop into a concept. The idea was selected that fit most of the criteria and boundaries. To make sure the demonstrator could be made and show as much of the unique qualities of the magnetic soft material.

Future idea directions

For the future idea diretions, the criteria were not as strict as for the demonstrator. These opportunities have been identified during the ideation, but do not specifically show all the unique qualities of the material, but are opportunities for which the material is suitable. Some of these ideas do also not fall into the boundaries set up in the design criteria chapter but might deserve extra research to see if they could be made possible, due to the current lack of knowledge on the material.

4.3.2 Ideation

This chapter shows some of the brainstorming sessions for the ideation, as well as inspiration and some of the methods mentioned in the subchapter before this one. Throughout this chapter some ideas are highlighted, the generated ideas can be found in Appendix J.

4.3.2.1 Creative session

The idea generation was kicked off with a creative session. The purpose was to create some first ideas and have new perspectives on magnetic soft materials. The sessions set-up was based upon the first two diamonds (problem statement and idea generation) of the revisited CPS model of Tassoul and Buijs (2007). For this session, the boundaries were not taken into account.

Two IDE bachelor students were the participants during this session. I was the facilitator. All the intermediate results of this session can be found in Appendix I.

First flower associations (Tassoul, 1999-2009) (p. 17) were done about the four characteristics of magnetic soft materials: fast, remote (wireless), reversible and heatless. This was done to wake up the knowledge the participants already have about these subjects.

After that, it was explained what magnetic soft materials are and videos of MIT were showed to give the participants a feel for the material. After this, a brainstorm was done about the characteristics of magnetic soft materials. Another brainstorm was done about different locations and domains in life.

These two brainstorms were afterwards combined as input for a guided fantasy (Tassoul, 1999-2009) (p. 71). The participants were asked to draw product ideas of products that they would encounter in a world made out of magnetic soft materials visiting the different locations thought of before (see figures 4.2).

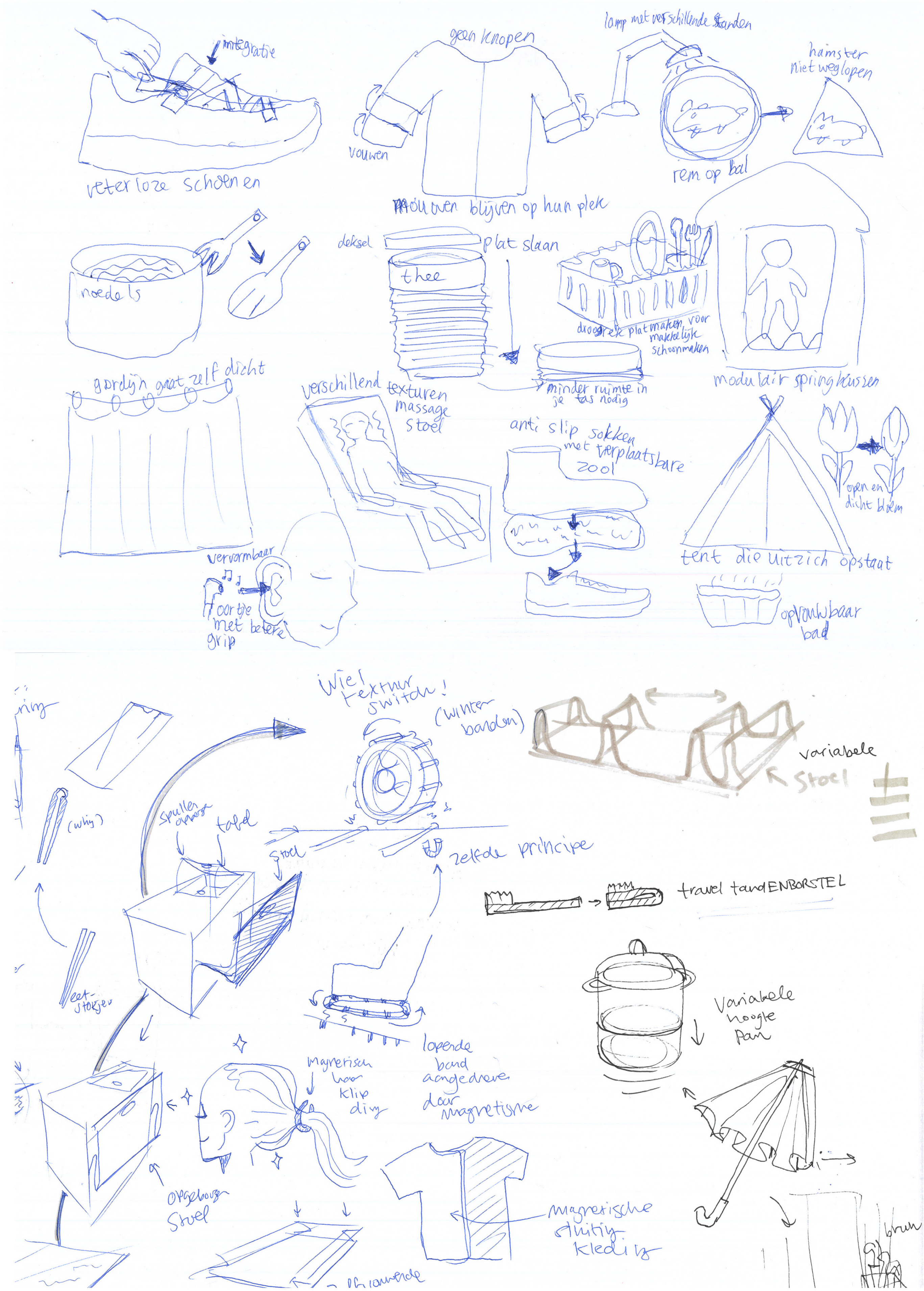

Figure 4.2: *Generated ideas during the guided fantasy*

A C-box (Tassoul, 1999-2009) (p. 88) which is normally used to select promising ideas, was now used as a conversation tool to discuss all the individual ideas. During the conversation four identified types of ideas and idea mechanisms were identified at the end of the session:

Texture transformations
One of the most interesting concepts during the idea generation was the texture change of magnetic soft materials. For example, there is an anti-slip sock with a retractable texture (see figure 4.3 right) throug which the sole could be made smooth again so there was less resistance with the floor or shoe which prevented the user from slipping on a slippery surface. A tire changing from summer to winter tire by the change of texture can be seen in figure 4.3 left. Both ideas increase and decrease their surface roughness.

Opening and closing motion
Multiple objects presented opening and closing motions that were multifunctional. For example, a fork (see figure 4.5) that through a magnetic field could transform into a spoon.

Foldable and space-saving products
There were also many ideas where the magnetic field folded the material in or out to save space. For example, see figure 4.4 (left) where a box could be transformed into a table and chair for compact living situations. Another foldable product that was interesting was a drying rack that could be folded in (see figure 4.4 right). The advantage of this was that it could be easier to clean, by folding in all the edges that normally stick out.

Personalised fits
The last identified idea direction was personal fit, which is also a type of texture change. For example, a pair of shape-changing earphones, where the embodiment through a magnetic field can change to fit the shape of the ear of the user (see figure 4.6).

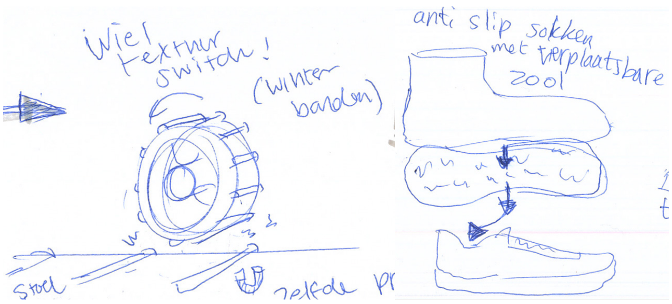

__Figure 4.3:__ (right) The texture of tires made out of magnetic soft materials. So the tires can be switched from summer to winter tires by the change of a magnetic field. (left) Socks where the non-slip sole can be retracted with magnetic soft materials to slip easily into your shoes

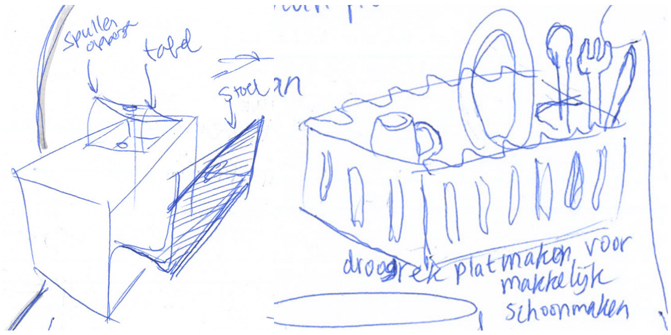

__Figure 4.4:__ (left) A box with multiple functions. Here a table and chair pop out of the shape. (right) A drying rack for dishes that can be folded in to be easier to clean

__Figure 4.5:__ A spoon fork combination that can switch between two modes

__Figure 4.6:__ The magnetic field activates the magnetic soft material in the embodiment of earphones, to make the embodiment adjust to the shape of the ear of the user

4.3.2.2 Inspirational topics

There was looked into different topics for extra inspiration for individual brainstorming. Topics that were explored were haptics, kirigami and bistable mechanisms.

Haptics

Haptics as a domain is interesting for magnetic soft materials. As for shape-changing materials that do not require heat to change shape, there was already thought of using it inside the body. However, it can also be used on the skin as a feedback mechanism.

A TED talk by robotion Jamie Paik (2019) showed a button that gave feedback as different stiffnesses and forces to press on to give feedback on the material shown on a picture on a computer (see figure 4.7). The type of robot that she used could be replaced by magnetic soft materials (see figure 4.8). By changing the magnetic field strength emitted by an electromagnet, the stiffness of the button could change.

Other haptic products that were thought of were compact braille boards (see figure 4.9). This depends on the magnetic field strength needed to pop up the magnetic soft material and if micro coils can emit such fields.

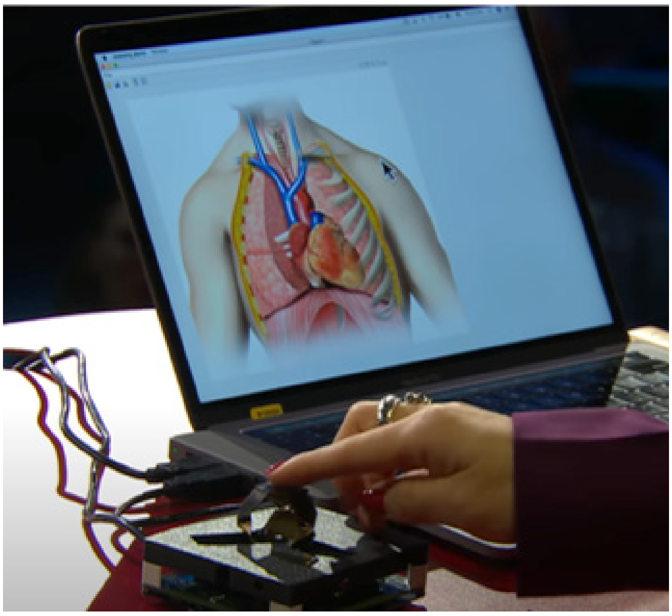

Figure 4.7: The reformable robot presented in the TED talk (Paik, 2019)

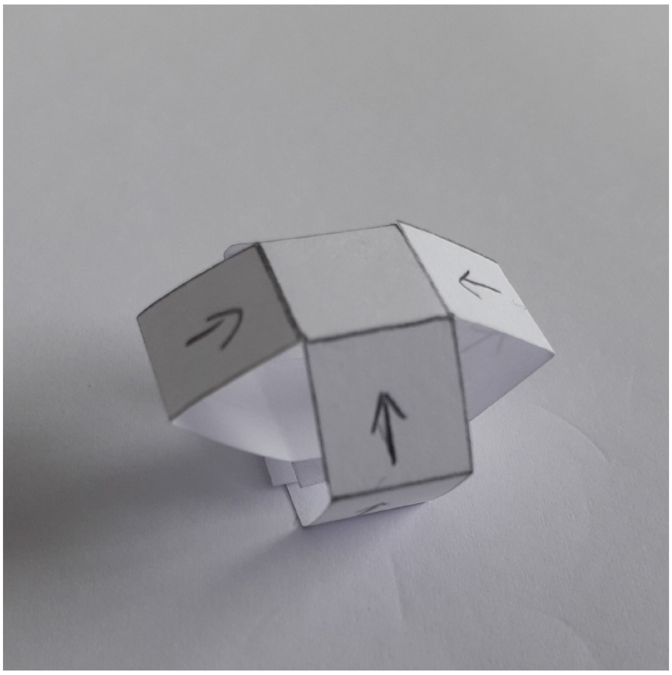

Figure 4.8: The button in 3D as if made from magnetic soft material with the magnetization pattern directions

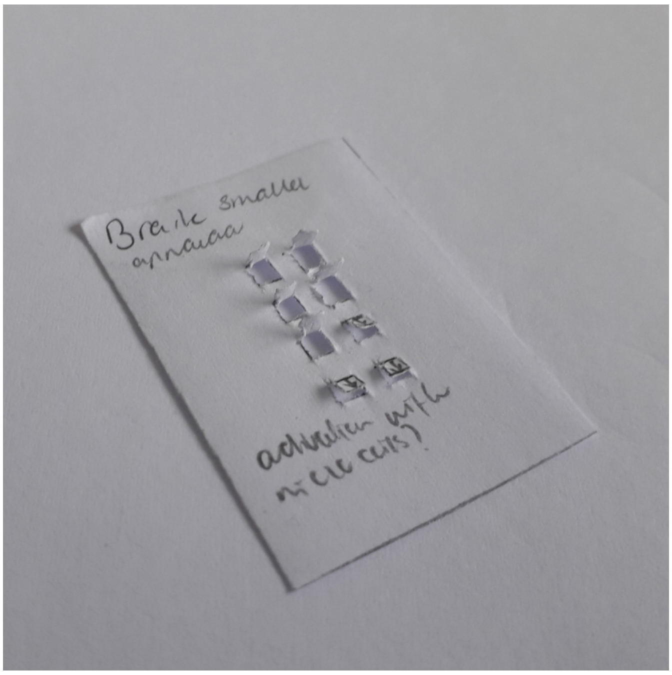

Figure 4.9: The compact braille concept with the magnetization patterns on it

Kirigami

Kirigami is like origami but then with the addition of cuts in the folded material. What is interesting from the examples in figure 4.10, is that they are made from a 2D sheet that is folded with maximum angles of 90°. This is possible to create with 2D printed magnetic soft material from the MIT study. Kirigami adds to different types of shape transformations that could be made with magnetic soft materials.

Example D in figure 4.10 and the two buttons in figure 4.11 are also interesting. If they are made out of magnetic soft materials, they could function in a dynamic interface, where inactive buttons are retracted.

The braille board idea from the haptics session could also be seen as kirigami.

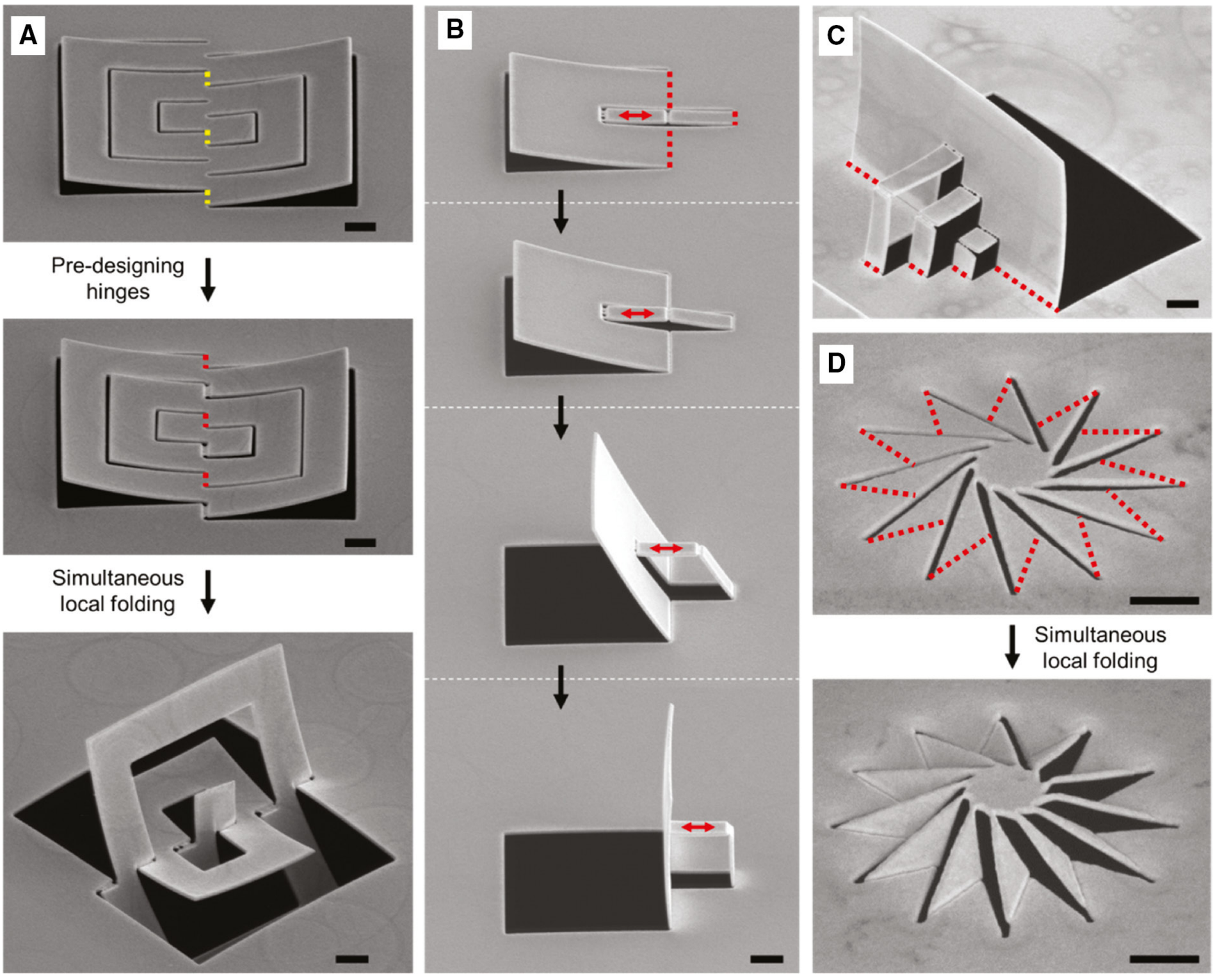

Figure 4.10: *Kirigami from the study of Li and Liu (2018)*

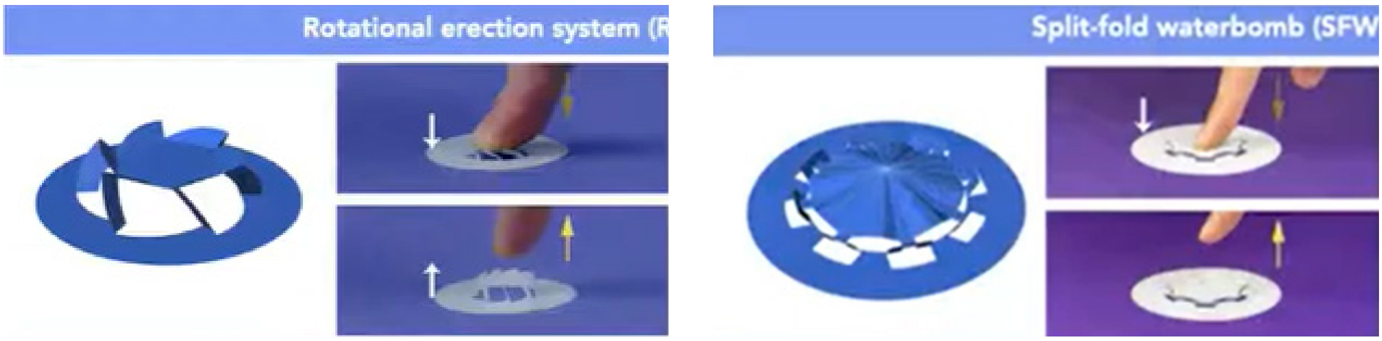

Figure 4.11: *Kirigami buttons (SIGCHI, 2020)*

Bistable mechanisms

Bistable mechanisms are mechanisms that have multiple stable positions or rest positions. For a bistable mechanism to go from one state to another, it should be put over a centre of motion. In figure 4.12 a collage of bistable mechanisms can be seen.

Bistable mechanisms are interesting because magnetic soft materials do lose their activated shape after they are released from their magnetic field. To bring bistability in their mechanisms their activated shapes could be preserved.

One of the ideas inspired by this can be seen in figure 4.13. The magnetic soft material is used as a mixer tab mechanism. In one state it closes off one side of the water supply and in the other state the other side. An electromagnet switching direction could push and pull the magnetic soft material in the two states. The magnetic field for the shape transformation could be on the outside of the water tubes. It can be interesting if the moving material could prevent calcification of the mechanism when it casts off the calcification by turning its shape inside out.

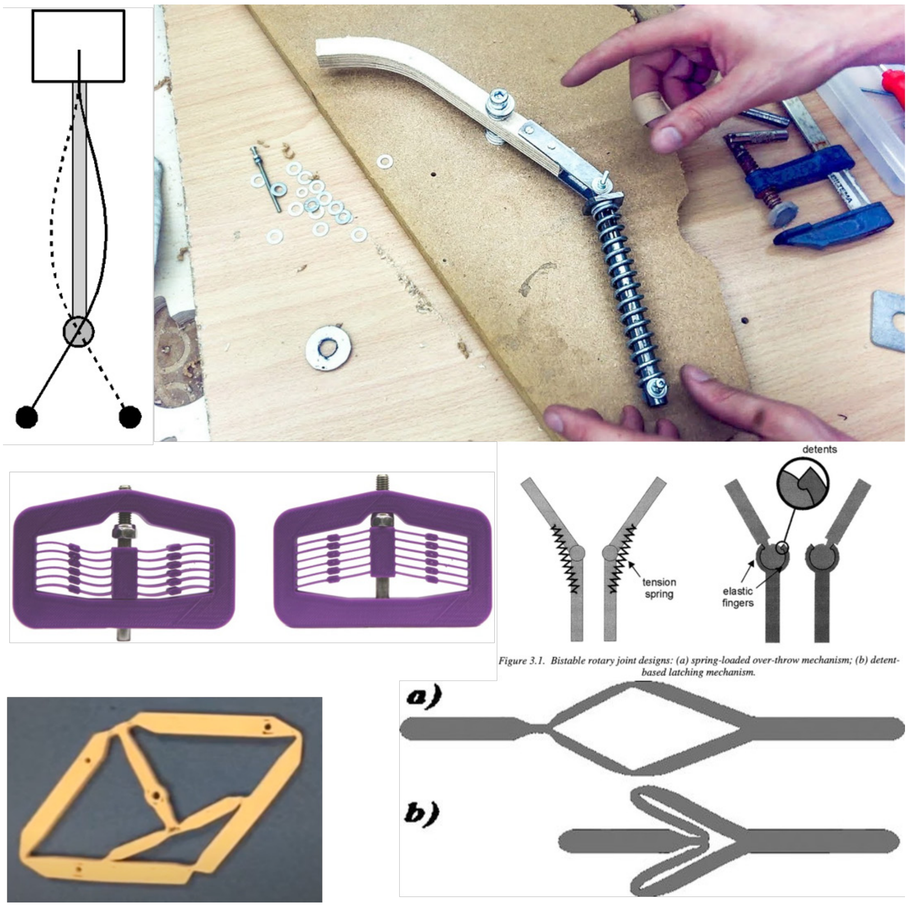

Figure 4.12: *Collage of bistable mechanisms (Alqasimi, Lusk, & Chimento, 2016; Lichter, 1999; Technology, 2015; Wintergatan, 2018; Zirbel, Tolman, Brian P, & Howell, 2016)*

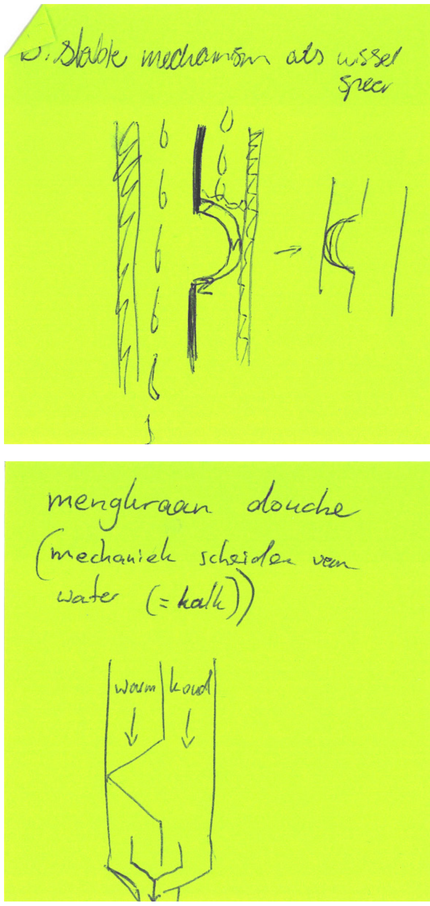

Figure 4.13: *Mixer tap with magnetic soft material as the switching mechanism*

4.3.2.3 Activation magnetic fields

Magnetic soft materials have to be activated by an external magnetic field, to change their shape. At this moment magnetic soft materials are only made and activated in laboratory settings. When magnetic soft materials are going to be used outside of the laboratory, it should be known how they could be activated for their product design.

The two types of magnetic fields that are used the most for magnetic soft materials are permanent magnets and electromagnets.

Figure 4.14 shows an example of a permanent magnet in the study by MIT ((MIT), 2018). The permanent magnet is moved and rotated by hand to change the activated shapes of the magnetic soft material.

Figure 4.15 shows a rose that is activated by two electromagnetic fields (TV, 2020). The magnetic field could be switched to create the opposite shape transformation

Both the permanent magnet and the electromagnet have advantages and disadvantages in their use. For this comparison, it is considered that they are used the way that they are presented here and are not attached to mechanisms that can move them.

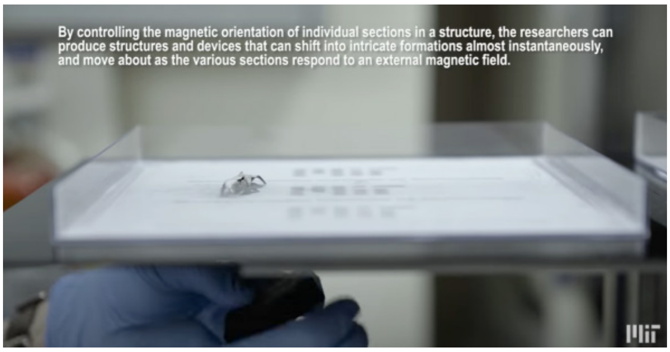

***Figure 4.14:** The magnetic soft material is activated by a permanent magnet that is moved and rotated under the glass plate by a researcher ((MIT), 2018)*

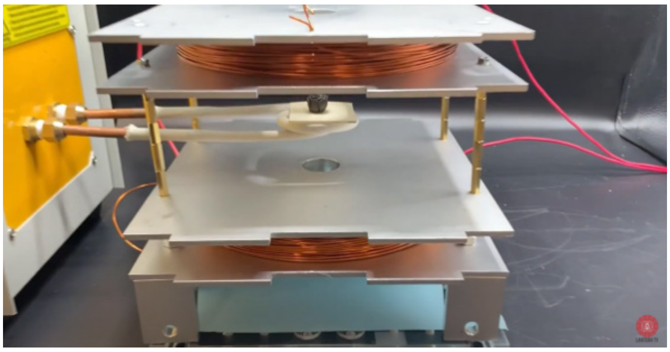

***Figure 4.15:** The magnetic field for activation is created by the two electromagnet coils (TV, 2020)*

Permanent magnet
- User has to become trained in how to move the magnet
- More continuous movement than the electromagnet

Electromagnet
- Can be controlled by a device
- Easier flipping of magnetic field
- Could be integrated into the product itself

Another type of magnetic activation that maybe could be used in later projects is the magnetic milli-robot swarm platform from the study by Hsu, Zhao, Gaudreault, Foy, and Pelrine (2020). This board has small magnetic fields that can be switched on and off to make swarm robots move in the preferred directions over the entire board. It has tiny North and South poles that are created from neighbouring wires, where the current runs in opposite directions (see figure 4.16). This could be an interesting mechanism. Questions by this technique are:
- What magnetic field strength can the platform create?
- What magnetic field do magnetic soft materials require to be activated?

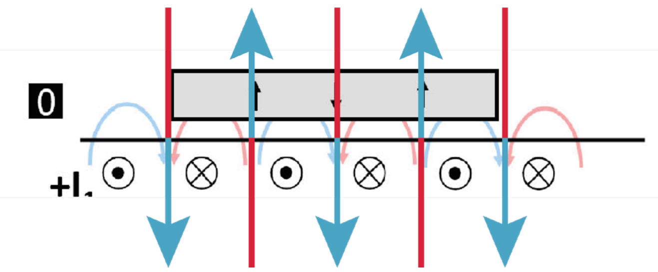

***Figure 4.16:** The current of the neighbouring wires run in opposite directions. The place where the magnetic fields of two wires meet, both turn up or down, creating small local magnetic fields. (Hsu et al., 2020)*

4.3.2.4 MIT shape transformation analysis

To understand more about the shape transformations and the magnetization patterns of magnetic soft materials, prototypes were made from MITs magnetic soft materials shapes (Y. Kim et al., 2018).

The 2D printed shapes (see figure 4.17 A-F) were printed, cut out and folded (see figure 4.18). Some of the 3D shapes (see figure 4.17 J-K) were analysed on paper (see figure 4.19). Some of these 3D shapes were also designed into product mechanisms like surface levelling, elevators and catapults (see figures 4.20 and 4.21).

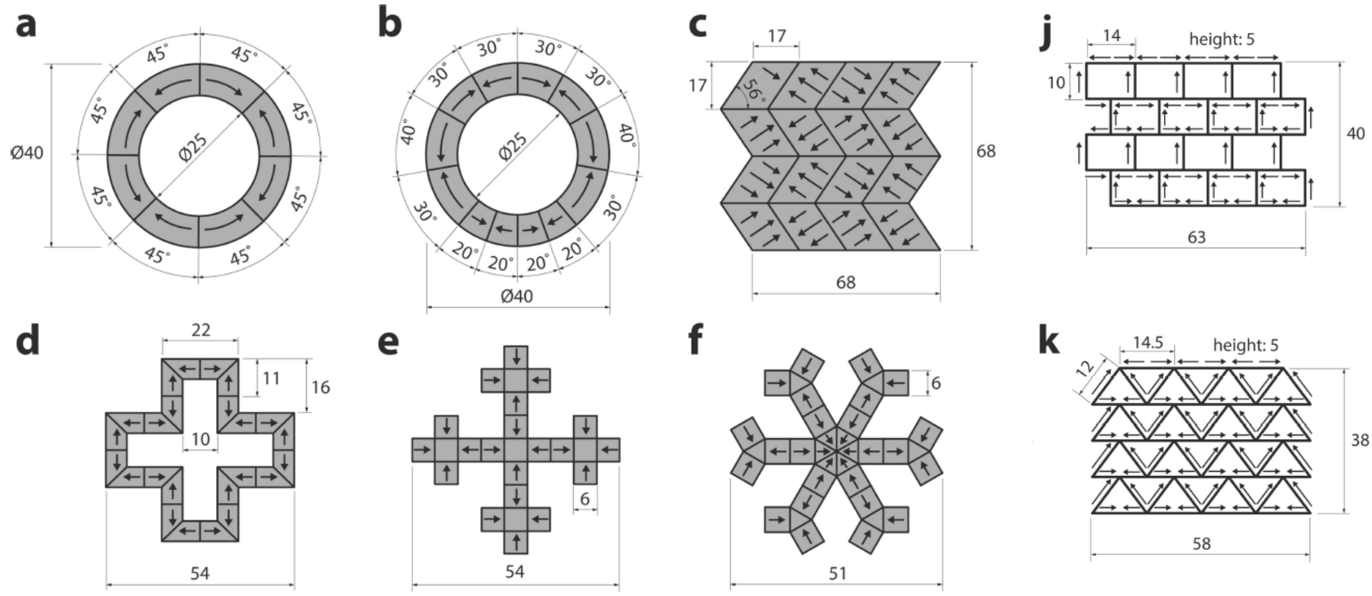

__Figure 4.17:__ A-F: Printed 2D shapes MIT. J and K: Printed 3D shapes MIT (Y. Kim et al., 2018)

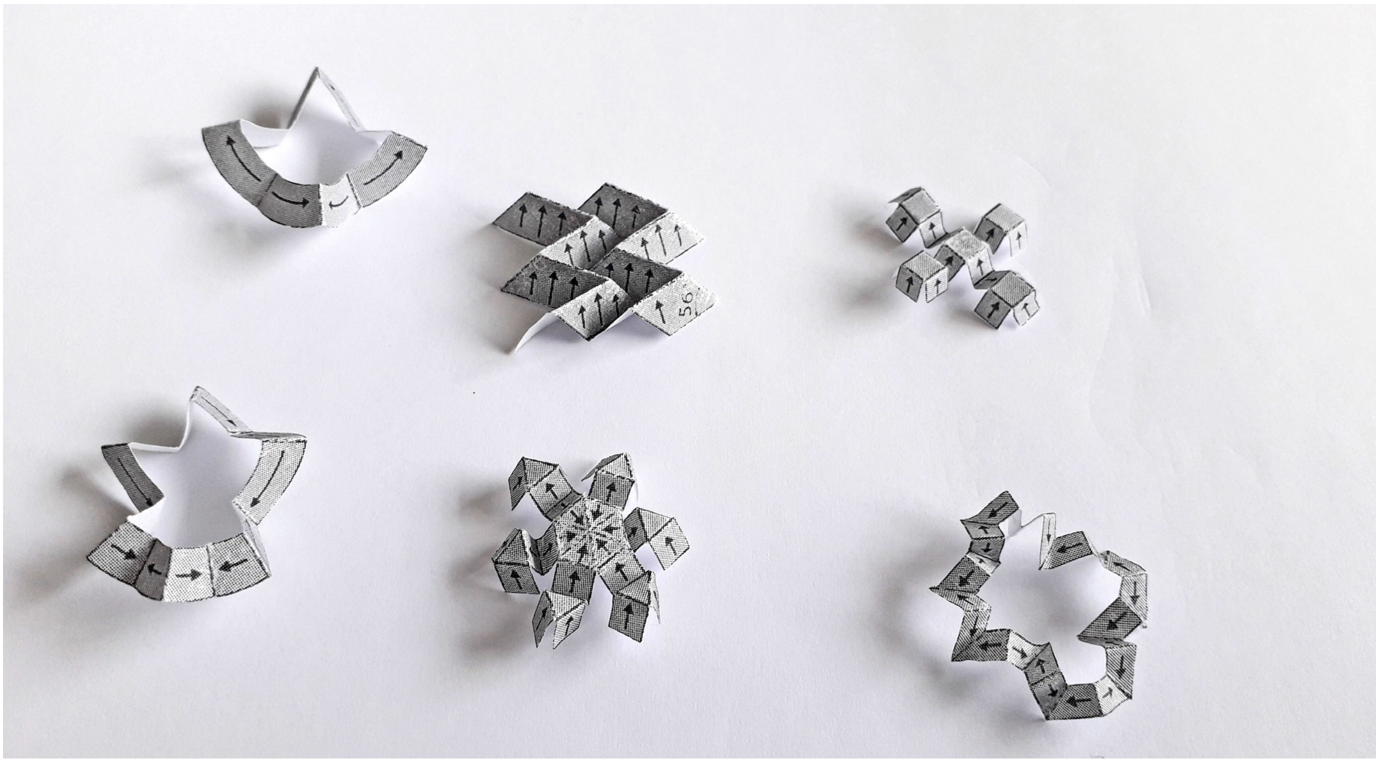

__Figure 4.18:__ Prototypes from the MIT 2D shapes. The arrow represent the magnetic field direction, so all the arrows have to point in the same direction.

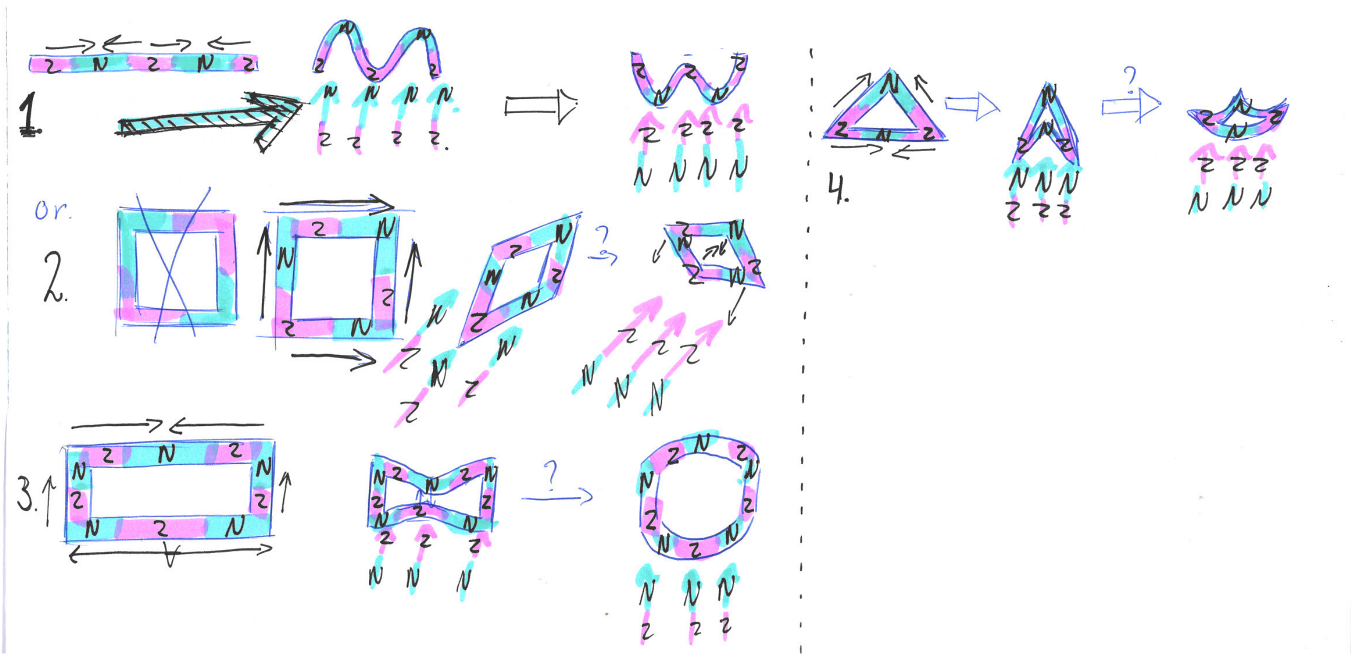

Figure 4.19: *3D MIT shape transformation analysis. The arrows from MIT were translated into North and South poles. While keeping the shape in the same orientation, two magnetic field directions were thought out.*

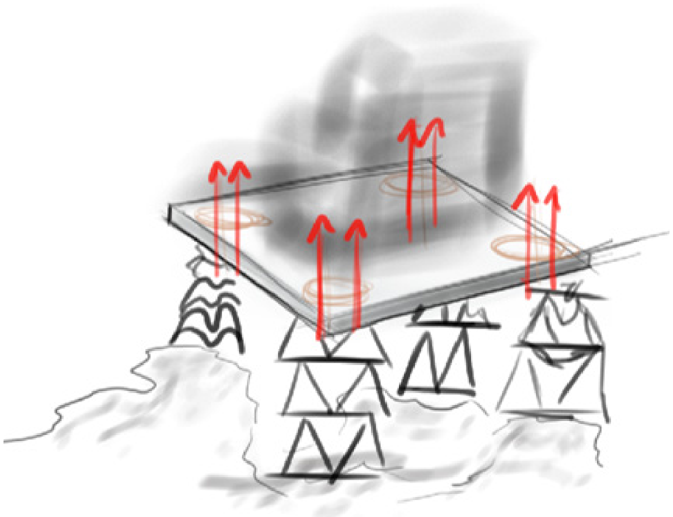

Figure 4.20: *Surface levelling mechanism. Every 3D mechanism is individually controlled by its electromagnet.*

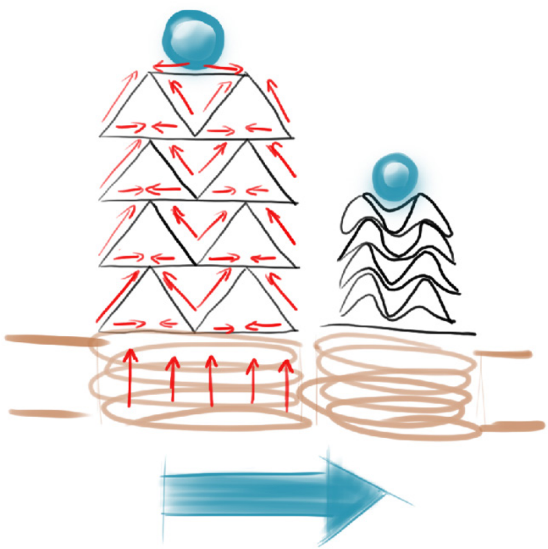

Figure 4.21: *The elevator or catapult. A magnetic field can pull the structure down. If the magnetic field strength is gradually weakened or strengthened, the movement can be as one of an elevator. If the magnetic field on the other hand is flipped in an instant or released, and the elastomer itself pops back into shape, objects on top might be fired away as a catapult.*

The last brainstorm was done with only using MITs 'M'-shape that was part of the proof of principle shape. The input for the brainstorm was how the shape could be used as a product that showed all the magnetic soft material characteristics. The ideas can be found in Appendix J. The most interesting ones were the mixer tap (figure 4.13), a reversible spoon for tubes (figure 4.22) and a cleaning tool for hard to enter spaces (figure 4.23).

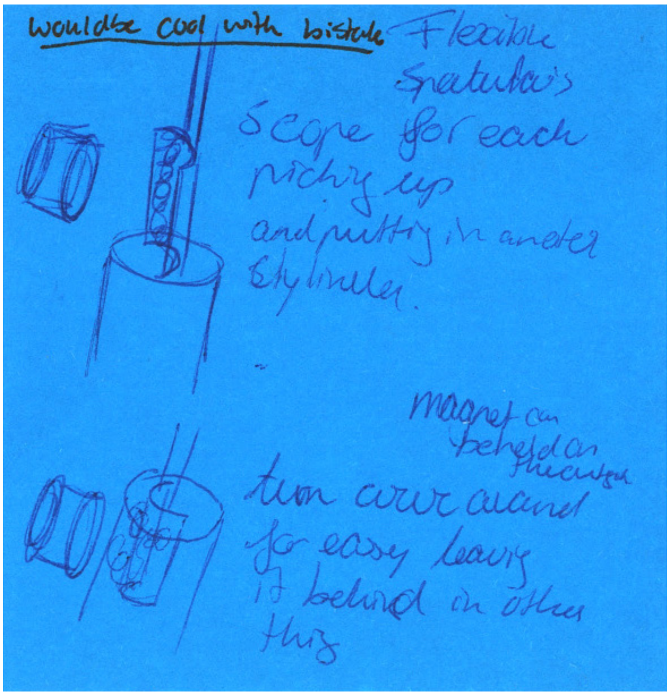

Figure 4.22: Reversible cylindrical spoon. To scoop pastes out of tubes. Put the spoon in another tube and flip the spoon inside out by flipping a permanent magnet on the outside of the tube to release the paste easier to the wall of the tube.

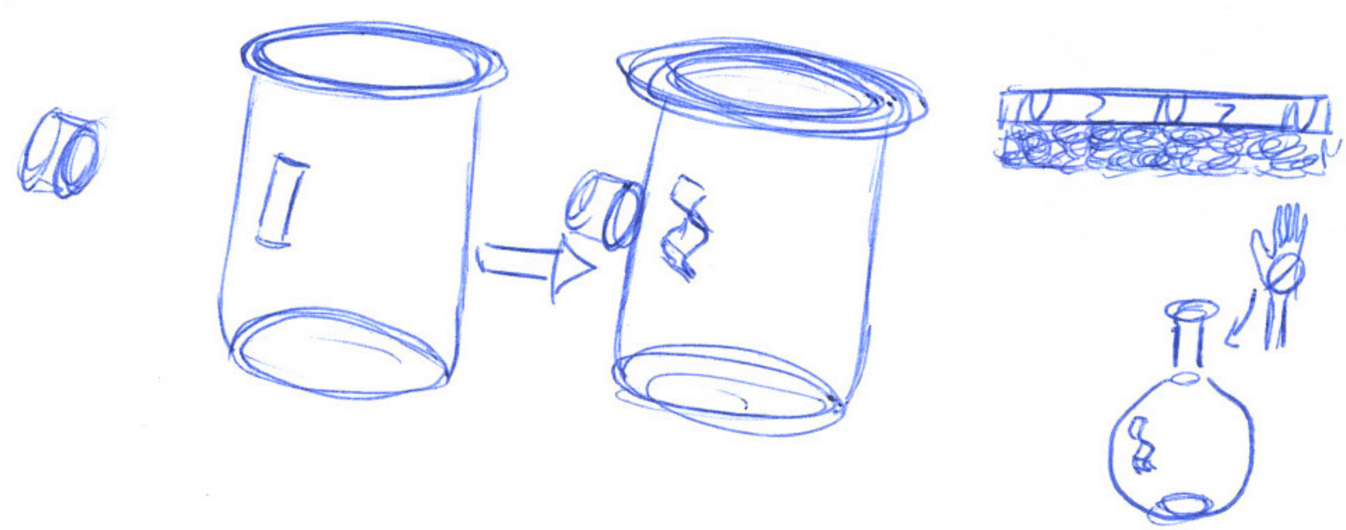

Figure 4.23: The cleaning tool for hard to enter spaces. Through the elastomer, the material can be rolled up and placed in spaces where the hand cannot come. By a permanent magnet, the wiping can be controlled on the outside and by moving the magnet closer and further away the magnetic soft material can contract and relax to create a scrubbing motion.

4.3.3 Choice for demonstrator

In this chapter, the chosen idea is presented that will be turned into a concept. The generated ideas can be found in Appendix J. Many ideas or idea directions were interesting, but due to a lack of knowledge, mostly on the required magnetic field strength, forces and loads, were not chosen because it is not known if they can be realised yet. For selected ideas and idea directions that are interesting for future research see chapter 4.5.

The idea that will be further developed into a concept is the cleaning tool for hard to enter spaces (figure 4.23), because:

- It can be made with the 'M'-shape as the working mechanism
- It is small, under the 6 centimetres
- It does not carry a high load
- The elastomer is used to get it into hard to enter spaces.
- Through cleaning and wiping motions the product is constantly moving
- The scrubbing motion of the 'M' is determined by the closeness of the magnet and shows the reversible and dynamic movement
- It is remotely controlled from the outside

Therefore it checks all the characteristics and boundaries.

It also has extra advantages, like that it could be demonstrated in a translucent vase where the functioning of the magnetic soft material can be observed and played with.

This idea is further elaborated into a concept in the next chapter.

4.4 Conceptualization

The chosen idea of the cleaning tool will be further developed into a concept. Because the 4D printer set-up is not fully developed, the concept will not be fabricated at the end of this project.

4.4.1 Use scenario

In figure 4.24 the use scenario of the cleaning tool can be seen. Due to the elasticity of the silicone base of the magnetic soft material, the tool can be rolled up by hand and be placed through the narrow opening of the vase. A permanent magnet on the outside is used to move the cleaning tool on the inside of the vase. Because of the flexible material and the pulling of the magnetic field of the permanent magnet, the magnetic soft material that is touching the glass, takes over its curve for optimal surface contact. Due to the magnetization pattern in the tool, the flat shape can contract into an 'M' and many variants within, according to the magnetic field strength. This creates a surface texture to scrub the inside of the vase with.

The cleaning tool can be easily taken out of the vase, by using tweezers with a small permanent magnet attached to one side. This magnet, can attract the magnetic soft material and be held to pull out of the vase. Again the flexibility of the magnetic soft material helps to get out of the vase.

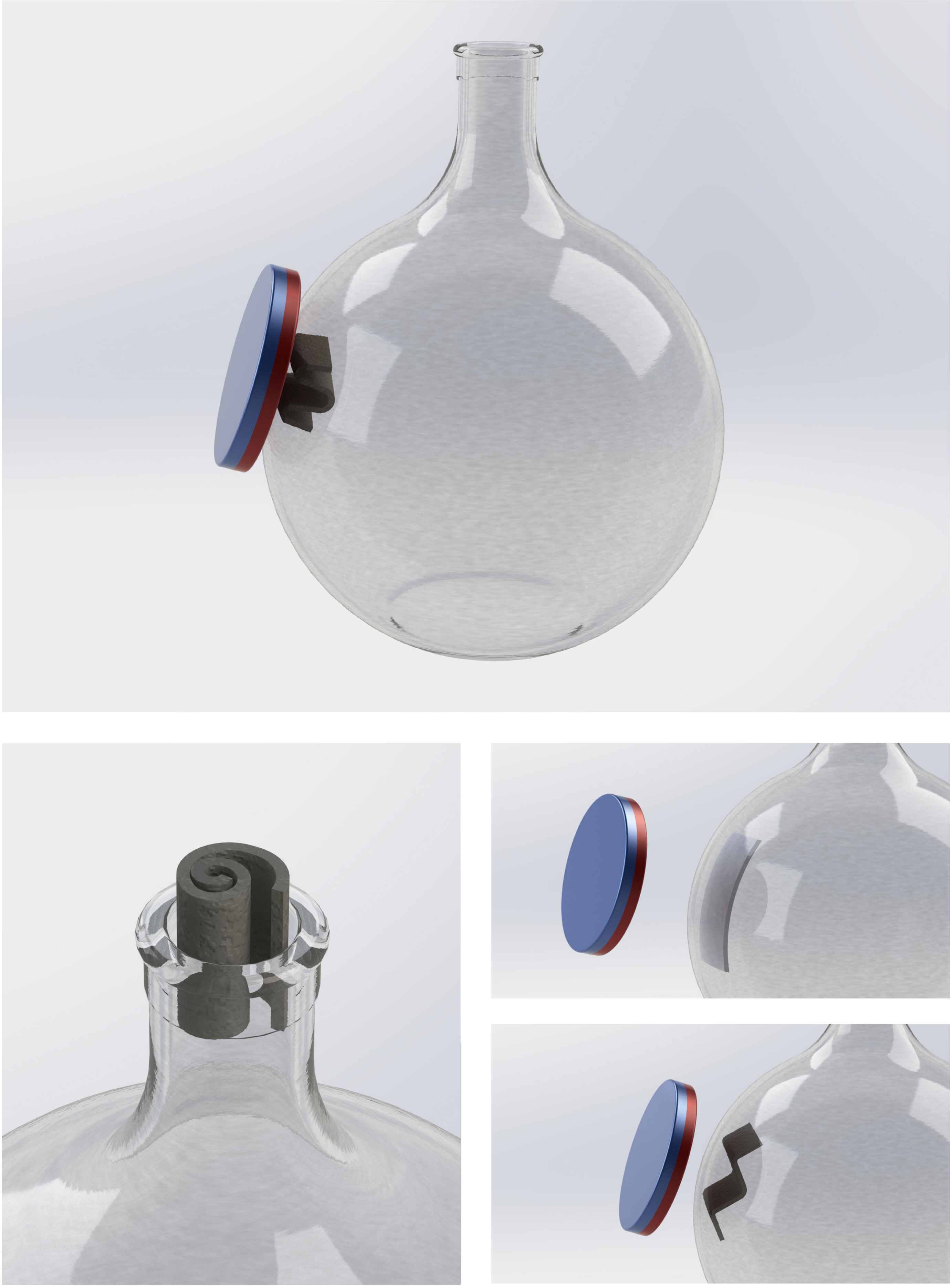

Figure 4.24: *The cleaning tool is rolled up and put inside a vase. Depending on the distance of the magnet to the material, the strength of the shape transformation differs according to the magnetic field strength. The position of the magnet relative to the vase moves the tool over the inner surface of the vase.*

4.4.2 Shape transformations

To create the M-shape with magnetic soft material, it has an alternating magnetization pattern going North-South-North-South-North. When the North pole of a permanent magnet is facing the cleaning tool, the two South poles (being the opposite to the North pole) get attracted, while the three North poles of the tool are repelled (see figure 4.25).

To print this M-shape transforming magnetic soft material, a schematic is drawn in figure 4.26. It is supposed that it will be printed with an electromagnet at the nozzle and that it, therefore, can be printed in a continuous line as explained in figure 3.41. So the rectangle is printed with a continuous zigzagging line of ink over the length of the shape, the direction of the electromagnet is switched four times (figure 4.26 white arrows). In these four regions, the magnetic fields of the magnetic particles lay in opposite directions (figure 4.27). The dimensions of the rectangle in the user scenario renders (figure 4.24) are 30 x 13 mm. This could be larger depending on the object it is used for. The 30 mm is 10 mm shorter than the 40 mm of MIT (Y. Kim et al., 2018) and therefore is probably possible.

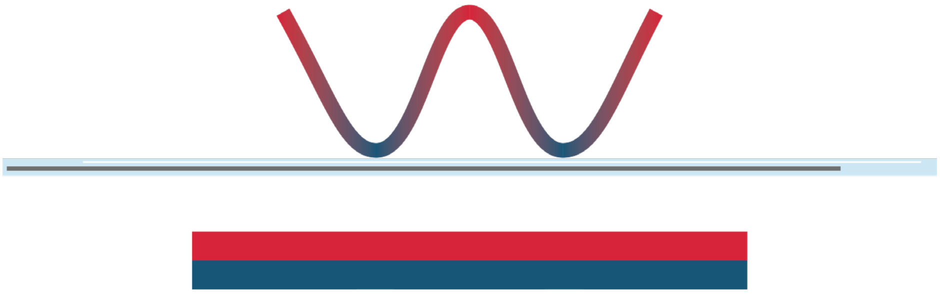

Figure 4.25: *The South poles of the magnetic soft material are attracted to the North pole of the magnet that is facing the material. Making the material contract into an M-shape.*

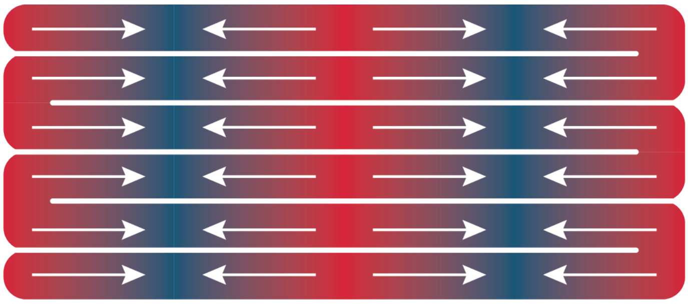

Figure 4.26: *The schematic representation of the print process of the M-shape. The path is the path that the printer head travels, the red and blue colours the magnetization pattern and the arrows the direction of the magnetic field of the electromagnet attached to the nozzle during the print process.*

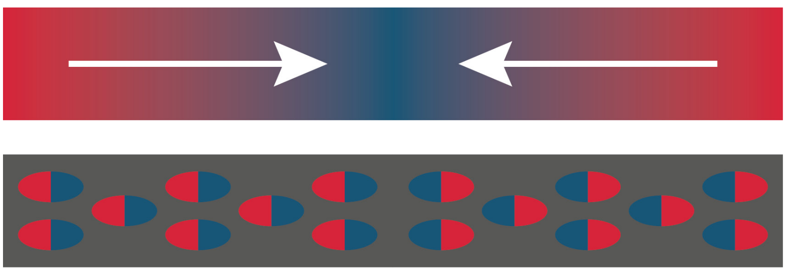

Figure 4.27: *How the particles are oriented in the ink in comparison with the schematic North and South poles of the magnetic soft material*

4.4.3 Assembly

The magnetic soft material from itself will not glide well of the glass, because it has a rubber texture from the silicone. This was also seen in activating the proof of principle, that was put into water to weaken the resistance. The silicone does also not absorb water or soap to clean with. Therefore it is necessary to connect some fabric to the rectangle of magnetic soft material. This could be connected with flexible silicone glue, but that will make it hard to separate the fabric from the magnetic soft material, to for example wash the fabric after cleaning. It is also hard during the recycling process by hand or by shredder. Therefore connectors were designed that could keep the two materials together, without glue and could be easily taken out by hand or break in a shredder (figure 4.28). The small connectors can be 3D printed and with the circular side be pushed, first through the fabric and then through the silicone. The elasticity of the rubber keeps the connectors in place (figure 4.29). The connectors were only placed at the magnetic North poles of the material so that they will not be in the way when the South poles are attracted to the cleaning and will not scratch the glass.

Figure 4.28: *Magnetic soft material (grey) with fabric (brown) that is connected with the designed connectors (white)*

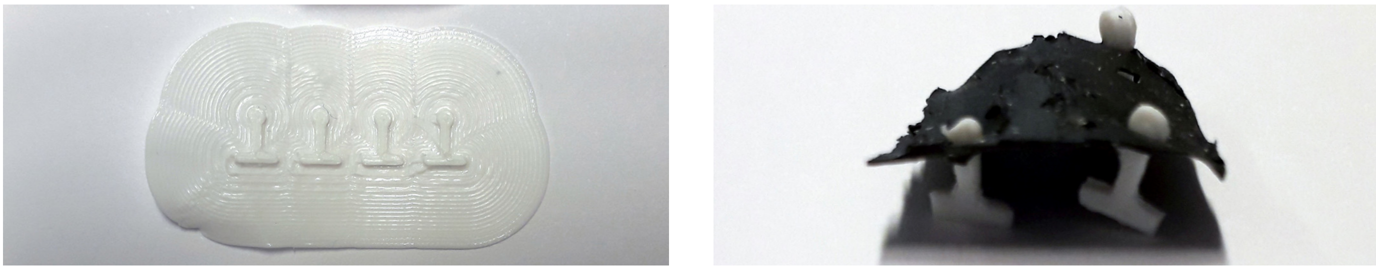

Figure 4.29: *Prototype of the designed connectors. The best size had a thickness of 0.8 mm for the straight parts and a diameter of 1.6 mm for the circle on top.*

4.4.4 Final thoughts

I think that this concept can show all of the unique characteristics of the magnetic soft material, like the fast, remote less and reversible shape transformations. It can also help people who are newly introduced to magnetic soft materials to understand how it works with its simple shape. Because the material is not printed yet and there is not much known about the forces, the thickness of the magnetic soft material and the fabric are not determined yet, as well as the magnetic field strength of the permanent magnet that controls the shape transformations.

4.5 Future idea directions

During the idea generation, interesting ideas have been introduced. However, they were not further explored or developed during this project, because they did not fulfil all the criteria or knowledge is lacking on the behaviour or capabilities of magnetic soft materials. Six idea directions have been selected that can be used for future projects and were not fully represented in the applications found in the literature review. Therefore, biomedical products are not mentioned, because there is already a focus on that domain regarding magnetic soft materials. For example target drug delivery (Goudu et al., 2020; Hu et al., 2018), but also ideas like heart-pumping devices.

Haptics
Haptics can be an interesting domain for the applications of magnetic soft materials. They are safe by not heating up and could convey information to the user by their shape-changing capability, Here the influence of magnetic fields on health could be researched more and if a shape change can be felt on the skin. Some ideas in the haptics domain can be seen in figures 4.8 and 4.9.

Personalized fits
Products with personalized fits provided by magnetic soft materials could be interesting for the same reason as the haptics and have the same knowledge gap on the health and strength of the transformed shape. Through texture change caused by the local placement of magnetic fields, products could be altered to fit the user. An idea can be seen in figure 4.6.

Texture change
Texture change was one of the most interesting idea directions from the creative session. It can have mechanical functions, like changing surface roughness, but it can also be used for dynamic product experiences, where a characteristic of a cute sphere transforms into a scary sphere with spikes. Texture change can also be used for haptic experiences, like in the study of Ion, Kovacs, Schneider, Lopes, and Baudisch (2018).

Replacement of vulnerable mechanical parts
This idea is mostly inspired by the mixer tab of figure 4.13. It would be interesting to see if due to the dynamic shape transformations of the magnetic soft material, it can be used in places where corrosion or calcification often damage the current system. Here especially the force that magnetic soft materials can resist is important to know for replacements.

Milli devices and scaffolding
Another idea direction is milli devices and scaffolding. This idea is based upon the brainstorm session with MIT structures (figures 4.20 and 4.21) and the autofocus and optical image stabilization in figure 2.4 from the literature review. Because magnetic soft material can create functional structures and movements out of one piece, it could be used for functional units in smaller devices where mechanical parts are not possible or too expensive to make.

Toys
The last idea direction is to make or incorporate magnetic soft materials in toys. The shape transformations can spark wonder and play. Some ideas for toys can be found in Appendix J.

5. Conclusion

Magnetic soft material is a recently developed material, that can change its shape by aligning the fields of its magnetic particles with an external magnetic field. This graduation project has addressed literature, researched and identified multiple ink composition factors (see figure 3.2), started a setup for a 4D printer system, broadened the existing solution space by adding new product directions and succeeded in making working magnetic soft material.

The parameters that have been studied in this research were mixing orders, ingredient ratio's, the influence of ingredients on the ink, which particles had the best magnetization and how the ingredients influenced the viscosity of the ink. The ink viscosity is one of the most important factors for the 4D printing process of magnetic soft materials. A too high viscosity ink cannot be extruded and can cause different problems, like expanding tubes and stopping motors. The current viscosity of the magnetized NdFeB12,5%SiO_M lays out of reach for the 3D printer with a resting viscosity of 56.000 Pa.s. The viscosity that seems to be the current limit of the 3D printer system is 11.000 Pa.s of the Fe31,25%SiO_nM ink. For a continuation of this research, the current 3D printer system should be reconsidered.

Even though the printer setup was not fully developed at the end, working magnetic soft material was made with a syringe and a ring magnet. It had the shape of a strip with an alternating magnetization pattern of North-South-North. It could stand up like a bow or lift the ends of the strip as its inspiration the 'M'-shape made in the research of Y. Kim et al. (2018). The strip also moved immediately as its position relative to the external magnet was changed. This strip proved that magnetic soft material can be made and that the research at the faculty of IDE could be continued.

The ideation of this project resulted in interesting idea directions for magnetic soft material applications to inspire researchers and designers. These directions were haptics, personalized fits, texture change, replacement of vulnerable mechanical parts, milli devices and scaffolding, and toys. Those domains broadened the currently existing domains in which biomedical applications currently seem the most promising with for example target drug delivery. Also, a concept for a demonstrator was developed that could show all the unique magnetic soft material characteristics, fast, remote, reversible and heatless shape transformations to inspire designers and researchers. It was a cleaning tool that could reach hard to enter spaces. The shape transformation was based upon the 'M'-shape from MITs research and the proof of principle from this research that showed that this simple shape transformation could work. Current limitations for the generation and conceptualization of ideas are uncertainties of how strong the magnetic field should be for shape transformations, how strong the magnetic field may be without affecting the health of its user and the environment, and how much force the transformed shapes can withhold. These still form a knowledge gap that should be addressed in later research. As well as how different shapes and sizes of the material influence these factors.

This graduation project formed the start of the research into magnetic soft materials at the faculty of IDE. Although the first parameters have been studied and the material was made successfully, many parameters and questions have not been explored yet. The research into magnetic soft materials has just begun and still needs more work to integrate them in future applications on a large scale.

5.1 Recommendations

The main recommendations are already stated in the conclusion. However, during the project, other topics and questions appeared as well, but could not be addressed. This chapter lists a few of those.

Recyclability

One of the main points is the recyclability of magnetic soft material. It is important to think of how the material can be recycled, especially because it contains NdFeB particles which are rare earth materials. In the standard recycling process, the material will probably be incinerated, because the silicone and the magnetic particles cannot be separated easily. The magnetic particles will then probably be lost. It is important to think about these questions before products are made from this on a large scale. Without the knowledge of the to recycle them, many rare earth materials could be lost.

Expense NdFeB and SmCo particles

During this project, the costs for NdFeB and SmCo particles were a few hundred euros for 100 grams. This could be due to the small amounts that were ordered. However, the costs are very high to use the material in normal consumer products (biomedical products excluded). It might be worth looking into alternative magnetic particles, that are also no rare earth materials. So the price can be lower and rare earth materials will not get lost due to wrong recycling.

The durability of magnetic soft material

The durability of moving silicone, the magnetization pattern and the aging of the magnetic particles might also be researched to get a sense of how long magnetic soft materials will last and if something in the recipe or design should be adjusted to that. This point was not yet placed in the factors of making magnetic soft materials. It can also be interesting to see if the material fails on certain locations which can be addressed to the magnetization pattern. For example, where two similar magnetic poles face each other and push each other away.

5.2 Reflection

I started this project because I was interested to learn more about materials and material research. After all, it was a topic I had always been interested in. I have learned a lot during this project. In this section, I will reflect on the five personal learning goals that I had at the start of the project.

Get more experience with material testing, optimizing and manufacturing techniques

This learning goal went well throughout the project. I have had instructions in multiple material testing devices, which I have used throughout the project. The most emphasis has been on the rheometer with which the viscosity of the ink was measured. I have also gained a deeper understanding of 3D printers and G-code. I feel that I have grown in setting up and structuring the test plans for the experiments that were performed during this project.

Developing my prototyping skills

I think that this learning goal went well, even though I first did not see it myself. I have developed many prototypes by using syringes and ring magnets as a manual 4D printer. I had also made multiple iterations of the screw connection for the electromagnet to make sure it fits.

Learning more research (project) based skills

Here I have learned a lot. Especially how research projects differ from design projects or design courses. At the start of the project, my supervisory team and I had fixed some outcomes of this research project. However some steps took longer than I had planned, tests did not go well the first time or showed other results than were expected, etc. This stressed me out throughout the project because the time to reach the goals got smaller. I have talked about this with the supervisory team of my project and they told me that this was how research worked and we adjusted the outcomes for the project. I was not accustomed to this because of the hard deadlines during other courses. The nature of research might have taken the longest to learn during this project.

Learning to start a design project from a given material

This part of the project was challenging. In my first planning, I thought that I would have made working magnetic soft material at the midterm, before starting the design process. This was not the case and the proof of principle was accomplished in the last test of this project. Therefore lots of facts about magnetic soft materials were unknown. This made it harder to think of what was possible and what was not. It took some time to get started with this process. I am happy with the list of future idea directions for magnetic soft materials to show more potential for the material. The idea for the demonstrator was simple but could work and can clearly show the characteristics of the magnetic soft material to people who want to understand it as well as showing a function for the material.

Learning scientific discussion and presentation skills

This skill has also improved over the project. In my midterm evaluation, I had stated that many of the outcomes during the first tests had been compared and judged by observation. This was no scientific way to show and present outcomes. It was also hard to give others an impression of the results. This had improved after this statement. For example, the viscosity of all the new inks got measured and compared with each other.

During this graduation project, I have also helped with writing a paper. From that, I have also learned a lot about the publication process in the scientific field.

Acknowledgements

I want to thank the many people who have helped me throughout my graduation project. Without their help, this project would not have come where it is now.

At first, I want to thank my supervisory team for all their help, support, feedback and reminding me about the nature of a research project.
Sepideh: for giving me the opportunity for this project and the research project in which I had the opportunity to learn more about material research. Also for always helping me with both technical project problems and mental support.
Zjenja: for all your help on the 4D printer system and for calling my attention back to the bigger scope of my project, by not losing focus on the project structure.
Andres: for developing and making the electromagnet, finding a magnetizer and all your other input in the problems with the 4D printer system.
Houman: for having helped me with almost every test and waiting throughout all the scale calibrations, hoping that it would not give an error.

I would also like to thank Anton Lefering from the Reactor Institute, who has magnetized multiple syringes of ink as well as performed the quantitative measurements.

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Appendix

A. Project brief

Designing magnetic soft materials for 4D printing — project title

Please state the title of your graduation project (above) and the start date and end date (below). Keep the title compact and simple. Do not use abbreviations. The remainder of this document allows you to define and clarify your graduation project.

start date 15 - 03 - 2021 — 31 - 08 - 2021 end date

INTRODUCTION **

Please describe, the context of your project, and address the main stakeholders (interests) within this context in a concise yet complete manner. Who are involved, what do they value and how do they currently operate within the given context? What are the main opportunities and limitations you are currently aware of (cultural- and social norms, resources (time, money,...), technology, ...).

Magnetic Soft Material is a new type of shape memory materials that is currently being developed. Shape memory materials have the ability to change their shape to a 'programmed' shape, when they are triggered by an external stimulus. Where in more common shape memory materials this stimulus is a temperature change, in magnetic soft materials this stimulus is a magnetic field.

The magnetic soft material consists of an elastomer with incorporated magnetic particles. The magnetic fields of these particles are positioned in different magnetization patterns that determine the shape change of the material. When the magnetic soft material is placed in an external magnetic field the magnetic fields of the particles will align with it. In doing this they pull the elastomer along with them and the composite assumes a new shape. (see figure: 1)

Magnetic soft materials have some unique qualities in comparison with heat activated shape memory materials. They can be remotely activated, require no heat, can change their shape in multiple ways (due to the direction of the magnetic field), and transform quick from one shape to another in a short time. This makes magnetic soft materials interesting and usable for other locations and applications than heat activated shape memory materials. Some already explored applications for magnetic soft materials are biomedical devices, soft robotics, actuators and metamaterials, because of these unique qualities. However, because of them being a new material, there is not a lot of literature available yet on applications and there are still many applications to discover.

Magnetic soft materials are fabricated by first preparing the magnetic soft material ink to print structures with. After the ink is prepared it is loaded into a 3D printer of which a permanent magnet or an electro magnet is attached around the nozzle. While the object is printed, the magnetic particles align with the magnetic field of the nozzle, to create the magnetization patterns in the material. (see figure: 2) The applied magnetic field during printing adds an extra dimension to the printing process. That is why printing will be called 4D printing. The details of this process and more precisely how certain parameters influence the performance of magnetic soft materials is still largely unknown. Like for example the ink compostion, mixing order, printing speed, nozzle diameters or the magnetic field strength caused by the magnet attaced at the nozzle. This makes the material inaccessible for designers to work with and more research is needed on the fabrication process and the applications of magnetic soft materials before more people can start experimenting and designing with them.

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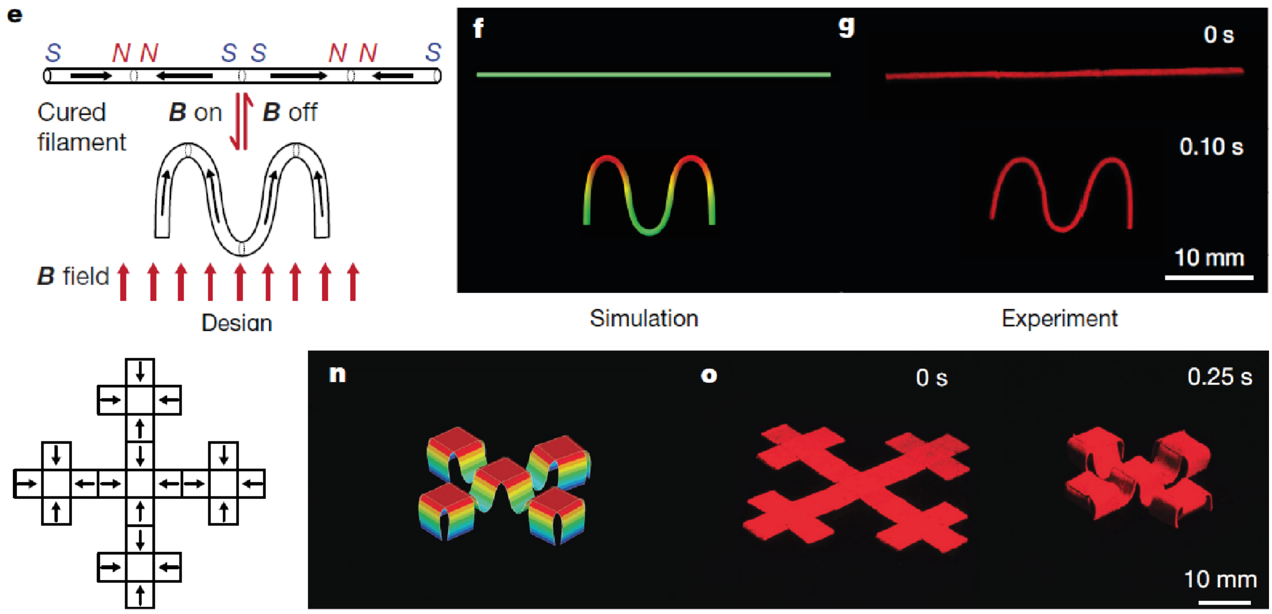
Initials & Name SJM van Vilsteren 4842 Student number 4543483

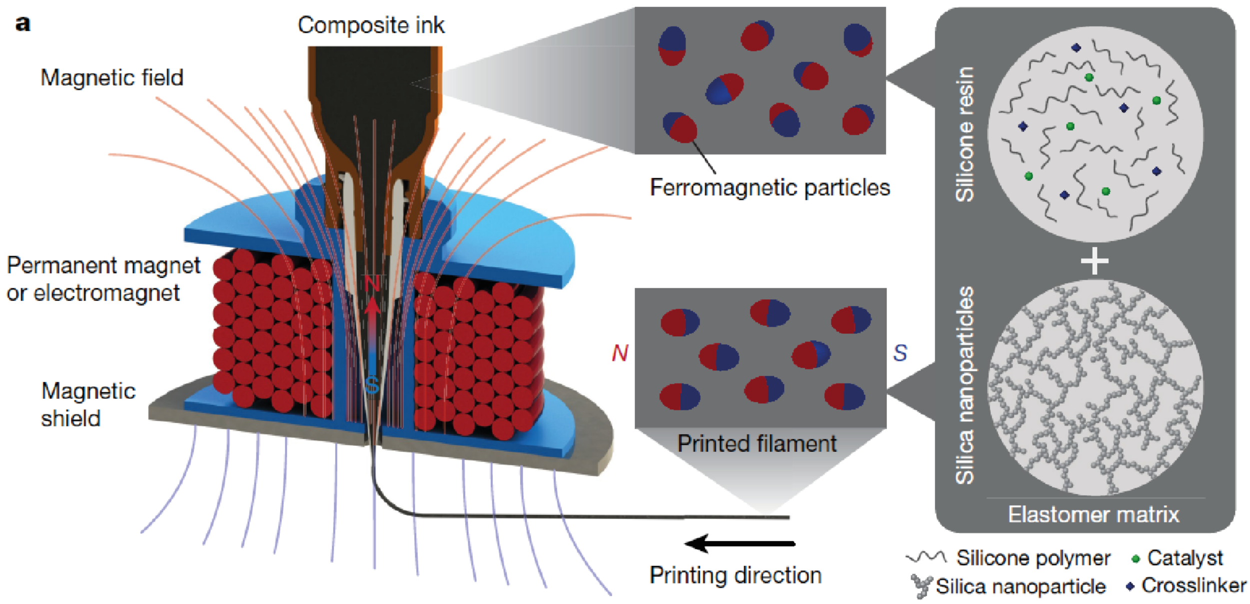
Title of Project Designing magnetic soft materials for 4D printing

introduction (continued): space for images



Kim, Y., Yuk, H., Zhao, R., Chester, S. A., & Zhao, X. (2018). Printing ferromagnetic domains for untethered fast-transforming soft materials. Nature, 558, 274-279. doi:10.1038/s41586-018-0185-0

image / figure 1: Working principle of magnetic soft material (Y. Kim, Yuk, Zhao, Chester, & Zhao, 2018)



Kim, Y., Yuk, H., Zhao, R., Chester, S. A., & Zhao, X. (2018). Printing ferromagnetic domains for untethered fast-transforming soft materials. Nature, 558, 274-279. doi:10.1038/s41586-018-0185-0

image / figure 2: 4D printing of magnetic soft material (Y. Kim, Yuk, Zhao, Chester, & Zhao, 2018)

PROBLEM DEFINITION **

Limit and define the scope and solution space of your project to one that is manageable within one Master Graduation Project of 30 EC (= 20 full time weeks or 100 working days) and clearly indicate what issue(s) should be addressed in this project.

Magnetic soft material is a new material, unknown to designers. Lots of parameters of this material are unknown, like the material composition, ink preparation, manufacturing processes, etc. Not a lot of research is done about magnetic soft materials (also in 4D printing them); only in the last 5 years a number of papers have been published (Kim, Yuk, Zhao, Chester, & Zhao, 2018; Ma et al., 2020). So, much more detailed research is needed to enable and inspire designers to work with magnetic soft materials.

The first topics that have to be investigated are how certain parameters in the material composition, the material properties, the material ink preparation process and the manufacturing process (eg. 4D printing) influence the material performance and its shape memory behaviour.

Next to that, there is also a lack of guidelines or inspirational objects and projects made with magnetic soft materials, that show the possibilities and advantages of this material to designers. These demonstrators could introduce the new material to designers and the guidelines could lower the threshold for designers to start working with this new material.

Kim, Y., Yuk, H., Zhao, R., Chester, S. A., & Zhao, X. (2018). Printing ferromagnetic domains for untethered fast-transforming soft materials. Nature, 558, 274-279. doi:10.1038/s41586-018-0185-0

Ma, C., Wu, S., Ze, Q., Kuang, X., Zhang, R., Qi, H. J., & Zhao, R. (2020). Magnetic Multimaterial Printing for Multimodal Shape Transformation with Tunable Properties and Shiftable Mechanical Behaviors. Applied Materials and Interfaces. doi:10.1021/acsami.0c13863

ASSIGNMENT **

State in 2 or 3 sentences what you are going to research, design, create and / or generate, that will solve (part of) the issue(s) pointed out in "problem definition". Then illustrate this assignment by indicating what kind of solution you expect and / or aim to deliver, for instance: a product, a product-service combination, a strategy illustrated through product or product-service combination ideas, ... In case of a Specialisation and/or Annotation, make sure the assignment reflects this/these.

The aim of this project is to develop magnetic soft material, identify and investigate the parameters of the ink and 4D printer settings that influence the performance of the material, through systematic testing. The results will be translated into a demonstrator that shows the possibilities opened up by these materials. It is equally important to propose a set of guidelines for designers on how they can get started and design with magnetic soft materials.

In this project, research will be done on the parameters of the composition of the ink of the magnetic soft material as well as on the 4D printer setting parameters. Like for example the material composition (materials and ratio's), mixing orders, printing speed, the nozzle diameter, magnetic field strength during the printing, printing patterns, etc. This will be done to see which parameters have a big influence on the performance of the magnetic soft material. It also helps to understand which parameters could be changed to design a specific shape change and see what are the possibilities.

The outcomes of this project are a 4D printed magnetic soft material demonstrator and a set of guidelines for designers. The purpose of the demonstrator is to show designers the potential of magnetic soft materials and how they could be used in an inspiring design. The guidelines are for designers and researchers who want to work and make objects with magnetic soft materials.

The demonstrator will first be focussed on creating a single hinge mechanism of the magnetic soft material. After that is achieved the crated structures will become more complex like for example grippers or micropumps.

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PLANNING AND APPROACH **

Include a Gantt Chart (replace the example below - more examples can be found in Manual 2) that shows the different phases of your project, deliverables you have in mind, meetings, and how you plan to spend your time. Please note that all activities should fit within the given net time of 30 EC = 20 full time weeks or 100 working days, and your planning should include a kick-off meeting, mid-term meeting, green light meeting and graduation ceremony. Illustrate your Gantt Chart by, for instance, explaining your approach, and please indicate periods of part-time activities and/or periods of not spending time on your graduation project, if any, for instance because of holidays or parallel activities.

start date 15 - 3 - 2021 31 - 8 - 2021 end date

	Calender week	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	Total
	Project week	1	2	3	4	5	6	7	8	9	10		11	12	13	14	15	16	17	18	19			20	21	22	
	Working days this week	5	5	4	4	5	5	4	4	3	5		4	5	5	5	5	5	5	5	5			5	5	2	100
1. Ink preparation	Literature study magnetic shape memory materials											B										B	B				840 h
	Initial experimenting MSM											R										R	R				
	Learning about material testing											E										E	E				
	Parametric study MSM ink											A										A	A				
2. Ink printing	Learning about 4D printing											K										K	K				
	Preparing and making the 4D pringing setup																										
	Parametric study MSM 4D printing																										
3. Developing guidelines	Experimenting with MSM																										
	Developing MSM design guidelines																										
	Listing unique characteristics and limitations of MSM																										
4. Demonstrator ideas	Idea generation applications MSM																										
	Choice of Concepts for demonstrator																										
5. Demonstrator development	Demonstrator concept development																										
	Demonstrator prototyping																										
	Demonstrator testing																										
	Finalizing design guidelines																										
6. Project documentation	Documentation of project process and results																										
	Presentation																										
	Poster																										

The Gantt Chart above shows the general planning of my graduation project. I first start with the experiments about the ink composition and preparation (1), together with an ongoing literature study about magnetic shape memory materials. This graduation project succeeds my research project on magnetic shape memory materials, for which I have already done a large part of the literature study on magnetic soft materials (MSM) and the first initial experiments. I will simultaneously start with research into 4D printing MSM and preparing the printer setup (2). The parametric study of the 4D printing will come later in the process after sufficient experience about the MSM ink has been gained. From these parametric studies the insights will be used to experiment in making some MSM objects and based on this a start will be made with creating the design guidelines (3). In the next phase the unique characteristics and limitations of MSM will be written down to serve as a part of the criteria for the demonstrator that will be developed in the following phase. In this stage (4) new ideas will be generated on the capabilities of MSMs. One idea will be selected to be further developed in a demonstrator object (5), so as to obtain extra insight for creating the design guidelines. Everything needs to be documented, which will be an ongoing task throughout the entire project (6). In the last four weeks the poster and presentation will be made to finish the project.

In the period of my graduation there are seven days that are non working days on the TU Delft according to the TU Delft calender due to national holidays. These days are: 02/04, 05/04, 27/04, 05/05, 13/05, 14/05, 24/05. I will also take tree weeks of the project to reflect on my work or go on a holiday. The important dates are:
-Kick-off meeting: 15-03-2021
-Mid-term meeting: 17-05-2021
-Green light meeting: around calendar week 29
-Graduation ceremony: around calendar week 35

Initials & Name SJM van Vilsteren 4842 Student number 4543483

Title of Project Designing magnetic soft materials for 4D printing

MOTIVATION AND PERSONAL AMBITIONS

Explain why you set up this project, what competences you want to prove and learn. For example: acquired competences from your MSc programme, the elective semester, extra-curricular activities (etc.) and point out the competences you have yet developed. Optionally, describe which personal learning ambitions you explicitly want to address in this project, on top of the learning objectives of the Graduation Project, such as: in depth knowledge a on specific subject, broadening your competences or experimenting with a specific tool and/or methodology, ... Stick to no more than five ambitions.

I was interested in materials, the tools and techniques to make things with them, even before I started with my bachelor. During my bachelor and master, I was very interested in the material and manufacturing courses and projects. In a project about extrusion blow moulding, my team and I played with the unique characteristics of this manufacturing method to come up with product concepts. This was very fun and inspiring to do. However, during my course Advanced Embodiment Design, I had to find out certain limitations due to the characteristics of a material. To set up a test for it and be able to qualify my results were a challenge.

Because I was interested in materials I wanted to learn more about them, so I started with a research project on Magnetic Shape Memory Materials during my electives. During that project, my time was mainly spent on writing a literature review about Magnetic Shape Memory Polymers and Elastomers, how they work, how they are made and what makes them unique and what applications are known. Because these materials are quite new, no one ever has experimented with them at TU-Delft, so there was no time during the project to start to make or to print the magnetic soft materials. Actual making the magnetic soft ink and printing it into an object is the next stage of the project; hence, I decided to extend my research project to my graduation project. This way I could learn more about the fabrication, manufacturing, testing and designing with specific materials. These things are translated in my personal learning goals during this project:

- Get more experience with material testing, optimizing and manufacturing techniques
- Developing my prototyping skills
- Learning more research (project) based skills
- Learning to start a design project from a given material
- Learning scientific discussion and presentation skills

FINAL COMMENTS

In case your project brief needs final comments, please add any information you think is relevant.

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B. Test Documentation

This Appendix contains the documentation of all the tests that were performed during this project. The are sorted into five categories. In later tests, multiple categories can be found in one test. In this Appendix, ink names are documented in an older style used in this project. In this table, the old and new names can be compared.

NdFeB12,5%SiO_nM	T8E1 nM
NdFeB12,5%SiO_M	T8E1 M
Fe25%SiO_nM	T6E3 IC
NdFeB25%SiO_nM	T6E3 NdFeB
Fe31,25%SiO_nM	T7E1
Fe37,5%SiO_nM	T6E4
Fe50%SiO_nM	T3E1

B.a Ink development

This part contains the earlier ink development tests in which different ink parameters are explored, like mixing orders, Ecoflex ratio's, vol% or wt% calculation of magnetic particles and the influence of fumed silica on the print stability.

Magnetic Soft Materials: Experiment 1st try

Dr. Sepideh Ghodrat
Houman Yarmand
Sanne van Vilsteren
26-01-2021

First try

For the first try the plan was to make 100 grams of the Magnetic soft material as is described in the MIT paper. For the amounts of the ingredients that were needed we used the weight percentages at the end of the method for preparing the ink (see the yellow marked text in figure 1).

In the MIT recipe they used:

- Ecoflex 00-30 part B
- Fumed silica nanoparticles
- SE 1700 base
- SE 1700 catalyst
- NdFeB microparticles

METHODS

Ink composition and preparation. The magnetic ink was prepared first by blending two silicone-based materials—SE 1700 (Dow Corning Corp.) and Ecoflex 00-30 Part B (Smooth-on Inc.)—in a 1:2 volume ratio. Ecoflex 00-30 Part B, a softer elastomer than SE 1700, was used to achieve the preferred mechanical properties of the composite material. Fumed silica nanoparticles (amorphous, 20–30 nm; US Research Nanomaterials Inc.), which corresponds to 12.5 wt% with respect to Ecoflex Part B, were added to achieve required rheological properties for direct ink writing. After mixing the blend in a planetary mixer (AR-100, Thinky) at 2,000 r.p.m. for 2 min, 20 vol% NdFeB microparticles (287.5 wt% with respect to Ecoflex Part B) with an average size of 5 μm (MQFP-B-2007609-089, Magnequench) were added into the elastomer mixture and then mixed thoroughly at 2,000 r.p.m. for 3 min, followed by defoaming at 2,200 r.p.m. for 1 min. The composite ink was then magnetized by impulse magnetic fields (about 2.7 T) generated by an impulse magnetizer (IM-10-30, ASC Scientific) to impart magnetic polarities to the ferromagnetic particles embedded in the elastomer matrix. Both SE 1700 and Ecoflex 00-30 are platinum-catalysed, addition-curing silicones, so 10 wt% SE 1700 catalyst with respect to SE 1700 base was added into the magnetized ink and then mixed at 2,000 r.p.m. for 30 s before printing. The final concentrations of components were as follows: 21.78 wt% Ecoflex 00-30 Part B, 2.72 wt% fumed silica nanoparticles, 11.71 wt% SE 1700 base, 1.17 wt% SE 1700 catalyst and 62.62 wt% NdFeB microparticles. For imaging purposes, about 2 wt% fluorescent colourants (Ignite PMS 805C, Smooth-on Inc.) were added to this final composition.

Figure 1 The used weight percentages for the calculations for the preparation of the ink are marked in yellow

In our experiment we did not have the SE 1700 and we decided to add the weight percentages of SE 1700 base and catalyst together and replace it by Ecoflex part A. This resulted in the following weight percentages that can be transferred to grams directly:

- 21,78 wt% Ecoflex 00-10 part B
- 2,72 wt% fumed silica
- 12,88 wt% Ecoflex 00-10 part A
- 62,62 wt% iron powder (carbonyl iron: 12)

Method

- We started by measuring the Ecoflex part B
- Then we added the fumed silica
- This was mixed by hand due to a defect mixer
- WE STOPPED

We stopped after mixing the fumed silica with Ecoflex part B. The cause was that the viscosity of the mixture was not right. The volume of the fumed silica seemed too much to mix in to the Ecoflex part B.

The particle size of the fumed silica was not known to us and could be one of the causes of the wrong mixture.

An assumption of Sanne: Another option could be that the fumed silica was too light. During the dosing of the fumed silica, the fumed silica almost seemed to float or act like snow. It might be possible that the scale, therefore could not measure the top layer of the fumed silica.

Second try

For the second try we used the same recipe as written above, only this time we did not mix the fumed silica at the start to first have a look at how the Ecoflex part B would mix with the iron powder. For this try we used the same ratio as during the first try, only we made 53,34 g magnetic soft material instead of the 100 as attempted in the first try. This was due to the probability of running out of materials.

<u>Ingredients</u>

- 11,6 g Ecoflex 00-10 part B
- 33,4 g Iron powder (carbonyl iron: 12)
- 6,9 g Ecoflex 00-10 part A
- 1,45 g fumed silica

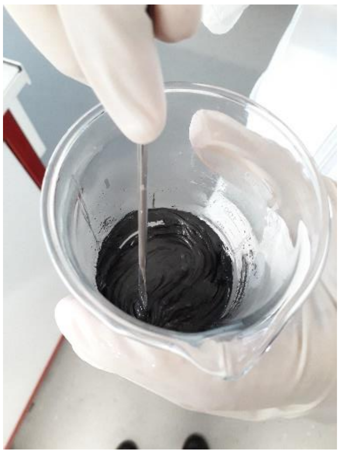


Figure 3 Ecoflex part B mixed with iron powder

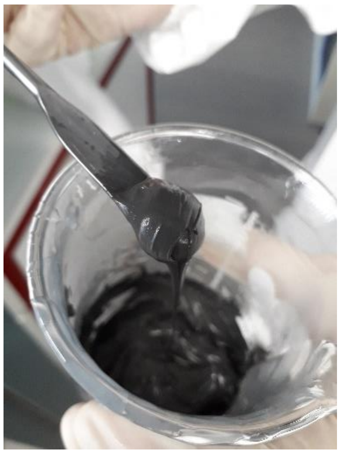


Figure 2 Ecoflex part B mixed with iron powder and Ecoflex part A

<u>Method</u>

- First we manually mixed the Ecoflex part B with the iron powder (figure 2)
- Then Ecoflex part A was added
- The mixture was again mixed by hand (figure 3)
- Because the mixture seemed right this time we added the fumed silica
- The mixture was mixed manually (figure 4)
- The mixture was put in a vacuum oven at 500 m bar for 27 minutes
- The mixture was spread out on a glass plate to cure
- The glass plate was put in an oven at 60°C for 20 minutes
- The magnetic soft material was pulled of the glass plate and cut into small strips

Figure 5 Ecoflex part B mixed with iron powder, Ecoflex part A and fumed silica

The structure of the mixture was a lot better then the first try. We still were not pleased with the viscosity of the material and found that it was to viscose. This could be due to the replacement of the SE 1700 with Ecoflex part B. It should also be mentioned that part A and B of the Ecoflex were already mixed for around forty minutes and that the curing of the material might also influence the viscosity of the ink.

Figure 4 the mixture was spread out on a glass plate to cure

One of the created strips was placed in the middle of a horse shoe magnet to see if it would react to the magnetic fields. By turning the magnet, not a lot happened. The strips were cut smaller and tried again. One strip gave a small shock when the magnet was placed at a certain position. That the material did not act as in the MIT is due to the fact that before the material was spread out on the glass plate, the magnetic particles were not magnetized or extruded wile being under a magnetic field. Because of this there were no predetermined non uniform magnetization patterns through which the material could contract in programmed shapes. Now the magnetic fields of the iron powder microparticles were randomly orientated.

When placing the material directly against the magnet the material stayed attached to the magnet due to the magnetic fields.

Magnetic Soft Materials: Experiment 2nd try

Dr. Sepideh Ghodrat
Houman Yarmand
Sanne van Vilsteren
02-01-2021

Keeping the same ratio's but change the oven curing time

The first experiment of today is to make the same mixture as the second time on 26-01-2021. Only due to a limited amount of iron powder (carbonyl iron: 12) we made only an amount of 12 grams.

Ingredients
- 2,6 g Ecoflex 00-10 part B
- 7,5 g Iron powder (carbonyl iron: 12)
- 1,55 g Ecoflex 00-10 part A
- 0,3 g fumed silica

Method
- After everything was mixed as the last experiment of 26-01-2021, the mixture was put in the vacuum oven at 575 bar for 15 minutes
- Then the mixture was spread out on a glass plate as before, but with next to it and extruded rod of the material made with a 10 ml syringe as an extrusion mould (see pictures 1 and 2) (2x)
- The two glass plates got a different treatment like oven curing time and magnetization

Figure 1 Magnetized samples on glass plate

Magnetized sample
- For the magnetized sample the spread out and extruded material were placed inside two horseshoe magnets.
- This was afterwards cured in the oven like before for 20 min at 60°C

Longer oven curing time sample
- This sample was directly placed inside the oven at 60°C for 40 minutes

Figure 2 Longer oven curing time sample

Findings

It was very hard to extrude the material because of its very low viscosity and grainy structure. To get the material extruded the force put on the syringe needed to be very high.

During the activation of the material with a magnet, there was no observant difference between the magnetized sample and the sample without the magnets. For now, we can not say if the magnets during the curing had any influence on the direction of the magnetic fields of the micro particles in the material.

Trying a different ratio of Ecoflex part A and B

For the second experiment of today the method was the same as in the second experiment of 26-01-2021. The parameter changed in this experiment is the weight ratio between Ecoflex part A and B. We used the 1:1 ratio as was written on the labels of the Ecoflex containers. We again made 12 grams.

<u>Ingredients</u>

- 2,1 g Ecoflex 00-10 part B
- 7,5 g Iron powder (carbonyl iron: 12)
- 2,1 g Ecoflex 00-10 part A
- 0,3 g fumed silica

<u>Samples</u>

From this mixture again two sample plates were made with an extruded rod and a spread-out sample. This was the same as the first test of today.

Plate one was put in the oven as it was and cured for 20 min at 60°C.

On plate two, a magnet was placed in the same manner as in in the first experiment of this day. This plate was also put in the oven and cured for 20 min at 60°C.

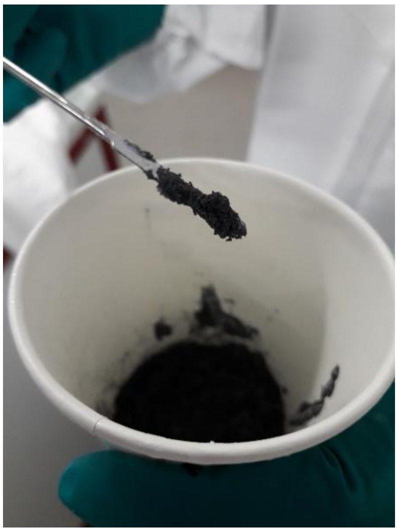


Figure 3 The grainy texture of our first experiment of today

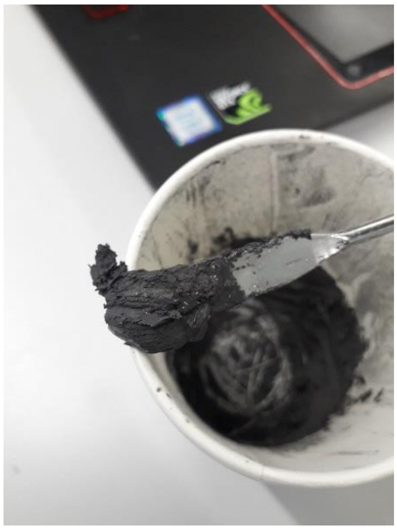


Figure 4 The smooth texture of the experiment with the 1:1 ratio between Ecoflex part A and B

Findings

This ratio worked better than the first one of today or the materials of last week (see figures 3 and 4). The material now had a more paste like structure and it seemed as if it had a higher elasticity. For the extrusion with the syringe, it worked easier now and less pressure was needed in in order to extrude it.

During the activation of the material with a magnet, there was no observant difference between the magnetized sample and the sample without the magnets. For now, we cannot say if the magnets during the curing had any influence on the direction of the magnetic fields of the micro particles in the material.

Both experiments of today again in the oven

Because all four material samples were not fully cured yet, the plates were put into the oven again. The settings were 30 min at 60°C.

Follow up research

Next week we want to continue with the 1:1 ratio of the Ecoflex 00-10 part A and B, because the viscosity is better. The parameter that we would like to change is the magnetic particle type. We are going to search for stronger alternatives that the carbonyl iron powder and/or for stronger magnets. We want to do this in order to try to get a magnetization profile in created magnetic soft material, which we could not detect in our experiments of today.

Magnetic Soft Materials: Experiment 3rd try

Dr. Sepideh Ghodrat
Houman Yarmand
Sanne van Vilsteren
05-01-2021

Introduction

The purpose of the experiments of today is to discover how certain ratios of the ingredients influence the performance of the material. In these experiments the method is the same as the second experiment during the first try. Things that differ from this method is that we left out the vacuum oven, because we forgot it by one sample and did want to compare all the samples made under the same conditions, to prevent influences of the method. The second thing that we changed was the curing time in the oven. This was changed to 120°C for 60 minutes, as we had later found in the MIT paper. At last, we decided to keep the ratio of the A and B part of the Ecoflex in the ratio 1:1 as we had discovered during the third and fourth experiment of the second try.

Ingredients for the different experiments

Experiment 1: 50% of the normal amount of fumed silica

<u>Ingredients</u>

- 2,1 g Ecoflex 00-10 part B
- 7,5 g Iron powder (carbonyl iron: 12)
- 2,1 g Ecoflex 00-10 part A
- 0,16 g fumed silica

Experiment 2: 200% of the normal amount of fumed silica

<u>Ingredients</u>

- 2,1 g Ecoflex 00-10 part B
- 7,5 g Iron powder (carbonyl iron: 12)
- 2,1 g Ecoflex 00-10 part A
- 0,65 g fumed silica

Experiment 3: Ecoflex ratio 2A : 1B

<u>Ingredients</u>

- 1,4 g Ecoflex 00-10 part B
- 7,5 g Iron powder (carbonyl iron: 12)
- 2,8 g Ecoflex 00-10 part A
- 0,3 g fumed silica

Experiment 4: Ecoflex ratio 4A : 1B

<u>Ingredients</u>

- 0,8 g Ecoflex 00-10 part B
- 7,5 g Iron powder (carbonyl iron: 12)
- 3,3 g Ecoflex 00-10 part A
- 0,3 g fumed silica

Experiment 5: 200% Iron powder

<u>Ingredients</u>

- 2,1 g Ecoflex 00-10 part B
- 15 g Iron powder (carbonyl iron: 12)
- 2,1 g Ecoflex 00-10 part A
- 0,3 g fumed silica

Experiment 6: Replace iron powder for NdFeB particles

<u>Ingredients</u>

- 2,1 g Ecoflex 00-10 part B
- 7,5 g NdFeB particles (<5 µm)
- 2,1 g Ecoflex 00-10 part A
- 0,3 g fumed silica

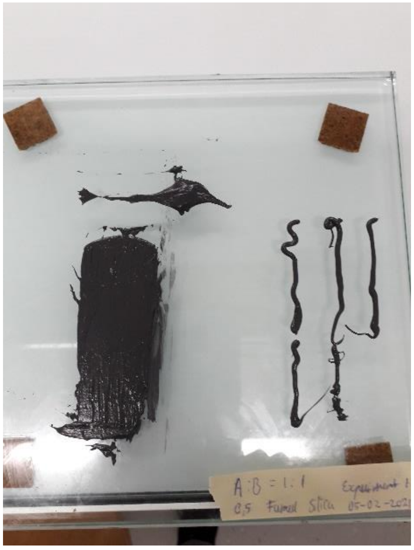


Figure 6 Experiment 1 50% fumed silica

Figure 1 Experiment 2 200% fumed silica

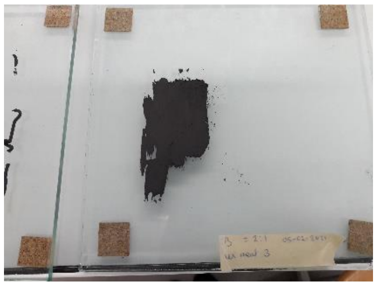


Figure 2 Experiment 3 Ratio 2A : 1B

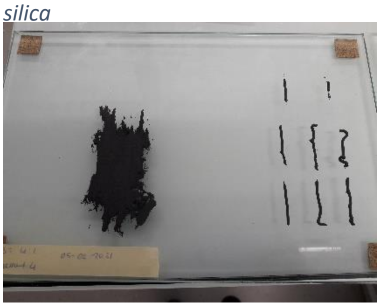


Figure 4 Experiment 4 Ratio 4A : 1B

Figure 3 Experiment 5 200% iron powder

Figure 5 Experiment 6 NdFeB particles instead of iron powder

Results

Experiment 1: 50% of the normal amount of fumed silica

Very flexible, elastic and magnetic responsive

Experiment 2: 200% of the normal amount of fumed silica

Magnetic responsive, less flexible, less elastic

Experiment 3: Ecoflex ratio 2A : 1B

Average elasticity, good magnetic response

Experiment 4: Ecoflex ratio 4A : 1B

The elasticity is more than the elasticity of experiment 3, but less than the elasticity of experiment 1. This was remarkable considering that experiment 3 with less ratio difference between part A and B mixed worse than the 1:1 ratio.
Magnetic responsive

Experiment 5: 200% Iron powder

No elasticity, this stiffness is probably due to the amount of powder being to much for the elastomer matrix to contain.
It has magnetic response.

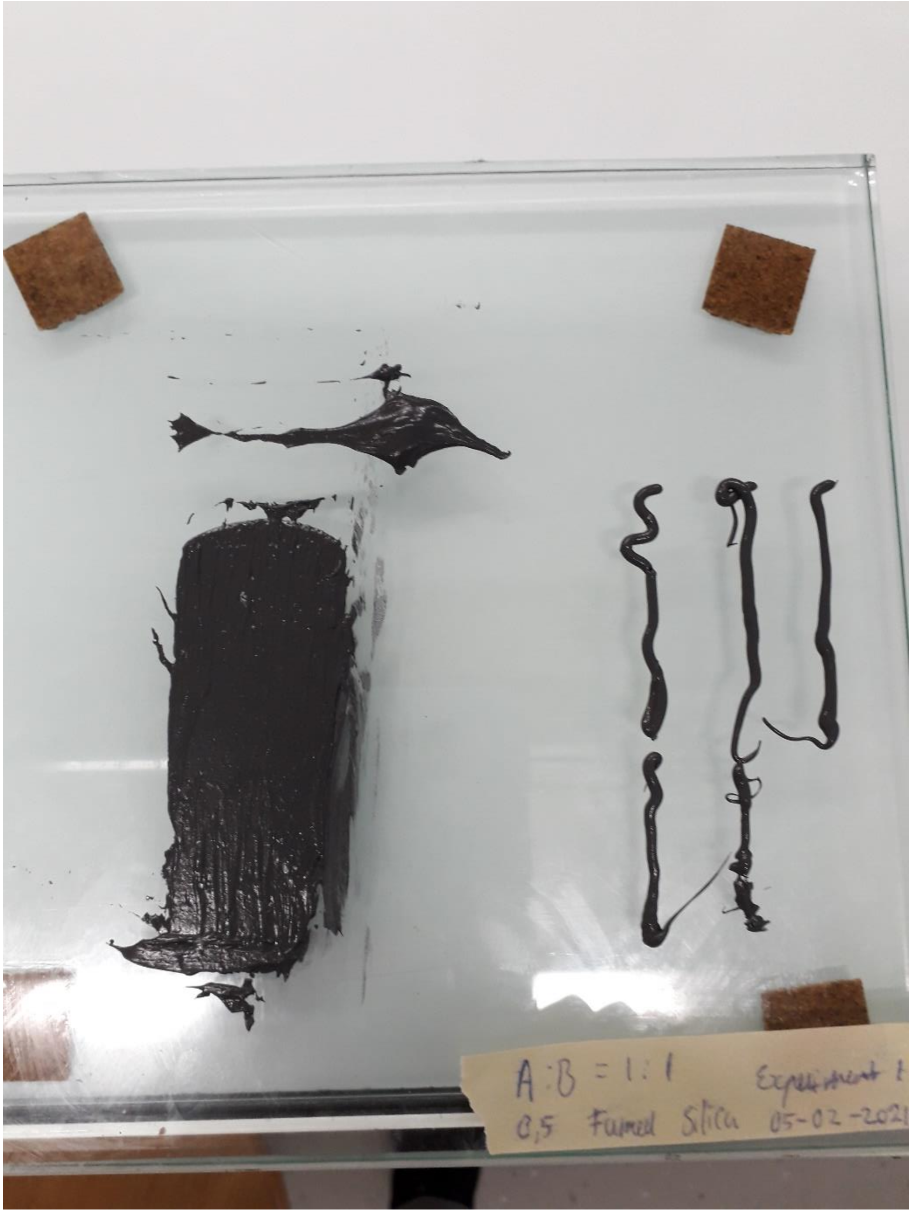
Experiment 6: Replace iron powder for NdFeB particles

Worst magnetization response, this was not what we expected, because NdFeB should have been a stronger magnet than the iron particles.
The elasticity is as absent as the material made in the third experiment. This could be because we replaced the same weight of particles instead of the volume. So due to a density difference the powder volume of the NdFeB could have been larger, which could have been to much powder for the elastomer matrix to contain as in experiment five of today.

Conclusion

The best result of today was experiment number 1.
The fumed silica influences the viscosity. The more fumed silica is added the more viscous the material becomes.

The 1:1 ratio of Ecoflex part A and B works the best.



Further research

The next step in this research will be to examine different types of magnetic particles and how they let the material response to magnetic fields.

We should also come up with test setup to determine the material properties like magnetic response and elasticity.

Magnetic Soft Materials: Experiment 4th try

Dr. Sepideh Ghodrat
Houman Yarmand
Sanne van Vilsteren
19-02-2021

Introduction

For the experiments of today, we used the first recipe that we made during the 3rd try with 50 wt% of the originally used amount of fumed silica. The purpose of this experiment is to see the difference in the performance of different magnetic particles. We will make ink mixtures by adding the same volume% or the same weight% as in comparison with the iron powder used in most of our previous experiments. The new particles all have a size of 5µm.

Ingredients for the different experiments

Experiment 1: same weight ratio Fe_3O_4 particles

<u>Ingredients</u>

- 2,1 g Ecoflex 00-10 part B
- 7,5 g Fe_3O_4 powder
- 2,1 g Ecoflex 00-10 part A
- 0,16 g fumed silica

Experiment 2: same weight ratio Samarium Cobalt particles

<u>Ingredients</u>

- 2,1 g Ecoflex 00-10 part B
- 7,5 g Samarium Cobalt particles
- 2,1 g Ecoflex 00-10 part A
- 0,16 g fumed silica

Experiment 3: same volume ratio Fe_3O_4 particles

<u>Ingredients</u>

- 2,1 g Ecoflex 00-10 part B
- 4,8 g Fe_3O_4 powder
- 2,1 g Ecoflex 00-10 part A
- 0,16 g fumed silica

Experiment 4: same volume ratio Samarium Cobalt particles

<u>Ingredients</u>

- 2,1 g Ecoflex 00-10 part B
- 8 g Samarium Cobalt particles
- 2,1 g Ecoflex 00-10 part A
- 0,16 g fumed silica

Experiment 5: same volume ratio NdFeB particles

Ingredients

- 2,1 g Ecoflex 00-10 part B
- 7,2 g NdFeB particles (<5 µm)
- 2,1 g Ecoflex 00-10 part A
- 0,16 g fumed silica

Method

For the method we did the same as the last time:

- Add the Ecoflex part B and the magnetic particles together
- Mix by hand
- Add the Ecoflex part A
- Mix by hand
- Add the fumed silica
- Mix by hand
- Extrude material with a syringe on a glass plate
- Spread the material out on the glass plate with a thickness of 100 µm
- Cure it in the oven at 120°C for 60 min.

Results

Experiment 1: same weight ratio Fe_3O_4 particles (figure 1)

During ink preparation

This mixture had too many particles for the Ecoflex to contain, because of this the ink was very grainy and did not stick together as one hole. Because of this, it did not extrude through the syringe and we could not make a 100 µm thick spread.

After curing

Even though there was a magnetic response of the material, the material itself was too stiff.

Experiment 2: same weight ratio Samarium Cobalt particles (figure 2)

During ink preparation

This mixture was very nice and had a nice viscosity. It was easy to extrude and spread out on the glass plate. The difference here with experiment 4 is not a lot in how well it worked because the weight difference in added particles was 8-7,5=0,5 gram, which is not a big percentage of the initially used weight.

After curing

This material had a good response but seemed to be a bit less than the iron-based powders. The elasticity was also good.

Experiment 3: same volume ratio Fe_3O_4 particles (figure 3)

During ink preparation

The volume ratio of this mixture worked better dan the weight ratio in experiment 1. The material was less viscous this time and was able to be extruded.

After curing

This material had both a good magnetic response and a good elasticity. It was far better than the stiff material from experiment 1.

Experiment 4: same volume ratio Samarium Cobalt particles (figure 4)

During ink preparation

This mixture was very nice and had a nice viscosity. It was easy to extrude and spread out on the glass plate.

After curing

This material like experiment 2 had a good response but less than the iron-based powders. The elasticity was also good. There was not a big difference between experiment 2 and 4. This might be because the added particle difference was not big in percentage according to the SmCo particles or the ratios as a whole.

Experiment 5: same volume ratio NdFeB particles (figure 5)

During ink preparation'

During the ink preparation, this ink mixture was also very nice with a good viscosity. The material was also able to be extruded with the syringe.

After curing

The cured material was not responsive as was the case in the last try of experiments. Through the addition of fewer particles than before (both in magnetic particles and fumed silica), the material had more viscosity which was very positive.

Figure 1: experiment 1

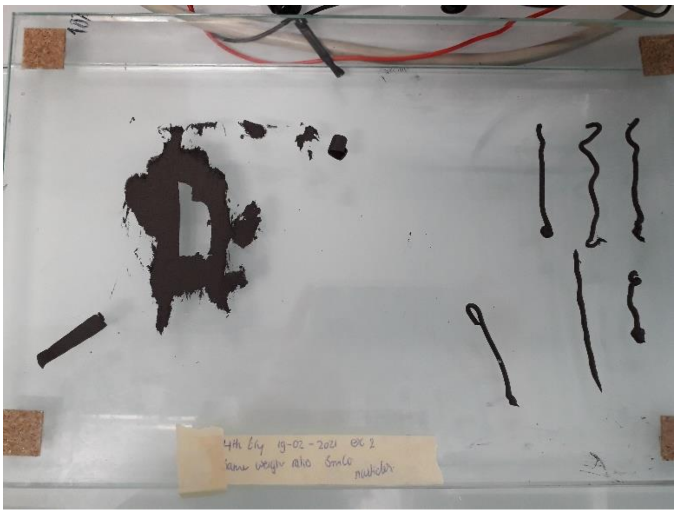


Figure 2: experiment 2

Figure 3: experiment 3

Figure 4: experiment 4

Figure 5: experiment 5

Conclusion

Of the four particles that we tested, the iron powder and the Fe_3O_4 powder showed the best magnetic response. The SmCo powder also showed a nice response and was especially good in its elasticity.

For a good elastic material, it is important when the particles are changed to do it according to the volume ratio. Otherwise, there might be too many particles for the matrix to contain and then the material gets stiff and brittle. Also, the viscosity will be very high.

Follow up research

For follow up research we first want to repeat our three best recipes: try 3; experiment 1, try 4; experiment 2 or 4 and try 4; experiment 3.

Besides this, we also want to change a next parameter to see which influence it has. Which parameter will be changed should still be decided on.

Another thing to think about is how we can quantify our results in the materials and how we can measure this.

Magnetic Soft Materials: Experiment 5th try

Dr. Sepideh Ghodrat
Houman Yarmand
Sanne van Vilsteren
24-02-2021

Introduction

The goal of the experiments of today is to repeat three of our best recipes until now. The other goal is to change one parameter again to see the effects. So, for the other experiments we used the same three recipes like the ones we wanted to repeat, but only added 50% of the magnetic particles.

Ingredients for the different experiments

Experiment 1: Iron carbonyl powder repeat T3E1

Ingredients

- 2,1 g Ecoflex 00-10 part B
- 7,5 g Iron powder (carbonyl iron: 12)
- 2,1 g Ecoflex 00-10 part A
- 0,16 g fumed silica

Experiment 2: Fe_3O_4 powder repeat T4E3

Ingredients

- 2,1 g Ecoflex 00-10 part B
- 4,8 g Fe_3O_4 powder
- 2,1 g Ecoflex 00-10 part A
- 0,16 g fumed silica

Experiment 3: Samarium Cobalt powder repeat T4E4

Ingredients

- 2,1 g Ecoflex 00-10 part B
- 8 g Samarium Cobalt particles
- 2,1 g Ecoflex 00-10 part A
- 0,16 g fumed silica

Experiment 4: 50% of the iron carbonyl powder

Ingredients

- 2,1 g Ecoflex 00-10 part B
- 3,75 g Iron powder (carbonyl iron: 12)
- 2,1 g Ecoflex 00-10 part A
- 0,16 g fumed silica

Experiment 5: 50% of the Fe_3O_4 powder

Ingredients

- 2,1 g Ecoflex 00-10 part B
- 2,4 g Fe_3O_4 powder
- 2,1 g Ecoflex 00-10 part A
- 0,16 g fumed silica

Experiment 6: 50% of the samarium cobalt powder

<u>Ingredients</u>

- 2,1 g Ecoflex 00-10 part B
- 4 g Samarium Cobalt particles
- 2,1 g Ecoflex 00-10 part A
- 0,16 g fumed silica

Method

For the method we did the same as the last time:

- Add the Ecoflex part B and the magnetic particles together
- Mix by hand
- Add the Ecoflex part A
- Mix by hand
- Add the fumed silica
- Mix by hand
- Extrude material with a syringe on a glass plate
- Spread the material out on the glass plate with a thickness of 100 µm
- Cure it in the oven at 120°C for 60 min.

Results

Experiment 1: Iron carbonyl powder repeat T3E1 (figure 1)

During ink preparation

Was as good as experiment 1 of the third try.

After curing

Same as before. It had a good elasticity and magnetic response.

Experiment 2: Fe_3O_4 powder repeat T4E3 (figure 2)

During ink preparation

With this experiment maybe something went wrong. The mixture seemed stickier and was harder to extrude than last Friday. We do not know what the cause of this is.

After curing

The cured material was less elastic than that of the Friday before. We do not know if the elastic response had improved or not. The magnet had to be close to the material, but then the response worked fine.

Experiment 3: Samarium Cobalt powder repeat T4E4 (figure 3)

During ink preparation

This ink was the same as Friday. It is nice to mix and to extrude and leaves a clean cup. When mixing the material sometimes has a grain-like feel and sound like sanding paper, but the mixture is well mixed.

After curing

Same as last Friday. It had a good elasticity and a medium magnetic response.

Experiment 4: 50% of the iron carbonyl powder (figure 4)

During ink preparation

This mixture had no big difference in accordance with the first experiment of today. It could be well extruded with the syringe.

After curing

Good elasticity and magnetic response. It might be a bit more elastic than the material of experiment 1, but we are not sure. The magnetic response is also good, but the magnet has to be closer to the material for it to respond.

Experiment 5: 50% of the Fe_3O_4 powder (figure 5)

During ink preparation

This ink was better to extrude than the ink of experiment 2 with the same magnetic particles.

After curing

The material had more elasticity than the material of experiment 2.

Experiment 6: 50% of the samarium cobalt powder (figure 6)

During ink preparation

As in experiment 4, this ink mixed very nice and was good to extrude.

After curing

The material had a worse magnetic response and the magnet had to be closer compared to experiment 3. The elasticity looked similar to the material of experiment 3.

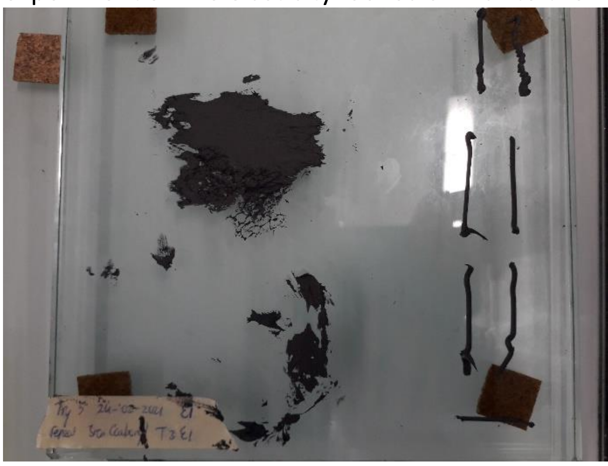


Figure 1: experiment 1

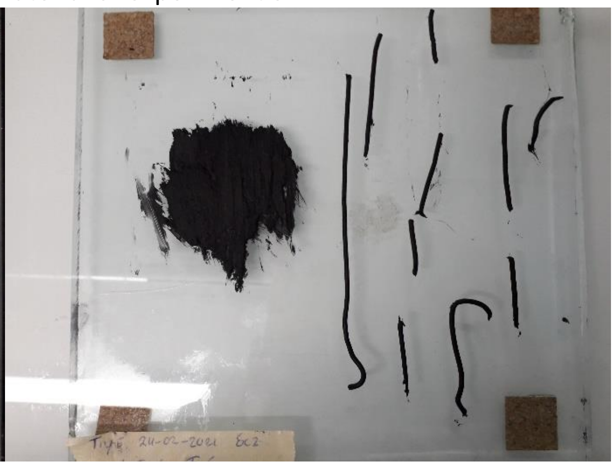


Figure 2: experiment 2

Figure 3: experiment 3

Figure 4: experiment 4

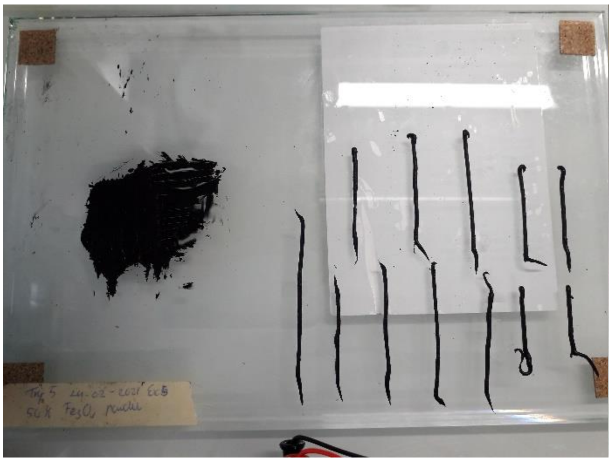


Figure 5: experiment 5

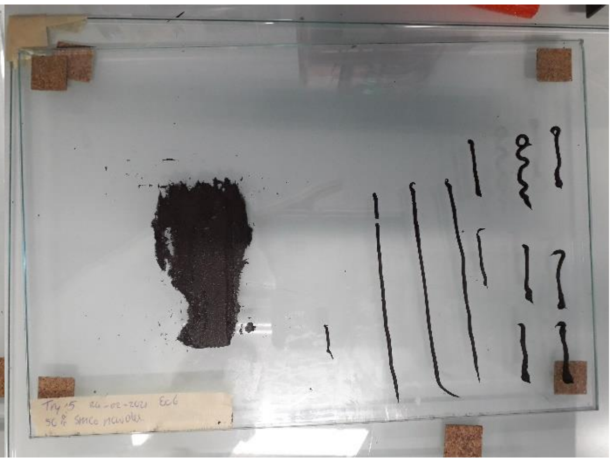


Figure 6: experiment 6

Conclusion

The repeated recipes seemed to behave the same way as the original ones. This only was not the case for experiment 2 with the Fe_3O_4 particles, but we could trace back where it went wrong.

The lesser number of magnetic particles made the material more elastic and less magnetic responsive as we thought. The ink with fewer particles as also less viscous and easier to extrude.

Further research

For further research, we want to start investigating material properties or starting with making test setups. This is important to later characterize the material.

We also want to use better machines for the mixing so that manual irregularities are more eliminated form the outcome. Also, the use of a magnetizer and 3D printer will be important next steps.

Magnetic Soft Materials: Experiment 6th try

Houman Yarmand
Sanne van Vilsteren
28-04-2021

Introduction

This experiment takes place after the first three static mixing nozzle tests. In those, it was concluded that the static mixing nozzle could mix the ink and that the mixed ink cured well. However, it was hard to extrude the ink through the nozzle, because of the high viscosity and therefore during those tests, only half the amount of fumed silica and magnetic particles were used. This resulted in an ink with a lower viscosity which was easier to extrude but did not hold its shape very well. Before optimizing the nozzle and building the 3D printer setup, first, the ink properties should be optimized so that the printer could be set up around that.

To optimize the ink, we should know what to optimize the ink to. What should it do and what should it not do? To make this list of criteria different there was looked at images from other studies.

One of the important parameters for the ink is the amount of shape stability through the viscosity of the ink. In Figure 1 there are multiple dog bone-shaped specimen, printed with different amounts of fumed silica from the study of Zhou et al. (2019). What can be seen is that the left picture with the lowest viscosity does not keep its shape as the higher viscous ink at the right picture does. This shape stability of the right picture is important for printing with magnetic soft materials. In figure 2 and 3 the 3D structures of the study by Kim, Yuk, Zhao, Chester, and Zhao (2018) can be seen. Their walls have a thickness of one extruded line of ink, which can keep its shape while it is stacked multiple layers high. Also, if the material should be able to make sharp bends when activated, it should be able to print gaps between the ink as can be seen in figure 4. If the ink stability and therefore also the viscosity, is low, this printed gap will flow closed and would prevent the material from making sharp bends.

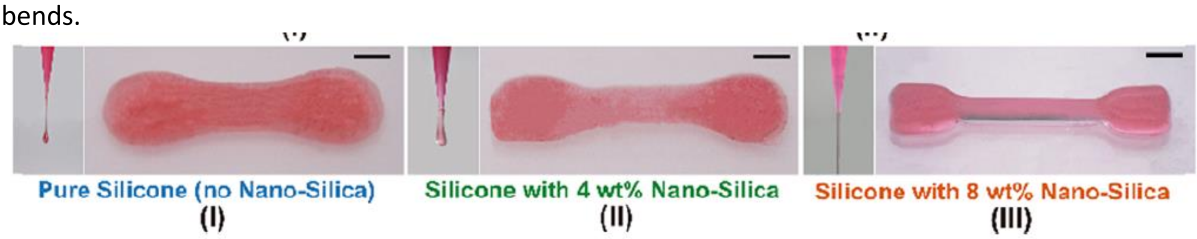


Figure 1 Different shape stabilities due to different ink viscosities (Zhou et al., 2019)

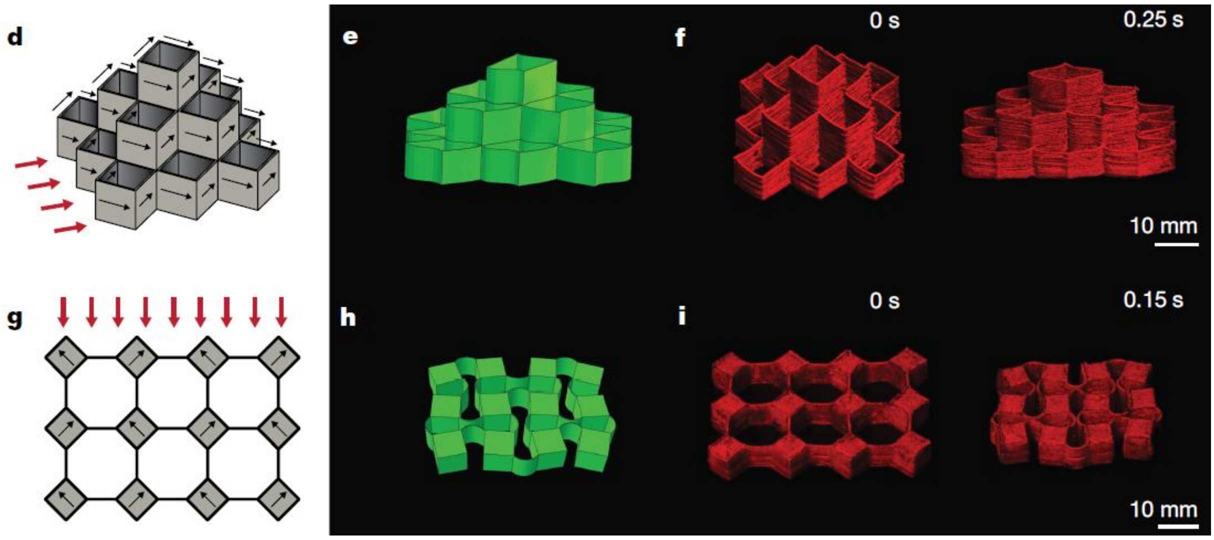


Figure 2 3D structures with thin walls (Kim et al., 2018)

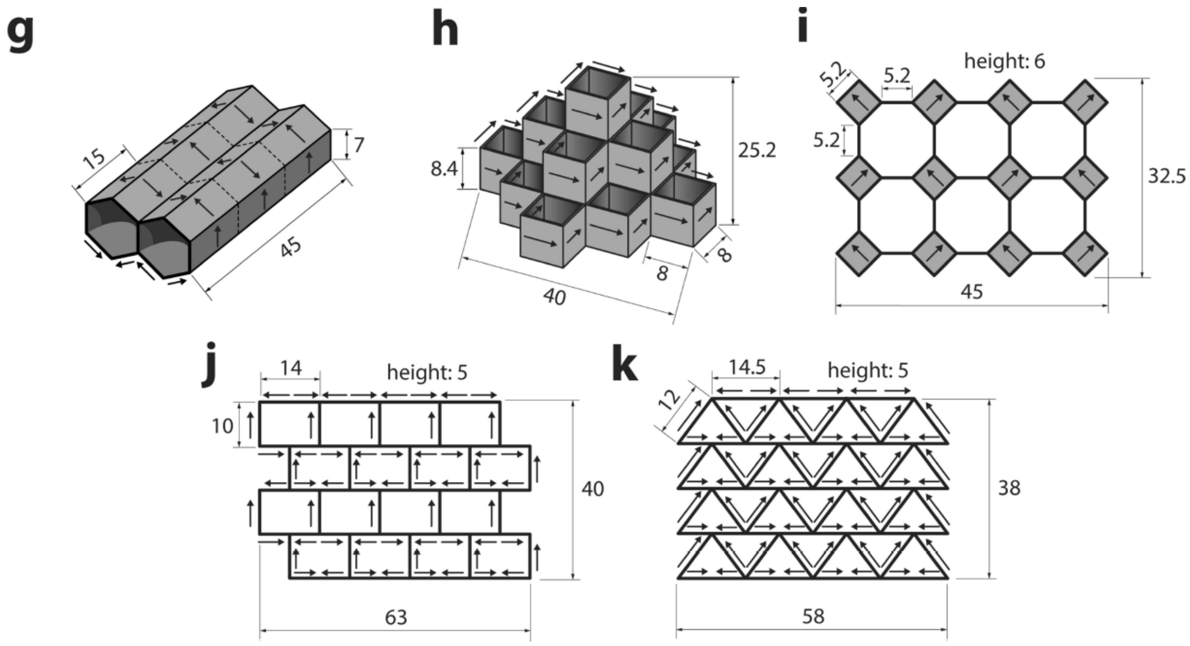


Figure 3 Dimensions printed 3D structures from the MIT paper (Kim et al., 2018)

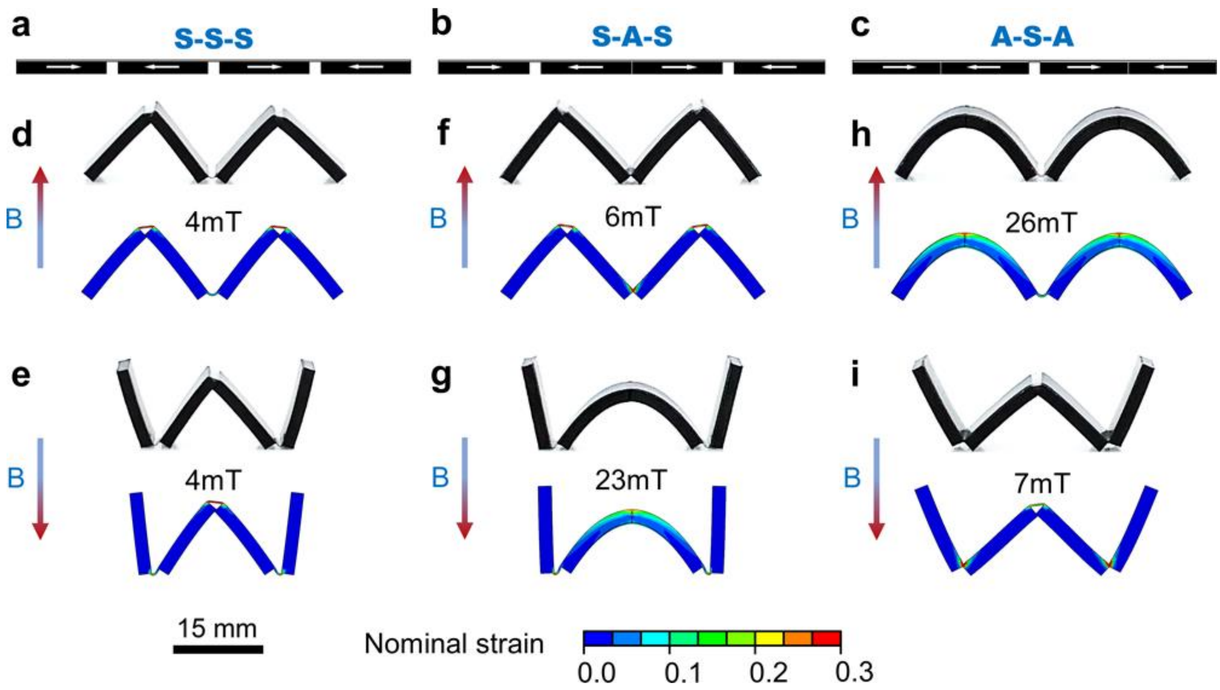


Figure 4 Sharp and soft bends of magnetic materials (Wu et al., 2019)

Shape stability is not the only important factor for magnetic soft materials, but it is one of the most important factors for the fabrication and shape qualities. The performance of the cured material is also important. For this, the number of magnetic particles has influence as well as the elasticity of the magnetic soft material.

For this, the number of magnetic particles is important for the strength of the possible magnetic field. Our developed ink has not been magnetized until now so therefore we have to look at other studies to get some insights. Below our wt% are compared with those of the MIT paper and the paper in which they both developed magnetic soft materials as well as magnetic shape memory polymers. The weight percentages do correspond quite a lot, except for the fumed silica.

Our recipe T3E1 (Ecoflex A:B = 1:1 only 50% used fumed silica in comparison with the MIT recipe)

Silicon	4,2	/11,86	x100wt%	=34,66wt%
Fumed silica	0,16	/11,86	x100wt%	=1,35wt%
Magnetic particles	7,5	/11,86	x100wt%	=63,24wt%

MIT recipe (Kim et al., 2018)

Silicon	21,78	+11,71	+1,17wt%	=34,66wt%
Fumed silica	2,72wt%			=2,72wt%
Magnetic particles	62,62wt%			=62,62wt%

Magnetic soft material recipe in the paper in which they also developed magnetic shape memory polymers (Ma et al., 2020)

Silicon	100%	90,9%	=30,98wt%
Fumed silica	+10wt%	9,09%	=3,40wt%
Magnetic particles	+20vol%*		=62,62wt%

**For this vol% the wt% was not given, because the MIT paper also uses 20vol% of the same type of particles (NdFeB), I have chosen to use this wt%. I know that the densities of the other materials are not completely the same but it is used to give some insights into where our recipe is positioned.*

The starting point for this test is the ink T3 E1 which hold its shape very well. Of this ink, multiple extruded lines will be stacked and judged on how well it holds its shape. From this onward, there will be decided how to change the recipe for the next time and repeat the experiment. In the end, these all will be cured and judged on how well they hold their shape.

The shape we will make in this experiment is a square with sides of 2,5 cm. This is bigger than the squares and shapes than in for example figure 3, but during this experiment, we will use syringes that have larger nozzles than those that are used for the shapes in figures 2 and 3. That larger size will therefore mimic the same ratios as in figures 2 and 3.

Materials

Utensils

- Syringes
- Scales
- Glass plate
- Oven
- Cups
- Mixing spatulas

Experiment 1: Iron carbonyl powder repeat T3E1

- 2,1 g Ecoflex 00-10 part B
- 7,5 g Iron powder (carbonyl iron: 12)
- 2,1 g Ecoflex 00-10 part A
- 0,16 g fumed silica

Method

Ink preparation

- Add the Ecoflex part B and the magnetic particles together
- Mix by hand
- Add the Ecoflex part A
- Mix by hand
- Add the fumed silica
- Mix by hand

Extrusion and shape building

- Draw a square of 2,5 by 2,5 cm on paper
- Lay this under the glass plate
- Load the syringe with the ink
- Trace the drawn square with the ink on the glass plate
- Extrude another layer of ink on the square
- Keep on building ink layers until you run out of ink

Take a picture of the made structure with the ink. Also, measure the height of the made structure.

After the open cube is built the structure should stay at least 30 minutes out of the oven, to see how the shape holds itself. After this, the structures can be cured in the oven for 60 minutes at 120°C.

After they have cooled down the structures should be judged on how well they held their shape. For this also take a picture of the structure after it has been cured and compare it with the picture taken before, when the structure was just built. Also, measure the height of the structure again and compare that with the height measured before.

How to adjust the ink

If this first ink does not perform well, we could double the amount of fumed silica to get at the same level as the MIT recipe.

If the material does keep its shape well, we could lower the total amount of added particles. This will be done by changing the amount of fumed silica, so that the magnetic properties of the ink can stay the same.

Experiment 2: Experiment 1 with 200% fumed silica (the same amount of fumed silica as in the MIT paper)

This ink was decided on, because the first ink performed well and we wanted to see how an ink with the same ratios as the MIT paper behaved.

- 2,1 g Ecoflex 00-10 part B
- 7,5 g Iron powder (carbonyl iron: 12)
- 2,1 g Ecoflex 00-10 part A
- 0,30 g fumed silica

Experiment 3: Experiment 1 with 50% fumed silica

Because increasing the amount of fumed silica in experiment 2 made it too hard to extrude the ink, we decided on lowering the amount of fumed silica in relation to experiment 1.

- 2,1 g Ecoflex 00-10 part B
- 7,5 g Iron powder (carbonyl iron: 12)
- 2,1 g Ecoflex 00-10 part A
- 0,08 g fumed silica

Experiment 4: Experiment 1 with 75% fumed silica

The ink of experiment 3 had a too low viscosity and not a lot of shape stability, it was easier to extrude. Therefore, we decided to make the fourth recipe with an amount of fumed silica between the amounts of experiment 1 and 3.

- 2,1 g Ecoflex 00-10 part B
- 7,5 g Iron powder (carbonyl iron: 12)
- 2,1 g Ecoflex 00-10 part A
- 0,12 g fumed silica

Results

Experiment 1: Iron carbonyl powder repeat T3E1

This ink extruded well through the syringe and was easy to use. During the entire process, the shape did not change (see figures 5 and 6). The measured height of the square directly after the extrusion was approximately 1 cm. After waiting for more than 30 minutes and curing the material in the oven for an hour the shape had not changed (see figure 6) and the height still was 1 cm. The layers were also bonded together (see figure 6).

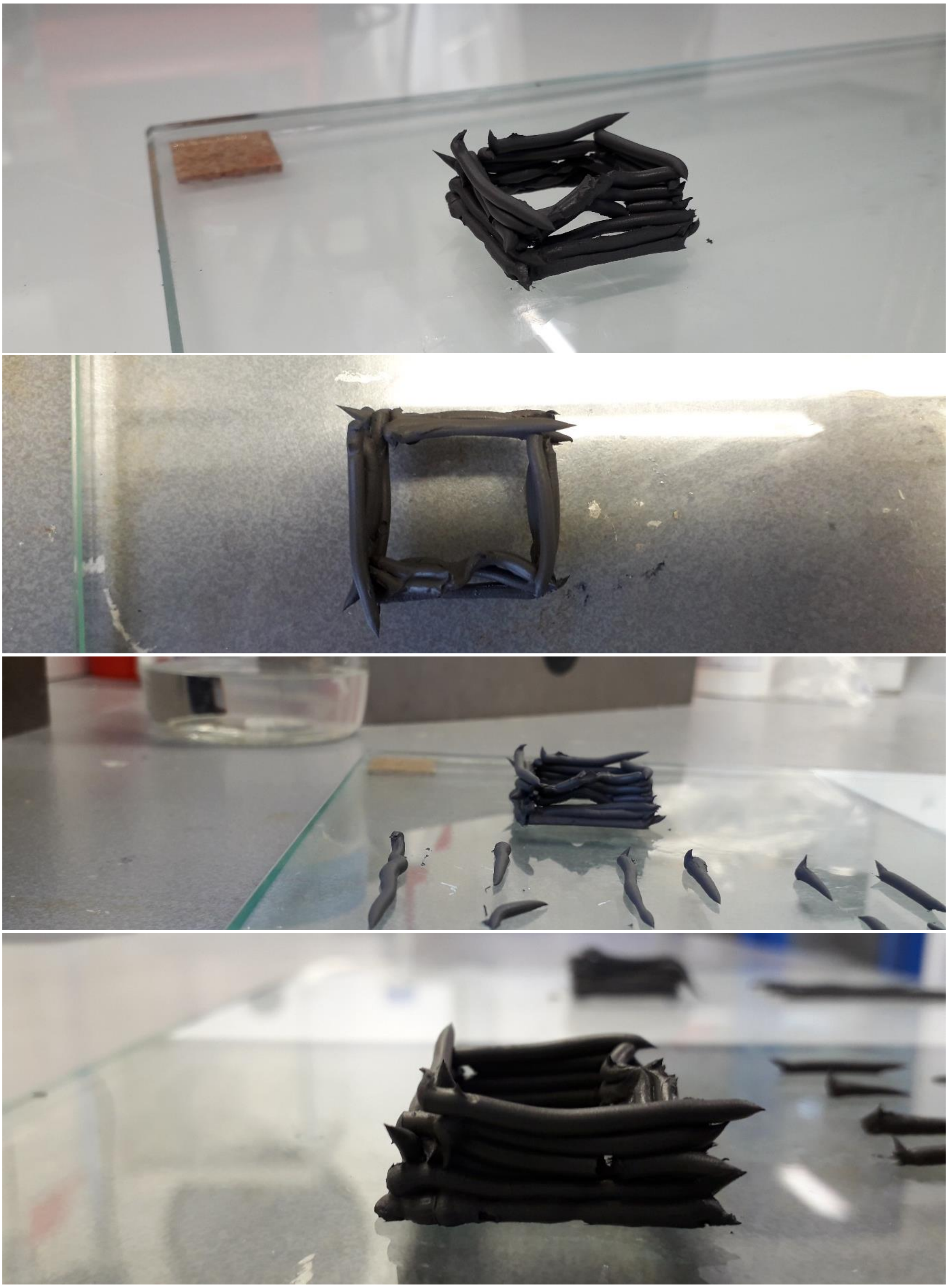


Figure 5 Experiment 1 after extruding

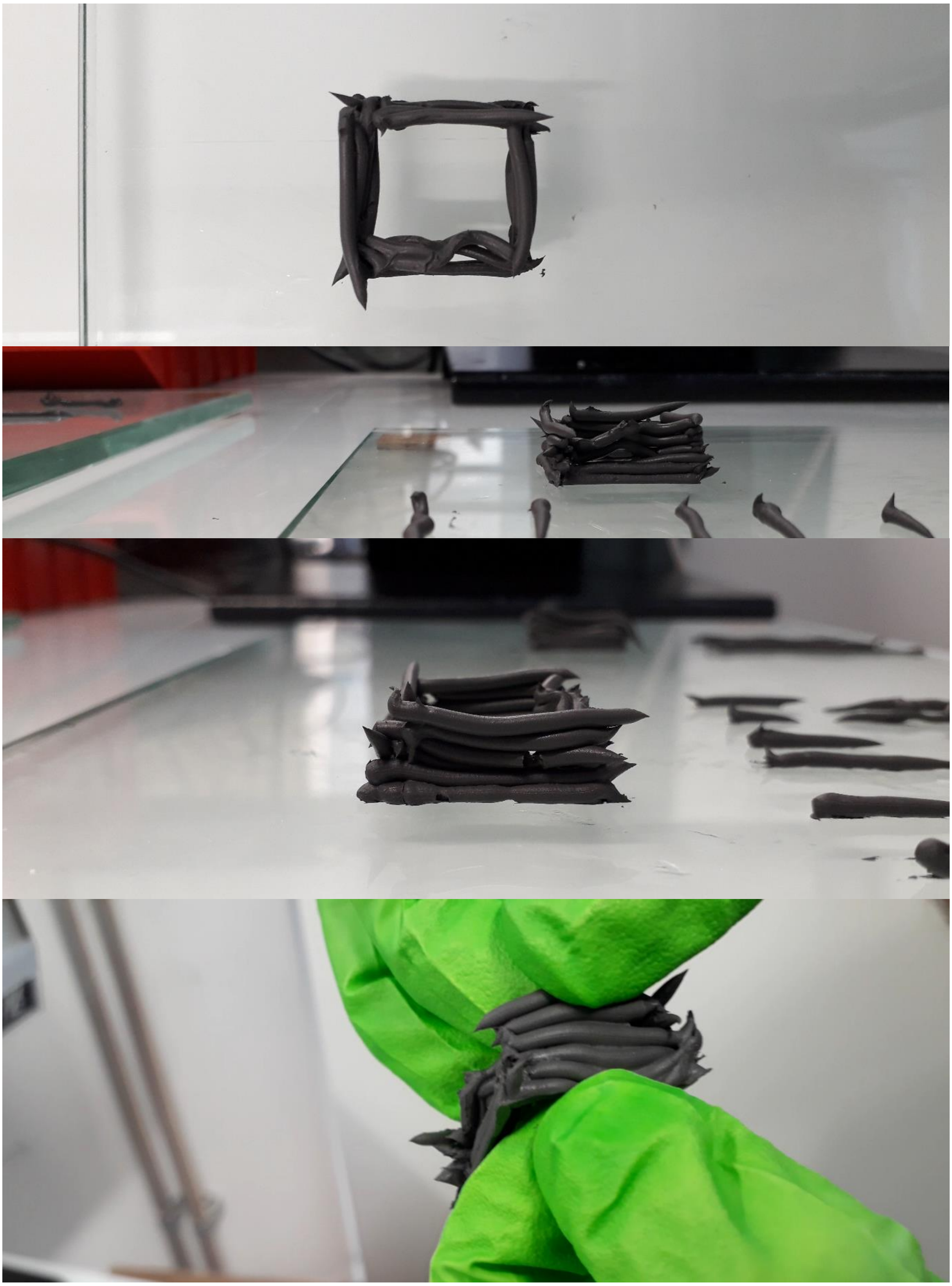


Figure 6 Experiment 1 after curing in the oven

Experiment 2: Experiment 1 with 200% fumed silica (the same amount of fumed silica as in the MIT paper)

This ink was more viscous than the one of experiment 1 and was harder to extrude. During the process, the piston of the syringe broke and the experiment stopped. The one layer that was extruded on top of the square held its shape very well. (see figure 7)

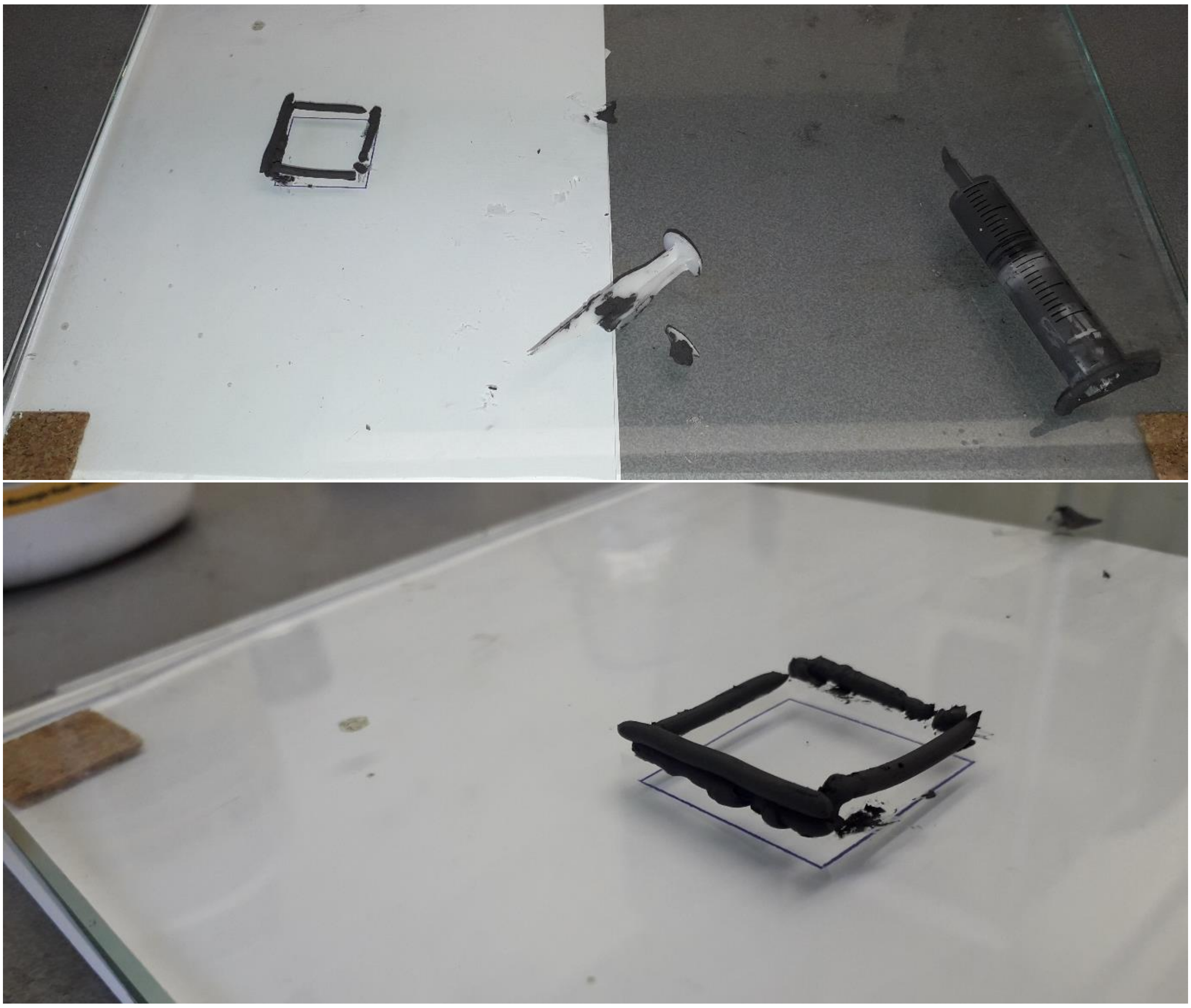


Figure 7 Experiment 2 after extruding

Experiment 3: Experiment 1 with 50% fumed silica

The ink of experiment 3 was less viscous and therefore easier to extrude. This structure was less stable than the previous two experiments. During the extrusion by hand, some of the walls did collapse and fell down (see figure 8). In the time between the extrusion and the oven, some of the extruded lines dissolved more into each other (see figure 9). This ink felt too unstable.

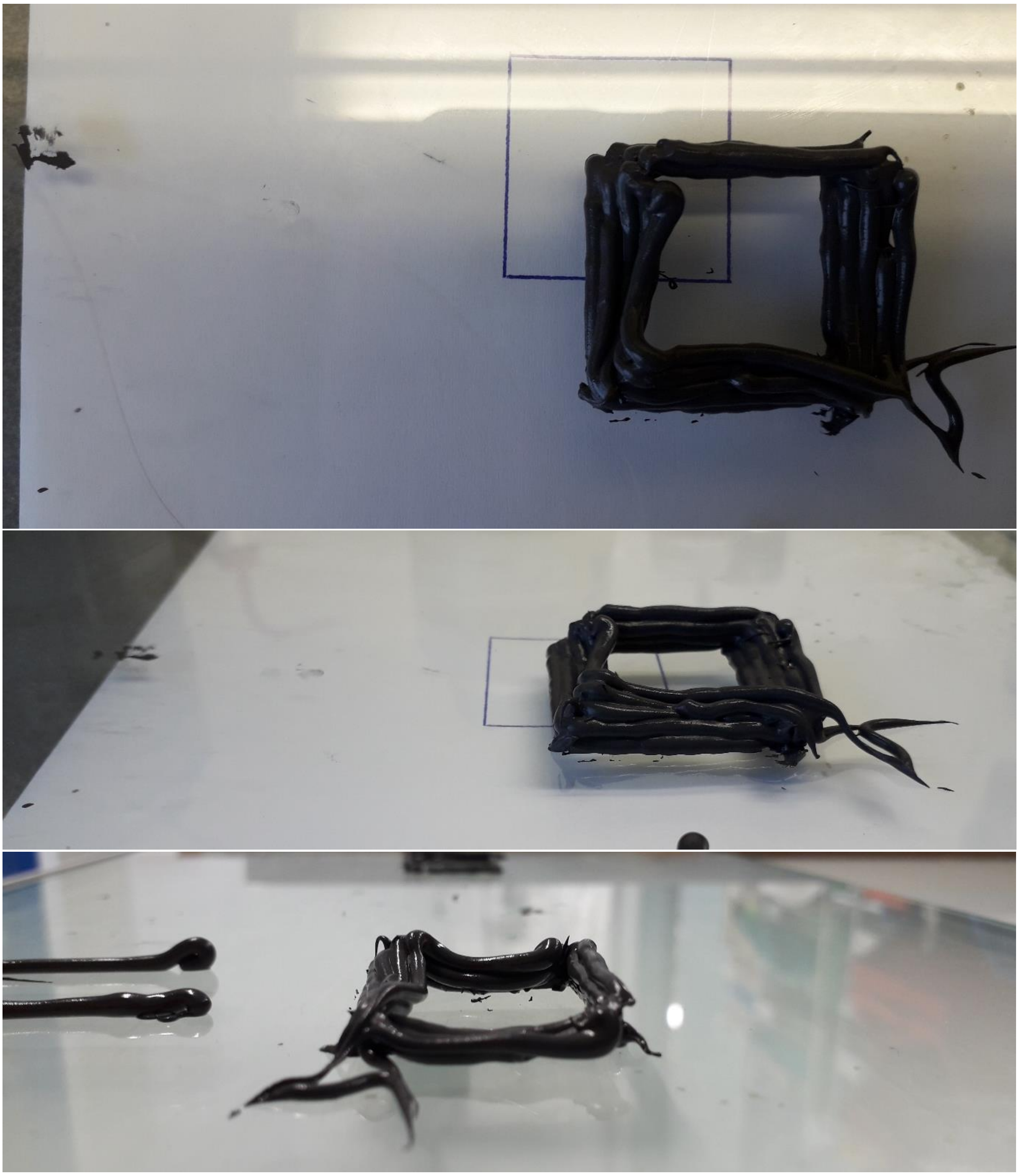


Figure 8 Experiment 3 after extruding

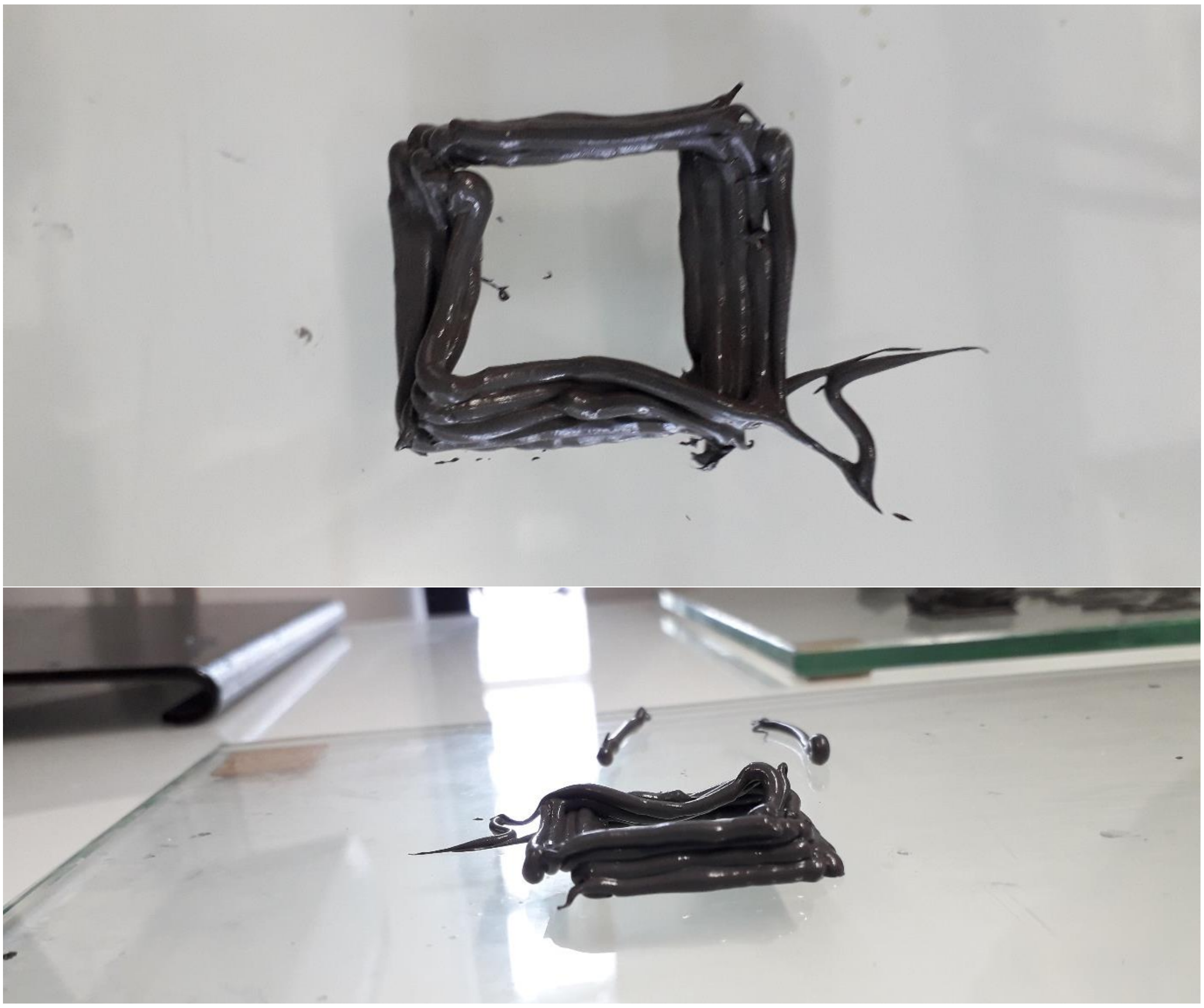


Figure 9 Experiment 3 after curing in the oven

Experiment 4: Experiment 1 with 75% fumed silica

There was not a big difference between the inks of experiment 1 and 4. The ink of experiment 4 was a bit softer but still functioned well. In figure 10 the extruded square can be seen directly after it was created. One of the sides was collapsed, but this was due to a fault during the process. The structure stood approximately 1 cm high. After the curing (see figure 11) this height still was the same. Also, the shape did not change during the waiting time. The layers were also bonded together (see figure 11).

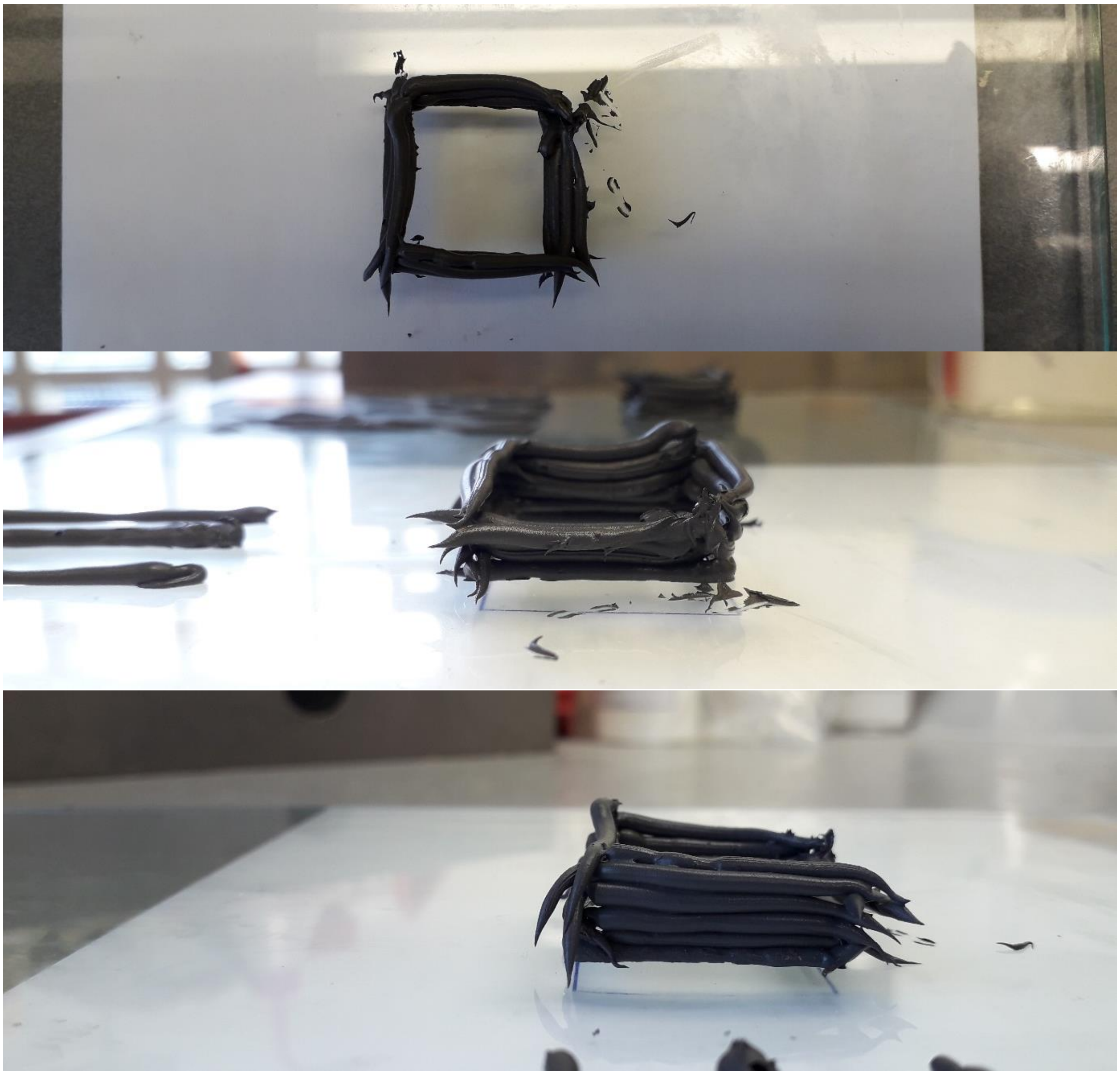


Figure 10 Experiment 4 after extruding

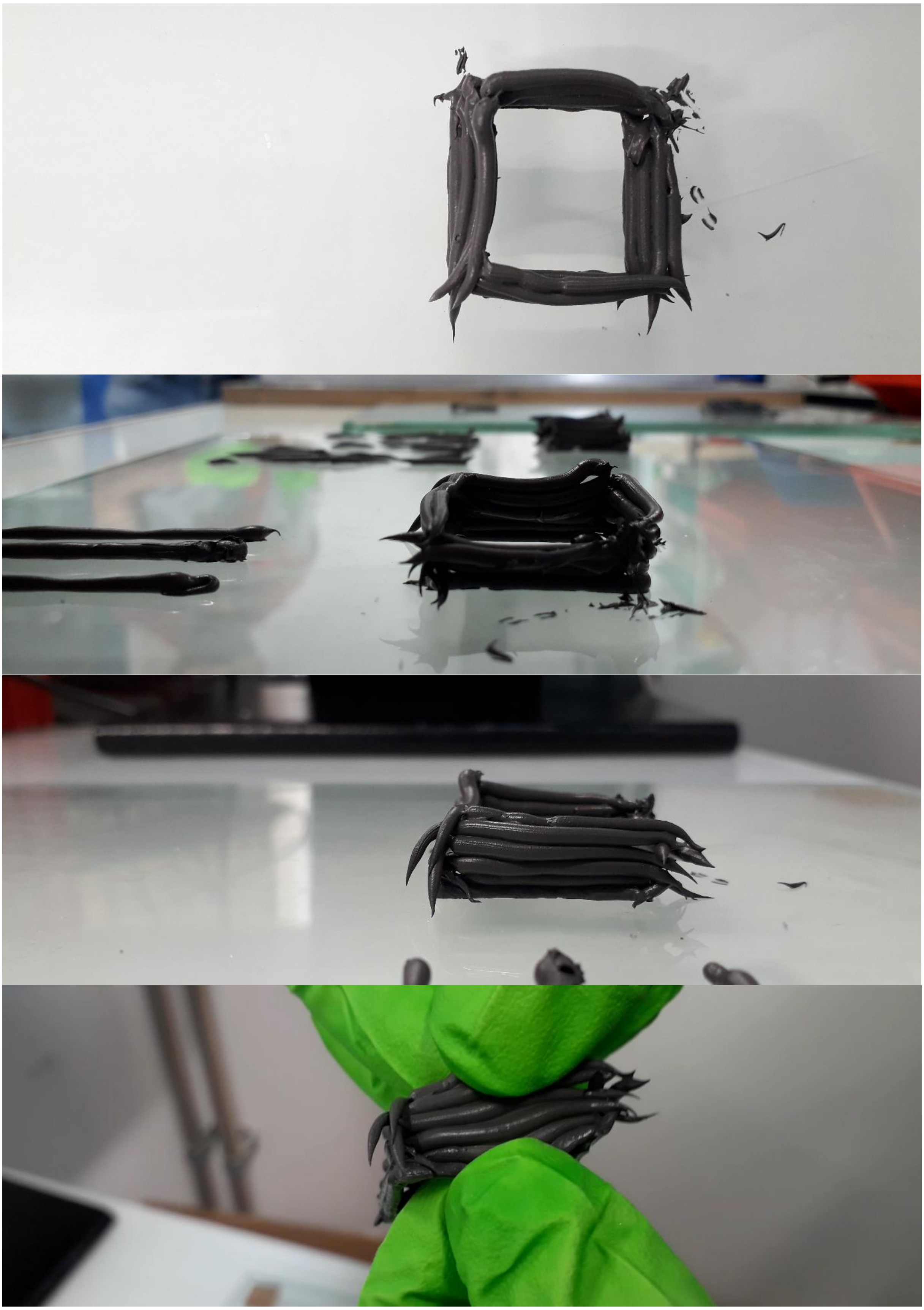


Figure 11 Experiment 4 after curing in the oven

Conclusion

Out of the four recipes that were tested today, recipes 1 and 4 had the best properties that showed the preferred behaviour, as described in the introduction. Those were both extrudable and had good shape stability.

The amount of fumed silica that was used in the MIT recipe seems to be too much in our recipe. For further research, the recipe that we are going further with lay between recipe 1 and 4, in which only the amount of fumed silica was changed.

Further research

For further research about the ink and the fabrication process, the next steps will be to optimize the static mixing nozzle for mixing an ink with a recipe falling in the spectrum between today's recipes 1 and 4.

Another thing that should be tested is the extrusion of the material at a 3D printer to finetune the ink and see the real behaviour of the ink in the system.

References

Kim, Y., Yuk, H., Zhao, R., Chester, S. A., & Zhao, X. (2018). Printing ferromagnetic domains for untethered fast-transforming soft materials. *Nature, 558*, 274-279. doi:10.1038/s41586-018-0185-0

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B.b Nozzle tests

These are the manual tests of extruding ink through the static mixing nozzle. They are done to see which ink viscosity could get through the static mixing nozzle, which mixing mechanism had the least resistance, the force needed for extrusion and if the ink was mixed well enough to cure.

Static mixing nozzle test

Sanne van Vilsteren
Houman Yarmand
01-04-2021

Introduction

The current option we have to use as a 3D printer for this project can extrude two materials at the same time. Due to this, there is an opportunity to load both cells with another component of the Ecoflex, a two-compound silicon. Opportunities for mixing the two compounds afterwards are that the silicon will not cure before it is printed. This makes it able to print the material for a longer time and print larger structures of the magnetic soft material. However, to print with two materials (Ecoflex part A and B) they still have to be mixed before printing so that the silicon can cure. For this, there is a static mixing nozzle. This is a long nozzle that will mix the two components before printing. Before deciding to use and further equipping this printer for magnetic soft materials, it first should be tested if the static mixing nozzle does work for the ink we have made so far.

For this test to succeed the material should:

- Not cure during the mixing process (clumping ink out of the nozzle)
- Be able to cure well after printing (mixed well before)

For these tests the “Static mixing nozzle silicone v2” Solidworks file will be modified. This nozzle already fits on the 3D printer. The test however will be performed with two syringes instead. The extruding diameter of the nozzle will be modified to a diameter of 0,4 mm. This is a common size for 3D printing nozzles and is also used in the study by Kim, Yuk, Zhao, Chester, and Zhao (2018). In case this size does not extrude well through resistance three other nozzles will also be printed with diameters of 0,6, 0,8 and 1,0 mm.

The ink that is going to be used in this experiment is the iron carbonyl ink as first made during try 3 experiment 1. Only the procedure to make this ink will be different as done in the previous experiments to make separate Ecoflex A and Ecoflex B inks. The fumed silica and iron particles will be divided in half and mixed in with the Ecoflex before the extrusion. This way the fumed silica and iron carbonyl particles will be mixed well beforehand and the only thing that the static mixing nozzle has to mix are the two Ecoflex compounds.

Materials

Utensils

- Printed static mixing nozzles for silicones adapted for syringes and with diameter openings of 0,4, 0,6, 0,8, and 1,0 mm
- 10 ml syringes
- Scale
- Glass plate
- Oven
- Cups
- Mixing spatulas
- Measuring cup

Materials

- Water

- 4,2 gram Ecoflex part A
- 4,2 gram Ecoflex part B
- 2 x 0,16 gram fumed silica
- 2 x 7,5 gram iron carbonyl powder

Procedure

Prepare and print the static mixing nozzles

The static mixing nozzle Solidworks file (see figure 1) should be adapted to the printing preferences. Due to this the nozzle size (see figure 2) is changed to a diameter of 0,4 mm. In case this nozzle is too small to extrude ink there are also prepared three other sizes (0,6, 0,8 and 1,0 mm). The two openings at the top (see figure 3) are made wider so that a 10 ml syringe can be mounted on top of it. These different size nozzles are printed from PLA.

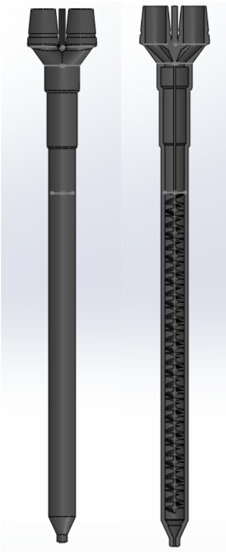


Figure 1 Static mixing nozzle outside (left), inside (right)

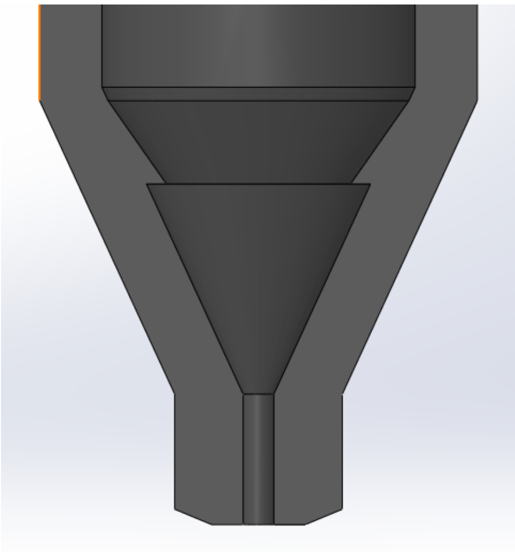


Figure 2 extrusion part of the static mixing nozzle

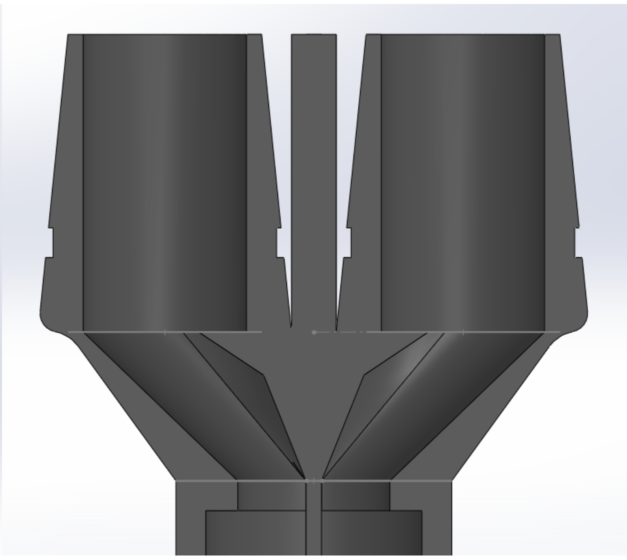


Figure 3 part to insert the two ink compounds

Determine the volume inside the static mixing nozzle

The support should be removed from the nozzles before going further in the experiment. After that, it is recommended to run water throughout the system to see if the nozzle is open or not.

After that, the entire nozzle should be filled with water and this water should be poured into a measuring cup to get an indication of the volume of ink that should be made for this experiment.

Before using the nozzle for the test is better to let it dry overnight so that the water will not interfere with the silicone mix.

Prepare the ink

- Ecoflex part A and Ecoflex part B should be poured into different cups
- Measure the half amount of the iron carbonyl particles and add this to the cup of Ecoflex part A
- Measure the other half of the iron carbonyl particles and add these to a cup of Ecoflex part B
- Mix the Ecoflex part A and the iron carbonyl particles manually
- Mix the Ecoflex part B and the iron carbonyl particles manually
- Measure the half amount of the fumed silica and add this to the cup of Ecoflex part A
- Measure the other half of the fumed silica and add this to the cup of Ecoflex part B
- Mix the contents in the cup of Ecoflex part A manually
- Mix the contents in the cup of Ecoflex part B manually

At the end of this step, there should be two cups with a mixture of Ecoflex part A or B with half of the amount of the fumed silica and the iron carbonyl particles.

Prepare for extrusion

- Load one 10 ml syringe with the Ecoflex A mixture
- Load another 10 ml syringe with the Ecoflex part B mixture
- Measure the openings of the static mixing nozzle, because of the inaccuracy of the 3D printing, to be sure through which size nozzle the ink is extruded
- Mount both of the syringes at the top of the static mixing nozzle (start with the smallest diameter size as this one is preferred to use)

Extrusion

- Keep the static mixing nozzle upright
- Press simultaneously on both the syringes pistons with the same force with separate hands
- Move the static mixing nozzle to extrude a line of magnetic soft material on the glass plate

Eventually follow up

There is a chance that the ink cannot be extruded through the small opening of 0,4 mm. If this is the case the Ecoflex A and Ecoflex B syringes can be mounted on the second smallest nozzle (0,6mm) and the procedure can be run again.

Control group

Because this mixing order has not been made previously it is good to also make a mixing control group without the static mixing nozzle (some samples of the original mixing order of Try3Experiment1 are still present and therefore do not have to be made again). For this, an equal amount of the Ecoflex A mixture and the Ecoflex B mixture should be made and mixed by hand. These should also be cured so that they can be compared with the mixed ink out of the static mixing nozzle.

Results

Figure 4 Printed static mixing nozzles first attempt, closed nozzles

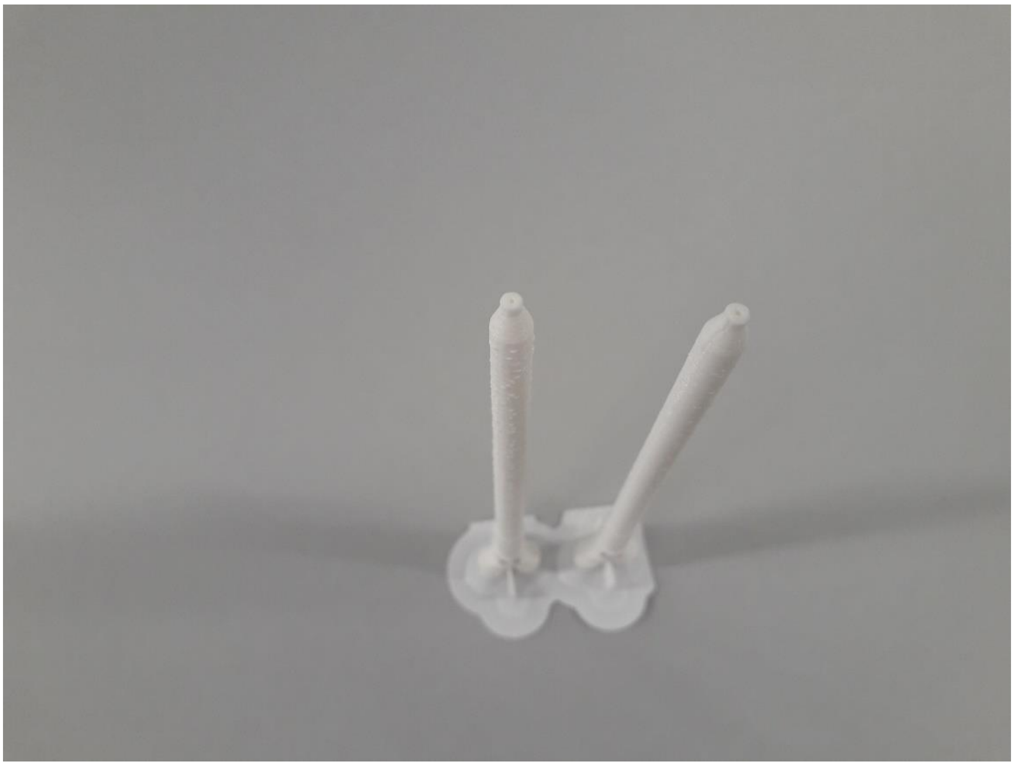


Figure 5 Printed static mixing nozzles third attempt open nozzles with a diameter of 0.4 mm

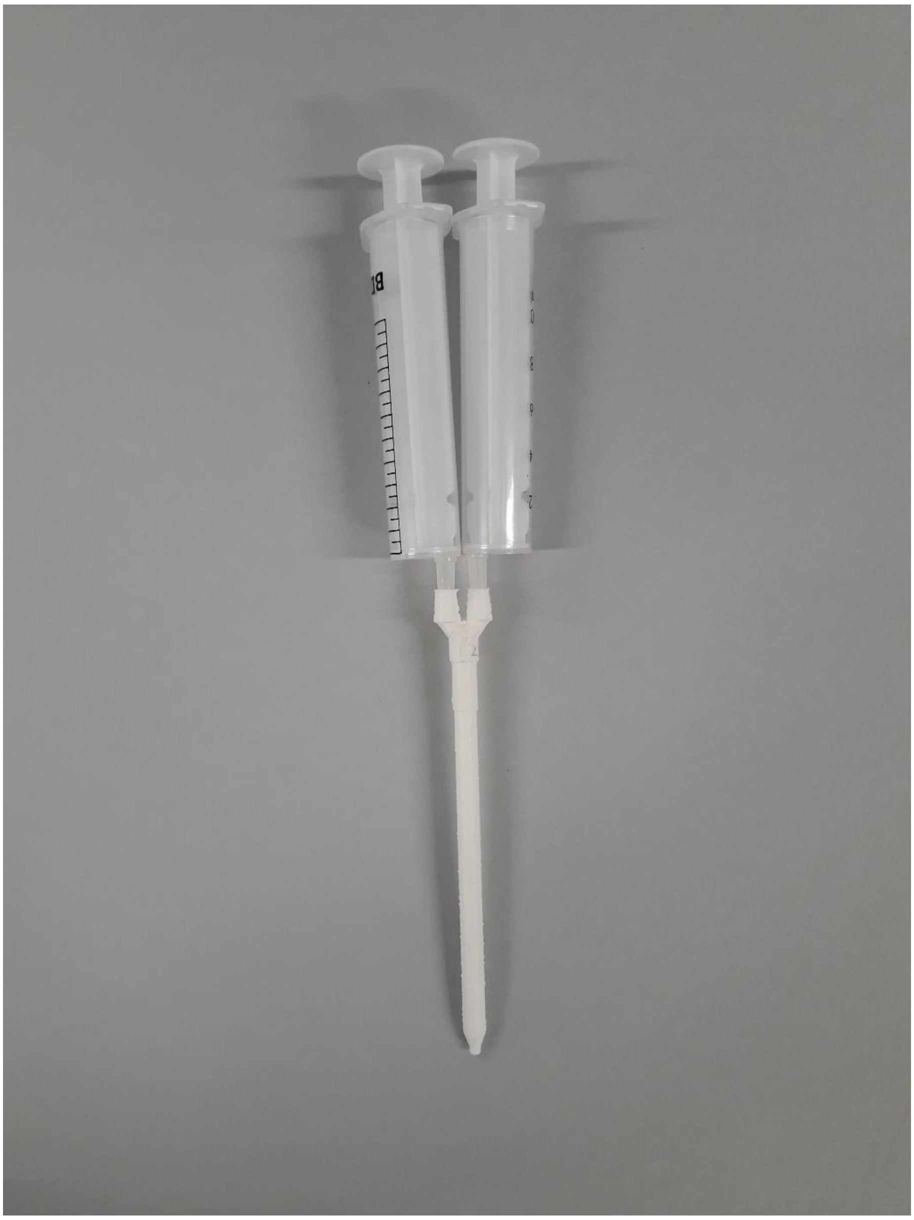


Figure 6 Syringes and static mixing nozzle assembly

The first print of the static mixing nozzles were not usable. Through the lower accuracy of the printer, the small openings at the nozzle were printed closed (see figure 4). After some experimenting with other size openings, the ones with a Solidworks model with a nozzle diameter of 1.2 mm and 1.4 mm got a printed diameter of around 0.4 mm (see figure 5). How the nozzle, later on, will be attached during the experiment can be seen in figure 6.

Figure 7 Test if the static mixing nozzles are hollow by running water through them

It was tested if the static mixing nozzles were hollow before the start of the experiments. The printed static mixing nozzles were hollow. In figure 7 it can be seen that the water drips out of the nozzle.

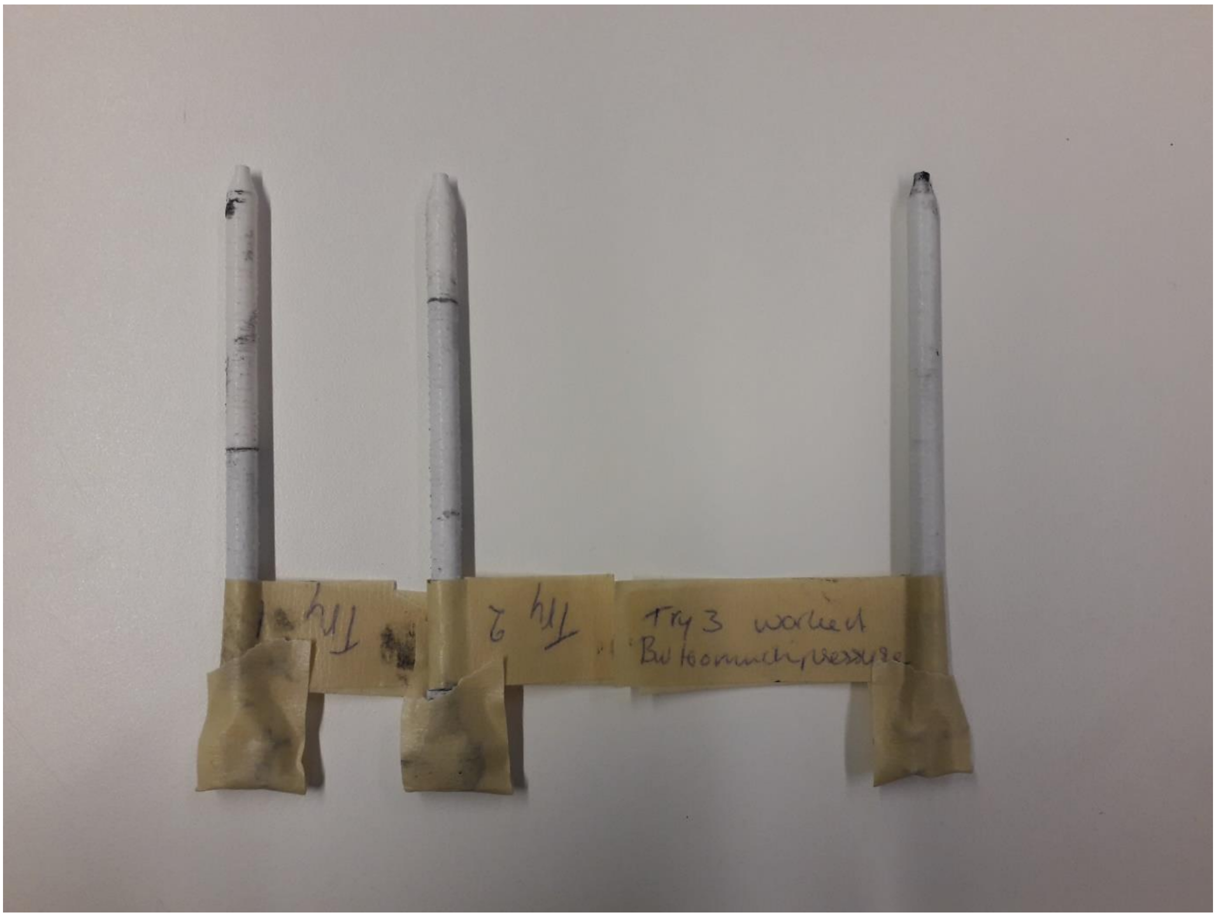


Figure 8 Nozzles used during the test. The black lines show how far the ink came inside the static mixing nozzle. In try3 the ink came out of the nozzle and therefore has no black line.

Figure 8 shows the three nozzles that were used during this test. Try one was performed with the ink as described in the method. Unfortunately, it took far too much pressure to get the ink extruded through the nozzle. The black line on the nozzle shows how far the material had come inside the nozzle. Because the ink had not reached the opening yet, we also tried to extrude premixed ink through a 0,4 mm diameter needle (see figure 9). This was possible. Some of this premixed ink with the new mixing order was also cured on a glass plate (see figure 10).

For try 2 only half of the amount of the iron particles in relation to the recipe was used. This made the materials a bit better to extrude, but the force needed was still too much. The material did not come out at the nozzle (see figure 8).

For the third try half the amount of both the iron particles and the fumed silica was used in comparison to the recipe described earlier. This time the ink could be extruded and was also cured on a glass plate (see figure 10).

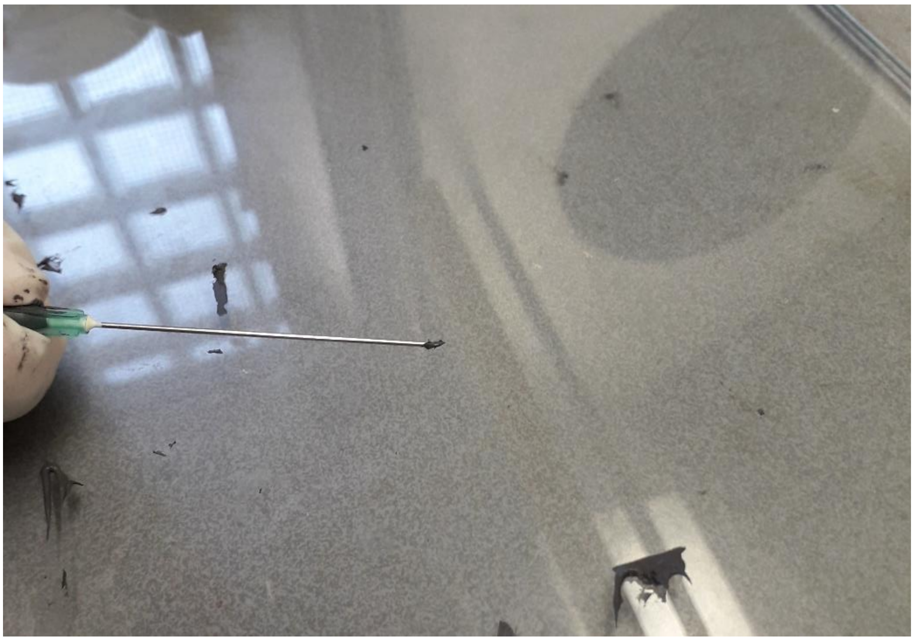


Figure 9 Test if the first material could be extruded through a needle with a 0.4mm diameter

Figure 10 Glass plate with on the left the cured manually mixed material with the new mixing order and on the right the uncured ink of the third try that got extruded through the static mixing nozzle.

Conclusion

Try 1: The viscosity was too high and therefore the material could not be extruded. There was probably too much resistance inside the static mixing nozzle.

The material could be extruded through a 0,4 mm diameter needle and therefore could be used with a 0,4 mm diameter nozzle at a 3D printer.

The new mixing order of the material could still cure well when mixed manually.

Try 2: The viscosity of this ink was still too high to be extruded through the static mixing nozzle. However, it came father in the tube than the material of try 1.

Try 3: This ink composition was able to be extruded through the static mixing nozzle. For the extrusion still, a lot of force was needed. Because the materials were less viscous the material became more liquid and spread out more on the glass plate before curing which is not preferable for the printing of the magnetic soft material. Also, the material was not cured after it came out of the oven. Even after four days, the material at the tip of the nozzle still is not cured. This can have different causes:

1. The mixing inside the static mixing nozzle does not work or is not long enough for this material.
2. Too little material was made and extruded. This could have resulted in the extruded material having been only one of the Ecoflex components. Maybe more material had to be extruded to come at a well-mixed ink.

Further research

For further research at least try 3 should be repeated with more material to exclude the second option of why the material has not cured.

Furthermore, a discussion should be had on these results if the nozzle should be adjusted or if we are going to use a different printer for this project.

References

Kim, Y., Yuk, H., Zhao, R., Chester, S. A., & Zhao, X. (2018). Printing ferromagnetic domains for untethered fast-transforming soft materials. *Nature, 558*, 274-279. doi:10.1038/s41586-018-0185-0

Static mixing nozzle test 2

Sanne van Vilsteren
Houman Yarmand
08-04-2021

Introduction

In the last test, the ink made in the third experiment extruded by a static mixing nozzle did not cure. This could have had multiple reasons. One of them was that the well-mixed material was not extruded yet. Due to this reason, the third experiment will be repeated to eliminate this reason and come up with the next steps for the 3D printer.

Materials

Utensils

- Printed static mixing nozzles for silicones adapted for syringes 0,4mm diameter
- 10 ml syringes
- Scale
- Glass plate
- Oven
- Cups
- Mixing spatulas
- Measuring cup

Materials

- Water
- 4,2 gram Ecoflex part A
- 4,2 gram Ecoflex part B
- 2 x 0,08 gram fumed silica
- 2 x 3,75 gram iron carbonyl powder

Procedure

Prepare the ink

- Ecoflex part A and Ecoflex part B should be poured into different cups
- Measure the half amount of the iron carbonyl particles and add this to the cup of Ecoflex part A
- Measure the other half of the iron carbonyl particles and add these to a cup of Ecoflex part B
- Mix the Ecoflex part A and the iron carbonyl particles manually
- Mix the Ecoflex part B and the iron carbonyl particles manually
- Measure the half amount of the fumed silica and add this to the cup of Ecoflex part A
- Measure the other half of the fumed silica and add this to the cup of Ecoflex part B
- Mix the contents in the cup of Ecoflex part A manually
- Mix the contents in the cup of Ecoflex part B manually

At the end of this step, there should be two cups with a mixture of Ecoflex part A or B with half of the amount of the fumed silica and the iron carbonyl particles.

Prepare for extrusion

- Load one 10 ml syringe with the Ecoflex A mixture
- Load another 10 ml syringe with the Ecoflex part B mixture
- Measure the openings of the static mixing nozzle, because of the inaccuracy of the 3D printing, to be sure through which size nozzle the ink is extruded
- <u>Level the two syringes so that they have the same amount of ink in them. This is important so that you can be sure that both syringes have loaded the nozzle with the same amount of ink. That is important because the Ecoflex only becomes a silicone if the two are mixed together. From earlier research, it was already found out that the mixing ratio of 1:1 (which is also the instruction on the Ecoflex bottles itself) work the best.</u>
- Mount both of the syringes at the top of the static mixing nozzle (start with the smallest diameter size as this one is preferred to use)

Extrusion

- Keep the static mixing nozzle upright
- Press simultaneously on both the syringes pistons with the same force with separate hands
- Move the static mixing nozzle to extrude a line of magnetic soft material on the glass plate

Control group

For the control group, a mixture of all the ingredients can be made and mixed by hand as was normally done before. This ink can then be extruded again with the help of a syringe.

Oven

Both the inks should be cured in the oven at 120°C for one hour.

Results

It was still hard to extrude the ink through the static mixing nozzle and too much force should have been needed for the 3D printer to print these silicones.

Figure 1 Cured ink. On the left: Cured ink mixed and extruded by the static mixing nozzle. On the right: ink mixed by hand and extruded with the help of a syringe (control group)

The inks both the one mixed by the static mixing nozzle as well as the ink mixed by hand cured.

Through the low viscosity, the ink spread out more on the glass plate than previous inks made with more magnetic particles and fumed silica (see figure 1).

Conclusion and further research

The ink mixed by the static mixing nozzle cured and therefore the static mixing nozzle can mix the ink. What still is unknown is if the ink mixed by the static mixing nozzle and the ink of the control group have the same material properties and therefore if the ink mixed by the static mixing nozzle is mixed well enough. This should be measured in the next test. The print quality of the static mixing nozzles also was not optimized and therefore should be improved to repeat this test. If the quality of the nozzle is better the insides might be smoother. This might prevent additional resistance during the extrusion and can lower the pressure needed for the extrusion.

A thing that worked from this test is to level the amount of Ecoflex part A and B before assembling the syringes on the static mixing nozzle. This way it is easier to put the same amount of force on both pistons. It is also easier to keep track of if the same amount of ink is extruded.

Another thing that should be measured in the next test is the force needed to extrude both inks through the static mixing nozzle.

Static mixing nozzle test 3

Sanne van Vilsteren
Houman Yarmand
14-04-2021

Introduction

In the last test, we found out that the static mixing nozzle could mix the A and B component of the magnetic soft material ink. This however still took a lot of force. In the third static mixing nozzle experiment, a better 3D print of the nozzle will be used (see figure 1). This might decrease the force needed to extrude the ink through the static mixing nozzle. Also, a static mixing nozzle will be tested with a different inner construction of the mixing mechanism.

Figure 1 Smoother print static mixing nozzle v2

Materials

Utensils

- Printed static mixing nozzles for silicones adapted for syringes 2,4mm diameter
- 10 ml syringes
- Scales
- Glass plate
- Oven
- Cups
- Mixing spatulas
- Measuring cup
- Rheometer

Materials

- Water
- 4,2 gram Ecoflex part A
- 4,2 gram Ecoflex part B

- 2 x 0,08 gram fumed silica
- 2 x 3,75 gram iron carbonyl powder

Procedure

Prepare the ink

- Ecoflex part A and Ecoflex part B should be poured into different cups
- Measure the half amount of the iron carbonyl particles and add this to the cup of Ecoflex part A
- Measure the other half of the iron carbonyl particles and add these to a cup of Ecoflex part B
- Mix the Ecoflex part A and the iron carbonyl particles manually
- Mix the Ecoflex part B and the iron carbonyl particles manually
- Measure the half amount of the fumed silica and add this to the cup of Ecoflex part A
- Measure the other half of the fumed silica and add this to the cup of Ecoflex part B
- Mix the contents in the cup of Ecoflex part A manually
- Mix the contents in the cup of Ecoflex part B manually

At the end of this step, there should be two cups with a mixture of Ecoflex part A or B with half of the amount of the fumed silica and the iron carbonyl particles.

Prepare for extrusion

- Load one 10 ml syringe with the Ecoflex A mixture
- Load another 10 ml syringe with the Ecoflex part B mixture
- Measure the openings of the static mixing nozzle, because of the inaccuracy of the 3D printing, to be sure through which size nozzle the ink is extruded
- <u>Level the two syringes so that they have the same amount of ink in them. This is important so that you can be sure that both syringes have loaded the nozzle with the same amount of ink. That is important because the Ecoflex only becomes a silicone if the two are mixed together. From earlier research, it was already found out that the mixing ratio of 1:1 (which is also the instruction on the Ecoflex bottles itself) work the best.</u>
- Mount both of the syringes at the top of the static mixing nozzle

Extrusion

- Keep the static mixing nozzle upright
- Press simultaneously on both the syringes pistons with the same force with separate hands
- Move the static mixing nozzle to extrude a line of magnetic soft material on the glass plate
- **Measure: In previous experiments, it took too much force to extrude the ink by hand and we had to press in through the static mixing nozzle by pushing the pistons of the syringes on a table. To measure the force needed to extrude the ink, the pistons of the syringes should be pressed against a scale when extruding the ink to see the maximum force needed.**

- <u>For the extrusion, through both the nozzles the same syringes with the same batch of magnetic soft material ink were used</u>

Oven

Both the inks should be cured in the oven at 120°C for one hour.

Results

It still took too much force to extrude the ink by hand through both types of static mixing nozzles. Therefore we had to press the syringes against the table surface to extrude the ink.

This force needed was measured for both static mixing nozzles.
The first static mixing nozzle that we also used in the previous experiments (v2), had a maximum weight of 19,7 Kg while extruding the ink through it (see figure 2).

Figure 2 Maximum amount of Kg during the extrusion through static mixing nozzle v2

The second static mixing nozzle, with a different inner construction (v1), had a maximum weight of 15,3 Kg while extruding the ink through it (see figure 3).

Figure 3 Maximum amount of Kg during the extrusion through static mixing nozzle v1

The ink mixed by both of the nozzles cured (see figure 4).

Figure 4 Cured ink. Left: cured ink static mixing nozzle v2. Right; cured ink static mixing nozzle v1.

Conclusion

Both the static mixing nozzles mixed inks got cured with makes them both applicable to use for the mixing of the ink.

However, the second static mixing nozzle that we tested (v1) only used 75% of the force needed for the first static mixing nozzle (v2). Due to this, the experiments will be continued with static mixing nozzle v1, because the force still was too high to let the ink be extruded by a 3D printer.

Further Research

For further research, the design of the v1 static mixing nozzle could be changed, so that there is less resistance in the nozzle and the force needed to extrude the ink can be lower.

But first, the ink viscosity should be optimized to get ink that has the right print stability for our purpose. Then the 3D printing process and the static mixing nozzle could be optimized to make printing with that ink possible.

Static mixing nozzle test 4

Sanne van Vilsteren
Houman Yarmand
07-05-2021

Introduction

The purpose of this experiment is to see if the optimized ink for printing from the 6th try of ink preparing experiments can be extruded through the static mixing nozzle. For this we again need to measure the extrusion force needed, to see if this is possible for a 3D printer. The ink should also be characterized more by measuring the viscosity of the unmixed ink components and the mixed ink.

For this experiment, the recipe of T3E1 will be used and T6E4, which came out the best during the ink preparation test try 6, for the printing stability. If those two do not work, we could take a look at T6E3 which had uncertain ink stability, but has the potential to work in a 3D printing setup.

For the viscosity test, we will use the same procedure as was presented in the work by Kim, Yuk, Zhao, Chester, and Zhao (2018), with the exception that we use a different geometry with a diameter of 40 mm instead of 20 mm. The measuring of the pressure will again be done by using a scale.

Materials

Utensils

- Printed static mixing nozzles v1 for silicones adapted for syringes 2,4mm diameter printed fine (0,1 mm layer height)
- 10 ml syringes
- Scales
- Glass plate
- Oven
- Cups
- Mixing spatulas
- Rheometer

Materials

Experiment 1: Iron carbonyl powder repeat T3E1

- 4,2 g Ecoflex 00-10 part B
- 2 x 7,5 g Iron powder (carbonyl iron: 12)
- 4,2 g Ecoflex 00-10 part A
- 2 x 0,16 g fumed silica

Experiment 2: Repeat 75% fumed silica T6E4

- 4,2 g Ecoflex 00-10 part B
- 2 x 7,5 g Iron powder (carbonyl iron: 12)
- 4,2 g Ecoflex 00-10 part A
- 2 x 0,12 g fumed silica

Experiment 3: Repeat 50% fumed silica T6E3

- 4,2 g Ecoflex 00-10 part B
- 2 x 7,5 g Iron powder (carbonyl iron: 12)

- 4,2 g Ecoflex 00-10 part A
- 2 x 0,08 g fumed silica

Method

Prepare the ink

- Ecoflex part A and Ecoflex part B should be poured into different cups
- Measure the half amount of the iron carbonyl particles and add this to the cup of Ecoflex part A
- Measure the other half of the iron carbonyl particles and add these to a cup of Ecoflex part B
- Mix the Ecoflex part A and the iron carbonyl particles manually
- Mix the Ecoflex part B and the iron carbonyl particles manually
- Measure the half amount of the fumed silica and add this to the cup of Ecoflex part A
- Measure the other half of the fumed silica and add this to the cup of Ecoflex part B
- Mix the contents in the cup of Ecoflex part A manually
- Mix the contents in the cup of Ecoflex part B manually

At the end of this step, there should be two cups with a mixture of Ecoflex part A or B with half of the amount of the fumed silica and the iron carbonyl particles.

Prepare for extrusion

- Load one 10 ml syringe with the Ecoflex A mixture
- Load another 10 ml syringe with the Ecoflex part B mixture
- Level the two syringes so that they have the same amount of ink in them. This is important so that you can be sure that both syringes have loaded the nozzle with the same amount of ink. That is important because the Ecoflex only becomes a silicone if the two are mixed together. From earlier research, it was already found out that the mixing ratio of 1:1 (which is also the instruction on the Ecoflex bottles itself) work the best.
- Mount both of the syringes at the top of the static mixing nozzle

Extrusion

- Press simultaneously on both the syringes pistons with the same force with separate hands
- Move the static mixing nozzle to extrude a line of magnetic soft material on the glass plate
- **Measure: In previous experiments, it took too much force to extrude the ink by hand and we had to press in through the static mixing nozzle by pushing the pistons of the syringes on a table. To measure the force needed to extrude the ink, the pistons of the syringes should be pressed against a scale when extruding the ink to see the maximum force needed.**
- Not all the ink should be extruded so that some from the syringes and some of the static mixing nozzle can be used to measure the viscosity.

Oven

Both the inks should be cured in the oven at 120°C for one hour.

Measure viscosity

The viscosity of the ink should be measured in three conditions. Those are:

- Ink component A separately
- Ink component B separately
- The ink after it is just mixed

This way we can get an insight into how the viscosity changes over the printing project.

The measuring procedure:

- 40-mm-diameter steel plate geometry.
- Steady-state flow experiments with a sweep of shear rates (0,0-100 s^{-1})
- The inks were brought to a temperature of 25°C for one minute before the experiment started.
- The gap height between the geometry and the plate always was 0,5 mm.

Results

https://biosci.mcdb.ucsb.edu/biochemistry/tw-lig/logarithms/logarithmic-axes.htm

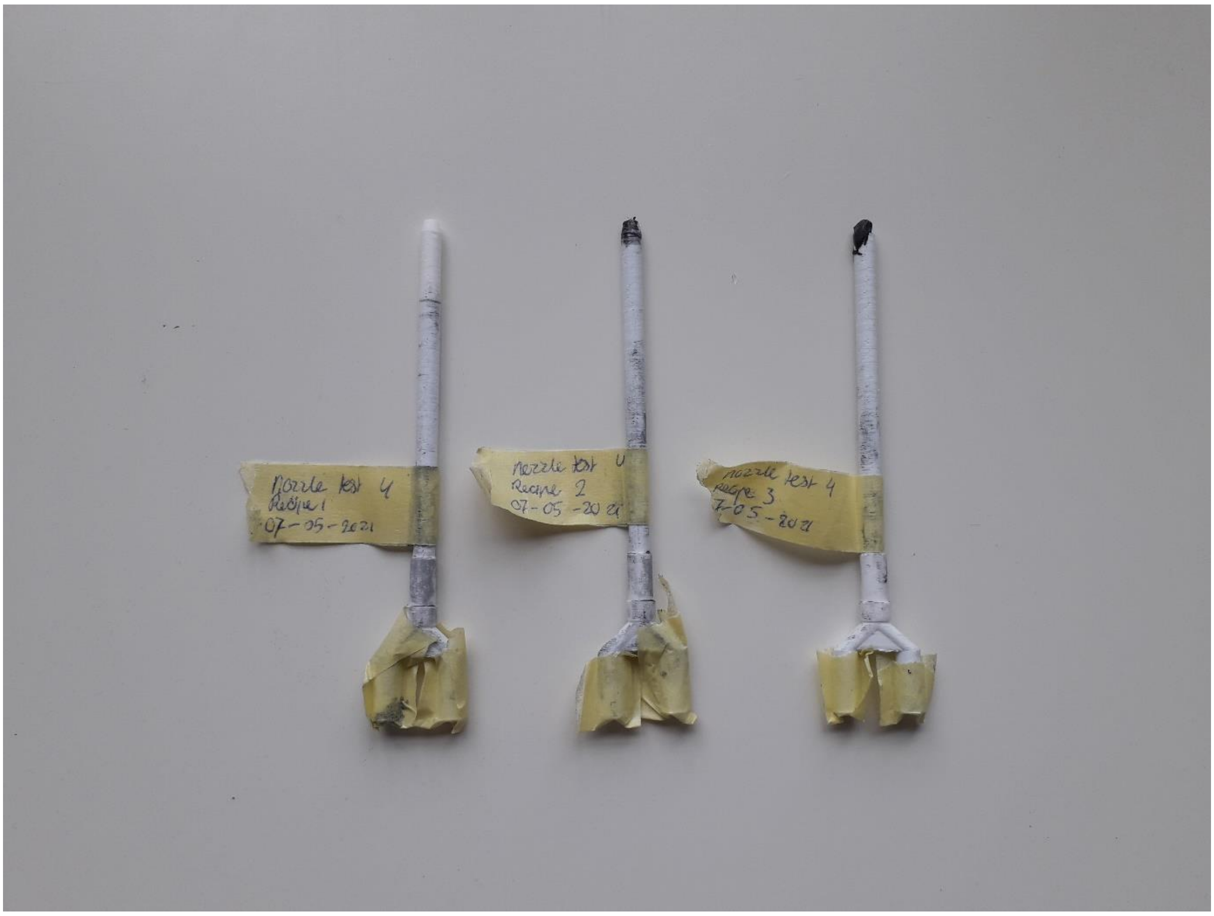
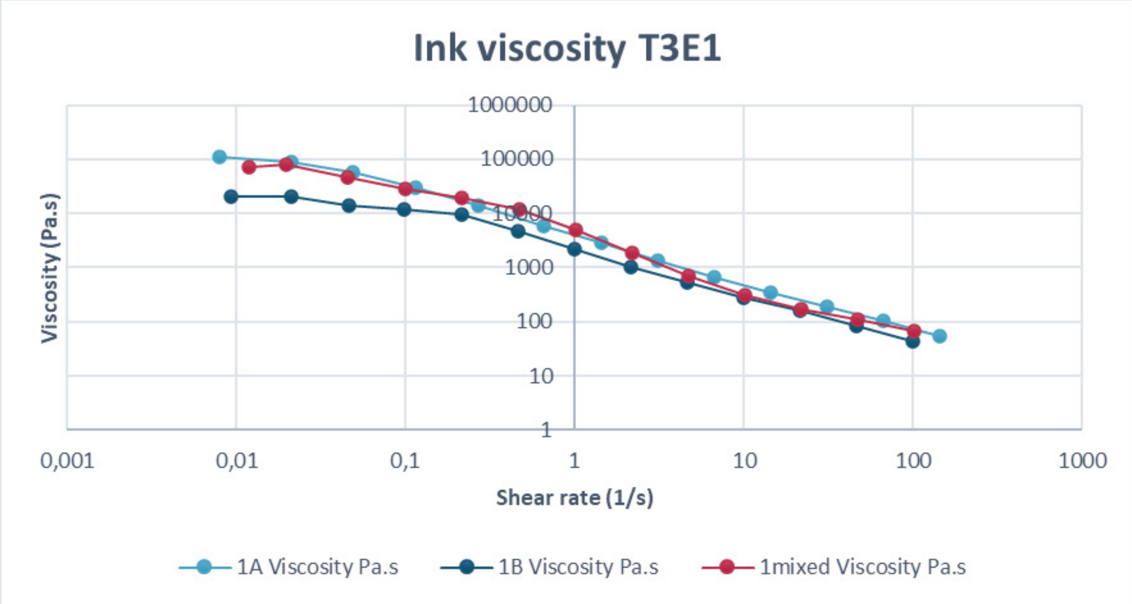


Figure 1 3 used nozzles for the mixing and extrusion of the different inks. From left to right T3E1, T6E4 and T6E3

Experiment 1: Iron carbonyl powder repeat T3E1

Maximum measured kg: The scale showed an error which means that the maximum 24 kg of the scale was exceeded. As can be seen in figure 1 the ink did not get extruded through the nozzle because too much force was needed. Therefore, we think that T3E1 will not be suitable for 3D printing.

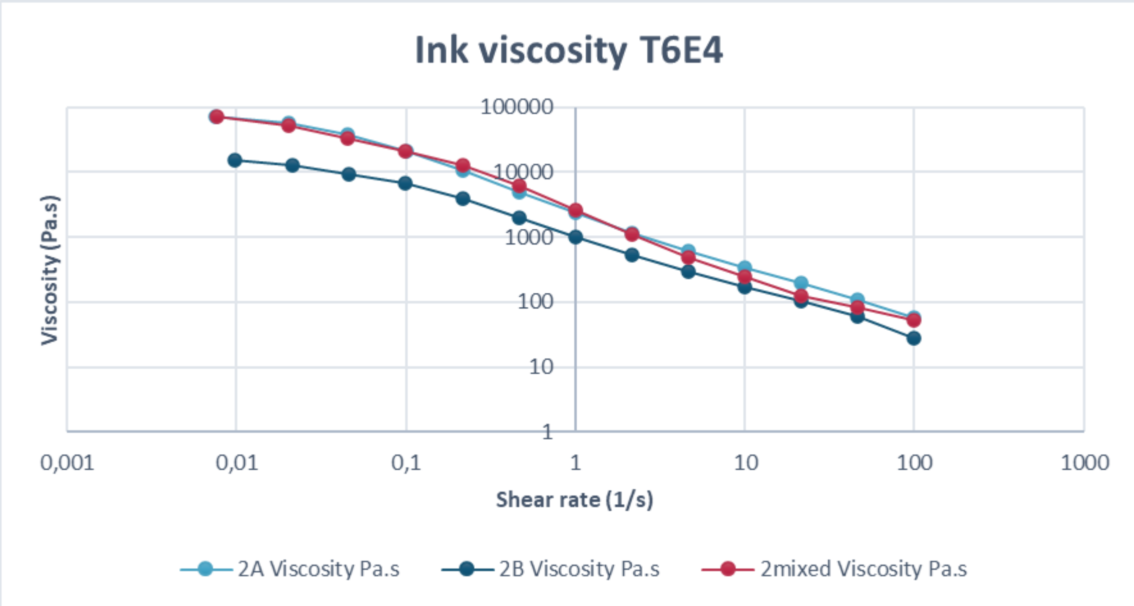


These are the viscosity measurements from the T3E1 in (component A, component B and the mixed ink). What can be seen is that component A has a higher viscosity than component B. Furthermore, shows the declining graph that the ink has shear-thinning properties, which are recommended for printing silicones. That makes sure that while the ink is extruded it will be more viscose, which means that less force is needed. But when no force is exceeded on the ink internal links will be build up which gives the ink its stability after printing. (Zhou et al., 2019)

Experiment 2: Repeat 75% fumed silica T6E4

Maximum measured kg: 22,566 kg
This ink got extruded through the static mixing nozzle. We had however not extruded a lot and the ink that we wanted to cure did not cure in the given time. Because of this we opened the static mixing nozzle and saw that some parts have cured (see figure 2). To be sure of this and because this ink also showed good print stability in the 6th ink test, this test should be repeated to know for sure if the ink can be cured right.

Figure 2 Sawn open static mixing nozzle v1 with T6E4 ink

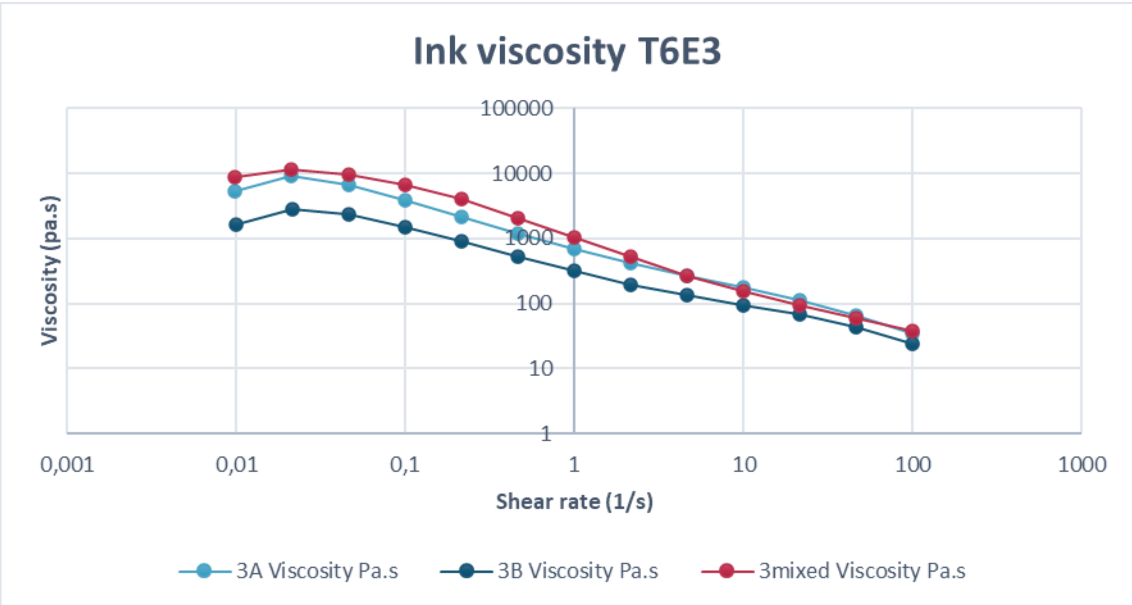


The graphs of the T6E4 ink show the same shear thinning behaviour as the T3E1 ink. The viscosity is a bit lower than that of T3E1 which can be seen in the Mixed ink viscosity graph.

Experiment 3: Repeat 50% fumed silica T6E3

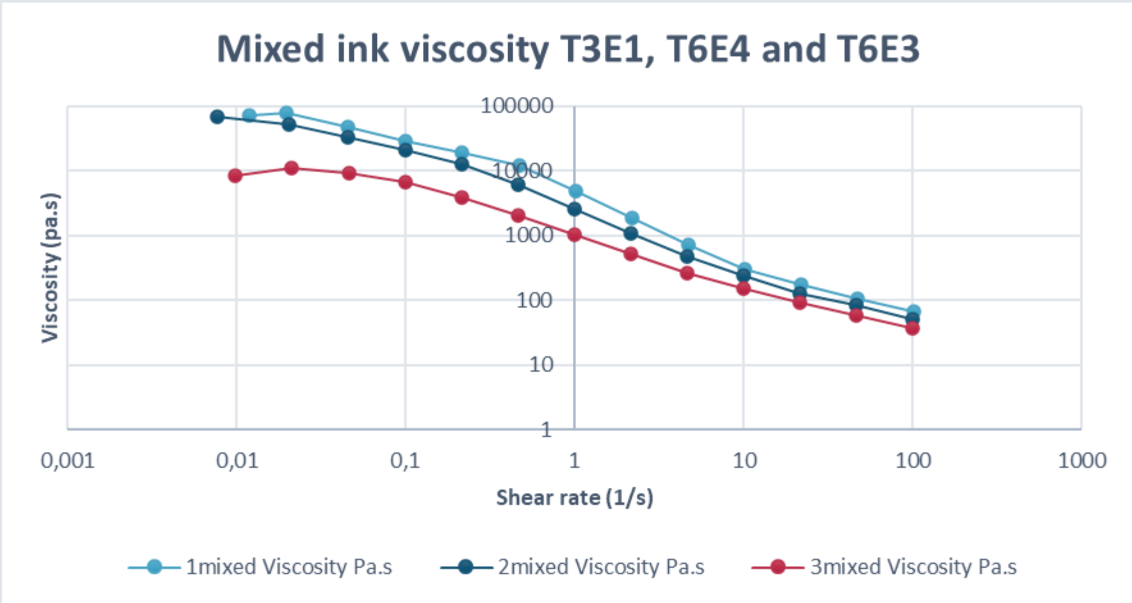
Maximum measured kg: 19,069 kg

The T6E3 ink could be extruded and had the lowest force needed for it. This ink also did cure after it was mixed with the static mixing nozzle which makes this ink suitable for 3D printing.



This ink also shows the shear-thinning properties and has the lowest measured viscosity of all the tree inks that were tested in this experiment.

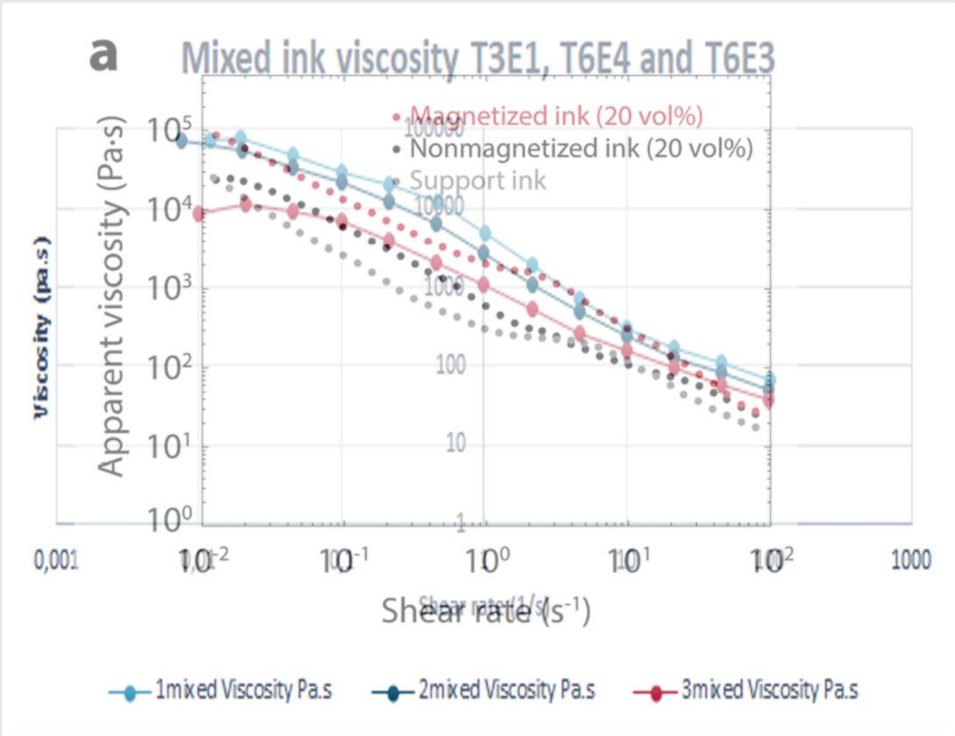
Compared



Legenda:

- T3E1 = 1mixed
- T6E4 = 2mixed
- T6E3 = 3mixed

In this graph, all the mixed inks are compared with each other. The viscosities correspond with the pressure that was needed to extrude the inks through the nozzles as well as the amount of fumed silica that was added. The T6E3 has a more distant viscosity than the T3E1 and the T6E4.
In the graph below the graph of the mixed inks is combined with the graph of the MIT paper (Kim et al., 2018). What can be seen is that T3E1 and T6E4 are a kind of following the viscosity of the magnetized ink of the MIT paper. T6E3 is more following the viscosity values of the non-magnetized ink.



Conclusion

Right now, the T6E4 ink performs the best through its good print stability as was found out in the ink test try 6 and it is also extrudable. Because T3E1 is too hard to extrude we will not continue with that ink.

Further Research

For further research, the nozzle extrusion test for ink T6E4 should be repeated to see if it cures.

The next steps will be to try to extrude the ink through the nozzles on a 3D printer, to see if the printer can produce the force needed to extrude the ink T6E4 through the v1 nozzle.

References

Kim, Y., Yuk, H., Zhao, R., Chester, S. A., & Zhao, X. (2018). Printing ferromagnetic domains for untethered fast-transforming soft materials. *Nature, 558*, 274-279. doi:10.1038/s41586-018-0185-0

Zhou, L.-y., Gao, Q., Fu, J.-z., Chen, Q.-y., Zhu, J.-p., Sun, Y., & He, Y. (2019). Multimaterial 3D Printing of Highly Stretchable Silicone Elastomers. *ACS Applied Materials & Interfaces, 11*, 23573-23583. doi:10.10.21/acsami.9b04873

Static mixing nozzle test 5

Sanne van Vilsteren
Houman Yarmand
28-05-2021

Introduction

In the last test, we chose to continue with the ink T6E4, because it could be extruded through the nozzle. This was not the case for T3E1, that was too viscous. During Static mixing nozzle test 4, we however think that we have not extruded enough ink to be mixed well, because the ink had not cured after the curing process in the oven. Due to this, we will repeat the extrusion experiment with T6E4, to see if it can cure. The sawn open static mixing nozzle from the last experiment showed some promising results.

Materials

Utensils

- Printed static mixing nozzles v1 for silicones adapted for syringes 2,4mm diameter printed fine (0,1 mm layer height)
- 10 ml syringes
- Scale
- Glass plate
- Oven
- Cups
- Mixing spatulas

Materials

Experiment 1: Repeat 75% fumed silica T6E4

- 4,2 g Ecoflex 00-10 part B
- 2 x 7,5 g Iron powder (carbonyl iron: 12)
- 4,2 g Ecoflex 00-10 part A
- 2 x 0,12 g fumed silica

Method

Prepare the ink

- Ecoflex part A and Ecoflex part B should be poured into different cups
- Measure the half amount of the iron carbonyl particles and add this to the cup of Ecoflex part A
- Measure the other half of the iron carbonyl particles and add these to a cup of Ecoflex part B
- Mix the Ecoflex part A and the iron carbonyl particles manually
- Mix the Ecoflex part B and the iron carbonyl particles manually
- Measure the half amount of the fumed silica and add this to the cup of Ecoflex part A
- Measure the other half of the fumed silica and add this to the cup of Ecoflex part B
- Mix the contents in the cup of Ecoflex part A manually
- Mix the contents in the cup of Ecoflex part B manually

At the end of this step, there should be two cups with a mixture of Ecoflex part A or B with half of the amount of the fumed silica and the iron carbonyl particles.

Prepare for extrusion

- Load one 10 ml syringe with the Ecoflex A mixture
- Load another 10 ml syringe with the Ecoflex part B mixture
- Level the two syringes so that they have the same amount of ink in them. This is important so that you can be sure that both syringes have loaded the nozzle with the same amount of ink. That is important because the Ecoflex only becomes a silicone if the two are mixed together. From earlier research, it was already found out that the mixing ratio of 1:1 (which is also the instruction on the Ecoflex bottles itself) work the best.
- Mount both of the syringes at the top of the static mixing nozzle

Extrusion

- Press simultaneously on both the syringes pistons with the same force with separate hands
- Move the static mixing nozzle to extrude a line of magnetic soft material on the glass plate
- Measure: In previous experiments, it took too much force to extrude the ink by hand and we had to press in through the static mixing nozzle by pushing the pistons of the syringes on a table.

Oven

The extruded ink should be cured in the oven at 120°C for one hour.

Results

The extruded T6E4 ink did cure (see figure 1).

Figure 1 Cured T6E4 ink mixed with static mixing nozzle v1

Conclusion

The T6E4 ink cured by the use of the static mixing nozzle 1. This means that the ink can be tried to be used on the double syringe 3D printer.

Further Research

For the next step in the research, it should be found out if the 3D printer has enough force to extrude this ink through static mixing v1. Then there can be decided if that printer can be used or if we have to adjust the static mixing nozzle.

B.c Magnetizer tests

These are the testes in which different inks got magnetized to see the effect of magnetization. For example, which particles are permanently magnetized, does the viscosity of the ink increase after magnetization and the proof of principle of magnetic soft material.

Magnetizer experiments: try 1

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Houman Yarmand
Andres Hunt
Sepideh Ghodrat
10 to 12-05-2021

Introduction

This test will be conducted during the first visit to the reactor institute where the particles in the ink for magnetic soft materials will be magnetized for the first time. Many things could be tested this first time using the magnetizer, but for now, is important to get the critical questions related to the magnetizing of the particles answered. Therefore, the focus of this test lays upon the question:

- Which of the magnetic particles has the strongest magnetic field after magnetizing?
    - Iron carbonyl powder
    - Fe_3O_4
    - SmCo
    - NdFeB

As this study is based on the study of Kim, Yuk, Zhao, Chester, and Zhao (2018), the strength of the magnetic field produced by the magnetizer will be 2,7 T. More information about the magnetizing process is not given.

Materials

Utensils

- 10 10 ml syringes
- Scales
- Cups
- Mixing spatulas
- Tape
- pen

Materials

Experiment 1.1 cured ink Iron carbonyl powder (ingredients needed 2x)

- 2,1 g Ecoflex 00-10 part B
- 7,5 g Iron powder (carbonyl iron: 12)
- 2,1 g Ecoflex 00-10 part A
- 0,12 g fumed silica

Experiment 1.2 cured ink Fe_3O_4 (ingredients needed 2x)

- 2,1 g Ecoflex 00-10 part B
- 4,8 g Fe_3O_4 powder
- 2,1 g Ecoflex 00-10 part A
- 0,12 g fumed silica

Experiment 1.3 cured ink SmCo (ingredients needed 2x)

- 2,1 g Ecoflex 00-10 part B
- 8 g Samarium Cobalt particles
- 2,1 g Ecoflex 00-10 part A
- 0,12 g fumed silica

Experiment 1.4 cured ink NdFeB (ingredients needed 2x)

- 2,1 g Ecoflex 00-10 part B
- 7,2 g NdFeB particles (<5 µm)
- 2,1 g Ecoflex 00-10 part A
- 0,12 g fumed silica

Experiment 2 uncured ink Iron carbonyl powder (Maybe ingredients needed 2x)

- 4,2 g Ecoflex 00-10 part B
- 2 x 7,5 g Iron powder (carbonyl iron: 12)
- 4,2 g Ecoflex 00-10 part A
- 2 x 0,12 g fumed silica

Methods

Preparation beforehand needed for all the experiments

Cut off the taps on the 10 ml syringes. Otherwise, the syringes will be too broad and will not fit in the magnetizer. The tube in which the syringes will be loaded to be magnetized has a diameter of 2,3 cm.

Experiment 1

Mixing procedure

- Add the Ecoflex part B and the magnetic particles together
- Mix by hand
- Add the Ecoflex part A
- Mix by hand
- Add the fumed silica
- Mix by hand

Filling syringes

- After an ink recipe is mixed this should be loaded in a 10 ml syringe of which the tabs are cut off
- The piston of the syringe should be pushed forward so that all the ink is compressed at the end of the syringe near the opening
- The opening of the syringe should then be taped off
- Mark the syringe with which ink recipe is in it so that all the syringes can be separated from one another
- Let the ink cure in the syringe (see figure 6)

Experiment 2

Mixing procedure

- Ecoflex part A and Ecoflex part B should be poured into different cups

- Measure the half amount of the iron carbonyl particles and add this to the cup of Ecoflex part A
- Measure the other half of the iron carbonyl particles and add these to a cup of Ecoflex part B
- Mix the Ecoflex part A and the iron carbonyl particles manually
- Mix the Ecoflex part B and the iron carbonyl particles manually
- Measure the half amount of the fumed silica and add this to the cup of Ecoflex part A
- Measure the other half of the fumed silica and add this to the cup of Ecoflex part B
- Mix the contents in the cup of Ecoflex part A manually
- Mix the contents in the cup of Ecoflex part B manually
- At the end of this step, there should be two cups with a mixture of Ecoflex part A or B with half of the amount of the fumed silica and the iron carbonyl particles.

Filling syringes

- After the two ink components are made those should be loaded separately in different 10 ml syringes of which the tabs are cut off
- The opening of the syringe should then be taped off
- Mark the syringes with which ink recipe is in which so that they can be separated

Magnetization

All the samples have been magnetized in the Reactor Institute with a maximum field of 3T.

Indicating magnetization and the strength

To check the magnetization the cured ink samples should be taken out of the syringes. This can be done by sawing them open. Then with a block magnet, there should be tested if the sample has a distinct north and south pole (see figures 7, 8, 9 and 10). If one side is attracted to one of the poles and pushed off the other.

Making the first magnetization patterns

To make the first magnetization patterns the magnetized uncured A and B component of the T6E4 ink should be mixed together in a 1:1 ratio. This mixed ink should be put into a syringe. At the tip of the syringe, a ring magnet should be mounted (see figure 1 and 4). This can be done by using tape. Then lines should be extruded on a glass plate (see figure 3).

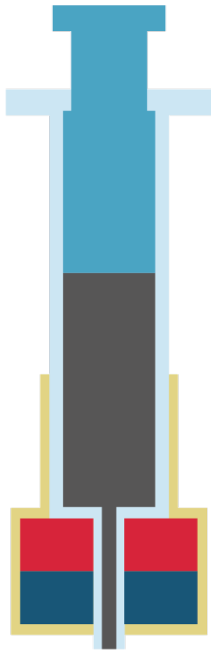

Figure 1 Schematic representation of the syringe with the attached magnet at the nozzle

In our experiment, we wanted to create some simple magnetization patterns as the extruded line in the MIT paper (Kim et al., 2018) (see figure 2).

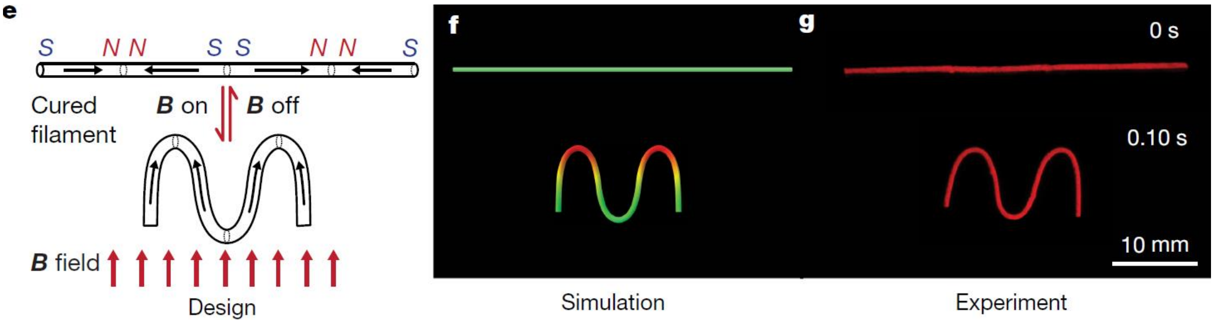

Figure 2 Magnetization pattern in one extruded rod

To make these with a permanent magnet two lines should be extruded separately in opposite directions as shown in figure 3 and joined together in the middle.

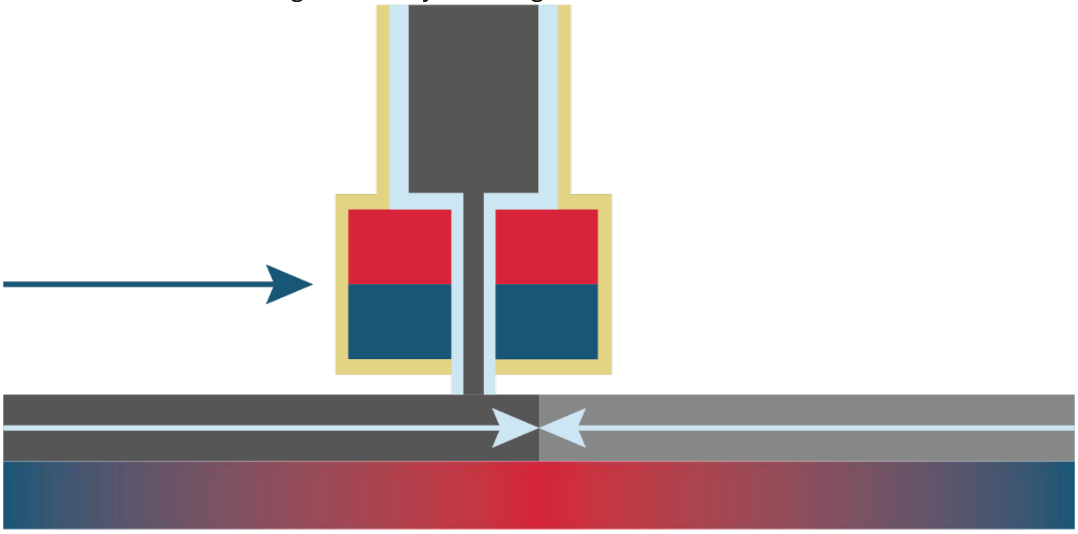
Figure 3 Extrusion of two different lines of ink that join in the middle to create a magnetization pattern


Figure 4 Syringe with mixed ink and a ring magnet at the nozzle

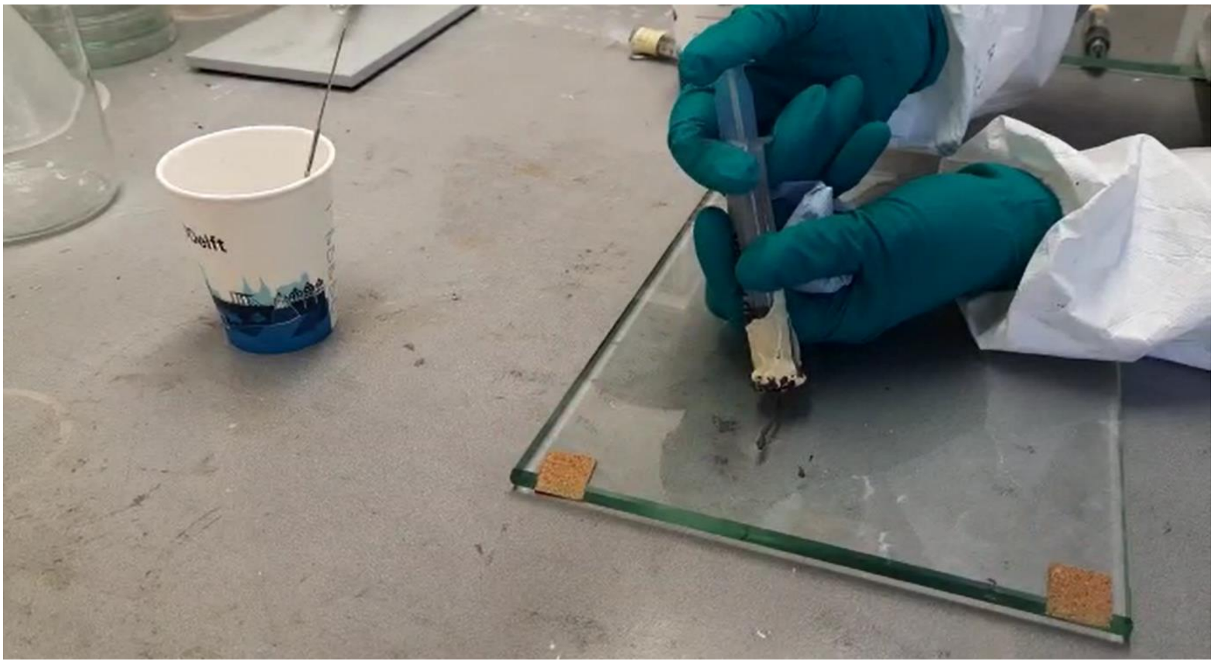

Figure 5 Extrusion of the mixed magnetized ink through the syringe with the mounted on ring magnet

Results

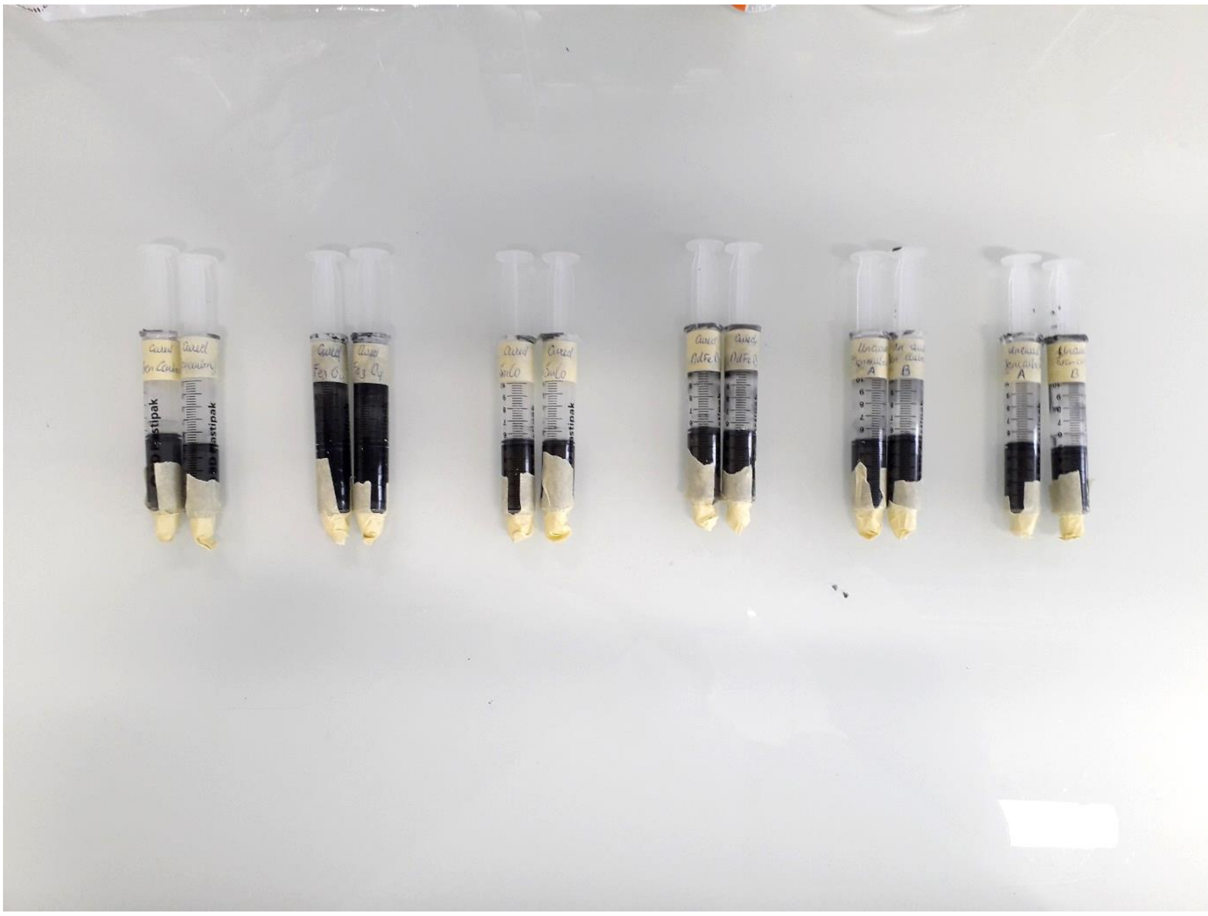

Figure 6 Magnetized ink samples in the syringes with the cut of tabs

Experiment 1

The magnetized samples were taken out of the syringes and tested if they had gotten detectable distinct magnetic poles (see figures 7, 8, 9 and 10). This was the case for the NdFeB particles and the SmCo particles. Of those, the NdFeB particles showed a response over a bigger distance than the SmCo particles.

The distinction of the magnetic poles could be detected the best when the poles should repel each other and therefore the sample should be pushed further away from the magnet. In the experiment of figure 8 when the same poles came near each other, the sample turned around quickly so that it could be attracted. In figure 10 the sample rolled away because it was pushed by the same magnetic field.

Iron carbonyl powder and Fe_3O_4 did not show distinctive north and south poles, in figures 7 and 9 they were attracted to the magnet. That was the same for the NdFeB and the SmCo particles, but in figures 8 and 10 they were still attracted to the magnet. This was interesting because the NdFeB and SmCo particle samples were repelled or turned around. Also, the gap between the magnet and the sample in the case of the iron carbonyl and Fe_3O_4 particles had to be very small (1 to 2 mm) to be attracted by the magnet.

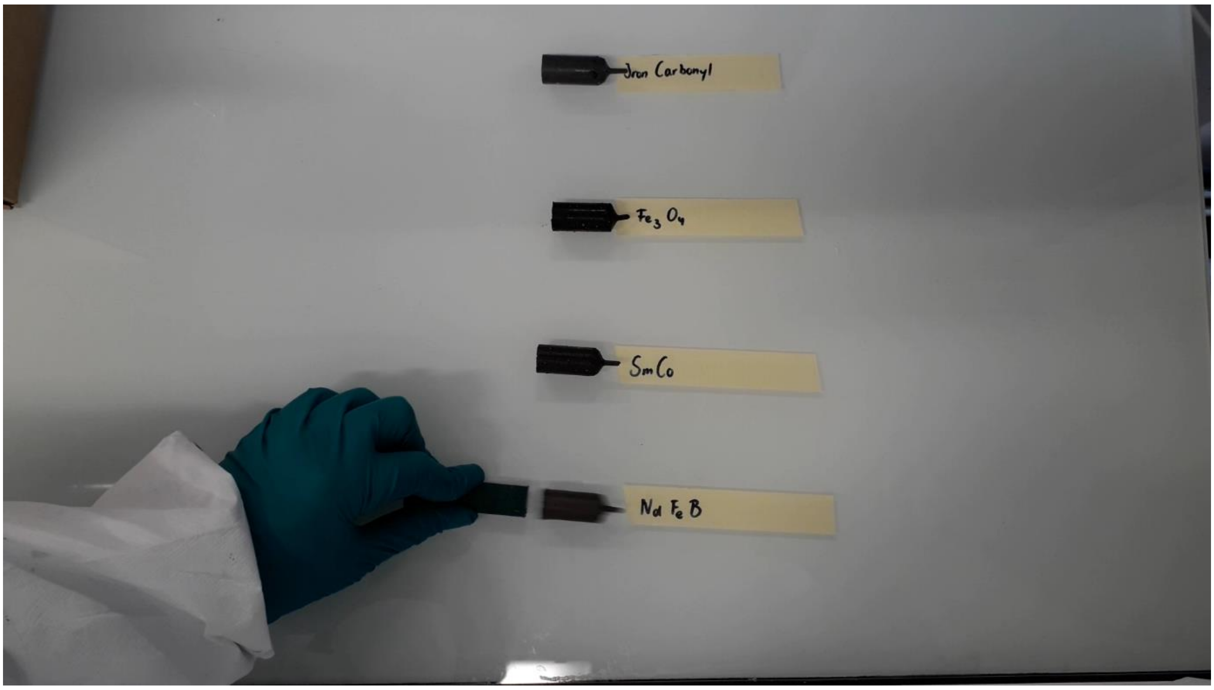

Figure 7 Attracting the samples

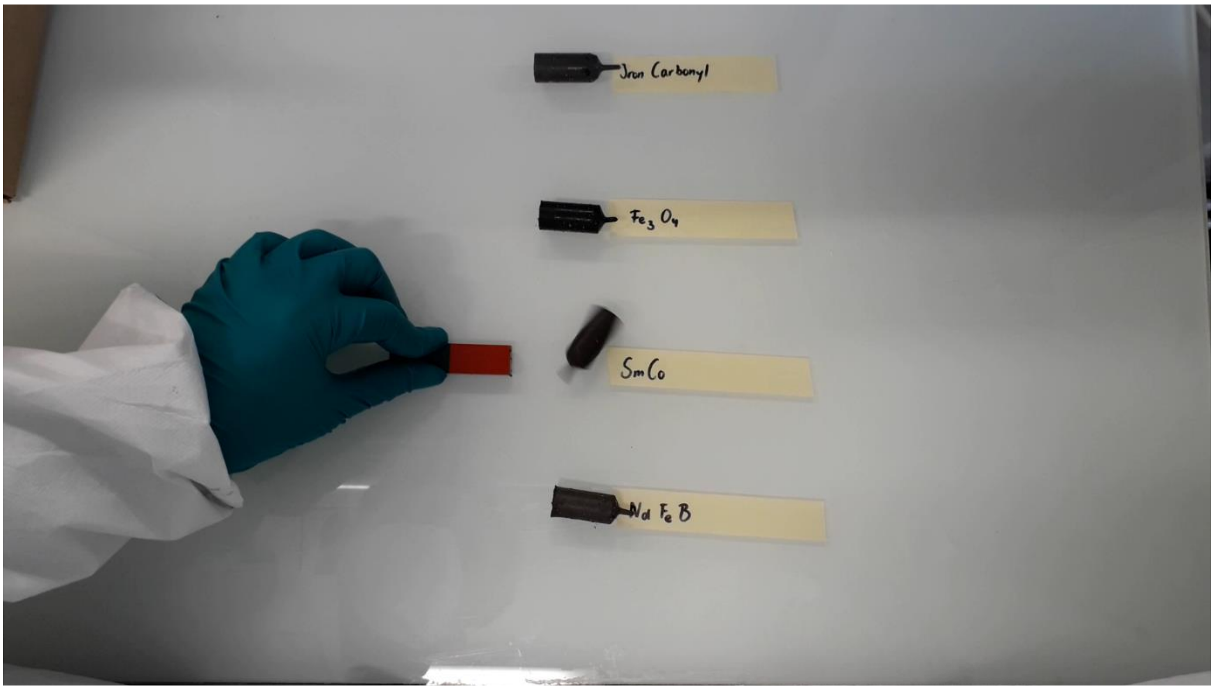

Figure 8 Repelling the samples, makes them turn around so that two opposite poles can attract each other

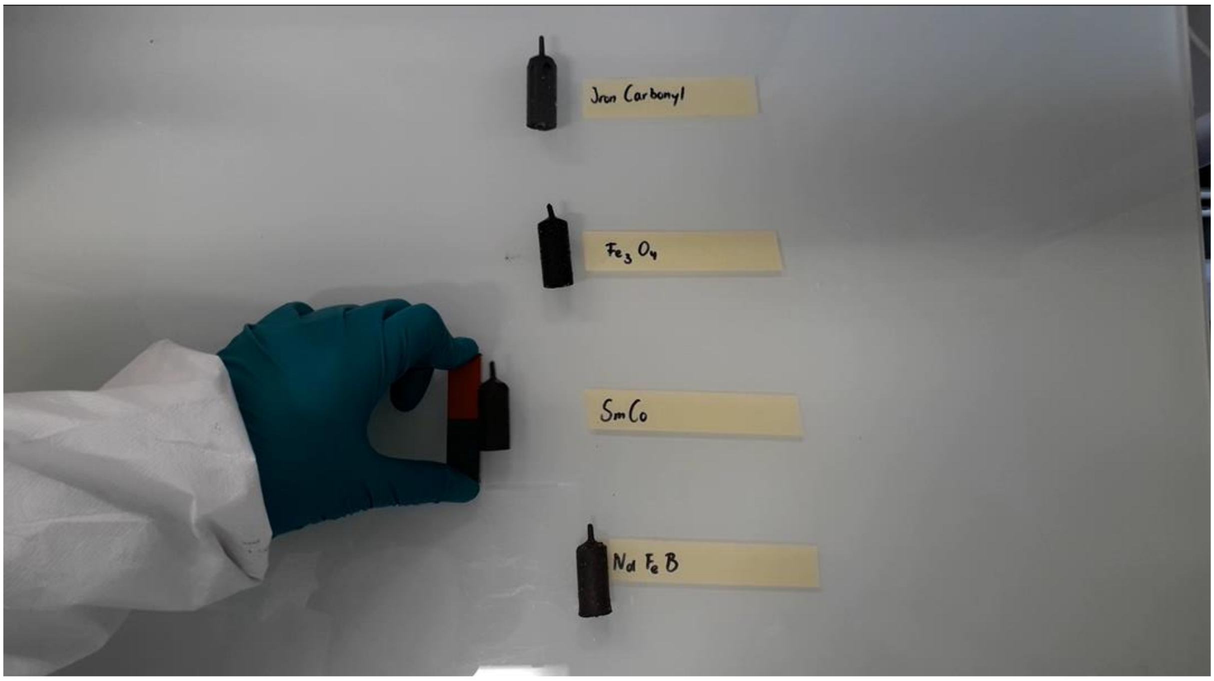

Figure 9 Attracting the samples

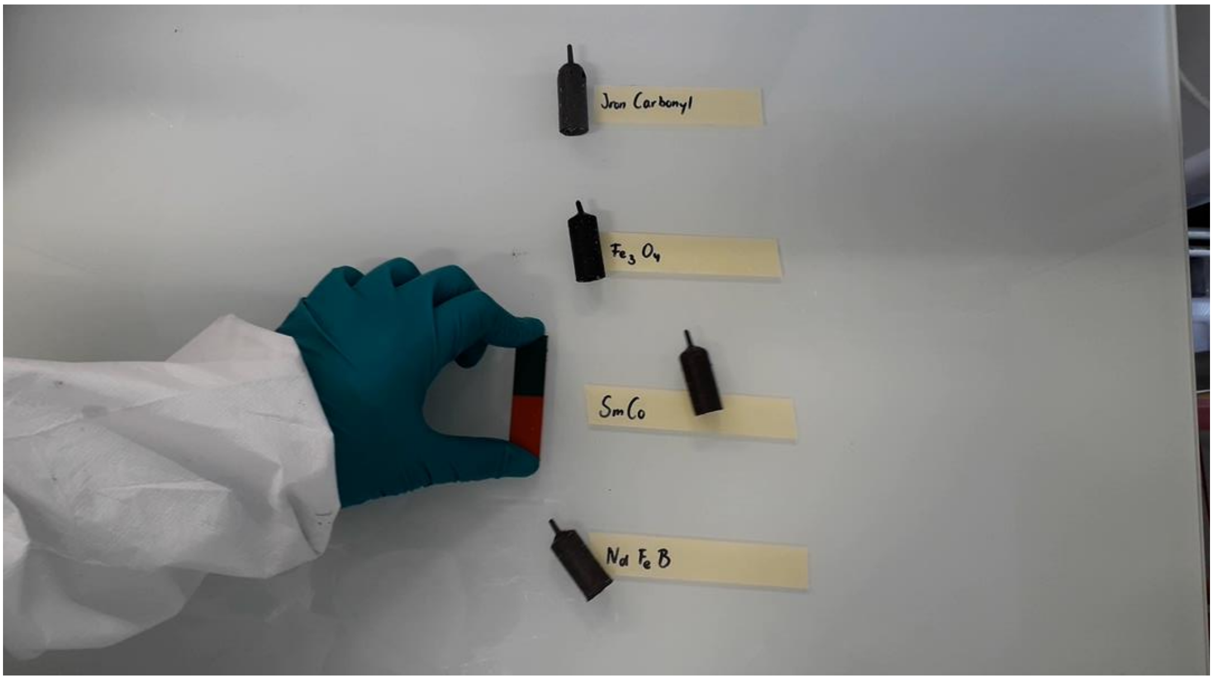

Figure 10 Repelling the samples, makes them role away. The NdFeB particles have a big field strength and turn to the magnet.

Experiment 2

During the first extrusion of the ink, a lot was learned. We had not used a magnetic shield between the extruded ink and the ring magnet attached to the nozzle. During the extrusion, with strong ring magnets near to the ink, the magnetic field was in some cases so strong that it pulled off the ink of the plate so that it would attach to the magnet (see figure 11). Due to this later in this explorative research part, we moved the magnet higher up the syringe.

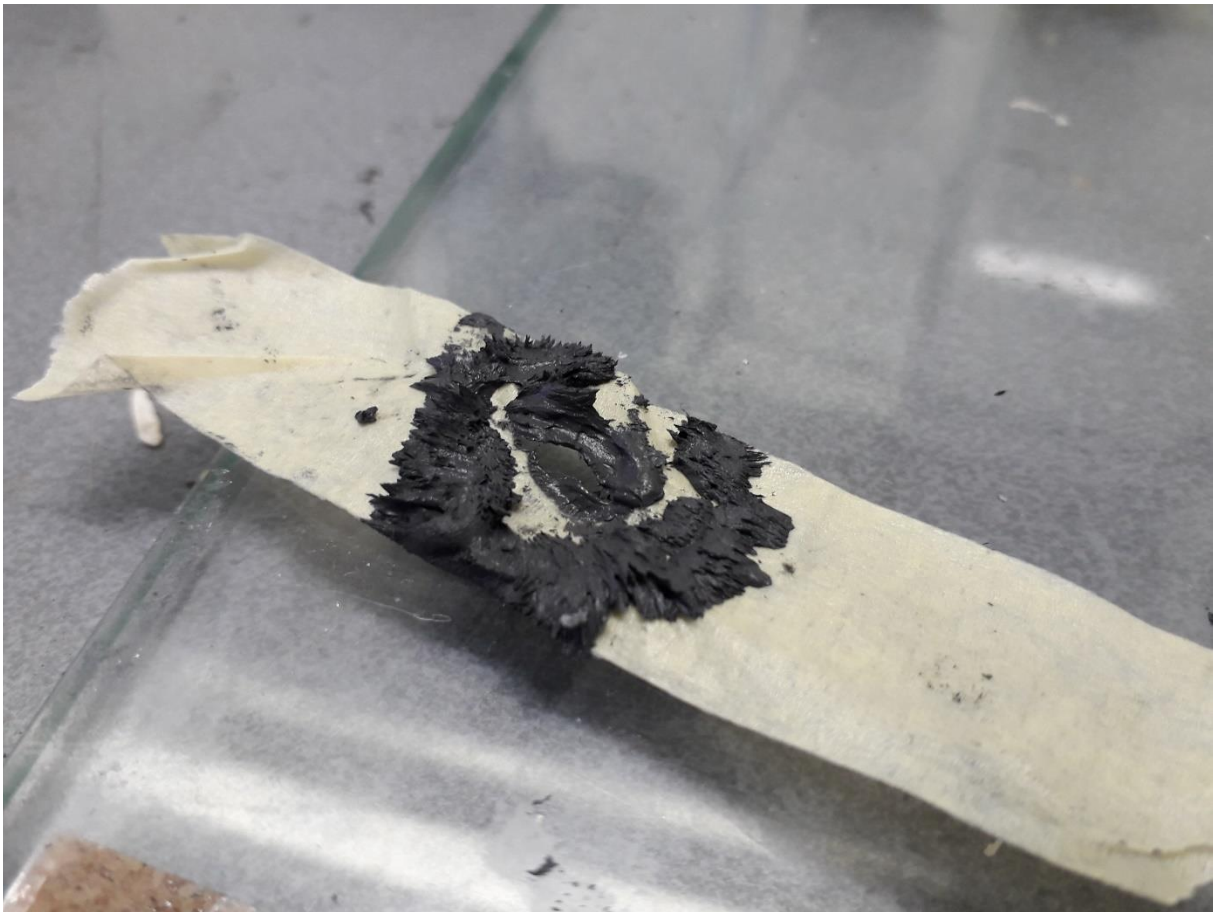

Figure 11 Ink that got attracted by the magnet that was attached to the syringe. The spikes in the ink show the magnetic field of the ring magnet

It was hard to say if the printed samples worked. This was due to the material not showing distinct north and south poles in the first experiment. One of the extruded strips seam to show some shape transformation. In figure 12 this strip is shown on a disc magnet and the ends of the strip curl up. This however does not say that much.

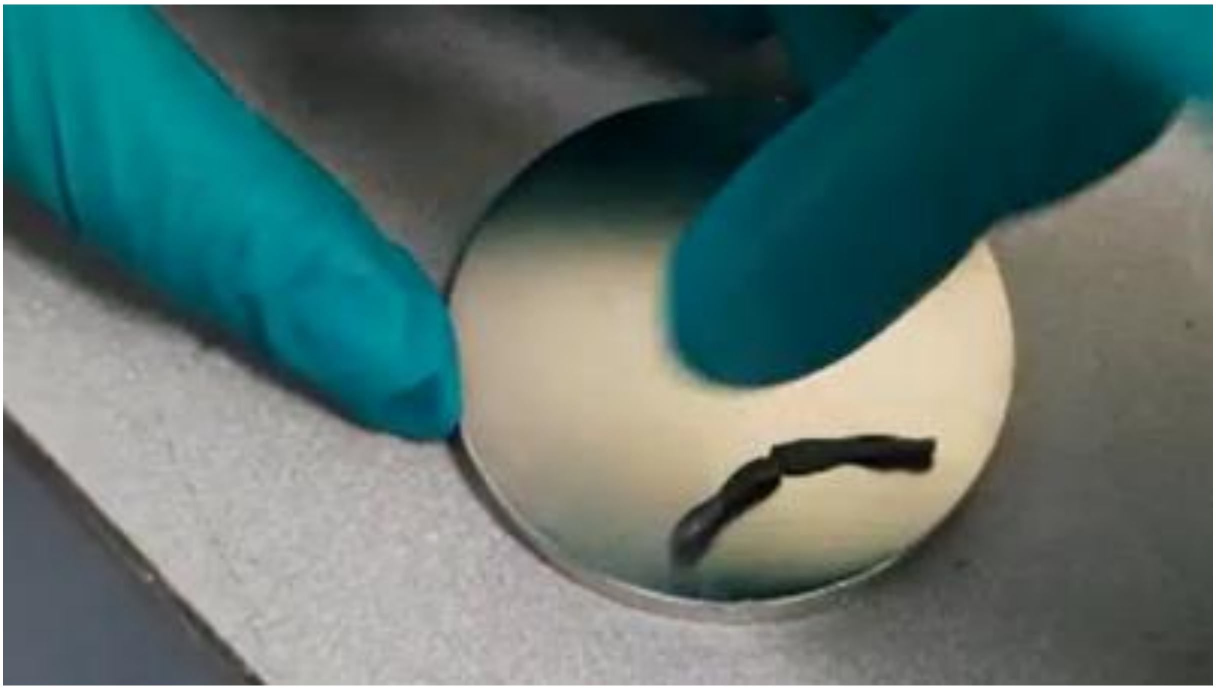

Figure 12 The ends of the extruded strip curl up in the magnetic field of the Neodymium magnet

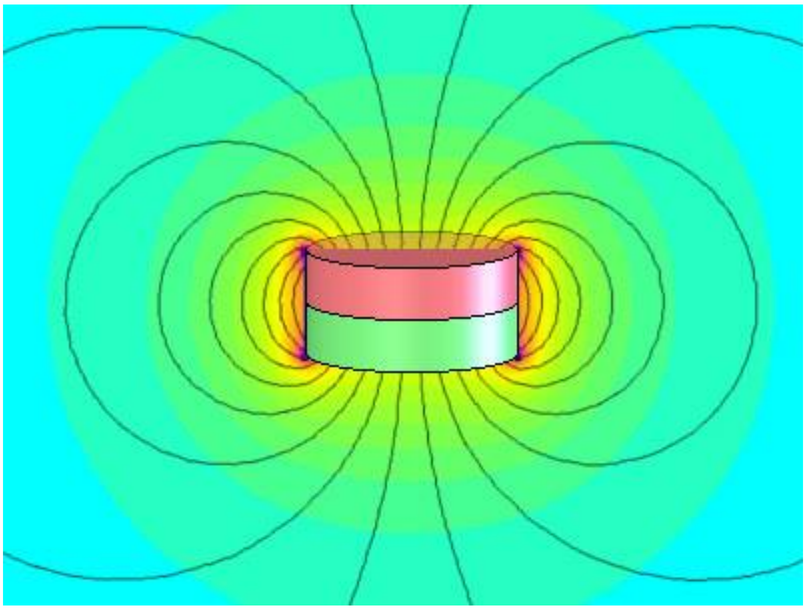

Figure 13 Magnetic field of a disc magnet (K&J Magnetics, n.d.)

Conclusion

The NdFeB and SmCo particles did show distinct north and south poles, and there the effect of the magnetizer can be seen. Because the NdFeB particles have become the strongest magnets, they show the most potential for this project, therefore we will continue with the NdFeB particles.

That the iron carbonyl powder and Fe_3O_4 did not show these distinct north and south poles might be due to the low coercivity of these particles. This means that through a relatively low magnetic field strength they can become demagnetized.

During the extrusion, we found out that a magnetic shield is required for printing the magnetization patterns.

Another thing that we should think of in the futures is to create and magnetic activation field with parallel magnetic field lines. Right now, we tried to activate the samples with a disc magnet, and as can be seen in figure 13, those field lines are not parallel. If we guaranty a parallel field the next time, we know what we can or should expect of the printed samples, regarding the shape transformation.

Further Research

To show the effect of the magnetizer in magnetizer experiment 1B the same four cured samples will be made.

A magnetic shield should be made or bought for the extrusion of the materials.

A parallel magnetic field should be created for the activation of the samples.

For tests about magnetization, the NdFeB particles should be used. Even though in following tests iron carbonyl particles can show up, to test certain things for which the magnetization is not required. This we will do to lower the costs of the experiments as well as not wasting rare earth metals which have a high impact on the environment.

References

K&J Magnetics, I. (n.d.). Magnets with an OFF Switch. Retrieved from https://www.kjmagnetics.com/blog.asp?p=magswitch

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Magnetizer experiments: try 1B

Sanne van Vilsteren
Houman Yarmand
28-05-2021

Introduction

In this experiment, we will remake some of the samples from try 1. However, this time the samples will not be magnetized so that we can compare the results of the magnetization to the non-magnetized samples.

Materials

Utensils

- 4 10 ml syringes
- Scale
- Cups
- Mixing spatulas
- Tape
- pen

Materials

Experiment 1.1 cured ink Iron carbonyl powder

- 2,1 g Ecoflex 00-10 part B
- 7,5 g Iron powder (carbonyl iron: 12)
- 2,1 g Ecoflex 00-10 part A
- 0,12 g fumed silica

Experiment 1.2 cured ink Fe_3O_4

- 2,1 g Ecoflex 00-10 part B
- 4,8 g Fe_3O_4 powder
- 2,1 g Ecoflex 00-10 part A
- 0,12 g fumed silica

Experiment 1.3 cured ink SmCo

- 2,1 g Ecoflex 00-10 part B
- 8 g Samarium Cobalt particles
- 2,1 g Ecoflex 00-10 part A
- 0,12 g fumed silica

Experiment 1.4 cured ink NdFeB

- 2,1 g Ecoflex 00-10 part B
- 7,2 g NdFeB particles (<5 µm)
- 2,1 g Ecoflex 00-10 part A
- 0,12 g fumed silica

Method

Mixing procedure

- Add the Ecoflex part B and the magnetic particles together
- Mix by hand
- Add the Ecoflex part A
- Mix by hand
- Add the fumed silica
- Mix by hand

Filling syringes

- After an ink recipe is mixed this should be loaded in a 10 ml syringe
- The piston of the syringe should be pushed forward so that all the ink is compressed at the end of the syringe near the opening
- The opening of the syringe should then be taped off
- Mark the syringe with which ink recipe is in it so that all the syringes can be separated from one another
- Let the ink cure in the syringe

Comparing the magnetized samples with the non-magnetized samples

In this step, the magnetized and the non-magnetized samples that were made from the same recipes should be compared. This can be done by showing the difference in their response on a permanent magnet and showing magnetization patterns with flux paper or iron fibres.

Results

Figure 1 NdFeB particles are not attracted by the magnetic field, which was the case for this orientation of the magnet in experiment 1

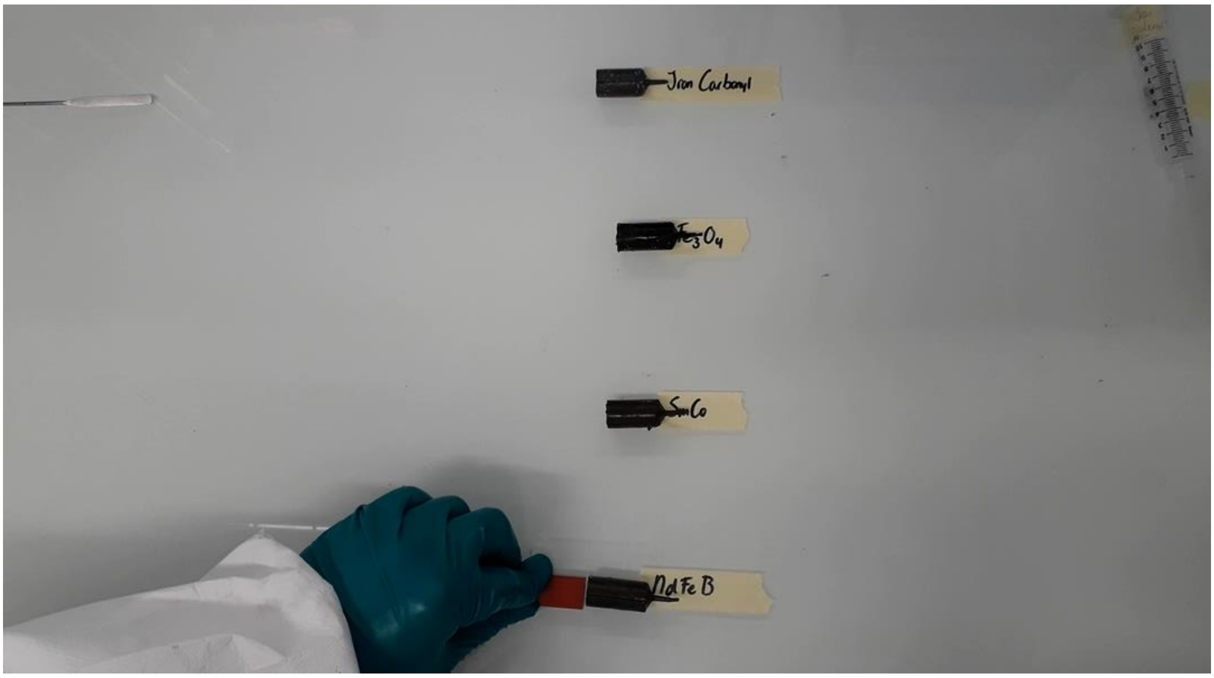


Figure 2 The NdFeB sample is not pushed away as was the case in magnetizer experiment 1 with this orientation of the magnet

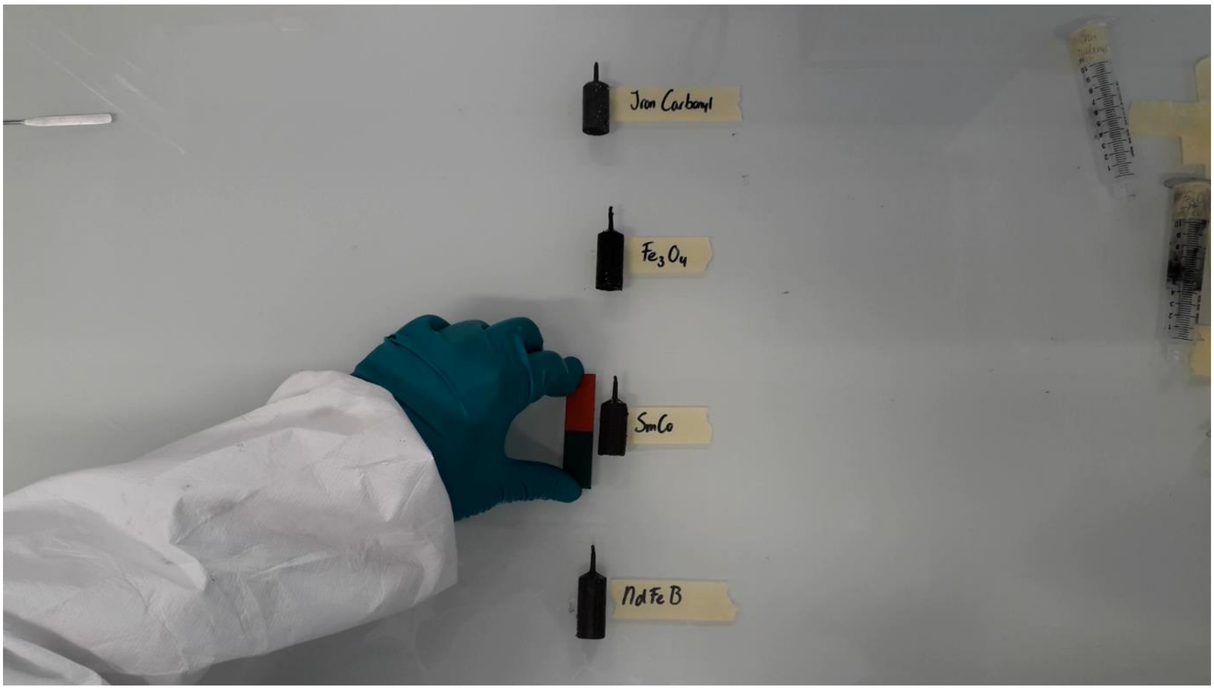


Figure 3 SmCo particles are not attracted by the magnetic field, which was the case for this orientation of the magnet in experiment 1

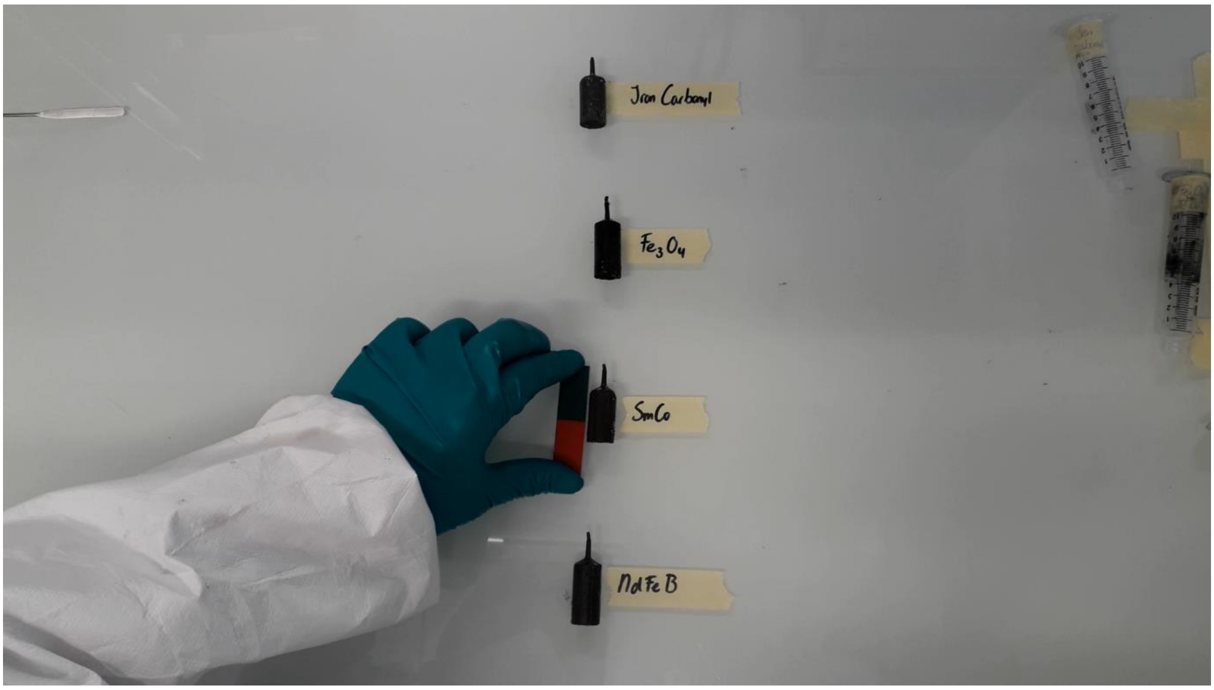


Figure 4 The SmCo sample is not pushed away as was the case in magnetizer experiment 1 with this orientation of the magnet

The NdFeB particles and the SmCo particles this time did not respond to the magnet. When the magnet held close to them, at a millimetre distance (see figures 1 to 4), the NdFeB and SmCo samples did not move.

The iron carbonyl powder and Fe_3O_4 did show a kind of similar responses as in experiment 1. They were attracted to all the orientations of the magnet.

Conclusion

The magnetizer does influence the behaviour of the magnetic particles.

Magnetizer experiments: try 2

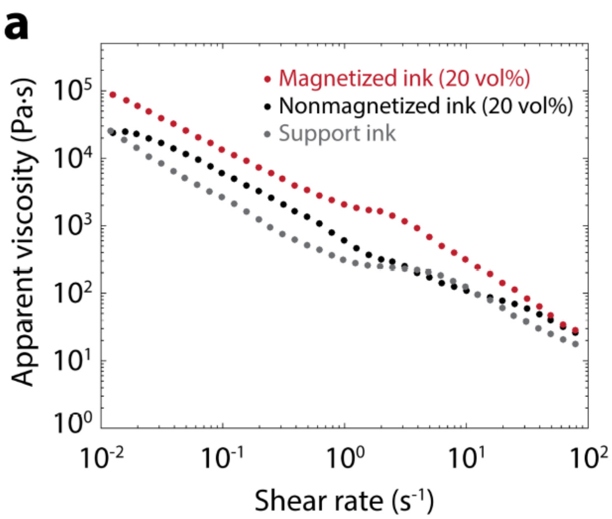
Sanne van Vilsteren
Houman Yarmand
Sepideh Ghodrat
30-08-2021 and 02-09-2021

Introduction

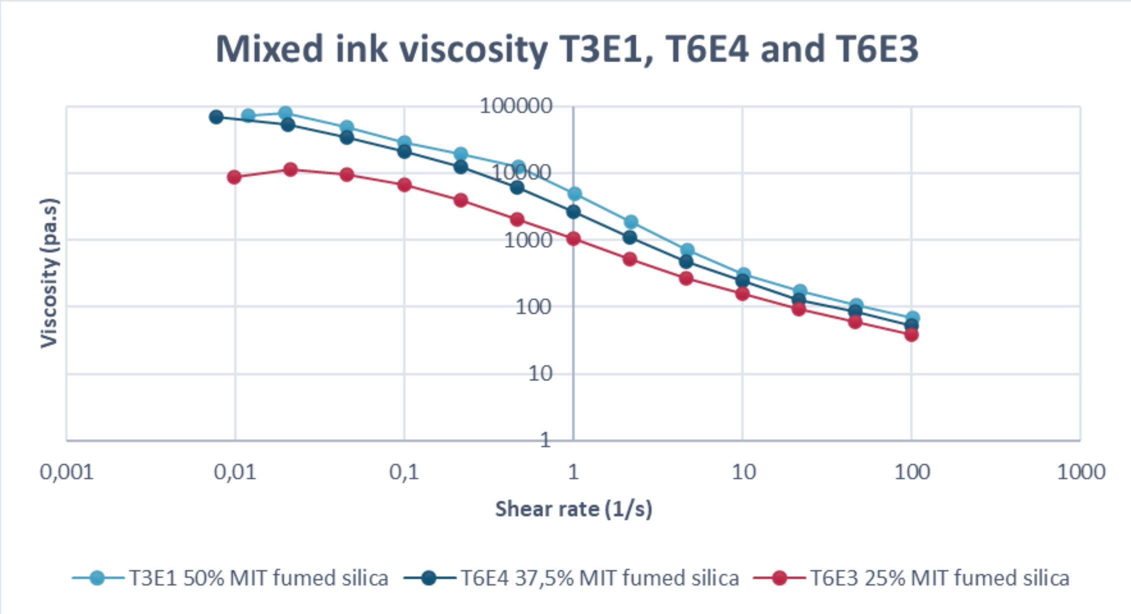
The purpose of these tests is to see if the magnetic particles agglomerate when a two-component ink is mixed together. How much the ink's viscosity increases when it goes from non-magnetized to magnetized particles and if the first magnetic soft materials can be made with a magnetization pattern.

Also, the magnetic properties of the magnetized NdFeB sample of magnetizer test 1 will be measured as well as the non-magnetized NdFeB sample from magnetizer test 1B that will be magnetized before its measurement. This will be done to see if some of the magnetizations degrade over time.

For these tests, a new ink will be made. This is due to the expected viscosity increase, based on the MIT paper. In the figure below can be seen that the viscosity of the ink of MIT increased by 65.000 Pa·s from 25.000 Pa·s to 90.000 Pa·s (Kim, Yuk, Zhao, Chester, & Zhao, 2018).



One of the inks that worked to extrude without problems was the T6E3 which had the lowest viscosity (see figure below). It has a lack of a good print stability, but for this project, there is no time left to improve it. The viscosity of the T6E3 at the start is 8063,14 Pa·s. This is already less than the viscosity increase of MIT. To lower the viscosity, but keep the magnetic performance the same, the fumed silica could be adjusted but can never reach a value by subtracting 65.000 Pa·s. Therefore for this test, one ink will be made with only the half amount of fumed silica in comparison with the amount of fumed silica in T6E3. The fumed silica will not be removed, because it gives the shear-thinning properties to the ink which are necessary for printing.



Materials

Utensils

- 3 10 mL syringes with tabs
- 6 10 mL syringes with cut off tabs
- scale
  - One with three decimals to make the ink
- Cups
- Mixing spatulas
- Tape
- Pen
- Rheometer
- Neodymium ring magnets that fit around the syringes
- Magnetic shield
- Glass plate

Recipes

The amount that was made in a previous experiment for 3D printing had a 30% increase relative to the first recipe's. That way 17 mL of ink of one component could be made which fills a bit more than 1,5 syringes. For this experiment, it is preferred to have 20 mL of ink of one component. Then around two times, 5 mL can be used for the viscosity tests and 10 mL to try to make working magnetic soft material. For that, the increase will be 50% instead of 30%.

Also, the Iron carbonyl powder will be changed again to NdFeB particles, which can be magnetized permanently and will be used for the working magnetic soft material.

Density Iron carbonyl powder: 7,86 g/mL (Merck, n.d.)
Density NdFeB: 7,6 g/cm^3 (magnets, 2018)
22,5/7,86*7,6 = 21,7 g

Experiment 1: two component T6E3 50% fumed silica and NdFeB particles: T8E1

- 12,6 g Ecoflex 00-10 part B
- 2 x 21,7 g NdFeB powder
- 12,6 g Ecoflex 00-10 part A
- 2 x 0,12 g fumed silica

50% increase (results in 20 mL of ink)

- 18,9 g Ecoflex 00-10 part B
- 2 x 32,55 g NdFeB powder
- 18,9 g Ecoflex 00-10 part A
- 2 x 0,18 g fumed silica

Method

Prepare the ink

- Ecoflex part A and Ecoflex part B should be poured into different cups
- Measure the half amount of the NdFeB particles and add this to the cup of Ecoflex part A
- Measure the other half of the NdFeB particles and add these to a cup of Ecoflex part B
- Mix the Ecoflex part A and the iron carbonyl particles manually
- Mix the Ecoflex part B and the iron carbonyl particles manually
- Measure the half amount of the fumed silica and add this to the cup of Ecoflex part A
- Measure the other half of the fumed silica and add this to the cup of Ecoflex part B
- Mix the contents in the cup of Ecoflex part A manually
- Mix the contents in the cup of Ecoflex part B manually

At the end of this step, there should be two cups with a mixture of Ecoflex part A or B with half of the amount of the fumed silica and the NdFeB particles.

Prepare the syringes

- Cut off the tabs of all the syringes, otherwise, they will not fit in in the magnetizer
- Fill all the syringes with the cut of tabs and 2 of the whole syringes up to 5 mL, so only halfway full. Otherwise not every part of the ink will see the magnetic field in the magnetizer and might not get magnetized.

At the end of this step, there should be around four half-filled syringes of each ink component that is made.

Measure viscosity

This time the viscosity of the ink should be measured in six conditions instead of 3 in the previous experiments. Those are:

- Ink component A non-magnetized separately
- Ink component B non-magnetized separately
- The non-magnetized ink after it is just mixed
- Ink component A magnetized separately
- Ink component B magnetized separately
- The magnetized ink after it is just mixed

This way we can get an insight into how the viscosity changes over the printing project.

The measuring procedure:

- 40-mm-diameter steel plate geometry.

- Steady-state flow experiments with a sweep of shear rates (0,0-100 s^{-1})
- The inks were brought to a temperature of 25°C for one minute before the experiment started.
- The gap height between the geometry and the plate always was 0,5 mm.

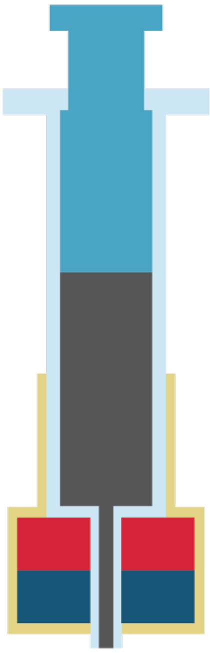


Figure 1 Schematic representation of the syringe with the attached magnet at the nozzle

Check on agglomeration

The magnetized ink components should be mixed and checked under the microscope on agglomeration.

Making the first magnetization patterns

To make the first magnetization patterns the magnetized uncured A and B components of the T6E4 ink should be mixed in a 1:1 ratio. This mixed ink should be put into a syringe. At the tip of the syringe, a ring magnet should be mounted (see figures 1 and 4). This can be done by using tape. Then lines should be extruded on a glass plate (see figure 3).

In our experiment, we wanted to create some simple magnetization patterns as the extruded line in the MIT paper (Kim et al., 2018) (see figure 2).

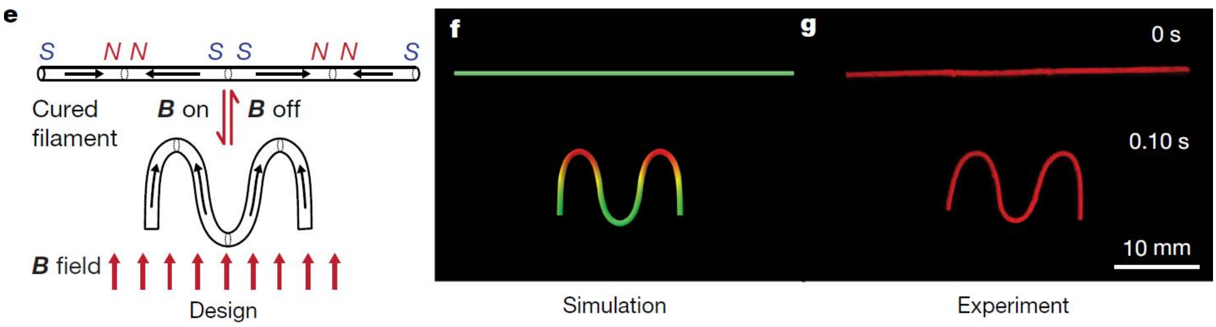


Figure 2 Magnetization pattern in one extruded rod

To make these with a permanent magnet two lines should be extruded separately in opposite directions as shown in figure 3 and joined together in the middle.

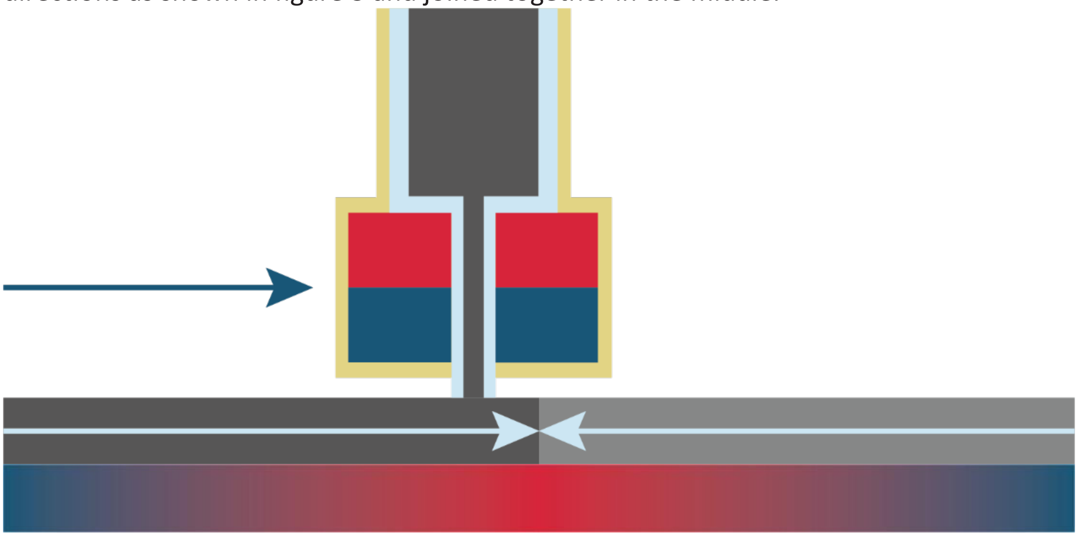


Figure 3 Extrusion of two different lines of ink that join in the middle to create a magnetization pattern

Figure 4 Syringes with mixed ink and a ring magnet at the nozzle. (left) Small NdFeB magnet at the nozzle. (right) Larger ferrite magnet, further up the syringe to prevent the magnetic field from pulling the extruded ink of the glass plate.

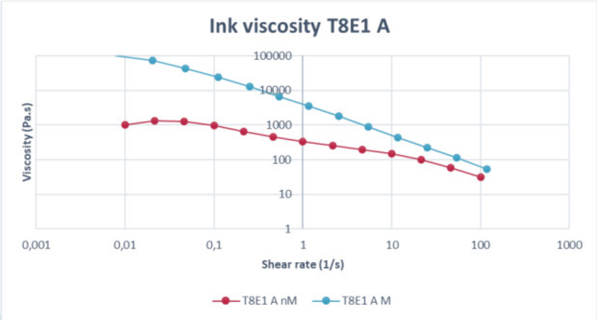
Results

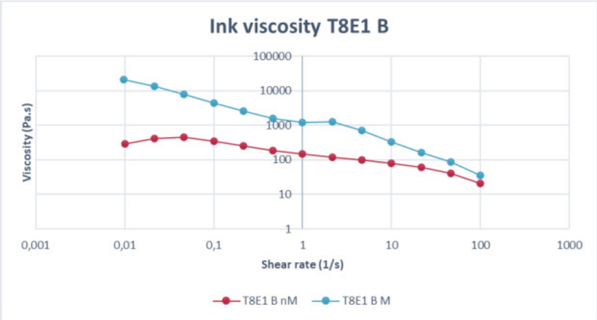
Viscosity

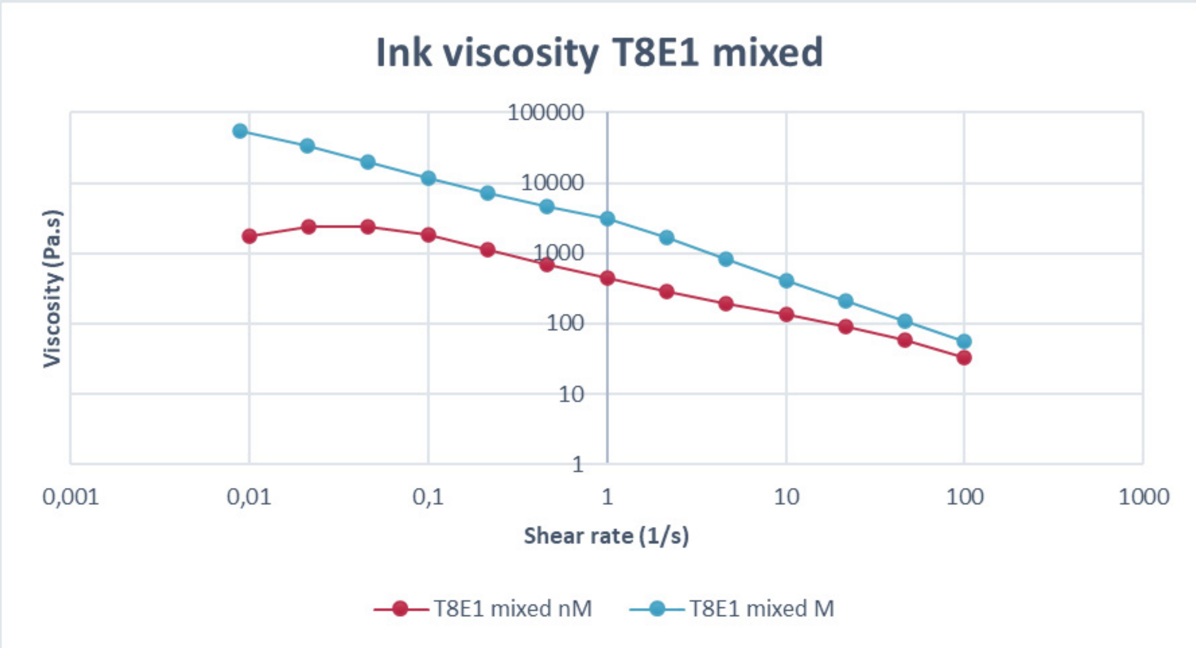
The viscosity of the non-magnetized (nM) T8E1 ink has increased a lot due to its magnetization (M) as was expected. The increase in viscosity for the three conditions is:

Part A: 99033,8 Pa·s
Part B: 21147,9 Pa·s
Mixed: 53978,5 Pa·s

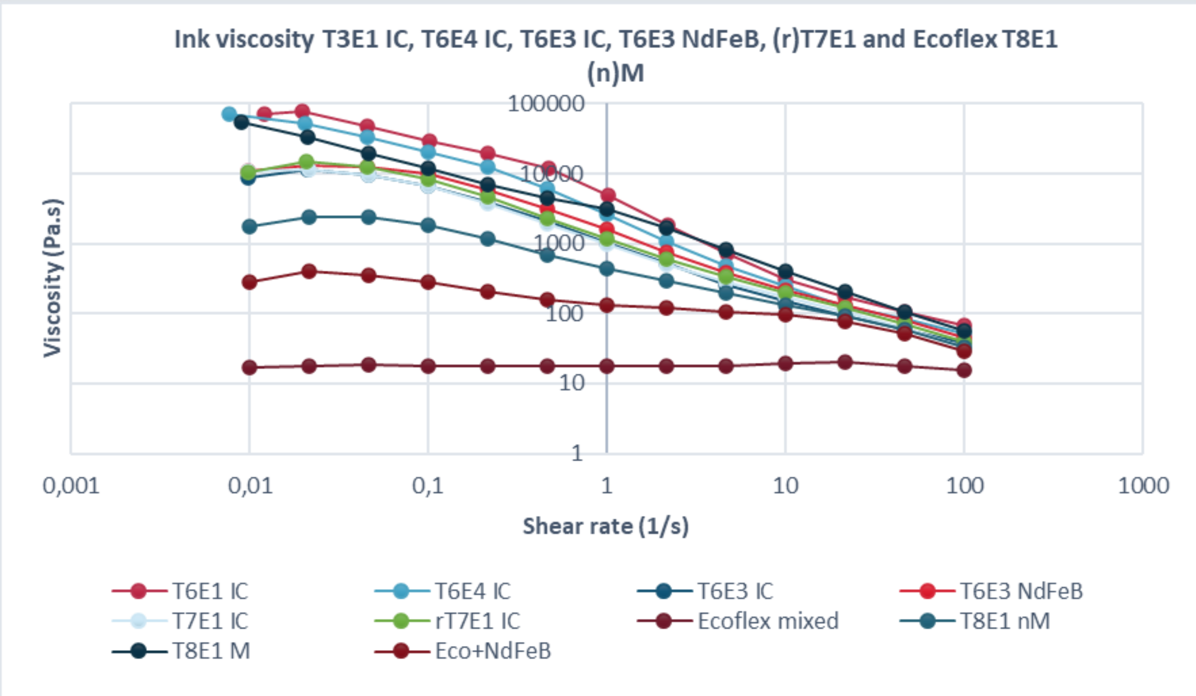
The mixed magnetized inks increase in viscosity only differentiates 16,96% from the increase of viscosity from the MIT paper.



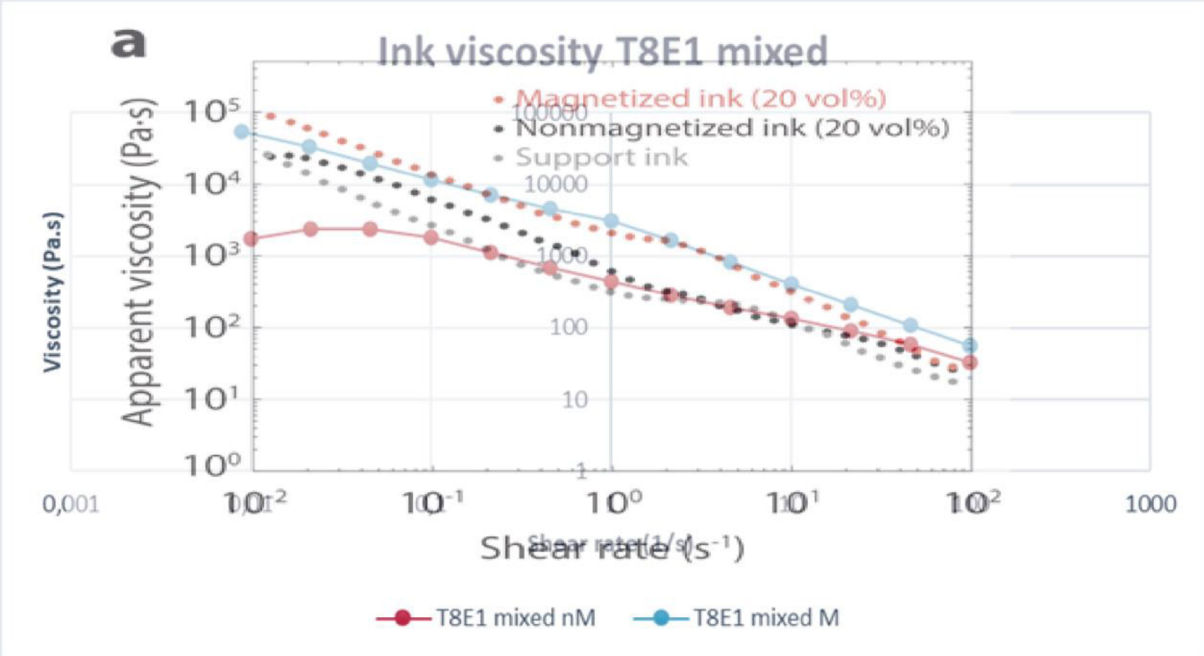




Ink viscosity T8E1 mixed
Viscosity (Pa.s)
100000
10000
1000
100
10
1
0,001
0,01
0,1
1
10
100
1000
Shear rate (1/s)
T8E1 mixed nM
T8E1 mixed M

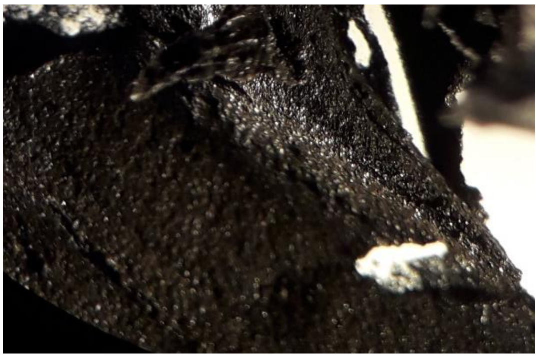


Ink viscosity T3E1 IC, T6E4 IC, T6E3 IC, T6E3 NdFeB, (r)T7E1 and Ecoflex T8E1 (n)M
Viscosity (Pa.s)
100000
10000
1000
100
10
1
0,001
0,01
0,1
1
10
100
1000
Shear rate (1/s)
T6E1 IC
T6E4 IC
T6E3 IC
T6E3 NdFeB
T7E1 IC
rT7E1 IC
Ecoflex mixed
T8E1 nM
T8E1 M
Eco+NdFeB



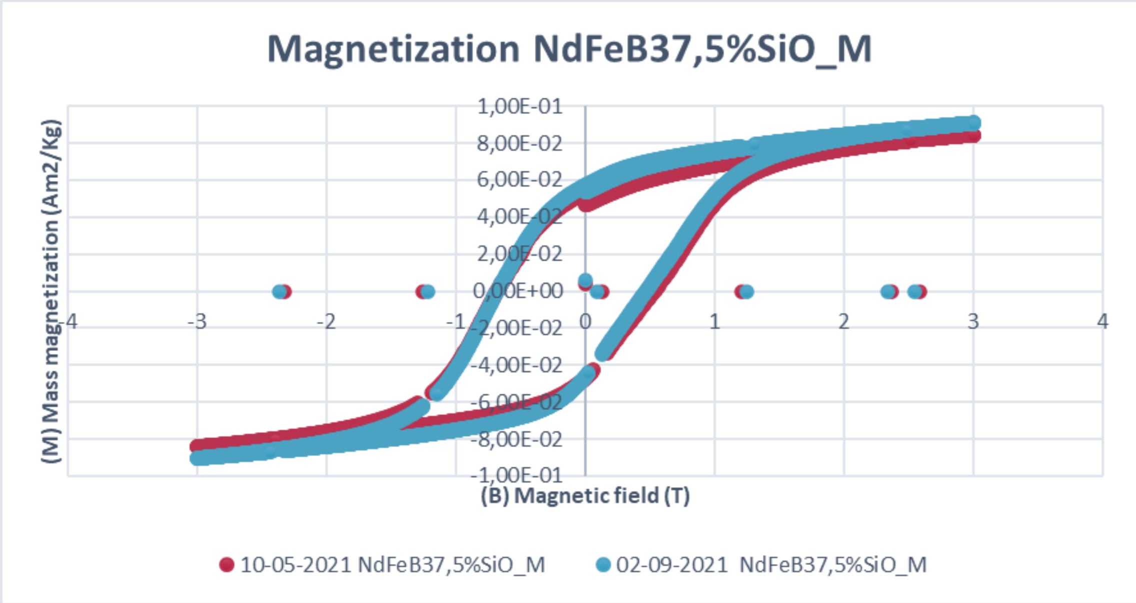
The magnetized T8E1 almost follows the same viscosity values of the magnetized ink from MIT, it only starts lower. The non-magnetized ink differs more from MIT's non-magnetized ink. When comparing the magnetized T8E1 with the other inks that were measured and tested in this project, it can be compared the best with the values of the higher viscosity inks T3E1 and T6E4. T3E1 never got extruded through the static mixing nozzle because too much force was needed. T6E4 until now is never successfully extruded through the 3D printer system. The rT7E1 ink, which has a lower viscosity than the magnetized T8E1 already seemed to be the limit of the 3D printer system. I, therefore, wonder, if this magnetized ink can even be used in the 3D printer system that we have right now. I think that that should be thought about before continuing with this project. If the viscosity increase through the magnetic field always will be in the range of 50.000 Pa·s, the viscosity will always end up in the higher viscosity segment of the inks that did not get extruded.

Agglomeration



The first picture is a microscope picture of unmagnetized ink and the second of the magnetized ink of this test. From the comparison of these two pictures, we cannot conclude if the particles do agglomerate or not. What was noticed throughout the experiment was that the particles still stayed distributed throughout the material.

Change in magnetic properties over time



The magnetization of the earlier magnetized sample was lower than the sample that was measured today by 6,9%. This can indicate the ageing of the magnetic properties. However, two different samples were measured and there could be slight differences in the number of particles or their distribution. For further research, I suggest measuring the change of magnetic properties of the same sample over time to eliminate these uncertainties.

Magnetic shield

The magnetic shield, made of first grade Non-Oriented Electrical Steel from Nippon Steel (Japan), was, unfortunately, no magnetic shield. It got attracted to the ring magnet that was used for the making of the magnetic soft material. During the extrusion of the ink lines, the magnetic field of the ring magnet pulled the ink from the glass plate. This means that other material should be used, like aluminium, or that a ticker one might be needed. This should be found out in later research.

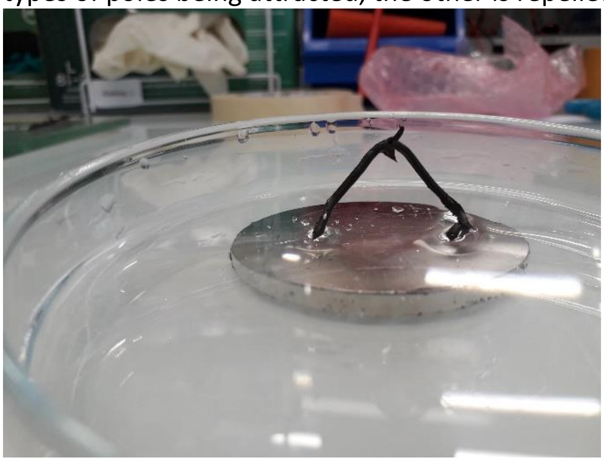
Ring magnets extrusion

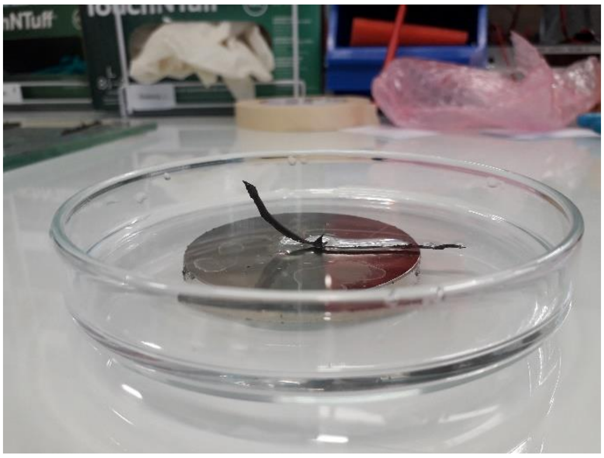
Because the magnetic shield did not work, the ferrite magnet that can be placed higher on the syringe (farther away from the printed material) did work the best to print the magnetic soft material. The neodymium magnet pulled the ink from the glass plate unless the syringe was moved very fast.

Working magnetic soft material

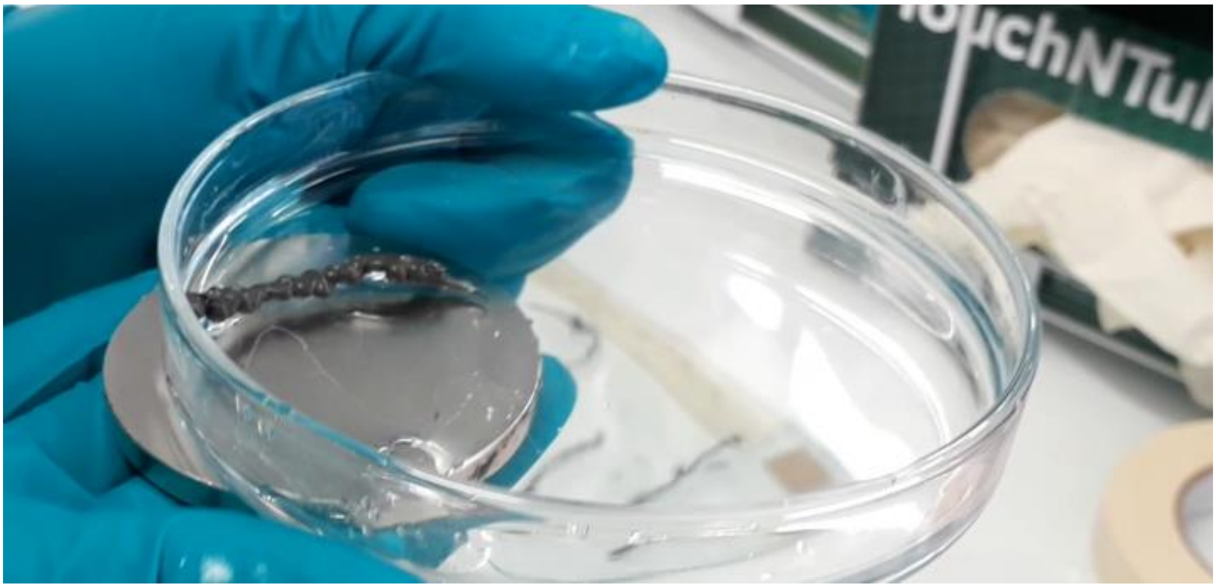
These are the three plates with printed magnetic soft material on top of there. The first two plates are made with the syringe with the neodymium magnet (figure 4 left) the last plate is made with the syringe with the ferrite magnet (figure 4 right). The strips that are printed with the neodymium magnet, have a magnetization pattern with one North and one South pole (→). Those are more jumping around the glass plate or only moving up on one side that is repelled by the magnetic field while the other side is kept down by the attraction of the magnet.

The ones that are printed with the ferrite magnet have a magnetization pattern of North-South-North (→←). These can form standing bows or ends that are standing up. Due to one of the two types of poles being attracted, the other is repelled.





It was noticed that the shape of the magnetic soft material, like the length and the thickness, influenced the movements of the magnetic soft material. When one side is too long it can be too heavy to be lifted as in the picture on the right, even though this might also be a problem with the magnetic field under which it was made, because it was not properly shielded. The thicker structure shown below was stiffer and therefore could not bend as easily as other materials and therefore did not move upwards as much.



Conclusion

This test provided the proof of principle of the magnetic soft material and proofs that it can be made. This means that the research into this material can be continued because there is a result. The material now is still very uncontrollable, so a better controllable manufacturing process is recommended. It was also shown how the thickness and length of the material influenced the movement of the magnetic soft material.

The viscosity increase of the non-magnetized T8E1 ink to the magnetized T8E1 was 53978,5 Pa·s. This was a 16,96% negative difference with the 65000 Pa·s increase of MIT but lays in the same number range. As described in the results, this increase makes the viscosity of the ink come in the region of inks that had a too high viscosity to be extruded through the static mixing nozzle or in the 3D printer system. It can at least been tried once to extrude it once. However, it might be good to reconsider the type of 3D printer system that is used.

What should change is the material that is used for the magnetic shield. The one that was used now was attracted to the magnet, and the magnetic field passed through. Therefore another material should be tried for the next test in which magnetic soft material ink is extruded.

The magnetization did not have a noticeable result on the agglomeration of the magnetic particles after mixing. This might have to be looked at with a better microscope. The magnetization of the NdFeB particles seems to degrade over time, but more measurements are needed to be sure of this.

Further research

One of the first things that should be found out is a new material that works for the magnetic shield of for printing magnetic soft materials. Because this one does not work and makes it hard to print without pulling the ink from the glass plate and influencing the printed magnetization patterns.

It can be tried to also magnetize the Ecoflex, NdFeB mixture because it showed shear-thinning properties itself. It can be checked if the increase of the viscosity stays in the same order of magnitude.

Later on, the 3D printer system should be tested again or discussed on the use, depending on the viscosity experiment outcomes.

The change of magnetic properties over time should in the future be measured with the same sample, to have clearer results.

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B.d 3D printer tests

In the 3D printer tests, the dual syringe printer was tested and improved, while also new types of ink were made that could be extruded by the system.

3D printer: try 1

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Zjenja Doubrovski
Houman Yarmand
02-06-2021

Introduction

This test is the first test of this project in which a 3D printer will be used to extrude the ink. For this experiment, we will use a double syringe 3D printer which will be able to extrude two components of the ink. We will use that in combination with the static mixing nozzle. The static mixing nozzle will mix the two components of the ink so that it can cure after it is printed.

In this test, we will use the T6E4 ink, which had good print stability. However, in static mixing nozzle test 4, we discovered that still a large force is needed to extrude this ink through the static mixing nozzle.

This experiment will find out if the 3D printer has enough force to extrude the T6E4 ink through static mixing nozzle v1 and if we can use the double syringe printer for this project, or not.

Materials

Utensils

- 2 60 ml syringes
- Scale
- Cups
- Mixing spatulas
- Tape
- Pen
- Double syringe 3D printer
- Clear rubber tubes (from the syringe to static mixing nozzle)
- The original static mixing nozzle v1 that can be used in the double syringe 3D printer

Experiment 1: two component T6E4

- 12,6 g Ecoflex 00-10 part B
- 2 x 22,5 g Iron powder (carbonyl iron: 12)
- 12,6 g Ecoflex 00-10 part A
- 2x 0,36 g fumed silica

Method

Prepare the ink

- Ecoflex part A and Ecoflex part B should be poured into different cups
- Measure the half amount of the iron carbonyl particles and add this to the cup of Ecoflex part A
- Measure the other half of the iron carbonyl particles and add these to a cup of Ecoflex part B
- Mix the Ecoflex part A and the iron carbonyl particles manually
- Mix the Ecoflex part B and the iron carbonyl particles manually

- Measure the half amount of the fumed silica and add this to the cup of Ecoflex part A
- Measure the other half of the fumed silica and add this to the cup of Ecoflex part B
- Mix the contents in the cup of Ecoflex part A manually
- Mix the contents in the cup of Ecoflex part B manually

At the end of this step, there should be two cups with a mixture of Ecoflex part A or B with half of the amount of the fumed silica and the iron carbonyl particles.

Prepare the syringes

- Load one 60 ml syringe with the Ecoflex A mixture
- Load another 60 ml syringe with the Ecoflex part B mixture
- Level the two syringes so that they have the same amount of ink in them (this is not necessary because the pistons are individually powered)

Prepare the 3D printer

- Attach the tubes to the syringes
- Attach the syringes to the static mixing nozzle
- (because the design is not optimized and the syringes do not have a Luer lock system, it is helpful to secure the tubes extra with the help of zip ties)
- Then the syringes should be put in the printer
- The mechanical parts that push the pistons of the syringes forward should be brought up, and the red clips should be attached.

Results

During the extrusion of the ink through the tubes, the motors started to make a ticking sound. The extruder was looked into and the motor skipped steps during the extruding. This happened while the ink was being extruded through the tubes, and had not yet reached the static mixing nozzle yet.

To tune the extrusion we found that when one of the syringes get the command to extrude a certain amount of ink, the number 241,66 = 1 ml.

Conclusion

The extrusion of the T6E4 ink took too much force for the printer and the motor skipped steps.

Further research

Because the printer did not have enough force for the ink to be extruded, we will do one more test to be certain of that. During the next test, the software of the extruder will be updated so that it is capable to send more power to the motors. Also the ink viscosity will be lowered by changing from the T6E4 to the T6E3 ink. Finally also the size of the syringes will be changed to 10 ml syringes, so that a smaller surface with the same force can produce a higher pressure. If the 3D printer after the alteration of these 3 parameters still is not able to extrude the ink, we will switch to another paste printer.

3D printer: try 2

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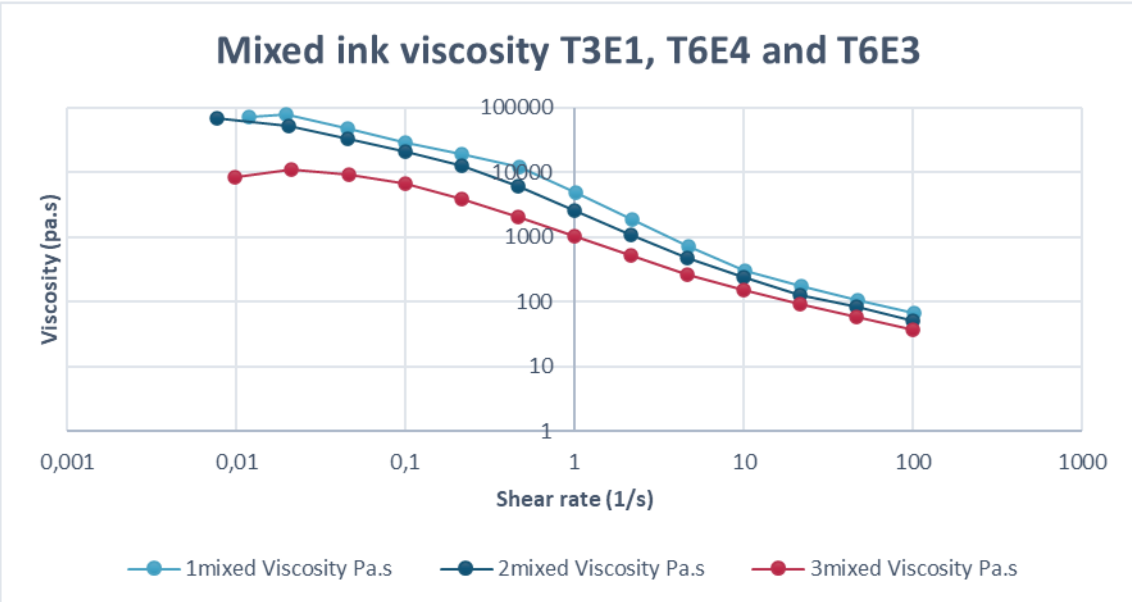
Zjenja Doubrovski
Houman Yarmand
11 and 14-06-2021

Introduction

In 3D printer: try 1 the dual syringe printer did not have enough force to extrude the ink through the tubes to the static mixing nozzle. Due to the limited time of this project, we will adjust 3 parameters at once today. This way we can decide if we could continue forward with this printer or should switch to another one, after which the electromagnet development can be started.

The parameters that will be changed today are the inks viscosity, the syringe size and the extrusion force of the 3D printer.

The ink viscosity will be lowered so that less force is needed to extrude the ink through the system. To lower the viscosity, we will change from the T6E4 to the T6E3 ink. T6E3 only has 66% of the amount of fumed silica that the T6E4 recipe has. This difference in fumed silica has a big difference in the starting viscosity of the inks (see the table below 2mixed is T6E4 and 3mixed is T6E3).



The 60 mL syringes that fit in the dual syringe extruder part of the 3D printer will be switched for 10 mL syringes (see figure 1). This will be done because the 10 mL syringes have a smaller diameter, which means a smaller surface that has to press to the ink. According to the formula:

$$P = \frac{F}{A}$$

P = pressure (N/m²)

F =force (N)

A = surface (m²)

When a smaller surface is used the same force can deliver a higher pressure in the system.

Figure 1 White 3D printed insertion pieces to convert the 60 mL syringe place to a 10 mL syringe

The last thing that will be altered at the 3D printer is the software of the dual syringe extruder part. The firmware will be changed so that it enables the motors to exert a higher force on the system.

By the change of these three parameters, we will test if the 3D printer now has enough force to extrude the ink from the syringes through the tubes to and through the static mixing nozzle. I this is not the case after this test, we will switch to a single syringe paste printer.

Materials

Utensils

- 2 10 ml syringes
- Scale
- Cups
- Mixing spatulas
- Tape
- Pen
- Double syringe 3D printer
- Clear rubber tubes (from the syringe to static mixing nozzle)
- The adjusted static mixing nozzle v1 of which the tube attachment nozzles have a tapered shape so that the tube could go on better
- 2x syringe mounting piece (convert 60 ml syringe place to a 10 ml syringe)
- 2x syringe piston enlarging piece (convert the 10 ml syringe piston end to one that will fit at the 60 ml syringe spot)

Experiment 1: two component T6E3

- 12,6 g Ecoflex 00-10 part B

- 2 x 22,5 g Iron powder (carbonyl iron: 12)
- 12,6 g Ecoflex 00-10 part A
- 2x 0,24 g fumed silica

Method

Prepare the ink

- Ecoflex part A and Ecoflex part B should be poured into different cups
- Measure the half amount of the iron carbonyl particles and add this to the cup of Ecoflex part A
- Measure the other half of the iron carbonyl particles and add these to a cup of Ecoflex part B
- Mix the Ecoflex part A and the iron carbonyl particles manually
- Mix the Ecoflex part B and the iron carbonyl particles manually
- Measure the half amount of the fumed silica and add this to the cup of Ecoflex part A
- Measure the other half of the fumed silica and add this to the cup of Ecoflex part B
- Mix the contents in the cup of Ecoflex part A manually
- Mix the contents in the cup of Ecoflex part B manually

At the end of this step, there should be two cups with a mixture of Ecoflex part A or B with half of the amount of the fumed silica and the iron carbonyl particles.

Prepare the syringes

- Load one 10 ml syringe with the Ecoflex A mixture
- Load another 10 ml syringe with the Ecoflex part B mixture
- Level the two syringes so that they have the same amount of ink in them (this is not necessary because the pistons are individually powered)

Prepare the 3D printer

- Attach the tubes to the syringes
- Attach the syringes to the static mixing nozzle
- (Because the design is not optimized and the syringes do not have a Luer lock system, it is helpful to secure the tubes extra with the help of zip ties)
- Insert the 60 to 10 ml conversion pieces into the printer where the syringes will normally be installed
- Then the syringes should be put in the conversion pieces
- The mechanical parts that push the pistons of the syringes forward should be brought up
- The syringe piston enlarging pieces should be put over the end of the 10 ml syringe pistons
- At last, the red clips should be attached over the syringe piston enlarging pieces

Results

The ink got extruded through the entire system (see figure 2). However, sometimes one of the motors stopped turning and started ticking. After a while, the ticking motor started turning again.

This ticking of the motors happened with both motors at different times in the extrusion process. For one single motor related to a syringe, I think the motor stopped turning around 3 to 4 times along the extrusion of the ink, from the tip of the syringe until the end of the static mixing nozzle.

Finally, when the ink of both syringes had reached the static mixing nozzle through the tubes, with a constant turning the ink got extruded through the static mixing nozzle.

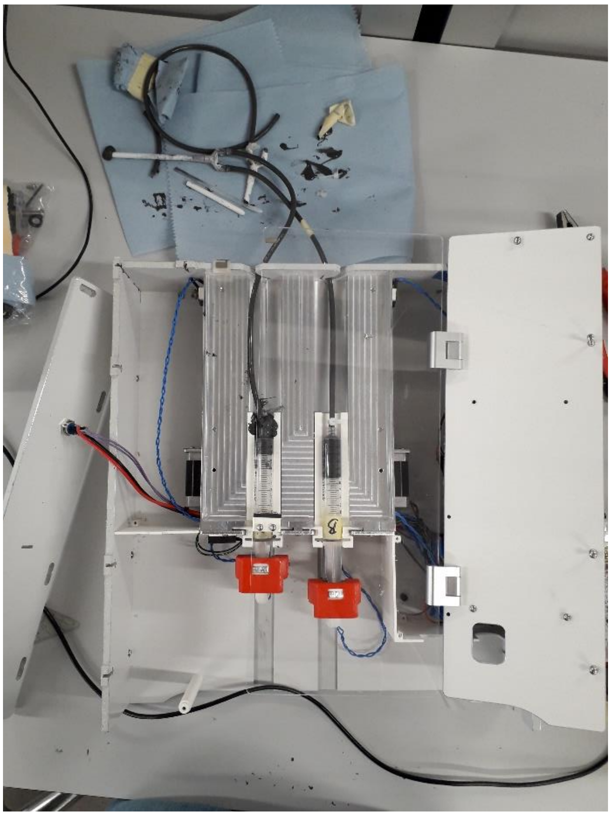


Figure 2 Extruded ink through the system and the static mixing nozzle

Conclusion

Even though the ink got extruded through the entire system, the system is not reliable enough because of the motors.

The motors started turning very smoothly but one after the other stopped turning, but after some undetermined time started turning again very smoothly.

Because of this the ink curing quality and the even extrusion of the ink can not be guaranteed. An uneven extrusion of the syringes makes the mixing ratio between the A and B component of the ink differ from the 1:1 ratio. This can make certain parts of the ink not cure good enough, if even not cure at all.

Also, when only one syringe is extruding instead of two, the amount of ink that is being extruded through the static mixing nozzle is not constant, because not the same volumes are moving through the system. This could finial cause extruded rods of varying thicknesses which will result in a bad printing quality.

Further research

Due to the time limits of this project, we might want to consider using another 3D printer. Even though the solution of the dual syringe extruder is very nice, to maintain a constant ink quality with the same properties (if everything works well), it would take too much time to adjust and/or optimize the 3D printer to work reliably.
Because of this as a next step, I think it is wise to look at a single syringe 3D printer. On the one side, it is unfortunate that we can not have a constant condition of the ink, because the mixed ink in a single syringe printer will start curing during the process. On the other hand, the ink has a smaller distance in the system before it can be printed. This is because there are no tubes and static mixing nozzles involved. This will probably lower the extrusion force needed, as well as lower the amount of waste created in the process and ink needed.

3D printer: try 3

Sanne van Vilsteren
Zjenja Doubrovski
16-06-2021

Introduction

This test was not planned and rather spontaneous. The force of the 3D printer was measured before this test and increased. From static mixing nozzle test 4, we knew that 19,069 kg was needed for extruding the T6E3 through the static mixing nozzle. This is around 19,069/2*10 = 95,345 N of force needed per syringe, excluding the additional force through the resistance in the tubes. The force measured of the updated firmware of the second test was only 80 N, which is not enough to get the ink extruded through the system. This day the motor power was increased to a measured value of 180N, which is more promising to work due to its force exceeding 95 N.

Due to the spontaneous nature of this test, there was no new ink prepared and remainders of the second 3D printer test were used. There was also no static mixing nozzle near to be used, so the ink was only extruded through the tubes. In the previous tests, the motors already started to skip steps in the tubes, so the purpose of this test was to see if the motors kept on turning while extruding and to decide if one more 3D printer test could be done with the dual syringe printer.

Materials

- The remaining unmixed ink components of the T6E3 ink used in 3D printer test 2
- Two clear rubber tubes (to attach to the syringes)
- 2 10 ml syringes
- 2 zip ties
- 2x syringe mounting piece (convert 60 ml syringe place to a 10 ml syringe)
- 2x syringe piston enlarging piece (convert the 10 ml syringe piston end to one that will fit at the 60 ml syringe spot)

Method

Prepare the syringes

- Load one 10 ml syringe with the Ecoflex A mixture
- Load another 10 ml syringe with the Ecoflex part B mixture

Prepare the 3D printer

- Attach the tubes to the syringes
- (It is helpful to secure the tubes extra with the help of zip ties because we do not have Luer-lock tube to syringe connection pieces yet)
- Insert the 60 to 10 ml conversion pieces into the printer where the syringes will normally be installed
- Then the syringes should be put in the conversion pieces
- The mechanical parts that push the pistons of the syringes forward should be brought up
- The syringe piston enlarging pieces should be put over the end of the 10 ml syringe pistons
- At last, the red clips should be attached over the syringe piston enlarging pieces

Printer settings

The extrusion speed that was used, was the command F2.

Results

The ink got extruded smoothly through the tubes without motors that skipped steps.

Conclusion

The ink got extruded through the tubes without stopping motors. The motors turned very smoothly the entire test without skipping steps. This makes it hopeful to use this printer again for one more test to see if it can also extrude the ink through the static mixing nozzle without motors skipping steps.

Further research

For the next test the second 3D printer test will be repeated, but then with the new firmware that has a force of 180 N per syringe. If the motors run do not skip steps during that test, the project will be continued with the dual syringe printer.

3D printer: try 4

Sanne van Vilsteren
Houman Yarmand
Sepideh Ghodrat
18-06-2021

Introduction

In this test, for the last time, there will be tested if the dual syringe printer can extrude the T6E3 ink through the tubes and the static mixing nozzle. Last test the force of the printer was increased from 80 N to 180 N. This makes a better change to extrude the T6E3 ink, for which at least 95 N was needed with the exclusion of the tubes.

Materials

Utensils

- 2 10 ml syringes
- Scale
- Cups
- Mixing spatulas
- Tape
- Pen
- Double syringe 3D printer
- Clear rubber tubes (from the syringe to static mixing nozzle)
- The adjusted static mixing nozzle v1 of which the tube attachment nozzles have a tapered shape so that the tube could go on better
- 2x syringe mounting piece (convert 60 ml syringe place to a 10 ml syringe)
- 2x syringe piston enlarging piece (convert the 10 ml syringe piston end to one that will fit at the 60 ml syringe spot)

Experiment 1: two component T6E3

- 12,6 g Ecoflex 00-10 part B
- 2 x 22,5 g Iron powder (carbonyl iron: 12)
- 12,6 g Ecoflex 00-10 part A
- 2x 0,24 g fumed silica

Method

Prepare the ink

- Ecoflex part A and Ecoflex part B should be poured into different cups
- Measure the half amount of the iron carbonyl particles and add this to the cup of Ecoflex part A
- Measure the other half of the iron carbonyl particles and add these to a cup of Ecoflex part B
- Mix the Ecoflex part A and the iron carbonyl particles manually
- Mix the Ecoflex part B and the iron carbonyl particles manually
- Measure the half amount of the fumed silica and add this to the cup of Ecoflex part A
- Measure the other half of the fumed silica and add this to the cup of Ecoflex part B
- Mix the contents in the cup of Ecoflex part A manually

- Mix the contents in the cup of Ecoflex part B manually

At the end of this step, there should be two cups with a mixture of Ecoflex part A or B with half of the amount of the fumed silica and the iron carbonyl particles.

Prepare the syringes

- Load one 10 ml syringe with the Ecoflex A mixture
- Load another 10 ml syringe with the Ecoflex part B mixture

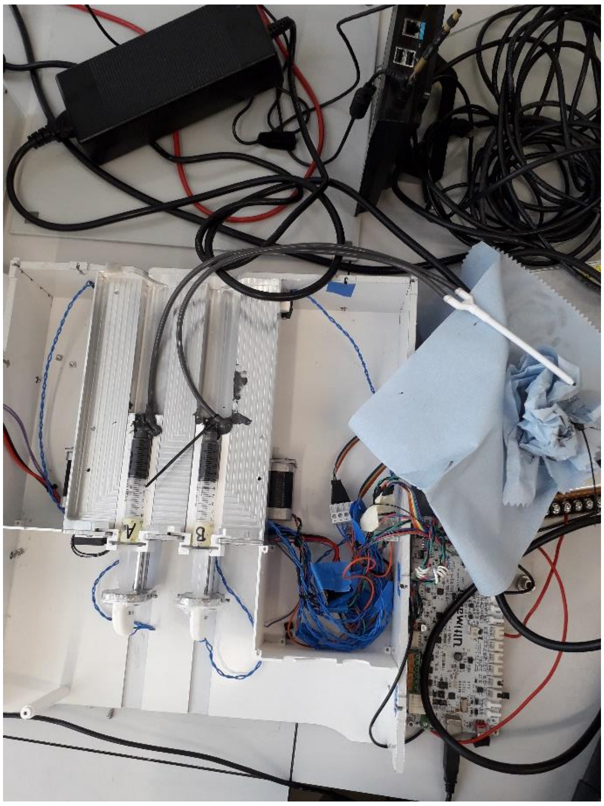
Prepare the 3D printer

- Attach the tubes to the syringes
- Attach the syringes to the static mixing nozzle
- (It is helpful to secure the tubes extra with the help of zip ties because we do not have Luer-lock tube to syringe connection pieces yet)
- Insert the 60 to 10 ml conversion pieces into the printer where the syringes will normally be installed
- Then the syringes should be put in the conversion pieces
- The mechanical parts that push the pistons of the syringes forward should be brought up
- The syringe piston enlarging pieces should be put over the end of the 10 ml syringe pistons
- At last, the red clips should be attached over the syringe piston enlarging pieces

Printer settings

The extrusion speed that was used, was the command F2.

Results



The ink got extruded through both the tubes and the static mixing nozzle, without the motors skipping steps. They ran smoothly the entire time.

In the pictures can be seen that the ink got extruded through the static mixing nozzle, but also that the tubes popped off of the syringes. This is because they are not fixed well yet and only slipped on.

Conclusion

The dual syringe printer has enough force to extrude the T6E3 ink. This means that this printer can be used in this project to print with magnetic soft material.

Further research

The next steps in the research will be to see how the printer prints with the ink and tune the printer settings. Also, the ink performances, like print stability, should be analysed to maybe optimize the ink.

3D printer: try 5

Sanne van Vilsteren
Houman Yarmand
29-06-2021

Introduction

The purpose of this test is to see if an ink with unmagnetized NdFeB particles behaves in the same way as the ink with unmagnetized iron carbonyl particles and to know if there are any differences. For this test, the T6E3 ink will be used, which was successfully extruded in the previous 3D printer test. The alterations made for this ink will be that the iron carbonyl particles will be changed for NdFeB particles by their vol%. In this test the extrusion force through the static mixing nozzle and the ink's viscosity will also be measured, to see if there are any differences between the iron carbonyl and the NdFeB based ink.

Materials

Utensils

- 2 10 ml syringes
- 2 scales
    - One with three decimals to make the ink
    - One with a range of up to 24 kg to measure the force needed to extrude the ink through the syringe
- Cups
- Mixing spatulas
- Tape
- Pen
- Double syringe 3D printer
- Clear rubber tubes (from the syringe to static mixing nozzle)
- 2x the adjusted static mixing nozzle v1 of which the tube attachment nozzles have a tapered shape so that the tube could go on better
- 2x syringe mounting piece (convert 60 ml syringe place to a 10 ml syringe)
- 2x syringe piston enlarging piece (convert the 10 ml syringe piston end to one that will fit at the 60 ml syringe spot)
- 2x female Luer lock 4,8 mm connector from the syringe to the tube

Experiment 1: two component T6E3 (iron carbonyl powder changed for NdFeB particles)

Density Iron carbonyl powder: 7,86 g/mL (Merck, n.d.)
Density NdFeB: 7,6 g/cm^3 (magnets, 2018)
22,5/7,89*7,6 = 21,7 g

- 12,6 g Ecoflex 00-10 part B
- 2 x 21,7 g NdFeB particles
- 12,6 g Ecoflex 00-10 part A
- 2x 0,24 g fumed silica

Method

Prepare the ink

- Ecoflex part A and Ecoflex part B should be poured into different cups
- Measure the half amount of the NdFeB particles and add this to the cup of Ecoflex part A
- Measure the other half of the NdFeB particles and add these to a cup of Ecoflex part B
- Mix the Ecoflex part A and the iron carbonyl particles manually
- Mix the Ecoflex part B and the iron carbonyl particles manually
- Measure the half amount of the fumed silica and add this to the cup of Ecoflex part A
- Measure the other half of the fumed silica and add this to the cup of Ecoflex part B
- Mix the contents in the cup of Ecoflex part A manually
- Mix the contents in the cup of Ecoflex part B manually

At the end of this step, there should be two cups with a mixture of Ecoflex part A or B with half of the amount of the fumed silica and the NdFeB particles.

Prepare the syringes

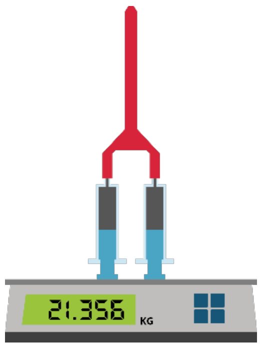
- Load one 10 ml syringe with the Ecoflex A mixture
- Load another 10 ml syringe with the Ecoflex part B mixture

Prepare for extrusion

- Level the two syringes so that they have the same amount of ink in them. This is important so that you can be sure that both syringes have loaded the nozzle with the same amount of ink. That is important because the Ecoflex only becomes a silicone if the two are mixed. From earlier research, it was already found out that the mixing ratio of 1:1 (which is also the instruction on the Ecoflex bottles itself) work the best.
- Mount both of the syringes at the top of the static mixing nozzle

Measure extrusion force

- **Measure: In previous experiments, it took too much force to extrude the ink by hand and we had to press in through the static mixing nozzle by pushing the pistons of the syringes on a table. To measure the force needed to extrude the ink, the pistons of the syringes should be pressed against a scale when extruding the ink to see the maximum force needed (see the figure on the right).**



Measure viscosity

The viscosity of the ink should be measured in three conditions. Those are:

- Ink component A separately
- Ink component B separately
- The ink after it is just mixed

This way we can get an insight into how the viscosity changes over the printing project.

The measuring procedure:

- 40-mm-diameter steel plate geometry.
- Steady-state flow experiments with a sweep of shear rates (0,0-100 s^{-1})
- The inks were brought to a temperature of 25°C for one minute before the experiment started.

- The gap height between the geometry and the plate always was 0,5 mm.

Prepare the 3D printer

- Attach the tubes to the syringes with the use of the female Luer lock connectors
- Attach the tubes to a new static mixing nozzle
- Insert the 60 to 10 ml conversion pieces into the printer where the syringes will normally be installed
- Then the syringes should be put in the conversion pieces
- The mechanical parts that push the pistons of the syringes forward should be brought up
- The syringe piston enlarging pieces should be put over the end of the 10 ml syringe pistons
- At last, the red clips should be attached over the syringe piston enlarging pieces

Printer settings

The extrusion speed that was used, was the command F2.

Results

Maximum extrusion force

21,730 Kg

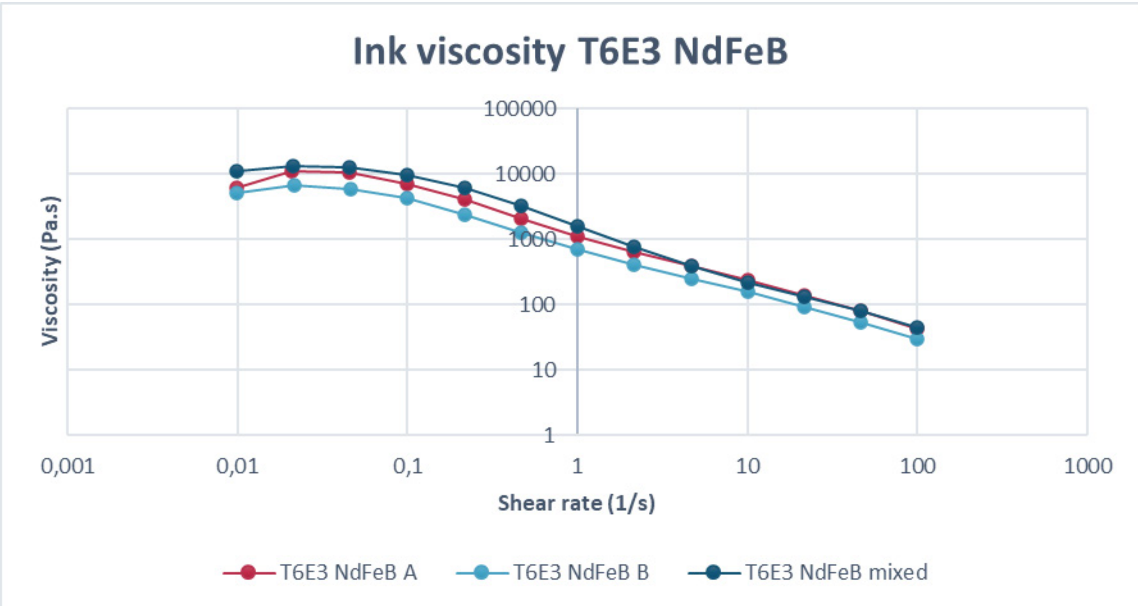
The maximum measured extrusion force of the T6E3 NdFeB ink was 21,730 Kg. This equals 21,73/2*10 = 106,85 N per syringe.

This value lays in between the extrusion Kg measured of the T6E4 (22,566 Kg) and the T6E3 (19,069 Kg) that had iron carbonyl particles.

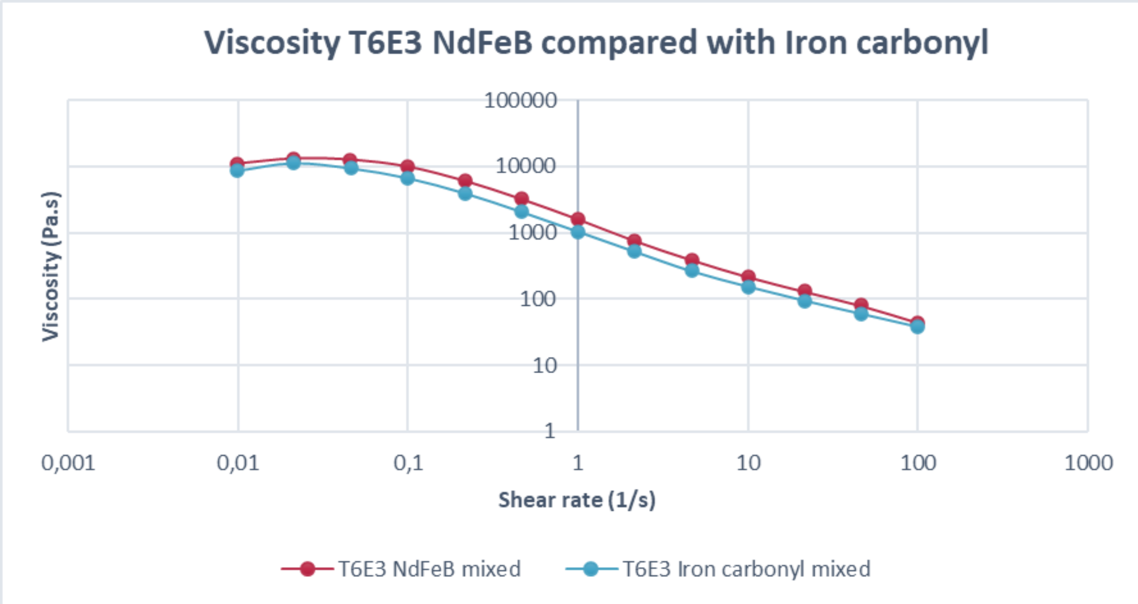
Ink curing after extrusion through the static mixing nozzle

The T6E3 NdFeB ink was cured after it was extruded and mixed through the static mixing nozzle.

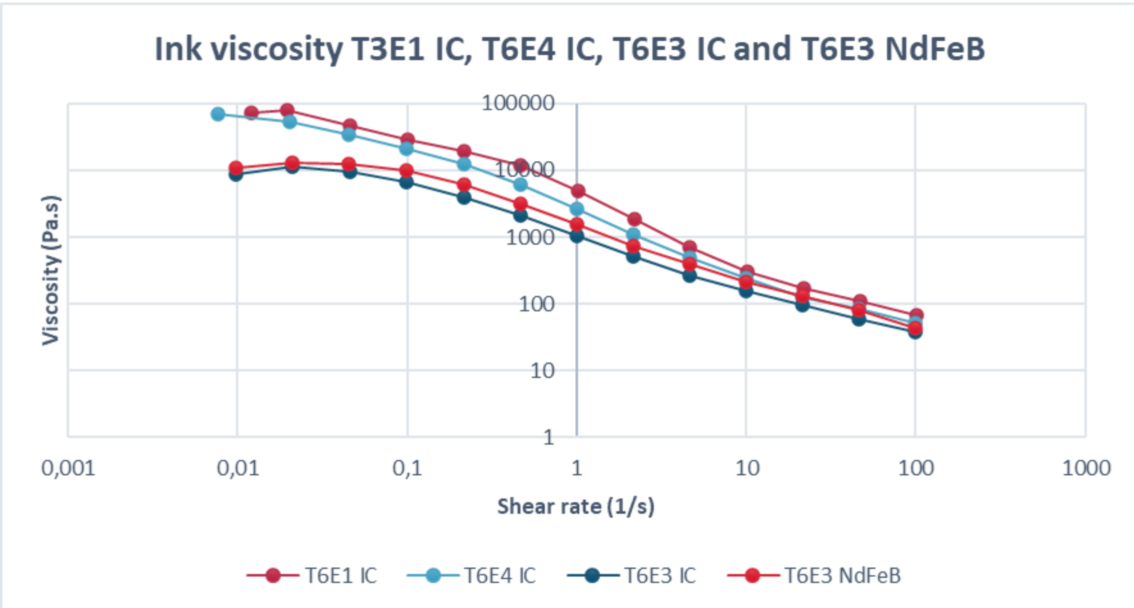
Viscosity measurements



The individual and mixed ink components of the T6E3 NdFeB ink follow the same order for their viscosity in comparison with the earlier measured inks in Static mixing nozzle test 4. Where the mixed viscosity is the highest, and ink component A viscosity is above the viscosity of ink component B.



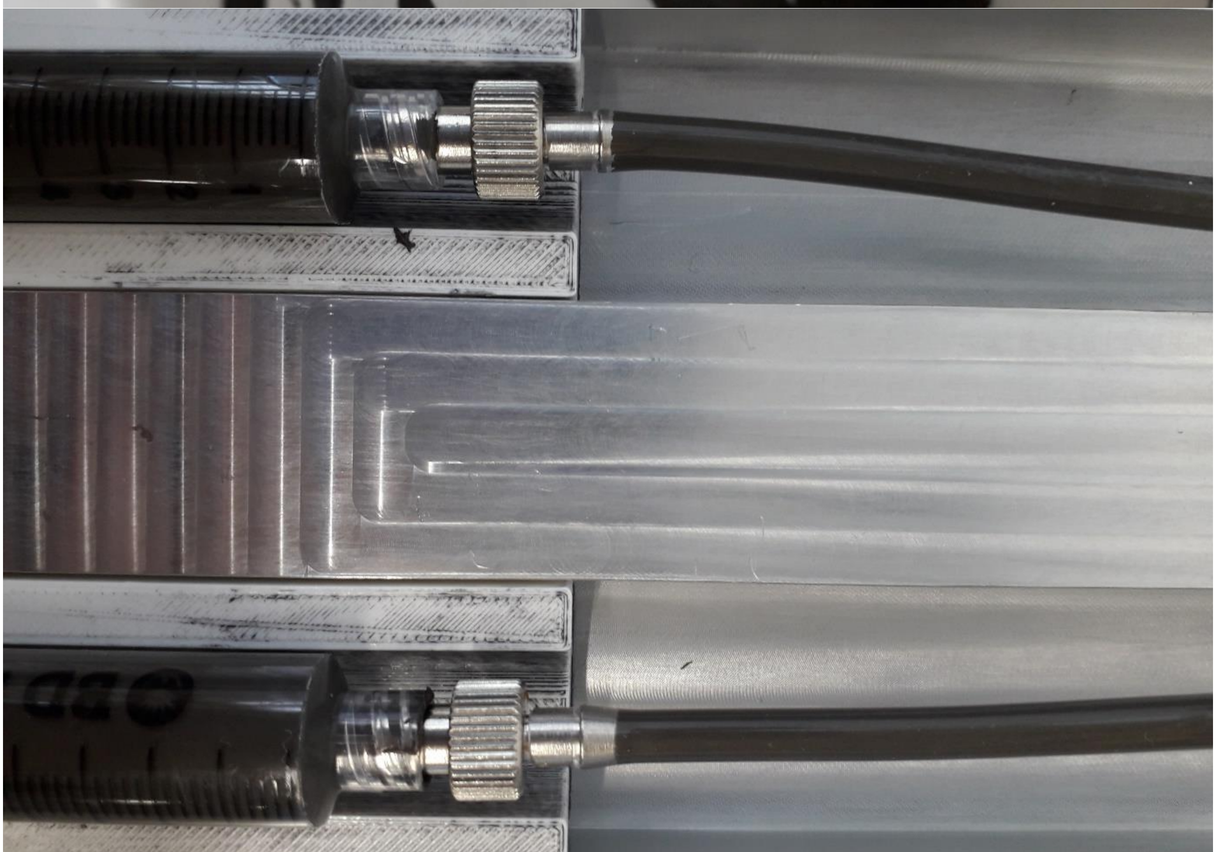
In this graph the ink viscosity of the original T6E3 with iron carbonyl particles with compared with the altered T6E3 with NdFeB particles. What can be seen is that the viscosity of the NdFeB based T6E3 is a bit higher. This can be because of the particles, but also because of conversion of the vol% of the particles, because these have been calculated with values found on the internet, instead of being specific to the particles that were used in this experiment.



When the NdFeB based T6E3 is placed in the graph next to the viscosities of the other mixed inks measured in Static mixing nozzle test number 4, there can be seen that it does fall in the same order of magnitude of the T6E3 iron carbonyl based.

Extrusion with the 3D printer

The NdFeB based T6E3 was successfully extruded through the syringes, hoses and the static mixing nozzle of the printer. The motors kept running smoothly. The ink that came out of the static mixing nozzle cured. The print stability however was not very good (unfortunately there was no good before and after picture taken.



After the extrusion of the syringes stopped, the ink kept coming out of the static mixing nozzle. This is probably due to build up pressure in the system. The hoses connecting the syringes with the static mixing nozzle are very flexible and have expanded as can be seen in the figure above. The upper syringe is filled with ink component A (the more viscous one) and this can explain why that tube has expanded more than the one coming from ink component B.

The diameters of the hoses were measured near the nozzles and near the static mixing nozzle:

Diameter of hose A	Near the syringe:	5,6 mm
	Near the static mixing nozzle	5,25 mm
Diameter of hose B	Near the syringe	5,25 mm
	Near the static mixing nozzle	5 mm

Conclusion

The NdFeB based T6E3 ink kind of behaved similar to the Iron Carbonyl based T6E3 ink. It was able to be extruded through the static mixing nozzle manually and with the 3D printer. In both cases the mixed ink cured.

Where the two inks differed from each other was the extrusion force and viscosity that are related to each other. The NdFeB based ink had a higher viscosity and extrusion force than the Iron Carbonyl based ink. However, the viscosity was in the same range as the Iron Carbonyl based T6E3 ink when it was compared to the T6E1 and T6E4. This difference can be due to not using the correct material densities when substituting the particles. The densities have not been measured and the product-specific densities could not be found.

Further research

For further research, due to the still weak ink viscosity and knowing that the printer does work right now, it should be tested if the T6E4 ink (one step higher in viscosity than the T6E3) could be extruded with the printer. This was also the purpose of 3D printer try 1, but due to a too low force, this was not possible then. If that ink can be extruded, it is a good base to base the viscosity and the in for the final magnetization on, because of its good print stability.

What should also be found out is at which values in the G-code the 3D printer extrudes 1 mL. Also, other printer settings should be found out like printing speed and extrusion per distance, to tune the printer for working with it.

References

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Merck. (n.d.). Iron 44890. Retrieved from https://www.sigmaaldrich.com/NL/en/product/aldrich/44890

3D printer: try 6

Sanne van Vilsteren
08-07-2021

Introduction

The purpose of this test is to see if the 3D printer system can extrude the T6E4 ink. This ink was made for the first time in ink development try 6 and had a good print stability. It could also be extruded through the static mixing nozzle in Static mixing nozzle test 4. In the first 3D printer test it was also the purpose to see if this ink could be extruded through the printer. Only due to less force of the printer at that time and the larger syringes it the printer was too weak to extrude the ink. In this test, it will be tried again to extrude the T6E4 through the static mixing nozzle with help of the 3D printer.

For this test, no new ink will be made. There is still T6E4 ink remaining from 3D printer: try 1, which will be reused for this experiment.

Materials

Utensils

- 2 10 ml syringes
- Cup
- Tape
- Pen
- Remaining T6E4 ink from 3D printer try 1
- Double syringe 3D printer
- Clear rubber tubes (from the syringe to static mixing nozzle)
- 2x the adjusted static mixing nozzle v1 of which the tube attachment nozzles have a tapered shape so that the tube could go on better
- 2x syringe mounting piece (convert 60 ml syringe place to a 10 ml syringe)
- 2x syringe piston enlarging piece (convert the 10 ml syringe piston end to one that will fit at the 60 ml syringe spot)
- 2x female Luer lock 4,8 mm connector from the syringe to the tube

Recipe T6E4

- 12,6 g Ecoflex 00-10 part B
- 2 x 22,5 g Iron powder (carbonyl iron: 12)
- 12,6 g Ecoflex 00-10 part A
- 2x 0,36 g fumed silica

Method

Prepare the syringes

- Load one 10 ml syringe with the Ecoflex A mixture
- Load another 10 ml syringe with the Ecoflex part B mixture

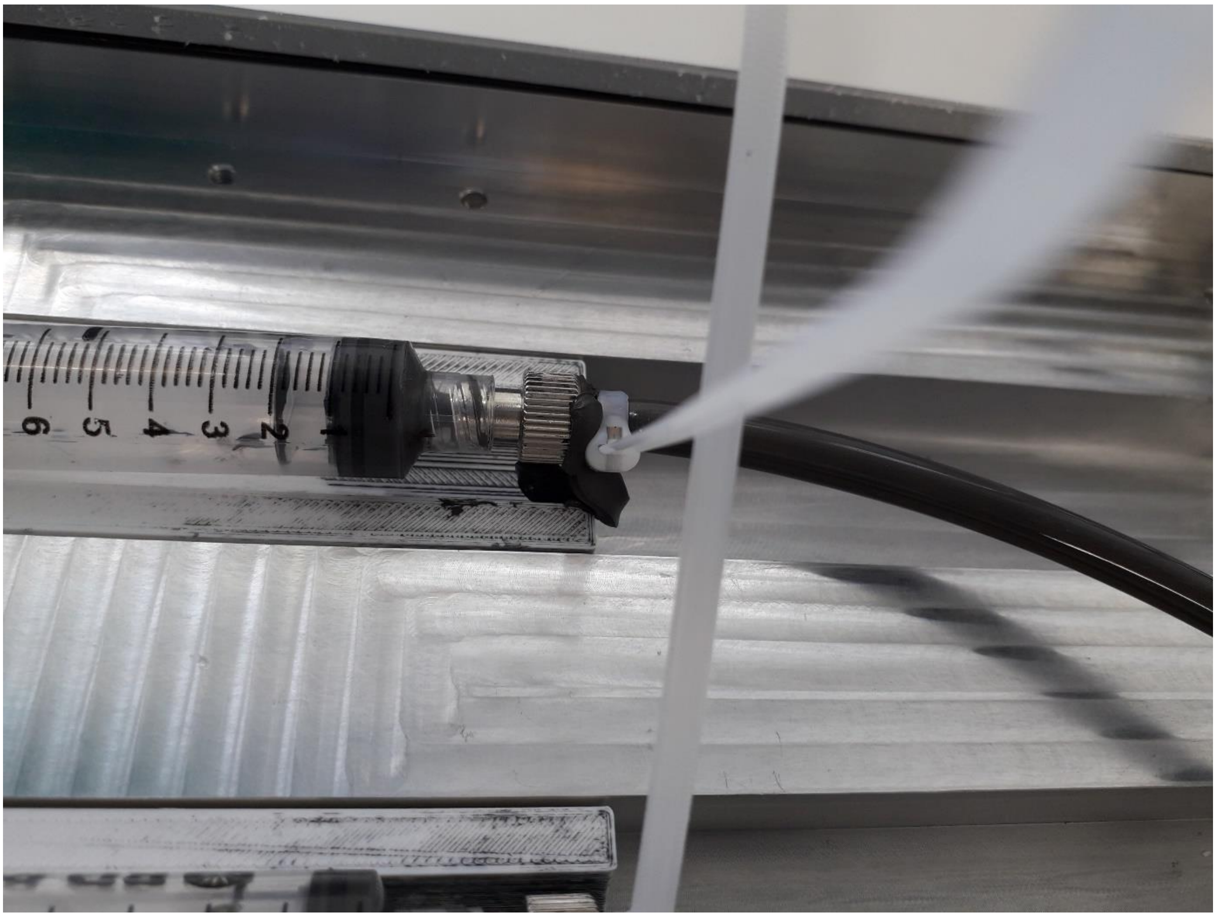
Prepare the 3D printer

- Attach the tubes to the syringes with the use of the female Luer lock connectors
- Attach the tubes to a new static mixing nozzle
- Insert the 60 to 10 ml conversion pieces into the printer where the syringes will normally be installed
- Then the syringes should be put in the conversion pieces (see the figure on the right)
- The mechanical parts that push the pistons of the syringes forward should be brought up
- The syringe piston enlarging pieces should be put over the end of the 10 ml syringe pistons
- At last, the red clips should be attached over the syringe piston enlarging pieces

Printer settings

The extrusion speed that was used, was the command F2.

Results



Motors:
During the entire experiment, the motors kept running smoothly, so the printer was no limiting factor anymore during this test.

Tubes:
However, the problem during this extrusion lay in the tubes in combination with the viscosity of the ink components. As can be seen in the upper left picture, the tube on the left side (ink component A) has quite expanded in comparison with the right tube of ink component B. Due to this, the volume of the ink component A was kept at the beginning of the tube. That caused ink component A to have a delay at coming to the static mixing nozzle (upper right picture). Also by finding a way with less resistance, part A found a way between the tube and the zip ties to escape the tube on the other side (bottom picture). In the meantime, because B was also extruded further and had already passed through the static mixing nozzle, the ink found another way of least resistance and went the way where part A of the ink should go through the static mixing nozzle (upper right picture).

The elasticity is too much and this is a problem for the system. Because ink component A has a delay it will probably not be mixed 1:1 with ink component B which will result in an uncontrollable ink quality.

Also is ink component A probably too viscous, because it did not reach the static mixing nozzle.

Conclusion

The printer works fine, but the ink component A is too viscous and the tubes are too elastic. The T6E4 can not be used in the current printer system.

Further research

In the introduction, the choice to try T6E4 was explained, mainly due to its print stability. However, because this ink can not be used the print stability of the ink is not solved. That is why in the next ink development I want to make an ink between T6E4 and T6E3 with the amount of the fumed silica to get the viscosity between the two inks. I hope that that will make the ink less viscous to be able to go through the system, but also a bit more viscous to increase the print stability.

It is also a good idea to search for stiffer tubes, like for example steel enhanced tubes. That way the tubes will not expand that much and the build-up volume problem might be solved.

Another thing not entirely mentioned in the results is that part B had already reached the static mixing nozzle before part A. This was because I extruded the part A and B at the same rate with the syringes. I think that in the future it could be good to extrude parts A and B separately up to the static mixing nozzle. After that, they could be extruded at the same time to also control the mixing in the static mixing nozzle better.

3D printer: try 7

Sanne van Vilsteren
Houman Yarmand
13-07-2021

Introduction

The conclusion of 3D printer: try 6 was that the T6E4 was too viscous to be extruded by the 3D printer system, but T6E3 was also not ideal to use as ink due to its low print stability. This will be the final test in this project with a new type of ink to try if the ink can be extruded through the system, while also having a good print stability. For that, the average amount of fumed silica ink will be taken from the T6E3 and the T6E4 to create a new ink T7E1. Depending on the outcome of this test it will be decided what inks will be made for the magnetizer. Depending on this test will also almost for sure be decided if for this graduation project it will be possible to print 3D structures or not, depending on this inks print stability and ability to be extruded. There is no other time left in this project to further optimize the ink.

Materials

Utensils

- 2 10 ml syringes
- 2 scales
  - One with three decimals to make the ink
  - One with a range of up to 24 kg to measure the force needed to extrude the ink through the syringe
- Cups
- Mixing spatulas
- Tape
- Pen
- Double syringe 3D printer
- Clear rubber tubes (from the syringe to static mixing nozzle)
- 2x the adjusted static mixing nozzle v1 of which the tube attachment nozzles have a tapered shape so that the tube could go on better
- 2x syringe mounting piece (convert 60 ml syringe place to a 10 ml syringe)
- 2x syringe piston enlarging piece (convert the 10 ml syringe piston end to one that will fit at the 60 ml syringe spot)
- 2x female Luer lock 4,8 mm connector from the syringe to the tube

Experiment 1: two-component T7E1

T6E3 had 0,24 g fumed silica.
T6E4 had 0,36 g fumed silica.
So, this new ink T7E1 will have 0,30 g fumed silica

- 12,6 g Ecoflex 00-10 part B
- 2 x 22,5 g Iron powder (carbonyl iron: 12)
- 12,6 g Ecoflex 00-10 part A
- 2x 0,30 g fumed silica

20% increase:

- 15,1 g Ecoflex 00-10 part B
- 2 x 27 g Iron powder (carbonyl iron: 12)
- 15,1 g Ecoflex 00-10 part A
- 2x 0,36 g fumed silica

Method

Prepare the ink

- Ecoflex part A and Ecoflex part B should be poured into different cups
- Measure the half amount of the NdFeB particles and add this to the cup of Ecoflex part A
- Measure the other half of the NdFeB particles and add these to a cup of Ecoflex part B
- Mix the Ecoflex part A and the iron carbonyl particles manually
- Mix the Ecoflex part B and the iron carbonyl particles manually
- Measure the half amount of the fumed silica and add this to the cup of Ecoflex part A
- Measure the other half of the fumed silica and add this to the cup of Ecoflex part B
- Mix the contents in the cup of Ecoflex part A manually
- Mix the contents in the cup of Ecoflex part B manually

At the end of this step, there should be two cups with a mixture of Ecoflex part A or B with half of the amount of the fumed silica and the NdFeB particles.

Prepare the syringes

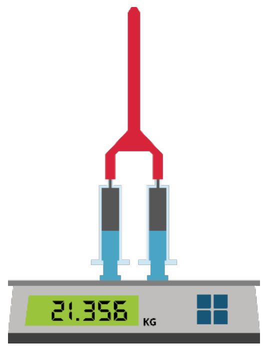
- Load one 10 ml syringe with the Ecoflex A mixture
- Load another 10 ml syringe with the Ecoflex part B mixture

Prepare for extrusion

- <u>Level the two syringes so that they have the same amount of ink in them. This is important so that you can be sure that both syringes have loaded the nozzle with the same amount of ink. That is important because the Ecoflex only becomes a silicone if the two are mixed. From earlier research, it was already found out that the mixing ratio of 1:1 (which is also the instruction on the Ecoflex bottles itself) work the best.</u>
- Mount both of the syringes at the top of the static mixing nozzle

Measure extrusion force

- **Measure: In previous experiments, it took too much force to extrude the ink by hand and we had to press in through the static mixing nozzle by pushing the pistons of the syringes on a table. To measure the force needed to extrude the ink, the pistons of the syringes should be pressed against a scale when extruding the ink to see the maximum force needed (see the figure on the right).**



Measure viscosity

The viscosity of the ink should be measured in three conditions. Those are:

- Ink component A separately
- Ink component B separately
- The ink after it is just mixed

This way we can get an insight into how the viscosity changes over the printing project.

The measuring procedure:

- 40-mm-diameter steel plate geometry.
- Steady-state flow experiments with a sweep of shear rates (0,0-100 s^{-1})
- The inks were brought to a temperature of 25°C for one minute before the experiment started.
- The gap height between the geometry and the plate always was 0,5 mm.

Prepare the 3D printer

- Attach the tubes to the syringes with the use of the female Luer lock connectors
- Attach the tubes to a new static mixing nozzle
- Insert the 60 to 10 ml conversion pieces into the printer where the syringes will normally be installed
- Then the syringes should be put in the conversion pieces
- The mechanical parts that push the pistons of the syringes forward should be brought up
- The syringe piston enlarging pieces should be put over the end of the 10 ml syringe pistons
- At last, the red clips should be attached over the syringe piston enlarging pieces

Printer settings

The extrusion speed that was used, was the command F2. This was later changed to F1 when part A extruded slower. This time thicker, but still elastic tubes were used to hopefully prevent the expansion of the tube. Also, the inks got extruded individually up to the static mixing nozzle until they were extruded together.

G-code settings that were used (some multiple times to get ink at the end of a tube):

- F2 A178 (2ml) B10 (not that much)
- F1 A178 B10 (maybe a slower speed will keep up the delay of the ink at the end of its extrusion)
- F1 A20 B178 (to get B at the same level as A)
- F1 A45 B1 (because of the big delay of part A)

Results

Maximum extrusion force

23,396 Kg

The maximum measured extrusion force of the T7E1 ink was 23,396 Kg. This equals 23,396/2*10 = 116,96 N per syringe.

This value lays in between the extrusion KG measured of the T6E1 (above the 24 Kg) and the T6E4 (19,069 Kg) which is strange because the only different thing is the amount of added fumed silica. The amount of fumed silica added to the T7E1 lays below the T6E1 and the T6E4 so according to earlier viscosity and force measurements, this value should be below the ones of the T6E1 and the T6E4.

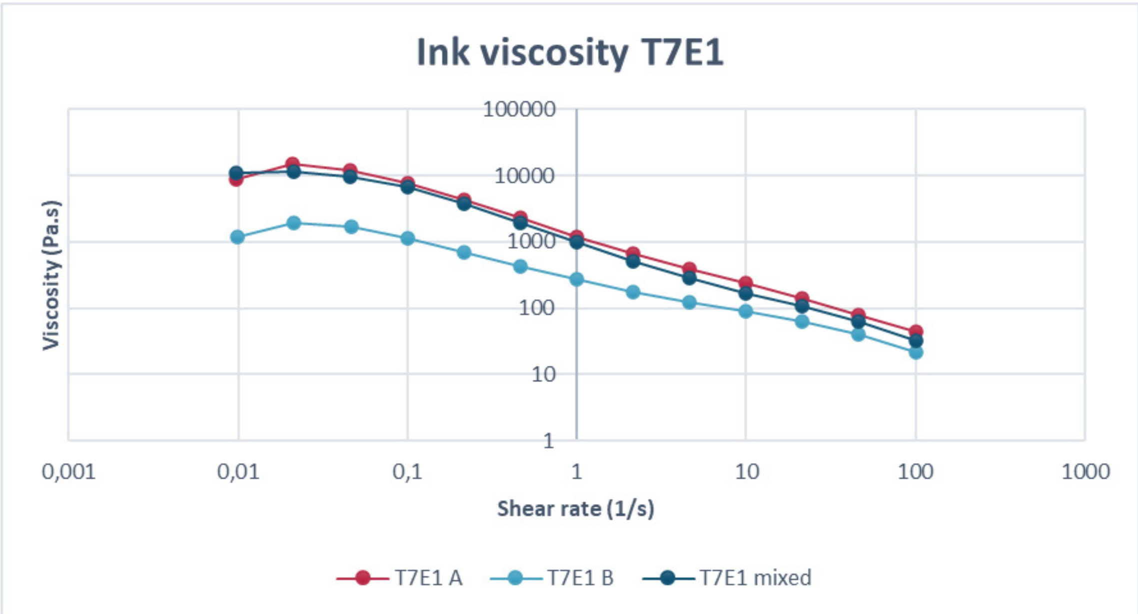
What was different with this ink though was (in comparison with the T6E1 and T6E4), that Houman (who extruded all the inks through the static mixing nozzles) found this one easy to extrude and was surprised when the ink was already extruded through the static mixing nozzle.

The high extrusion force could be a cause of the lesser surface quality of the static mixing nozzle.

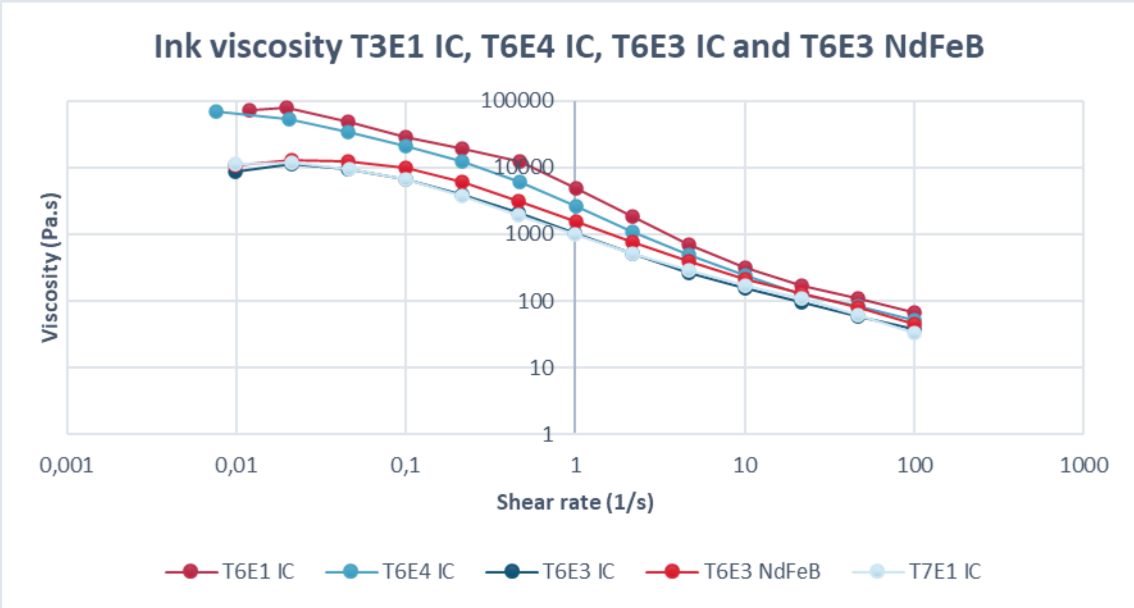
Ink curing after extrusion through the static mixing nozzle

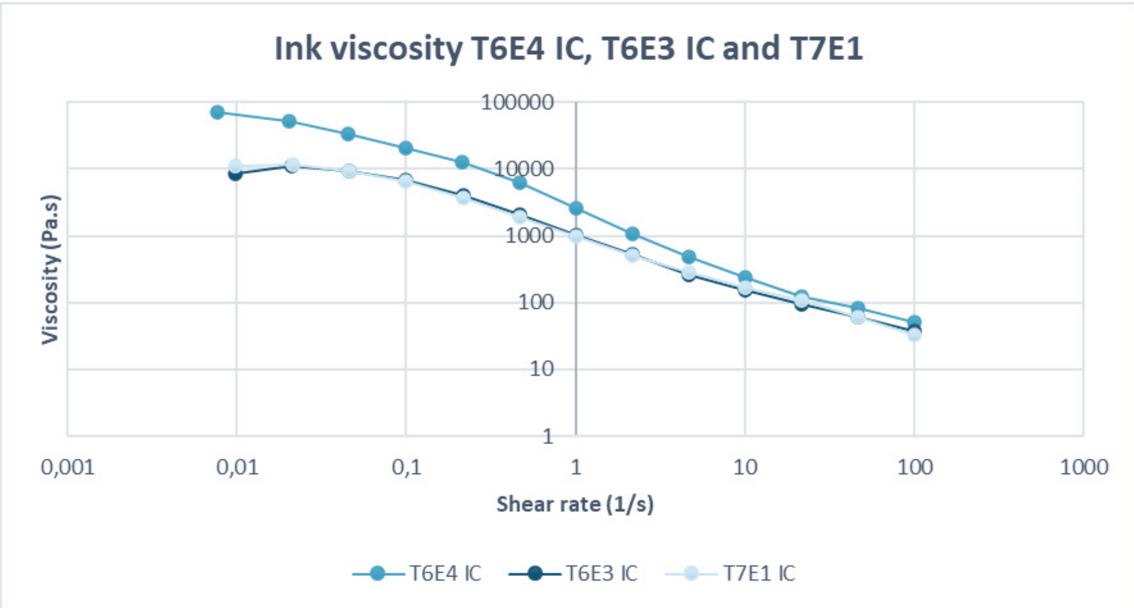
The T7E1 was cured after it was extruded and mixed through the static mixing nozzle. The extruded ink had already a bit of print stability because the extruded ink has a more defined extruded shape, and the ink did not blend together in an unrecognizable shape of ink.

Viscosity measurements



The viscosity graphs of the T7E1 follow the normal order and shape of the previously measured ink viscosities. With the mixed viscosity and part A on top and part B below. What Is different, is that part A seems to have a higher viscosity than the mixed ink, which was never the case before. Also, the starting viscosities of A and B lay farther apart than previously measured ink viscosities.



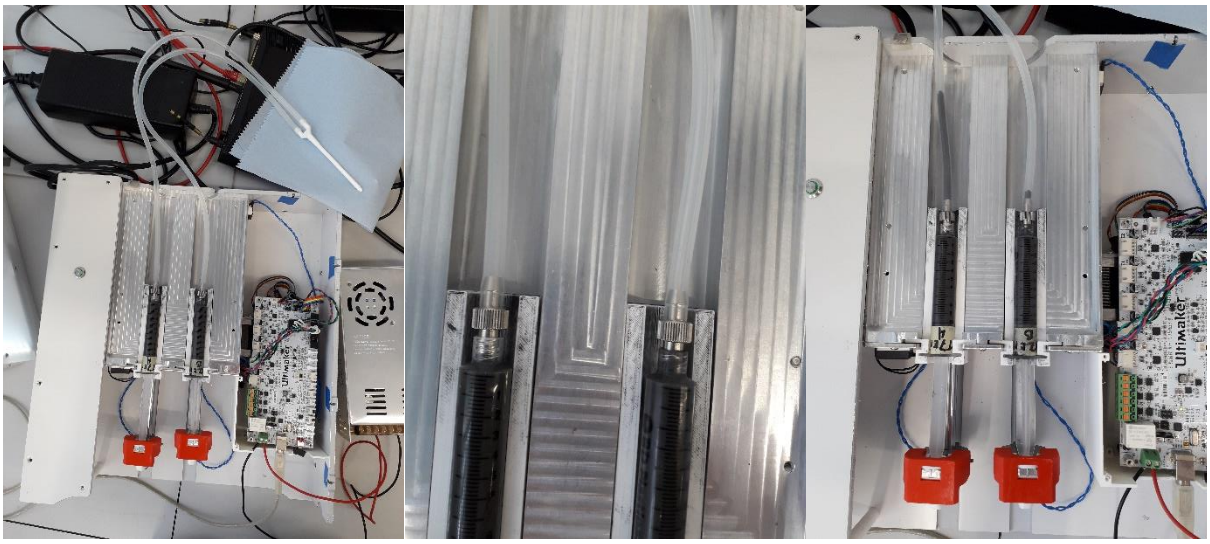


These two graphs show the relation of the mixed T7E1 ink with the other mixed ink viscosity values. As said in the introduction, there was aimed at a viscosity in between the T6E3 and the T6E4. This has not succeeded. The viscosity values of the T7E1 are more comparable with the T6E3, and therefore probably has a too low viscosity for a good print stability.

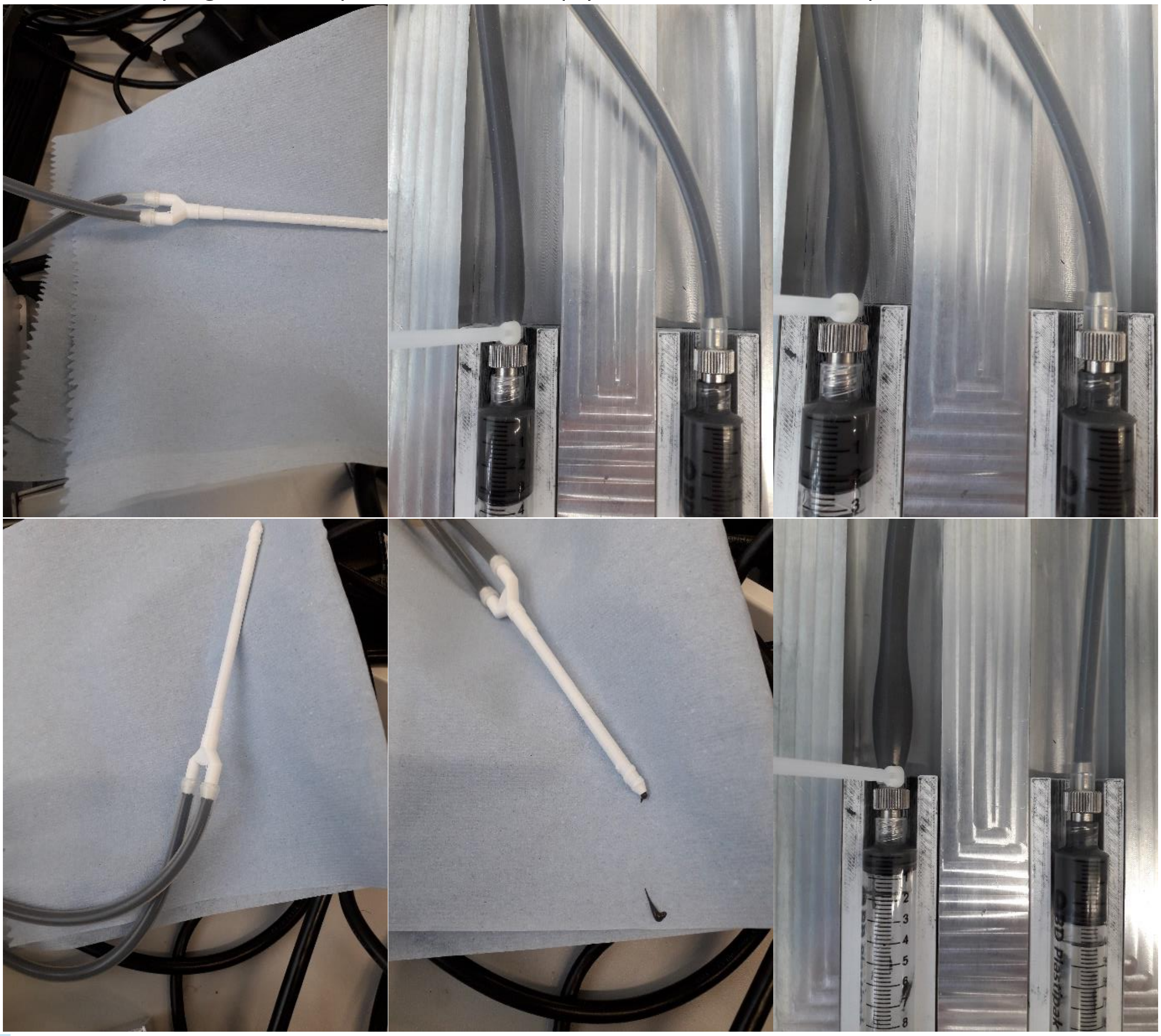
Extrusion with the 3D printer

For this experiment, other tubes were used with an outer diameter of 6,4 mm when they were empty.

First part A was extruded, because of its high viscosity, it often needs the most time for extrusion and problems can occur more often.

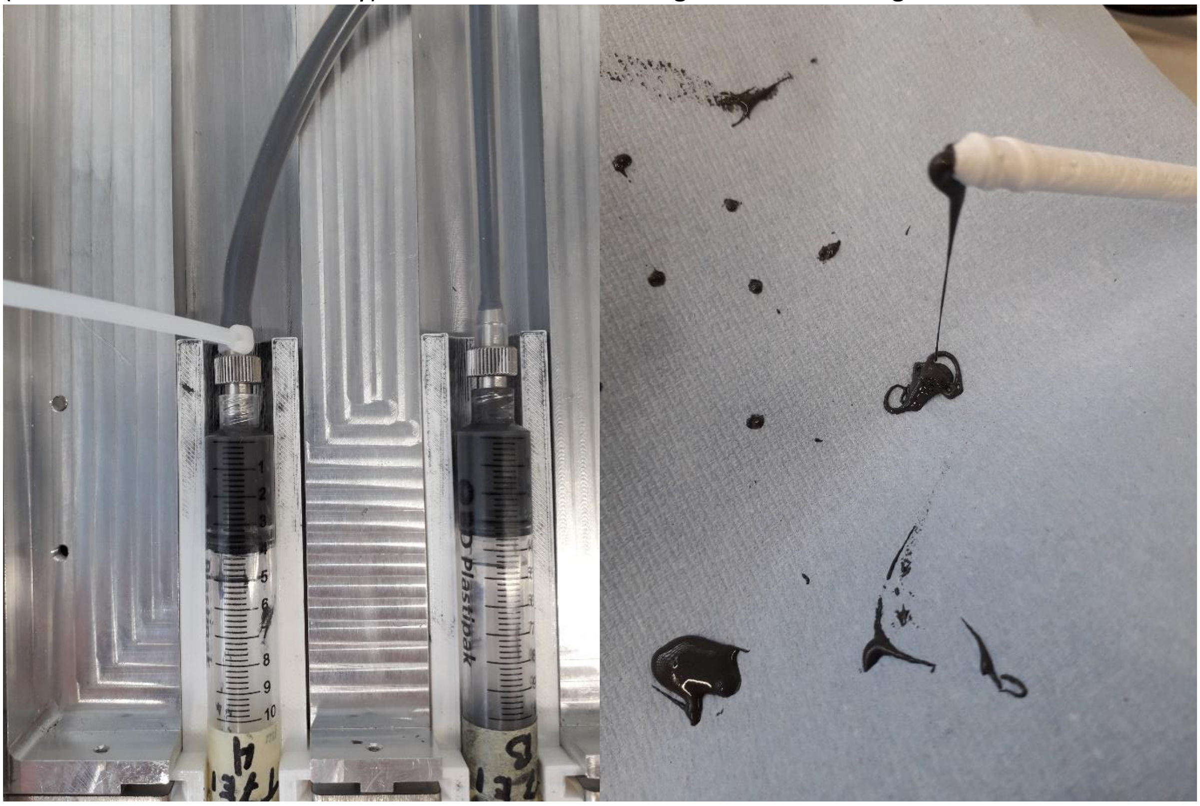


First, the extrusion went well, but at the end when part A almost reached the static mixing nozzle, the tube started to expand. Due to this the volume of A did not go further to the static mixing nozzle but built itself up at the beginning of the tube. Especially the last picture shows the big contrast with ink part B. B was there already extruded through the static mixing nozzle, which part A just reached. The amount of extra built-up volume in the tube of part A can also almost be read from the different levels of the syringes. Where part A is almost empty and started fuller than part B.



In the end, the ink of part A was retracted into the syringe to relieve the tube from the ink. Here can also be seen that there was at least 2 mL of ink in the swelling of the tube. Still, the tube of part A is expanded when compared to the part B tube.

The ink coming out of the static mixing nozzle had a very low viscosity. This could be because part B (which has the lowest viscosity) was first extruded through the static mixing nozzle.



The diameters of the hoses were measured near the nozzles and near the static mixing nozzle:

Diameter of hose A	Near the syringe:	8,3 mm
	The thickest part:	12,3 mm
	Near the static mixing nozzle:	6,5 mm
Diameter of hose B	Near the syringe:	6,5 mm
	Near the static mixing nozzle:	6,3 mm (original tube size)

Conclusion

The T7E1 ink was very strange and did not do, what was expected at the beginning before this experiment started. The extrusion force was higher than expected, while it extruded well. The viscosity was lower, while it was expected higher. Despite its low viscosity, which from previous experiments would mean that it could be extruded easily on the 3D printer, it had problems as if though the ink was very viscous.

It could be that this happened because the static mixing nozzle for the extrusion force test did not have a good surface quality. Or maybe that the new tubes have different qualities than the previous one which caused more resistance for the ink.

What has not changed was how and with what the viscosity measurements were taken. Here the ink had a too low viscosity as what was expected, and a larger difference between the starting viscosities of parts A and B. It could be that something went wrong during the measurements of the different ink components, but we are not aware of that.

Further research

For further research, I would have liked to repeat this experiment, because of its many questionable outcomes. However, due to the time constraints of this project, that is not possible anymore. Therefore to end this project, I will make an ink with a low viscosity that can be extruded and mixed through the 3D printer and will focus on making 2D structures as a demonstrator for this project.

The next test will then be, that maybe 2 different inks will be made with a lower amount of fumed silica than the T6E3 to be magnetized. The graph from the MIT research showed an increased viscosity due to the magnetized particles, and to save time, a lower viscosity ink should be made to compensate for that difference.

Another problem is still the too elastic tubes on the 3D printer. For this stiffer ones should be found to use in the future.

3D printer: try 8

Sanne van Vilsteren
Houman Yarmand
19-08-2021

Introduction

This test has two main purposes. One is to test new tubes with a stiffer wall and see if they do not swell up under the pressure of the ink. For this test a higher viscosity ink than the T6E3, because it is expected that later the magnetized inks will have a higher viscosity than the T6E3. That made the opportunity to repeat the T7E1 from the previous experiment that had questionable outcomes. Now it can be checked if something had gone wrong in the process last time, or if it belongs to the qualities of ink T7E1.

Also, the viscosity of the Ecoflex will be measured in this test, to see how the inks with the particles differ from it or are similar.

If the extrusion through the tubes goes fine, there might also be tried to print the first line of ink.

Materials

Utensils

- 2 10 ml syringes
- 2 scales
  - One with three decimals to make the ink
  - One with a range of up to 24 kg to measure the force needed to extrude the ink through the syringe
- Cups
- Mixing spatulas
- Tape
- Pen
- Double syringe 3D printer
- New clear tubes with a higher wall stiffness (from the syringe to static mixing nozzle) https://www.arestho.nl/transparante-pvc-slang.html
- 2x the adjusted static mixing nozzle v1 of which the tube attachment nozzles have a tapered shape so that the tube could go on better and one in which the syringes can be inserted directly for the pressure test
- 2x syringe mounting piece (convert 60 ml syringe place to a 10 ml syringe)
- 2x syringe piston enlarging piece (convert the 10 ml syringe piston end to one that will fit at the 60 ml syringe spot)
- 2x female Luer lock 4,8 mm connector from the syringe to the tube

Experiment 1: two-component T7E1

Original recipe from 3D printer try 7

- 12,6 g Ecoflex 00-10 part B
- 2 x 22,5 g Iron powder (carbonyl iron: 12)
- 12,6 g Ecoflex 00-10 part A
- 2x 0,30 g fumed silica

Method

Prepare the ink

- Ecoflex part A and Ecoflex part B should be poured into different cups
- Measure the half amount of the NdFeB particles and add this to the cup of Ecoflex part A
- Measure the other half of the NdFeB particles and add these to a cup of Ecoflex part B
- Mix the Ecoflex part A and the iron carbonyl particles manually
- Mix the Ecoflex part B and the iron carbonyl particles manually
- Measure the half amount of the fumed silica and add this to the cup of Ecoflex part A
- Measure the other half of the fumed silica and add this to the cup of Ecoflex part B
- Mix the contents in the cup of Ecoflex part A manually
- Mix the contents in the cup of Ecoflex part B manually

At the end of this step, there should be two cups with a mixture of Ecoflex part A or B with half of the amount of the fumed silica and the NdFeB particles.

Prepare the syringes

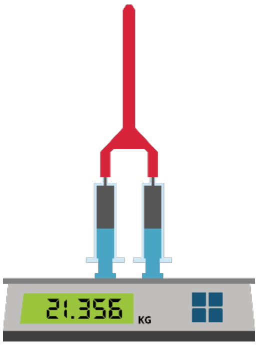
- Load one 10 ml syringe with the Ecoflex A mixture
- Load another 10 ml syringe with the Ecoflex part B mixture

Prepare for extrusion

- <u>Level the two syringes so that they have the same amount of ink in them. This is important so that you can be sure that both syringes have loaded the nozzle with the same amount of ink. That is important because the Ecoflex only becomes a silicone if the two are mixed. From earlier research, it was already found out that the mixing ratio of 1:1 (which is also the instruction on the Ecoflex bottles itself) work the best.</u>
- Mount both of the syringes at the top of the static mixing nozzle

Measure extrusion force

- **Measure: In previous experiments, it took too much force to extrude the ink by hand and we had to press in through the static mixing nozzle by pushing the pistons of the syringes on a table. To measure the force needed to extrude the ink, the pistons of the syringes should be pressed against a scale when extruding the ink to see the maximum force needed (see the figure on the right).**



Measure viscosity

The viscosity of the ink should be measured in three conditions. Those are:

- Ink component A separately
- Ink component B separately
- The ink after it is just mixed

This way we can get an insight into how the viscosity changes over the printing project.

This time also the viscosity of the Ecoflex components, separately and mixed, will be measured to see if it behaves differently without additional particles mixed in.

The measuring procedure:

- 40-mm-diameter steel plate geometry.

- Steady-state flow experiments with a sweep of shear rates (0,0-100 s^{-1})
- The inks were brought to a temperature of 25°C for one minute before the experiment started.
- The gap height between the geometry and the plate always was 0,5 mm.

Prepare the 3D printer

- Attach the tubes to the syringes with the use of the female Luer lock connectors
- Attach the tubes to a new static mixing nozzle
- Insert the 60 to 10 ml conversion pieces into the printer where the syringes will normally be installed
- Then the syringes should be put in the conversion pieces
- The mechanical parts that push the pistons of the syringes forward should be brought up
- The syringe piston enlarging pieces should be put over the end of the 10 ml syringe pistons
- At last, the red clips should be attached over the syringe piston enlarging pieces

Printer settings

First part A was extruded because this part often has the most trouble with extrusion through its higher viscosity. For this the following settings were used until A was extruded to the static mixing nozzle:
F2 A178 (2 mL) B10

After A was extruded till the static mixing nozzle, B was extruded up to the static mixing nozzle with the settings:
F2 A5 B356 (4 mL)

When both ink components have arrived at the static mixing nozzle the following settings were used to push the ink through the static mixing nozzle:
F2 A45 (0,5 mL) B45 (0,5 mL)

It was also tried to extrude ink while printing with the following 3 snippets of g-code:
Try 1:
G0 F10000 X0 Y0 Z1
M400
G1 F4 A10 B10
G1 F300 X10 Y0 Z1
M400

Try 2:
G0 F10000 X100 Y100 Z170
M84
G0 F10000 X0 Y0 Z1
M400
G1 F1 A10 B10
G1 F30 X10 Y0 Z1

M400

Try 3:
G0 F10000 X0 Y0 Z1
M400
G1 F1 A20 B20
G1 F30 X10 Y0 Z1
M400

Results

Maximum extrusion force

20,990 Kg

The maximum measured extrusion force of the T7E1 ink made this time was 20,990 Kg. This equals 20,990/2*10 = 104,95 N per syringe.

This extrusion force still lays between the T3E1 and the T6E4 with respectively above the 24 Kg and 19,069 Kg. This is still strange because the amount of fumed silica added in the recipe of the T7E1 is below both of these inks. This time in comparison with the T7E1 from 3D printer try 7, is that the value is lower. In the last test, the extrusion force that was measured was 116,96N.

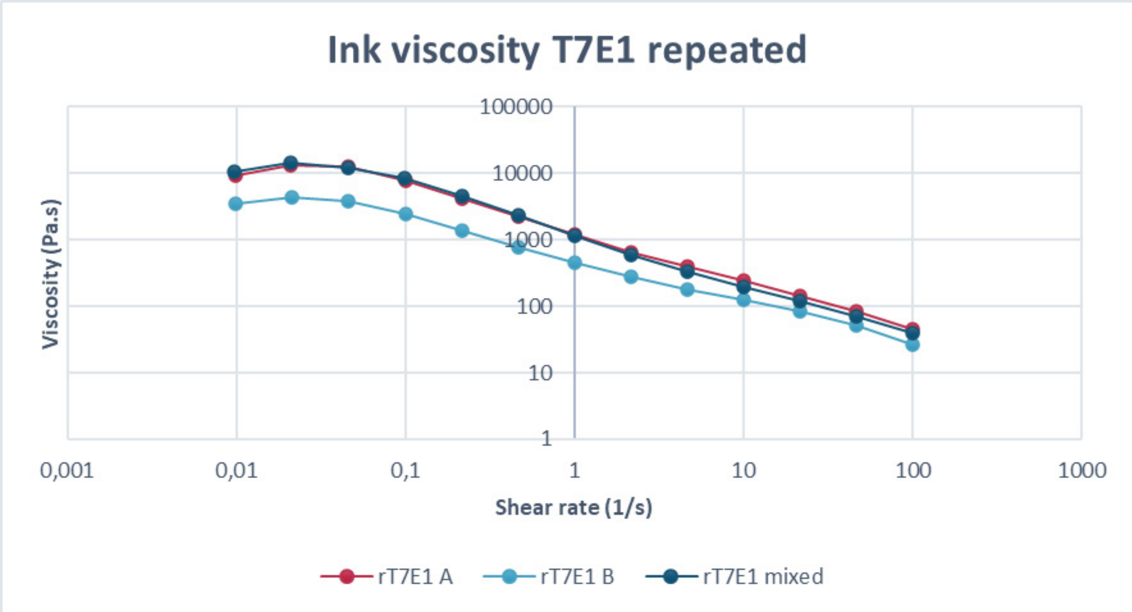
The surface quality of the static mixing nozzle used in this test was better than the one that was used in the previous test.

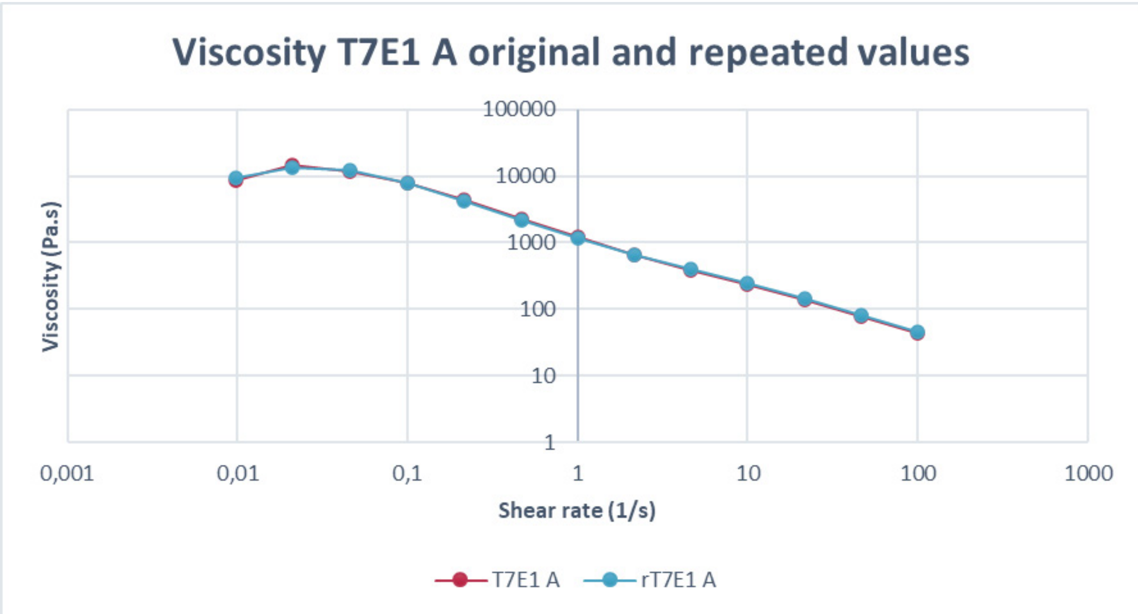
Ink curing after extrusion through the static mixing nozzle (static mixing nozzle test)

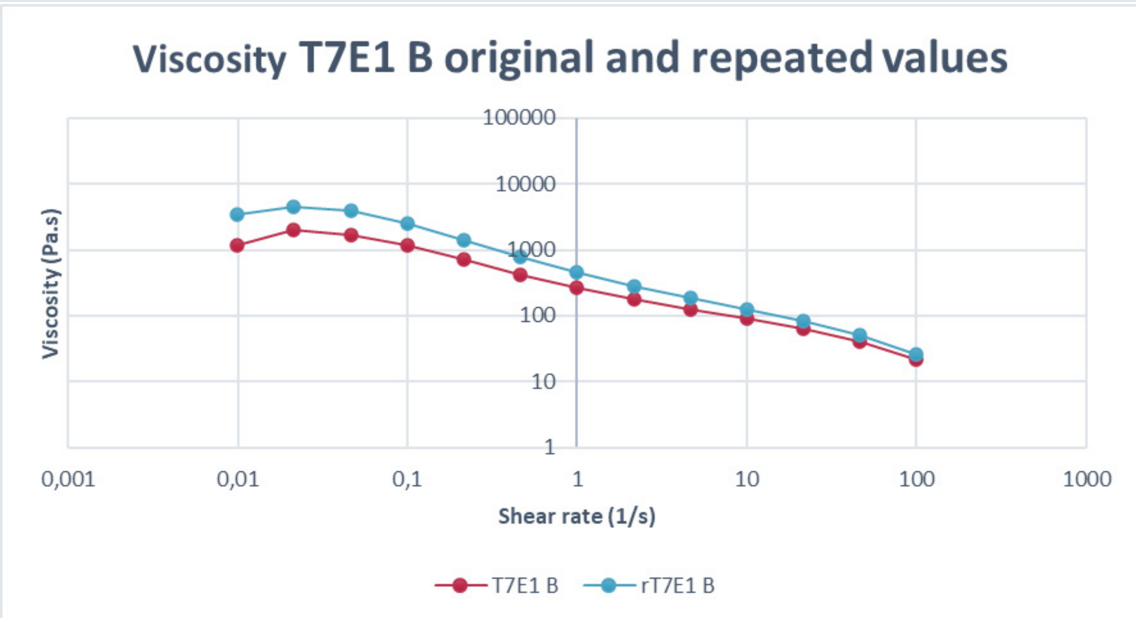
The T7E1 was cured after it was extruded and mixed in the static mixing nozzle extrusion force measurement test.

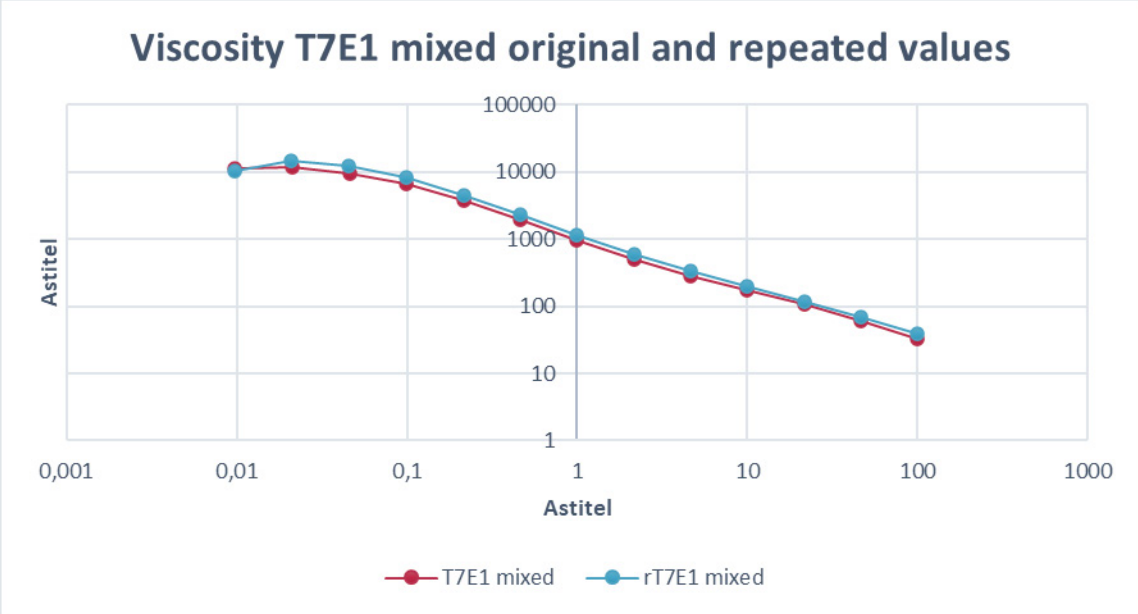
Viscosity measurements

When comparing the repeated T7E1 (rT7E1) viscosity measurements with the T7E1 from 3D printer test 7 (T7E1), there might have been a mistake last time in making the B component of the ink. In the table where the two B components are compared (**Viscosity T7E1 B original and repeated values**), the two graphs do deviate from each other. For component A this is not the case and there the ink components from experiments 7 and 8 seem identical. The difference in component B is probably also the cause of the difference in the graphs of the mixed inks (**Viscosity T7E1 mixed original and repeated values**).

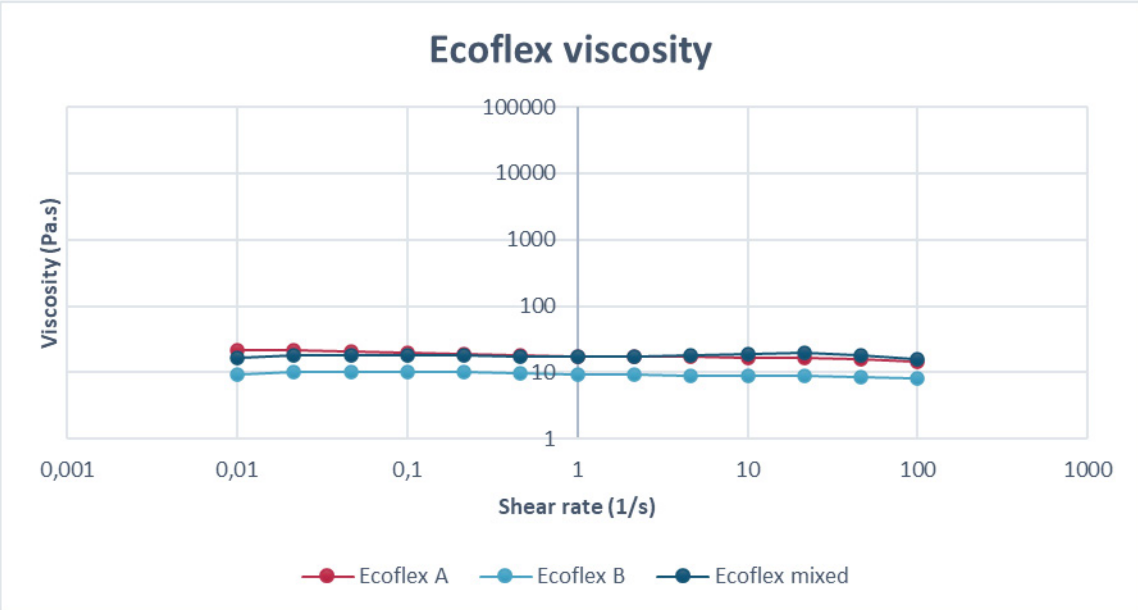

Ink viscosity T7E1 repeated
Viscosity (Pa.s)
100000
10000
1000
100
10
1
0,001
0,01
0,1
1
10
100
1000
Shear rate (1/s)
rT7E1 A
rT7E1 B
rT7E1 mixed


Viscosity T7E1 A original and repeated values
Viscosity (Pa.s)
100000
10000
1000
100
10
1
0,001
0,01
0,1
1
10
100
1000
Shear rate (1/s)
T7E1 A
rT7E1 A

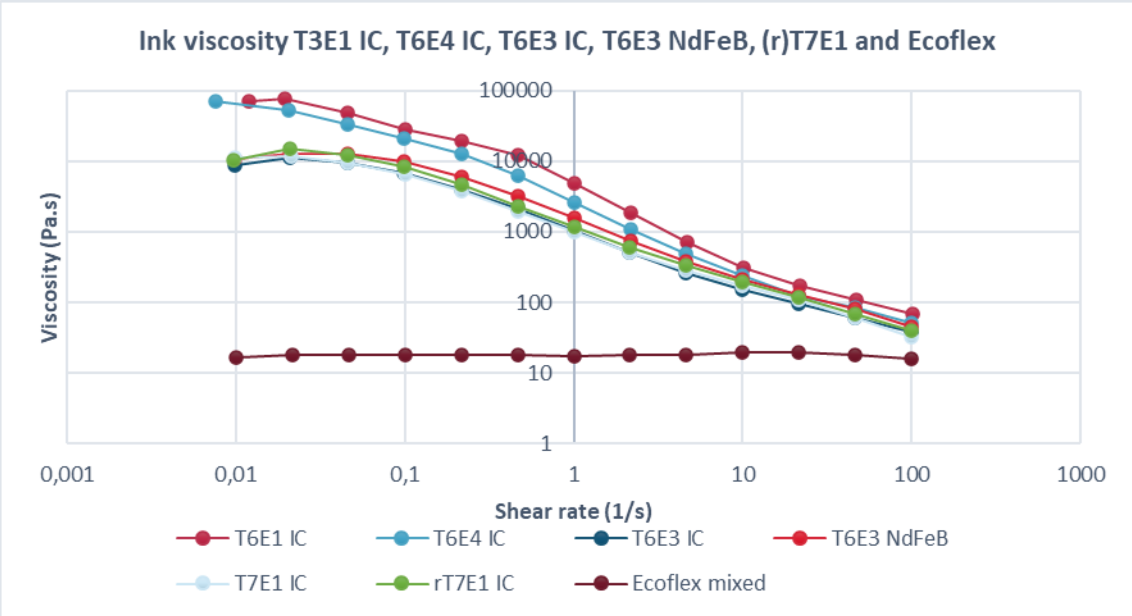

Viscosity T7E1 B original and repeated values
Viscosity (Pa.s)
100000
10000
1000
100
10
1
0,001
0,01
0,1
1
10
100
1000
Shear rate (1/s)
T7E1 B
rT7E1 B



The viscosity of Ecoflex components A, B and mixed was also measured in this test:



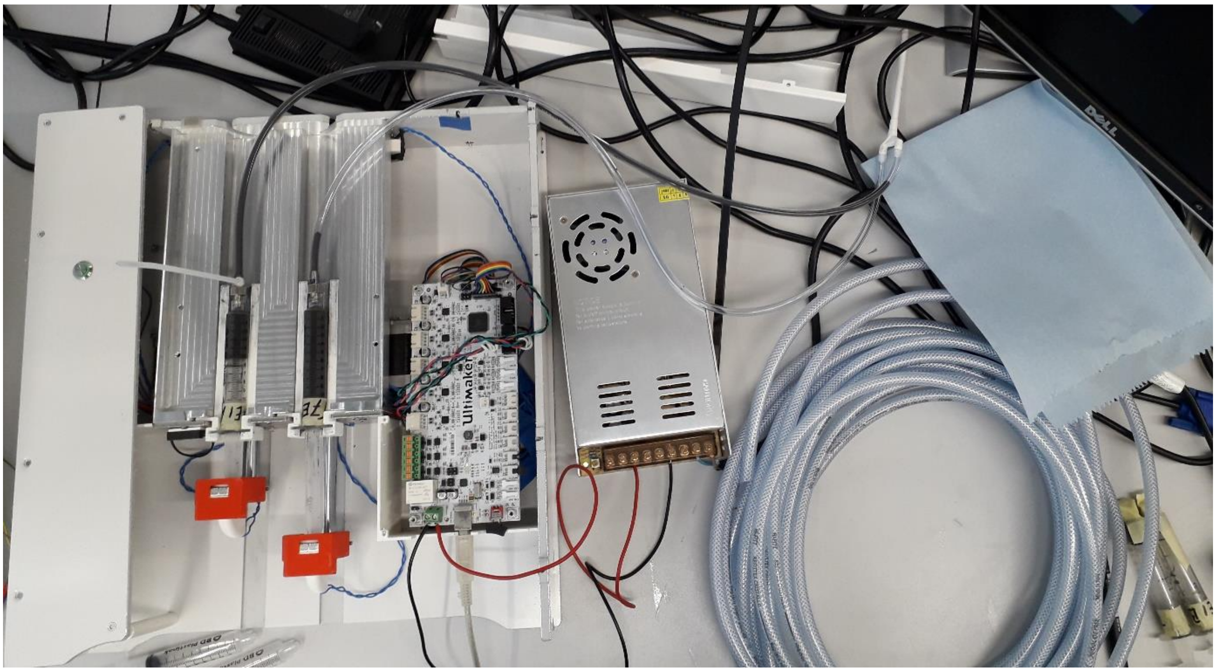
When the Ecoflex measurements are compared with all the other inks (**Ink viscosity T3E1 IC, T6E4 IC, T6E3 IC, T6E3 NdFeB, (r)T7E1 and Ecoflex**). It can be seen that the viscosity of the Ecoflex stays constant. This means that Ecoflex does not have shear-thinning properties, which are important for 3D printing with silicones (Zhou et al., 2019). In the study by Zhou et al. (2019), it was explained that the fumed silica was added to the silicones to give them shear thinning properties. This makes a silicone less viscous during extrusion, but when it is not moved after printing inner bonds in the material will be restored to increase the viscosity and give the material some print stability. I do not know if the magnetic particles also give some of these properties to the ink, but for the magnetizer test, I think it will be best to keep some fumed silica in the recipe after this test.



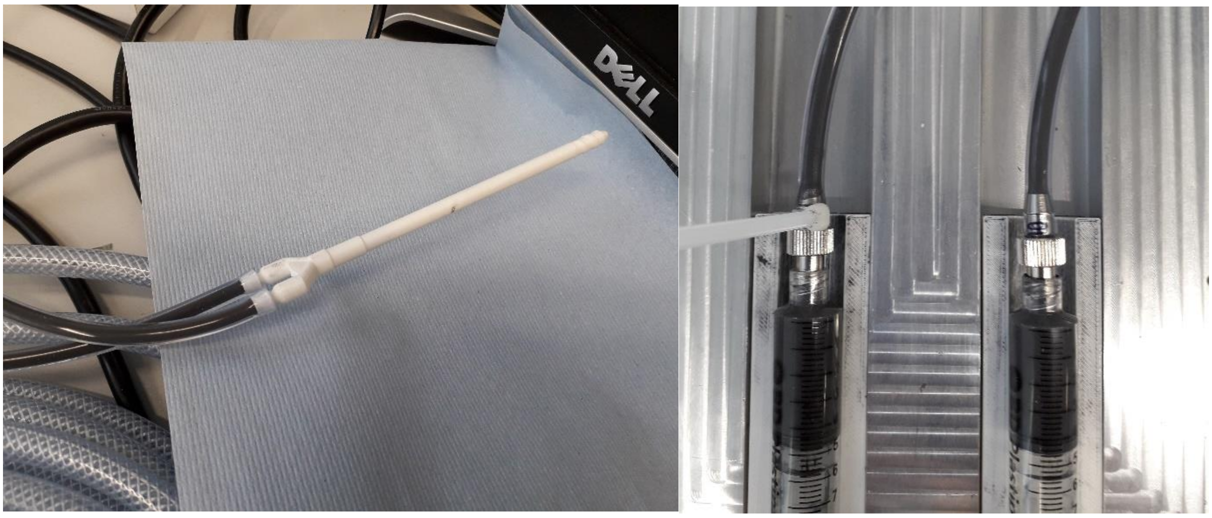
Extrusion with the 3D printer

For this experiment again new tubes were tested: [https://www.arestho.nl/transparante-pvc-slang.html.](https://www.arestho.nl/transparante-pvc-slang.html) These had a stiffer wall and measured 6 mm diameter on the outside before extrusion. Even though the wall was stiffer we still could get the tubes over the Luer lock connections and the connection of the static mixing nozzle.

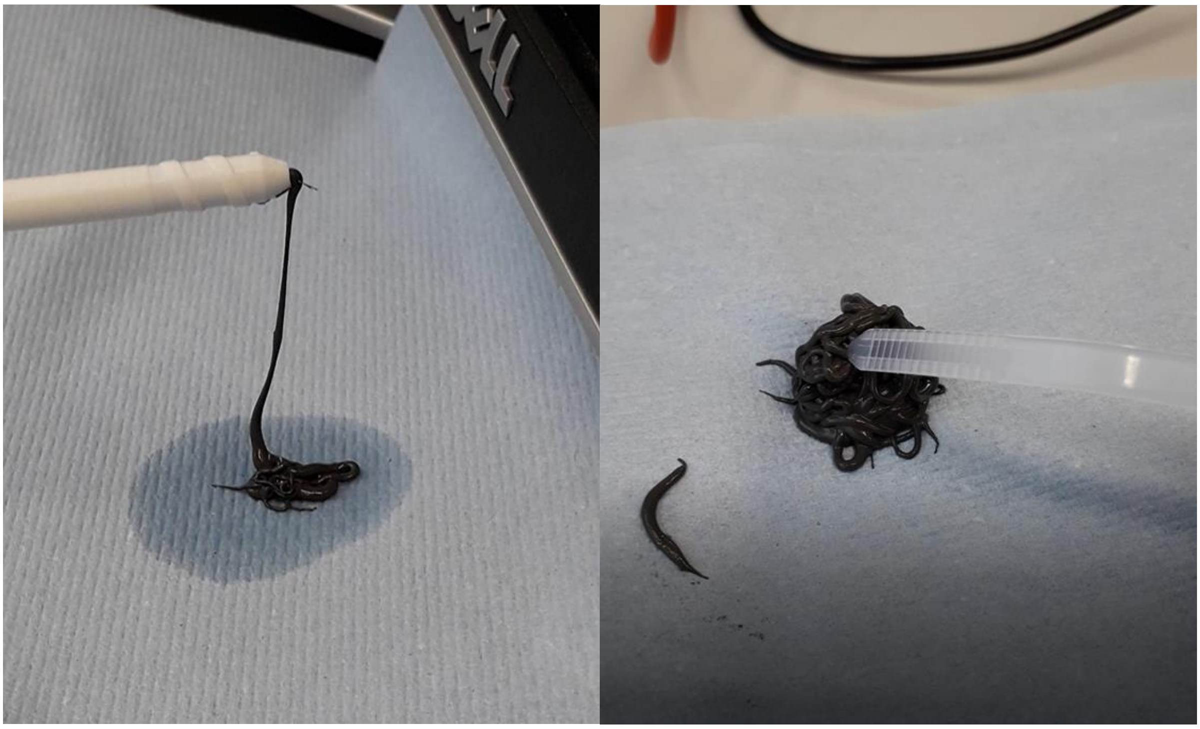
First Ecoflex part A was extruded through the tube because this mostly has the most trouble through its high viscosity. The extrusion went well, only at the end sometimes the motor of A stopped turning, but without regularity. In the end, the motor turned well.



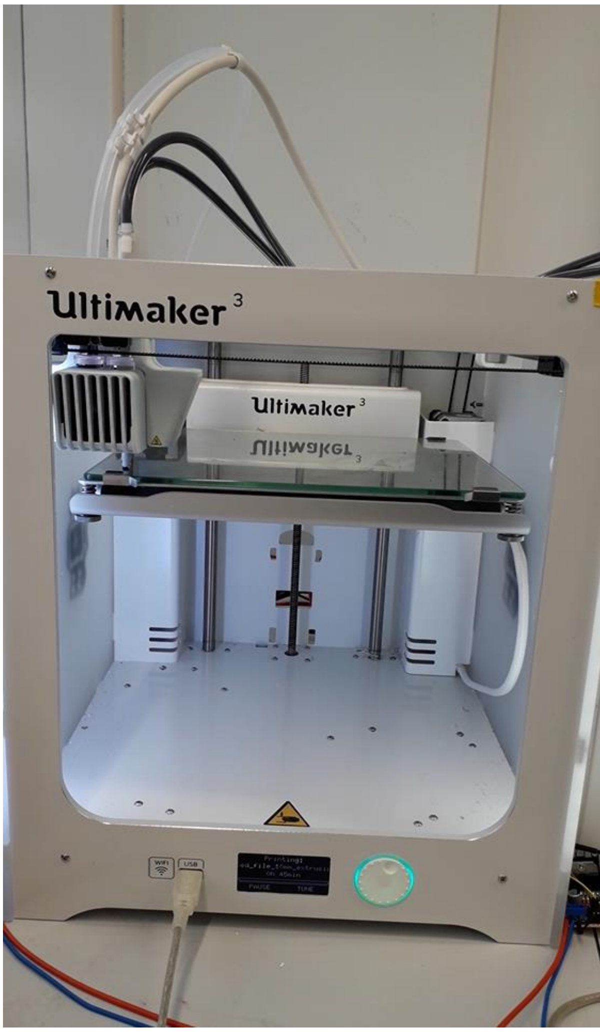
After that Ecoflex B was extruded. This went smoothly without failing motors.

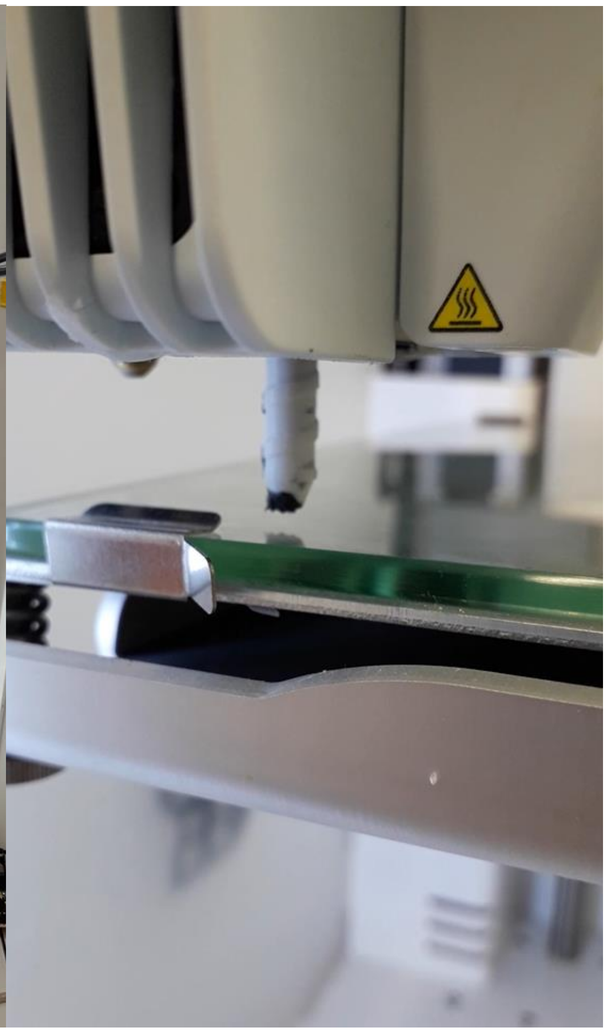


The new tubes worked and were stiff enough. In the picture above the connection of the tubes to the syringes can be seen when the entire tubes are filled with ink, and they have not swollen through the extrusion. The maximum measured diameter was 6,1 mm.



The ink got extruded through the static mixing nozzle with the printer setup and the ink did cure. This means that both ink components, A and B, can be extruded through the static mixing nozzle together in the 3D printer system and got mixed. The lines on the pile of ink are still individual which means that the ink has a bit of print stability which. Due to the not swollen tubes and the extruded ink, it was also tried to print the first line with it on the 3D printer.





However, when it was tried to extrude ink from the syringes and draw a line of the ink on the glass plate of the 3D printer (pictures above), no ink came out of the static mixing nozzle. It was tried several times with no effect. This part of the g-code was tried again: F2 A45 (0,5 mL) B45 (0,5 mL) to get ink out of the static mixing nozzle, but still, nothing happened.

After some minutes, the static mixing nozzle started dripping ink (picture on the right). So, there is some delay in the system, preventing the ink to react directly.

Conclusion

When comparing the T7E1 viscosity measurements with the ones of the T7E1 from experiment 7, there might have been that a wrong amount of particles was added to Ecoflex part B. A difference in the viscosity can only be spotted in the part B measurements, the part A measurements are almost identical.

The viscosity measurements of Ecoflex showed now shear-thinning properties, which means that one or both of the added particles add that to the Ecoflex. It is assumed that the fumed silica adds it due to the study of Zhou et al. (2019), but it is not known if the magnetic particles also add something.

The tubes did not swell during the extrusion and the motors did not fail during the extrusion of the T7E1 ink. Which makes the extrusion system now almost complete and working.

The new problem is that there is a delay in the extrusion of the ink through the system of which the origin is not identified yet.

Further research

In further research, the cause of the delay in the ink extrusion should be found out. This should be controllable to print shapes with the 3D printer.

References

Zhou, L.-y., Gao, Q., Fu, J.-z., Chen, Q.-y., Zhu, J.-p., Sun, Y., & He, Y. (2019). Multimaterial 3D Printing of Highly Stretchable Silicone Elastomers. *ACS Applied Materials & Interfaces, 11*, 23573-23583. doi:10.1021/acsami.9b04873

B.e 3D printer tuning

In these tests, the values were found for different G-code commands. The A and B values to extrude 1 mL of ink, the speeds of different F values and the tests to see if a 3D printer pin could be turned on.

3D printer tuning 1

Sanne van Vilsteren
8 and 12-07-2021

Introduction

To use the 3D printer to print shapes with the values in the G-code, these values should first be found. This document focuses on the A, B and F values in the G-code. A and B are for the amount of extrusion of the A and B syringe respectively. Being a certain distance that is covered depending on the input values. F is the speed at which the volume of A and B is extruded and how fast the piston is moved.

During the measuring of the volume of the syringes by controlling A and B, empty syringes were used. During the speed measurements, the syringes were filled with water and the volume was confirmed.

Volume: A and B values for 1 mL ink extrusion

For the extrusion values of A and B, there was first started with the value 77. This value came from an earlier test with ink inside the syringes, but it was not constant.

The measured length to extrude 1 mL in a 10 mL syringe was measured to be 6 mm. This value was used to calculate new A and B values to try. For example, A77 had a distance of 3,5 mm. To calculate a new A value:

$$\frac{6}{3{,}5} * 77 = 132$$

$$\frac{mm\ for\ 1\ mL\ extrusion}{measured\ mm} * A\ value\ used$$

The first measurements were a bit off with the rest of the measurements, this could be because the system first has to be prepared for extrusion. As can be seen in the top image there is for example some space between the pistons and the buttons, which first has to disappear before the piston can be pushed forward. After the second measurement, the values for the third test were quite good with A91 and B93. A later seemed to go a bit too far, whereby the travelled distance did not match the 6 mm for 1 mL of extrusion. That is why it was lowered to A89 and that worked better.

Because the motors and spindles of the 3D printer work the same there was decided on A89 and B89 to extrude 1 mL of ink at the end of the experiment.

The measured values can be found in the table below. The A89 and B89 values were tested and measured to be 6 mm more in the end than are included in the table.

G-code A value	measured moved distance (mm)	G-code B value	measured moved distance (mm)	remarks
77	3,5	77	4	
132	8,7	115,5	7,5	
91,03448276	6	92,4	6	seemed to work
repeat		repeat		A is a bit off
89		repeat		seems okay
repeat		repeat		
measured on syringe				
1 mL =	6	mm		

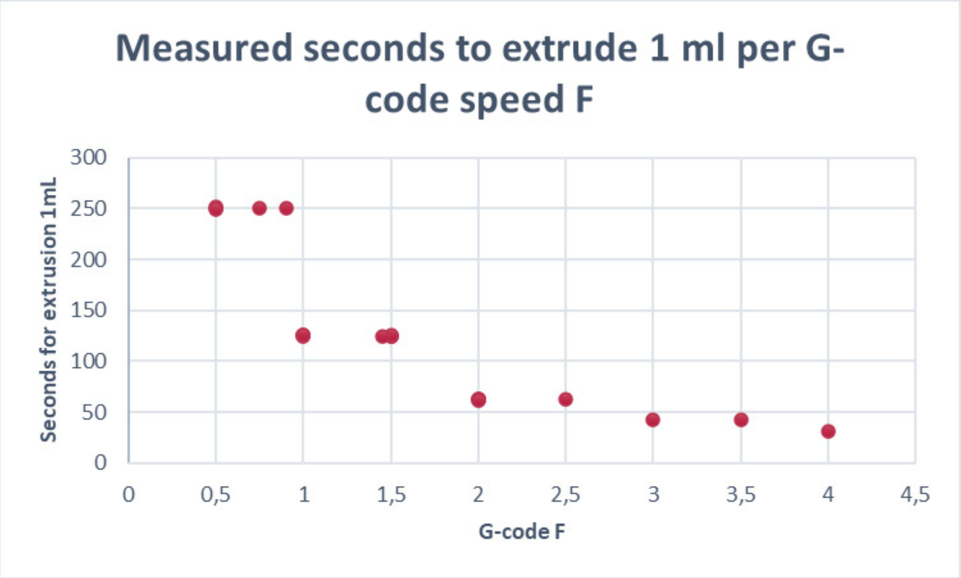
Speed: the number of seconds needed to extrude 1 mL ink for different F values

For the speed of the extrusion through the syringes, the F command in the G-code should be used. To measure the speed belonging to the F value the A89 and B89 values were used to extrude 1 mL of the previous experiment. There was first started with F2 and F1 because we had used these values before in testing the 3D printer.

<u>Measuring procedure</u>
During the extrusion of 1 mL, the time was kept with a stopwatch from the beginning that the motors started turning till they turned off.

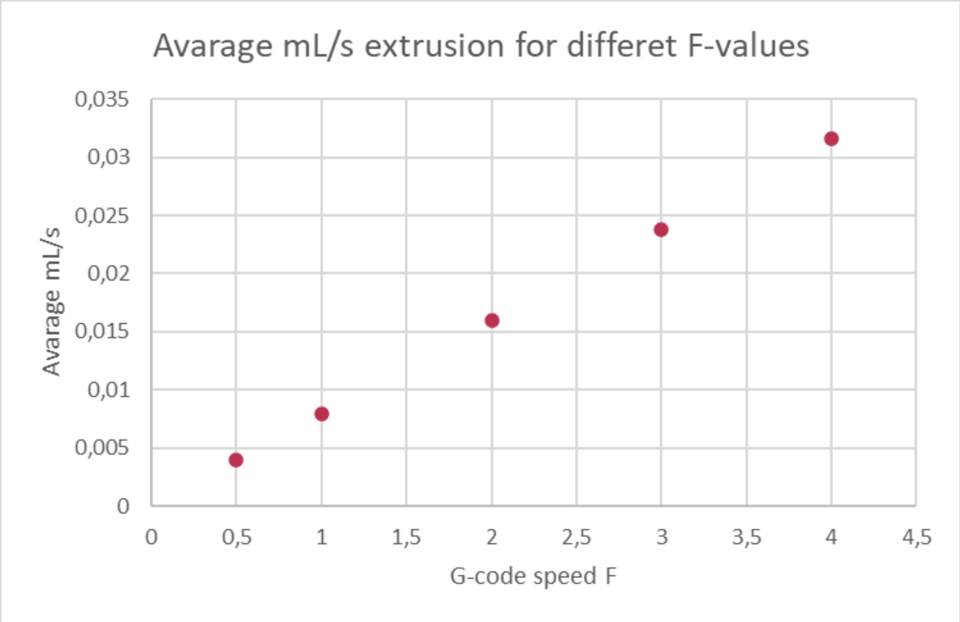
This was first done for the values F2, F1 and F0,5. The amount of time that it took was very constant and the number of millimetres was corresponding with the earlier measured volume.

When the time was measured for the extrusion of 1 mL with F1,5, the time seem to correspond with the time needed to extrude 1 mL with F1. Due to that more half values and larger values were measured. (see the table and graph below). It was concluded that the 3D printer could only use whole numbers for controlling the extrusion speed.



Speed extrusion	amount extrusion A	amount extrusion B	seconds	mm
F2	A89	B89	62,62	6
F2	A89	B89	62,17	12,3
F2	A89	B89	62,91	6
F2	A89	B89	62,46	
F1	A89	B89	125,4	6
F1	A89	B89	124,94	
F1	A89	B89	125,38	
F0,5	A89	B89	250,24	6
F0,5	A89	B89	251,09	
F0,5	A89	B89		
F1,5	A89	B89	124,62	??
F1,5	A89	B89	124,73	???
F1,5	A89	B89		
F1	A89	B89	125,79	
F2	A89	B89	61,83	
F0,5	A89	B89	249,37	
F1,5	A89	B89	125,78	
F1,45	A89	B89	124,96	
F0,75	A89	B89	250,3	
F0,9	A89	B89	250,28	
F3	A89	B89	41,97	
F2,5	A89	B89	62,45	
F4	A89	B89	31,64	
F3,5	A89	B89	42,11	

After that, all the values of the whole F values were sorted and the average amount of mL/s was calculated. The graph below where the mL/s is plotted against the F value shows a linear relation, through which the other values could be calculated.



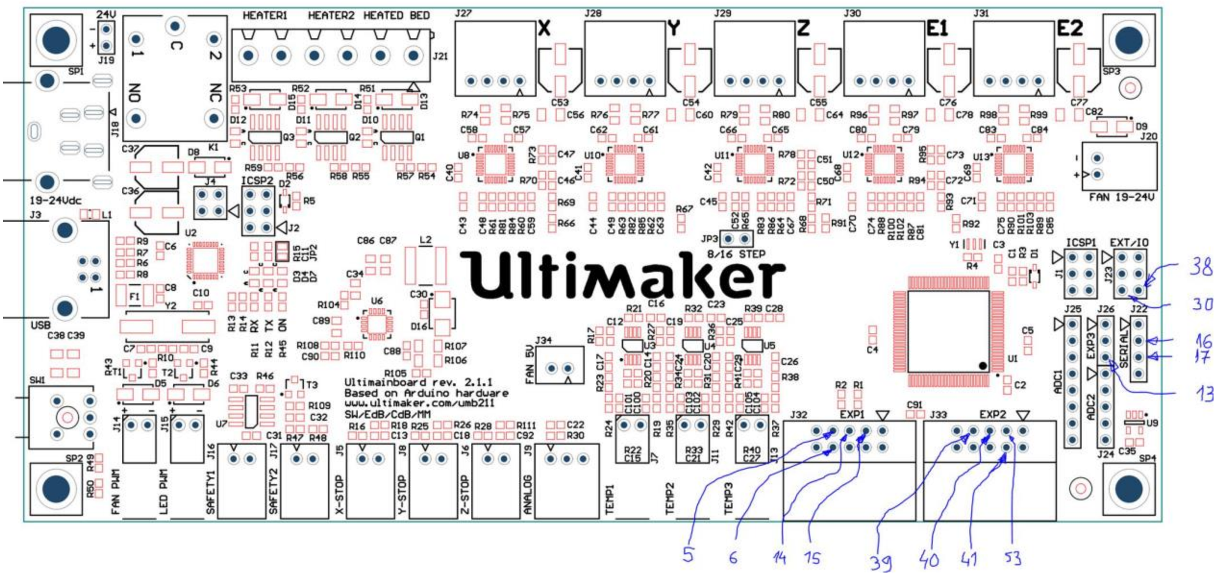
F	Speed extrusion	amount extrusion A	amount extrusion B	seconds	mm	mL/s		avarage mL/s
1	F0,5	A89	B89	250,24	6	0,003996164		0,003996302
1	F0,5	A89	B89	251,09		0,003982636		
1	F0,5	A89	B89	249,37		0,004010105		
1	F1	A89	B89	125,4	6	0,007974482		0,007975959
1	F1	A89	B89	124,94		0,008003842		
1	F1	A89	B89	125,38		0,007975754		
1	F1	A89	B89	125,79		0,007949758		
2	F2	A89	B89	62,62	6	0,015969339		0,016026723
2	F2	A89	B89	62,17	12,3	0,016084928		
2	F2	A89	B89	62,91	6	0,015895724		
2	F2	A89	B89	62,46		0,016010247		
2	F2	A89	B89	61,83		0,016173379		
3	F3	A89	B89	41,97		0,023826543		0,023826543
4	F4	A89	B89	31,64		0,031605563		0,031605563

3D printer tuning 2

Sanne van Vilsteren
Andres Hunt
Zjenja Doubrovski
10, 12, 13 and 16-08-2021

Introduction

To control the electromagnet with the 3D printer, the electromagnet has to be connected to the pins of the circuit board of the 3D printer. This was done and the electromagnet was connected to pins 50 and 52 through the H-bridge. However, when the G-code was run there was no output of 5 volts measured at the assigned pins. In this test, multiple other general-purpose pins of the 3D printer will be tested to see which can be used to connect the electromagnet. These pins are indicated in the picture below.



Materials

- The Ultimaker 3 that is used in this project
- Multimeter

Method

To turn the pin on, the following G-code was used:

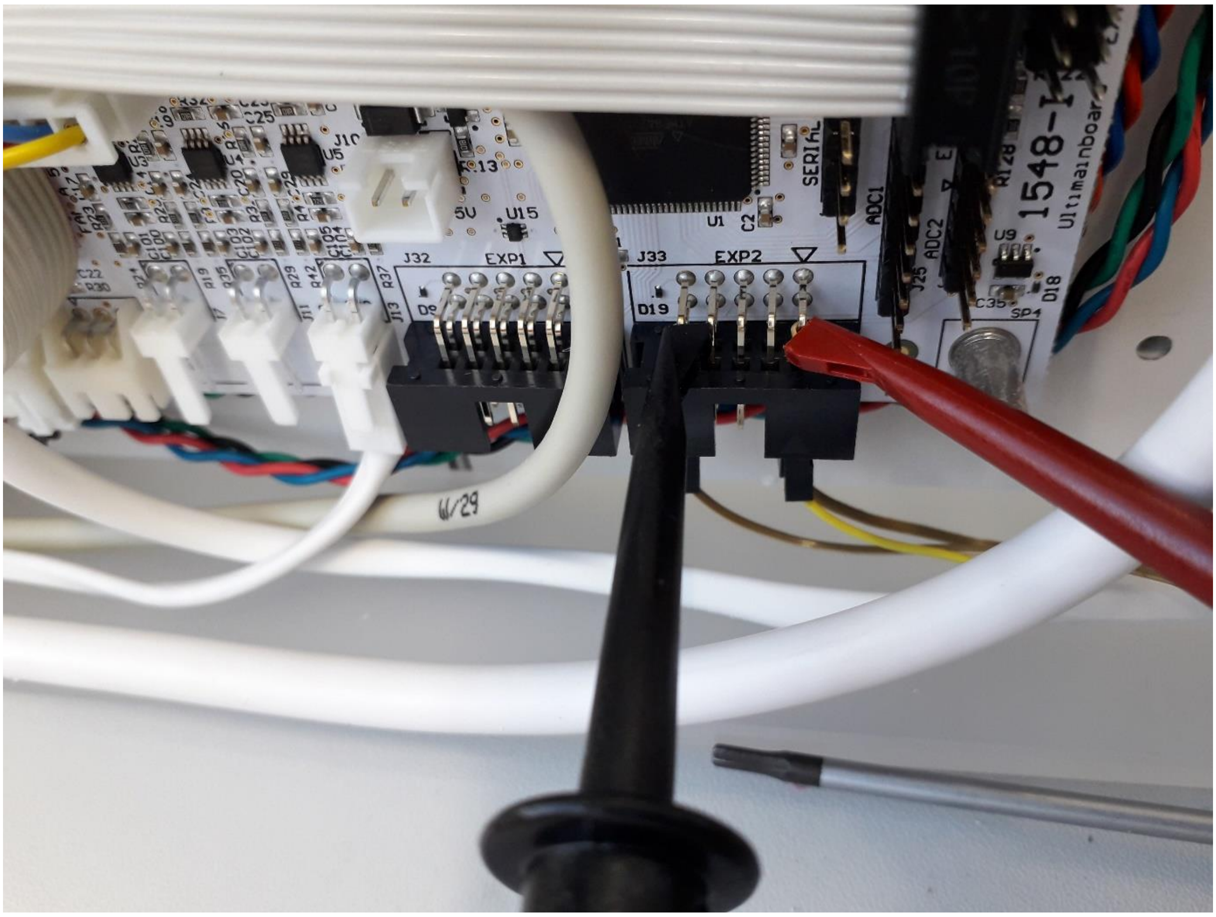
```
G0 F10000 X178 Y6 Z170
M107
G0 F10000 X100 Y100 Z170
M84
M400
M42 P5 S255
G1 F120 X0 Y0
M400
M42 P5 S255
```

G1 F120 X10 Y0
G4 P1000
M400
M106

M104 S0
M104 T1 S0

More about this entire G-code can be read in the Appendix called G-codes. The red **P5's** in the G-code determine which pin is turned on. If another pin has to be turned on the 5 has to be replaced with another number. The pins that were tested are: 5, 6, 13, 14, 15, 16, 17, 24, 30, 38, 39, 40, 41, 50, 52 and 53.

A value of S255 was sent to the pin which should result in an output of around 5 V if the pins work properly. This was tested by connecting the pin and a ground pin to a multimeter to measure the voltage over the pins (see picture below).



Results and conclusion

There was no voltage measured over any of the mentioned pins above (see picture below). First, it was thought that it could be a problem with a group of pins, that they might be used for the dual syringe extruder extension of the printer. This was because with the same code Zjenja could turn on these pins on another Ultimaker 3. Now because not one of the pins responded, we think that there might be another firmware installed on the 3D printer of this project, to control the dual syringe extruder, which was a prototype of Ultimaker. This was the last test on testing the 3D printer pins for this project. If it is continued, it might be needed to look into the firmware of the printer to be able to control the electromagnet with the G-code.

C. Viscosity measurements data

During the project, the viscosity of multiple inks has been measured. For this, the same stings were used as in the research of MIT. In figures C.1 and C.2, the geometry and procedure settings can be seen that were used.

This Appendix will further show all the tables and graphs of the measured inks sorted on the amount of fumed silica. The Excel files have been transferred to the chair and mento of this project.

More combined graphs of different inks can be found in the test documentation in Appendix B.

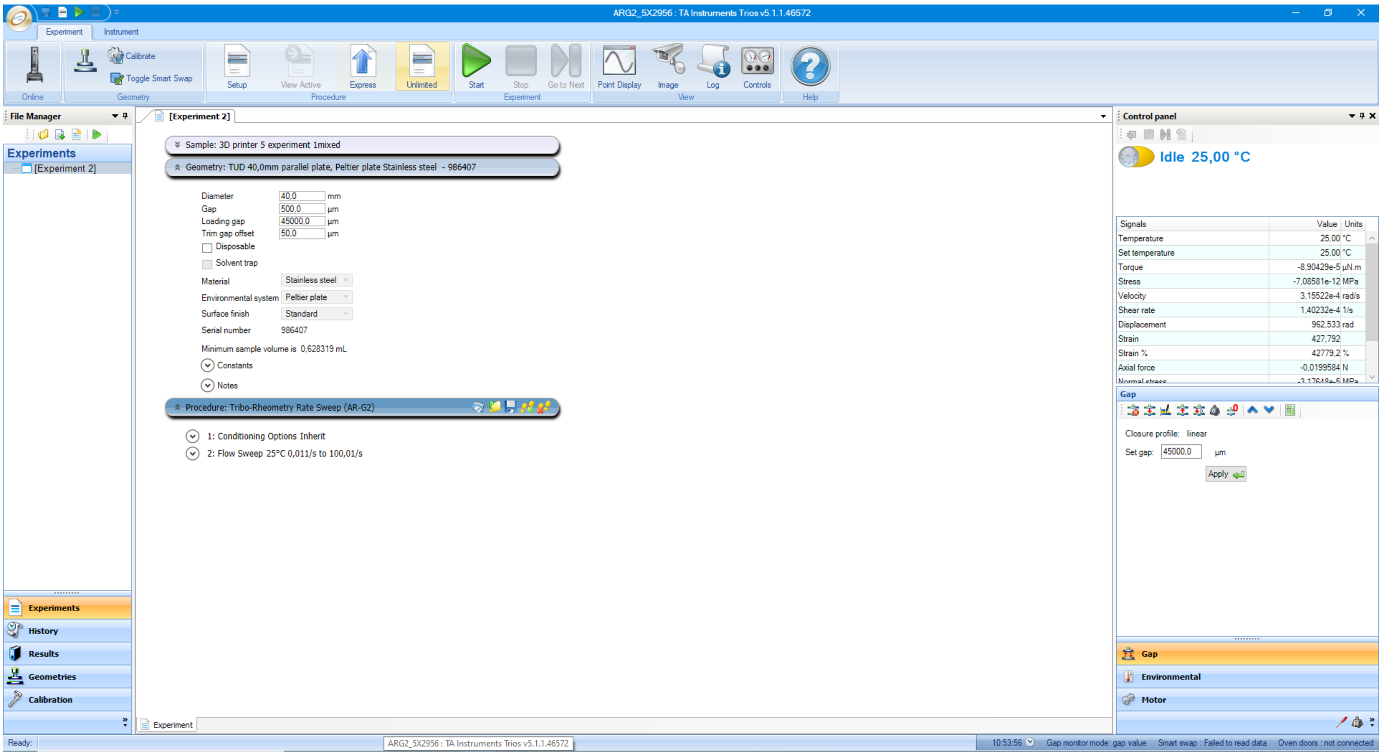


Figure C.1: The geometry of the steel plate in the program to operate the AR-G2

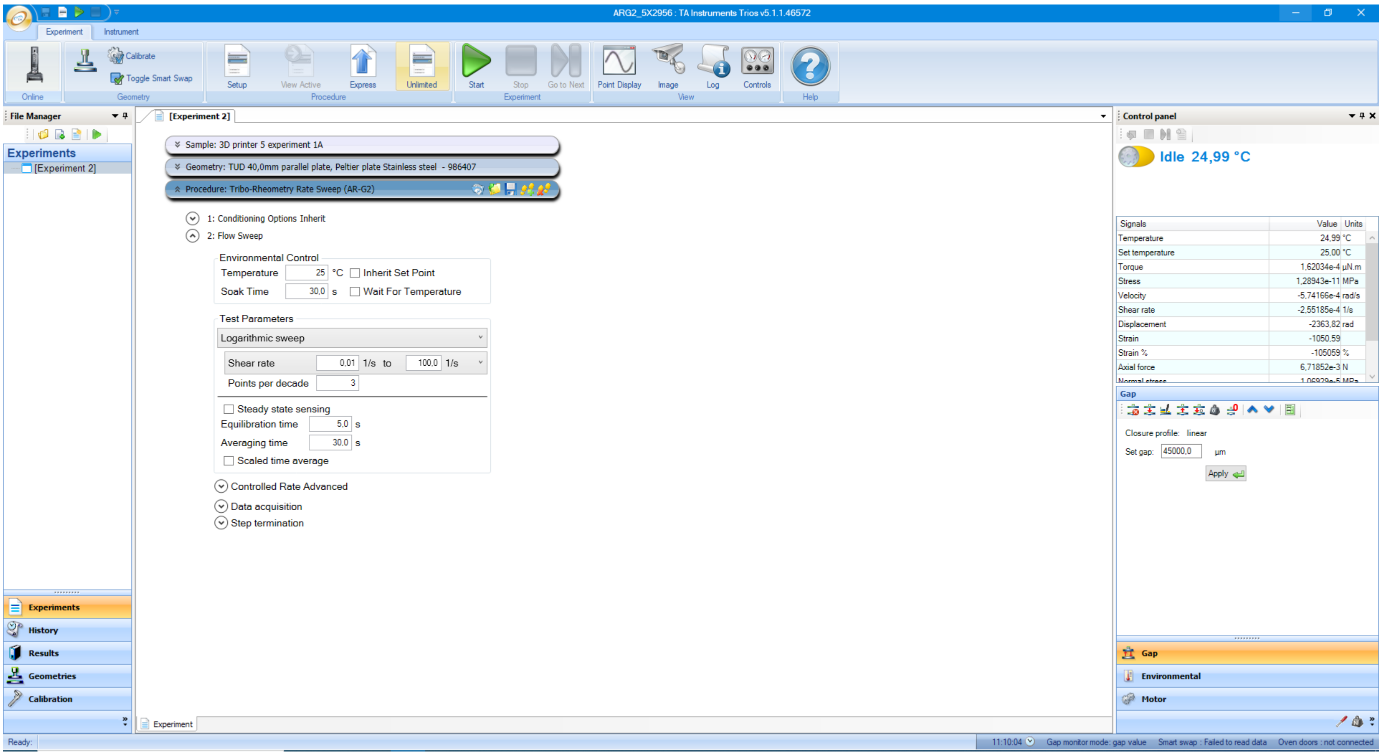
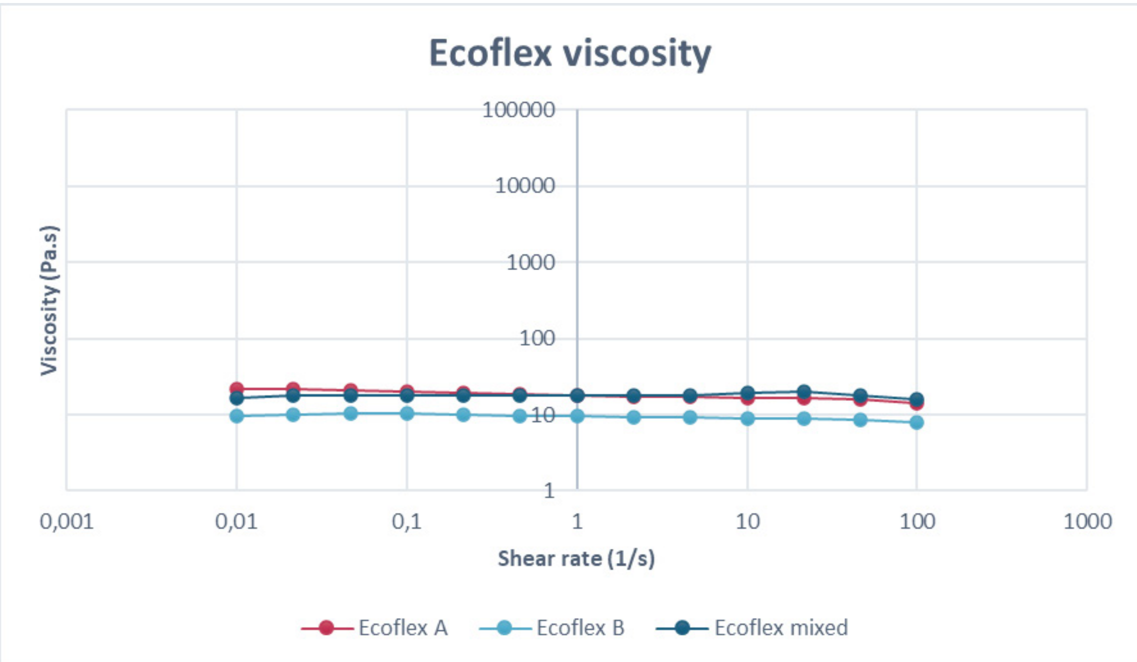


Figure C.2: *The viscosity test procedure settings in the program to operate the AR-G2*

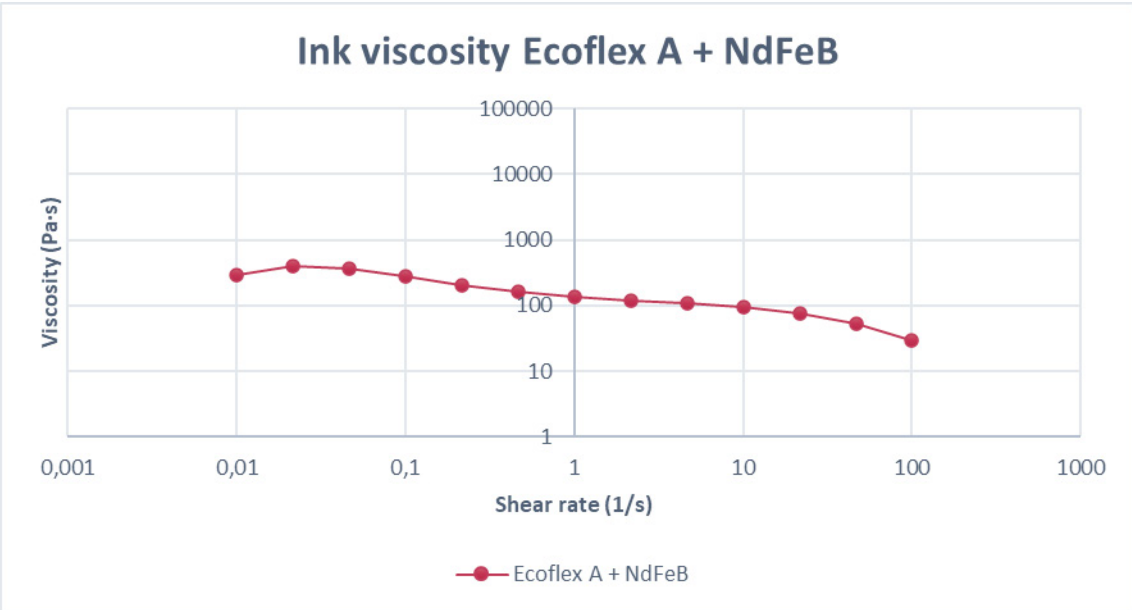
C.a Ecoflex

Ecoflex A		Ecoflex B		Ecoflex mixed	
Shear rate	Viscosity	Shear rate	Viscosity	Shear rate	Viscosity
1/s	Pa.s	1/s	Pa.s	1/s	Pa.s
0,0099842	21,5085	0,0099964	9,5051	0,00999588	16,7569
0,0215588	21,3265	0,021553	10,1727	0,0215412	17,9605
0,0464156	20,9441	0,0464153	10,3453	0,0464176	18,2222
0,100008	20,1678	0,100003	10,3135	0,100003	18,0837
0,215438	19,2068	0,215448	10,0917	0,215444	17,8717
0,464164	18,4069	0,464162	9,77395	0,46416	17,7275
1,00001	17,7917	1,00001	9,50413	1	17,6477
2,15444	17,352	2,15444	9,29109	2,15443	17,7146
4,6416	17,0706	4,64159	9,11759	4,64158	18,1371
10	16,8593	10	8,96313	9,99995	19,376
21,5443	16,4779	21,5443	8,77879	21,5443	20,0363
46,4159	15,6609	46,4159	8,47085	46,416	18,1115
100	14,2643	100	8,02627	100	15,7089



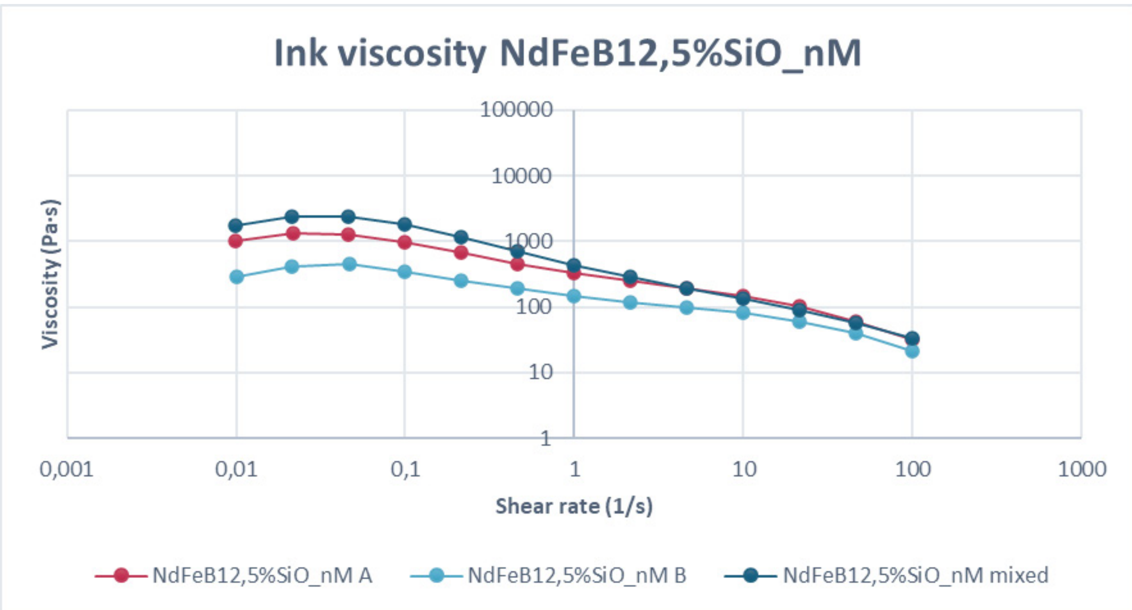
C.b Ecoflex A + NdFeB

Ecoflex A + NdFeB	
Shear rate	Viscosity
1/s	Pa.s
0,00996727	285,856
0,0215369	402,105
0,0464104	358,822
0,100004	277,721
0,21545	204,139
0,464169	159,836
1,00001	134,46
2,15443	118,759
4,64159	107,546
10	95,6025
21,5447	76,0283
46,4168	53,0751
100,001	29,6265



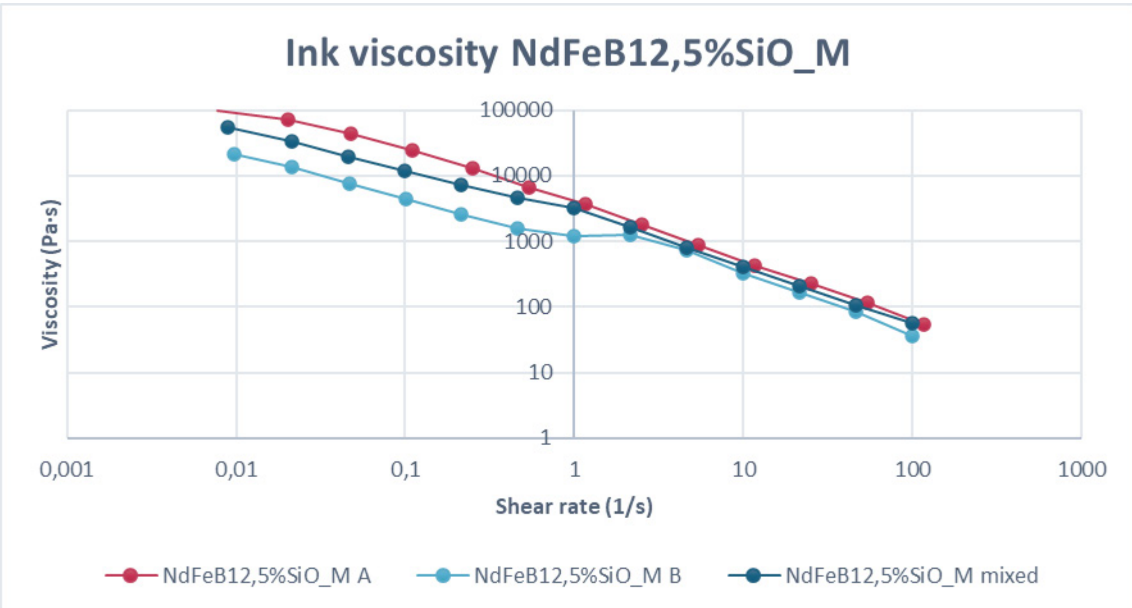
C.c NdFeB12,5%SiO_nM

NdFeB12,5%SiO_nM A		NdFeB12,5%SiO_nM B		NdFeB12,5%SiO_nM mixed	
Shear rate	Viscosity	Shear rate	Viscosity	Shear rate	Viscosity
1/s	Pa.s	1/s	Pa.s	1/s	Pa.s
0,00996338	1020,2	0,00997957	292,4	0,00994518	1742,54
0,0215144	1354,7	0,0214842	414,781	0,0214922	2417,26
0,046382	1271,84	0,046403	447,68	0,0463469	2419,7
0,0999915	979,228	0,100005	350,238	0,0999201	1843,56
0,215448	667,754	0,215451	252,017	0,215473	1152,94
0,464189	461,951	0,464167	188,86	0,464234	701,711
1,00004	336,011	1,00001	148,009	1,00012	441,195
2,15451	253,522	2,15445	118,954	2,15461	290,823
4,64168	196,955	4,64161	97,5095	4,64194	194,688
10,0006	147,983	10	81,304	10,0006	133,672
21,5461	100,972	21,5447	61,1546	21,545	91,022
46,4176	58,8953	46,4171	40,682	46,4169	58,3132
100,003	32,03	100,003	20,9851	100,002	32,8358



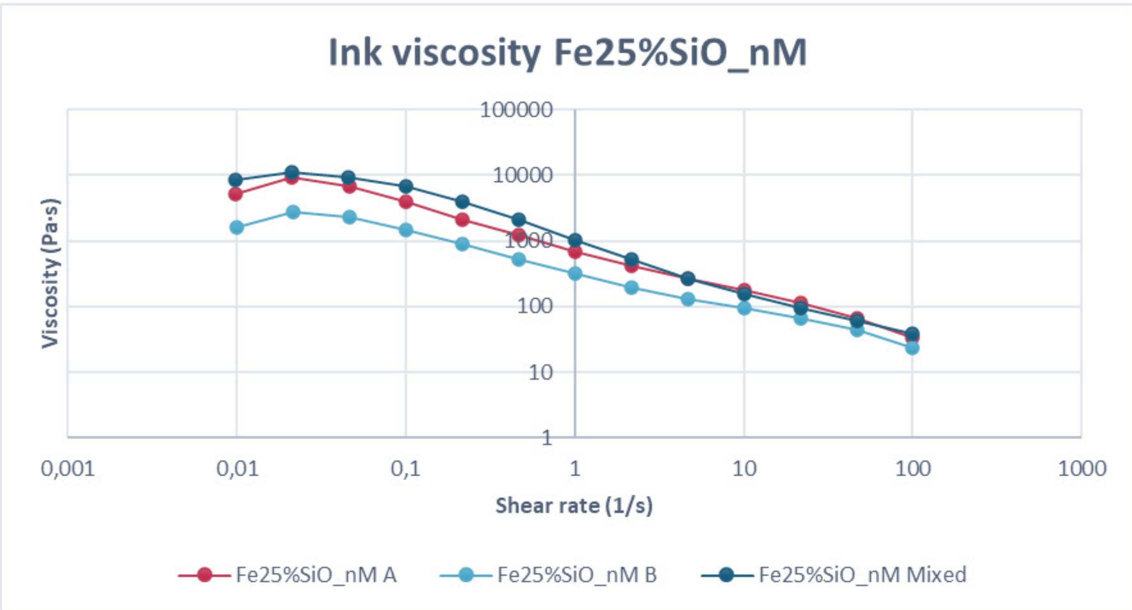
C.d NdFeB12,5%SiO_M

NdFeB12,5%SiO_M A		NdFeB12,5%SiO_M B		NdFeB12,5%SiO_M mixed	
Shear rate	Viscosity	Shear rate	Viscosity	Shear rate	Viscosity
1/s	Pa.s	1/s	Pa.s	1/s	Pa.s
0,00779437	100054	0,00970797	21440,3	0,00888853	55721
0,0204036	73250,5	0,0214211	13566,5	0,0211795	33172,3
0,0478829	44125,8	0,0464046	7745,66	0,0460027	19549,1
0,110894	24312,4	0,100117	4369,06	0,0996095	11771,4
0,251644	12867,3	0,21567	2528,87	0,215259	7163,27
0,543606	6547,48	0,464313	1593,59	0,46394	4589,16
1,16892	3626,8	0,999475	1222	0,999749	3181,25
2,51816	1825,91	2,15199	1260,07	2,15591	1674,22
5,42356	884,773	4,64405	721,689	4,64327	815,036
11,6814	436,751	10,0022	331,185	10,0016	410,947
25,1646	225,018	21,546	165,893	21,5472	209,283
54,2163	117,074	46,4181	85,1369	46,4186	106,534
116,797	53,22	100,004	35,6775	100	56,8328



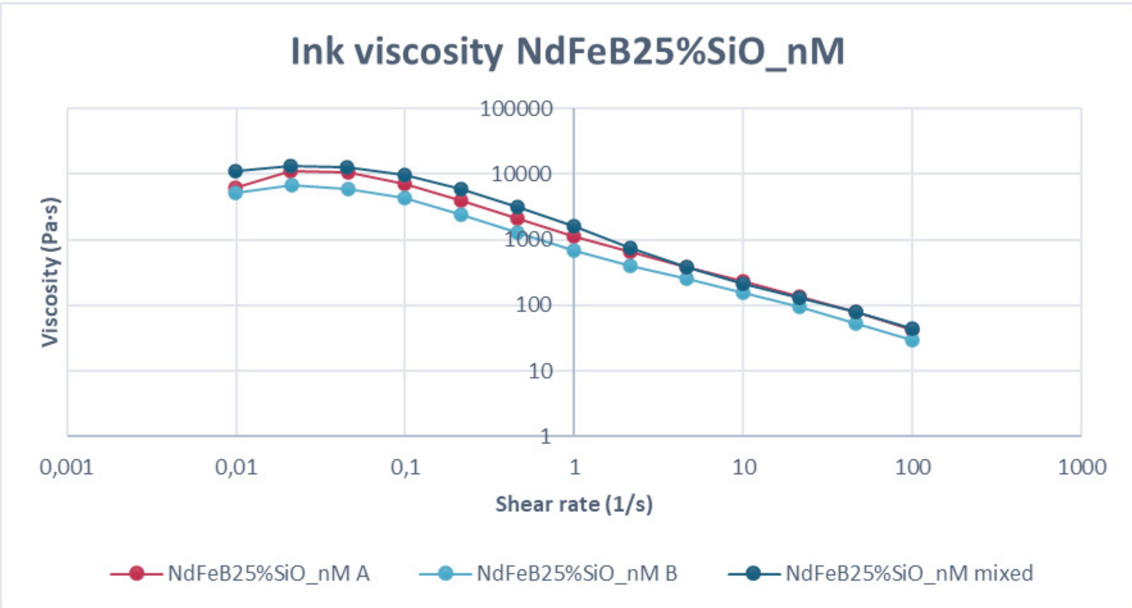
C.e Fe25%SiO_nM

Fe25%SiO_nM A		Fe25%SiO_nM B		Fe25%SiO_nM Mixed	
Shear rate	Viscosity	Shear rate	Viscosity	Shear rate	Viscosity
1/s	Pa.s	1/s	Pa.s	1/s	Pa.s
0,00981305	5276,7	0,0099043	1619,84	0,0098316	8663,14
0,0211749	9353,34	0,0214736	2802,46	0,0210365	11344,2
0,0463253	6711,69	0,0463857	2307,29	0,0459129	9454,12
0,100068	3880	0,0999958	1500,01	0,0995897	6725,39
0,215592	2155,37	0,215479	889,135	0,215539	3973,54
0,464366	1206,7	0,464219	524,978	0,464491	2077,88
1,00022	696,389	1,00009	316,652	1,00052	1045,08
2,15468	424,646	2,15455	198,062	2,1553	521,674
4,642	270,142	4,64167	132,153	4,64238	264,194
10,0006	176,663	10	94,4263	10,0006	154,613
21,546	113,934	21,5445	67,7309	21,5452	94,3737
46,417	66,2232	46,4168	43,9688	46,4161	59,8485
100,003	34,4666	100,002	23,9228	100,002	38,0127



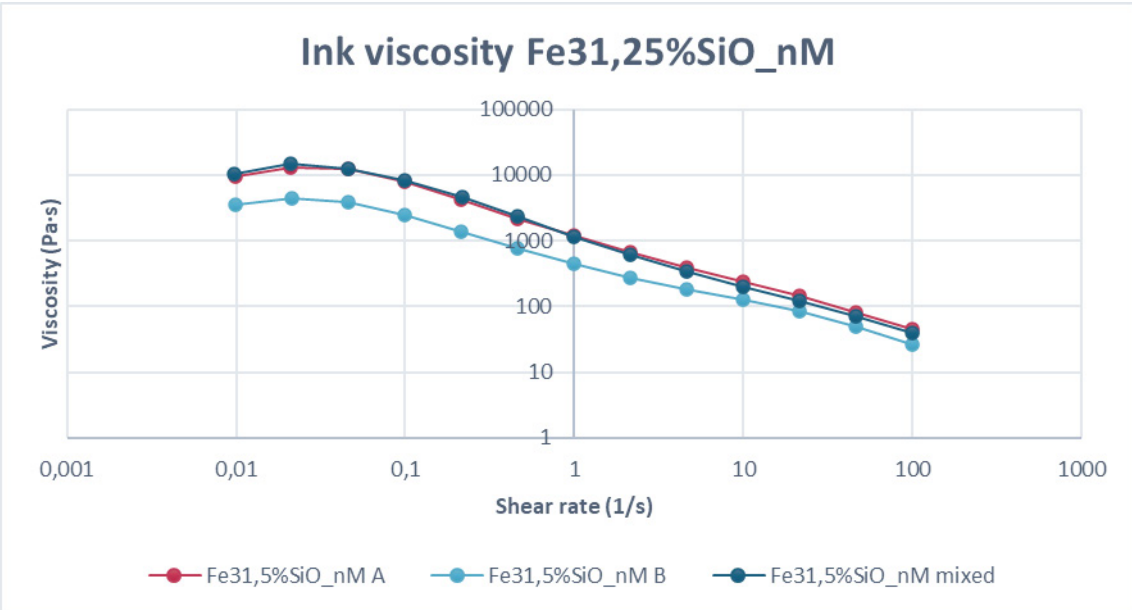
C.f NdFeB25%SiO_nM

NdFeB25%SiO_nM A		NdFeB25%SiO_nM B		NdFeB25%SiO_nM mixed	
Shear rate	Viscosity	Shear rate	Viscosity	Shear rate	Viscosity
1/s	Pa.s	1/s	Pa.s	1/s	Pa.s
0,00981857	6210,02	0,00989679	5081,99	0,00980512	10942
0,0208796	11209,5	0,0213882	6639,17	0,02108	13019,1
0,045828	10438,8	0,0460385	5916,07	0,0455391	12582,6
0,0997434	7081,33	0,0997941	4243,1	0,0993399	9819,51
0,215666	3998,17	0,215624	2413,17	0,215493	5999,23
0,464496	2081,78	0,464371	1268,82	0,464649	3192,33
1,00029	1120,17	1,00018	697,849	1,00101	1581,79
2,15473	646,247	2,15456	405,175	2,1559	749,819
4,64199	389,024	4,64175	250,756	4,64272	388,101
10,0009	237,588	10,0007	154,75	10,0011	215,997
21,547	137,852	21,5454	93,2996	21,5455	129,884
46,4169	79,49	46,4168	52,8802	46,4173	79,8741
100,001	42,6606	100,003	29,8538	100,002	44,4841



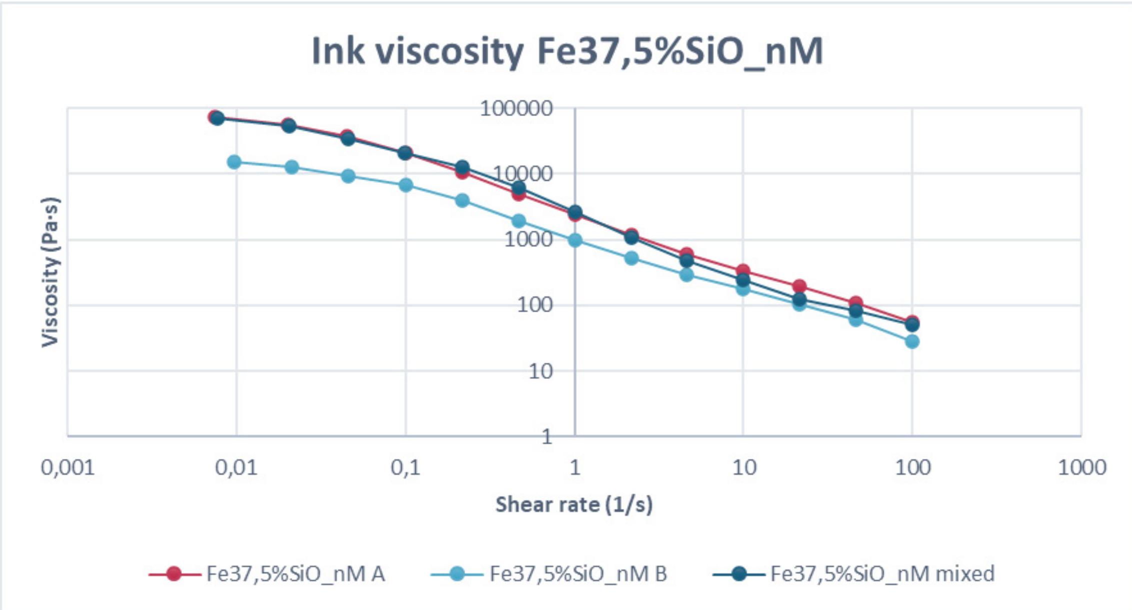
C.g Fe31,25%SiO_nM

Fe31,5%SiO_nM A		Fe31,5%SiO_nM B		Fe31,5%SiO_nM mixed	
Shear rate	Viscosity	Shear rate	Viscosity	Shear rate	Viscosity
1/s	Pa.s	1/s	Pa.s	1/s	Pa.s
0,00983061	9367,22	0,00991948	3494,71	0,00976268	10520,7
0,0207908	13287,5	0,0214651	4452,72	0,0208656	14804,6
0,0456158	12455	0,0462616	3866,22	0,0457696	12393,9
0,0999115	7916,92	0,0999873	2504,87	0,0996388	8410,04
0,215713	4238,6	0,215525	1387,79	0,21574	4593,72
0,464553	2203,81	0,464262	772,24	0,464573	2320,04
1,00041	1186,28	1,0001	447,966	1,00048	1170,66
2,15507	661,965	2,15454	273,528	2,15509	606,603
4,64183	393,625	4,64154	182,873	4,64209	334,985
10,0011	242,941	10,0002	125,962	10,0008	196,596
21,547	144,047	21,5446	84,1217	21,5461	119,087
46,4175	83,0505	46,4168	50,6168	46,4181	70,0189
100,002	45,7305	100,003	25,9769	99,9953	39,4141



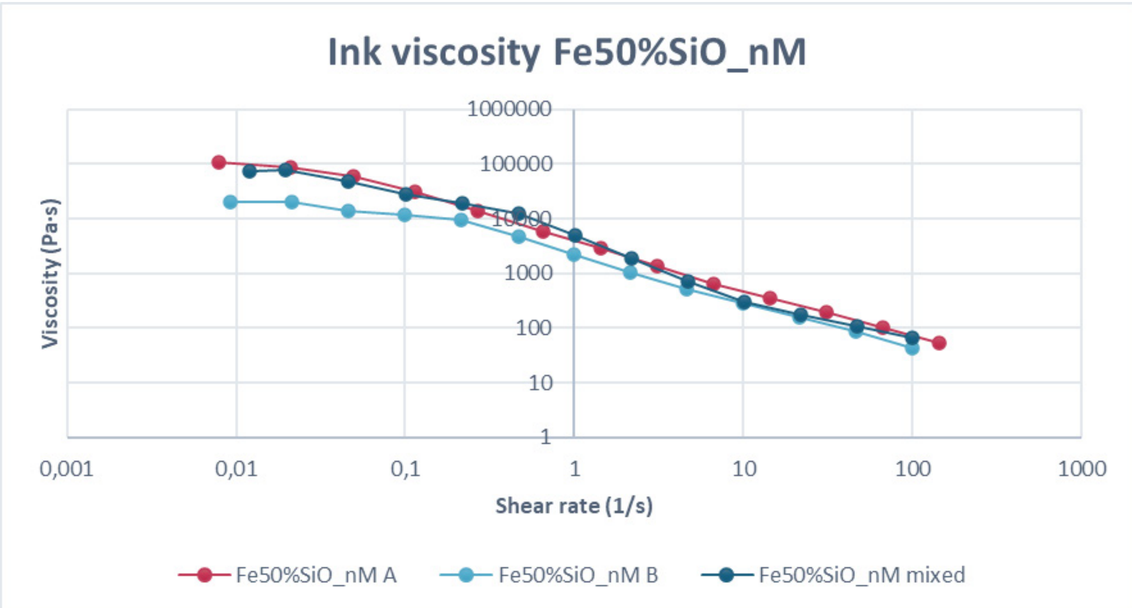
C.h Fe37,5%SiO_nM

Fe37,5%SiO_nM A		Fe37,5%SiO_nM B		Fe37,5%SiO_nM mixed	
Shear rate	Viscosity	Shear rate	Viscosity	Shear rate	Viscosity
1/s	Pa.s	1/s	Pa.s	1/s	Pa.s
0,00748685	72045,3	0,00971492	15449	0,00764364	70225,2
0,0202896	55834	0,0214024	12826,4	0,0204014	52651,7
0,045285	37040,6	0,0461031	9229,1	0,0455674	33503,2
0,0999698	21325,8	0,0996966	6647,83	0,0991483	20830,5
0,216583	10480,8	0,215674	3911,09	0,215119	12537,3
0,465251	4928,62	0,464569	1963,77	0,465929	6182,1
1,00102	2381,46	1,00035	988,508	1,00272	2643,17
2,15559	1161,3	2,15462	524,332	2,15669	1087,16
4,64278	608,777	4,6419	297,868	4,64378	484,714
10,0005	335,265	10,0005	175,043	10,0013	243,896
21,5468	195,077	21,5456	103,06	21,5458	125,478
46,4204	109,135	46,4168	59,7926	46,4157	84,007
100,003	56,6961	100,001	28,3473	100,004	51,6237



C.i Fe50%SiO_nM

Fe50%SiO_nM A		Fe50%SiO_nM B		Fe50%SiO_nM mixed	
Shear rate	Viscosity	Shear rate	Viscosity	Shear rate	Viscosity
1/s	Pa.s	1/s	Pa.s	1/s	Pa.s
0,00793335	109973	0,00927007	20433,8	0,0119457	72075,2
0,0210363	88391,2	0,0211742	20135	0,0196488	78024,9
0,0490445	58024,3	0,0459132	13897,1	0,0457595	47662,8
0,114476	30639,3	0,098342	11929,5	0,1005	28638,9
0,267424	13752,8	0,214336	9567,24	0,216023	19343,7
0,653264	5894,88	0,46576	4757,99	0,470352	12126,1
1,44148	2833,22	1,00088	2186,88	1,01515	4921,04
3,10499	1332,06	2,15538	1037,99	2,17697	1849,56
6,6866	649,918	4,64245	523,533	4,68306	708,719
14,4023	346,99	10,0002	283,217	10,0828	309,234
31,0286	194,91	21,5454	161,782	21,718	172,187
66,8489	105,234	46,4189	85,6019	46,7893	108,05
144,014	54,7908	100,005	44,1323	100,802	67,4496



D. Ordered materials

This is a list of the ordered materials for the magnetic soft material ingredients as well as ordered equipment like the tubes for the 3D printer, multiple permanent magnets and materials to show magnetic fields.

Ingredients

The list of all the used magnetic particles and fumed silica and where they were ordered. The Ecoflex was not ordered on a special website. That is why it does not have a link.

NdFeB particles (5 μm)
https://www.nanochemazone.com/product/neodymium-iron-boron-magnetic-powders/
These are the NdFeB particles that were ordered with a size of 5 μm. To order these a quote should be requested.

Iron carbonyl powder (5-9 μm)
https://www.sigmaaldrich.com/NL/en/product/aldrich/44890

SmCo particles
https://www.sigmaaldrich.com/NL/en/product/aldrich/339229?cm_sp=Insite-_-caSrpResults_srpRecs_srpModel_smco%20powder-_-srpRecs3-2

Fe3O4 particles (5 μm)
https://www.sigmaaldrich.com/NL/en/product/aldrich/310069?cm_sp=Insite-_-caSrpResults_srpRecs_srpModel_fe3o4%20powder-_-srpRecs3-1

Ecoflex 00-10

Fumed silica
https://www.sigmaaldrich.com/NL/en/product/aldrich/s5130?context=product

Tubes

Used to connect the syringes with the static mixing nozzle in the 3D printer system.

PVC tubes (Ø 3x6 mm)
https://www.arestho.nl/transparante-pvc-slang.html

Reinforced tubes (PN30 4 x 10mm) (not used)
https://www.arestho.nl/refitexx-cristallo-transparant.html

Magnets

All the magnets and magnetic detectors have been ordered at supermagnete.nl

Schijfmagneet Ø 35 mm, hoogte 5 mm (S-35-05-N)
https://www.supermagnete.nl/schijfmagneten-neodymium/schijfmagneet-35mm-5mm_S-35-05-N

Schijfmagneet Ø 35 mm, hoogte 20 mm (S-35-20-N)
https://www.supermagnete.nl/schijfmagneten-neodymium/schijfmagneet-35mm-20mm_S-35-20-N

Schijfmagneet Ø 60 mm, hoogte 5 mm (S-60-05-N)
https://www.supermagnete.nl/schijfmagneten-neodymium/schijfmagneet-60mm-5mm_S-60-05-N

Ringmagneet Ø 19,1/9,5 mm, hoogte 6,4 mm (R-19-09-06-N)
https://www.supermagnete.nl/ringmagneten-neodymium/ringmagneet-19.1mm-9.5mm-6.4mm_R-19-09-06-N

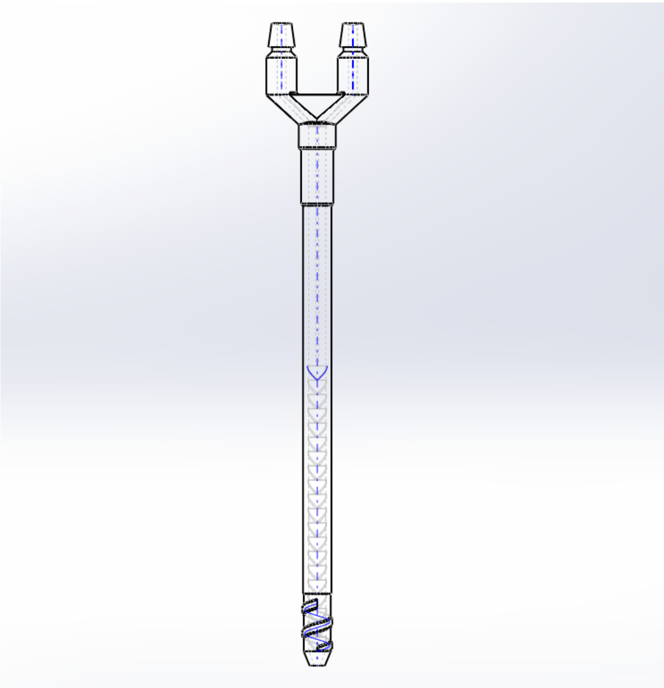
Ringmagneet Ø 60/20 mm, hoogte 10 mm (FE-R-60-20-10)
https://www.supermagnete.nl/ringmagneten-ferriet/ringmagneet-60mm-20mm-10mm_FE-R-60-20-10

Fluxdetector klein
https://www.supermagnete.nl/school-magneten/fluxdetector-klein_M-04

IJzervijlsel
https://www.supermagnete.nl/school-magneten/ijzervijlsel_M-22

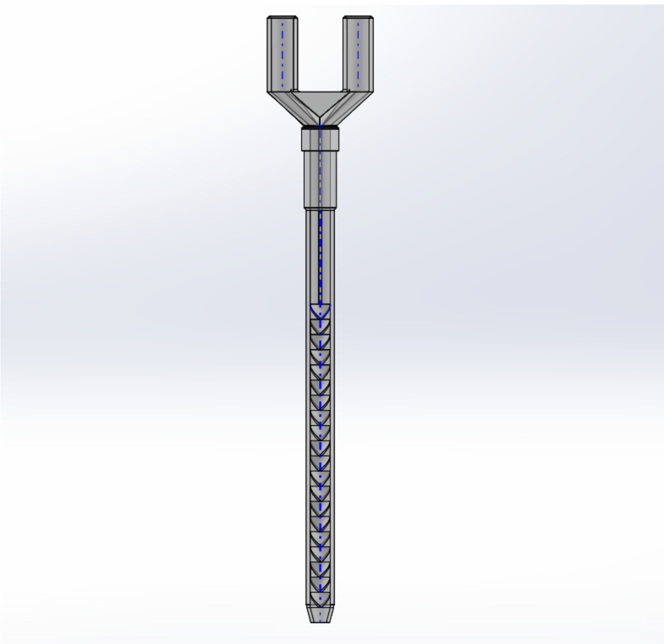
E. 3D printed parts overview and file names

This appendix has a list of the final 3D printed models for the 3D printer system and their names. The files are transferred to the chair and mentor of this project.



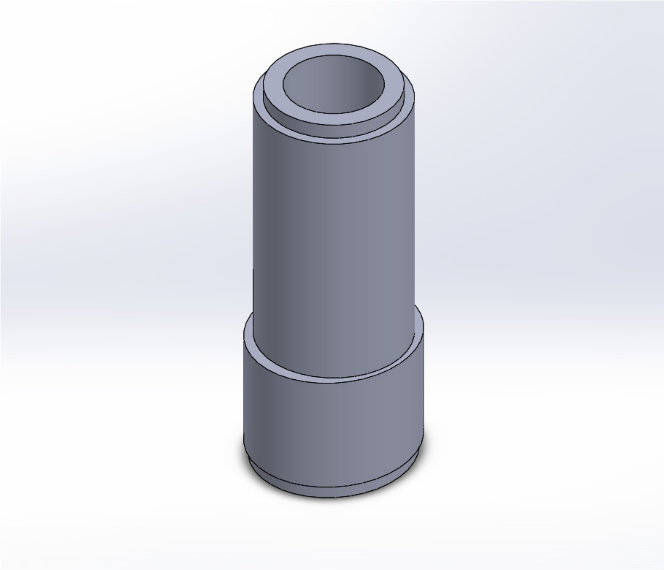
Extended static mixing nozzle with electromagnet connection

This is the static mixing nozzle that is used for printing, with connectors for the tubes and thread at the nozzle to connect the electromagnet to. It should be printed upright, standing on the two connectors for the tubes, without support. The support would otherwise clog the insides of the static mixing nozzle where the ink comes through. It should also be printed with a layer height of 0,1 mm. With that setting, the inner mixing mechanism is the smoothest, which provides less force needed in the system. They also should be printed one at a time, otherwise, the surface quality decreases significantly, which also adds up the required force to push the ink through the system.

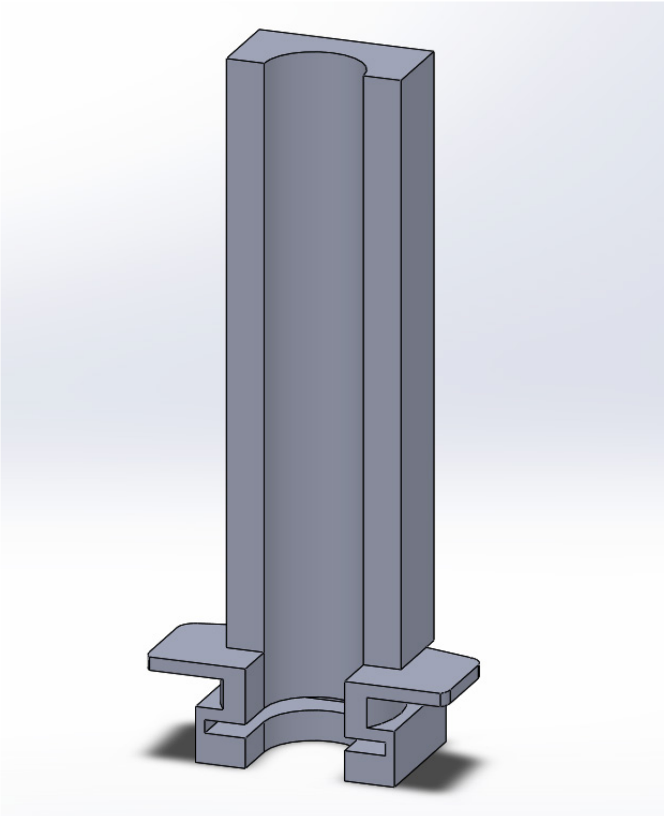


Static mixing nozzle for extrusion force tests

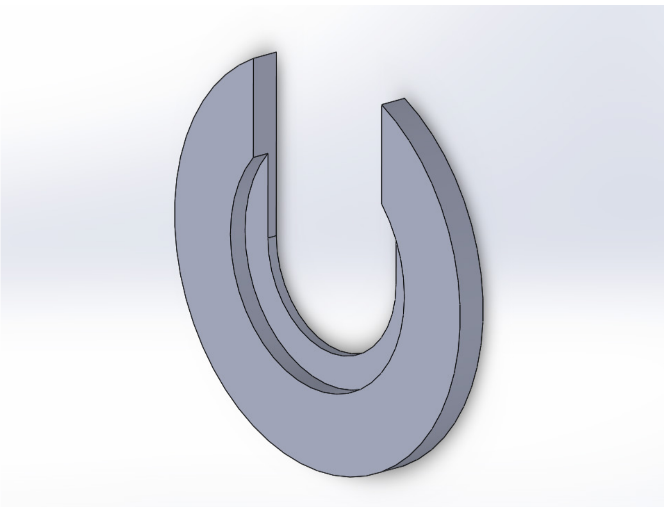
This is the static mixing nozzle in which two syringes can be inserted immediately and is used for the extrusion force measurements. For print instructions see the extended static mixing nozzle with electromagnet connection.



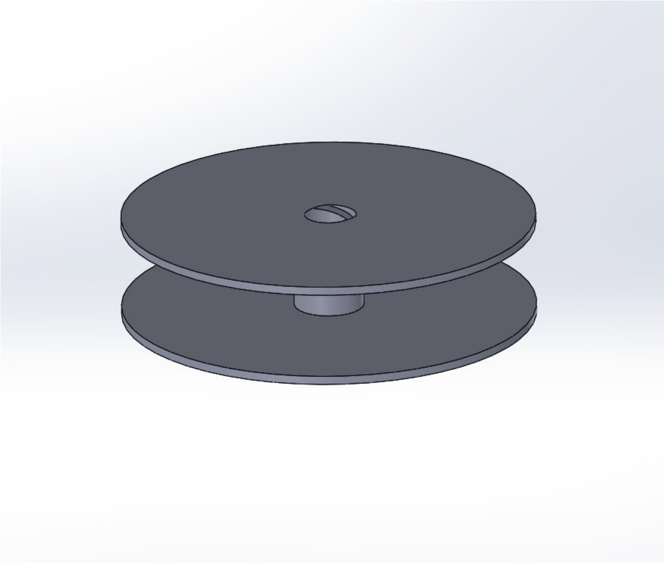
Stabilizer nozzle in printer
The stabilizer for the static mixing nozzle. Is a piece that is inserted in the print core of the 3D printer. It helps to limit the movement of the static mixing nozzle.



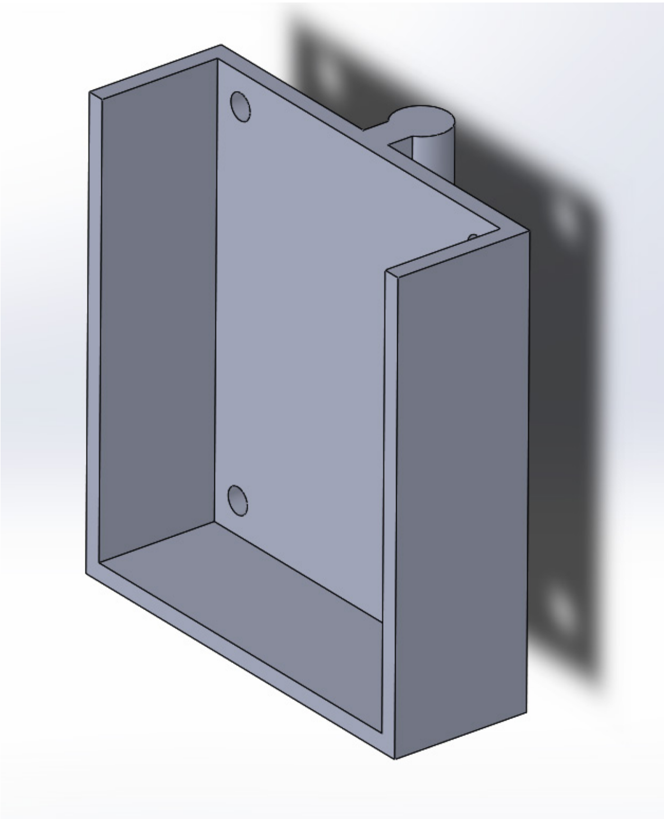
Syringe conversion 3D printer 60 mL to 10 mL
The part that is used to put a 10 mL syringe in the slot of a 60 mL syringe in the dual syringe extruder.



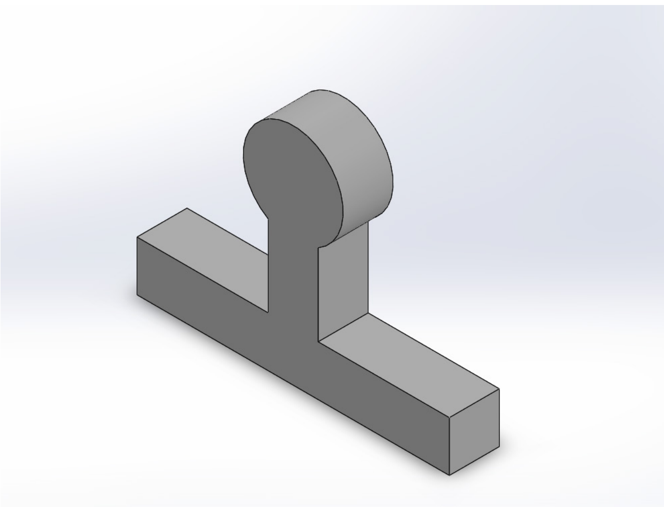
Piston conversion 3D printer 60 mL to 10 mL
This is used to make the end circle of the 10 mL syringe as big as the circle at the end of the piston of a 60 mL syringe. This is used to secure the red clips over the ends of the pistons.



Electromagnet coil with thread
This is the spindle on which the coil is winded. It has a thread in the inner opening to screw it to the static mixing nozzle. This design right now should be printed with support, to support one of the spindles end stops. To print it the overhang angle for the support should be adjusted so that there is no support generated inside the thread. The thread is, as the tread on the static mixing nozzle, designed with an angle of 45° so that it can be printed without support. This part should also be printed with a layer height of 0,1 mm so that the tread can be printed as smooth as possible.



H-bridge holder
The H-bridge holder can snap the H-bridge to the cable clips of the 3D printer. This one was designed for another H-bridge than the one that was connected to the electromagnet at the end of the project. In case of continuing this project, the size of this part should be altered. This one is designed to be printed so that the cylinder of the connection part stands upright.



Connector – Concept
This is the file with the tiny connector from the concept for the demonstrator that was developed at the end.

F. 3D printer setup

This Appendix shows the 3D printer system and how everything should be set up and connected. The first part is about the system without the electromagnet because that worked during this project. At the end of this section, the theoretical connection of the electromagnet to the system will be presented.

3D printer setup cable connections

For the 3D printer setup connections without the electromagnet see figure F.1. The system exists out of four devices, a computer, router, 3D printer (Ultimaker 3) and the dual syringe extruder. All these devices need power and are therefore plugged in.

The computer is used to send G-code to the 3D printer. This is done by connecting both the computer and the 3D printer to the same router with ethernet cables. The router does not have to be connected to the internet but is now used as a communication device.

The dual syringe extruder is connected to the Ultimaker 3 with a USB cable. The dual syringe printer is controlled by the 3D printer, which sends instructions through the USB cable. When turning on the system, first the 3D printer has to be turned on and started, before the dual syringe printer is started. Otherwise, the 3D printer will not recognize the dual syringe extruder.

From the dual syringe extruder also the tubes do run that connect the syringes to the static mixing nozzle in the 3D printer.

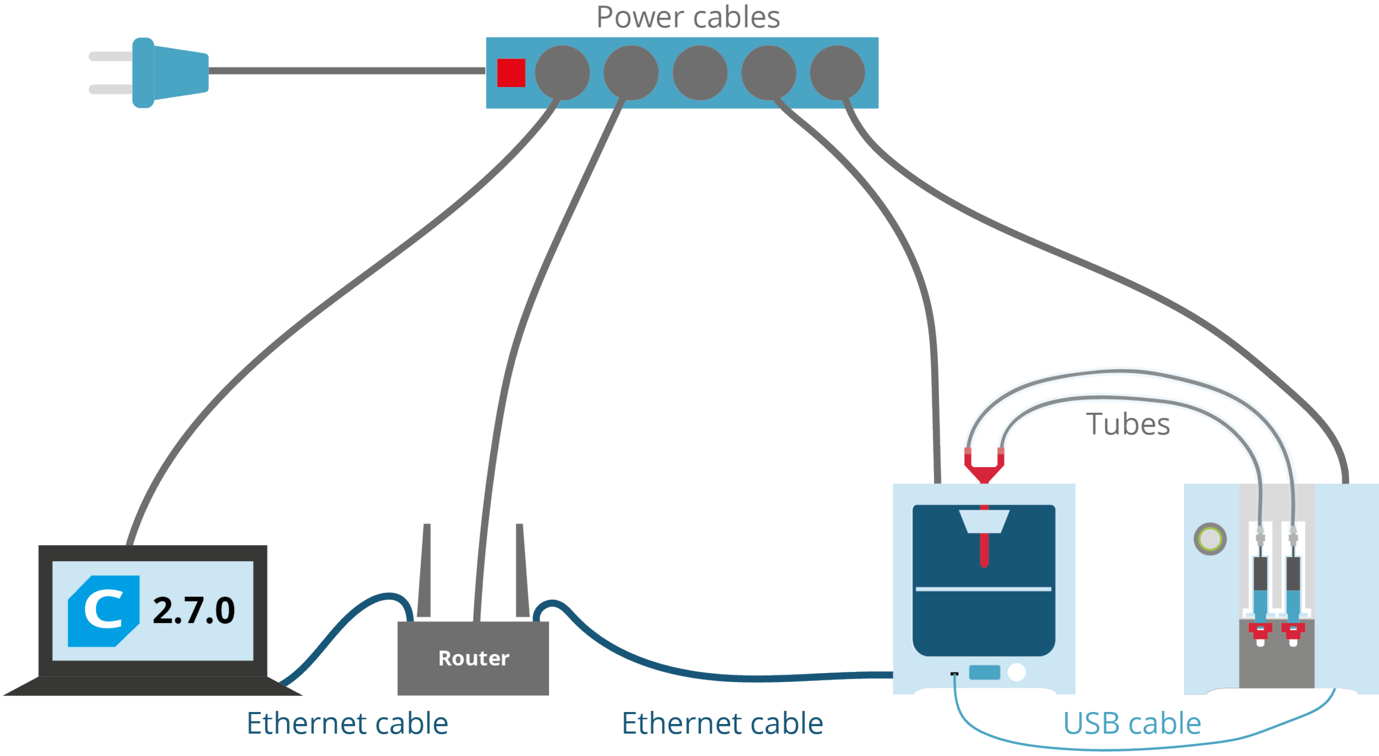


Figure F.1: *The 3D printer set up with all the cable and tube connections*

Dual syringe extruder; Installing syringes

This section will walk through the physical setup of the ink extrusion system.

First, the ink guiding system should be assembled (figure F.2):

- Connect the Luer lock connection to the syringe (2x)
- Connect the Luer lock connection to the tube (2x)
- Connect the other side of the tube to the static mixing nozzle (2x)
- Click the 10 mL syringe in the conversion piece (white) (2x)

Figure F.2: *The ink guiding system: conversion pieces, 10 mL syringes, Luer lock connections, tubes, static mixing nozzle*

When that is done, the system can be put into the dual syringe extruder (figures F.3 and F.4):

- Turn the dual syringe extruder on (the button is under the device), the button to move the plates should be blinking green
- Press the button for a few seconds, when it is released the two plates that will push against the pistons of the syringes should move down
- Put the conversion pieces with the syringes in the slots for the 60 mL syringes
- Press the button again for a few seconds. When it is released it should bring the plates upwards until they touch the pistons. The button should stop blinking and shine a solid green
- Then the enlargers of the pistons should be put over the pistons. These are also used to fit the 10 mL syringes in the system
- Over those, a red cap should be placed. These help to pull the pistons down when a negative value is placed in the G-code

Now the dual syringe printer is prepared for printing.

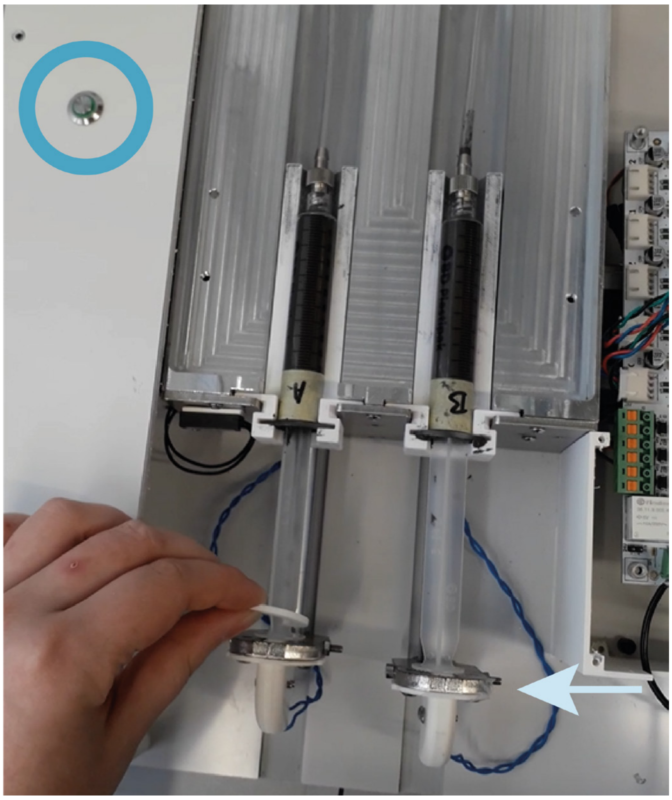


Figure F.3: *The button in the circle moves the plates up and down and shows the light on the status of the plates. One of the plates is pointed to with an arrow*

The static mixing nozzle must be placed into the empty slot of the printer head. It is put through an empty printer core (figure F.7). The thicker white piece that can be seen in picture F.6 is a stabilizer that is put inside the empty printer core. It has a smaller diameter than the printer core to limit the static mixing nozzle's movement during printing.

To get everything out of the extruder, first, the red caps over the pistons should be removed. Then the syringes should be taken out. After that, the plates can be brought down again by pressing the button. That cannot be done before, because those plates contain buttons that sense when the system is prepared for extrusion.

To remove the static mixing nozzle from the printer head the ring in figure F.5 should be pressed down. This releases the grip from the static mixing nozzle. The static mixing nozzle will not break so soon when this ring is pressed down.

Figure F.4: *The end of the piston with the plate against it, the white enlargement piece on top of it and the red cap over it*

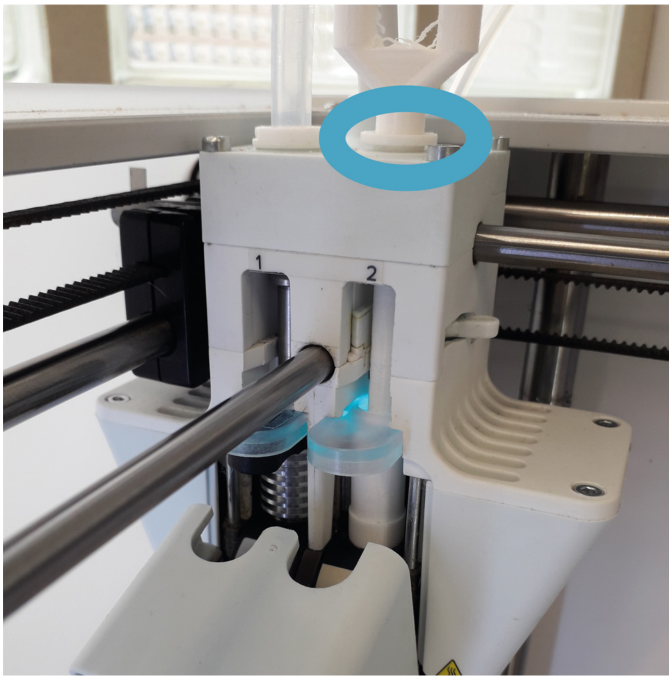


Figure F.5: *The ring that should be pressed down when the static mixing nozzle is removed*

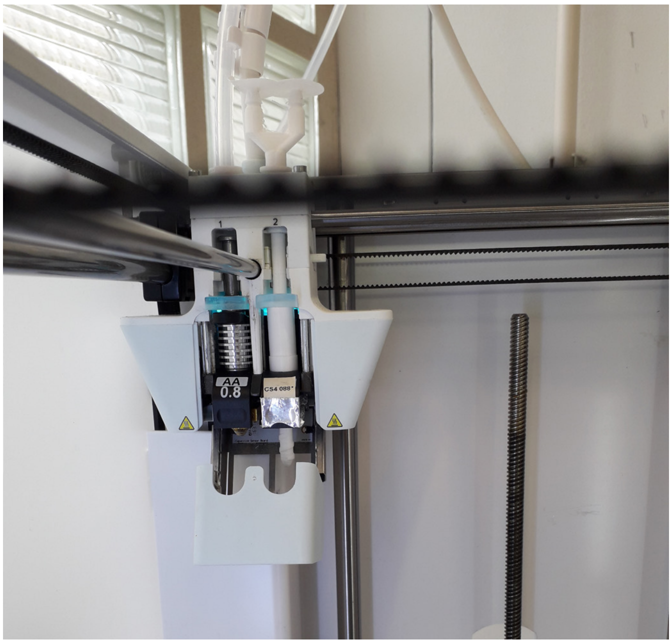


Figure F.6: *Printer core inside the printer head, with stabilizer and static mixing nozzle*

Figure F.7: *Empty printer core*

Cura 2.7.0; connection printer and computer

Cura 2.7.0 has to be used to send G-code from the computer to the 3D printer.

In settings --> printer --> manage printers, the 3D printer can be found to connect to the computer (figure F.8). When the computer is once connected manually, it will connect to the printer automatically next time when the ethernet cable is plugged in.

When the 3D printer is connected, a G-code can be loaded, by clicking on the folder in the upper left corner. When it is selected the button “Save to file” will change into “Print over network”. This button should be clicked to start the 3D printing process.

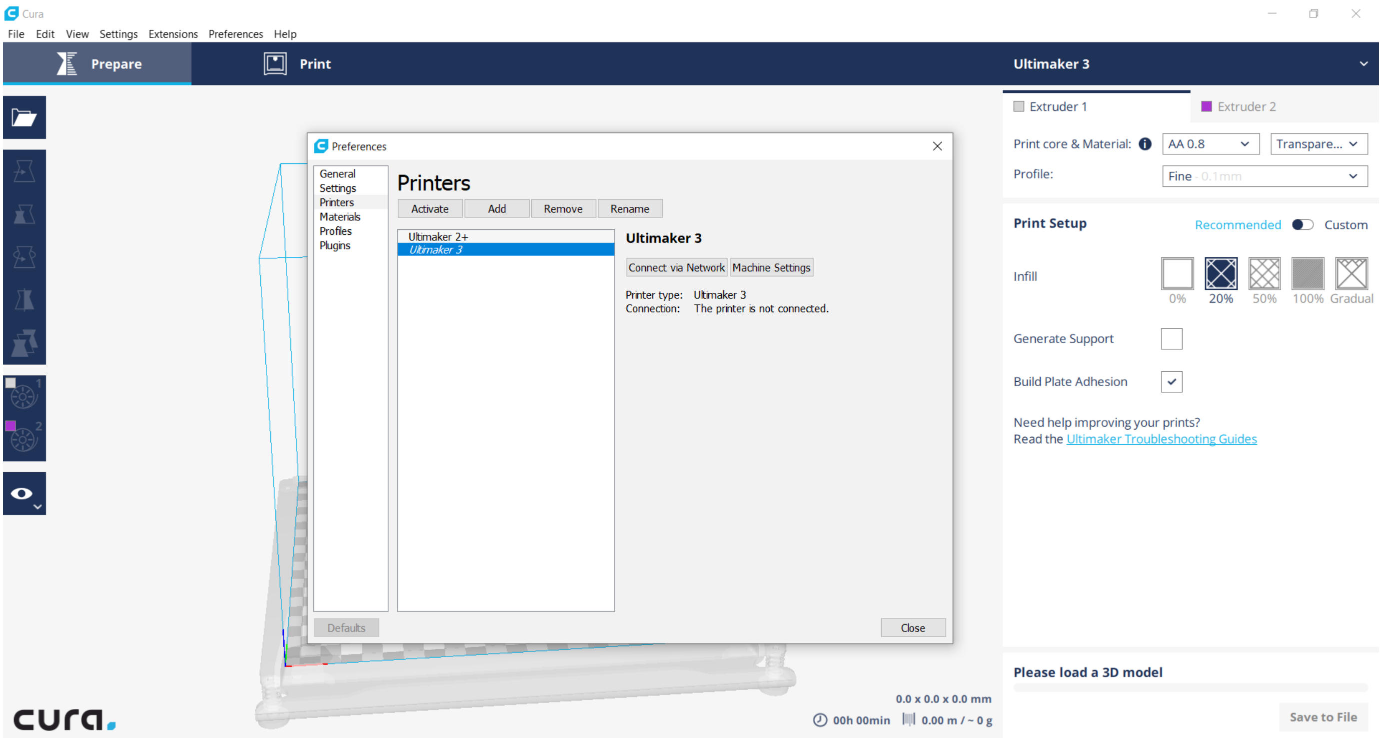


Figure F.8: The main screen of Cura 2.7.0 with the popup screen of “manage printers” to connect to the 3D printer

Connection Electromagnet

To connect the electromagnet to the 3D printer system that was explained previously, the electromagnet with a magnetic shield should be turned on the static mixing nozzle that is already placed in the 3D printer.

In figure F.9 the electrical connections of the electromagnet can be seen. The electromagnet, 3D printer and power supply are all connected to an H-bridge. The red wires are connected to the higher power supply that provides the electromagnet with enough power to create a magnetic field of 50 mT. The blue wires are connected to the ground. At the last, there are the green and yellow wires that conduct 5 volts from the 3D printer to communicate the settings for the electromagnet. One of them communicates if the electromagnet should be turned on or off. The other determines the direction of the magnetic field by turning the direction of the current running through the electromagnet.

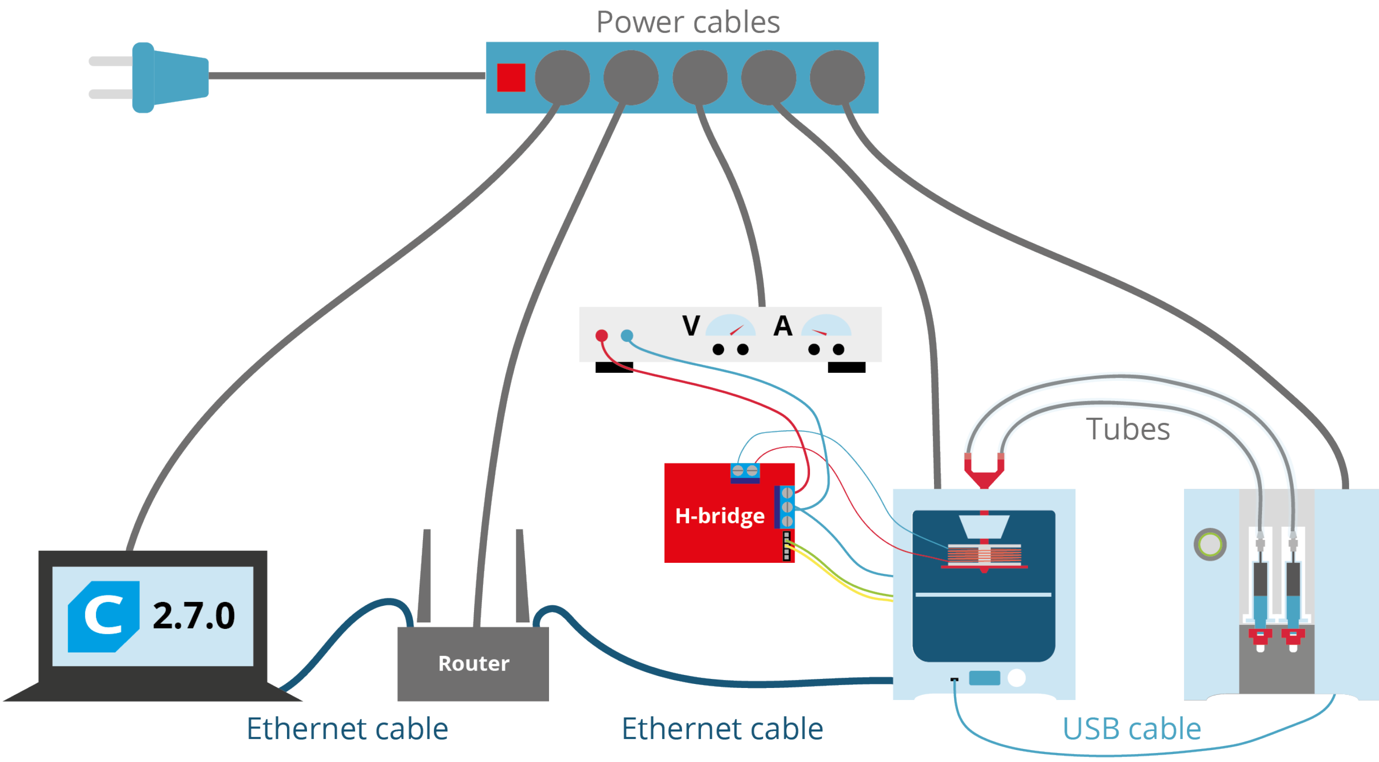


Figure F.9: *The 3D printer set up with all the cable and tube connections including the electromagnet*

G. G-codes

This Appendix contains the two G-codes that were used to find the values for the volume and speed of the extrusion, as well as the G-code with which multiple pins of the 3D printer were tested on their output to connect the electromagnet. The G-codes are built upon existing G-codes. The bold parts are the parts that were modified by me for the functions of the tests. The existing G-code around the bold part make the 3D printer prepare and finish the print. The G-code commands of the pin testing code, including the already existing code, are all written out and can be found after the code used in this project. For the explanations of the different G-code commands, the RepRap website was used (RepRap, 2021).

G.a Measuring volume and speed G-code

This code was used to measure the volume and the speed of the A, B and F parameters. With A and B the distance that the pistons are moved is controlled. Depending on which size syringe is used, a different A and B value is needed to extrude 1 mL of ink. In the case of the 10 mL syringes, this was 6 mm. However, for the 60 mL syringes which have a larger diameter, less distance might be needed. If the value in the front of A and B are the same, the distance that the pistons are moved should also be the same, because they have the same thread and motors to be moved. When A and B are positive (A89 B89), the pistons move upward and push ink out of the syringes. Negative A and B values can also be used (A-89 B-89). This will move the pistons downwards, pulling them out of the syringes again. The A and B values were not tested with decimals. A and B can also move different distances in the same command (A89 B45). That was used in later 3D printer tests when it was wanted to extrude components A and B separately up to the static mixing nozzle before printing.

The value of F determines the time over which the movement of A and B takes place. The F value does not work with decimals (see Appendix B.e.1).

In the bold command, G1 F1 A89 B89, the speed is controlled with the F1 command and the distance with A89 and B89. This line is enclosed by two lines with the command M400, which means, wait until all the tasks above are ready. This is needed when there is also a movement added to this G-code. Before the start of the next command, both the movement of the printer head and the extrusion of the ink should be ready. Therefore the M400 command is used.

To measure and find different values for the A, B and F, the numbers behind the letters were changed regularly.

```
;START_OF_HEADER
;HEADER_VERSION:0.1
;FLAVOR:Griffin
;GENERATOR.NAME:Cura_SteamEngine
;GENERATOR.VERSION:2.3.1
;GENERATOR.BUILD_DATE:2016-11-04
;TARGET_MACHINE.NAME:Ultimaker 3
;EXTRUDER_TRAIN.0.INITIAL_TEMPERATURE:60
;EXTRUDER_TRAIN.0.MATERIAL.VOLUME_USED:5429
;EXTRUDER_TRAIN.0.MATERIAL.GUID:506c9f0d-e3aa-4bd4-b2d2-23e2425b1aa9
;EXTRUDER_TRAIN.0.NOZZLE.DIAMETER:0.4
;BUILD_PLATE.INITIAL_TEMPERATURE:32
;PRINT.TIME:2718
;PRINT.SIZE.MIN.X:0
;PRINT.SIZE.MIN.Y:0
;PRINT.SIZE.MIN.Z:0
;PRINT.SIZE.MAX.X:215
;PRINT.SIZE.MAX.Y:215
;PRINT.SIZE.MAX.Z:200
;END_OF_HEADER
;Generated with Cura_SteamEngine 2.3.1

G0 F10000 X178 Y6 Z190
M107
G0 F10000 X100 Y100 Z190
M84
M400
G1 F1 A89 B89
M400
G4 P1000
M400
M106

M104 S0
M104 T1 S0
;End of Gcode
;SETTING_3 {"global_quality": "[general]\\nversion = 2\\nname = empty\\ndefiniti
;SETTING_3 on = ultimaker3\\n\\n[metadata]\\ntype = quality_changes\\nquality_ty
;SETTING_3 pe = draft\\n\\n[values]\\n\\n", "extruder_quality": ["[general]\\nve
;SETTING_3 rsion = 2\\nname = empty\\ndefinition = ultimaker3\\n\\n[metadata]\\n
;SETTING_3 type = quality_changes\\nextruder = ultimaker3_extruder_left\\nqualit
;SETTING_3 y_type = draft\\n\\n[values]\\nbrim_width = 0\\n\\n", "[general]\\nve
;SETTING_3 rsion = 2\\nname = empty\\ndefinition = ultimaker3\\n\\n[metadata]\\n
;SETTING_3 type = quality_changes\\nextruder = ultimaker3_extruder_right\\nquali
;SETTING_3 ty_type = draft\\n\\n[values]\\n\\n"]}
```

G.b Checking 3D printer pins output

This G-code was used to check if the pins at the printer board could be turned on. If an output of around five Volts was measured, the electromagnet could be connected (see Appendix B.e.2) the code of M42 P24 S255, would have been used to turn on or off, or switch the magnetic field of the electromagnet.

M42 is the code that is used to turn on a general-purpose pin (RepRap, 2021), on the board of a 3D printer. The P... tells the printer which pin needs to be controlled. The S0...255 stand respectively for an output of 0 to 5 Volts from the pin. In this G-code, only 255 is used to check if a pin could be turned on.

In the G-code, there is also an added line, G1 F120 X10 Y0. This is a general movement command which is used to move the printer head while extruding filament (RepRap, 2021). The X and Y are the coordinates the printer head has to move to and F is the speed in mm/min. Before the movement is started, the pin is turned on to be sure that it is supposed to be on when the movement of the printer head starts. This line is added in to give the printer some time over which the pin has to be turned on. Otherwise, the printer is finished printing immediately after putting on the pin and there is no time left to measure. There are two movements between the M400 waiting commands as explained in the other code. There are two movements to hear the 3D printer switch direction, to know when the test is almost finished.

```
;START_OF_HEADER
;HEADER_VERSION:0.1
;FLAVOR:Griffin
;GENERATOR.NAME:Cura_SteamEngine
;GENERATOR.VERSION:2.3.1
;GENERATOR.BUILD_DATE:2016-11-04
;TARGET_MACHINE.NAME:Ultimaker 3
;EXTRUDER_TRAIN.0.INITIAL_TEMPERATURE:60
;EXTRUDER_TRAIN.0.MATERIAL.VOLUME_USED:5429
;EXTRUDER_TRAIN.0.MATERIAL.GUID:506c9f0d-e3aa-4bd4-b2d2-23e2425b1aa9
;EXTRUDER_TRAIN.0.NOZZLE.DIAMETER:0.4
;BUILD_PLATE.INITIAL_TEMPERATURE:32
;PRINT.TIME:2718
;PRINT.SIZE.MIN.X:0
;PRINT.SIZE.MIN.Y:0
;PRINT.SIZE.MIN.Z:0
;PRINT.SIZE.MAX.X:215
;PRINT.SIZE.MAX.Y:215
;PRINT.SIZE.MAX.Z:200
;END_OF_HEADER
;Generated with Cura_SteamEngine 2.3.1

G0 F10000 X178 Y6 Z170
M107
G0 F10000 X100 Y100 Z170
M84
M400
M42 P24 S255
G1 F120 X0 Y0
M400
M42 P24 S255
G1 F120 X10 Y0
M400
G4 P1000
M400
M106

M104 S0
M104 T1 S0
;End of Gcode
;SETTING_3 {“global_quality”: “[general]\\nversion = 2\\nname = empty\\ndefiniti
;SETTING_3 on = ultimaker3\\n\\n[metadata]\\ntype = quality_changes\\nquality_ty
;SETTING_3 pe = draft\\n\\n[values]\\n\\n”, “extruder_quality”: [“[general]\\nve
;SETTING_3 rsion = 2\\nname = empty\\ndefinition = ultimaker3\\n\\n[metadata]\\n
;SETTING_3 type = quality_changes\\nextruder = ultimaker3_extruder_left\\nqualit
;SETTING_3 y_type = draft\\n\\n[values]\\nbrim_width = 0\\n\\n”, “[general]\\nve
;SETTING_3 rsion = 2\\nname = empty\\ndefinition = ultimaker3\\n\\n[metadata]\\n
;SETTING_3 type = quality_changes\\nextruder = ultimaker3_extruder_right\\nquali
;SETTING_3 ty_type = draft\\n\\n[values]\\n\\n”]}
```

G0 F10000 X178 Y6 Z170: G0 is linear movement without extrusion, F is the speed of the printer head (mm/min), XYZ the coordinates were to move to
M107: Turns of the fan of the 3D printer
G0 F10000 X100 Y100 Z170: G0 is linear movement without extrusion, F is the speed of the printer head (mm/min), XYZ the coordinates were to move to
M84: "Stop the idle hold on all axis and the extruder. In some cases, the idle hold causes annoying noises, which can be stopped by disabling the hold. Be aware that by disabling idle hold during printing, you will get quality issues. This is recommended only in between or after print jobs." (RepRap, 2021)
M400: Waits until all previous commands are completed
M42 P24 S255: M42 to turn on a general-purpose pin. P… is the pin that you control and S is the output given to the pin going from 0 to 255
G1 F120 X0 Y0: G1 is a linear movement with extrusion, F is the speed of the printer head (mm/min), XYZ the coordinates where to move to.
M400: Waits until all previous commands are completed
M42 P24 S255: M42 to turn on a general-purpose pin. P… is the pin that you control and S is the output given to the pin going from 0 to 255
G1 F120 X10 Y0: G1 is a linear movement with extrusion, F is the speed of the printer head (mm/MIN), XYZ the coordinates where to move to.
G4 P1000: G is waiting for or a delay, P is the number of milliseconds the printer has to wait
M400: Waits until all previous commands are completed
M106: puts the fan on

M104 S0
M104 T1 S0: is used to set the temperature of the extruder. The number after the 'S' is the temperature to which the temperature has to be set. In this case, it is put off. The 'S' is in degrees of Celsius.

M400 is added multiple times in the G-code. This is done to make sure that when also the extrusion of the ink is included in the G-code, it finishes both the movement of the printer head as well as the extrusion of the ink.

G.c Example command block for working 3D printer system with electromagnet

During this project, it was not accomplished to complete the entire extrusion and electromagnet system. If the system works, a block of G-code commands to print a single line with one magnetic field direction should look like the code in the blue block below. First, with M42 the pin is turned on, which turns on the electromagnet. Then M42 is used again to control another pin, with which the direction of the magnetic field is determined. 255 is used for one direction and 0 for the other. Then the extrusion of the ink from the syringes is turned on with the G1 F1 A20 B20. Finally, also the movement is started with the G1 F30 X10 Y0 Z1. The two G1 commands should be adjusted to each other with the values measured in the volume and speed tests of the 3D printer in combination with how much ink should be extruded. The numbers in this block have not been calculated and are only there to show the structure of the G-code. The block is ended with M400 which makes sure that those four commands are ready before starting the next movement.

```
M400
M42 P50 S255
M42 P52 S255
G1 F1 A20 B20
G1 F30 X10 Y0 Z1
M400
```

H. Electromagnet coil design

The electromagnets coil was designed by my mentor Andres Hunt. It was designed so that it could reach the magnetic field strength of 50 mT. That was the same field strength that MIT used (Y. Kim et al., 2018). The design dimensions can be seen in figure H.1. It has a magnetic field strength in the core of 64,9 mT and 50 mT at the edge. It is made with 300 to 350 metres of 0,2 diameter Lacquered copper wire that is near the AWG32 rating. To be able to use only one power supply, the coil was made of four different spun coils they were connected as in figure H.2. It is intended to be operated with a current of 0,3 A and a maximum voltage of 27.

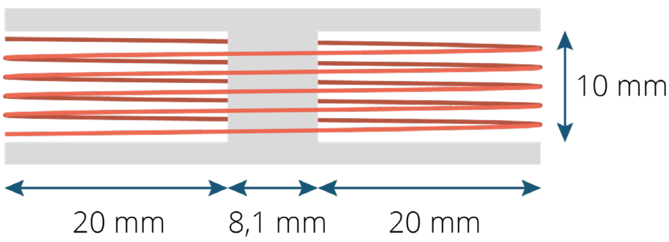


Figure H.1: *The dimensions of the electromagnet coil*

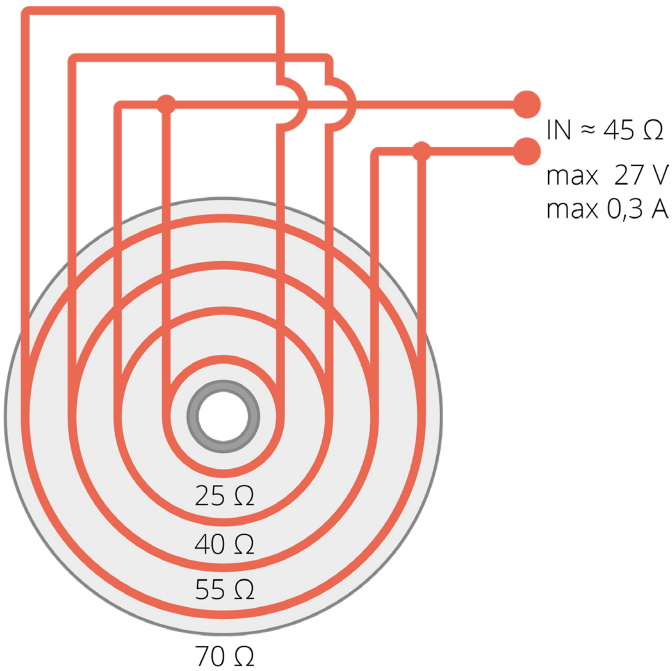
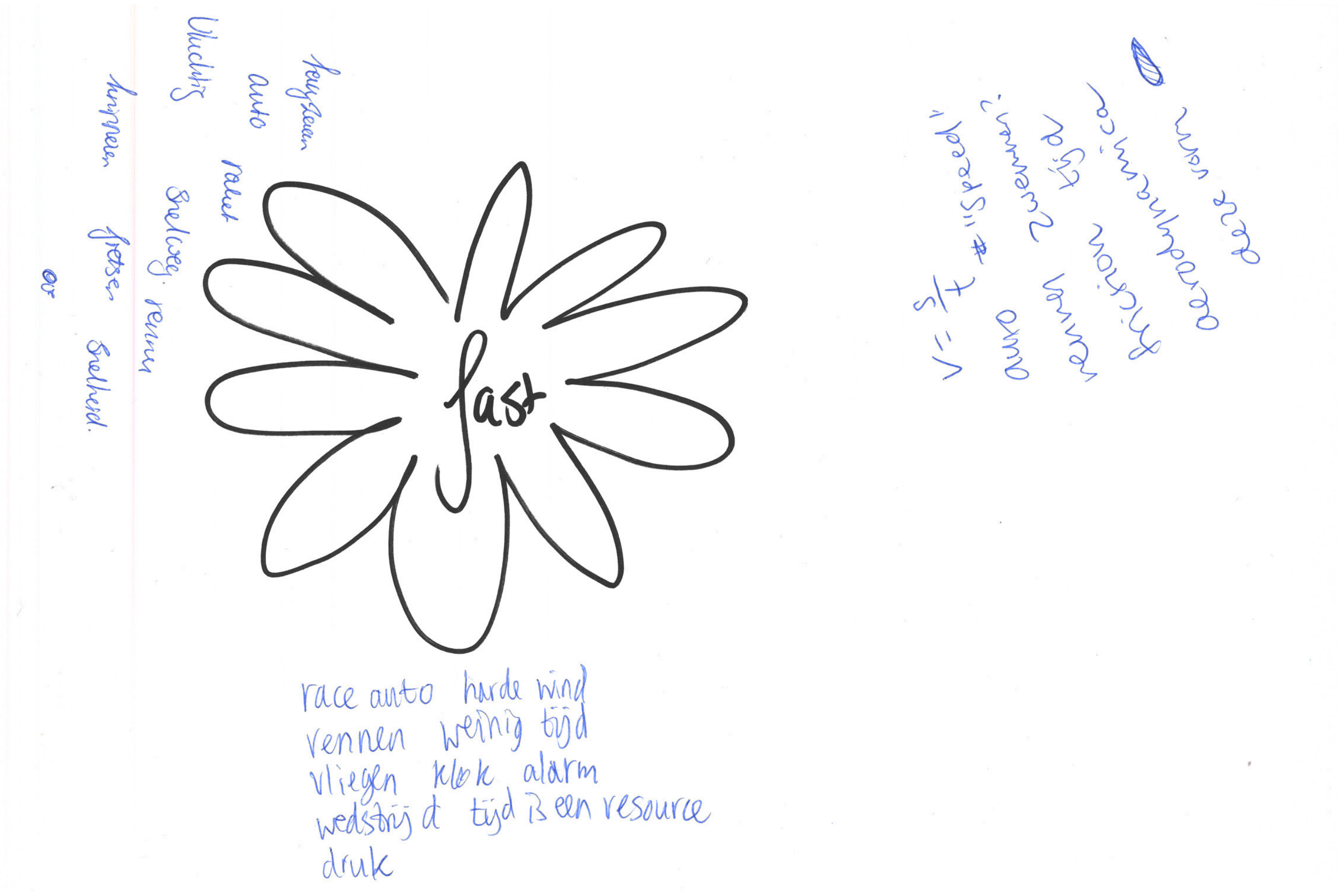


Figure H.2: *The connections of the four spun coils of the electromagnet*

I. Creative session

This attachment has the lists of words and the C-box that were generated during the creative session.

I.a Flower association: Fast

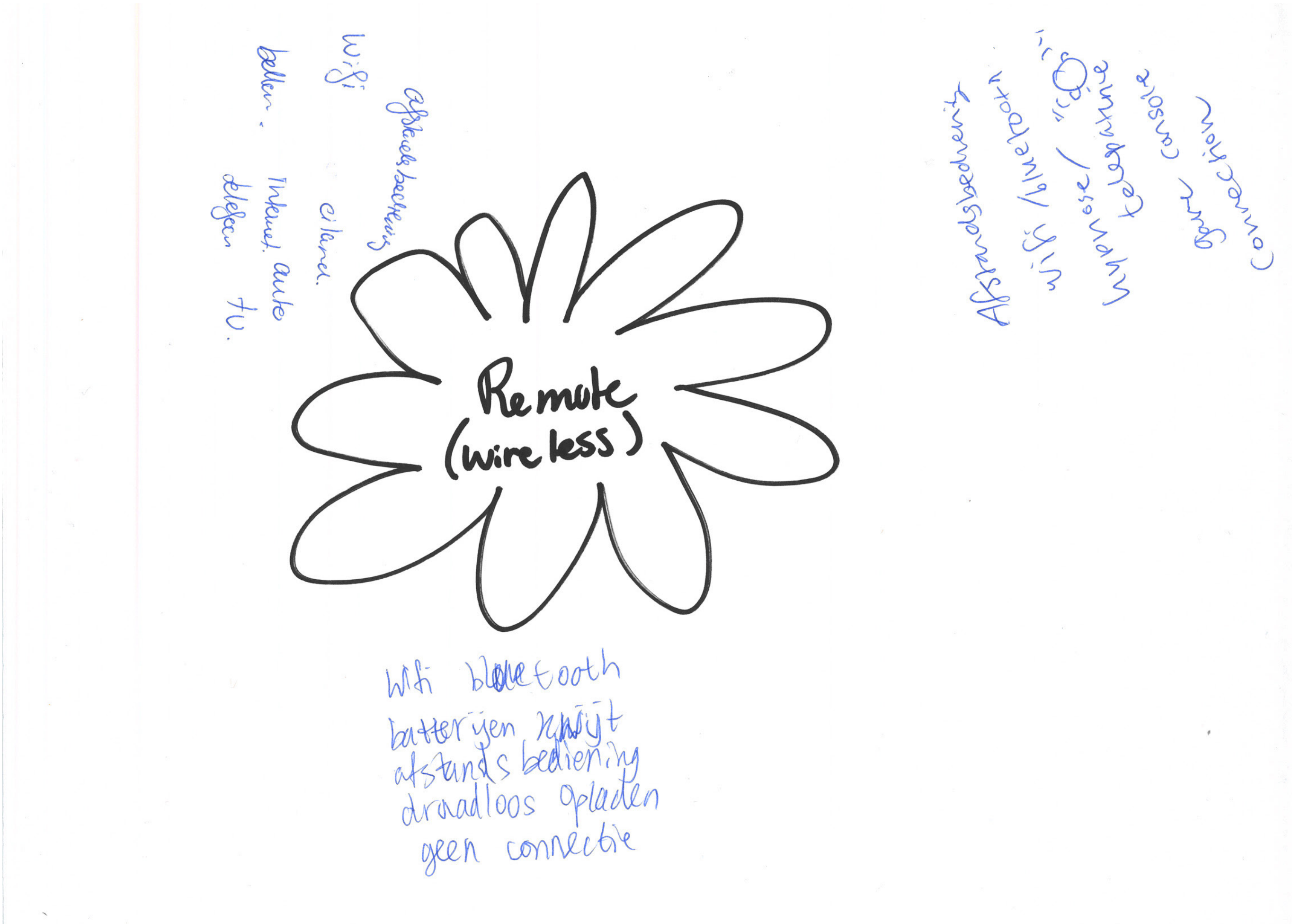


- Racecar
- Strong wind
- Running
- Limited amount of time
- Flying
- Clock
- Alarm
- Contest
- Time is a resource
- Pressure

- V=s/t
- Car
- Running
- Swimming
- Friction
- Time
- Aerodynamics
- Fluid forms
- Slow
- Car

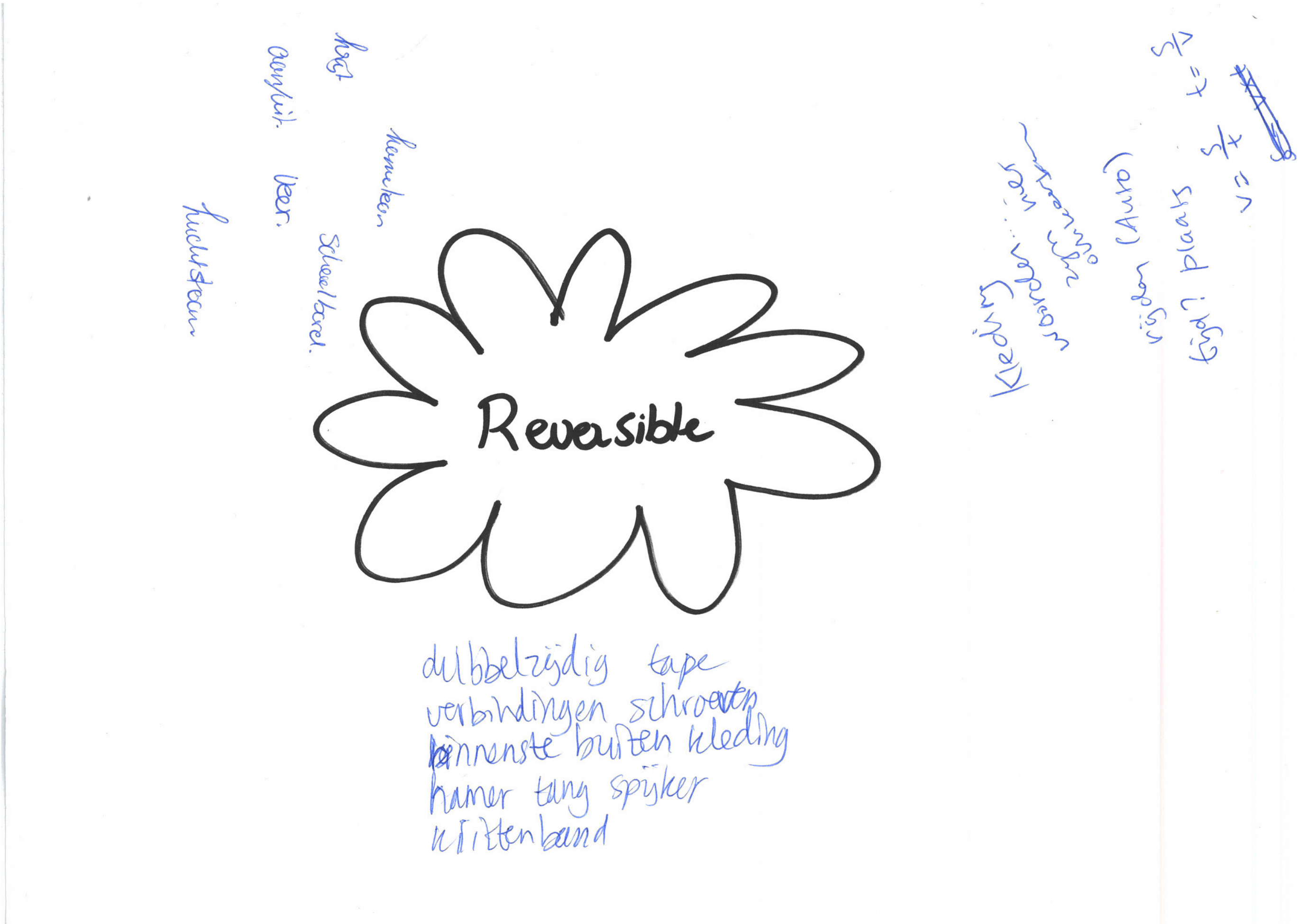
- Rocket
- Volatile
- High way
- Running
- Blinking
- Cycling
- Public transport
- speed

I.b Flower association: Remote (wireless)



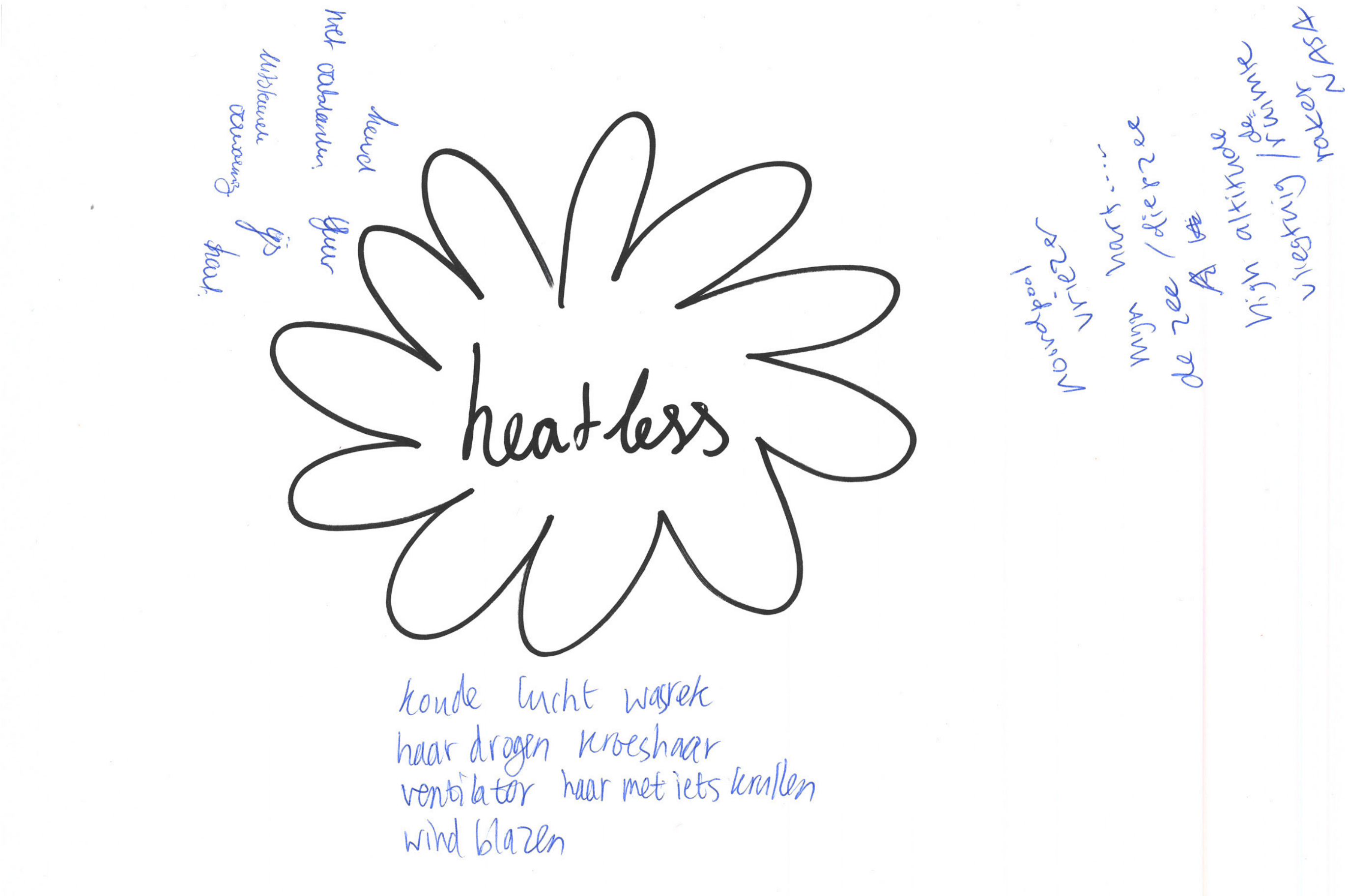
- Wifi
- Bluetooth
- Batteries
- Remote control
- Wireless charging
- No connection
- Lost
- Remote control
- Wifi/Bluetooth
- Hypnosis
- Telepathy
- Came console
- Connection
- Remote control
- Wifi
- Island
- Calling
- Internet
- Car
- Phone
- Television

I.c Flower association: Reversible



- Double-sided tape
- Linkage screws
- Inside out clothes (reversible clothes
- Hammer
- Pliers
- Nails
- Velcro
- Clothes
- Words... are not reversible
- Driving (car)
- Time? Place
- V=s/t
- T=s/v
- Chameleon
- Chalk
- Blackboard
- On/off
- Spring
- Airflow

I.d Flower association: Heatless



- Cold air
- Laundry rack
- Drying hair without heat resulting in some curls and flyaways
- Fan
- Blowing wind
- North pole
- Freezer
- My hart...
- The sea
- Deep in the sea
- High altitude
- Aeroplane
- Space
- Rocket
- NASA
- Cold
- Fire
- Not burning
- Ice
- Turned off heater
- Hart

I.e Magnetic soft materials

- Pullable
- Stretchable
- Flexible
- Elastic
- Origami
- Rollable
- Springy
- Foldable
- Clothing
- Crushable/de-crushable
- Pressing in

- Magnetically manipulable/pullable
- Automatic
- Versatile
- Conductive?
- Energy-absorbing

- Magnetic
- Remember activation shapes
- Dynamic material properties
- Quick transformations
- Wireless

- Waterproof
- Insulation
- Durable (fatigue)

- House
- Kitchen
- With your grandmother
- Skincare routine
- Office (home)
- Mall

- Laundry service
- Restaurant
- Barber
- (IKEA) furniture store

- Zoo
- Street/public areas
- Garden
- Park
- Farm

- Hospital --> waiting room
- Dentist
- Operating room
- Laboratory

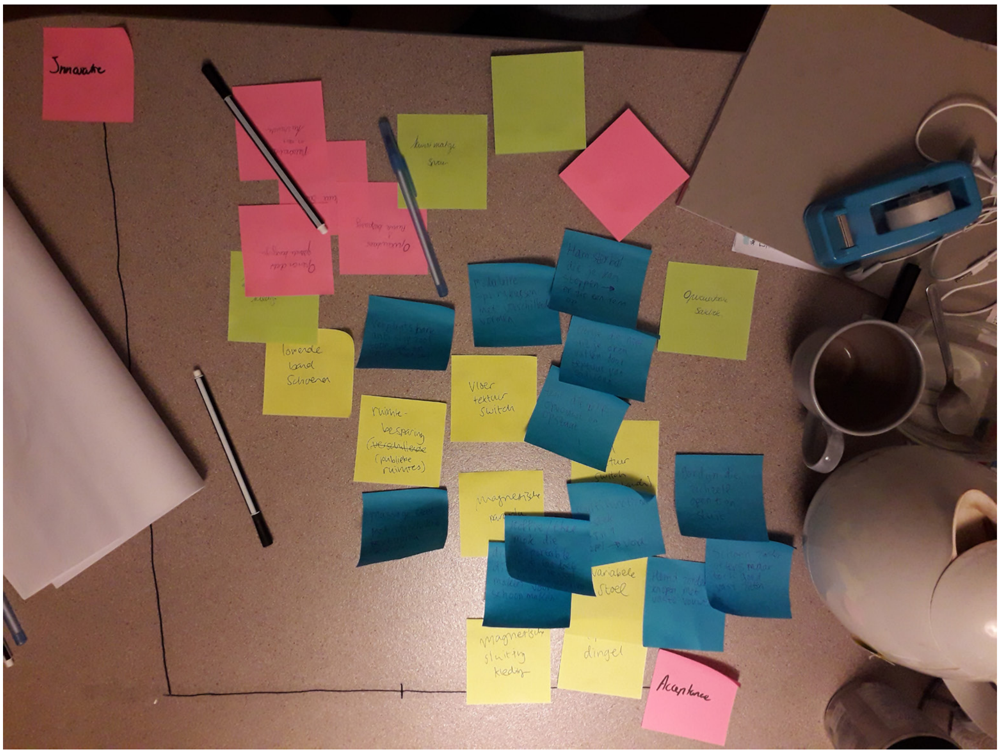
- Cinema
- Museum
- Concert
- Studio (music)

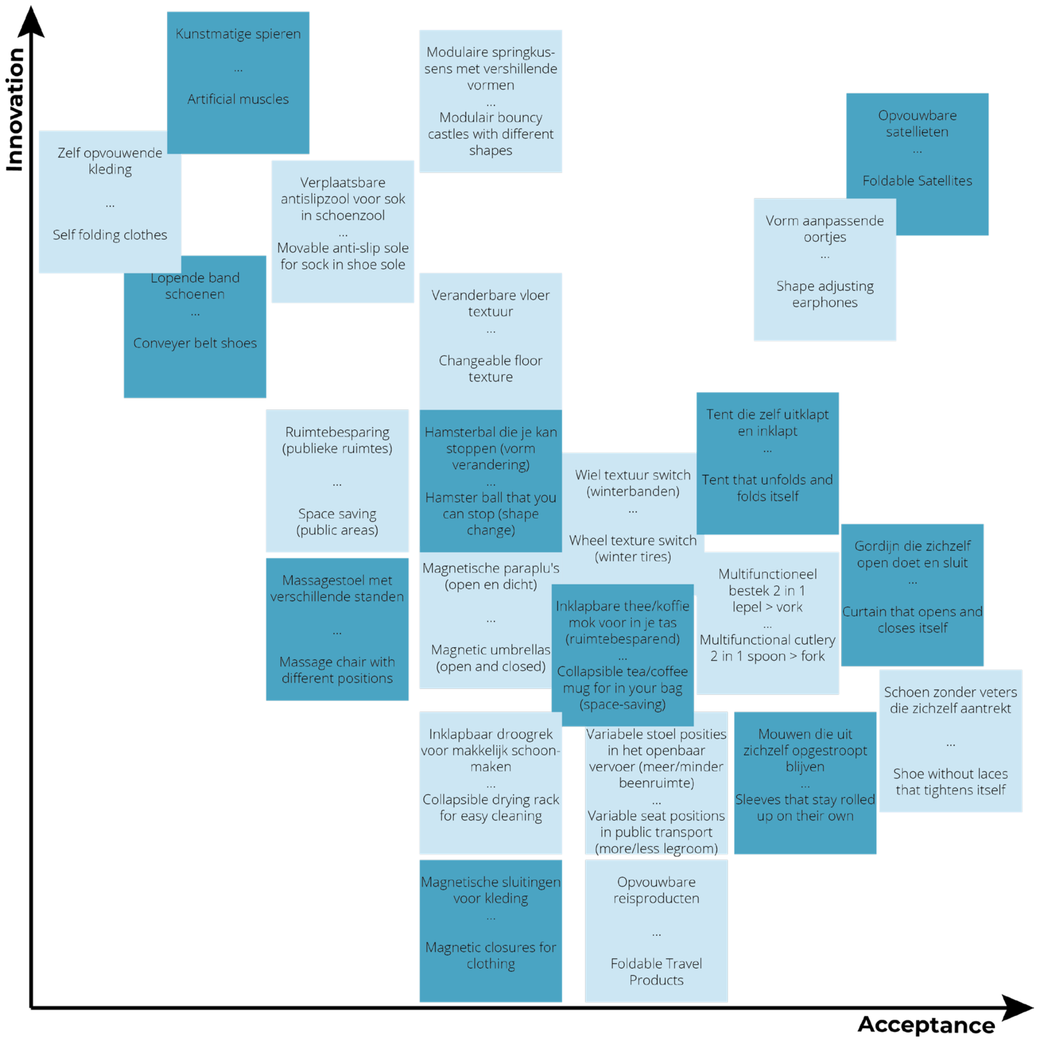
- Aquatics
- Swimming pool
- Fitness

- School
- Office (work)
- Factory
- Library/study room

- Aerospace
- Car
- Station
- Schiphol Airport
- Subway

I.g C-box

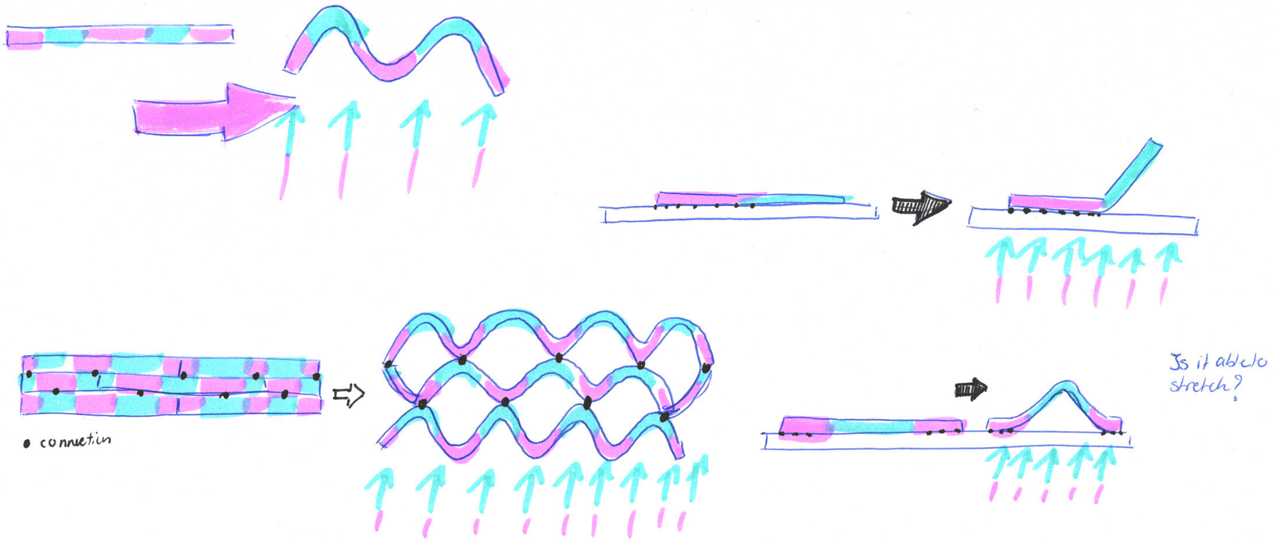


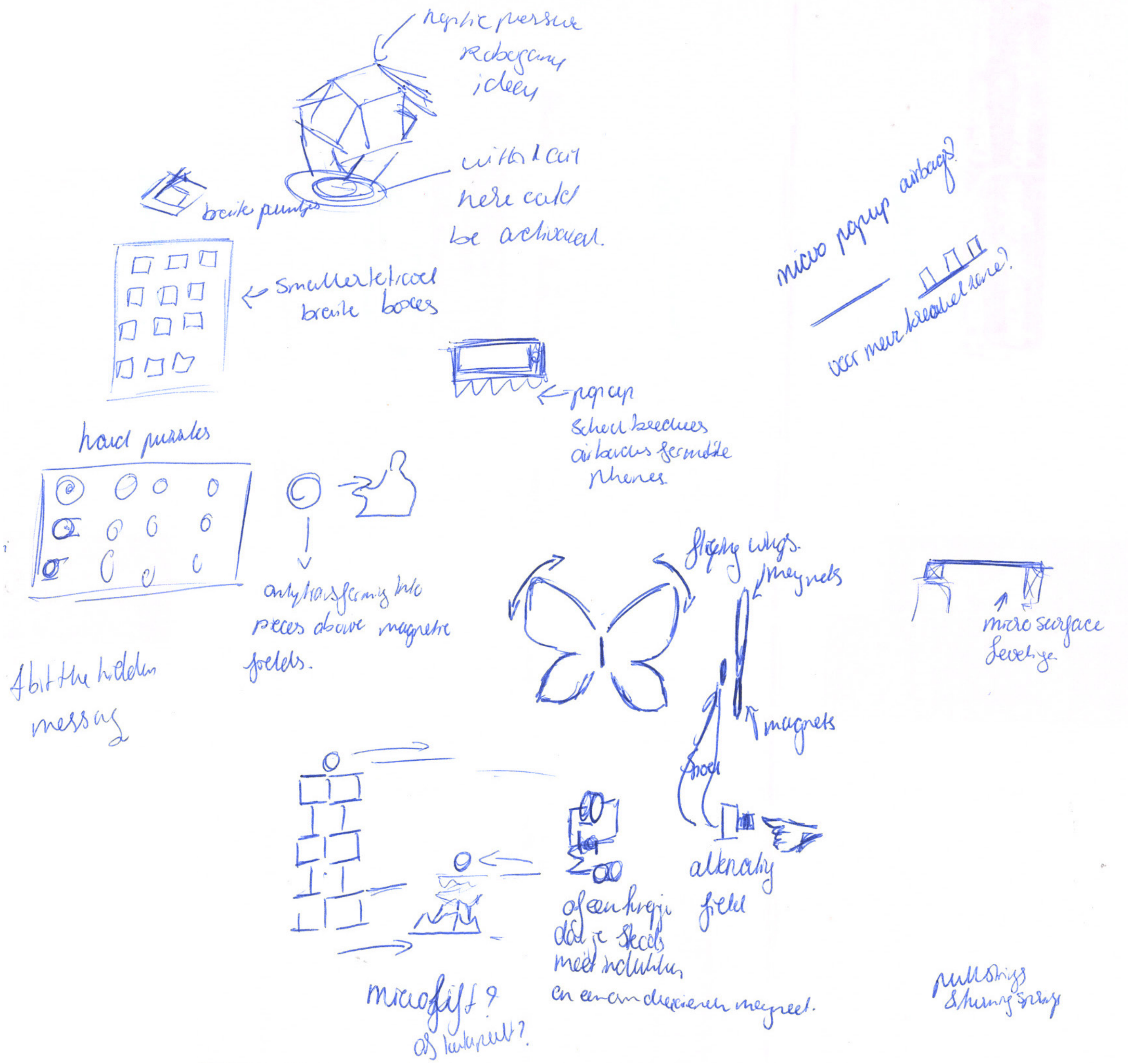


Innovation
Acceptance
Kunstmatige spieren ... Artificial muscles
Modulaire springkussens met vershillende vormen ... Modulair bouncy castles with different shapes
Opvouwbare satellieten ... Foldable Satellites
Zelf opvouwende kleding ... Self folding clothes
Verplaatsbare antislipzool voor sok in schoenzool ... Movable anti-slip sole for sock in shoe sole
Vorm aanpassende oortjes ... Shape adjusting earphones
Lopende band schoenen ... Conveyer belt shoes
Veranderbare vloer textuur ... Changeable floor texture
Ruimtebesparing (publieke ruimtes) ... Space saving (public areas)
Hamsterbal die je kan stoppen (vorm verandering) ... Hamster ball that you can stop (shape change)
Wiel textuur switch (winterbanden) ... Wheel texture switch (winter tires)
Tent die zelf uitklapt en inklapt ... Tent that unfolds and folds itself
Massagestoel met verschillende standen ... Massage chair with different positions
Magnetische paraplu's (open en dicht) ... Magnetic umbrellas (open and closed)
Inklapbare thee/koffie mok voor in je tas (ruimtebesparend) ... Collapsible tea/coffee mug for in your bag (space-saving)
Multifunctioneel bestek 2 in 1 lepel > vork ... Multifunctional cutlery 2 in 1 spoon > fork
Gordijn die zichzelf open doet en sluit ... Curtain that opens and closes itself
Schoen zonder veters die zichzelf aantrekt ... Shoe without laces that tightens itself
Inklapbaar droogrek voor makkelijk schoonmaken ... Collapsible drying rack for easy cleaning
Variabele stoel posities in het openbaar vervoer (meer/minder beenruimte) ... Variable seat positions in public transport (more/less legroom)
Mouwen die uit zichzelf opgestroopt blijven ... Sleeves that stay rolled up on their own
Magnetische sluitingen voor kleding ... Magnetic closures for clothing
Opvouwbare reisproducten ... Foldable Travel Products

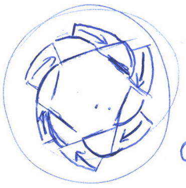
J. Generated ideas

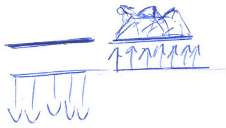
This attachment contains most of the brainstormed ideas during this project.


connection
Is it able to stretch?


hand puzzles
popup
micro popup airbags?
flapping wings
magnets
magnets
alternating field
more surface

Alternative buttonsor signs.



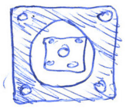


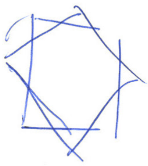
Ook een fijf achtig iets.
also rotating!! even
when only using a fieler
going upward.
might be interesting not that
special
But the rotational movement
is interesting.

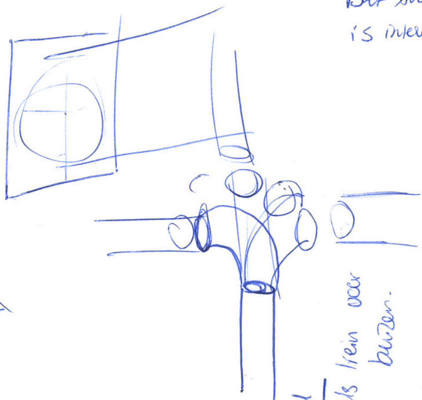
In kwade gebieden
waar mechanica niet werkt.

In koude gebieden?
waar mechanica niet werkt
Quantum computers?

Pakking.
opvouwen
en uittrekken en plaatsen
bestemming.
inwendelmenden



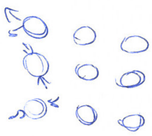


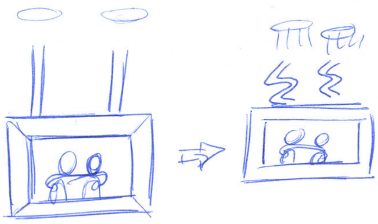


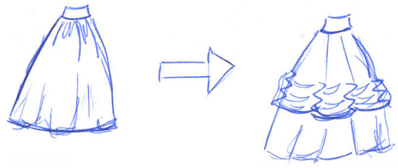
Wissel
zoals trein voor
buizen.

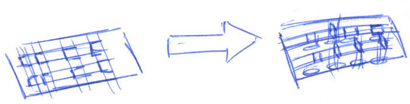
Voor verstelbare filters.

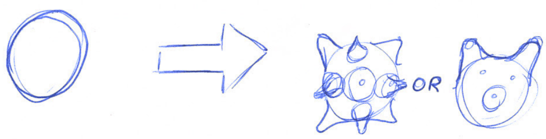
Zelf schoonmakende of ~~...~~ filters.

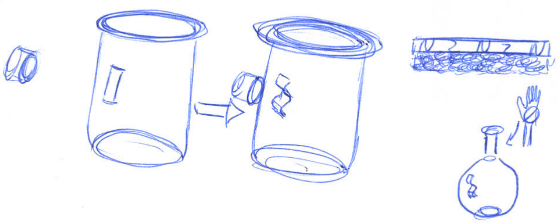


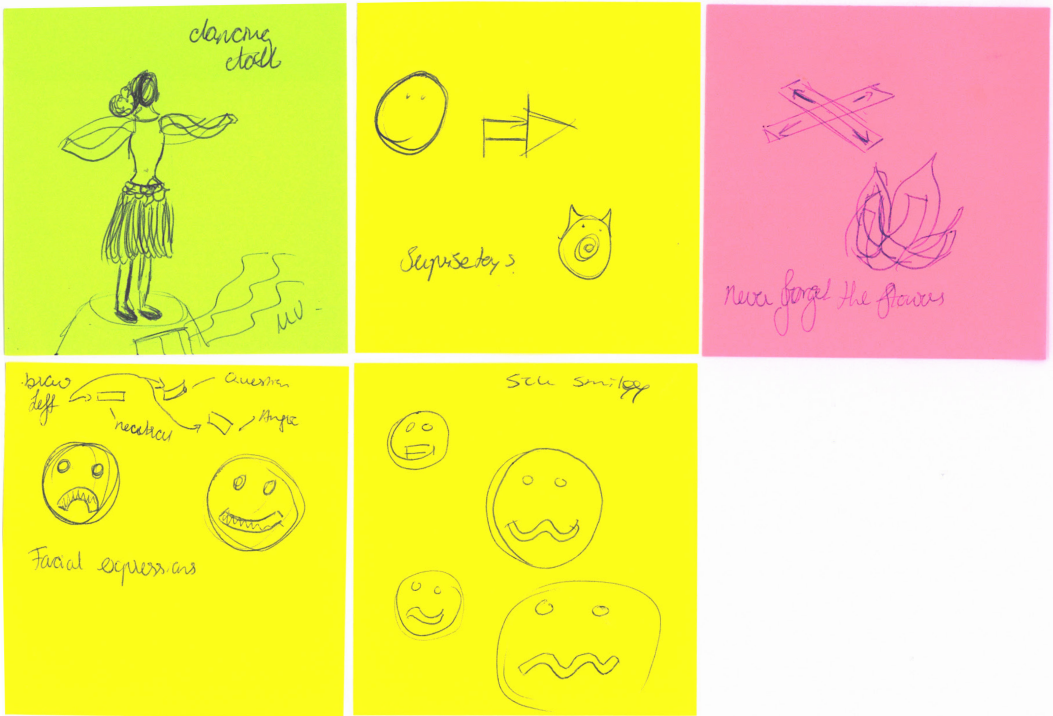











dancing doll
Facial expressions
never forget the flowers


Secret mesage
What if
this was constantly moving?
imitating waves of the sea
calm sea waves
Creating waves in a aquarium
Chang button surface
or feel the activated caps lock
on your computer or shift for bold letters

TU Delft