Clamping mechanism

of memory shape material for delayed female sterilization

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by

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Abstract

Female sterilisation is difficult to access in many low- and middle-income countries. Opt Medical wants to solve this problem by developing a clip that can be placed around the Fallopian tubes during C-sections without immediately affecting the fertility. By activating the clip at a suitable time, a clamping mechanism occludes the Fallopian tube leading to scar tissue formation and eventually sterility. This requires the development of a Nitinol clamping mechanism as described in this study. Part 1 contains a study on the shaping and training of Nitinol, part 2 describes the development of a simple and affordable way to test the clamping force and part 3 focuses on the iterative design process of the Nitinol clamping mechanism.

In Part 1 an extensive research on Nitinol is performed with the purpose of gaining sufficient knowledge on technical specification of the material to find the best method and setting of processing Nitinol. The preliminary experiments are performed as creative processes to find methods for connecting Nitinol and train the wires into complex shapes. The connection methods evaluated here are crimping an aluminum tube around two wire ends and soldering copper-plated Nitinol wires. The shaping is realized using 3D printed moulds and plaster. The further experiments are performed on coiled Nitinol wires in various shaping conditions to understand the dependencies between the shaping conditions and the final output. As the results, the crimping method for connecting the Nitinol wire is more sufficient than soldering. The layer of copper does not allow to make a connection. Complex shapes can be produced using 3D printed plastic moulds together with embedding them into plaster. Plaster keeps the wire in a desired shape during the shaping process in high temperature, in which the plastic part melts. The time and temperature of the shaping process have influence on the final activation temperature together with the manufacturing method of the wire, constraining method of the sample and wire's supplier. Because of large number of variables, the test for individual type of wire has to be performed to choose the settings which will lead to specific activation temperature.

In Part 2 the testing equipment is being developed based on the principle of multiaxial testing machine. The concept assumes that the samples are tensed and examined for the exerted force while undergoing the structural transformation. The test set-up includes five spring scales (distributed around the sample in equal distances), five aluminum arms with slots to move and fix the scales in certain position (to control the pretension), additive manufactured parts to ensure the fixation of the scales and keep them at the same level. In principle, the sample is clamped to the scales. Due to the design, the scales can be moved and provide tension on the samples. The tension is displayed on the scales and it corresponds to the stretching of the coil. While heated, Nitinol samples change their shape. If the pre-tension force is equal or lower than the maximum potential force of the sample, the deformation takes place. That maximum force is recorded to compare the designs.

In Part 3 several prototypes of various shapes are made from Nitinol and tested. They are grouped in categories of turned and flat wire, and numerous shapes. Besides that, there are samples of circular coils with various wire thickness, mandrel size and pitch value. The samples are tested for exerted force using the test set-up described in Part 2. Each sample is pre-tensed by the spring scales and heated with a heat gun from above. When the pre-tesion still allows for deformation, next measurement is taken with increased pretension value. The procedure is repeated until the sample does not deform when heated. The samples with the highest measured force are the round coils made of wire thickness 0.5 mm. When scaled down to the size of Fallopian tubes, they show the ability to close its lumen.

Contents

1

Introduction

1.1. Problems and ethics related to female sterilization

Nowadays the female sterilization process is available in most Western countries. In low income countries however, women often do not have access to these procedures. In many cases, are not allowed to decide themselves on the matter of sterilization, and in cases where they can, the local healthcare system is often not equipped to provide sterilization in a clinical setting due to financial constraints [1]. The moment those women undergo surgical procedures is during childbirth. However the sterilization can not happen during such event. The reasons for that are extreme experience a woman is subjected in this moment followed by hormones level far from common stage, but also irreversibility of sterilization [2]. Even though a women would like to be sterilized during a childbirth, she might change her mind when the baby she is having dies prematurely [3].

1.2. Proposed solution

Opt Medical provides research and development in the field of contraceptives. Their recent research is focused on a solution which should be developed to ensure that a fitting option exists for every woman. They want to use adaptive and product-centered research methodologies to learn how to remove barriers to contraceptive uptake in low-income countries. Opt Medical is developing a solution for those women so they can decide to undergo sterilization whenever they are ready for such a decision without a need of additional surgery. The idea is to create a device to be placed over the Fallopian tubes during Cesarean section, and activate it later without a surgery. Because of that the solution is referred as for delayed female sterilization.

The innovative idea makes the possibility to implant a sterilization device over the Fallopian tube during the labor. The device does not prevent the insemination at this time. It is implanted inside a woman body without causing any changes in her fertility and ability of impregnation. The implant though is active and can be actuated without a surgery at any time after its implementation. The working principle is to squeeze the Fallopian tube to block the passage and prevent pregnancies.

The activation of the implant can be done using an electromagnetic field. Radio frequency signal can be sent to an implant and induce a current flow through the mechanism. Due to energy losses dissipated as heat, the implant's temperature is rising. Temperature sensitive shape-memory-alloy (SMA) can change the shape accordingly while heated. The clamping mechanism can be trained and designed to squeeze around the Fallopian tube. The activation through radio frequency together with the use of SMA, and more specific Nitinol, is already used in biomedical implants. Micro-actuators are little devices which are programmed to change shape when the temperature rises. In certain frequency, the flow through the circuit is induced. Then due to heat dissipation, the Nitinol structure changes its shape.

1.3. Conventional female sterilization methods

Female sterilization procedure is a surgical procedure that permanently prevents pregnancy by occluding the Fallopian tubes so the egg is stopped and cannot be transported from ovaries to uterus. There are several methods to perform the procedure. The most straightforward division is between an open surgery and laparoscopic surgery. The laparoscopy technique in comparison to the open one has a lower cost and faster recovery time [4].

The techniques for permanent sterilization include cutting and folding the tubes, removing sections of tubes or blocking the tubes with bands or clips. Figure 1.1 presents the methods. Also, the anatomical picture of women reproductive system is shown for identifying the scope region of sterilization.

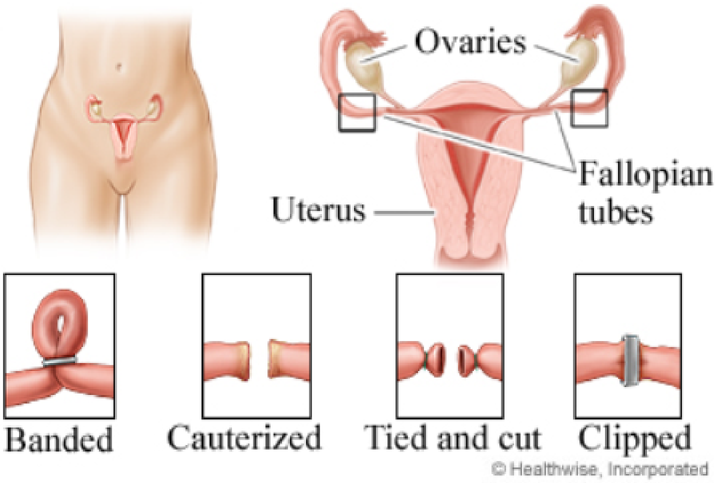

Figure 1.1: Common methods of female sterilization

Most commonly used clips are the Filshie clip and teh Hulka clip. The Filshie clip is a titanum clip lined with silicone developed for female sterilization. It bends around the Fallopian tube and the tubal necrosis occurs. The clip is small and its dimensions are: 14mm x 3.4 mm x 4 mm. The silicone lining applies continuous pressure to a Fallopian tube which results in developing a scar on the tube and blocking the passage [5]. The Filshie clip is introduced via laparoscopy with use of specially designed applicator device. The applicator is a laparoscopy instrument with a designated space for a clip at its tip. While a surgeon positions the clip over the Fallopian tube, he can apply pressure via the applicator and secure the clip on the tube. The Hulka clip is a spring loaded silicone clip of working principle the same as the Filshie clip. The procedure of bringing the Hulka clip into a female reproductive system is shown in figure 1.2. The Hulka clip is put around the tube with a laparoscopic device and clamped over the Fallopian tube. At the end, the surgical instrument is retrieved and the clip remains on the tube. All of the above are sufficient sterilization methods but all require expensive laparoscopy equipment. Those methods are widely used in the Western world.

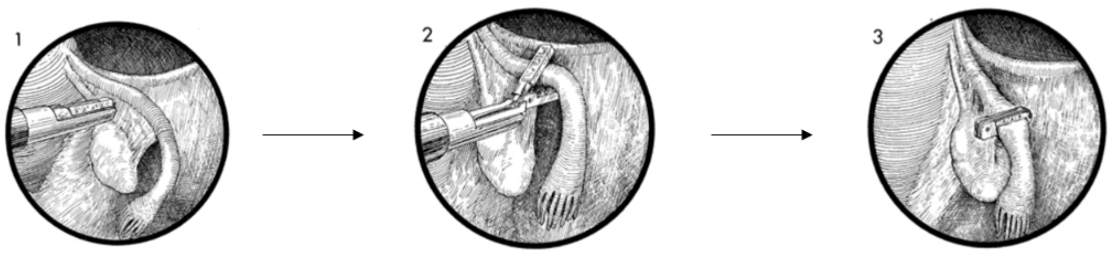

Figure 1.2: The procedure of applying Hulka clip over the fallopian tube

Another technique introduced only few years ago is Essure. It is a nonsurgical method as the device is brought to the Fallopian tubes via natural orifice in woman's body - a vagina. It consists of two tiny metal coils. One is inserted into one Fallopian tube, and one to another Fallopian tube. Eventually, scar tissue forms around the coils and blocks the Fallopian tubes. However, since 2019, the device is no longer distributed and used. Effective 31 December 2018, Ensure has been recalled from the American Market by the U.S. Food and Drug Administration (FDA). There have been cases of bleeding, pain and allergic reaction reported by the patients. In figure 1.3 the cross section of a Fallopian tube with a metallic coil inserted is shown.

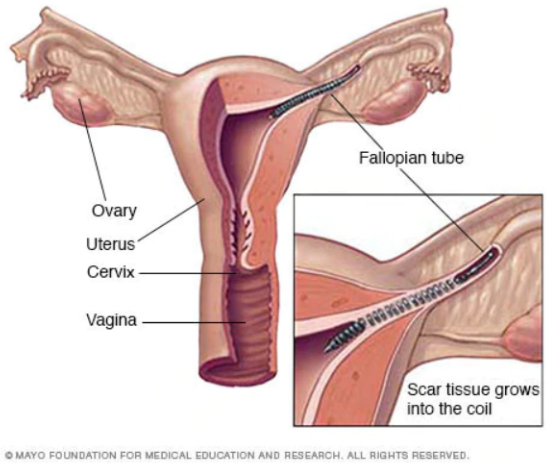

Figure 1.3: Cross section of female reproductive system with inserted Essure coil

The available sterilization methods are not suitable for the women in low-income countries due to the need of an additional surgery to perform the sterilization. The only one known method which does not require the surgery involves a device recalled from a market because it is not safe.

1.4. Challenge and approach

The solution proposed by Opt Medical includes an SMA clip but also the activation system. The activation system is based on a power transfer principle for heating purposes. It will result in heating the SMA clip which then changes its shape and the activation is initiated. It requires a transmitter and a receiver. The clip itself acts as the receiver and pick up the signal sent by the transmitter. Passing current is causing the heat dissipation which initiate the change in SMA structure. The performance is dependent on the closing force of the clip as well as its magnetic inductance and thermal properties for efficient power transfer. The conducted research exclusively focuses on the closing force, therefore the research question concerns only the shape of the clip and force exerted by it. The overall picture of the whole project proposed by Opt Medical is presented in figure 1.4. The marked part is the element on which the research in this thesis is focused on.

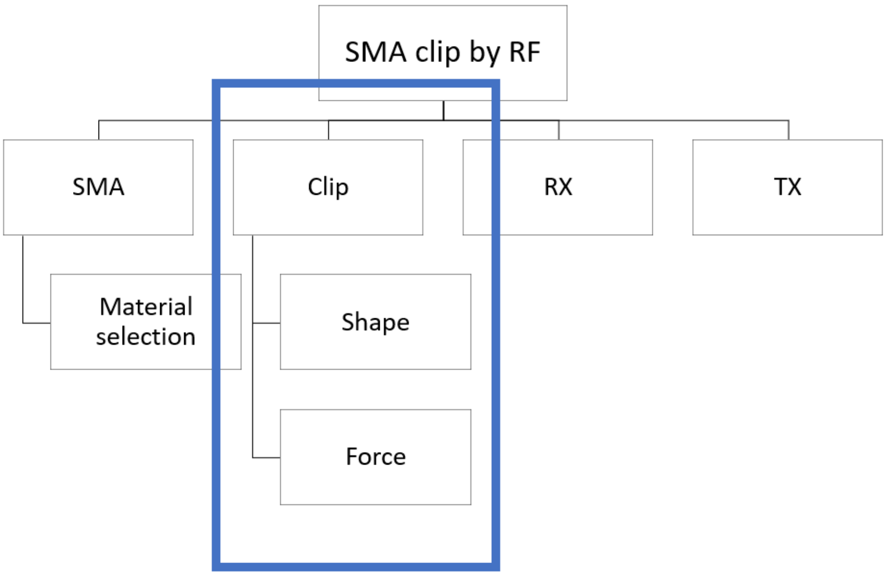

Figure 1.4: Elements of the project proposed by Opt Medical with the marked focus of this thesis

The aim of this project (included in this thesis) is to design and test a mechanical clamping mechanism of memory shape material for delayed female sterilization. The challenge is to find the relation between the design of the mechanism and the force exerted by the mechanism during the shape changing. it can be done by including two additional research questions:

- How are clips shaped from the SMA wire?
- How can the closing force of the shaped clips be measured?

Different shapes and sizes of the device must be manufactured and tested in order to find this relation. First, the SMA - Nitinol - has to be studied thus its properties can be used in a beneficial way. Secondly, the method of manufacturing shaped from that material has to be explored. Beside the shape design and development, the testing method is another goal of this project. There must be a satisfactory method found or developed to test the prototypes. A part of this project is also to provide a sufficient way to complete the measurements of the force exerted by the device.

1.4.1. Understanding Nitinol

The clamping mechanism which is the subject of this project is designed with shape memory material (SMA). Using such material gives the possibility to activate the mechanism with the use of heat. The activation is then executed by the shape change of the structure. In this case - the clamping over the Fallopian tube. The most common SMA used for medical implants is Nitinol. It is because of the biocompatibility and good response of the human tissue on the implants made of Nitinol. Nitinol is an extraordinary material which gives many possibilities to use its unusual properties. Nitinol is known by its ability to "remember" its prime shape. A structure made of Nitinol can be deformed but still transform to its original shape when heated up. However, this material is relatively new and used in the medical sector shorter than for example stainless steel. Due to the novelty of Nitinol, there are many questions and uncertainty in this subject. For better understanding this outstanding material and to transfer the knowledge into a design of a clamping mechanism, Chapter 2 is added to this report. It includes data obtained from scientific literature as well as the results of my individual work with Nitinol.

1.4.2. Contribution

Me, as a biomedical student interested in medical devices, I want to be a part of such an amazing initiative to help to overcome problems of people who cannot solve it for themselves. The privilege of studying and living in one of the most developed countries in the world, give me the opportunity to cooperate with Opt Medical and add my part to the solution.

1.5. Thesis lay-out

In this introduction the background on problems with female sterilization in low income countries are presented. It is followed by a possible solution and current methods for sterilization. Subsection "Challenge and approach" presents methodology of identifying the research questions and answering them. Further, it is explain why additional chapters are included to the report. Next, the thesis consists of three main chapters. Chapter 2, which covers the main knowledge on Nitinol properties and research on parameters influencing its behavior. Also it contains the method of shaping the Nitinol wire. Chapter 3 presents the work done in developing a testing set-up which can serve to verify the design of a clamping mechanism by measuring the force exerted by the Nitinol structure. Chapter 4 is devoted to the shape design and the resulting final design. The final design is a model of the most promising design found due to testing of many prototypes. Each chapter is structured in a way to have an introduction, method, result, discussion and conclusion sections. Besides that, the chapters 5 and 6 are the discussion and conclusion chapters of the whole thesis.

2
Nitinol

2.1. Introduction to Nitinol

This section covers the main knowledge on Nitinol properties and research on parameters influencing its behavior. The use of Nitinol for this project is essential. The design of the clipping mechanism is based on shape memory alloy. Excellent properties of Nitinol together with its biocompability make the choice obvious. It is a very complex alloy which is still being researched and tested thus there are many uncertainties concerning Nitinol. Knowledge from literature and scientific publications is gathered here for better understanding of this "magical" material. It covers the essentials as the indications of how to use it and how to profit from its benefits; also the explanation on the micro scale to comprehend the principle behind Nitinol's performance.

This chapter consists of preliminary experiments and proper experiments. The difference comes from the methodology of executing the experiments. First two preliminary experiments A and B relate to the work done in exploratory way. The methods of production are investigated. The two preliminary experiments are made to find the way of connecting the wire ends with each other (preliminary experiments A), and to find a method to manufacture unusual samples shapes (preliminary experiment B). They are conducted in "try and fail" way and evaluated based on the reliability and usefulness of the methods. The other experiments numbered from I to V are executed in systematic way. The results of those are obtained in structured tests, and the conclusions are drawn based on numerical values of the results.

2.1.1. Challenges of working with Nitinol

Due to its extraordinary properties, Nitinol is a material which requires very special processes of manufacturing. The same applies to connecting two ends of Nitinol wire in order to create a closed circuit. Regular processes of welding or soldering do not apply to Nitinol and alternative ways have to be found. It is the goal of this study to find an easy and available method to connect two ends of Nitinol wire. The need is to create a prototype which is a closed structure for a reason to investigate and test its ability to close the Fallopian tube. The requirements for the connection is that it does not break under tension applied to stretch the structure. The same tension must not restrain the mechanism from changing its shape when it is heated.

The last but not least objective of this study is to find a method to create any kind of Nitinol samples. The unusual or complex design cannot be limited by the lack of manufacturing method. An easy and efficient solution needs to be explored. In the next subsections, the answers for the mentioned challenges are studied by the mean of experiments. The discussion and conclusion provide the direct answers.

The first characteristic of Nitinol to be investigated is the dependency of the shaping process parameters on its activation temperature. The shaping temperature is often given in literature and studies as a range and not one specific value of temperature. Also, it varies for different studies. Moreover, time of shaping is often unspecified. An experiment needs to be performed to find the dependency of the those two parameters - time and temperature - on the final activation temperature of Nitinol products.

An important question raising while shaping Nitinol, is how important is the pre-tension of the samples when the are being shaped. How does it influence the activation temperature or if and how does it influence the final shape. If it does influence the outcome, the best method to provide a required fixation needs to be found and analyzed. Another experiment is planned to find a relation between pre-tensing Nitinol wire and the outcome of the shaping process.

Nitinol wires are offered in two options of manufacturing method: straight annealed (SA) and as drawn (AD). The practical distinction given by the supplier is the difference in proficiency of the user but it does not give an explanation of the final effect on prepared samples from such wires. The investigation needs to be carried out on dependency of manufacturing method of the wire on the final outcome of activatio temperature after pre-shaping the wire by a purchaser.

The suppliers of Nitinol do not provide extensive data on mechanical properties or chemical composition of the material. The only information given by them is the transition temperature. Unfortunately, they do not share the data in which conditions (i.e. time and temperature) the final given activation temperature can be achieved. There is no normalized method of testing the wires thus each manufacturer may reach the activation temperature using a different method of annealing and shaping Nitinol. Wires used in this project come from two suppliers: Flexmet and Kellogg's Research Labs. The researched information is: is the user able to obtain a specified activation temperature using the same shaping method for wires provided by various suppliers.

Even when the activation temperature is specified for a particular wire, it can be tweaked by aging the material. The effect of aging on the final outcome is also investigated in this section.

2.1.2. Background on Nitinol

Nickel-Titanium alloy also known as Nitinol is a material which gained recently its popularity among many technological fields including the medical. In the last few decades the use of Nitinol for medical applications boomed and there are many Nitinol medical devices available on the market [6]. Nowadays, it is used to produce among others: orthopeadic implants, cadiovascular stents, superelastic needles and orthodontics products. That happened due to outstanding Nitinol properties and its biocompability. The unique properties of Nitinol are the shape memory effect and superelasticity.

Nitinol was discovered in 1959 but commercialized years later. Due to difficulties with manufacturing, processing and machining, the alloy was not favorable. Only in 1980s the difficulties began to be resolved. The young age of Nitinol results with a limited research on methods of processing the material. The very challenging process of manufacturing Nitinol leads to varying quality between manufacturers. The material is sensitive to the process of annealing, aging and machining. Together with chemical composition they create a list of variables to set up a final behaviour of Nitinol [7].

Properties

Nitinol is a shape memory alloy (SMA) which contains Nickel and Titanium. Those two elements are present in the alloy in almost equal percentage. The small differences between the ratio give the alloy its distinguished properties and set the transition temperature rate.

The shape memory property is a result of a solid-state transformation called a martensitic transformation. This unique property is a resultant of a reversible solid state transformation. It occurs while the Nitinol is subjected to an external stimuli as temperature change or deformation.

Nitinol has to phases: austenite - the parent phase; and martensite - the daughter phase. The austenite phase occurs at high temperatures while Nitinol exhibits simple cubic crystalline structure. At low temperature Nitinol transforms to a more complex monoclinic crystalline structure. As the alloy is being heated, austenite starts to form at the austenite start temperature (A_s) and finishes at the austenite finish temperature (A_f). When the alloy is cooled down a transition from austenite to martensite occurs starting at martensite start temperature (M_s) and finishing at martensite finish temperature (M_f) [8].

The principle of the changes of crystalline structure of Nitinol is presented in figure 2.1, while in figure 2.2 the dependency of an austenite structure on temperature is shown. Also the latter figure presents the hysteresis effect which is caused by the difference in phase transformation temperatures when Nitinol is heated to when it is cooled down.

Another special property of Nitinol is superelasticity sometimes called pseudoelasticity. It is demonstrated when Nitinol is in the austenite phase. If the stress is applied on an item made of Nitinol, it will change its shape. However once the stress is retracted the result is a superelastic response and the item comes back to its shape. It is shown in figure 2.3 as a diagram. The crystaline structure represents Nitinol in austenite phase above A_s temperature. Typically Nitinol demonstrates superelasticity up to 8% strain. Above that, permanent deformation is expected [8]. However small differences can be noticed as the properties of Nitinol depends on thermomecanical processing.

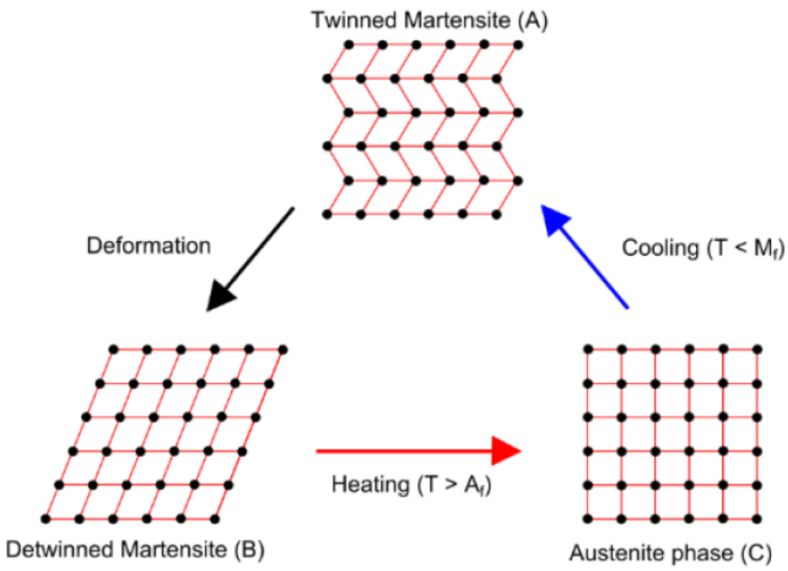

Figure 2.1: Crystalline structure changes in shape memory effect

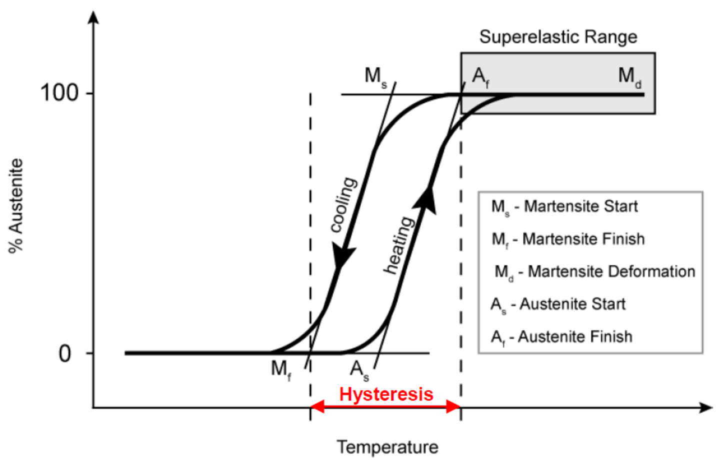

Figure 2.2: Dependency of an austenite structure on temperature, and hysteresis

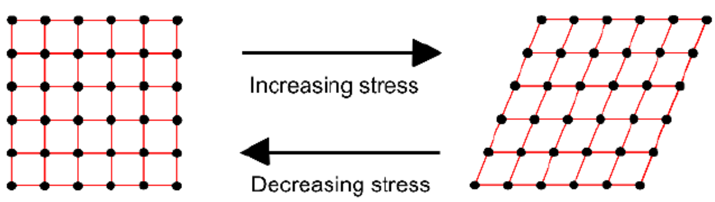

Figure 2.3: Schematic visualization of superelastic effect of Nitinol

Heat treatment of Nitinol

The heat treatment of Nitinol includes annealing and shape setting. In many publications those two names are used as synonyms which can lead to a confusion while working with Nitinol. Some sources (ex. [9]) give the explanation of the shape setting occurrence due to annealing process of the material. While in [10] the manufacturing process of Nitinol wire is described. The final steps in the production consist of cold drawing to a desired diameter and straight annealing. Straight annealing is a heating a pre-loaded (20-100 MPa) wire to a temperature between 450 and 700°C. This step is included in a manufacturing of a wire and not its shaping. A step for setting a shape is conducted in moderate temperature about 500°C, in short period of time. These two are to prevent the permanent deformation and to maintain the superelastic behavior. In this report the annealing refers to the final step of the manufacturing process - the straight annealing. The process of shaping the wire and training it to new and unique shape is called shape setting.

Manufacturing

In order to manufacture a Nitinol wire various steps are applied. First, Nitinol is forged and rolled into a bar at high temperatures. From there, the material is cold drawn to a wire using a die of a preferred diameter. After that, the wire might be annealed or not. Annealing causes recrystallization and ultimately restoring ductility in Nitinol. The wire which did not go through annealing process is labeled as as-drawn (AD). In this state, the wire is easier to form and has higher strength in comparison to a straight annealed (SA) product [8]. A scheme in figure 2.4 presents the process of drawing a Nitinol wire.

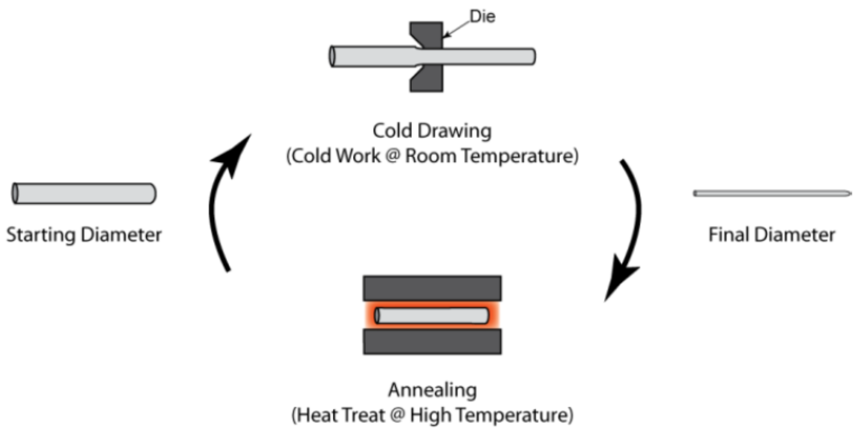

Figure 2.4: Process of manufacturing a Nitinol wire

Shape setting

Shape setting of the Nitinol consists of several steps which are invariably mentioned by amateurs in the internet as well as more science directed scholars and educators. Gathered information from various sources led to an identification of basic steps that have to be carried out to perform a successful shape setting of a Nitinol wire:

1. Restrain the wire in a desired shape
2. Heat up the wire to annealing temperature
3. Cool down the wire instantly in cold environment
4. Allow the wire to return to the room temperature

The steps listed above seem to be vague and general. The exact temperature and time varies from paper to paper and from one experiment to another. Despite the fact that the range is within 150°C(400 to 550°C) and the time never exceeds several dozen minutes, the exact parameters can be established only for an individual case. There are several reasons for that practice - wires differ from each other with dimensions, chemical composition and a principle of manufacturing. Also the final and desired transformation temperature differs per design and application and it depends on the process of shape setting.

Influence of chemical composition

Basic chemical composition of Nitinol is Nickel and Titanium. However, often certain additions are combined with Nitinol in order to shift the properties of final material. Typically it contains approximately 50 to 51% Nickel. Martensite start (M_s) temperature is very sensitive to changes in the ratio between Nickel and Titanium. As shown in figure 2.5 provided by Memry Corporation based on their own research. The temperature of the begining of martensitic transformation changes about 80K (80°C) per one atomic percent of Nickel.

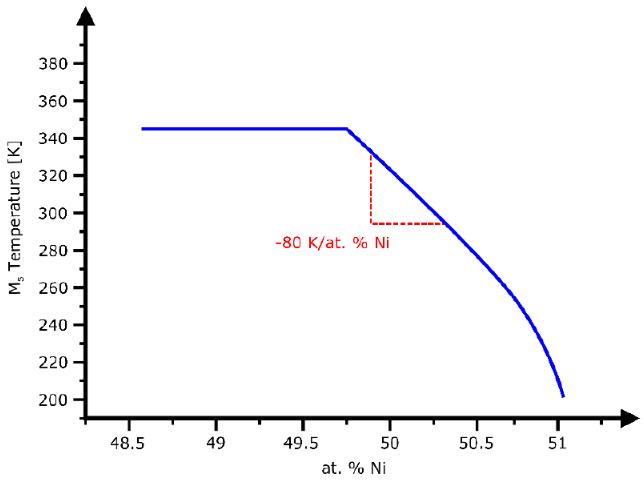

Figure 2.5

Also the other additions to the alloy can change its property. One of the examples is copper. Copper improves the fatigue life but limits recoverable deformation to 3%, when the rule of a thumb is that Nitinol can recover 12% deformation (once). The recovery deformation decreases with the number of cycles for pure Nitinol [11].

Aging

Aging is a second heat treatment, performed in order to change the transition temperature of Nitinol but keep the pre-defined shape. Aging causes the transition temperature to rise. In case of this heat treatment, the Nitinol sample does not need to be constrained. Sources mention temperatures between 375°C and 475°C for aging Nitinol.

Outcome

After the shape setting, the wire can be:

- stiff and springy
- soft and leaden
- brittle

If the wire comes out brittle it means an overheating has been occurred and the wire is no longer of any use. If the wire results to be soft and leaden when the memory shape has been set and the transition temperature is above room temperature and the Nitinol is ready to use. But when the Nitinol wire is soft and springy that means that the memory shape has been set but the transition temperature is near or below room temperature. If the super elastic Nitinol is desired then the job of heat treatment is complete. However if the shape memory effect is expected than a second heat treatment is needed.

Joining

As it is very unique material, connecting it is equally exceptional. Commonly known for joining Nitinol are laser, plasma, and resistance welding; crimping and soldering. High temperature of welding can cause effects such as recrystallization and grain growth leading to a decrease in mechanical strength and shifts in the transformation temperatures [8]. The connection (the example is shown in figure 2.6)can be very smooth in comparison to crimping and soldering. The crimping seems to be the most robust method but the effect is bulky and it creates a multi pieces construction. To crimp Nitinol, a tube which is used for this purpose must be plastic enough to clench around the wires. Soldering Nitinol can be done using silver bearing solders. Also, use of the flux is recommended as the top titanium-oxide-rich layer prevents from soldering. In order to achieve good results molten solder must have an access to oxide-free layer which can be done using flux [12]. The outcome of this method is also bulky and uneven. The Nitinol products which were crimped or soldered should not be used as implantable devices in their raw state. By raw stateit is meant the connection which is not covered by compatible material. The solder material is not suitable to be in contact with human tissue. Also, the bulky connection may may cause a hazard of rapturing the tissue. In figures 2.7 and 2.8 the examples of Nitinol connections using soldering and crimping methods respectively.

Figure 2.6: An example of welded Nitinol to stainless steel [13]

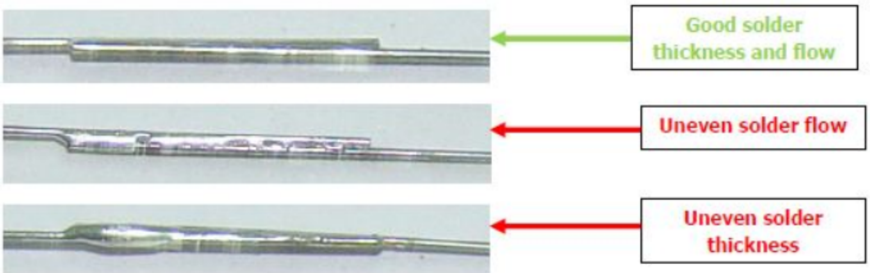

Figure 2.7: An example of connection made by soldeing Nitinol [14]

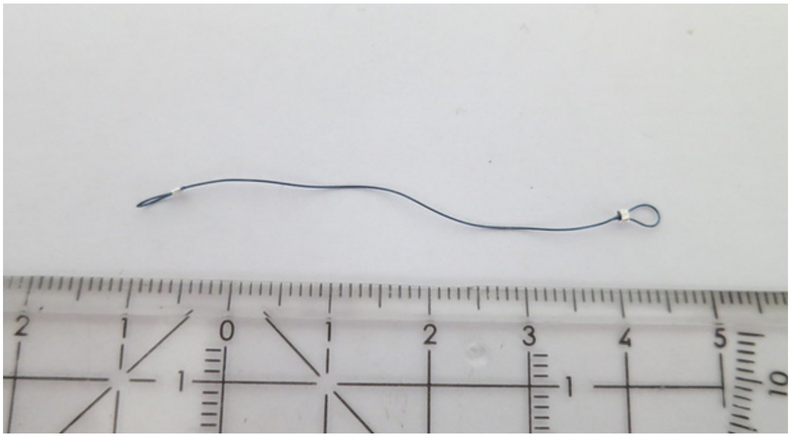

Figure 2.8: An example of crimped Nitinol wire [15]

2.1.3. Aim of the research on Nitinol

The aim of the study presented in this chapter is to gain knowledge in the subject of Nitinol and transfer it into the process of designing a clamping sterilization mechanism. Nitinol is the preferred material for the purpose of this project thus necessary information must be obtained to exploit Nitinol in the most beneficial way.

This process was exploratory. The observation of Nitinol and every other experiments led to conclusions used for the next experiment. The process in this chapter is presented in a linear way however in between them there were several iterations. Example of the iterations are types of the used wires. Through the process it became obvious that some wires are not preferable to work with, hence other types were purchased. The same situation was with the settings of shape setting Nitinol samples. The perfect set was established after experimenting with many wires.

2.2. Methods

This section is dedicated to methods of investigating Nitinol's properties. Below, the used materials and equipment are described. It is followed by an explanation of the methods used together with chosen settings in order to test optimal parameters of Nitinol wires.

2.2.1. Materials

All test and research on Nitinol is conducted using wires from a selections of seven types. The wires differ in diameter, chemical composition, method they were produced, and transition temperature given by the supplier. The materials used for the investigation in this chapter are listed in table 2.1.

Table 2.1: Available Nitinol wires used in the research

No.	Name	Name	Diameter [mm]	Production Method	Trans. temp.	Composition
1.	SMA-SA-0.15	Flexmet	0.15	Straight annealed	60 °C	NiTi
2.	SMA-AD-0.2	Flexmet	0.20	As drawn	60 °C	NiTi
3.	Kellogs 0.25	Kellogg's Research Lab	0.25	Straight annealed	45 °C	NiTi
4.	Kellogs 0.4	Kellogg's Research Lab	0.40	Straight annealed	45 °C	NiTi
5.	FLEXMET 0.43	Flexmet	0.43	As drawn	60 °C	NiTi
6.	FLEXMET AS	Flexmet	0.50	Straight annealed	60 °C	NiTi
7.	SMA-CU-0.66-AD	Flexmet	0.66	As drawn	60 °C	NiTiCu

2.2.2. Equipment

Major role in shaping and investigating Nitinol has a furnace as the material has to be heated above 500°C. Used in this project is 1R14-L Electrical Muffle Kiln. It is a digitally controlled, electrical furnace especially designed to heat up to 950°C. Due to its small dimensions, the kiln heats up in 20-30 minutes to temperature above 500°C. The picture of the device is shown in figure 2.9. This oven is safe and easy to use. It does not get hot on the outside while being used.

Additional equipment used for shaping Nitinol was: steel plates and screws to keep the wire in desired position.

In order to test the activation temperature, a heating plate is used. Available device for this purpose is a hot plate IKA C-MAG HS 7 (in figure 2.10) with included temperature sensor. The sensor can be placed inside a container with certain liquid and the temperature is measured constantly. The heating plate warms up transferring the heat to the container placed over it. The display presents the temperature measured by the sensor.

The most common liquid to use is water however some samples might have activation temperature above 100°C or around. In those cases vegetable oil is used. The samples' activation temperature is measured by putting the wire to the liquid. The liquid is transparent so any changes in wire's shape can be noticed. Temperature of the liquid in which the sample changes its shape is the activation temperature.

2.2.3. Preliminary experiment A

Connecting Nitinol

Two methods of connecting the wires seem to be applicable and reproducible in the lab settings. One is mechanically clamping two ends of the wire in a metal tube; another solution is soldering using settings especially designed for Nitinol.

The purpose of this study is to find the stronger connection between the given two. In this experiment, the settings of connecting methods are tweaked to find more reliable option.

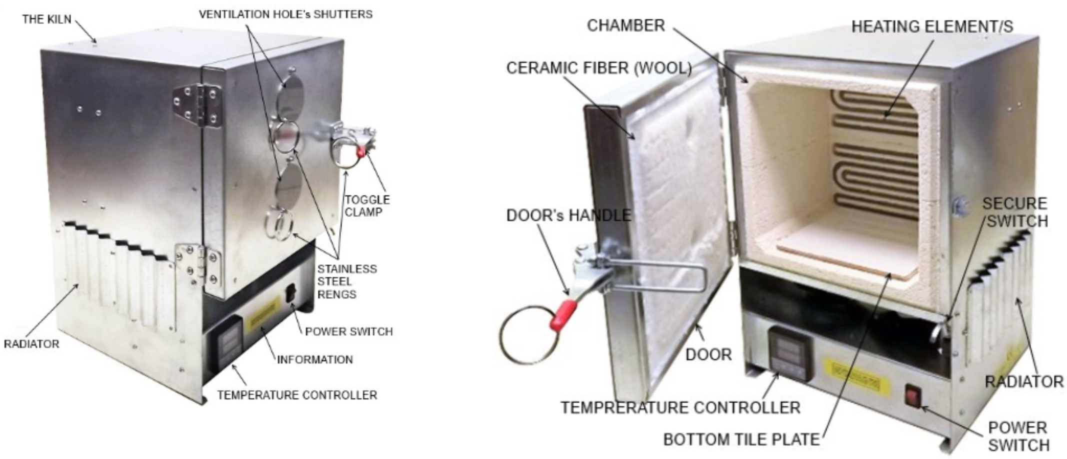

Figure 2.9: 1R14-L Electrical Muffle Kiln used for shaping Nitinol

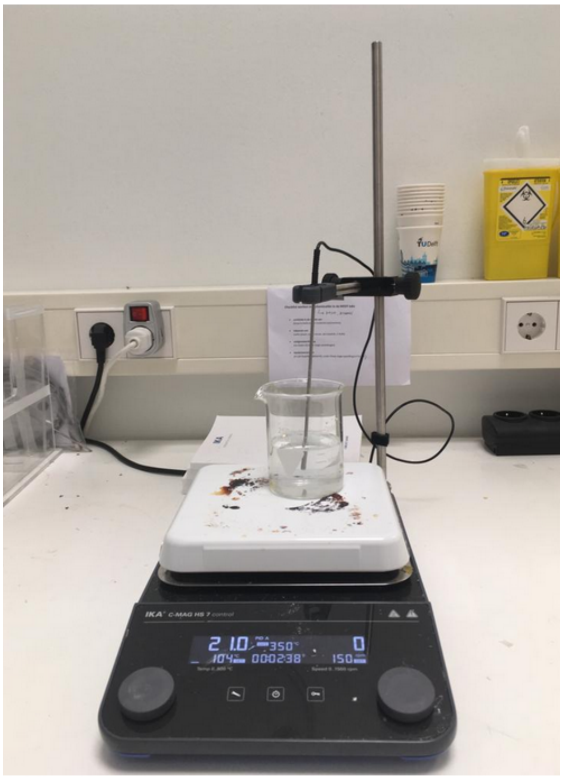

Figure 2.10: Hot Plate IKA C-MAG HS 7

Prepared samples are extended in radial direction to test just created connection if it can withstand applied force. The samples are widened by hands or pliers in case the sample's size prevents doing it by hands. The examined feature is resistance to detaching. The testing method seems unprofessional but it can give an initial impression of the strength difference between connection of two mentioned methods.

Crimping

Crimping is executed using an aluminum thin-walled tube and a crimping tool as shown in figure 2.11. It is done by pushing two ends of Nitinol wire to the tube and clamping it so the ends become restrained.

There are two methods to clamp together two wire ends. As shown in figure 2.12 cross sections of such connections. Those two methods are used in this section. The methods differs from each other with the contact area between the wire ends. The top scheme presents the case when the crimping tube's inner diameter

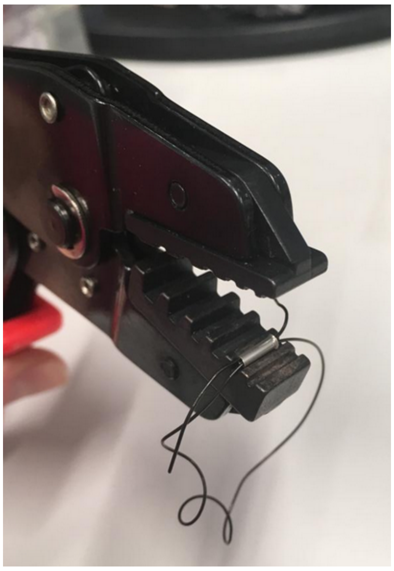

Figure 2.11: An example of connecting the Nitinol wire into a closed structure using crimping tool and thin aluminum tube

has a size close to an outer diameter of the wire. As a result, the two ends of the wire are placed inside the tube facing together and then squeezed inside the metal tube. The bottom scheme presents the case in which the inner diameter of crimping tube is at least twice as big as wire's outer diameter. It results in the connection where the two wire ends are deformed over each other and the connecting area is bigger.

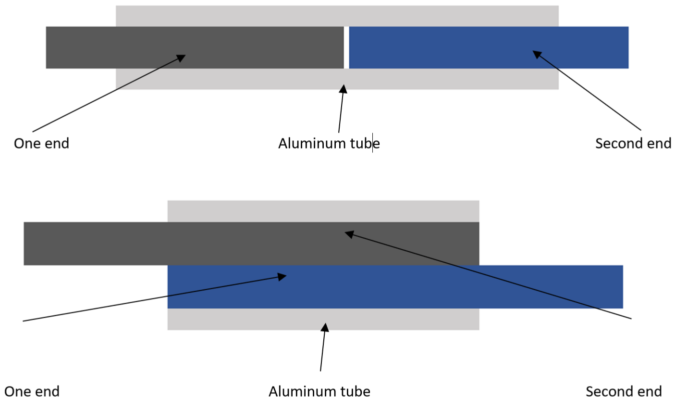

Figure 2.12: Two methods of crimping two ends of Nitinol wire depends on contact area between the ends

During this study two types of aluminum tubes were used: one of inner diameter of 1.4 mm and another one 0.9 mm. Both have wall thickness of 0.2 mm.

Soldering

Soldering is done using a soldering iron and material called solder. In principle the solder is heated up and melted. Then the ends of Nitinol sample are embedded into melted material. Once the solder solidifies, the sample creates a closed circuit. The materials and machines used for soldering purpose are:

- Soldering Iron (temperature 350°C) (as shown in figure 2.13);
- Solder: Tin and Silver (97/3);
- Nitinol coated with Copper;

Nitinol is coated with copper for the ease of soldering. As mentioned before, in order to attach molten solder over Nitinol wire, the top titanium oxide layer has to be removed. To prevent that step, Nitinol coated with Copper is used for this study. It is expected to provide a good connection between the solder and Nitinol.

Figure 2.13: Soldering Iron

2.2.4. Preliminary experiment B

Shaping Nitinol into unusual form

The ease of pre-shaping Nitinol occurs as long as the desired shape into which Nitinol is shaped is a coil. Winding up the wire around a screw and fixing it does not seem problematic. However, other shapes, especially flat ones cannot be made by this method. To solve the problem, an idea of pre-shaping Nitinol in plaster together with 3D printed parts, arose.

In the study of University of Washington [16], shape training of Nitinol is executed using 3D printed fixtures. Used printing material is stainless steel as the material can bear high temperatures. Due to lack of an access to a metal 3D printer as well as high costs of commercially purchasing stainless steel fixtures, polymer based fixtures were chosen to be used.

Rapid prototyping and quick prints are ideal for shaping Nitinol wire into unusual and complex shapes. However, commonly used PLA in filament based 3D printer, weakens already at 60°C. The problem of molten fixture and the same time, unfastening Nitinol can be solved by embedding the wire on a fixture into plaster.

Plaster in liquid state can be shaped into anything and in solid state it is hard and strong enough to keep Nitinol wire in a desired position. Moreover, plaster can withstand heat including temperatures as high as ones used for shaping Nitinol.

During heating process it is expected that the plastic fixture melts but the plaster surrounding keeps the wire in desired position.

To study the possibility of using such method several samples were made and examined for their convenience to shape Nitinol.

Fixtures are printed on 3D printer: Ultimaker 2+ Extended using PLA (polyactic acid) as the material. Next, Nitinol is wound around the fixture and embedded into plaster. The plaster block is placed inside the oven for the shaping purpose. After required time, the plaster block is cooled down and broken into pieces to release the Nitinol sample.

Figure 2.14 presents examples of 3D printed fixtures with Nitinol wound arund them. And in figure 2.15 the plaster blocks containing the PLA fixtures are shown.

2.2.5. Experiment I

Influence of time and temperature

Since the literature and other sources do not provide one specified set-up parameters to shape Nitinol, an additional test has to be performed. This test is executed in order to establish the influence of time and temperature of the shaping process on the activation temperature of the sample.

Twenty samples are prepared of Nitinol wire number 2 (SAM-AD-0.2) to be tested. Two conditions, time

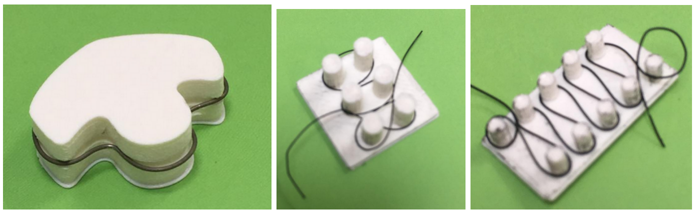

Figure 2.14: Constraining Nitinol samples on a screw

Figure 2.15: Constraining Nitinol samples on a screw

and of temperature, are variable. The time of the heating process varies between 5 and 10 minutes, and the temperature between 500°C and 550°C . It results in four conditions:

- Condition 1: temperature 500°C and 5 minutes of shape setting;
- Condition 2: temperature 500°C and 10 minutes of shape setting;
- Condition 3: temperature 550°C and 5 minutes of shape setting;
- Condition 4: temperature 550°C and 10 minutes of shape setting;

For each condition five samples were prepared which results in twenty samples made for the experiment.

The sample coils are made by wrapping the wire around an M3 screw and constraining them with bolts and nuts on a steel plate as shown in figure 2.16 placed below. After the oven time, the samples are placed in cold water.

After the shaping process the samples are tested with a use of water and oil on the heating plate. Each sample is placed inside a glass beaker filled with liquid. The heating plate is switched on to start the warming process. The thermometer is placed in the liquid and its temperature is displayed in real time. The temperature, in which the sample is completely converted to its final state, is recorded.

2.2.6. Experiment II

Influence of pre-tension

In order to investigate the influence of pre-tension on the wire samples during the shaping process twenty samples are used. Those are the same twenty samples as ones used in the Experiment I in subsection 2.2.5. They are made of wire number 2 and are made according to mentioned before method - i.e. using the settings of time and temperature of 5 and 10 minutes at 500 and 550°C. For this specific case, additionally the method of constraining the samples is described. There are five samples made using the Method 1; eleven samples using the Method 2; and four samples using the Method 3.

There are three different ways of pre-tensing the samples while fabricating them:

- Method 1: The sample is constrained by screws and nuts, as shown in figure 2.16;
- Method 2: The sample is constrained by screws and nuts and additionally the wire is pulled through holes, as shown in figure 2.17;

- Method 3: The sample is constrained by nuts on the screw, as shown in figure 2.18;

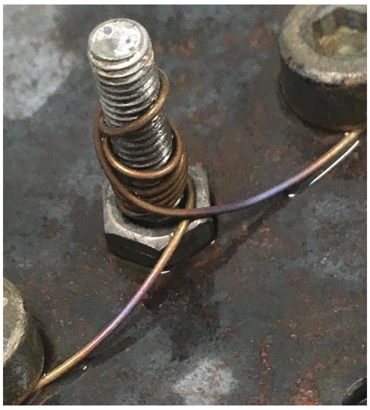

Figure 2.16: Method of constraining Nitinol samples by screws and nuts

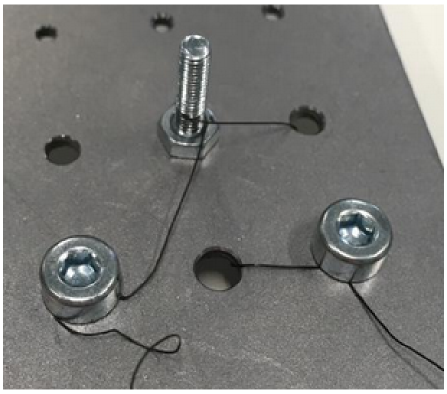

Figure 2.17: Method of constraining Nitinol samples by pulling it through holes

Figure 2.18: Constraining Nitinol samples on a screw

The methods are tested in order to detect if the constraining manner has any influence on the final result. There are six samples made using the method 1 (sample is constrained by screws and nuts), nine samples made using method 2 (sample is constrained by screws and nuts and additionally the wire is pulled through holes) and five samples made using method 3 (sample is constrained by nuts on the screw).

For this experiment the following outcomes are inspected: the activation temperature and ability to form a tight coil while heated. The activation temperature is tested using a hot plate. Each sample is placed inside a glass beaker filled with water or oil. The heating plate is switched on to start the warming process. The thermometer is placed in the liquid and its temperature is displayed in real time. The temperature, in which the sample is completely converted to its final state, is recorded.

The ability to form a tight coil is inspected visually. The oil in a glass beaker is heat up to 110°C - temperature higher than activation of individual samples. Each sample is placed in the beaker and its state is examined.

2.2.7. Experiment III

Difference between "as drawn" and "straight annealed" wires

Even though the supplier (Flexmet) of Nitinol wires announces the same value of transition temperature for both: "as drawn" and "straight annealed" wires, there are some differences. In order to confirm the supplier's assumption, additional test with SA (straight annealed) wire is conducted to compare the results with the outcome of the Experiment I section 2.2.5 executed with AD (as drawn) wire.

Four samples are made using wire number 6 of diameter 0.50mm. The wire is wound around a screw forming a coil as the samples in experiment I. The samples are constrained using the method 1 of fixing the wire on a plate with a use of nuts and bolts. Only one condition is used to shape the samples: 500 °C and 10 minutes of heat treatment in the oven.

The samples are tested for their activation temperature using the heating plate and the procedure is the same as for experiment 1. After the shaping process the samples are tested with a use of water and oil on the heating plate. Each sample is placed inside a glass beaker filled with liquid. The heating plate is switched on to start the warming process. The thermometer is placed in the liquid and its temperature is displayed in real time. The temperature, in which the sample is completely converted to its final state, is recorded. In order to compare the AD and SA wires, the average activation temperature of samples from this experiment is compared with the average activation temperature of five samples of condition 2 from the experiment 1.

2.2.8. Experiment IV

Difference between suppliers

Results from experiments I, II and III can already give some indication on conditions needed to train the Flexmet wires. In this experiment, the Kellogg's wires are tested. Twelve samples made from Nitinol wire number 4 (Kellogs 0.4) of diameter 0.4 mm, are prepared and trained in three different temperature settings and two time settings, thus six conditions. For each condition there are two samples to be tested. The time is 5 and 15 minutes and temperature is 400, 450 and 500°C.

- Condition A: 400°C and 5 minutes of heat treatment;
- Condition B: 400°C and 10 minutes of heat treatment;
- Condition C: 450°C and 5 minutes of heat treatment;
- Condition D: 450°C and 10 minutes of heat treatment;
- Condition E: 500°C and 5 minutes of heat treatment;
- Condition F: 500°C and 10 minutes of heat treatment;

After the shaping process the samples are tested with using water and oil on the heating plate. Each sample is placed inside a glass beaker filled with liquid. The heating plate is switched on to start the warming process. The thermometer is placed in the liquid and its temperature is displayed in real time. The temperature, in which the sample is completely converted to its final state, is recorded.

The purpose of this experiment is twofold. First, it enables the comparison between wires provided by Flexmet and by Kellogg's. The comparison is done by compiling the average activation temperature of Flexmet samples based on results from experiment III in subsection 2.2.7 for straight annealed wires and the average of results of condition E and F of this experiment. As a result the values of activation temperature of samples shaped at 500°C will be compared. The second purpose is to investigate the activation temperature of samples made from Nitinol number 4 depending on time and temperature of shaping.

2.2.9. Experiment V

Influence of aging

One of the methods of influencing the activation temperature is additional aging. The literature provides an aging temperature of 375°C [17] and this setting is used.

Six samples are made of Nitinol number 4 in various shapes. They are trained at 450°C for 10 minutes followed by quenching in ice cold water. Then each sample is studied to obtain its transition temperature. Both austenite start (A_s) and austenite finish (A_f) temperature are collected. As before, the equipment used to measure temperature is the heating plate. A glass beaker is filled with water or oil and placed on the plate. The thermometer is put to the liquid to display the liquid's temperature. The heating setting is switched on. Each sample is placed in the beaker and closely observed. The first moment of transitioning is referred to as the beginning of the state's change from martensite to austenite. Thus, the temperature in which small changes are noticeable is recorded as (A_s) temperature. The liquid is constantly heated to the moment when the sample has undergone the entire transformation of the shape. The temperature in which the remembered shape is complete is recorded as (A_f) temperature.

Then the samples are annealed at 375°C without being constrained. Three samples are aged for 60 minutes and other three for 90 minutes. After the aging process, the samples are cooled down in ice cold water.

The procedure of measuring the (A_s) and (A_f) temperature is the same as before the aging.

2.3. Results

In this section the results of tests described in section 2.2 "Methods" are presented. Each particular experiment is placed in a separated subsection.

2.3.1. Results of preliminary experiment A

Connecting Nitinol

Connecting Nitinol by crimping gave very positive results unlike the soldering. The samples which were made by crimping resisted the stretching and remained connected. Picture of example of crimping connection is shown in figure 2.19.

Figure 2.19: Nitinol sample connected using the crimping method - two ends are clamped together inside an aluminum tube

The soldering process was found to be very challenging. The heat applied from the soldering iron was transforming Nitinol sample into its austenite shape, so the sample could not be placed in a form to ease the connecting process. Furthermore, the solder did not stick to Nitinol easily. The wire ends had to be hold within the solder for about 60 seconds till the solder solidified and hold the pieces together. As the result, the connections became big and bulky (more than for the crimping method). The connections did not pass the stretching test and solder connections detached from the wire. Figure 2.20 presents the results from connecting Nitinol by soldering.

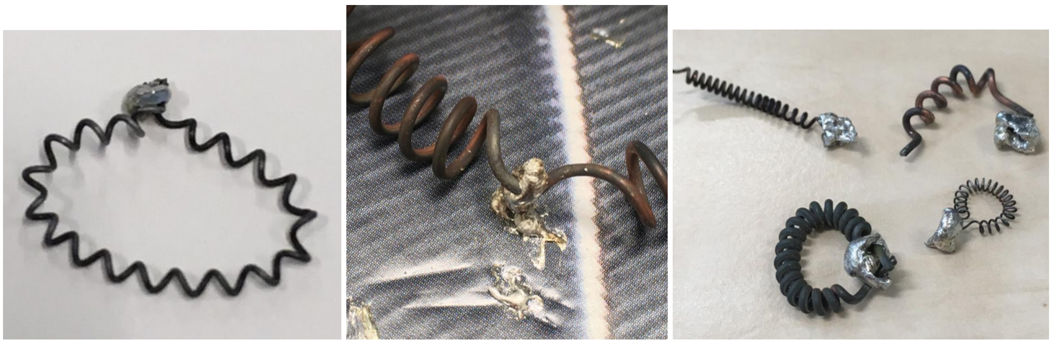

Figure 2.20: Nitinol sample connected using soldering - results are bulky and also very weak

2.3.2. Results of preliminary experiment B

Shaping Nitinol into unusual form

The study on shaping Nitinol using 3D printed moulds and plaster casting resulted in a design of robust method to prepare prototypes. Several trials using variations of time and temperature led to finding the process settings in which the shaping can be used.

Below in the figures results of the study are presented. In figure 2.21 and 2.22 two failure modes are shown. Picture in figure 2.21 plastic leaked through the cracks in plaster block. And in figure 2.22 the polymer mould did not melt and was still present when breaking the plaster block.

The figures 2.23 and 2.24 present the successful results of shaping Nitinol. There are two cases when the PLA moulds melted and the wire stayed in desired position.

Figure 2.21: Failing result of shaping Nitinol - leak of PLA

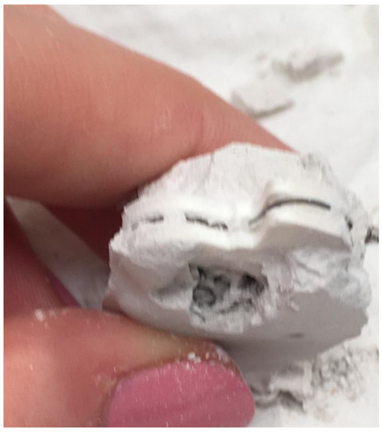

Figure 2.22: Failing result of shaping Nitinol - PLA not melted

Figure 2.23: Favorable result of shaping Nitinol

Figure 2.24: Nitinol sample after PLA melt and release from a plaster block

2.3.3. Results of experiment I

Influence of time and temperature

Sample number 19 got destroyed and could not be tested, so only nineteen samples in total were tested.

Figure 2.25 presents the results of the experiment I. The activation temperature is plotted base on the condition in which the samples were shaped. Many points are overlapping. The results are given for nineteen samples, however the plot shows only nine points. The lowest median of 83°C together with the lowest activation temperature were recorded for the samples shaped for 10 minutes at 500°C. The rise of the activation temperature to 85°C is followed by the samples shaped also in 500°C but for 5 minutes. The samples which were shaped in 550°C demonstrate higher activation temperature of 87°C for samples shaped for 5 minutes and 91°C for samples shaped for 10 minutes. For the samples shaped at 500°C , activation temperature decreases with the shaping time, while for the samples shaped at 550°C activation temperature rises with increasing the shaping time. For both temperature groups, the condition of longer shaping time (i.e. 10 minutes) results in smaller divergences of the recorded values.

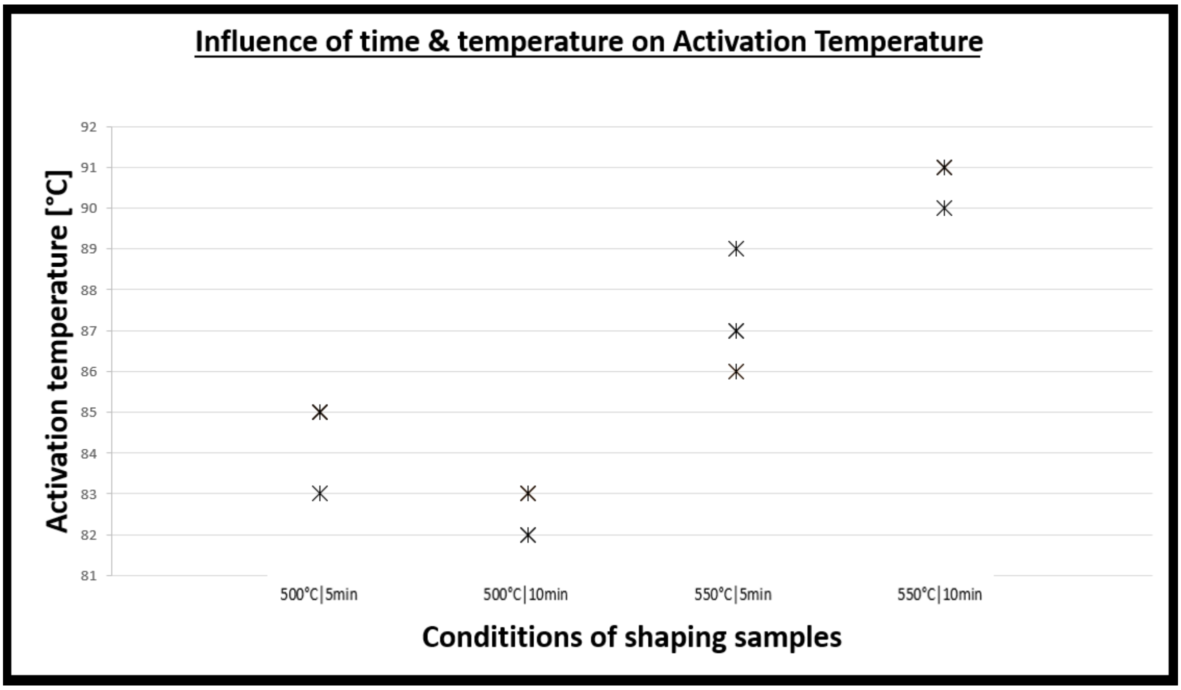

Figure 2.25: Results of experiment I plotted as activation temperature based on conditions of shaping Nitinol

2.3.4. Results of experiment II

Influence of pre-tension

In figure 2.26 the results are plotted to show the differences between activation temperatures based on the method of restraining the samples. The horizontal line drawn through bar diagrams represent the average activation temperature for each condition. The activation temperature values are the A_f temperature when the sample underwent the shape transformation completely. Recorded data for this experiment is listed in a table in appendix A.

The constraining methods used are: "Plate" which corresponds to a constraint by screws and nuts on a plate; "Holes" means a constrain by screws and nuts on a plate with additional pulling wire through the holes; and "Screw" describes samples made by winding the wire around a screw and constraining it with nuts.

For all four condition groups the activation temperature is the highest for the samples constrained on a screw. In all cases, activation temperature is also higher than the group average. The lowest activation temperature is recorded for the samples constrained on a plate by the use of bolts, nuts and additionally pulled through the holes, in three condition groups 1,2, and 3 - 500°C and 5 minutes; 500°C and 10 minutes; and 550°Cand 5 minutes.

At the visual inspection one sample did not coil back. It was the sample number 4 shaped for 5 minutes in 500°C, and constrained using the method of the plate together with pulling the wire through the holes.

2.3.5. Results of experiment III

Difference between "As drawn" and "straight annealed" wires

The median activation temperature of 68.5°C obtained from testing samples made of SA Nitinol wire is compared with the median activation temperature of samples made of AD Nitinol wire - 82.5°C. Both sets of samples were prepared using the same method as explained in section 2.2.7. Samples made of SA Nitinol wire exhibit lower activation temperature of about 14°C when the median values are compared.

2.3.6. Results of experiment IV

Difference between suppliers

The plot in figure 2.27 presents the results of the experiment IV. Two functions are plotted for the Kellogg's wires: one for the samples shaped for 5 minutes and one for samples shaped for 10 minutes. Additionally, the median activation temperature (68.5°C) of Flexmet samples shaped at 500°C for 10 minutes is added to the graph. Two dotted lines are the indications of activation temperature given by the suppliers. 45°C is given for

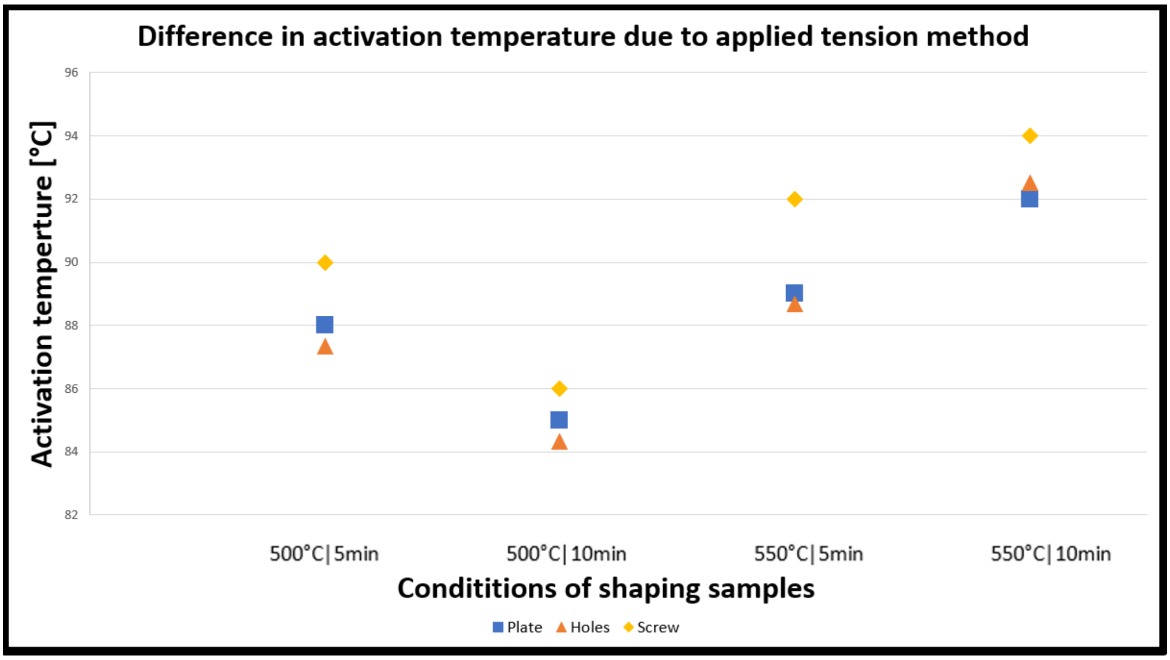

Figure 2.26: Results of experiment II plotted as activation temperature based on conditions of constraining samples

the Kellogg's Nitinol and 60°C for Flexmet Nitinol.

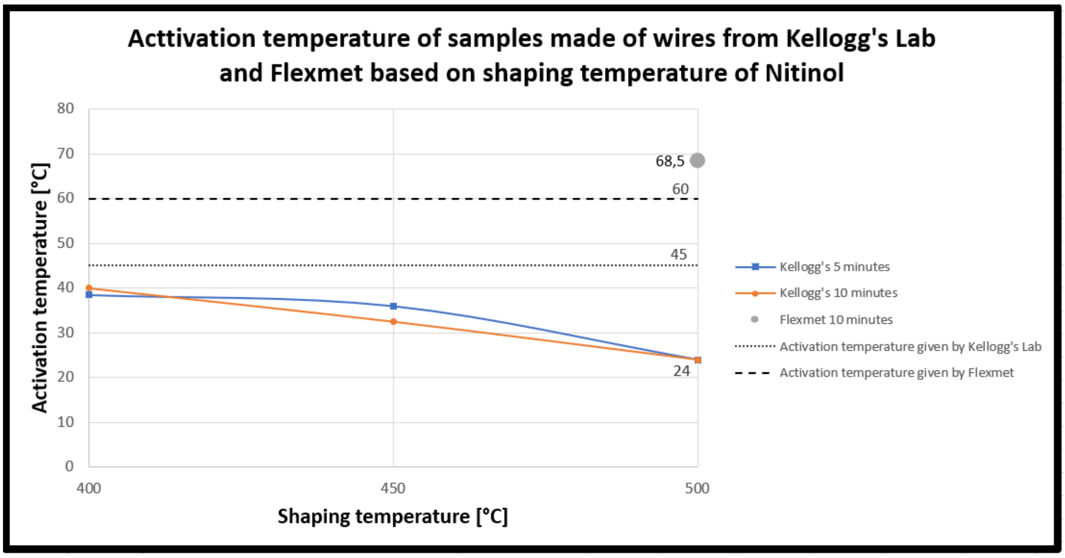

Figure 2.27: Results of experiment IV showing the difference in activation temperature between Nitinol supplied by Kellogg's Research Lab and Flexmet as a function of shaping time and temperature

Kellogg's samples shaped in 500°C resulted in activation temperature equal about 24°C which expresses room temperature. Those samples in room temperature could not be deformed and stayed in their pre-shaped forms. Samples shaped at 400 and 450°C could be deformed but only slightly.

For shaping temperature of 500°C the difference between measured activation temperature and specified by the supplier, is bigger for the Kellogg's samples and equals about 21°C . For Flexmet samples that difference is about 8.5°C . However, Kellogg's samples shaped at lower temperatures of 400°C have activation temperature close to specified 45°C.

2.3.7. Results of experiment V

Influence of aging

Figure 2.28 presents the results of experiment V. There are two variable conditions: the time of aging and the oorder of taking the measurements (ie. before and after th aging process). The plot presents two groups of results according to the used condition. For the samples aged for 60 minutes, only one sample shows a change in activation temperature after the heat treatment. This temperature rose from 35°C to 38°C. Remaining two samples did not exhibit any change in their activation temperature. In the group of 90 minutes aging, two samples had their activation temperature changed. For both samples the temperature dropped, from 40°C to 39°C for one sample, and from 43°C to 41°C for the other. For one sample no difference was recorded.

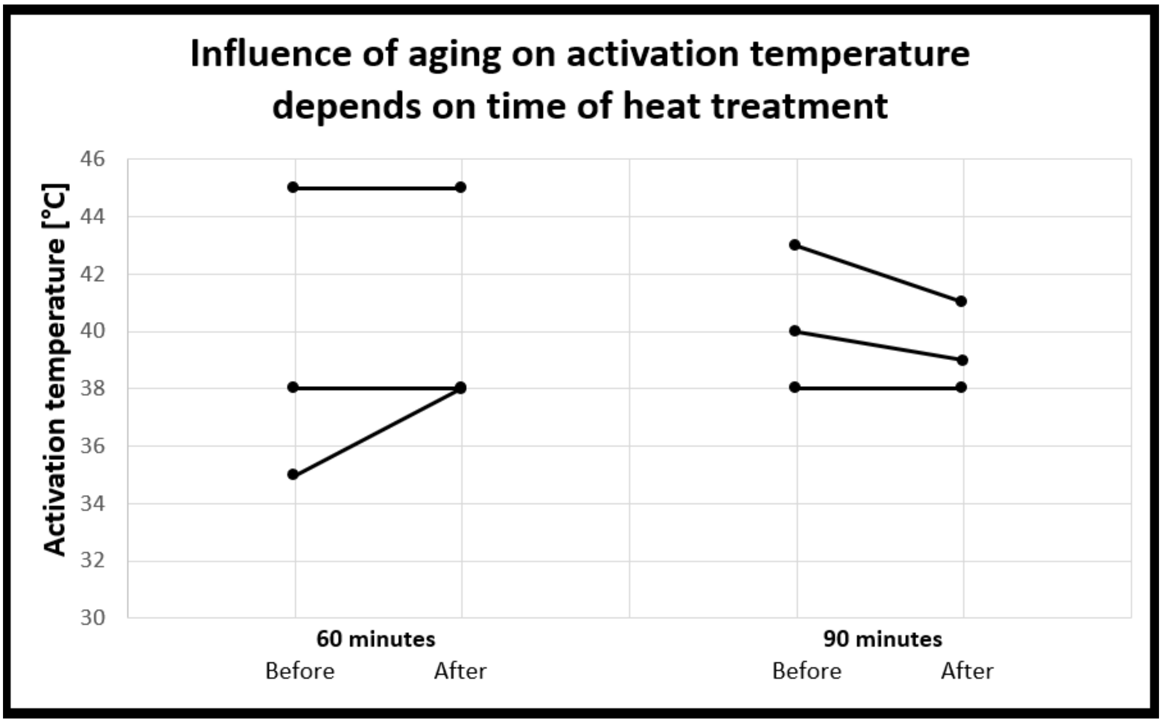

Figure 2.28: Results of experiment V showing the influence of aging on activation temperature depends on duration of aging

2.4. Discussion

Several tests were carried out in order to gain the most knowledge in Nitinol subject and also to distinguish the most best settings for handling Nitinol. This is very beneficial for development of the project of a sterilization device. Nitinol is chosen as the project's main material and the way of shaping it and the settings chosen for the process can noticeably contribute to the design process. The direct aim was to answer several research questions asked at the Nitinol introduction section 2.1.3.

2.4.1. Preliminary experiment A

Preliminary experiment A of connecting Nitinol resulted in better performance of crimped samples. This method is simple and reproducible. Soldering method failed mostly due to adjustments to the process, i.e. avoiding the use of flux and replacing the electrical current to soldering iron as the heat source. The electrical current can be used directly on a wire and by heating it, the solder can melt and connect two parts. With soldering iron, the solder is melted and packed around two wires which results with very poor adhesion of the solder. Moreover, the addition of copper over the Nitinol wire did not simplified the process. The solution of copper coating is not sufficient for soldering Nitinol. The crimping method is chosen for a prime method of connecting Nitinol in this study.

2.4.2. Preliminary experiment B

Preliminary experiment B on shaping Nitinol into complex structures resulted in finding the method of using 3D printed parts and plaster. Several requirements must be fulfilled to achieve a desired effect from the method.

1. Moulds must have possible smallest volume, as they occupy the space within a plaster block. Once the plastic melts, Nitinol wire can unwind at the empty space. The PLA melts earlier than the wire is shaped thus, it still tries to straighten when the empty space is created.

2. Using the ratio between plaster and water of 2:1, the time of hardening the material decreases and it is maximum ratio specified by the supplier to use. Also, keeping the equal amount of plaster and water for each sample, results in equal used volume of material isolating the sample. When the isolation layer is equal, the same shaping settings can be applied.

3. This requirement is related to the previous one (number 2), as using the similar size container provides similar sized plaster block.

4. Even when maximum plaster to water ratio is used, plaster block must be thoroughly dried. When too much moisture is trapped inside, the block brakes during the heating process. As shown in figure 2.21, the block ruptured, and PLA leaked through the gap.

5. This point is related to the previous one. Additional drying of the sample in the oven ensures that there is no more moisture left.

6. Sample in a block of plaster of a volume mentioned in previous points, needs 60 minutes to be shaped. Due to the isolation made of plaster, the additional time is required in comparison to the bare wire when 10 minutes of shaping is enough.

7. Sample must be cooled down and it is achieved the quickest using ice cold water for the quenching effect which is the requirement for successfully shaping Nitinol wire.

2.4.3. Experiment I

The first experiment on the influence of temperature and time of training Nitinol on activation temperature gave the first idea of upcoming challenges. The shaping settings of time and temperature indeed influence the final activation temperature of prepared samples. The relation is based only on four conditions of two sets of temperature and two sets of time. Taking into account two temperature values of 500 and 550°C, the higher the shaping temperature the higher the final activation temperature. For the used sets of time the pattern is not preserved, as in case of 500°C longer time results in lower activation temperature and in case of 550°C longer time results in higher activation temperature. The cause of those results seems to be as follow: 500°C is more suitable shaping temperature for the used wire. This statement can be based on the fact that the specified activation temperature for this wire is 60°C. The median activation temperature for the samples shaped in condition of 500°C and 10 minutes is 83°C, which is the lowest from the achieved measurements. Then, if 500°C is the more favorable shaping temperature than 5 minutes suggests to short time to achieve desired activation temperature. As the 550°C is too high for shaping the wire, the shorter time of 5 minutes results in lower activation temperature, closer to the specified one. For further experiments and studies it indicates those settings as currently most appropriate.

2.4.4. Experiment II

In each of condition group for this and previous experiment, there were three types of used constraints method. By looking individually on each group it is seen that the activation temperature value is dependent on the constraining method. In each group the sample constrained on a screw had the highest activation temperature. The most optimal results are the lowest ones, as the activation temperature should be as close to 60°C as possible thus the screw method gives the worst outcome. Already when preparing the samples, the wire constrained by this method seemed to be the loosest. Consequently, it can be said that the least tensed wire results in the activation temperature the furthest from desired (in a relation to its condition group). In case of this study it means the temperature the highest from the desired, but not enough data is collected to prove the hypothesis. For different shaping settings, the loosest wire could have had the lowest activation temperature if all the results were below it. The wire fixed on a screw ended up the loosest due to two reasons. One is the difficulty of securing the wire between two bolts as they had to be tighten in relation to each other only and any other fixed point. The second reason is the small area of the nuts. The wires could not be wound multiple times at the fixing point which also could decrease the tension of the whole wire. In three out of four trials, activation temperature was the lowest (regarding to the condition group) for samples constrained using the plate and pulling it through the holes. It seems that those samples were the most tensed as the additional pulling the wire gave them additional positioning.

2.4.5. Experiment III

After analyzing the results from experiments I and II, it became clear that achieving 60°C activation temperature from that wire was difficult because the obtained results were far from that number. Flexmet recommends that the user will straight anneal the wire on its own. To prevent that extra step straight annealed wire, also from Flexmet, was used and compared with the as-drawn wire. The samples made of the SA wire have the average transition temperature of 68.25°C which is so much closer to given 60°C and also about 14°lower than previously obtained values. Straight annealed wires are preferred for the further use.

2.4.6. Experiment IV

Comparison between two suppliers: Flexmet and Kellogg's Research Labs was caused by two reasons. One was that the result obtained so far on activation temperature were not at 60°C, and two that lower (even than 60°C) activation was wanted. Lower activation temperature benefits the ease and safety of testing the samples. Water in which the samples are transforming does not have to be as hot as in the range of 60-95°C, or oil of temperature above 100°Cdoes not need to be used. It facilitates the tests and prevents a tester from burns. Kellog's Reserach Lab provides wide selection of Nitinol and additional material was ordered from it. New wire number 4 in table 2.1 had to be tested if it gives similar results as the wire from Flexmet.

Firstly, the shaping conditions were added at this test to examined the influence of decreasing the shaping temperature. Those changes were chosen based on results from experiment I where 500°Cthe lower temperature limit. Below this value the activation temperature outcome was unknown.

The comparison between two suppliers could be done for the condition of 10 minutes at 500°C. In these settings, the Flexmet wires resulted in activation temperature closer to one specified by the supplier i.e. 60°C, when the measured one was 68.5°C. The Kellogg's wire had larger difference between measured and specified activation temperature. Supplier ensured 45°C, and measured was room temperature (given here as 24°C of the environment temperature in the lab that day). Thus at the setting of 500°C and 10 minutes, the Flexmet wire resulted better.

However, adding extra conditions during this test, provided with an additional information on the Nitinol behaviour at lower temperatures and longer shaping time. The samples shaped in 400°C had the activation temperature the closest to specified 45°C; 38.5°C and 40°C for shaped in 5 and 10 minutes respectively. The resulted activation temperature decreases to 36 and 32.5°C when the shaping temperature increases to 450°C. Hence for the Kellogg's wires, the shaping temperature has to be reduced to get the outcome the closest to the value specified by the supplier.

The aging test as experiment V was conducted on samples which turned out to have activation temperature lower than specified by the supplier, i.e. their activation temperature was in a range between 35 and 45°C when 45°Cshould be a norm. The samples were aged in order to increase the activation temperature to 45°C for all the samples. Two conditions of aging time gave two different results - aging in 60 minutes resulted in two samples without any change in activation temperature and one sample with an increased on. Two samples aged for 90 minutes had their activation temperature lowered after the process, and one sample did not show any change. The sample population was very small for this test however in case of rising the activation temperature, settings of 90 minutes at 375°C will be used.

2.5. Conclusion

The study on Nitinol resulted in number of recommendation to follow while preparing prototypes in further studies. Chosen settings are as follow: 500°C and 10 minutes of shaping process for a bare Nitinol wire. The method of pre-tensing the wire by use of screws and nuts on a plate with additional pull through the holes is chosen to be used for further experimentation and prototyping since it gives the best results. Straight annealed wires are preferred for the further use over the as-drawn wires. Wires provided by Kellogg's Research Lab give lower activation temperature hence they are more favorable to work with. Settings of 90 minutes at 375°C are recommended for aging process to increase activation temperature. The crimping method is chosen as a prime method of connecting Nitinol prototypes. Unusual or complex shapes can be made using the method of 3D printed moulds and plaster but the process has to follow requirements found in this study via experimentation.

3

Test set-up

3.1. Introduction

This chapter presents the work done in developing a testing set-up which can serve to verify the design of a sterilization device. The main purpose of the device is to stop the flow through the Fallopian tubes by mechanically clenching over the tubes. As the device clamps over the structure it applies certain force. The force to close tube's lumen successfully is known based on the research done by the author of this report for Opt Medical at the end of 2018 during an internship. During that research, a synthetic Fallopian tube was designed and tested in order to mimic the human tissue. The tube is made of silicone of the hardness close to the real Fallopian tube. There is a lack of studies in literature on mechanical properties of Fallopian tubes, thus a group of gynecologists surgeons evaluated the synthetic tube. They decided it resembles the human tissue. An experiment was conducted in order to assess a force needed to close its lumen. It resulted in a mean value of 3.55 N as a force to be applied on such a tube to stop the flow through its lumen [18]. This chapter is dedicated to work on a design and development of a set-up to measure the force exerted by the clamping mechanism. The right principle has to be chosen in order to provide the best method to examine the prototypes and final design of a sterilization device.

Existing solution

There is an available solution for testing materials in several directions. Such an equipment is Biaxial Testing Machine of Biomaterials developed by Zwick/Roell and it is available in biomaterials lab of KU Leuven - a univeristy in Belgium. It consists of four linear drives, controlled independently of each other in terms of position, force, or strain, which are integrated in the system. Force measurement is via load cells, two each in the X and Y directions. Figure 3.1 presents the configuration of that testing machine.

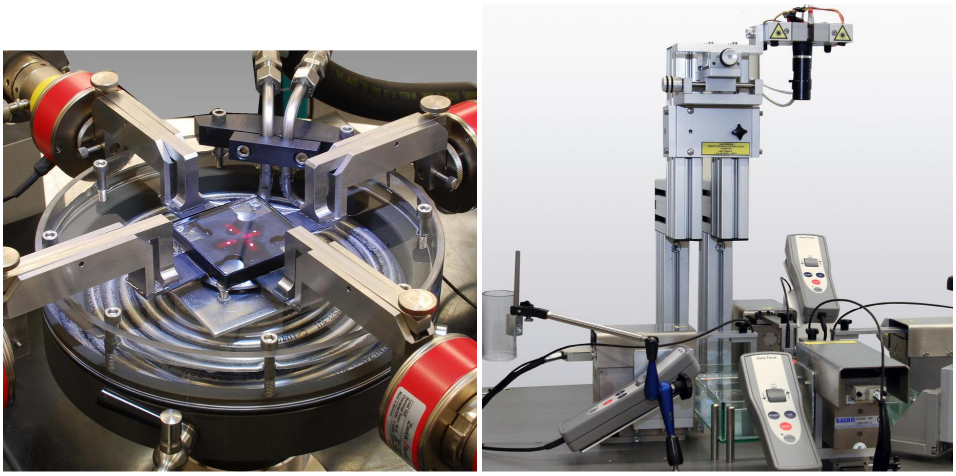

Figure 3.1: Zwick/Roell Biaxial Testing Machine for Biomaterials [19]

Requirements
Test set-up to be valuable to the project must be an easy and cheap alternative to multi axial set-up which can measure forces in different directions, which also is connected to a heated water tank and has high accuracy. The forces have to be measured in multiple directions as the clamping mechanism might affect the Fallopian tube from each direction around it. The feature of heating up the sample is necessary as the Nitinol transition appear with the increase of temperature. An important function is also that the set-up can be adjusted as the samples vary in shape and dimensions. Measurements in multiple directions in one plane have to be taken in order to get reliable indication about the forces that the samples can exert.

The requirements that the set-up has to meet are:

- measure the force in multiple directions
- be adjustable to various sample dimensions
- be suitable to keep the measured sample in a fixed position
- be suitable for heating up measured samples

Required force
The force exerted by the sterilization device must be higher than the force of the Fallopian tube which enables it to stay open. The Fallopian tube is round and there are infinitely many points in which the force can be measured i.e. the circumference of the tube. Since that is not possible, the measurement can be done in only few points. Figure 3.2 presents a schematic illustration of a tube on which certain pressures are acting. With P_i inside the tube there is intraluminal pressure marked, P_t is the tissue pressure and h indicates the thickness of the walls. The exerted force by the contraceptive device must overcome the P_i. None of the above is known thus the final design must be capable of stopping the flow in a synthetic Fallopian tube with geometry and material imitating the real tube which is mention in other part of this report.

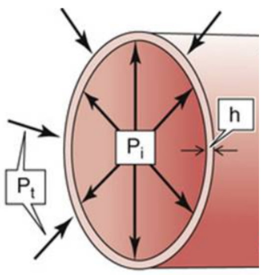

Figure 3.2: Scheme of a tube with intraluminal pressure acting on its walls [20]

3.2. Method

3.2.1. Concept

The concept of the test set-up is based on the Zwick/Roell Biaxial Testing Machine. A sample is placed in the center and it is surrounded by the units which provide the load and also can act as a measuring system. In the original Zwick/Roell machine, this function have the load cells. The alternative for them may be springs. The results of a brain storm for a system consisting of springs around a sample is shown in figure 3.3.

Conditions

The value which is measured during the shape changing of Nitinol structure is actually a displacement. To couple it with a force, the use of a spring seems to be the most beneficial. A spring is a common method to measure a force in a certain direction. A simple mechanism which contains a spring and indicating force is a spring scale as in figure 3.4. The expansion of the spring indicates the weight of a sample suspended on the hook which can be easily translated into force in Newtons.

The principle using the spring scales is as follow, the springs are pre-tensed to certain value and once the heat is applied on a Nitinol sample, the shape will change causing extra displacement of the spring. The

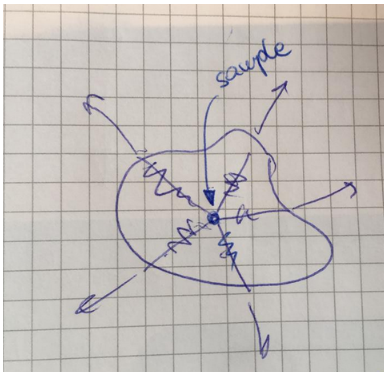

Figure 3.3: Scheme of distributing the spring scales around the sample in equal distance between them

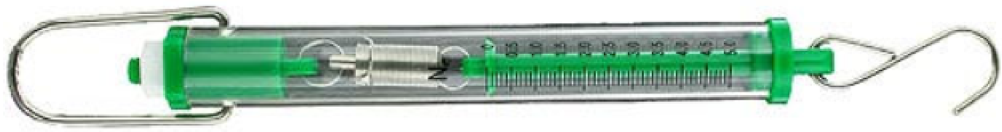

Figure 3.4: Spring scale

second scenario is that the shape will not change due to pre-tension equal or higher than maximum possible force exerted by a sample. According to this principle, the test set-up must be designed.

In reference to the requirements of the test set-up listed in section 3.1 those spring scales are the essence of the test set-up and around them the set-up is designed. But there are many other components needed in order to make the set-up workable and fulfilling the requirements.

To enable conducting measurements in several direction, the set-up must consist of multiple spring scales placed around the sample. The base for set-up can benefit from a circular shape so the angles between individual spring scales can be easily deducted. The spring scales have to be mounted in equal distance between each other and also in relation to the measured sample. The angle between the scales are equal (in case of five of them the angle in 72°.)

In order to pre-tense the springs, some kind of a holder or a pin must be added to keep the spring in desired position as it is stretched. As the samples have various dimensions, the starting position for each sample is different. The holder for pre-tensing the spring has to adjustable from sample to sample. Slot with adaptable holder is preferred.

The diameter of the wire used to manufacture the sample is below 1 mm. To fix the sample of such small dimension, very tiny hook, clamp, clip or a wire is needed. Connection between sample and the spring scale has to stay steady and inline so the measurement is reliable. Any small change in the line can cause the disturbance in the value read from a scale.

The remaining requirement concerns heating up the sample above transition temperature. It can be done using hot liquid, hot air or conduction. Conduction can be achieved by connecting the sample to a heating source by electrical network. However to have a uniform heat dissipation on a whole sample, the connections would have to cover the entire sample which could be influence the results of the test. By using hot liquid as water or oil, the test set-up needs to be water proof. Heat gun can provide high temperature hot air in seconds. Provided heat exceeds the required value to change samples' shape but this approach can guarantee that every sample receives sufficient heat to undergo the shape change.

Manufacturing
The prime methods to build such a test set-up are mechanical machining, laser cutting and 3D printing. The parts to be printed in PLA material are the components of the spring scales, connections between scales and clamps, positioning parts to keep the set-up and its components immobilized. They are shown in figure 3.5.

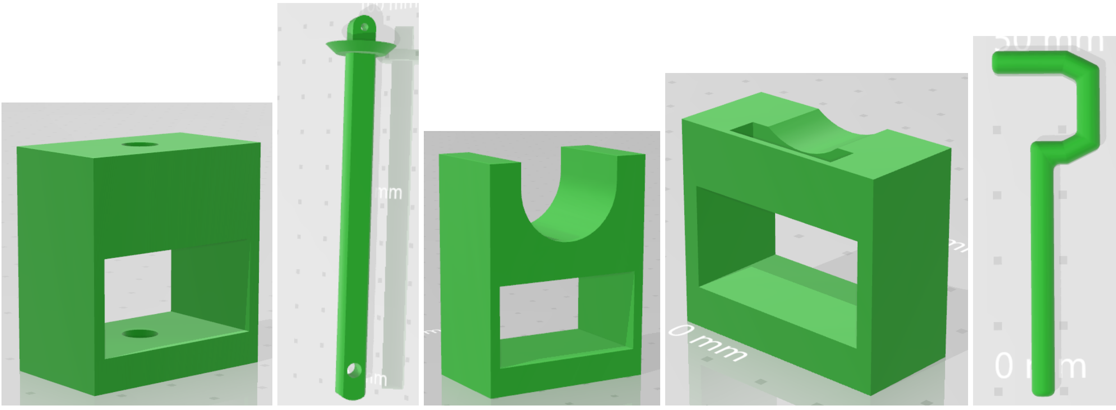

Figure 3.5: 3D printed parts of the test set-up

Machined parts are the aluminum arms which are the bases for the spring scales and allow to change the position of the springs and pre-tense them. Laser cut is the middle part of the set-up which connects the arms together to create one integrated system. The parts are connected using screws and nuts.

Two sets of parts: clips and spring scales are bought off the shelf. The spring calipers measuring up to 5 N each are purchased from OPITEC and clips from a brand MUELLER ELECTRIC.

The hooks connecting the spring scales with the clips are made of plastic. They are attached by mechanically squeezing the round metal end of the clip around the plastic hook. To reinforce this connection, additional clay is added to prevent the hook from melting when exposed to high temperatures and also to strengthen the connection. Used clay is an air-drying clay and it softens when it has contact with water. During the test, the samples are cooled down with ice, and it melts when touching hot metal. The water from melted ice can soften the clay. To prevent that, the PTFE tape is added around the hooks. It prevents water to get into contact with clay and also is heat resistant.

3.2.2. Validation Test
Before the test on prototypes is executed, the test set-up has to be validated. The sample is tested in three configurations of the set-up, ie. with three, four and five arms with spring scales on them to determine which configuration can be the most profitable for the set-up. In figure 3.6 the configurations are presented. There is an equal distance between the arms.

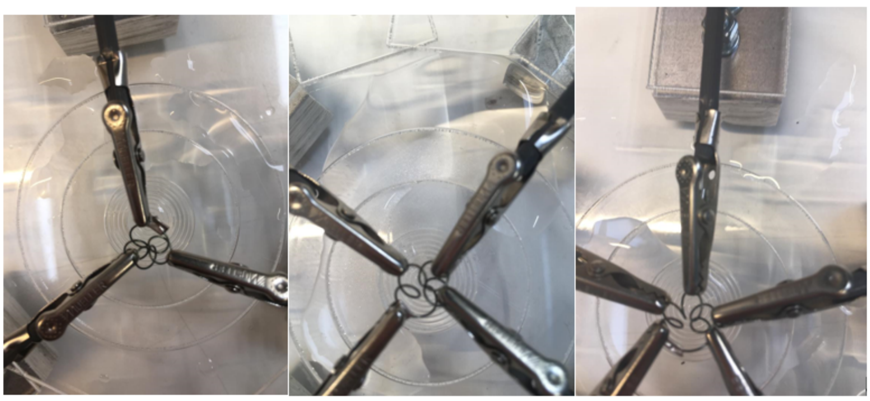

Figure 3.6: Illustration of a sample being tested in three different configurations of the test set-up: from left to right: with three, four and five arms

Force for each configuration is measured till the sample did not change to its trained shape while exposed to a rise of temperature or the sample comes back immediately after the heat is no more applied. The heat is generated with a heat gun. The used one in this experiment is Astro Pneumatic Tool® 9425 - Dual Temperature Heat Gun Kit, placed 3-5 cm above a sample. The temperature setting I is used (300°C). The exact procedure is as follow:

1. the sample is gripped by selected number of clamps in the most symmetrical way
2. the sample is deformed into most open state
3. spring scales are set to an equal pre-tension state while the sample is kept as centered on a plate (for the first measurement the the tension is set to 0 N); it is done by pulling the springs and fixating it at the distal end of an arm
4. the heat from a heat gun is a applied on the sample from above for 3 to 5 seconds
5. the values on the spring weights are recorded
6. the sample is cooled down with an ice cube

Next measurement is performed using the protocol but omitting the point 1 since the sample stays clamped. For each following measurement the value if each tension to which the spring scales are pre-set (point 3) changes by 0.5N. So if the first measurement was conducted with the scales set to 0N then for the second measurement they will be pre-set to 0.5 N on each arm, for third one they will be pre-set to 1 N and so on. Close to a maximum value in which the sample does not change its shape, the steps between measurements can be reduced to 0.1 N since this is the resolution of the spring scales. Thus if samples still returns to its hot state when the spring scales are set to 2N each, but do not returns when they are set to 2.5N the step size must be reduced. Somewhere between 2 and 2.5 N there is a maximum value of force to let the sample return to the hot state maximally. This value is recorded.

Two validation tests are planned to be executed to confirm the veracity of the results. In each test different sample is used. The samples differ in wire thickness, amount of coils and mandrel size to prepare the coils.

- **Validation test I** is executed with the use of a sample shown in figure 3.7 on the left. This sample is made in the lab. It is shaped and trained by the author.
- **Validation test II** is executed using the sample in figure 3.7 on the right. This sample is ordered pre-coiled wire from Kellogg's Research Labs.

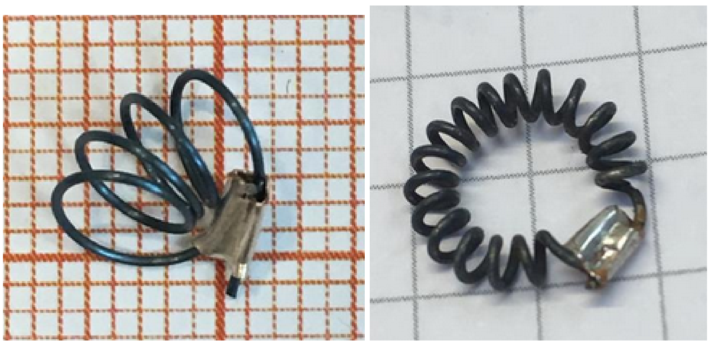

Figure 3.7: Samples used during the validation tests of the test set-up. On the left sample used for Validation test I and on the right, sample used for Validation test II

3.3. Results

3.3.1. Hardware

The test set-up is modular and can be used with three, four, or five spring scales at once. Its structure contains pre-ordered parts as spring scales and crocodile clamps, metallic parts in a form of arms holding the scales, 3D printed parts as supportive elements to keep the set-up aligned, and laser cut middle plate. A heat gun is an extra part of the system to provide the heat in order to change Nitinol's shape. For adjusting the position of the spring scales for an individual sample and to provide pre-tension, slots are drilled at the distal ends of the arms. In the slots there are screw which can be easily moved and fixed in preferred position. Figure 3.8 presents the set-up with five arms, the set-up with the heat gun placed above the sample, and the close up of one of the arms with indicated elements.

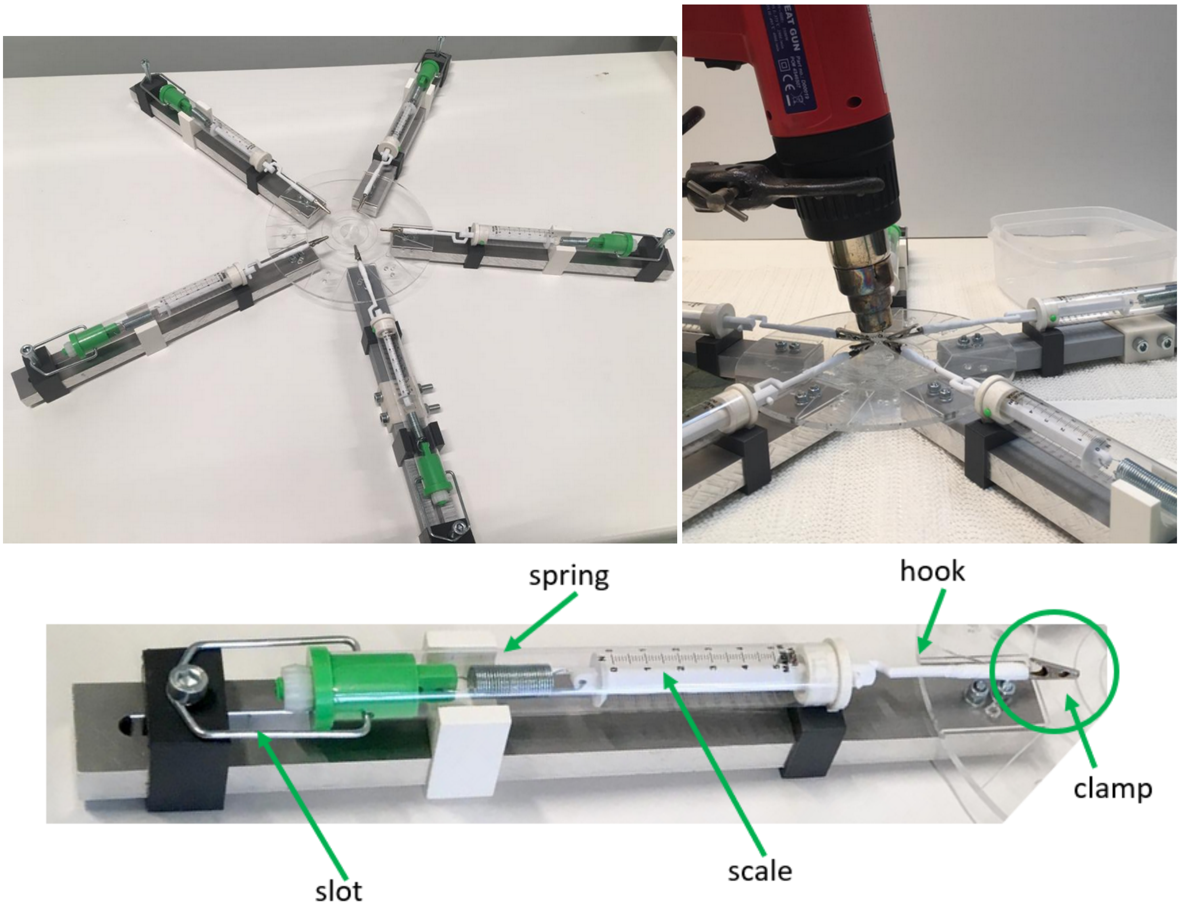

Figure 3.8: Test set-up with five arms, heat gun placed over a sample and a close view on one arm with indicated elements of the design

Due to redesigning internal parts of the spring scales, the friction was reduced. The spring scales are intended to be used in a vertical position and then the middle part fits perfectly to an end-part and no additional friction is spotted. The middle part is a block which is connected to the spring at one end and to the hook at another end, also serving as a value indicator. The end-part is located at the distal part of the spring scale. In case of this set-up, the scales are used horizontally, which results in extra friction. Re-designed parts are smaller and there is extra space between the middle part and the end part. The original part and the re-designed part are shown in figure 3.9. Green is the original part, where the middle and end block are close together. White is the re-designed part with additional space between the elements.

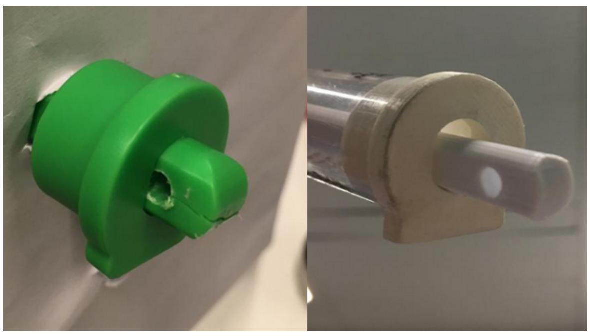

Figure 3.9: Original and re-design end parts of the spring scale. Green is the original part, where the middle and end block are close together. White is the re-designed part with additional space between the elements

3.3.2. Validation test

The raw results from the trial: Validation test I are gathered in table B.1 in appendix B, however table 3.1 shows the maximum values measured during the experiment and figure 3.10 presents the results plotted together.

In the table the maximum values of the force are presented, before heating ("Pre-tension") and after heating for each configuration of the test method, i.e. with three, four and five spring scales. The last row presents the difference between before and after heating which is related to the distance how much the sample moved while changing its shape. This values are added here to compare the conditions of the test method. The value of a change per arm in three various conditions is close to each other (0.33, 0.33 and 0.28 for the set-up of three, four and five arms respectively).

Table 3.1: Maximum force values in [N] from a validation test I

	3 ARMS	Per arm	4 ARMS	Per arm	5 ARMS	Per arm
Pre-tension	10.5	3.5	11.2	2.8	12	2.4
After heating	11.5	3.8	12.5	3.13	13.4	2.68
Difference	1	0.33	1.3	0.33	1.4	0.28

The graph 3.10 presents clear comparison of values received from the first validation test. The results presented are the force values measured with three, four and five arm. The are plotted together to see if the amount of the arms influences the measured force. The results are close to each other despite the amount of arms. To show the clear similarity in the results, additionally the linear trendlines are added. It can be seen how aligned they are.

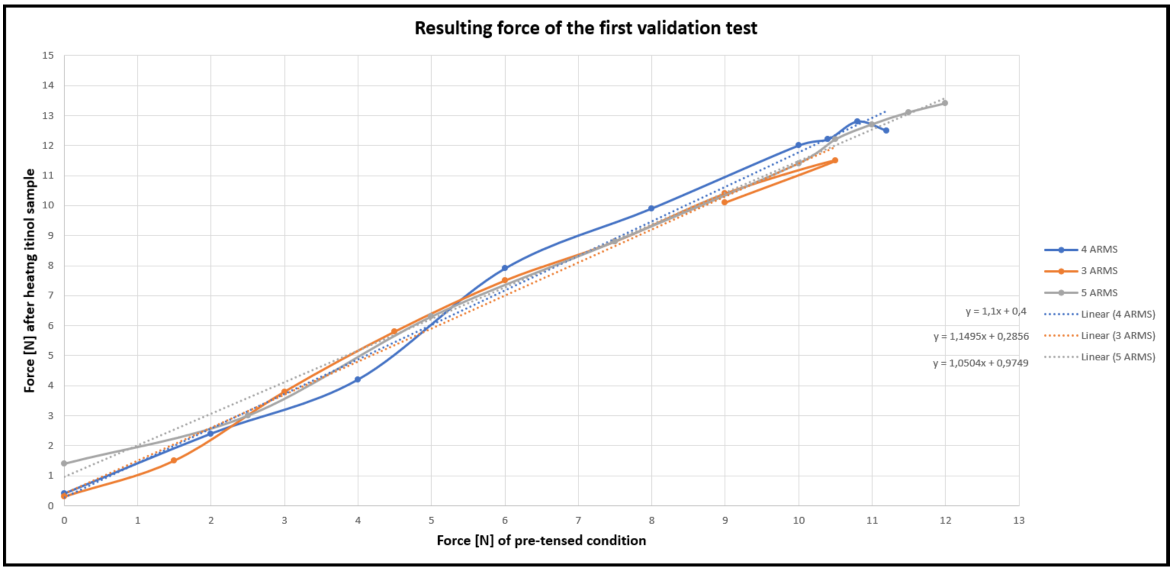

Figure 3.10: Plotted functions of the results from the test set-up validation I

The results of Validation test II are listed in table C.1 in appendix C but again the table 3.2 with maximum force values and a graph 3.11 with plotted results are added in this section. Since the results were pretty much aligned, no trendlines were needed to show the similarities between obtained values.

Table 3.2: Maximum force values in [N] from a validation test II

	3 ARMS	Per arm	4 ARMS	Per arm	5 ARMS	Per arm
Pre-tension	10.5	3.5	14.4	3.6	17	3.4
After heating	11	3.66	14.9	3.7	17.6	3.52
Difference	0.5	0.17	0.5	0.13	0.6	0.12

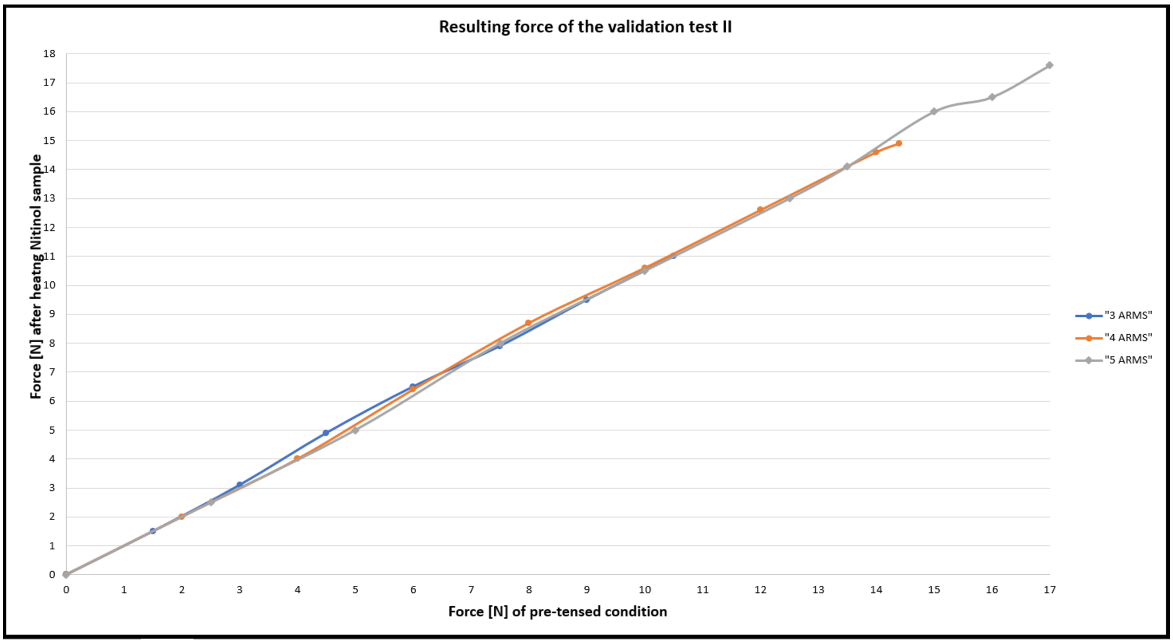

Figure 3.11: Plotted functions of the results from the test set-up validation II

3.4. Discussion

This chapter is dedicated to a development of a simple and affordable solution to measure the force of Nitinol samples in several directions during their structural transition. Such set-up was successfully delivered and tested which is a result of the work described in above subsections.

When it comes to the hardware, the biggest challenge was the connection between crocodile clamp and the hook extending from the spring scale. The heat applied on a sample was transferred to clamps and then to the plastic hook. The structure of the hook was softening and the connection was breaking or disassembling. To prevent that from happening several methods were tried: reinforcement with extra glue, adding heat resisted sleeves over the connection and also using the clay. The clay happened to be the best solution. It is a short term improvement. For the long term the connection has to be redesigned. Most favourably it should be entirely made of metal or heat resisting material so the problems with melting and softening would not happen. Another future consideration is a design of a hook, clamp and spring as one structure. Each connecting point between those modules increases the risk of detachment. Moreover, the alignment between those parts must be perfect so the test results cannot be disturbed.

The results from the validation tests confirm that the set-up is sufficient and accurate for the further testing of prototypes. The values for the same sample but different amount of arms indicated that the higher number of arms provides more reliable results. The values as shown in figures 3.10 and 3.11 are aligned however the maximum force is obtained for the condition of five (the highest) number of arms. With the lower number of arms the samples "gave up" at lower forces' values. It seems that the more arms the better force distribution.

3.5. Conclusion

The test set-up presented in this chapter is suitable to be used for the final measurements of the force exerted by prototypes and final design. Based on results obtained from validation tests, five arms will be used for each sample.

4

Shape design

4.1. Introduction

This chapter is dedicated to a design process leading to a result of the most suitable clamping mechanism. The mechanism is to be made from shape memory alloy and tested for the exerted force which is present when clamping occurs. The clamping is caused by rising the temperature of the mechanism thus allowing the alloy to go through a change of the shape and the internal structure. The complete process consists of methods of designing, manufacturing and testing prototype samples of clamping system.

The goal of this project is to design and test the mechanical clamping system which is to be included into the sterilization implant. It is done by designing and manufacturing scale-up prototypes and testing them for their clamping power. Those action lead to distinguishing the most satisfactory design and building it in a scale which is appropriate for sterilization purpose.

The clamping system has to fulfill certain requirements in case to be useful and to be desired as a part of the sterilization implant. At first it is required to clamp the Fallopian tube (after an activation) so no passage through its lumen is possible. After time, such closed tube will develop a scar tissue which will permanently close the lumen. Secondly, it has to fit over the Fallopian tube without causing any blockage before the activation. Thirdly, the system must imitate a closed circuit in order to enable the electrical current flow. The attaching of the device into the Fallopian tube is not a scope of this project. In real situation the system must be conductive but also be applicable for an installation inside a human body.

Starting point of the designing process is given by Opt Medical. The initial idea is provided to indicate what is the projected outcome of the sterilization implant as shown in figure 4.1. The figure presents the scheme of the device in two states: before and after activation. After an activation the Nitiol wire causes the deformation of the entire structure. The right picture of this figure presents the placement of the clip around the Fallopian tube. Figure 4.2 presents the disassembled device with a Nitinol wire as one of the parts to indicate the position of the wire within the sterilization clip.

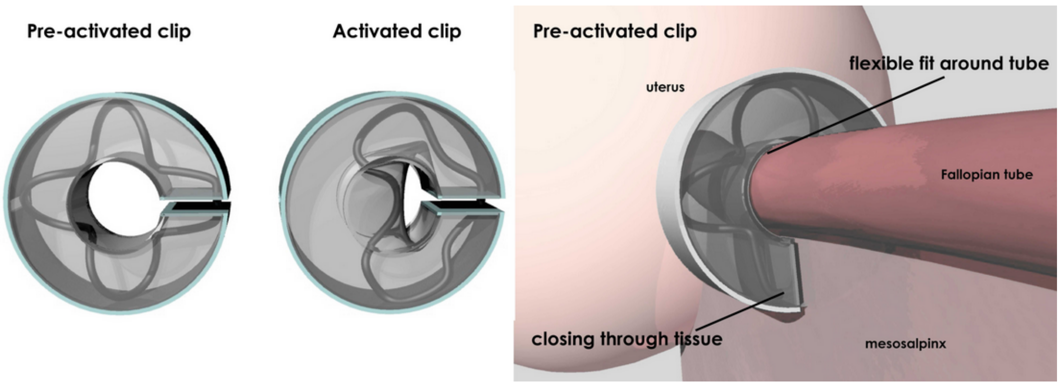

Figure 4.1: Initial design of the sterilization clip proposed by Opt Medical. The left side shows the clip in a pre-activation state and activated state. On the right side the clip is being placed over the Fallopian tube.

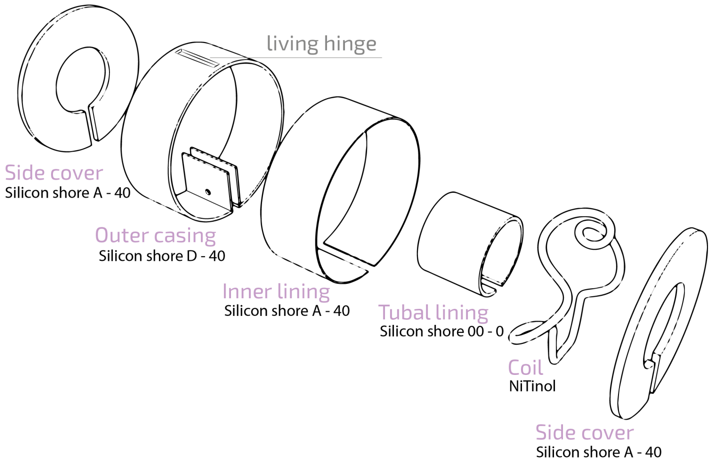

Figure 4.2: Scheme of a disassembled sterilization clip

4.2. Methods

This section is divided into four subsections. The first one is dedicated to the design process thus the method leading to creation of several models. The next subsection is related to the method of manufacturing. The third subsection describes used methodology of testing the samples - used materials, equipment and protocol of performing the measurements. The last subsection is about the iterative process of performing the steps motioned in subsections 4.2.1 (design), 4.2.2 (prototyping), 4.2.3 (measurement). Based on results presented in section 4.3 - Results, the final design is developed.

4.2.1. Design

The design process starts with identifying existing clamping mechanisms and solutions. The most common examples of systems used on a daily base are a stapler, laundry clip, trousers belt, paper clip and many others. Already recognizing the stuff laying around us gives a valuable insight for the first ideas and concepts. Those several methods of clamping are acknowledged and gathered during a brainstorm session. Figure 4.3 present the results of analyzing the concepts and translate them into solutions for the sterilization clip.

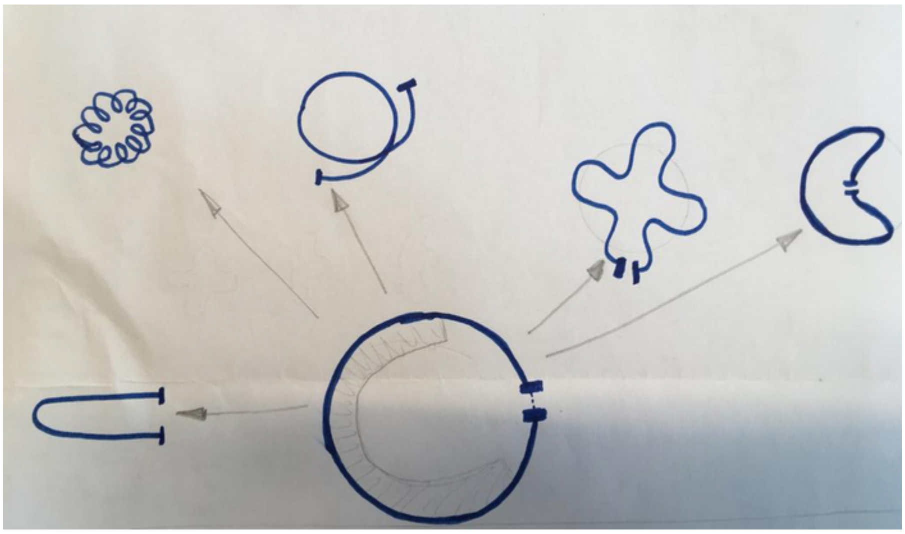

Figure 4.3: Results of a brainstorm

4.2.2. Prototyping

In order to train the Nitinol sample into a certain shape, the wire has to be pre-tensed in a desired shape and heated up. This section presents methods of manufacturing the prototypes based on their designs. In table

Table 4.1: Manufacturing methods of prototypes based on their designs

<table>
<tr><th></th><th>Turned wire</th><th>Flat Wire</th><th>3D shape</th></tr>
<tr><td>Symmetrical</td><td>Plate and screw</td><td rowspan="6">3D printed mould</td><td>3D printed mould</td></tr>
<tr><td>Expanding concept</td><td>3D printed mould with grooves</td><td></td></tr>
<tr><td>Cloud concept</td><td>Plate and screw</td><td>3D printed mould</td></tr>
<tr><td>Trident concept</td><td>3D printed mould with grooves</td><td>Plate and screw</td></tr>
<tr><td>Two connections concept</td><td>Plate and screw</td><td>3D printed mould</td></tr>
<tr><td>Egg concept</td><td>3D printed mould with grooves</td><td>Plate and screw</td></tr>
</table>

4.1 the methods of shaping for certain designs are presented. They are based on the shapes' designs which are only shown in a further part of this report in figure ... (i.e. results of the design section).

Coiled samples

For manufacturing coils the best solution is to use screws. The wire has to be wound around the screw and fixated at both ends so the wire is not able to wind off the screw when the heat is applied. The moment the sample is heated, the wire tends to unwind and change the shape into previously "remembered" one. The most common is to straighten shape. But once the wire is heated up enough, it remembers its austenit phase as a coil. Such prepared coils are clamped into desired patterns. The crimping it (i.e. make it a closed circuit sample) also determines its shape and and the way the samples will be deforming.

The steps to prepare a coiled sample are as follow:

1. Fix one end of the wire on a plate with a screw and tighten it hard with a nut
2. Choose a central screw size based on the final dimension of the coil you want to obtain
3. Wind the wire around the central screw as many times as wished final length
4. Fix the other end of the wire the same way as the previous one (with a screw tighten to a plate with a nut)
5. Place the plate with the wire in the oven preheated to 500°C
6. Let the wire sit in the oven for 5 to 15 minutes
7. Carefully remove the plate from the oven and place it in ice cold water
8. Release the coil from a screw
9. Cut off the ends of the sample to the desired length
10. Connect two ends of the wire with an aluminum tube and crimping tool

Flat wire and unusual shapes

For the flat shapes the solution is to use plaster. Plaster can withstand high temperatures of the oven and also keep the wire in a desired position.

First, to form a wire into a shape wanted as final, 3D-printed moulds are very useful. Plastic 3D parts melt inside the plaster block and do not intervene with the process as the plaster around the sample keeps the samples in shape.

The moulds are designed in 3D designing software - SolidWorks. Furthermore, the thin groove of a size of the wire diameter is included into the design. Depends on whether the mould is designed for a turned wire or a flat one, the groove can be placed flat or be wind around the mould. The difference is shown in figure 4.17 where moulds for the same type of a concept (trident) are presented but for different wire structure.

Moreover, figure 4.5 presents the models of moulds used to make symmetrical samples using flat wire. The wire is to be wound around the small circular blocks in order to receive desired shapes.

In figure 4.6 a wave structure is presented. Such mould can be used to manufacture multiple types of prototypes. For example a symmetrical model of a 3D symmetrical coil. To achieve that, the shown model must be doubled. By connecting the ends the prototype can be shaped. In figure 4.7 the picture of printed and shaped mould is presented. Using the same mould, the cloud concept of the flat wire and 3D wire can

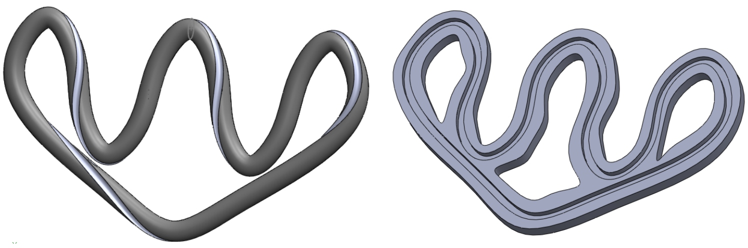

Figure 4.4: Models of 3D printed moulds to manufacture trident concept. On the left a model for turned wire and on the right the flat wire.

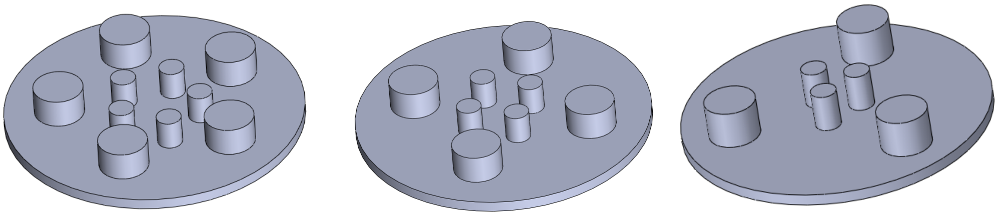

Figure 4.5: Models of 3D printed moulds to prepare symmetrical samples with flat positioned wire

be built. For the flat design, two eds of the wire can be connected below the wave in the same plane. The difference with the 3D model would be that the ends must be bent 90°and connect underneath the wave but in another plane.

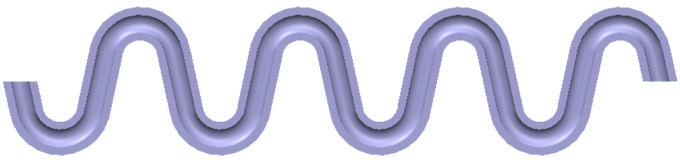

Figure 4.6: Wavy structure with grooves to create several types of prototypes

Figure 4.7: Printed mould shaped to make 3D symmetric model

The Nitinol wire can be pre-shaped into a printed mould and then embedded into plaster. The same amount of plaster and water is used for every sample to achieve the same volume as it would give the same time to heat up the samples inside. Quickly drying plaster from Knauff is used in a proportion 2:1 with water. For each sample 30 grams of plaster and 15 grams of water is mixed to prepare mixture. After drying time of the plaster, the sample can go to the oven to train the wire to its new shape. Since the plaster provides extra heat protection, the heating process must be elongated. The sample is put to the cold oven (room temperature) and as the temperature rises, two small vents are open. It allows to release all the remained moisture from the plaster block. This step is added to prevent the plaster from braking in high temperatures. As the oven reaches the necessary temperature, the vents are closed. The sample in plaster is kept in the oven for 60 minutes to be sure that the Nitinol reached shaping temperature.

The detailed procedure of preparation the prototypes with the use of 3D printed moulds and plaster is listed below:

1. Wind the wire around a printed mould or place it inside the grooves of it.
2. Prepare the plaster using 30 grams of powder and 15 grams of water according to the instructions given by the manufacturer. Prepare it in a small paper cup.
3. Dip the sample in the plaster and make sure it is all covered by the plaster and no plastic is exposed.
4. Let the plaster solidify for about 15 to 20 minutes.
5. Remove the small plaster block from a cup to speed up the drying process.
6. Let the block to dry overnight
7. Place the sample in a block in the oven
8. Open two small vents of the oven and start heating it up to 500°C.
9. After 20 to 30 minutes when the oven reaches 500°C , close the vents and count down 60 minutes.
10. Remove the plaster block from the oven and place it in iced cold water
11. Brake the plaster block to release the wire - note that you might need a hammer to brake the plaster.
12. Cut off the ends of the sample to the desired length
13. Connect two ends of the wire with an aluminum tube and crimping tool

Off-the-shelf coils

There is an additional group of samples prepared in order to examine the variables between the coils. The coils can differ with the wire thickness, mandrel size on which they were made and a pitch value between the coils. As manufacturing such coils would be extremely laborious and also could be far from needed accuracy, a set of pre-made Nitinol coils was purchased from Kellog's Laboratories. A batch of wires varying in those three characteristics which were mentioned before is added to this study. The type of wires and their features are listed in table 4.2. The lack of consistency in numbering the samples is a result of an incomplete delivery from the manufacturer.

The coils need are delivered loose and to be used in the study their ends must be connected to create a closed unified structure. It is done using the aluminum tubes and crimping device.

4.2.3. Measurement

The samples are tested with two methods. Methods of both are described in this section.

Fallopian tube test

In order to determine if the sample exerts enough force to stop the flow through the Fallopian tube, this test is conducted. The accurate mechanical properties of the Fallopian tubes are unknown and also there is access to the real tissue for the purpose of this project. But there is a synthetic representation of Fallopian tubes developed by the author of this report during the internship for Opt Medical [18]. The synthetic tubes are made with silicone and have the size corresponding to the real Fallopian tubes. They were tested by a group of surgeons who have an experience with the human tubes and could provide their opinions on prepared tubes. The examination on those tubes was visual as well as the surgeons examined the feel of the tubes in their hands. The synthetic Fallopian tubes are the most similar to the real tissue from any other available materials but the results from the tests on them can not be definitive. It must be noted that the tubes may vary from the human tissue.

Table 4.2: List of the coils purchased from Kellog's Laboratories

Label	Diameter [mm]	Mandrel size [mm]	Pitch [mm]	Length [mm]
2	0.25	0.5	0.75	18
4	0.25	1.15	0.75	20
6	0.25	2.4	0.75	24
7	0.25	2.4	1	24
9	0.5	0.5	1	18
11	0.5	1.15	1	20
12	0.5	2.4	0.75	24
13	0.5	2.4	1	24
14	0.5	3.2	1	26
15	1	0.5	1	18
16	1	0.5	3	18
17	1	1.15	1	20
18	1	1.15	3	20
19	1	2.4	1	24
20	1	2.4	3	24
21	1	4.75	1	32
22	1	4.75	3	32

As the prototypes are scaled up from the final device, the synthetic tubes were scaled as well to match the samples' sizes. As the anatomically correct Fallopian tube has outer diameter of 5 mm, the additional tubed were manufactured in 10mm, 12mm, 14mm, 16mm and 18mm of outer diameter and the inner diameter corresponds to the ratio of the real tube. The material and method to create those tubes were the same as for the samples examined by the surgeons. The tubes are presented in figure 4.8.

Figure 4.8: Synthetic Fallopian tubes in scale up sizes; from left to right: 18, 16, 14, 12, 10 and 5 mm outer diameter

This test is a pass/fail test. The prototype is placed around a tube on which it fits without squeezing it before applying the heat. Then, the heat is applied on the sample and as the prototype tightens around the tube, the water is injected to the tube. The examinations contains the check if the water flows through the tube or it is stopped due to the prototype clasped over the tube.

Force measurements

The measurements of the force is executed on the test set-up described in the chapter 3. The used configuration for the test is with five spring scales. However there are two configurations of the hooks' length as the prototypes have varying dimensions. Additional equipment and materials are the heat gun to provide the sufficient temperature to heat up the samples (used in this experiment is Astro Pneumatic Tool® 9425 - Dual Temperature Heat Gun Kit, placed 3-5 cm above a sample. The temperature setting I is used (300°C)) and ice cubes to cool down the samples between the measurements for ease and safety of handling the test set-up.

The test is performed by clamping the sample onto the set-up. The five clamps are distributed in the most symmetrical way as possible. The sample must be kept in the middle of the plate and the spring scales must be adjusted in position so that they all display the same force value in the beginning of measurements.

The protocol of the order of actions is as follow:

1. Clamp the sample according to the previous instructions (in the center of the plate, at five points distributed equally, and the spring calipers are at the same position);
2. Set the spring scales to 0 N each;
3. Place the heat gun directly over the sample, about 3 - 5 mm above it;
4. Switch on the heat gun for about 3 to 5 seconds;
5. Record the values displayed on the spring scales;
6. Place an ice cube over the sample;
7. Set the spring scales 0.5 N higher than previously;
8. Repeat the steps 3 - 7 until there is no more change in the sample's shape;
9. As the value - in which the sample does not change - is reached, adjust the pre-tensioning step to 0.1 N (point 7) less than the last measurement to confirm the exact maximum force value the sample can not overcome.

Samples
There are 41 samples to be tested. 15 of the Kellogg's coils and 26 of self made. The samples to be used at those tests are presented in the results subsection 4.3.2 as pictures of prepared prototypes.

4.2.4. Iteration I

Iteration I and Iteration II parts are added based on the results described in a further part of the report. It is to keep a correct structure of the thesis report, however the chronological order of the event is disturbed. For better understanding, it is recommended to come back to this part after reviewing the results section.

Five coils were investigated of wire thickness 0.5 mm and various pitches and inner diameter as shown in figure 4.9. Those are the same coils as the the ones used for preparing Kellogg's samples number 9, 11, 12, 13, 14.

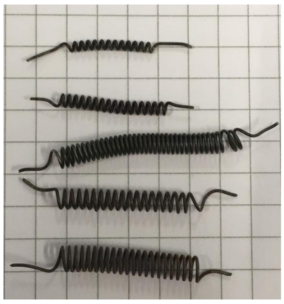

Figure 4.9: Kellogg's coils of wire diameter 0.5 mm

In order to obtain comparable numerical values on coils of different sizes, the samples have to be standardized. The factor over which the samples are normalized is the factor a. a is the ratio between the diameter of the coil and its pitch. The relation looks as follow: $a = \frac{d_{in}}{p}$, where d_{in} indicates the inner diameter and p a pitch thus the distance between the coils. This procedure was used to be able to compare the forces applied on the coils in dependency on the geometry of the samples.
The measuring method of the five samples is as follow: the samples are fixed at one side and on the other side they are clamped to the spring scale. The sample is measured for its length (x) and number of turns to establish the pitch (p), hence determine the a factor. In order to stretch the samples, certain force (F) is needed. The force on the spring scale was noted for each calculated a. The samples are heated after each measurement, to examine their ability to come back to their prime (set-up) shape (x_1) after applying pretension. This operation sets the boundaries of further measurement as the sample is no more tensed when the force is keeping it restricted. The inner diameter d_{in} is assumed to remain constant for the coil in both stages: pre-tensed stage and the prime shape stage. The scheme configuration of the coil before and after applying heat is shown in figure 4.10.

The coils are being evaluated in the linear state and not circle as they are shaped in the previous experiment. The length and the pitch are the same in both configuration as seen in figure 4.11, but the measurements can be done using only one spring scale which simplifies the process.

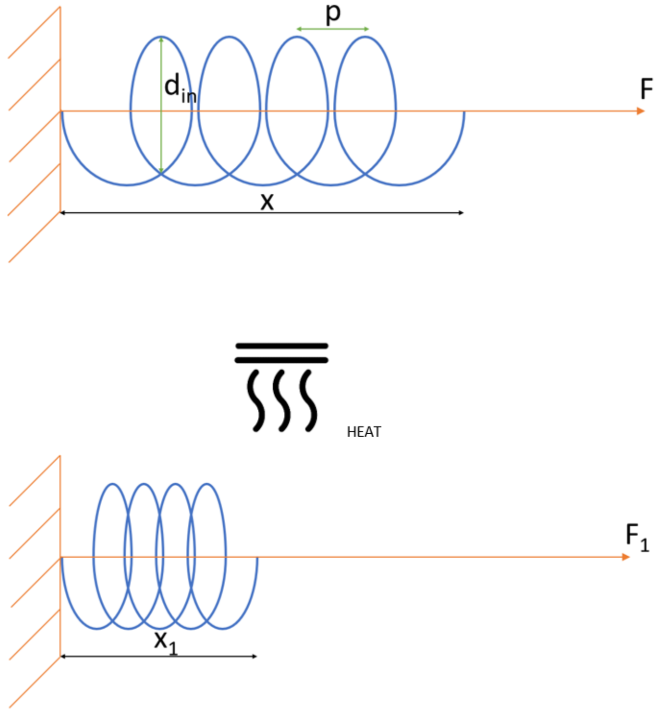

Figure 4.10: Configuration scheme of performing the measurements of the force dependent on a factor. Above: pre-tensed coil; below: heated coil in its prime shape

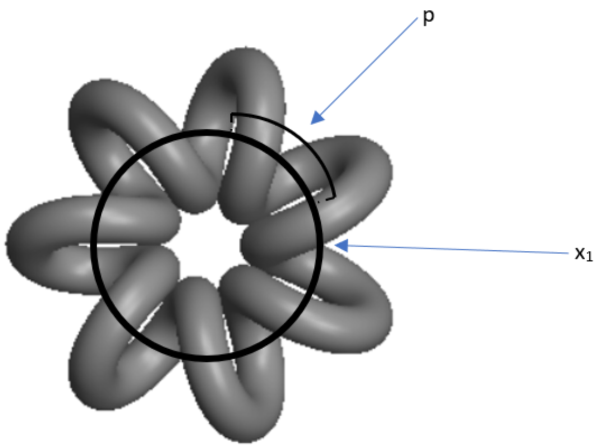

Figure 4.11: Relation of the coil length x_1 and pitch p in the circular shaped coil.

4.2.5. Iteration II

Iteration II is an attempt to compare two the most promising coils (based on the Iteration I): sample 9 and sample 11. Those two coils of the lowest mandrel diameter are chosen due to high forces presented during the force measurements of the samples, followed by high forced measured in the linear state at Iteration I and high deformation rate which is beneficial for the final purpose of the clamping mechanism.

They are tested in a similar way to the way presented in Iteration I. The samples are fixed at one side, and connected to the spring scale on another side. The initial length is measured. Then, with a step of 0.5N, the coil is stretched and its extension is noted. The sample is heated and the resulting length of the coil is again measured. After the last measurement sample is released, heated and measured at its final state to examined possible plastic deformation in samples. The initial length is subtracted from the resulting length after heating the sample with certain applied tension, and this value is used to calculate the spring constant.

Additionally, the samples are modeled in a 3D sketching software, to evaluate the dimensions of the pre- and post- activation state. Modelling and measuring the samples is a more accurate than building and measuring the samples, hence the sketched models are presented as the results. They are both designed to have seven turns, hence their length is 7 mm.

4.2.6. Final design

The final design is created based on the results obtained from the tests described in subsection 4.2.3. The shape and materials will be chosen to prepare the final sample which is desired to demonstrate sufficient ability to close the lumen of the Fallopian tube.

The final design sample will be tested using the 5 mm outer diameter synthetic Fallopian tube. The procedure will be as follow: the coil is stretched open and placed over the synthetic Fallopian tube. Heat from a heat gun is applied on the sample to cause the deformation. Water is put through the tube using at one side. The other side of the tube is examined for the water leaking through it.

The iteration on the final design is based on results presented in the section 4.3.3. Samples of Kellogg's coils of 0.5 mm wire diameter were chosen for further investigation due to high force values measured during the force measurements.

Eight out of ten best performing prototypes are made of coils and only two of a flat wire. The coils are closer investigated for the additional dimensions as length, pitch, mandrel size and amount of coils to find dependency between the high force and those parameters. To do that, five Kellogg's coils of equal wire thickness are chosen because they can be compared with each other.

The dimensions of the final design are based on the size of Fallopian tubes. The outer diameter of the Tube is about 5 - 5.5 mm. The final design in an open state must be of this dimension, firstly to prevent from squeezing the tube inwards; and secondly to stay firm on the Tube and not migrate within a body. The closed state diameter must be about 2 - 2.5 mm, however in case the design includes other parts like silicone casing, the length of the Nitinol wire can be larger than 2.5 mm. Then, additional casing squeezes the tube, and the wire provides its displacement in radial direction.

The calculation of circumferences of the Fallopian tube give the indication of the length of the final design. Thus, the length in an open state should be around 16.5 mm and in closed state about 7 mm.

4.3. Results

4.3.1. Designs

Next step is to realize how those ideas fit the requirements. New device must posses a metallic closed structure to enable a current flow. As the principle of the device must be applicable for an inductive power transfer, the coil is the first concept which comes to mind. There is no one simple design of a coil. There can be variations in pitch, mandrel size on which the coil was shaped as well as the number of the turns. This leads to a broad study on a coil design. On the other hand the solutions without a coil should not be rejected. A flat shaped wire can also be shaped into a prototype which meets the conditions. It can also be the hybrid of both.

Another criteria identified while studying the existing solutions is how the Fallopian tube is going to be clamped. It can be squeezed in one axis (as the tube is squeezed between two fingers), as it is for the current devices as Filshie or Hulka clips, but also the mechanism can apply the pressure around the tube. It could also deform the tube into unspecified shape as long as the lumen of the tube is closed. Moreover the squeezing can be performed in two or three planes which can make the design "3D".

Possible concepts and solutions are based on two criteria: how the tube will be deform and how the wire is shaped - turned or flat or 3D. The deformation of the tube can be specified as:

- symmetrically on the entire tube providing equal force around the structure.
- asymmetrically; squeezing the tube in certain axis only or in each direction but with varying force.

The symmetrical approach may be divided into radial and circumferential as shown in figure 4.12 which is based on anatomical representation of muscle contraction. An example is the heart where the contraction method depend on the fiber orientation. The circumferential direction results in twisting motion. Both ways affect the blood flow as it is pumped from the heart. The radial and circumferential contractions can be related also to the muscles of an eye, where the constriction is the effect of light intensity. In figure 4.13 the example of the muscle movement in the eye is shown.

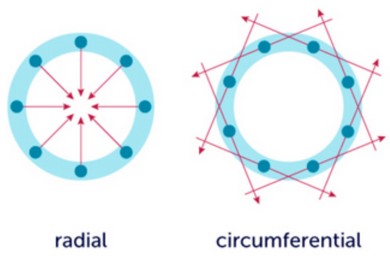

Figure 4.12: Different directions of symmetrical contraction [21]

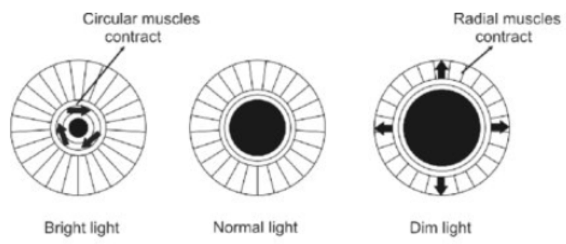

Figure 4.13: Radial and circular muscles contraction [22]

The asymmetrical approach can be presented in even more forms. It can occurs as an action in one particular direction as shown in figure 4.14, or in multiple directions but with varying force value as in figure 4.15. However it still must close the tube's lumen.

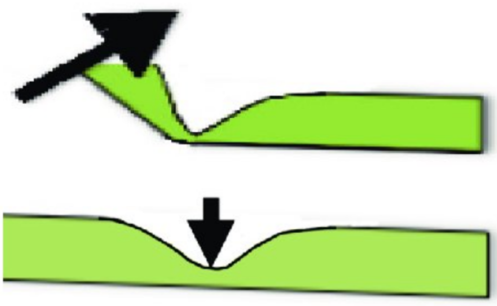

Figure 4.14: Compression of a tube by applying external force (bottom), and kinking a tube (top) [23]

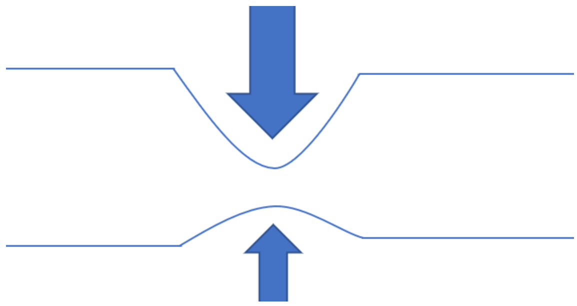

Figure 4.15: Different magnitude forces applied in the same direction

The designs were created based on principles and ideas mentioned in the method's subsection 4.2.1. To identify them in structural and clear way a matrix was proposed. The matrix covers two most important criteria of the way the Fallopian tube is deformed and how the wire is shaped. The tube can be deformed in symmetrical and asymmetrical way but for better division of the concepts, those categories were sub-divided. Thus, the symmetrical way consists of radial and circumferential deformation. The latter method can be used to develop an opposite effect. This effect is an expansion instead of a compression. Also the designs including coils represent the circumferential deformation.

The asymmetrical way also consists of multiple realizations:

"Cloud concept" where a coil or wavy structure is present on one side only and the deformation's principle is that the turns are approaching each other - simple visualization is included in figure 4.16.

"Trident" is a concept similar to the cloud one but it contains only three bumps and the deformation is as follow: the two outer bumps approaching the middle one. The sketch of it is shown in figure 4.17.

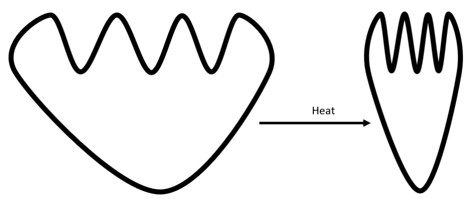

Figure 4.16: Sketch of a "cloud concept"

Concept **"Two connections"** - the idea behind this concept is to have two individually trained wires which exhibit different deformations. The name of this concept is based on the necessity of connecting two wires in two spots to create a closed circuit. The sketch is presented in figure 4.18, the dots represent the connection points.

"Egg" - the deformation causes clamping at one direction, as shown in figure 4.19.

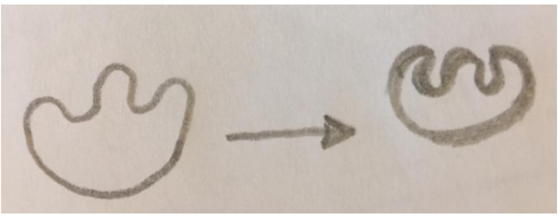

Figure 4.17: Sketch of a "trident concept"

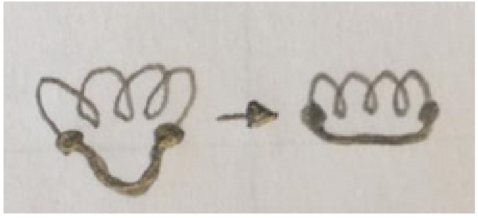

Figure 4.18: Sketch of a "two connection concept"

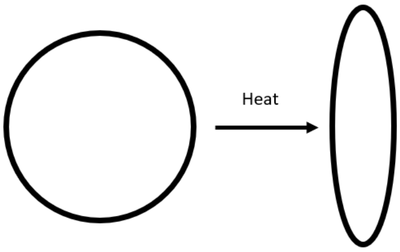

Figure 4.19: Sketch of an "egg concept"

To summarize the concepts presented above, the matrix was prepared. It is presented in figure 4.20. In this table the designs are sorted based on the concept type and the method of shaping the wire. For some resulting designs there are more than one solutions but they still fit the category.

Figure 4.20: Matrix including the designs. It is categorized due to the concept shape and the structure of the wire

4.3.2. Results of prototyping

Kellogg's

Figure 4.21 presents the connected coils from Kellogg's of wire thickness 0.25 mm. From left to right, the coils are: sample number 2, 4, 6 and 7. All of the four samples were connected using an aluminum tube and crimping tool.

Figure 4.21: Samples 2, 4, 6 and 7 (from left to right) of wire thickness 0.25 mm

Samples number 9, 11, 12, 13 and 14 are shown in figure 4.22. Those samples have all thickness 0.5 mm and they are all connected using aluminum tubes and crimping tool. Sample number 13 is not completely symmetrical even though it was made with the same method.

Figure 4.22: Samples 9, 11, 12, 13 and 14 (from left to right) of wire thickness 0.5 mm

The last seven samples (16, 17, 18, 19, 20, 21, 22) are shown in figure 4.23. Only samples 19 and 21 have the connections made with the use of an aluminum tubes and crimping tool. Samples 17 and 20 are joined with solder as the common for other samples method of crimping was unsuitable for those coils. Samples 16 and 18 could not be connected neither using crimping nor soldering due to large wire diameter together with small length of the coils. Bringing together two ends of the samples was impossible.

Figure 4.23: Samples 16, 17, 18, 19, 20, 21 and 22 (from left to right) of wire thickness 1 mm

Shaped prototypes

The prototypes which are shaped in the lab are presented in figures 4.24 and 4.25. The first table presents the samples of symmetrical shapes and second one presents samples of asymmetrical shapes. There are nine samples designed to change according to the radial principle. The nine samples include one made of coiled wire, five samples of flat wire, and three samples 3-dimensional. There are two samples of circular shape changing - one of coiled wire and one of flat wire.

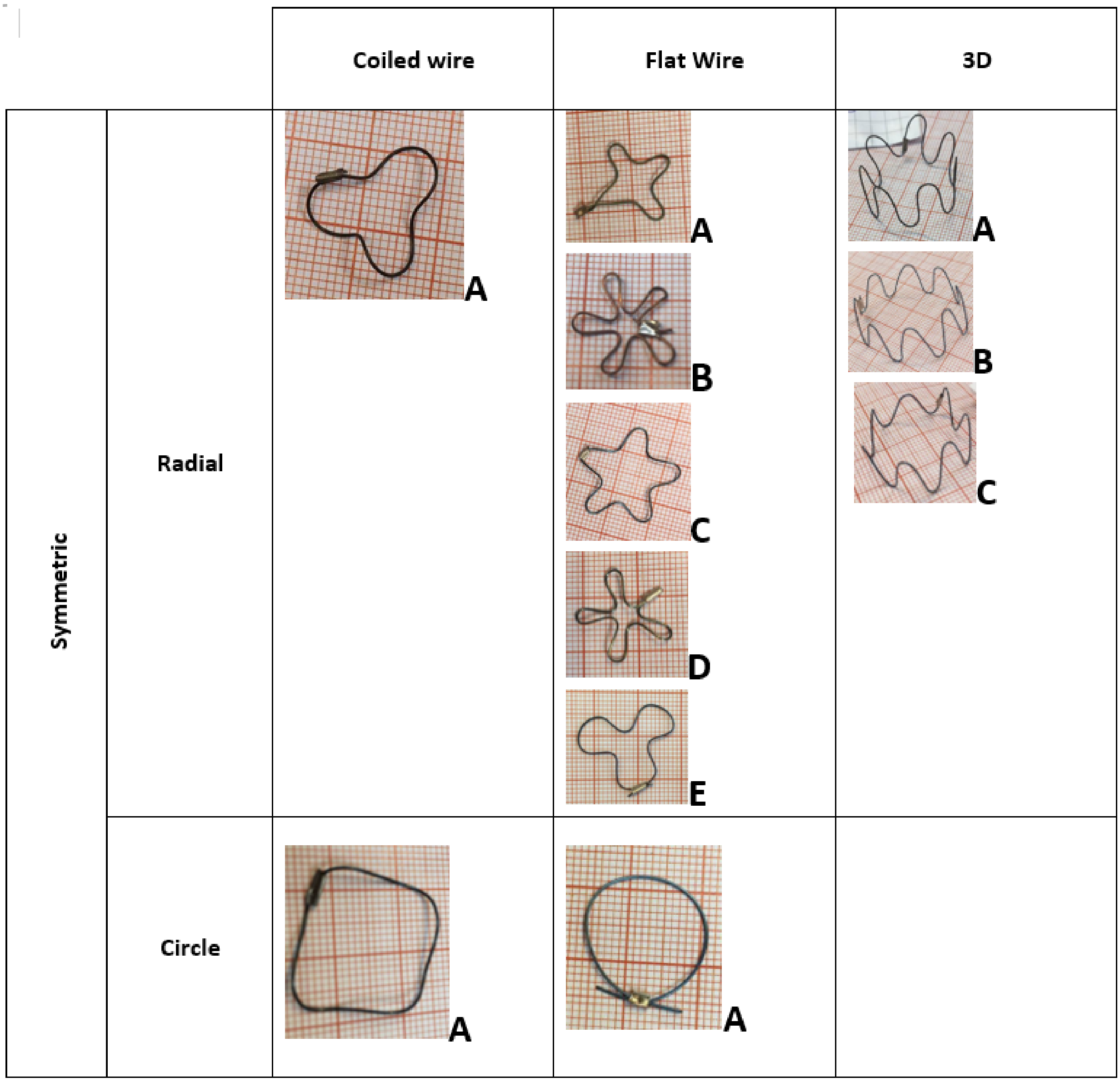

Figure 4.24: Symmetrical samples self- designed and shaped

Fifteen samples presented in figure 4.25 include four samples of cloud design. Two are made with coiled wire and two with flat wire. Next five samples represents trident concept. Three samples are made from coiled wire and two from flat wire. There is only one sample of Two connections concept. It is placed in coiled wire category but it includes both: coiled wire and flat wire. Last but not least five samples are of egg concept. Two samples are coiled and three samples are flat.

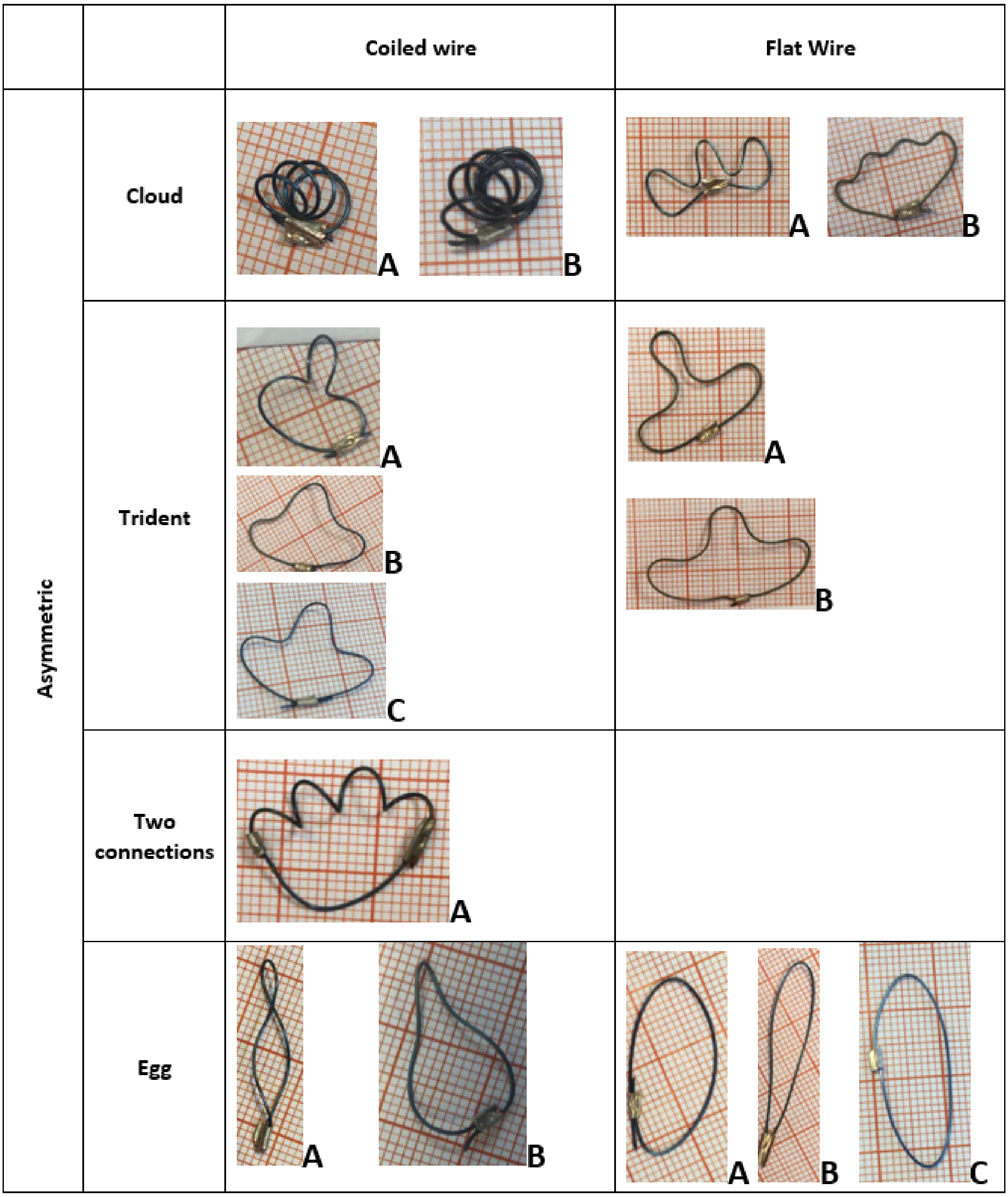

Figure 4.25: Asymmetrical samples self- designed and shaped

4.3.3. Measurements

Fallopian tube test

Samples tested for their ability to stop the flow within Fallopian tubes are listed in table 4.3. The first fifteen entries are of Kellogg's coils, arranged in the order of sample's number from smallest to largest. Below in a table, separated by a horizontal line are the self-made samples listed in the order of how they are presented in figures 4.24 and 4.25. The order is: the concepts, and within the concepts starting from coiled wire samples, then flat wire samples and then 3D samples. Samples marked as N/A in "Fallopian tube diameter" and "Pass/Fail" columns, were not tested and thus now results provided for them.

Table 4.3: Results from a test on synthetic Fallopian tubes

Sample Type	Sample	Fallopian tube diameter	Pass/Fail
Kellogg's coils	2	5	Fail
	4	10	Fail
	6	10	Pass
	7	10	Fail
	9	12	Fail
	11	10	Fail
	12	14	Pass
	13	14	Fail
	14	10	Pass
	16	N/A	N/A
	17	N/A	N/A
	18	N/A	N/A
	19	18	Fail
	20	N/A	N/A
	21	12	Pass
Own designs	Coiled wire Radial A		
	Flat wire Radial A		
	Flat wire Radial B	10	Pass
	Flat wire Radial C		
	Flat wire Radial D	12	Pass
	Flat wire Radial E		
	3D wire Radial A		
	3D wire Radial B		
	3D wire Radial C		
	Coiled wire Circle A		
	Flat wire Circle A		
	Coiled wire Cloud A	5	Pass
	Coiled wire Cloud B	10	Pass
	Flat wire Cloud A	10	Pass
	Flat wire Cloud B		
	Coiled wire Trident A		
	Coiled wire Trident B		
	Coiled wire Trident C		
	Flat wire Trident A		
	Flat wire Trident B		
	Coiled 2 Connections A		
	Coiled wire Egg A		
	Coiled wire Egg B		
	Flat wire Egg C		
	Flat wire Egg B	18	Pass
	Flat wire Egg A		

Force Measurements

First graph in figure 4.26 presents the results of force measurements on Kellogg's coils. The colors indicate the groups of wire thickness, so in yellow (2, 4, 6, 7) there are samples of 0.25 mm diameter, in green (9, 11, 12, 13, 14) the samples of 0.5 mm diameter and in orange (16-21) the samples of 1 mm wire diameter. The missing slots are for the samples which were planned to be measured but could not provide any results due to strength of the coils. Samples number 16 and 18 already failed to be connected into a circular structure. Those coils could not be shaped to a desired outcome. Remaining samples except sample number 17 had to be pulled open with a force higher than it could be provided by the test set-up.

The highest values of Kellogg's coils, were recorded for the samples of 0.5 mm diameter hence they are chosen for further investigation to design and manufacture the final prototype.

In figure 4.27 the results are presented of twenty six samples which were self-made. They are arranged in

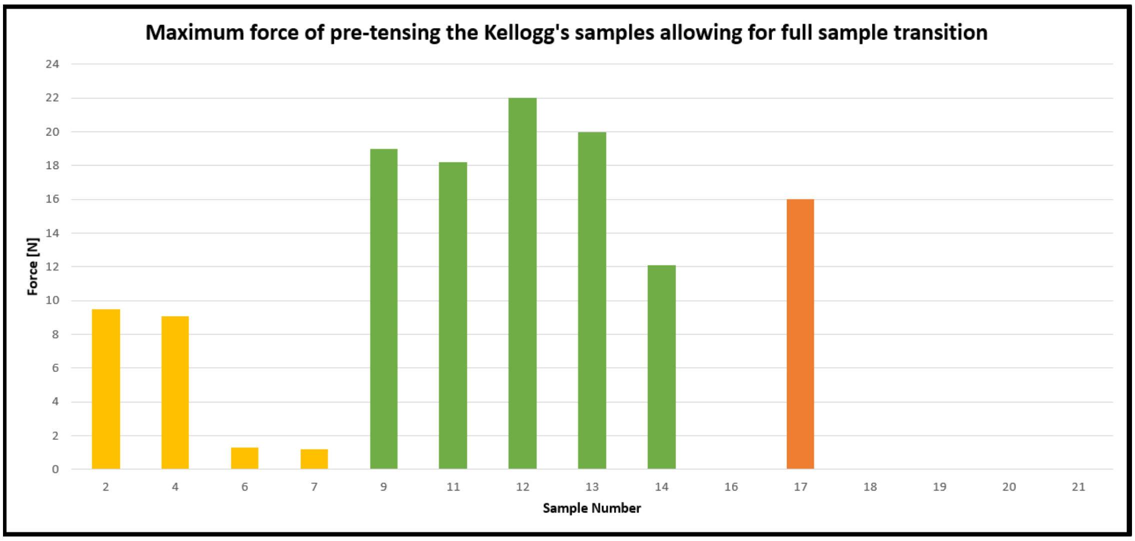

Figure 4.26: Results of force measurements on Kellogg's coils. The colors indicate the groups of wire thickness, in yellow samples of 0.25 mm diameter, in green the samples of 0.5 mm diameter and in orange the samples of 1 mm wire diameter.

the order of resulting force from the highest value on the left to the lowest on the right. Three last samples with the force value of 0, did not demonstrate any shape change while heated.

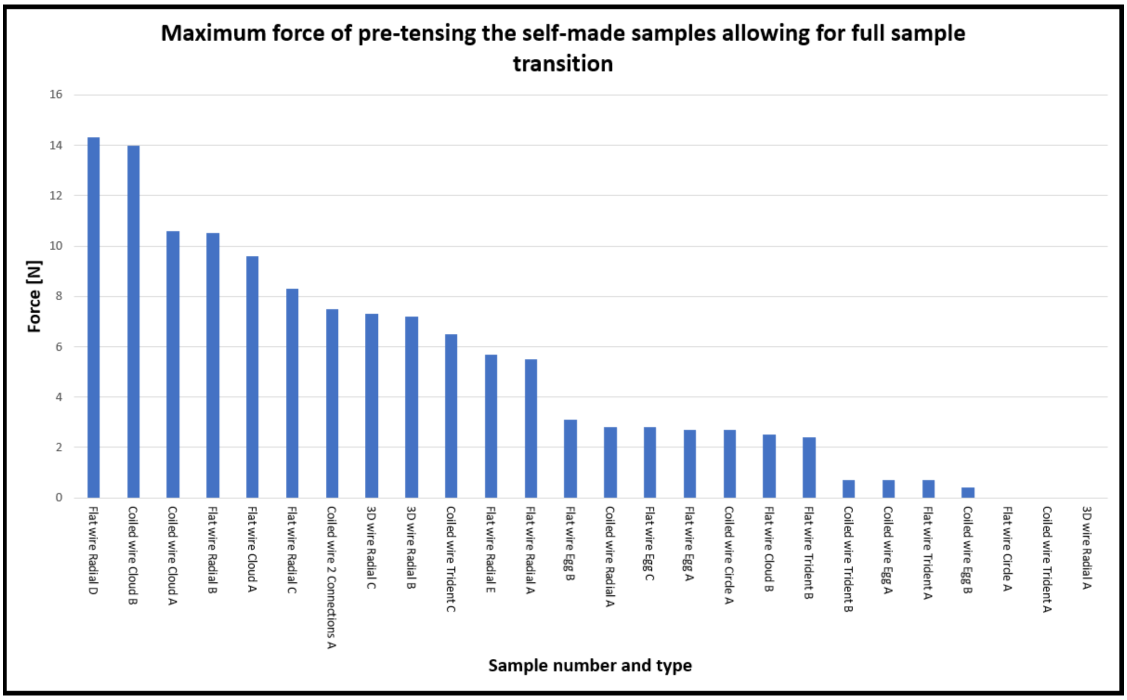

Figure 4.27: Results of force measurements on self-made prototypes

Figure 4.28 presents the collective results of ten samples out of all examined samples. There are presented in the order of highest measured force from left to the right. The colors of the bars represents the groups of samples, in green the Kellogg's coils are presented and in yellow the self-made samples. Additionally the pictures of the prototypes are included under the bars. Next to the 0.5 mm wire Kellogg's coils, the best scoring self-made samples are symmetric-radial flat wire samples B and D, together with asymmetric cloud concept coiled wire samples A and B.

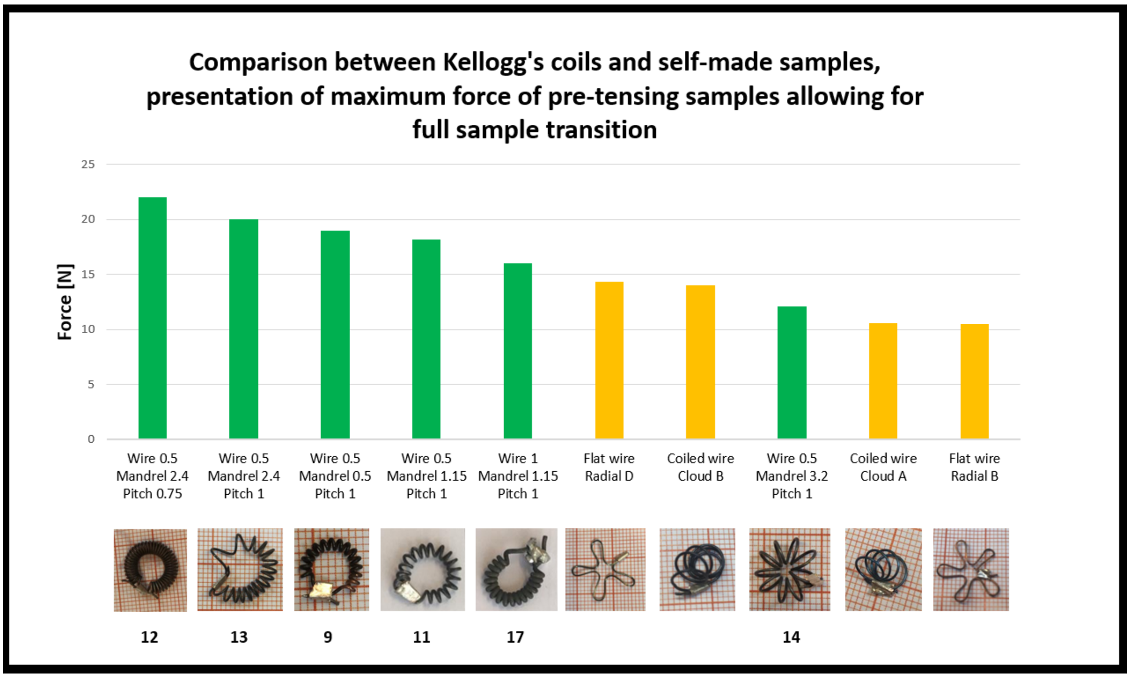

Figure 4.28: Collective results of ten prototypes which were measured the highest forces at the force test

4.3.4. Iteration I

For the five chosen samples, there is one narrow range of a for which they could be standardized. The value of that a is between 0.42 and 0.45. It is due to large differences between coils' mandrels diameters. The sample 14 has the mandrel diameter of 3.2 mm thus the a of about 0.45 is the minimum ratio the coils could be shaped to. When the smallest sample (of mandrel diameter of 0.5mm) could be measured for a of 0.24.
The results are shown in figure 4.29. The results of the five samples are presented in a form of dependency between a and pre-tensing force in N. The two coils of smallest mandrels' diameters (i.e. sample 9 and 11) could have been pre-tensed to the highest forces and still exhibit deformations when heated. Also the results show that the smaller the a, the higher the force.

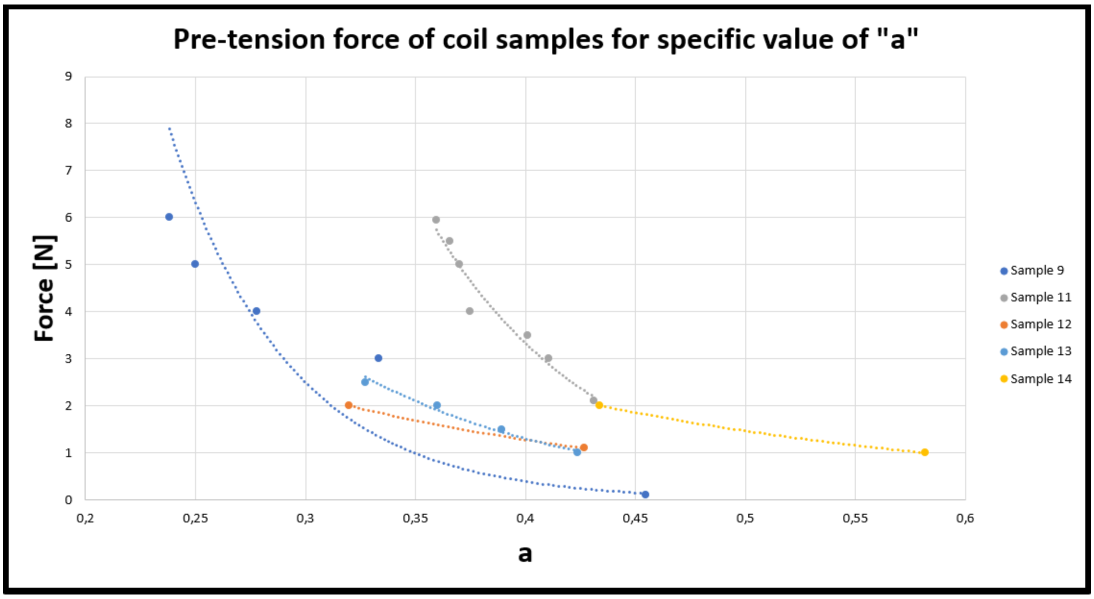

Figure 4.29: Pre-tension force of coil samples for specific a

Another measured value is the length of the samples in the pre-tensed state (x) and their length after applying heat, so the prime state (x_1). The results are presented in figure 4.30. The highest elongation is seen for samples 13, 11 and 9 - 205%, 197% and 182% accordingly.

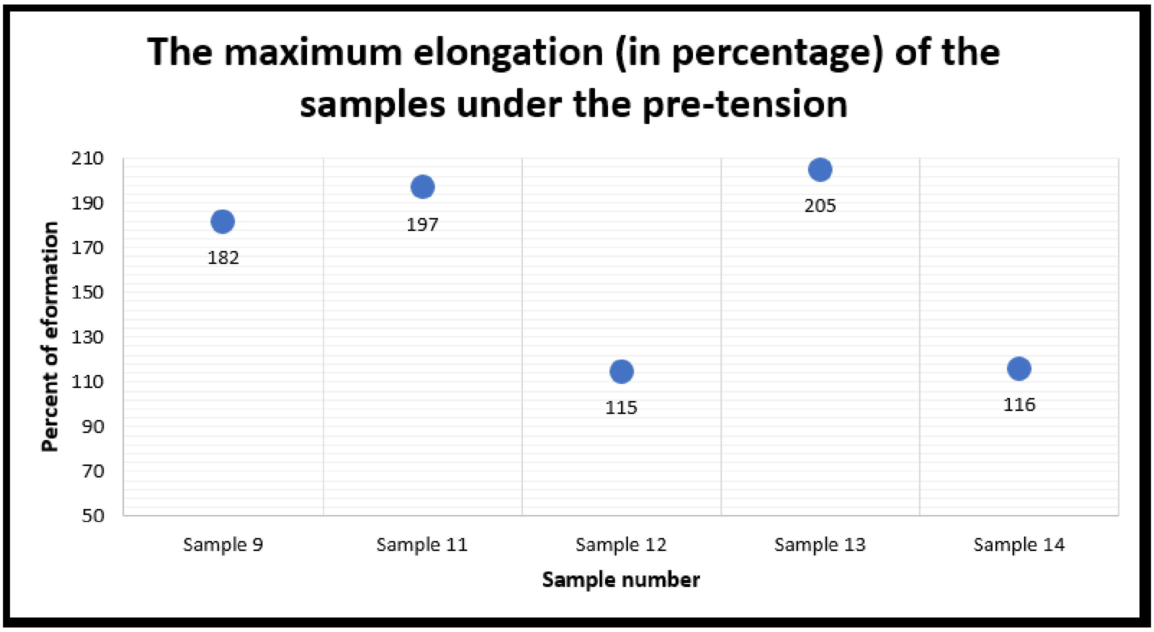

Figure 4.30: The maximum percent of elongation of the samples under the pre-tension

4.3.5. Iteration II

The resulted spring constant for certain pre-tensing force is shown for both samples: 9 ad 11 in figure 4.31. The spring constant is not constant. It decreases with increasing the tensing force. The values of spring constant are higher for the sample 9 - which has the inner (mandrel) diameter of 0.5 mm, pitch of 1 and wire thickness 0.5 mm.

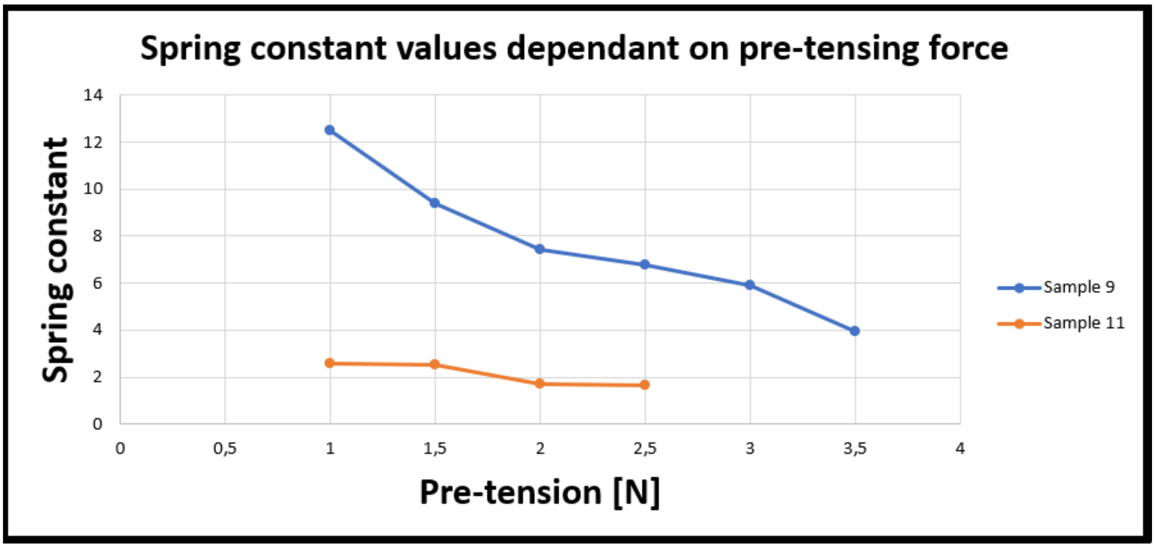

Figure 4.31: Spring constant dependent on pre-tensing force

The models of the coils are presented in figures below. Coils in the open state are shown in figure 4.32 - the sample 9 on the left and sample 11 on the right. The sample 9 has a mandrel diameter of 0.5mm and sample 11 has a mandrel diameter of 1.15mm. The length of the coils is equal which is indicated with the blue circles. When expended to the state shown in the figure, the most outer diameter is 6.5 mm for the sample 9 and slightly bigger 7.2 mm for the sample 11. The inner diameter is larger for the sample number 9: 4.5 mm.

For the sample 11, the inner diameter is equal 3.9 mm. In both cases the inner diameter is smaller than the outer diameter of the Fallopian tube (the size is between 5 and 5.5 mm).

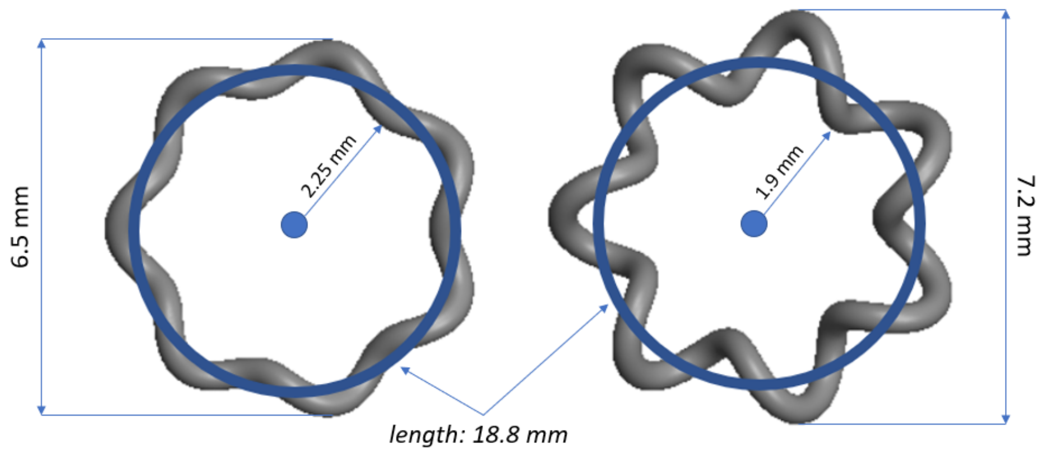

Figure 4.32: Two coils of wire diameter 0.5 mm and various mandrel diameter: on the left sample 1 and on the right sample 2 at the pre-activation state stretched to fit over the Fallopian tube. The blue circles indicate the length of the coils equal for both samples

In the figure 4.33, the same two coils are presented in the closed state (post-activation). They both have seven turns and pitch of both is 1 mm, therefore the length is 7 mm indicated with the black solid circle. The inner diameter are indicated with the dashed lines. Sample number 9 has an inner diameter of 1.2 mm (green dashed circle) when sample number 11 has an inner diameter of 0.60 mm (green dashed circle). The outer diameter is larger for sample 11 - 3.8 mm. Sample 9 has an outer diameter of 3.2 mm. The mandrel diameter of the coils is indicated with yellow arrow.

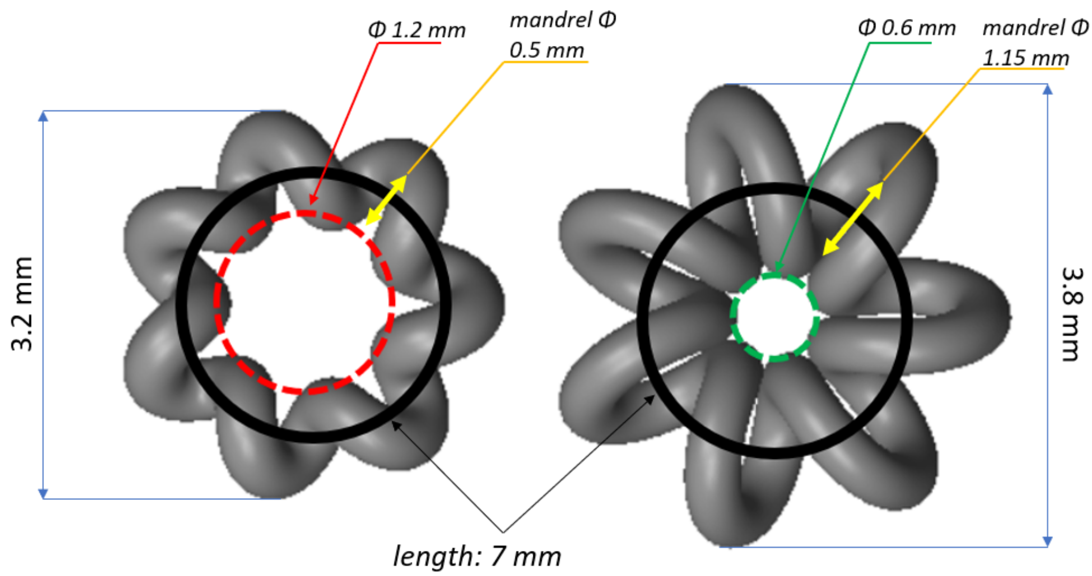

Figure 4.33: Samples number 1 and 2 with various mandrel diameter (indicated as yellow arrow). The inner diameter are indicated with dashed lines (red for sample 1, and green for sample 2), the black circles indicate the length of the coils: 7mm - equal for both samples

4.3.6. Final design

Based on test results and both iterations, the coils of 0.5 mm wire thickness, mandrel diameter of 1.15 mm and pitch of 1 mm is chosen to be used to build a final sample. It is presented in figure 4.34, placed over the synthetic Fallopian tube.

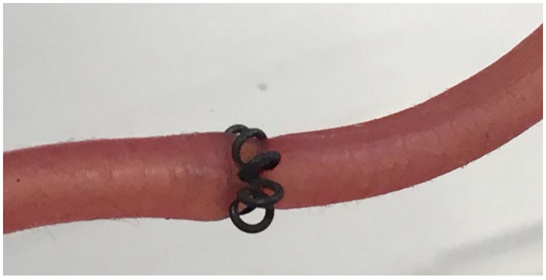

Figure 4.34: Final coil samples placed on synthetic Fallopian tube

The sample was tested for its ability to stop the flow withing synthetic Fallopian tube. The final design passed the test and the tube's lumen was sufficiently closed preventing water from going through the tube.

4.4. Discussion

4.4.1. Design

Designing process was based on the preliminary design provided by Opt Medical however the author was not limited from creating other solutions. In case the best results would have been obtained for completely different design, Opt Medical would include it in their design.

4.4.2. Prototyping

The prototypes of 3D wire are only made for the symmetrical radial group. It is due to the manufacturing limitation of such designs. Problematic turned out to be the plastic moulds. They could not be produced for samples in dimensions similar to other prototypes. Already, the made prototypes exceed the dimensions of other samples and could not be tested on Fallopian tubes. Also some other prototypes were not manufactured due to the complex design. Only samples which turned out to be sufficient for further examination are included in the results table.

Some of the thickest Kellogg's coils (1 mm wire diameter) could not have been connected and transformed into prototypes. They were to strong to squeeze them in order to clamp them as a next step. But since they are so big and strong, they could not benefit for the project of the sterilization device. Such forces are not required to clamp the Fallopian tube.

The method of using the 3D printed mould seems to be very promising. The mould can be printed in very small dimensions, however the angles between the pins are based on possibility to bend Nitinol. Therefore, the prototypes could not have been made due to the wire limitations and not manufacturing method.

4.4.3. Measurements

The test on Fallopian tube turned out to be an unnecessary step. The Nitinol wire is a part of a clamping mechanism and it is responsible for shape change and force generation but the whole device needs additional elements to provide a large clamping area - something like a silicone casing. The tests was not finish, as there was no relation between high potential force values obtained during the force measurement and ability to close the lumen.

Measured force is not a force exerted by sample to close it, but it is a potential force which indicates maximum tension under which the sample can be put. Above the force values recorded at the test, the sample will not change to its trained state when heated up. But for the purpose of this project, the force is a neat expression of differences between the prototypes.

The amount of variables between the prototypes does not help with finding relations on shapes and measured forces. For a better understanding the prototypes should at least were designed in a way to have similarity in a size and they could all close the lumen of the same silicone tube.

There is no correlation between the results from the two test: closing of Fallopian tube and force measurements. The reason is that the closing of the tube is caused by squeezing the sample to certain final state. If the inner area of the sample exceeds the dimension of Fallopian tube, there is no possibility the sample will be closed, even if at the force test the measured value was very high.

4.4.4. Iterations

Five coils of the same wire thickness but different pitch and mandrel were investigated. It seems that the bigger the mandrel used the higher the difference between the coil in cold martensitic state and hot austenitic state. But only for wires with the same pitch. The results are not surprising as the manufacturer provides a rule of a thumb that the smaller the mandrel the higher the forces. The additional test was carried out on two most promising samples. The smaller sample turned out to be stiffer, as its spring constant has higher values.

Initially the experiment would have contained the measurements of the relaxation of the sample coils after each measurements. It supposed to be perform as releasing the sample from a clamp so the sample would not have been pre-tensed anymore. The idea had to be changed as the constant releasing and clamping the sample caused a change in the results. Due to very small dimensions of the samples it is extremely difficult to clamp the coil in exactly the same spot as before. The length was changing followed by change in pitch and force. At the end the measurements were taken at once, so the sample was clamped all the time at the same spot.

After all measurements, the samples were released, heated and evaluated for their length. It was to check if the initial length changed after pre-tensing the samples. Indeed, all the samples encountered plastic deformations. It was noticed also already during the measurements when the length after the heating did not match the length from previous attempt. However, it could have been still caused by high pre-tension value, so that the additional measurement was take after releasing the tension.

Even though the at the second iteration, sample number 13 demonstrated high elongation rate, it was not chosen for further investigation. The samples 12, 13, and 14 have dimensions large enough to be dismissed from the additional analysis. Firstly, the implant is required to be small to not disturb the internal organs of female reproductive system. A coil made of samples 12, 13, and 14 would have significantly larger dimensions than the one made of coils number 9 or 11 due to the size of a mandrel. Secondly, the activation of the device is planned to be via power transfer. It is beneficial to reduce the amount of the wire to obtain the best activation settings. The last remark was not an official requirement for the device in this report, as the power transfer is very complex subject. The electrical characteristics will be coupled with the results of this report in another project.

4.4.5. Final Design

The final design is based on the results of the test on prototypes, thus the symmetrical coil is chosen to be the final design. It is manufactured in a way to have the inner area smaller than the inner diameter of synthetic Fallopian tube, which is about the size of $5mm^2$. In order to close the lumen, the diagonal of the sample inner area must be not larger than 2.5 mm long.

According to the design, the samples suppose to have about 6 to 7 turns to have the length providing the closing Tube's lumen.

4.5. Conclusion

Standardizing the coils dimensions to a which is the relation between coil's diameter and its pitch, resulted in comparison and choosing the most promising coils. Final design of the shape memory alloy clamping mechanism was found to be a symmetrical coil of wire diameter 0.5 mm, mandrel diameter 1.15 mm and pitch of 1 mm. The coil consists of seven turns which results in design suitable to place over the Fallopian tube in the stretched state. After activation, so when the heat is applied, the clamping mechanism is capable of closing the tube's lumen and thereby stopping the flow through the tube.

5 Discussion

5.1. Study on Nitinol

The study on Nitinol was useful and brought useful information for the further process of designing and developing the clamping mechanism. The type of a wire and settings of shaping it, were used in the another part of this report, i.e. Shape design. The most valuable finding from the part on Nitinol, was developing the method of training most complex and tricky designs. The method is a combination of two known approaches - using 3D printed moulds and using plaster. Instead of 3D printing metallic or ceramic part which could withstand the high temperatures in which Nitinol is shaped, the plastic parts can be printed instead. As they would melt in those high temperatures, a plaster casing is added. The Nitinol wire shaped according to the mould structure is embedded in plaster. As a result, plastic melts but plaster maintain the shape of a sample. The biggest advantage of this method is an ease of the procedure, low cost and freedom in shape designing. The research on Nitinol has allowed to find several gaps in the topic which are not covered in literature. Connecting Nitinol is still the part with many unknowns and it lacks a universal and simple solution. The properties of Nitinol are great but to achieve the most benefits from it, laborious and difficult research is required.

5.2. Test set-up

The test set-up development was successful and turned out to be a simple and cheap alternative to a precise multiaxial testing machine for biomaterials for the use in this project. The accuracy is high enough to compare diverse samples but not to obtain high quality results and precise numbers. The biggest disadvantage of this test set-up is its tendency to wear off with time and number of repetitions of the test. Many parts of the set-up are 3D printed from PLA material and they do not have resistance to wearing off. Also, the parts are not heat proof, which results in weakening of the structures, when the heat is applied. Even though the heat is not applied directly at the plastic parts, the heat transfer plays a role, and after several measurements the quality of the device decreases. Even though the test set-up provided data used for choosing the best shape, the reliability of it is questionable. The data is not validated by any other method, and the test set-up is not used in any other study. After a while, the set-up was loosing its accuracy. It was due to the wearing off of the components and weakening cause by exposing them to the heat. For more reliable results, the test set-up needs a redesign, including wear- and heat prove parts. The test set-up should be validated using calibrated devices.

5.3. Shape design

There were multiple creative designs of the clip, however the most suitable shape turned out to be a circular coil. Although, it was necessary to test the other shapes in order to eliminate them with a valid reason. For the coils the dependency was of the design on the force was found. For the samples of equal wire thickness the smaller the mandrel size, the higher the force they exert. The a factor was a good solution to compare the samples of different mandrel diameters. The forces for certain values of a could have been recorded and compared. The finding from this test was also that the larger the a (where $a = \frac{d_{in}}{p}$) the smaller the forces. Another limitation of the study concerns the method chosen to compare tested designs. The test should have been more structured and the number of variables should be minimized. The most important, the dimensions of the prototypes. They should be all designed to close the Fallopian tube and only then the force would be compared between them. Several designs seemed to be valuable, however the force measured during the test was not sufficient to further investigate those shapes.

6 Conclusion

Several shapes of the potential clamping mechanism were made and tested. It resulted in finding the strongest options, which after iterations, were translated into the final design - round coil of wire thickness 0.5 mm, with a mandrel diameter 1.15 mm and pitch 1 mm, consisting of seven turns. This design is sufficient to close the lumen of the Fallopian tube by itself. As the ability to close the lumen can be tested for the whole sterilization device and not only Nitinol wire, another method, different from the one proposed in this report, have to be found to establish exact clamping force. At the end, the clamping mechanism contains the Nitinol wire but has to be coupled with silicon casing for enlarging the area of clamping.

A

Appendix: Results table of Experiment I on Nitinol

Table A.1: Test results of the influence of time and temperature on activation temperature

No.	Temperature [°C]	Time [min]	Constraining method	A_s [°C]	A_f [°C]	Tight coil
1	500	5	Plate	85	88	yes
2	500	5	Plate and holes	85	88	yes
3	500	5	Plate and holes	85	88	yes
4	500	5	Plate and holes	83	86	**no**
5	500	5	Screw	85	90	yes
6	500	10	Plate	83	85	yes
7	500	10	Plate	83	84	yes
8	500	10	Plate and holes	82	84	yes
9	500	10	Plate and holes	83	85	yes
10	500	10	Screw	83	86	yes
11	550	5	Plate	87	89	yes
12	550	5	Plate and holes	86	88	yes
13	550	5	Plate and holes	86	88	yes
14	550	5	Plate and holes	87	90	yes
15	550	5	Screw	89	92	yes
16	550	10	Plate	90	92	yes
17	550	10	Plate and holes	91	93	yes
18	550	10	Plate and holes	91	93	yes
19	550	10	Plate and holes	N/A	N/A	N/A
20	550	10	Screw	91	94	yes

B

Appendix: Results table of validation test I on the test set-up

Table B.1: Results from a validation test I

No. of arms	Value on each arm	Sum	Sum after heating	Force on each arm
3	0	0	0.3	0.1
	0.5	1.5	1.5	0.5
	1	3	3.8	1.23
	1.5	4.5	5.8	1.93
	2	6	7.5	2.5
	2.5	7.5	8.8	2.93
	3	9	10.4	3.47
	3.5	10.5	11.5	3.8
4	0	0	0.4	0.1
	0.5	2	2.4	0.5
	1	4	4.2	1.23
	1.5	6	7.9	1.93
	2	8	9.9	2.5
	2.5	10	12	2.93
	2.6	10.4	12.2	3.47
	2.7	10.8	12.8	3.8
	2.8	11.2	12.5	3.8
5	0	0	1.4	0.28
	0.5	2.5	3	0.6
	1	5	6.3	1.26
	1.5	7.5	8.8	1.76
	2	10	11.4	2.28
	2.1	10.5	12.2	2.44
	2.2	11	12.7	2.54
	2.3	11.5	13.1	2.62
	2.4	12	13.4	2.68

C

Appendix: Results table of validation test II on the test set-up

Table C.1: Results from a validation test II

No. of arms	Value on each arm	Sum	Sum after heating	Force on each arm
3	0	0	0	0
	0.5	1.5	1.5	0.5
	1	3	3.1	1.03
	1.5	4.5	4.9	1.63
	2	6	6.5	2.16
	2.5	7.5	7.9	2.63
	3	9	9.5	3.16
	3.5	10.5	11	3.66
4	0	0	0	0
	0.5	2	2	0.5
	1	4	4	1
	1.5	6	6.4	1.6
	2	8	8.7	2.18
	2.5	10	10.6	2.65
	3	12	12.6	3.15
	3.5	14	14.6	3.65
	3.6	14.4	14.9	3.7
5	0	0	0	0
	0.5	2.5	2.5	0.5
	1	5	5	1
	1.5	7.5	8	1.6
	2	10	10.5	2.1
	2.5	12.5	13	2.6
	2.7	13.5	14.1	2.82
	3	15	16	3.2
	3.2	16	16.5	3.3
	3.4	17	17.6	3.52

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