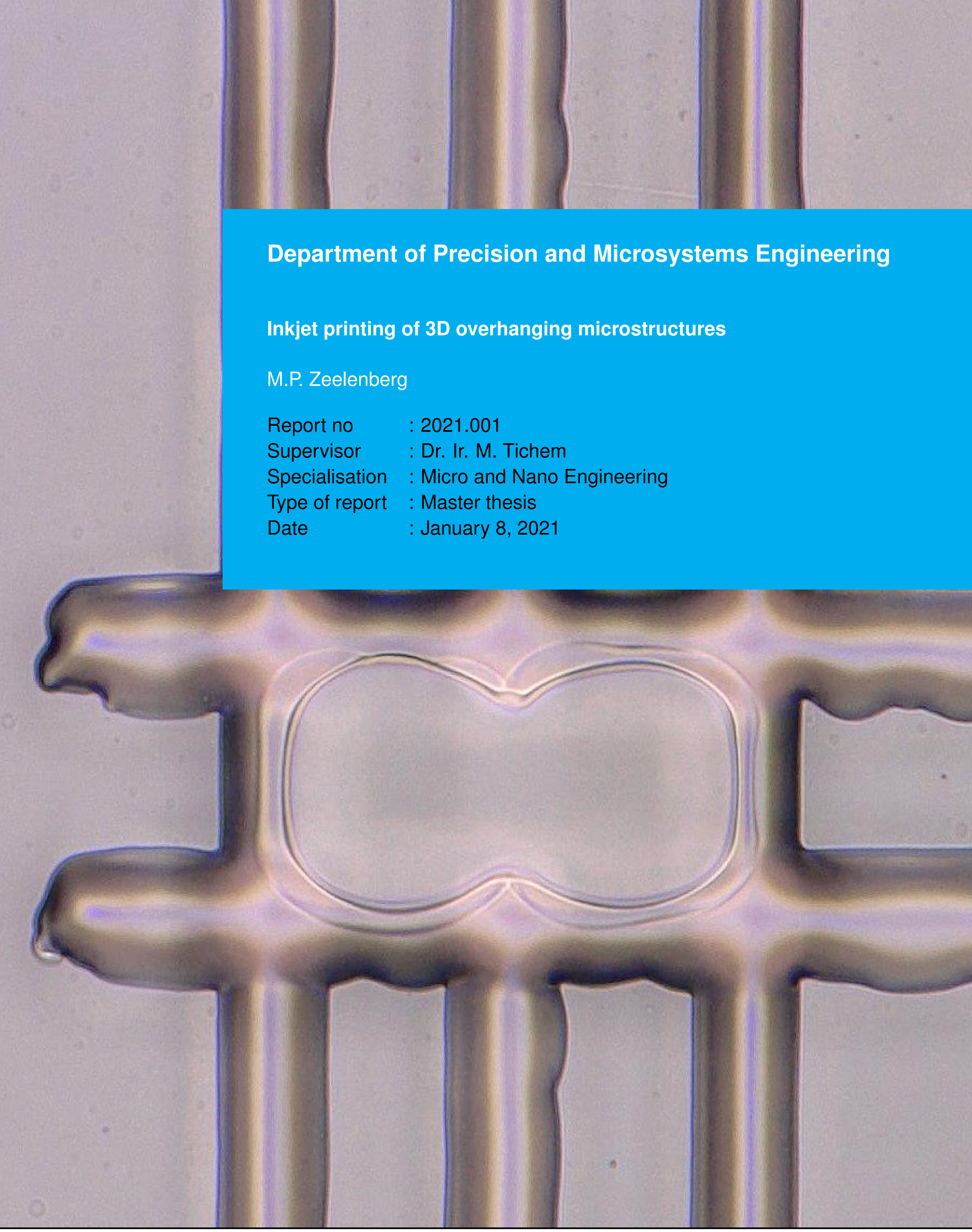

Department of Precision and Microsystems Engineering

Inkjet printing of 3D overhanging microstructures

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TU Delft Delft University of Technology

Challenge the future

Inkjet printing of 3D overhanging microstructures

by

Michiel Pieter Zeelenberg

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TU Delft

Preface

This is the result of my master thesis that I have been working on for the past year. I would like to thank the following people: Marcel Tichem for his guidance during the process. The lab technicians for their practical tips that made my life easier. My friends, family, roommates, and Vera for helping me stop thinking about small drops when needed. Last but not least I would like to thank my parents for their support during my master thesis and all six and a half years at the TU Delft. They give me the opportunity to grow as a person, thank you!

Michiel
Rotterdam, December 2020

Abstract

Freeform overhanging microstructures are used for various applications, for example in micro metamaterials. To create these structures industry uses a variety of manufacturing methods. There is a gap in manufacturing methods that can produce a microstructure at a high resolution and use multiple materials at individual locations. Inkjet printing can fill this research gap because multiple materials can be printed at individual points in structures by using multiple ink reservoirs. The goal of the research is to develop a method to produce overhanging microstructures using inkjet printing.

Using inkjet printing structures are produced bottom-up therefore a (sacrificial) support material is needed to support building material during the production. This material is later removed resulting in an overhanging structure. To allow removal of the sacrificial material both materials must cure differently. After considering different candidate materials, one building material (mr-UVCur26SF) and one sacrificial material (KL5315) were used during the manufacturing process. The sacrificial material is a positive photoresist and can be removed in a NaOH (1w%) solution after UV curing as opposed to the building material that solidifies after UV curing.

Two methods were used to increase the height of inkjet-printed structures. The first method is to print multiple layers of material on top of each other while curing the material between each layer, this is used to print the building material. This first method led to poor alignment between layers of sacrificial support material, therefore a new method is proposed to print the sacrificial material. Using the second method multiple lines of sacrificial material are printed next to each other without intermediate curing using a high cross-scan resolution of $4800[dpi]$ increasing the height of the structure.

Printing both materials close to each other to create an overhanging structure led to a loss of shape because the materials were attracted to each other. To solve this issue a new method is proposed using sacrificial dikes to prevent the building material from losing its shape. The first step of this method is to print and cure a grid of sacrificial support material. After that, the building material is printed in the free space between and on top of the dikes resulting in a microbridge once the sacrificial support material is removed. Using this method a microbridge with an overhanging part of approximately 50 $[\mu m]$ is created.

Contents

1 Introduction

1.1. General

Freeform overhanging microstructures are used for various applications, for example in micro metamaterials [1] and scaffolds for cell growth [2]. To produce these structures industry uses a variety of manufacturing methods.

Previously, a literature survey has been conducted on manufacturing methods of 3D structures on the micro- and nanoscale [3]. In the literature report, different manufacturing methods were investigated on throughput, use of multimaterial, and resolutions that can be reached. The following nine methods are described: Two photon lithography (TPL), Selective laser sintering (SLS) and Micro stereolithography (MSL), Microinjection molding (MIM), Inkjet printing (IJP), Fused deposition modeling (FDM), Laser induced forward transfer (LIFT), Nanoimprint lithography (NIL) and Electrochemical fabrication (EFAB). This survey showed that these manufacturing methods have their limitations.

One of the results of this literature survey is summarized in figure 1.1. Some methods can produce structures at a very small length scale but lack the ability to produce multimaterial structures. Others can produce multimaterial structures (using support material) but, do not have the best resolution. For example, Two photon lithography has a very good resolution but can not produce a multimaterial structure. Inkjet printing on the other hand can produce a structure with multimaterial at individual points but the resolution is not as good as with TPL.

This shows that there is a gap for manufacturing methods that produce structures with multimaterials at individual points and have a higher resolution. To benefit from this gap knowledge must be gathered about manufacturing methods. This research focuses on producing an overhanging microstructure using an inkjet printer.

Inkjet printing is an ideal method to create multimaterial structures because multimaterials can be printed within the same layer at different points, by using multiple ink reservoirs. The inkjet printing of multimaterial structures has many applications in manufacturing and engineering [4]. However, creating overhanging structures using inkjet printing is challenging because the structures are produced bottom-up and often the material has a low viscosity. This means that overhanging parts can collapse if not well supported. Since multiple materials can be used one of them could be a sacrificial support material. This support material will be printing during the process to make sure the structure does not collapse and is removed after the manufacturing is finished.

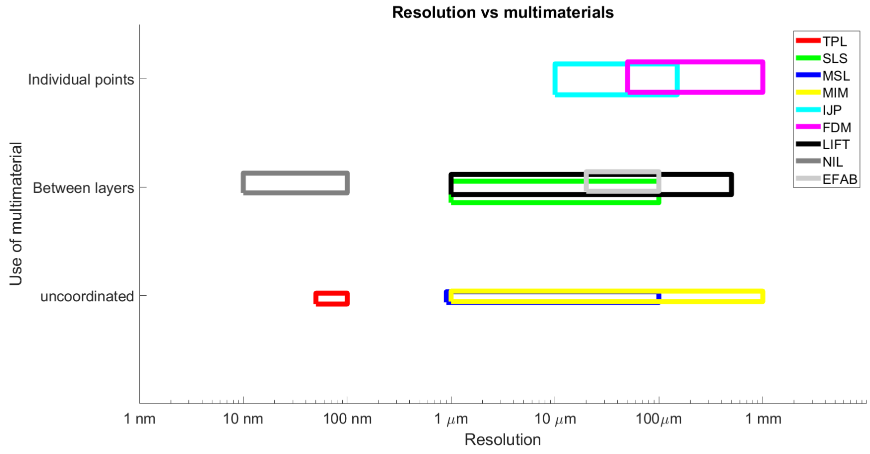

Figure 1.1: The resolution of some manufacturing methods and in what way multimaterials can be used, from Zeelenberg [3]

1.2. Problem statement and research questions

The goal of this research is

To develop a method to produce overhanging microstructures using inkjet printing

To accomplish this goal the following research questions are defined:

1. Which working combination of building material and sacrificial support material can be used?

   A compatible combination of building and sacrificial support material must be found. This will depend on the curing mechanisms of both materials and the removal of the sacrificial material. The building material should not be removed during the removal of the sacrificial material.

2. Which design and machine parameters affect the manufacturing process?

   Which parameters can be tuned so that structures can be built and better defined? What kind of results will the change in these parameters have on the structure?

3. How does the use of sacrificial material influence the characteristics of the building material?

   How does the combination of building and sacrificial material perform during experiments? What happens when the building material is deposited on the sacrificial material? And, what happens if the sacrificial material is removed from the substrate?

4. What are the limitations in shape and dimensions of the overhanging structures can one make using sacrificial material and an inkjet printer?

   What kind of structures can be made using inkjet printing and what is the size of these structures?

1.3. Approach

First, literature is found on the parameters that influence the printing process, and how they influence the manufacturing of structures. Also characteristics of the PiXDRO LP50, the inkjet printer used in this research, are investigated. Using this knowledge two materials, one building and one sacrificial, are chosen. Next, experimental prints are made, tuning the parameters to create stable lines for both materials. These lines are analyzed on height and width using optical microscopy and white light interferometry. The information gained from these experiments is used to create an overhanging structure using the two materials. The measurements of the overhanging structure are validated using a measurement made using a scanning electron microscope (SEM), to make sure an overhanging structure is created.

1.4. Report outline

First, general information about inkjet printing is presented. Secondly, the material selection is described, followed by the experimental procedure that is used. Next, the results of the first experiments are given and discussed. After that, procedures to create overhanging microstructures and produced overhanging microstructures are shown. To conclude a discussion of the results and a conclusion is given.

2

Inkjet printing

This chapter gives some background information on inkjet printing. Firstly, different types of inkjet printing are described. Secondly, the printability of materials is elaborated followed by information about the printer used during this research. To conclude, the parameters that influence the process are given.

Types of inkjet printing

To create objects using inkjet printing, liquid material is deposited on a substrate and cured to solidify it. The material is held in a reservoir and is ejected through a small nozzle at the end. In industry there are two main ways to print the material: continuous inkjet printing (CIJ) and drop on demand inkjet printing (DOD) [5], see figures 2.1 and 2.2. In CIJ, the material is continuously ejected from the nozzle. Using an electrostatic field the drops are either placed on the substrate or captured in the gutter. Material captured in the gutter is fed back to the reservoir to be used again [6]. In DOD printing the drops are only ejected when needed to create the structure. DOD printing can be done using heat (thermal DOD) or using a piezoelectric DOD printer. When using heat, a heating element vaporizes a small volume of material resulting in an increase of pressure in the cartridge followed by the ejection of a drop. Using a piezoelectric printhead, a piezoelectric actuator contracts increasing the pressure and ejecting a drop out of the nozzle.

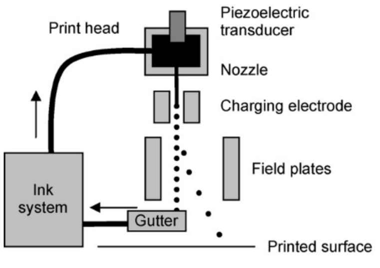

Figure 2.1: Schemetic illustration of a continuous inkjet printer, from Hon et al. [6]

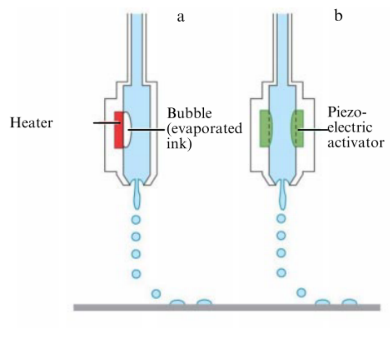

Figure 2.2: a) thermal DOD inkjet printing, b) piezo electric DOD inkjet printing, from Fritzler et al. [5]

Printability

Stable drops must be generated when jetting material from the nozzle to the substrate. This means that the material leaves the nozzle and forms one spherical drop without satellite drops. Whether stable drops are created depends on the following material properties: density, viscosity, and surface tension. Also, the diameter of the drop and flight velocity determined by the printer and printhead have an influence. These parameters can be combined in the Reynolds, Weber, and Ohnesorge number, see equations 2.1 till 2.3. The design space for printable materials can be made by plotting the Reynold and Weber number, see figure 2.3. However, it is a convention to rate printable materials by the Z factor, Z is given in formula 2.4. To print stable drops the Z value should be between 1 and 10 [7]. If the Z factor is lower than 1 the material is too viscous and can not form a drop. When the Z factor is higher than 10, satellite drops will be formed resulting in sputters on the substrate. Furthermore, the Weber number should be over 4, this will ensure that the drop has enough energy to escape the nozzle. Lastly, equation 2.5 should hold to prevent splashing when the drops hit the substrate. The value for $F(R)$ is given for a smooth flat surface and depends on the surface roughness only. Besides these dimensionless numbers, one could also only look at the viscosity, most commercial inkjet manufacturers specify a maximum viscosity of $20[mPas]$ [8]. So, when a material has a viscosity lower than $20[mPas]$ it should be printable using the PiXDRO printer.

$$Re = \frac{\rho d_{flight} v}{\eta} \tag{2.1}$$

$$We = \frac{\rho d_{flight} v_{flight}^2}{\sigma} \tag{2.2}$$

$$Oh = \frac{\eta}{\sqrt{\rho d_{flight} \sigma}} = \frac{\sqrt{We}}{Re} \tag{2.3}$$

$$Z = 1/Oh = \frac{Re}{\sqrt{We}} \tag{2.4}$$

$$We^{1/2} Re^{1/4} < F(R) < 50 \tag{2.5}$$

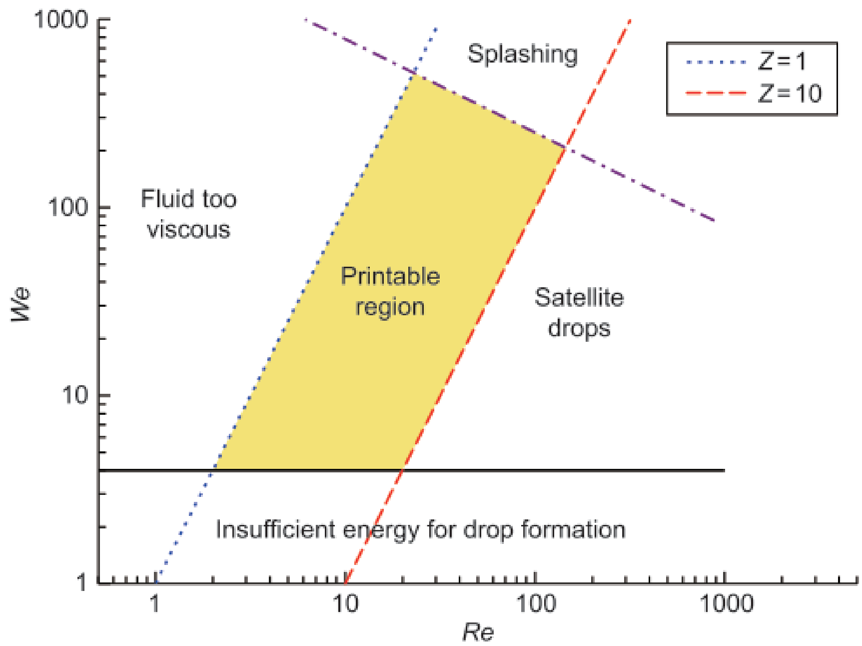

Figure 2.3: Designspace for printable materials using inkjet printing, from Derby [9]

Hardware

In this project a PiXDRO LP50 inkjet printer, which is a DOD printer made by SUSS MicroTech [10], is used in combination with a Dimatix 10 pL (DMC-11610) disposable cartridge, which is a piezoelectric actuated cartridge. This cartridge can hold up to $1.5[mL]$ of material, which is plenty when producing microstructures. The substrate bed of the printer can be heated up until $90[°C]$ and holds the substrate in place using vacuum clamping. Furthermore, the substrate can be exposed to UV light ($345[nm]$ to $385[nm]$) by the FireEdge™FE300 110x10AC365-3W [11] which has a peak irradiance of $3[W/cm^2]$ at $365[nm]$. The light exposes the full length of the substrate and has a width of $1[cm]$. So the substrate can be cured by heat and UV light without manually touching or moving the substrate. This is an advantage because the substrate does not have to be moved to a curing area outside the printer after printing each layer. The alignment between different layers will be better if the substrate is not moved during the printing.

The cartridge makes sure that the volume of the drop is always in the order of 10 pL. It is recommended by the supplier to change the resolution in steps of 25 drops per inch (dpi). The cartridge can be heated until $60[°C]$ [12], which can make the material more printable. However, it is recommended to keep the maximum temperature of the printhead below $50[°C]$ [13]. High temperatures can cause the nozzle to malfunction because the solvent might evaporate too fast. The reservoir of the cartridge can not be heated so the material should be able to flow to the printhead at ambient temperatures. The waveform to actuate the cartridge, and thus jetting the material can be tuned to create stable drops. This will further be explained in section 5.1.

Figure 2.4: PiXDRO LP50 printer by SUSS MicroTech, from SUSS MicroTech [10]

Parameters

Many parameters have an influence when creating microstructures using inkjet printing. These parameters are displayed in figure 2.5. On the left side the printer settings and material properties are shown. Arrows indicate what these inputs influence. During the report the influence of these parameters is elaborated in more detail. This flowchart is used as an orientation on which parameters can be tuned.

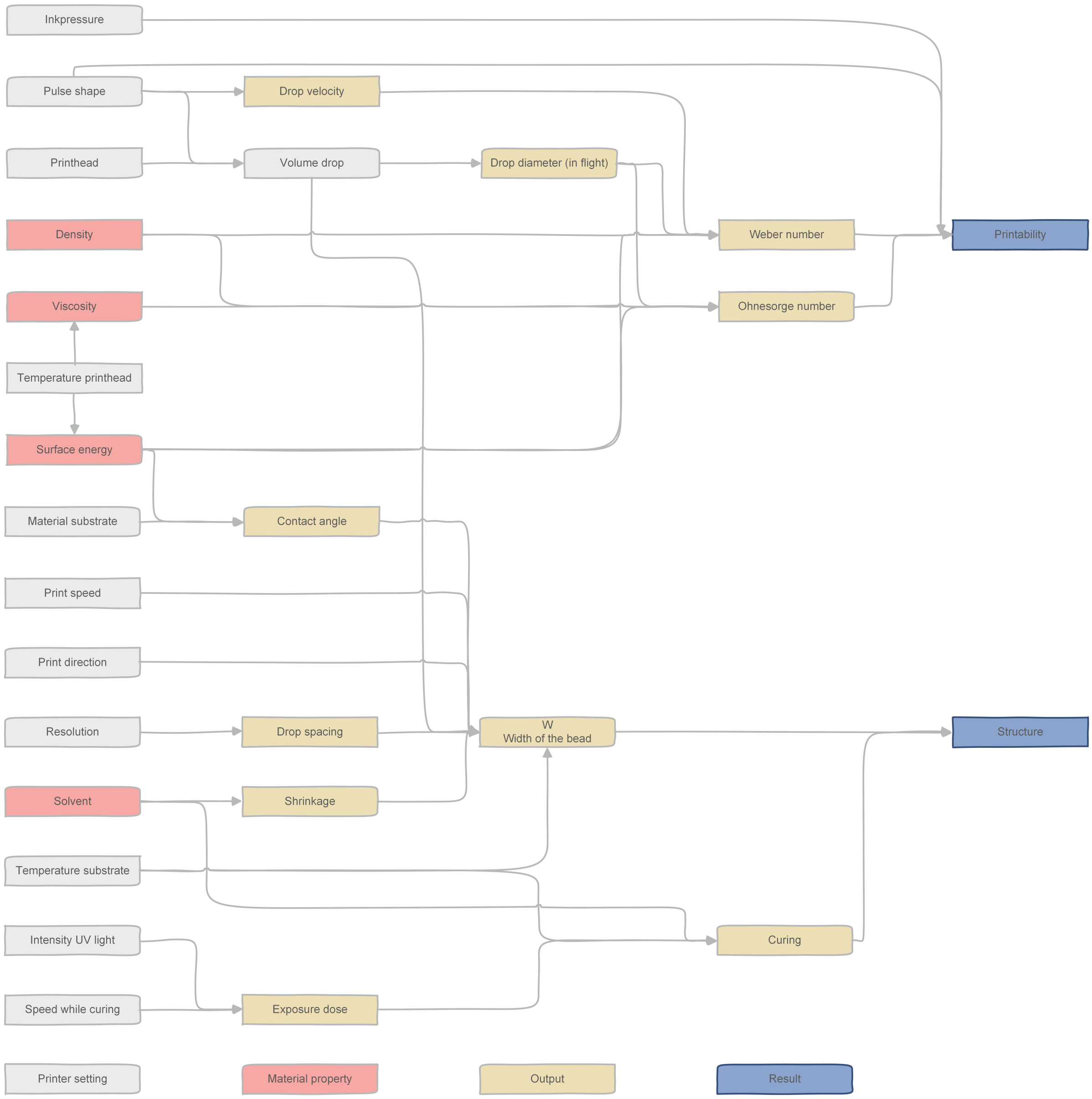

Figure 2.5: Parameters for inkjet printing of 3D structures

3

Material selection

In this chapter, the material selection is treated. First, the selection criteria are defined and elaborated. Then, candidate materials are presented and tested on the selection criteria. In the end, two materials, one building and one sacrificial are chosen.

3.1. Material criteria

The material criteria on which the materials are chosen are described below. These criteria are mostly based on the hardware of the printer and the safety measures in the lab. Another criterion is that the sacrificial and building material cure in a different way. By doing this the strength of one material can expose the weakness of the other. For example, if material A is removed easier after heating till $50°C$ and material B is harder to remove when heated till the same temperature, the strength of material B exposes the weakness of material A. A working combination of building and sacrificial material that looks most promising based on the criteria must be found. In this material selection only commercially available materials are considered.

Curing

Thermal

Thermal curing is done by heating the substrate which can reach a maximum temperature of $90[°C]$ as indicated in chapter 2. So materials that have a higher recommended curing temperature are less favourable because they need to be cured outside the printer or need longer curing at lower temperatures [14]. Furthermore, the heat is applied from the bottom of the PiXDRO printer. This is different from curing the substrate in an oven where heat is applied from all directions.

Sometimes photosensitive material, material which uses (UV) light to cure, needs a temperature treatment before it can be cured using light, this is called a prebake and can be given by the printer. However, it takes a lot of time to increase and decrease the temperature of the substrate. The substrate bed has no active cooling so cooling down happens by heat transfer to the environment. So, it would be convenient if the material does not need any thermal curing and if it does need thermal curing that this can be applied at a relatively low temperature. So that the temperature fluctuation is minimal.

If material needs a post exposure bake (PEB) after being exposed to (UV) light to complete the curing of the material, this can be done by heating the substrate in the PiXDRO printer or by heating it in an oven. This depends on the material and on the layer that needs the PEB. If for example the material that is printed first (usually the sacrificial material) needs a PEB, it would be best to do this PEB in the printer. This will result in better alignment between the layer(s) of sacrificial material and building material. When the second material needs thermal curing this can, depending on the number of layers be done outside the oven. However, it is important to keep in mind that the first material will have the same thermal treatment because it is deposited on the same substrate. It is preferred that the thermal curing is executed in the printer.

UV

The PiXDRO printer has UV light with wavelengths between $345[nm]$ and $385[nm]$. The material must cure when it is exposed to light with these wavelengths. The amount of energy on the substrate can be regulated by changing the intensity of the UV light and the speed at which the substrate travels through the illuminated area beneath the UV light. The wavelength at which materials cure is often displayed as i-line, which has a peak exposure of $365[nm]$.

Time

Curing takes time, some materials need thermal curing of a couple of minutes and some 30 minutes per layer. For now, the time it takes to cure a layer of a particular material is not taken into account unless it takes longer than one hour per layer. For the UV light, the time is not taken into account because the amount of energy that is needed to cure can be increased by increasing the intensity of the light.

Printability

One should be able to print the material so its properties must be inside the parameter space for the materials explained in chapter 2. The volume of the drop, which is determined by the printhead, influences the diameter of the drop when in the air. The velocity of the drop is determined by the waveform of the printhead. The waveform is material-specific and is later determined. The important material properties are density, surface tension, and viscosity. It is sometimes hard to determine all the values, therefore the printability is determined more qualitatively. When the viscosity is in the printable range and literature is found in which the material is printed, it is assumed that the material is printable.

Often materials are not printable in pure form because the viscosity is too high, to make it printable a solvent can be added to the material. Having a diluted material means there will be shrinkage because the solvent needs to evaporate. This results in a loss of precision because the shrinkage is hard to predict. So it is important to have a material that has preferably no solvent, which would result in minimal shrinkage. This will make it easier to create more precise structures because the height and shape of structures can better be predicted.

When a material is available in different viscosities, the highest printable viscosity is chosen. This will minimize the shrinkage because less solvent will evaporate. If the material turns out not to be printable, the temperature of the printhead can be increased resulting in a lower viscosity and making it printable again. However, the printhead temperature can not be higher than $50[°C]$ as indicated in chapter 2

Developer or remover

To create an overhanging structure sacrificial support material is used to support the building material during the production. After the printing and curing of the structure on the substrate, the sacrificial material must be removed. Depending on the material this is done by different (chemical) processes. The structure should not be removed in this process, so the developer or remover of the sacrificial material must not remove the building material. Due to safety reasons, it is important that the material can be handled safely in the lab and has a minimal risk of causing harm to the people handling it.

3.2. Candidate materials and selection

In table 3.1 and 3.2 on pages 12 and 13 the building and sacrificial candidate materials are displayed and their score on the criteria is given.

Selection of building materials

Five candidate building materials are considered in table 3.1. SU-8 can be cured using the printer but is not chosen because it can not be printed using the Dimatix printhead. InkOrmo and InkEpo are not chosen because they contain a solvent and need thermal curing which is not ideal. Suntronic and MR-UVCur26SF both look promising. Suntronic does not form an inhibition layer when cured but needs to be printed at a higher temperature which is at the limit of the printhead. MR-UVCur26SF can be printed at room temperatures and has no solvents, these advantages outweigh the disadvantages, shown in table 3.1 therefore MR-UVCur26SF is chosen as the building material.

Selection of sacrificial material

Three candidate sacrificial materials are considered in table 3.2. PMGI can be cured using the printer and should be printable. However, the advised curing temperature is far above the maximum temperature the printer can range, therefore it is not chosen as sacrificial material. Paraffin wax can be removed using water which is positive. The downside is that it can not be printed and for that reason it is not chosen. KL5315 is printable and can be cured in the printer. The recommended PEB temperature is just outside the range of the printer. So, the PEB should be performed at a lower temperature for a longer period of time. And, because the substrate bed has no active cooling, the substrate cools down slow this results in a longer period of time at higher temperatures. The downside of KL5315 is the harsh remover, after contact with the supplier Sodium Hydroxide is used as the developer which is less harsh. The concentration can be tuned so that it has minimal to no effect on the building material. In the end, KL5315 is the best of the three options and chosen as sacrificial material.

Summary

mr-UVCur26SF (Micro resist technology GmbH) as building material in combination with KL5315 (Kem-Lab Inc.) as sacrificial material look most promising to produce overhanging microstructures.

Candidate building material	Thermal curing	UV curing	Printability
Suntronic UV curing jettable insulator for low-K dielectric applications [15]	No thermal curing necessary [16]. This is positive.	No specification is found on the wavelength that is needed to cure the material however an advised dose to cure the material is $300[mJ/cm^2]$ [16]. This is not ideal	The material is developed for printing. However, the advised temperature for printing is $50°C$. The Dimatix printhead can print at this temperature however it is at the max of what is advised.
InkOrmo [17]	Lobos et al. [18] prebaked the material at $80-90[°C]$ for 5-15 minutes, this is in the range of the printer. No PEB has been applied to this material, which is positive.	The material cures using UV light with a wavelength ranging from $300[nm]$ to $410[nm]$ [19], this is in the range of the printer.	Is developed for printing at room temperature ($25[°C]$). It is mixed with a solvent to make it printable. This is not ideal.
InkEpo [20]	Lobos et al. [18] prebaked the material at $85-90[°C]$ for 5 minutes, this is in the range of the printer. A PEB at $100[°C]$ for 10 minutes is applied, this is just outside the range of the printer, not ideal.	The material cures using UV light with a wavelength ranging from $300[nm]$ to $390[nm]$ [19], which is positive.	Is developed for printing at room temperature ($25[°C]$). It is mixed with a solvent to make it printable. This is not ideal.
MR-UVCur26SF [21]	No thermal curing necessary. This is positive.	The material cures using UV light with a wavelength ranging from $365[nm]$ to $405[nm]$ [19], this is in range of the printer. When cured in the presence of oxygen an inhibition layer is formed, which is not ideal.	The material is developed for inkjet printing at room temperature in combination with nanoimprint lithography. It has no solvent [19], this is positive.
SU-8 [22]	Fakhfouri et al. [23] prebaked the material at $100[°C]$ for 15 minutes and applied a PEB at the same temperature and for the same time. The datasheet [22] bakes around the same temperature however, the time is shorter. This is just outside the range of the printer.	The material cures using UV light with a wavelength ranging from $350[nm]$ to $400[nm]$ [22]. This is positive.	Fakhfouri et al. [23] made it possible to print the material within the operating temperatures ranging from $60[°C]$ till $90[°C]$. This is outside the range of the printhead, This is negative.

Table 3.1: Candidate building materials and their score on the criteria

Material	Thermal curing	UV curing	Printability	Remover/ developer
Polydimethylglutarimide (PMGI) [24]	Recommended bake temperature is between $160[°C]$ and $210[°C]$ [24], this is outside the range of the printer. PEB is not necessary.	The material is a positive photoresist and can be removed using a developer after being exposed to i-line ($365[nm]$) UV light. This is positive.	PMGI is available in viscosities below $20[cst]$ so it should be printable [24]. This is positive.	Foulds et al. [25] used TMAH as a developer.
Paraffin wax	No thermal curing necessary, this is positive	No UV curing necessary, this is positive	Tse et al. [26] printed the material at the optimal printing temperature of $75[°C]$ This is outside the operating range of the printhead.	Nawada [27] used water at $70[°C]$ and 100% cyclohexane to remove the wax.
KL5315 [28]	Prebake the material at $90[°C]$ for 1 min. PEB is recommend at $115[°C]$, this is just outside the range of the printer.	The material is a positive photoresist and can be removed using a developer after being exposed to i-line ($365[nm]$) UV light. This is positive	The material is normally used for spin coating. But the viscosity is lower than $20[cst]$ so it should be printable, this is positive	Developed for .26N TMAH developers [28]

Table 3.2: Candidate building materials and their score on the criteria

4

Experimental procedure

In this chapter experimental procedures are explained.

4.1. Substrate selection

The criteria for choosing a substrate are determined by the process and the materials that were chosen in the previous chapter. During the process, the substrate is heated till $90[°C]$, exposed to UV light, and placed in a NaOH solution. The substrate must be able to survive these steps. Microscopes are used to visually look at the structures and perform measurements. To see if all the sacrificial material is removed it would be convenient to look from multiple angles at the structure so a transparent substrate is preferred.

Furthermore, the materials must adhere to the substrate so that the precision in the building process is secured. When looking at the website of MR-UVCur26SF [29] it is seen that many types of substrates can be used. A primer, mr-APS1, is advised to promote adhesion but it is not necessary to apply on the substrate. KL5315 adheres to a variety of substrates including silicon and glass [28]. But hexamethyldisilazane (HMDS) is advised as a primer to increase the adhesion.

Although primers are advised they are not used during the process because both materials are printed alongside each other. It would be a challenge to deposit both primers at the right location on the substrate. If both primers were deposited at the right location the alignment in the printer has to be perfect so both materials are placed on the right primer. However, the substrate is cleaned for 10 minutes using acetone in an ultrasonic cleaner, followed by 5 minutes of cleaning by isopropanol. In the end the substrate is blowdried.

In the lab silicon and glass substrates are widely available, and both materials should adhere to these substrates. When a glass substrate is chosen it is possible to look from the bottom through the glass substrate at the structure. By looking from the bottom one can look and see if there is sacrificial material left after the development step. Therefore it is chosen to print the microstructure on a glass substrate (VWR microscope slide, ground edge). For some first experiments silicon substrates were used due to technical reasons.

4.2. Printing

Images are printed by moving the substrate in y direction beneath the printhead, each pass is called a swath. After each swath the printhead moves in the x direction. Printing when the substrate moves up and down is called bi-directional printing. Only printing when the substrate moves down is called uni-directional. It is also possible to print when the substrate moves up, this is uni-directional reverse printing. These three types of printing are also possible when printing in the x direction.

During the ejection of a drop, and the landing on the substrate, the substrate moves. Due to this movement, the drop lands on a different position compared to a non-moving substrate. This error is called the Time of flight error (TOF-error) [30]. The TOF-error is different when printing uni-directional or uni-directional reverse. This results in a loss of precision between swath printed from top to bottom and from bottom to top. Therefore only uni-directional printing is used during this research.

Velocities and volumes of drops ejected by each nozzle are slightly different. This will give a loss in precision so in this research only one nozzle per printhead is used to print the material. This will give the best repeatability. Once a drop is ejected a test print is done so that the offset of the nozzle can be corrected. By compensating for this offset the image is printed at the right position on the substrate.

The cartridge of the building material is covered with tape to prevent it from curing by UV light coming from the environment. The cartridge of the sacrificial material is not covered.

4.3. Recipes

The printer used can only print two-dimensional images. This means that a three-dimensional structure is build up by printing multiple two-dimensional images on top of each other. Bitmap files consisting of only black and white pixels are printed in this research. Each black pixel corresponds to a single drop of material, a white pixel is the lack of material.

The resolution of the printer is defined by the drops per inch (dpi) that are placed in a specific direction. The dpi can be used to determine the distance between the centre of two drops. The in-scan resolution is the resolution in the print direction and the cross-scan resolution is the resolution perpendicular to the print direction. Using y uni-directional printing the in-scan resolution is in the y direction and the cross-scan resolution is in the x direction

To determine the centre to centre distance between lines in an image formula 4.1 can be used. In this formula dpi is the resolution, in drops per inch, of the printer in the x or y direction. N_{pixels} is the number of pixels in that specific direction. The amount of micrometers in an inch is 25400. Keep in mind that the spreading of drops is not taken into account, this will most likely result in longer structures.

Printing using a solvent can give (start-up) problems due to the evaporation of the solvent [13]. When the material is not jetted, the solvent can evaporate in the nozzle leading to inaccuracies in the printing process. To overcome this problem a startup zone is introduced in every image as can be seen in figure 4.1. In this startup zone the nozzle ejects a couple of drops so that at the end of the startup zone the drops are stable. After the startup zone a small blank area is placed before printing the intended line. The startup zone is located before every swath that is printed.

$$L[\mu m] = \frac{25400}{dpi}(N_{pixels} - 1) \tag{4.1}$$

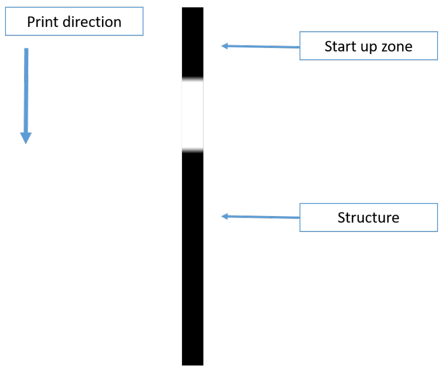

Figure 4.1: Single line with a startup zone located above the line

4.4. Measurements

In the beginning, drops are analysed using the advanced drop analyses system on the printer. This shows if stable drops are formed and what their volume and velocity is. If a nozzle ejects one coherent drop it is used for printing.

To see if a print is successful the first results can be analysed using a camera on the printer. This camera gives a top view of the substrate, uniform line formation and the position of the print are visible and one can determine whether the results are sufficient to continue building the structure. Using this camera it is also possible to take photos which can be used to see differences in a structure before and after curing. It is unfortunately not possible to zoom using this camera but the individual drops created in this research can be seen.

Once the structure is created it can be analysed using the Keyence digital microscope VHX-6000. The magnification of the Keyence is bigger than that of the camera on the printer so the results can better be interpreted. The substrate can be investigated from the bottom by looking through the glass which is not possible when the substrate is in the printer. Tuning the light helps the visual inspection of the printed structure.

After visual inspection, the substrate is scanned using a white light interferometer (Bruker), this gives a height profile of the structure. This can be used to determine the height and width of the structures. The data gathered is translated and rotated to compensate for possible misalignment of the substrate beneath the interferometer. Unfortunately, some noise is introduced when measuring the edges of the structures. So, to determine the width of a structure a threshold of $200[nm]$ has to be met. The height profile is measured at approximately the same position of the structure before and after development steps. It is difficult to find the same position because the substrate is removed from the scan area and placed back after development.

It is hard to look at the sides of the structure using the Keyence and interferometer. However, the building material is transparent so differences in the red sacrificial material, before and after development, should be visible. To make sure an overhanging structure is created the substrate is sputtered with gold, and a Jeol JSM-6010LA scanning electron microscope (SEM) is used to check the overhanging part. The results are compared with the visual photos and the data from the interferometer.

5
Results

The results are described in six different sections: stable drops, lines, planes, curing, height control, and the removal of material. These experiments are used to gain knowledge about the behaviour of the materials and to find the right settings for the printer to print patterns and lines. In the end a summary of the results is given.

5.1. Stable drops

Stable drops are a must when depositing material at a precise location using inkjet printing. First, the strategy to create stable drops is explained, followed by a description of the waveform that is used by this printhead and how this affects the drop formation. In the end, the waveform used for both materials is shown and the drop characteristics are explained.

Strategy

In chapter 2 some theory is introduced about stable drop formation during printing. Important print parameters are the velocity and the diameter of the drop, which can be influenced by tuning the waveform (time or voltage) and negative pressure in the cartridge. The viscosity and surface energy of the material can also be tuned by changing the printhead temperature.

Looking at the printability (figure 2.3) the following things can happen

- A material is too viscous, when this happens the printhead temperature can be increased to make the material less viscous and make it printable.
- It is also possible that the material does not have enough energy to leave the nozzle. When this happens material can be seen at the bottom of the printhead using the drop analysis system of the printer. To overcome this challenge, the voltage of the waveform can be increased. Increasing the voltage leads to an increase in the drops velocity [31]. A higher voltage also results in bigger drops, which could be undesired when building microstructures.
- The last thing that can happen is that satellite drops are formed, these are small drops that follow the main drop. This leads to inaccurate material deposition on the substrate. By decreasing the printhead temperature, with the lower boundary being the ambient temperature, the viscosity can be increased. A higher viscosity results in a smaller Reynolds number and possibly preventing satellite drops. However, some sources [13] claim that increasing the temperature can lead to more stable drops but it can also lead to more failures because the solvent will evaporate faster. It is known that the formation of satellite drops is proportional to the ligament length of the drop [32]. The ligament length is proportional to the velocity so it might be an option to decrease the velocity by changing the waveform of the printhead.

It is also possible that the material has a low viscosity and escapes the printhead at undesired moments and to prevent this from happening, the negative pressure in the cartridge can be increased. When this negative pressure is sufficient the material will not leak out of the cartridge, but when it is too high the material can not be printed.

Waveforms

An example of a waveform, i.e. changing voltage over time, to actuate the printhead is given in figure 5.1. Higher voltages lead to more deflection of the piezo actuator in the printhead. Steps 3 to 6 have the most influence on the drop formation [31]. After a small contraction in step 2, the voltage is decreased during step 3 filling the nozzle chamber. In step 5 the voltage is increased, contracting the actuator, increasing the pressure, and jetting the drop.

By changing the voltage, the amount of deflection of the actuator is controlled resulting in bigger or smaller drops. If the slope in step 5 is steeper the actuator contracts faster resulting in a higher drop velocity.

To create a stable drop it is advised to first find a voltage at which drops are formed [31]. In this research, the voltage and temperature are increased until drops are formed if there are no drops in the beginning. This strategy is chosen because the materials have a relatively high viscosity. Once drops are created the waveform is tuned to stabilise the drops creating one coherent drop. When tuning the waveform steps 3 to 6 are first tweaked because they have the most influence on the drop formation. The other time steps of the waveform are set close to the default values advised by the manufacturer depending on the stability of the drop.

The window at which a material is printable might be very narrow. Therefore, once a setting is found that creates stable drops, it will be used for all prints. As described earlier, only one nozzle is used because drops can have a slightly different size or volume per nozzle.

Drops need time to stabilise and form one coherent drop of material after being ejected from the nozzle. This has as a result that the printhead has to be located at a sufficient height above the substrate. This height is typically around $1mm$.

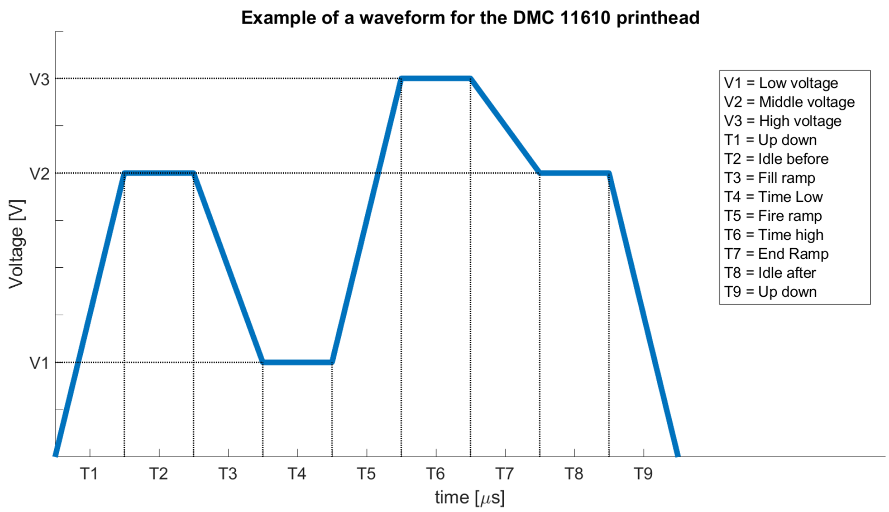

Figure 5.1: Example of a DMC printhead waveform. Based on: [13]

Results

The waves that jet stable drops from the nozzle to the substrate can be seen in figure 5.2. Some small variations in this waveform can also result in stable drops but, for all prints, this wave is used in combination with the printhead temperature and pressure shown in table 5.1. For the sacrificial material, KL5315, the temperature had to be increased to form stable drops. When the temperature is lowered the jetting stops. The building material, mr-UVCur26SF, leaks out the printhead when the pressure is closer to zero. The temperature is also higher than the recommended printing temperature $(25°C)$ this is done so that the temperature is always above the ambient temperature ensuring consistency throughout the project.

The volume, drop diameter, and drop velocity of both materials are also shown in table 5.1. The volume of both drops is in the order of $10[pL]$. To derive the diameter of the drops formula 5.1 is used. It shows the relation between the diameter (D) and the volume (V) of the drop when it is assumed that the drop has the shape of a perfect sphere.

Figure 5.3 shows unstable drops of sacrificial material being ejected using the standard waveform. Figures 5.4 and 5.5 show the ejected stable drops using the waves in figure 5.2 and the printer settings in table 5.1. In figure 5.4 the difference between the nozzles is clearly shown. Using the same settings the drop volume and drop velocity is different.

$$D = 2(\frac{3V}{4\pi})^{1/3} \tag{5.1}$$

Material	$Pressure[mbar]$	$T_{printhead}[^{\circ}C]$	$V_{drop}[pL]$	$D_{drop}[\mu m]$	$v_{drop}[m/s]$
KL5315	-0.5	38	14.2	30.0	5.04
mr-UVCur26SF	-8.0	28	12.3	28.6	4.33

Table 5.1: Printer settings and results when printing single drops

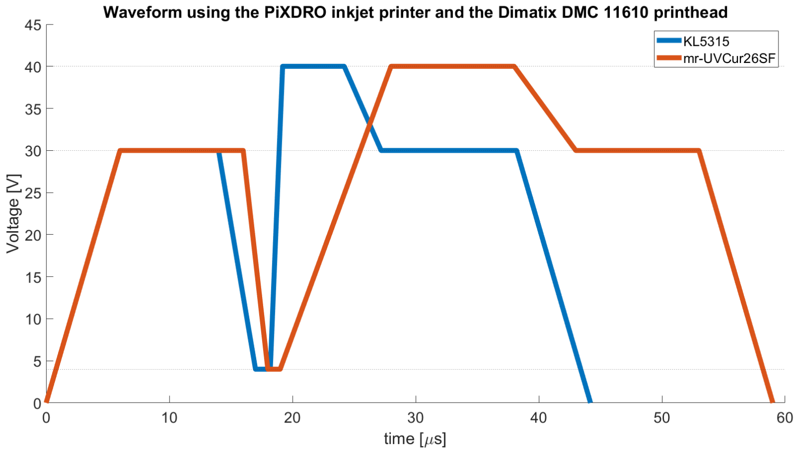

Figure 5.2: Waveform used to print KL5315 and mr-UVCur26SF

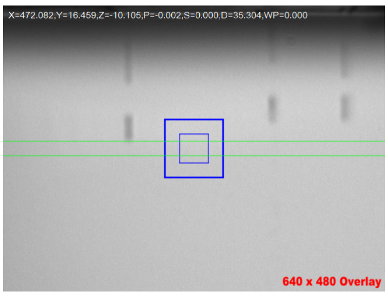

Figure 5.3: Unstable drops of sacrificial material

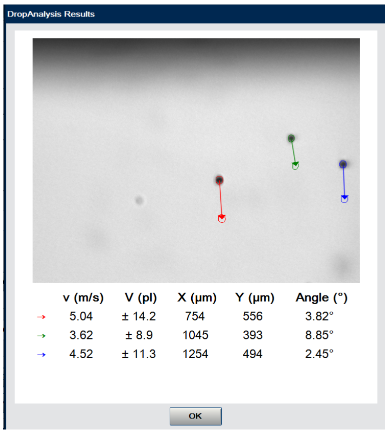

Figure 5.4: Advanced drop analysis of KL5315, the sacrificial material, only the left drop is used to produce structures

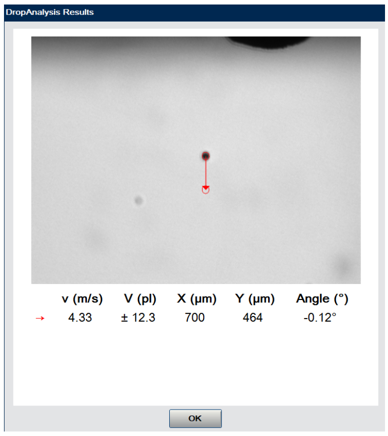

Figure 5.5: Advanced drop analysis of mr-UVCur26SF, the building material

After printing single drops the contact angle is determined using the interferometer. The results are shown in table 5.2. The contact angle of the sacrificial material is not determined using a single drop because it was too small. Due to evaporation the drop shrunk a lot. The contact angle of the sacrificial material is determined by measuring the angle of lines printed.

Material	Contact angle $[deg]$	$D_{droponsubstrate} [\mu m]$
KL5315	9.2	60
mr-UVCur26SF	8.8	83

Table 5.2: Contact angle of different material and the diameter of a single drop on a substrate

Discussion

The waves that are used to print stable drops only work for the nozzles that are used. The drops of sacrificial material printed by other nozzles are unstable using this waveform. If a different printhead is used it might be that all nozzles jet unstable drops using this waveform.

It is interesting that the measurements show that the contact angles of both materials are close to each other. This would suggest that the diameter on the substrate of individual drops would be almost the same since the volume of the drops is in the same order of magnitude. However, the diameter of the materials on the substrate differs significantly. This would suggest that the contact angle of KL5315 was bigger before the solvent evaporated and that the contact angle measurements are inaccurate. Only considering the diameter on the substrate suggests that KL5315 has a higher surface energy since mr-UVCur26SF wets the surface more than KL5315.

5.2. Lines

Theory

When printing drops of material close enough to each other lines can be formed. The centre to centre drop spacing is determined by the resolution in dpi and the number of micrometers in an inch, see formula 5.2

$$\Delta x[\mu m] = 25400/dpi \tag{5.2}$$

Soltman et al. [33] described the following five different line behaviours when printing. The relation between the drops spacing and the time interval between drops (delay) can be seen in figure 5.6

- Isolated drops, the drops are too far apart to interact and form a line.
- Scalloped line, the drops are close enough the interact but the one still sees the individual drops. These lines are smaller than the diameter of the drops.
- Uniform line, the drops are at the right spacing and form a uniform, stable line.
- Bulging line, the drops are too close together bulges are form separated by straight lines. These lines are wider than the diameter of drops.
- Stacked coins, when the temperature of the substrate results in the evaporation time of drops to be shorter than the jetting period, a single drop is dried before the next drops lands on the substrate, this looks like stacked coins.

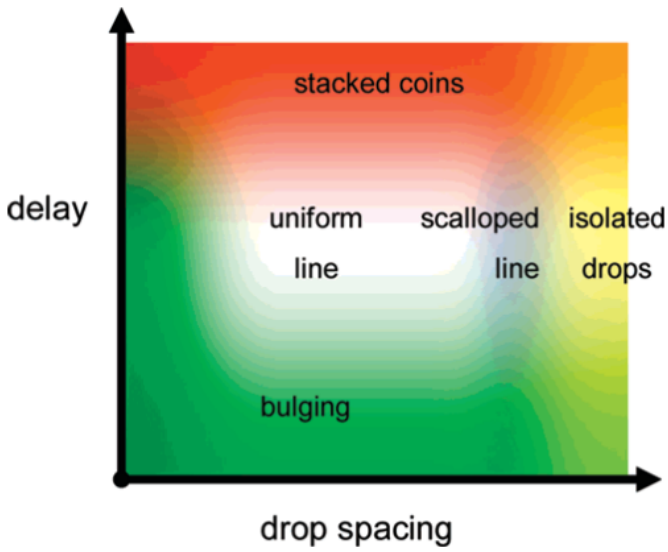

Figure 5.6: Relations between drop spacing and the delay between drops, from Soltman et al. [33]

Besides this more qualitative relation between different printer settings and line behaviour, models exist that predict the width of lines. However, the stability of lines is still mostly determined by practical trial-and-error iterations [34]. These models (most of the time) assume perfect conditions such as conservation of volume, stable contact angle, neglection of gravitational forces, and constant cross-section. Stringer et al. [35] gives a formula to calculate the width of a line, see formula 5.3. The formula assumes conservation of volume and determines the width of line based on the initial drop volume expressed by the diameter d_0. The other two parameters are the centre to centre distance between drops (Δx) determined by the resolution of the drop and the contact angle of the material. The sacrificial material contains a solvent to be able to print the material. This solvent will evaporate after being printed therefor the conservation of volume condition does not hold so the formula is not used for the sacrificial material. However, the formula is used to predict the width of the building material.

$$w = \sqrt{\frac{2\pi d_0^3}{3\Delta x(\frac{\theta_b}{sin^2(\theta_b)} - \frac{cos(\theta_b)}{sin(\theta_b)})}} \quad (5.3)$$

Strategy

To determine the resolution at which stable straight lines are formed, a single line is printed using a different resolution in the in-scan direction. Since the line is only one pixel wide the cross-scan resolution does not matter. The printer settings shown in table 5.3 are used during the printing of a single line in this experiment.

Setting	Value
Print speed	25 [mm/s]
Cross-scan resolution	500 [dpi]
In-scan resolution	varied [dpi]
Print direction	uni-directional
$T_{substrate}$	$40[°C]$

Table 5.3: Printer setting for printing a single line

Sacrificial material

The result of varying the in-scan resolution and printing lines using KL5315 can be seen in figure 5.9. Stable uniform lines are created from 500 dpi or higher. Below this resolution either no line is formed at all ($300[dpi]$), or a scalloped line ($400[dpi]$) is formed. Increasing the resolution does not seem to give a bulging effect, the lines are still uniform. During this experiment the resolution is not high enough to create bulging lines. When printing a single line of sacrificial material a resolution of 500 dpi is chosen so that uniform lines are printed with the smallest width.

Although not preferred the sacrificial material in figure 5.9 is printed on a silicon substrate due to technical issues. When printing on a glass substrate the results might have been different.

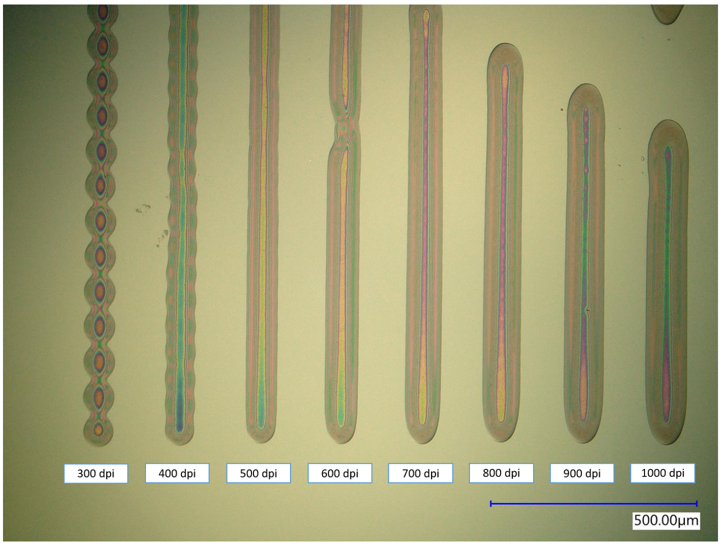

Figure 5.7: Lines created at various in-scan resolutions using KL5315, the sacrificial material

Building material

The results of varying the in-scan resolution and printing lines with mr-UVCur26SF can be seen in figure 5.11. Looking at this figure it is seen that no stable line is created when the in-scan resolution is set to 300 or 400 dpi. For now, printing using an in-scan resolution of $700[dpi]$ is used to further print because this gave the most stable lines.

When looking at the width of the lines in table 5.4 it can be seen that the actual width is not the same as the predicted width using formula 5.3 and the values found in the previous section.

A cluster of material can be seen at the bottom of the lines. Hsiao et al. [34] says that empirical evidence suggests that the formation and stability of a line are at least partly related to the liquid flow from the impacting drop to the pre-existing drop. The pressure difference between two following drops could be the reason for this. The pressure difference can be reduced by increasing the jetting frequency and print speed. However, increasing the print speed resulted in unstable lines.

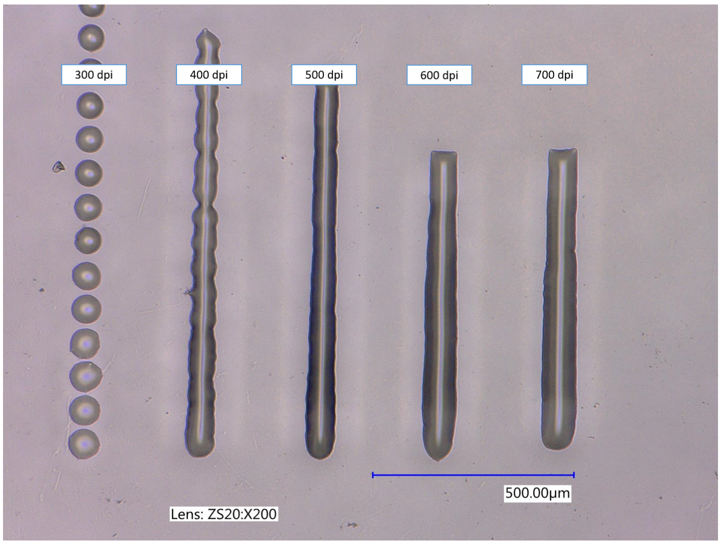

Figure 5.8: Lines created at various in-scan resolutions using mr-UVCur26SF, the building material

In-scan resolution $[dpi]$	Stringer width $[\mu m]$	Printed width $[\mu m]$
600	106.2	74
700	112.8	82

Table 5.4: Width of single lines of building material printed and predicted

Discussion

It would be better to print the sacrificial material on a glass substrate to see if the results are the same. The contact angle will be different on glass resulting in a different width of the line.

Predicting the width of the building material is not accurate in this case. This might come due to the contact angle measurements which might not be precise enough. It could also be possible that the building material shrinks when it is solidified. During this research formula 5.3 not used because predicting the width is more precise using experimental test prints.

Creating smaller lines is possible when changing the volume of the drops which is mostly determined by the printhead, and in this case it is not possible to change it. Changing the surface energy so that the contact angle increases could also help to make the line smaller and higher.

5.3. Planes

Strategy

The in-scan resolution for both materials is determined in the previous section, this is used to determine the right corresponding cross-scan resolution. To find the cross-scan resolution three lines are printed next to each other, by varying the cross-scan resolution the lines are placed closer and closer together until at one point they interact creating a plane. The resolution is varied from 300 to $700[dpi]$ in steps of 100. All other printer settings are shown in table 5.5

Setting	Sacrificial material	Building material
Print speed	25 [mm/s]	25 [mm/s]
Cross-scan resolution	varied [dpi]	varied [dpi]
In-scan resolution	500 [dpi]	700 [dpi]
Print direction	uni-directional	uni-directional
$T_{substrate}$	$40[°C]$	$40[°C]$

Table 5.5: Printer setting for printing planes

Sacrificial material

In figure 5.9 the results for the sacrificial material can be seen. At a cross-scan resolution of $300[dpi]$ individual lines can be seen. At higher resolution the lines are printed close to each other but still a stacked coins effect is visible. This might come due to the evaporation of the solvent before the second line is printed.

In figure 5.10 a startup zone is also visible before the lines are printed. In the startup zone the drops are unstable and are not printed at the right position. After the startup zone the structures are more stable but instability can still be seen in the structures.

In this experiment the sacrificial material is printed on a silicon substrate due to technical issues which might give different results compared to lines printed on a glass substrate.

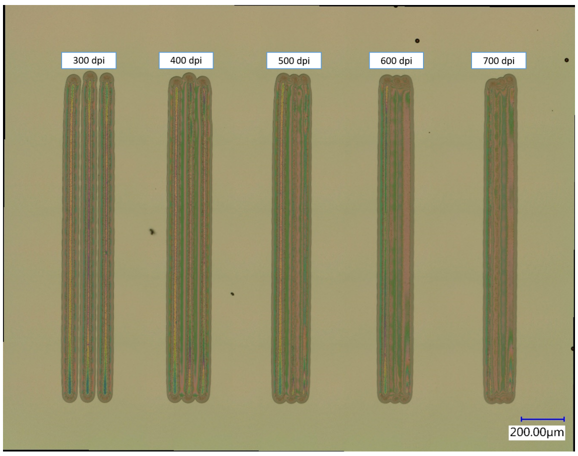

Figure 5.9: Planes created using KL5315 by varying the cross scan dpi from 300 (left) till 700 (right)

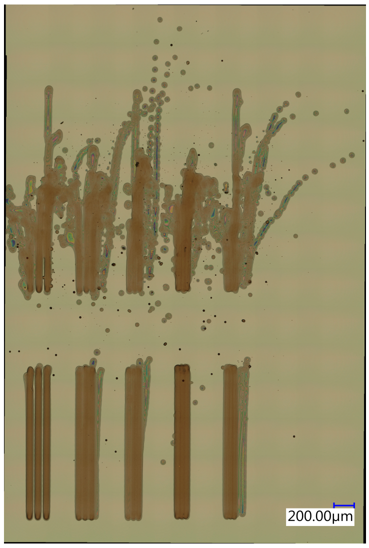

Figure 5.10: Start up zone above the structures is needed to stabilise the drops

Building material

In figure 5.11 the result of the experiment using the building material can be seen. At low cross-scan resolution individual lines can be seen. Printing at a cross-scan resolution of $800[dpi]$ results in a bulging edge. The lines printed at a resolution of $700[dpi]$ have a uniform edge and the material does not seem the cluster at the bottom of the line.

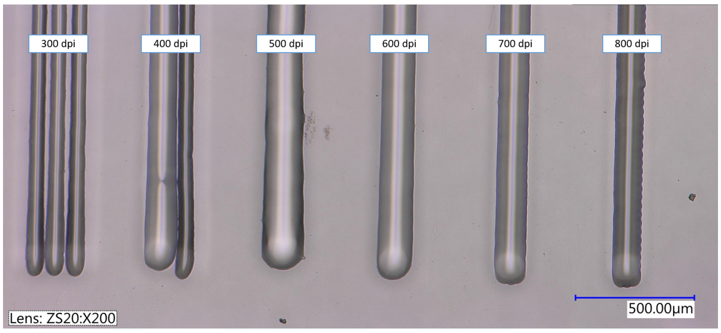

Figure 5.11: Planes created using MR-UVCur26SF in-scan resolution in set to 700 dpi, the cross-scan resolution of the lines is given

Discussion

When creating planes using the sacrificial material a cross-scan resolution of $500[dpi]$ is chosen because creates a uniform plane. For now the stacked coins effect is visible in the planes but when multiple layers are printed on top of each other this might disappear.

For the building material a cross-scan resolution of $700[dpi]$ seems to give the best result. Taking smaller steps in the resolution might give a better result. It could be considered to refine the experiment using the smallest step size in resolution ($25[dpi]$).

5.4. Curing

Theory

The sacrificial material (KL5315) is a positive photoresist when the material that is exposed to UV light can be removed using the developer. Material that is not exposed is supposed to remain on the substrate. The material is used as a sacrificial material, therefore all the material will be exposed to make sure everything is removed during development. To cure the sacrificial material it first needs a prebake at $90[°C]$, then a UV exposure dose of $60[mJ/cm^2]$ according to the datasheet [28], and after that a PEB. All these three steps are followed when printing the sacrificial material.

The building material (mr-UVCur26SF) needs an exposure dose of more than $500[mJ/cm^2]$ to develop [29]. However, the material is designed for NIL processes. Layers made using NIL are usually thinner than layers created using inkjet printing. Most likely a higher UV dose is needed before the material is cured. One of the characteristics of curing mr-UVCur26SF in the presence of oxygen is that it forms an inhibition layer [19]. This is a small liquid layer that will stay on top of the material when cured. This layer can later be removed during the development of the material.

The intensity of the $3[W/cm^2]$ UV light in the printer set-up can be varied. The light exposes the whole length of the substrate bed and has a width of $1[cm]$. To determine the dose the material receives formula 5.4 is used. P is the power of the light, w the width of the exposed area, and v is the speed of the substrate when passing the exposed area. The same exposure dose can be given to a material when changing intensity and speed. However, curing with higher speed and intensity might give mechanical or heat problems because of gasses inside the structure [36].

$$Dose = P * intensity * w/v \tag{5.4}$$

Process

The temperature and curing dose have an influence on the curing of the sacrificial material. The curing temperature of the sacrificial material is equal to the maximum temperature of the substrate bed and reached during the thermal curing cycle. To speed up the process, the temperature of the substrate bed is changed during the printing of the sacrificial material.

The curing dose is set above the recommended dose because a thicker layer is printed and this helps to remove all the material. If the material can not be removed later on in the process the dose can be changed.

mr-UVCur26SF, the building material, is exposed to UV light after one layer is printed. After the exposure, another layer is printed on top of the first one to see if the material is fully cured. If the extra layer breaks the first layer it will create a messy, poorly defined line. The dose is increased until no breakout occurs.

Sacrificial material

In figure 5.12 lines printed on a glass substrate at different temperatures can be seen. At lower temperatures the lines are better defined. The line created at a substrate temperature of $40[°C]$ has uniform edges just as the line printed at $60[°C]$. However, printing more structures on a $60[°C]$ substrate gives poorly defined lines. At higher temperatures the solvent evaporates faster which gives less time for the drop to develop resulting in less uniform lines. Another issue that arose when printing at higher temperatures was an increase in the printhead temperature because the printhead is close to the substrate. This led to unstable drops.

When the substrate temperature is $90[°C]$ it is exposed to the UV light. The sacrificial material is cured at lower intensity (10%) and lower speed $(10[mm/s])$ using equation 5.4 this results in an exposure dose of $300[mJ/cm^2]$. After which the material cooled down to $40°C$. When a higher dose was given by increasing the intensity the heat problems described in [36] were visible, see figures 5.13 and 5.14. Therefore it is preferred to cure at a lower speed and intensity.

Building material

In the beginning, the material was cured using an exposure dose of $2400[mJ/cm^2]$ and an extra layer was added as can be seen in figure 5.15. This was not enough to cure the material so the dose is increased and checked again. Finally, a dose of $[1620 \cdot 10^3 mJ/cm^2]$ was sufficient to cure a line of

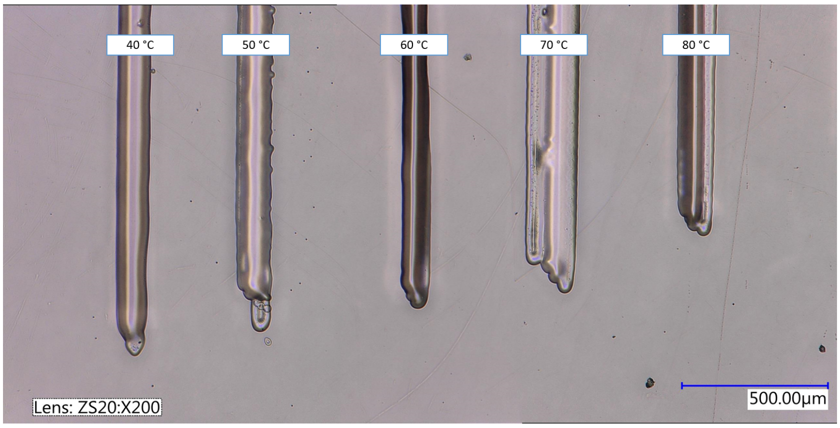

Figure 5.12: Printing multiple lines on top of each other at different substrate temperatures

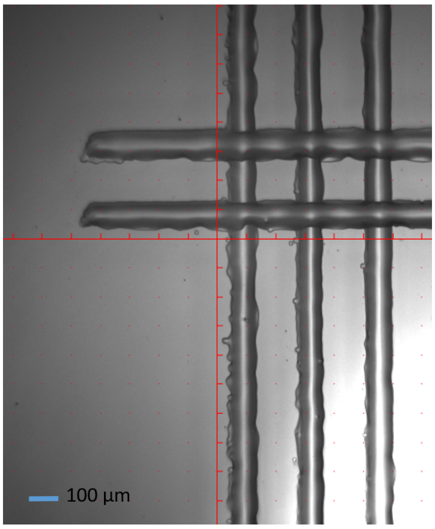

Figure 5.13: KL5315 before curing

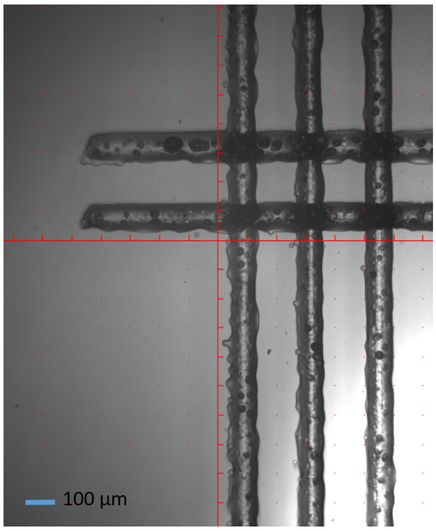

Figure 5.14: KL5315 after curing at to high intensity

building material that is way higher than the $500mJ/cm^2$ which was advised. Because the dose needed to cure the building material is high it is manually cured by placing the substrate below the UV light and turning it on for 10 minutes at 90% intensity. The layer is thicker ($\approx 6[\mu m]$) than a layer produced using NIL which would result in a higher exposure dose.

Discussion

All lines printed in figure 5.12 have imperfections. During multiple prints at higher temperatures imperfections were seen. But, since printing the sacrificial material is challenging at all temperatures it is hard to tell if what the optimal temperature is. Printing at lower temperatures showed the best results overall but can also have imperfections

Curing the building material by hand leads to inaccuracies. Since the exposed area that is cured is $1[cm]$ wide it is challenging to get an equal amount of energy to the whole structure. Printing smaller drops might help in getting a lower minimal exposure energy leading to more accurate curing. It might be better to do multiple curing cycles per layer so that the amount of energy can better be regulated.

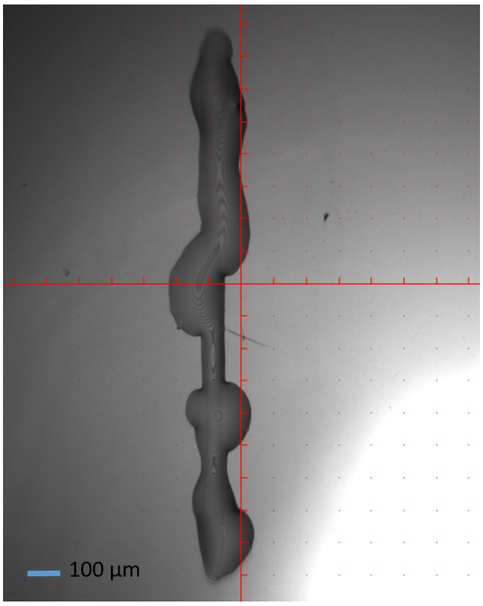

Figure 5.15: Spreading of building material when an extra layer is added

5.5. Height control

Process

To create higher structures multiple lines or planes can be placed on top of each other. After each layer, the material is cured and a new layer is placed on top of the old one. This process is schematically shown on the left-hand side of figure 5.16. An advantage of this method is that height can be created without increasing the width of the structure. One of the downsides is that the alignment between different layers must be precise so that the drops land on top of each other to increase the height. Another downside is that the material has to be cured after each layer which takes time. A different method that is proposed to create height in the structure is shown on the right-hand side of figure 5.16 and is, for now, called the high-resolution method. Multiple lines of material are printed next to each other using a cross-scan resolution of $4800[dpi]$ without intermediate curing. The centre to centre distance between drops is reduced to $5.3[\mu m]$. The in-scan resolution is increased to $600[dpi]$ to increase the height. Using this method the same amount of material can be printed on the substrate in one cycle as would normally be printed in multiple layers, resulting in an increase in height. However, the width of the structure increases because the drops are placed on top of each other with a slight offset which is the same as the centre to centre distance and because of the spreading of the material. This method only works if the drops do not spread too much over the surface.

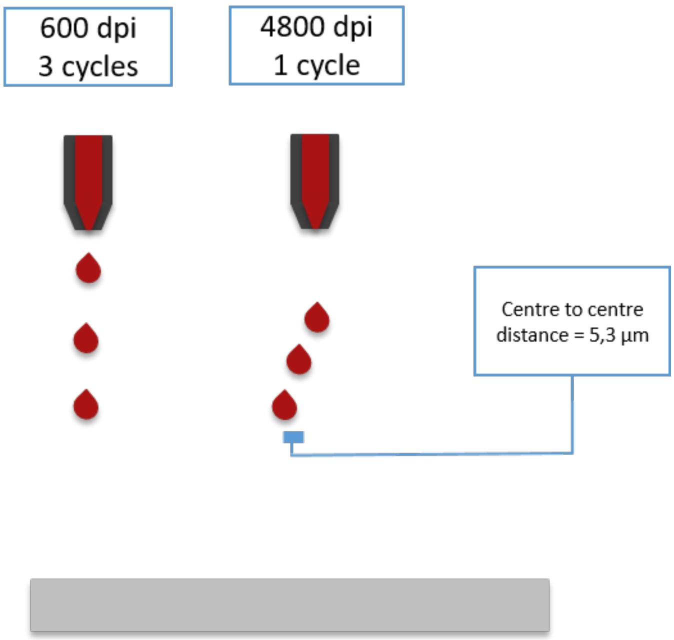

Figure 5.16: Schematic explanation of creating height by printing multiple lines on top of each other (left) and by increasing the amount op drops per inch (right)

Sacrificial material

In figure 5.17 lines of sacrificial material are printed on top of each other to create multi-layer structures. Looking at the lines the colour gets darker the more lines were printed on top of each other. The width of the lines stays the same when a layer is printed on top of another one. The height of the lines was measurable when three or more layers were printed on top of each other. When one or two lines are printed it was not possible to accurately measure the height. It was difficult to print the lines on top of each other because the sacrificial material is often unstable. This results in curved lines that were not printed on top of the previous line, contaminating the substrate. These lines were printed on a silicon substrate due to technical issues.

Figure 5.18 shown lines created using the high resolution methods. The lines are bulging and thus is the width hard to predict. The width of the line is also changing at different places on the substrate. These lines have a measurable height when the lines are three or more pixels wide. Although the width of the lines is bigger and the edge is not uniform the method is faster compared to printing layers on top of each other because it is printed in one cycle.

The height and width of lines printed using different printer settings and different methods can be found in table 5.7. It can be seen that the values differ a lot from each other. And it is counter-intuitive that a line of 5 pixels wide is smaller than a line of 4 pixels wide. This shows that the process is hard to reproduce.

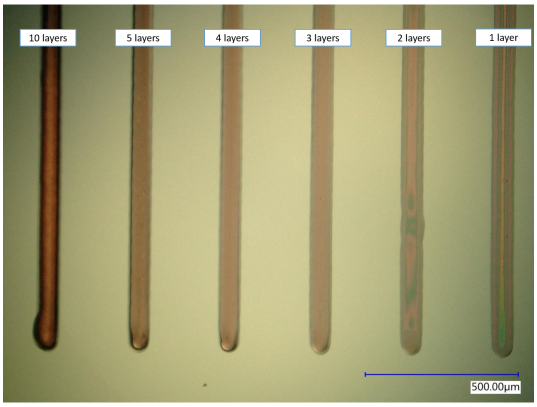

Figure 5.17: lines of KL5315 printed on top of each other using 500 dpi from left to right: 10, 5, 4, 3, 2, and 1 layer

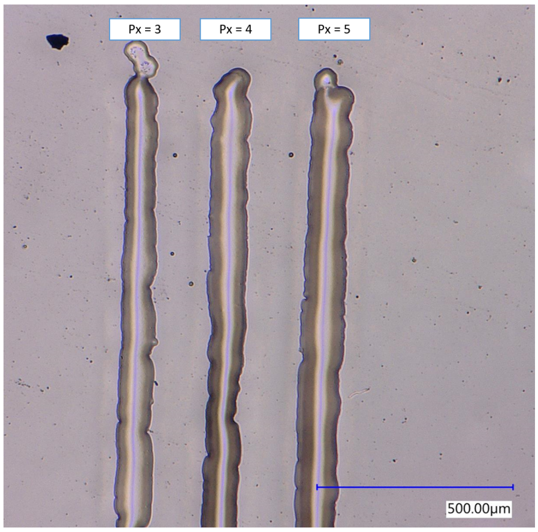

Figure 5.18: 3, 4, and 5 lines of KL5315 printed with a resolution of $4800[dpi]$ in cross section direction and $600[dpi]$ in in-scan direction

$Pixels_x$	N_{layers}	In-scan resolution $[dpi]$	Cross-scan resolution $[dpi]$	$Height[\mu m]$	$Width[\mu m]$
1	3	500	-	2.3	55.6
3	1	500	4800	3.3	68.8
1	4	500	-	3.8	55.1
4	1	500	4800	3.5	93.6
1	5	500	-	5.1	54.1
5	1	500	4800	4.6	90.2
3	1	600	4800	4.2	58.2
4	1	600	4800	4.9	81.1
5	1	600	4800	3.8	122.5

Table 5.6: Height and width measurements of different beads of KL5315 (SM)

The height profile of two lines, one printed normal and one at using a high cross-scan resolution is shown in figure 5.19. The height profile of the line created using the high-resolution methods is smoother than the profile of lines printed on top of each other.

To determine the maximum height that can be created using the high-resolution method the number of lines in the cross-scan direction is increased. Figure 5.20 shows the top view of the different lines. The cross-section of all the lines can be seen in figure 5.21. A line consisting of 30 pixels in the cross-scan direction is printed twice. It can be seen that the highest structure can be made using 15 pixels in the cross-scan direction.

Building material

The building material has a measurable height when one single layer is printed on the substrate. It is not suited for printing using a high cross-scan resolution because it spreads too much. The lines printed using the building material are also more stable and predictable than the lines printed using the sacrificial material so there is no need to print using a high cross-scan resolution. In figure 5.22 multilayer lines are printed on top of each other and in table 5.7 the height and width of the lines are

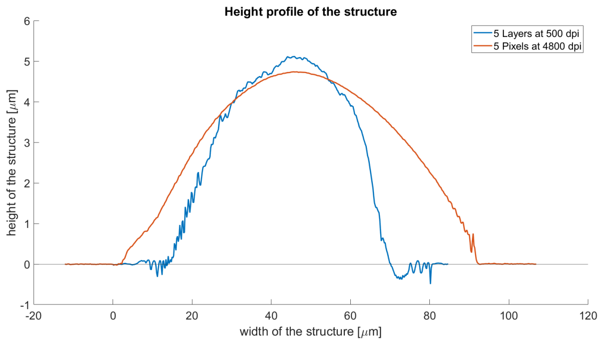

Figure 5.19: Height profile of 5 lines printed on top of each other and 5 lines printed using the high resolution method

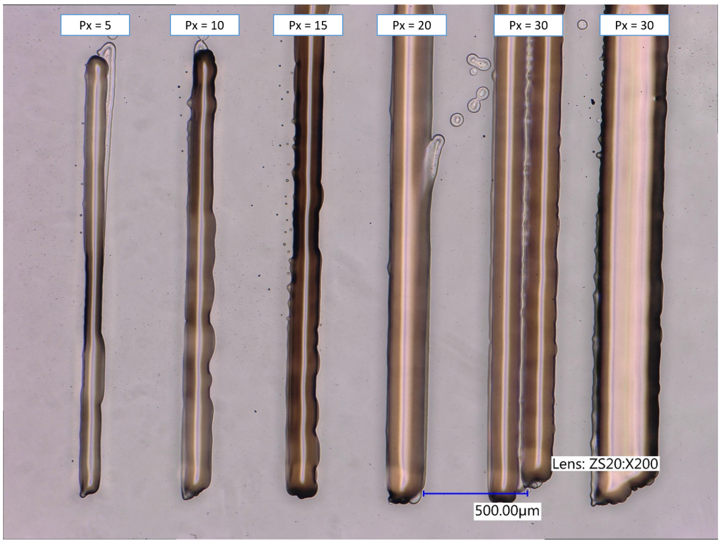

Figure 5.20: Lines of sacrificial material with different width printed using the high resolution method

given. The width of the structure increase when a second layer is added on top of the first layer, but when a third layer is added the width is the same. The height increases with every layer added.

$Pixels_x$	N_{layers}	In-scan resolution $[dpi]$	Cross-scan resolution $[dpi]$	Height $[\mu m]$	$Width[\mu m]$
3	1	700	700	6.1	130.3
3	2	700	700	9.1	165.4
3	3	700	700	12.3	162.3

Table 5.7: Height and width measurements of different lines of building material

Discussion

Creating reproducible lines that have a measurable height using the sacrificial material is a challenge. Printing the lines on top of each other makes it easier to predict the width but when an error is made in the alignment a new structure must be made. During the experiments it was challenging to print 3 or more lines right on top of each other. Increasing the startup zone led to better stability but this sometimes resulted in a startup zone that was twice the length of the actual structure. The surface is also quite rough which is likely because the layer is already cured before the next layer is printed.

Printing sacrificial material using the high-resolution method gives most of the time a structure with a measurable height and a smooth height profile. However, the width and height are harder to predict using this method. The step size of the printer is smaller than the centre to centre distance when printing at a resolution of $4800[dpi]$. The step size is not an integer and might have caused an error.

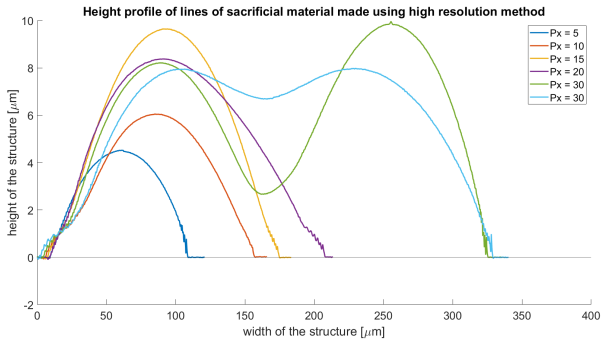

Figure 5.21: Height profile of the lines of sacrificial material printed using the high resolution method

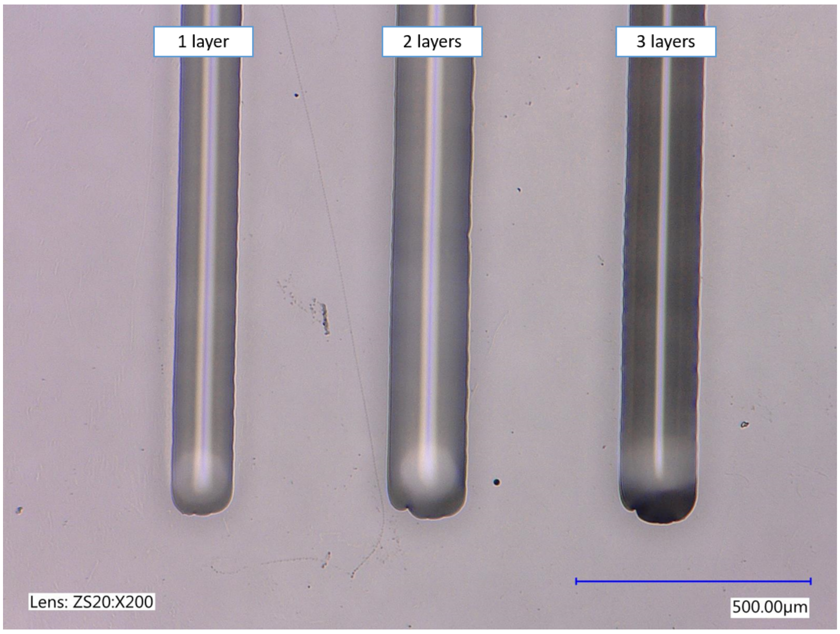

Figure 5.22: Lines of MR-UVCur26SF printed on top of each other at a resolution of $700[dpi]$ in both directions. From left to right 1,2, and 3 layers

However when printing at $5080[dpi]$, resulting in a centre to centre distance of $5[\mu m]$, the result was still hard to reproduce.

Printing more lines next to each other creates a higher structure and leads to a wider structure. However, once the material spreads too much the peak of the lines drops which can be seen when 30 lines were printed.

It is chosen to print the sacrificial material using the high-resolution method because this method was more reliable. The fact that the first method (printing layers on top of each other) was printed on a silicon substrate has no influence on the place where the material is printed. The drops of sacrificial material are not stable enough to be printed on the same position multiple times in a row.

The Building material is printed by placing layers on top of each other using intermediate curing. Although the width increased when the second layer was printed on the first layer this did not happen when a third layer was added. This increase in width might be due to a small misalignment between the first and the second print. The building material needs fewer layers than the sacrificial material to create the same height. The shrinkage of the sacrificial material is bigger and is possible caused by the solvent which evaporates.

5.6. Removal of Material

Background

Sacrificial material that is exposed to UV can be removed by the developer. However, it is not possible to use the recommended developer for the sacrificial material. Therefore sodium hydroxide is used which should also develop the material. The building material should not be removed during the removal of the sacrificial material.

Process

Multiple concentrations of sodium hydroxide are used to determine when the sacrificial material is removed. To find the right concentration this sacrificial and building material was printed on three substrates. First, three lines of sacrificial material were printed and cured followed by one line of sacrificial material which is also cured. All printing and curing steps are done using the settings previously described.

These substrates were all developed for 1 minute at different concentrations. A control is also performed to see if the sacrificial material is not removed by simply placing it in water. Before development the height profile of the building material is measured to be compared with the height profile after the development.

Results

The results of the experiment can be seen in figure 5.23. The sacrificial material is not removed by 1 minute in $0.1 w\% NaOH$. This means either more time or a higher concentration is needed for the development. When the substrate was placed in water for one minute, one of the three lines of the sacrificial material was moved. This means that the adhesion between the sacrificial material and the substrate is not ideal. However, the goal is to remove the sacrificial material so the adhesion is not of most importance.

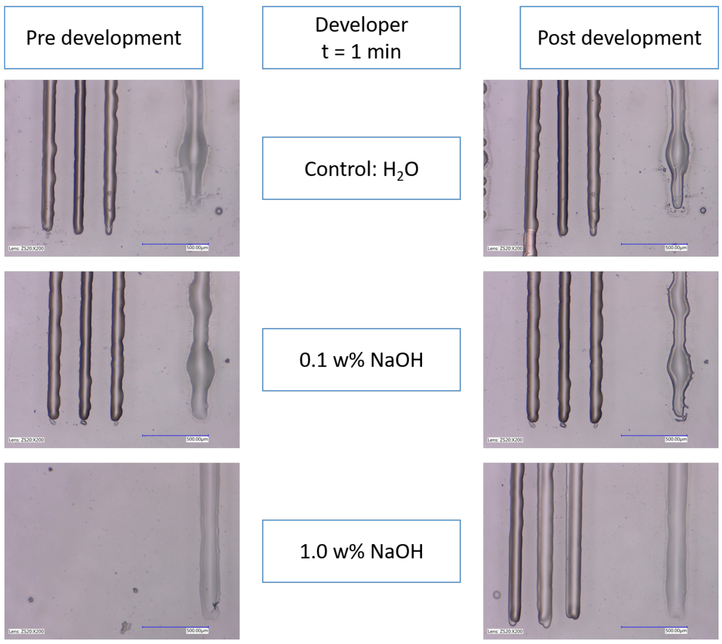

Figure 5.23: Development of lines using different developers, on the left side of each substrate the sacrificial material is printed on the right side the building material

Interesting is the difference in height profile at the building material before and after the development, see figures 5.24 till 5.26. This is most likely due to the removal of the small inhibition layer. The height is almost the same but the edge has shifted. Also some blobs appear on the side of the line.

Discussion

Maybe the maximum exposure dose of the sacrificial material is not reached which would speed up the process. The curing dose used for the sacrificial material is larger than the recommended energy. However, the thickness is also bigger than used in the datasheet due to printing using the high-resolution method.

The building material is affected by the developer as can be seen by the height profile. However, the structure is also affected by water. This most likely has something to do with the inhibition layer.

The structure developed in $1w\%NaOH$ is lower than the other structures which could have an influence. No extra blob is seen in this height profile. However, the surface shows imperfections not seen in the pre-development profile.

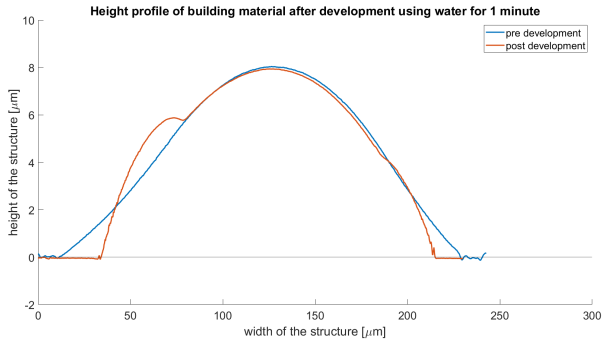

Figure 5.24: Height profile of the building material before and after development using water

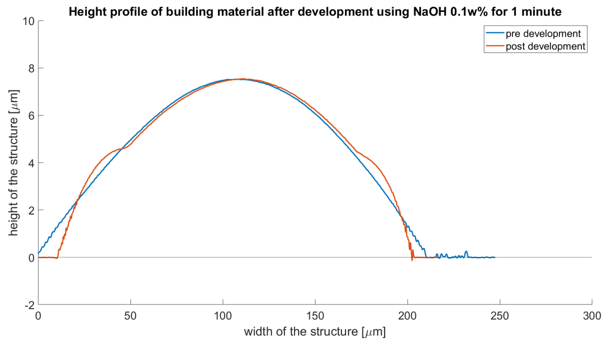

Figure 5.25: Height profile of the building material before and after development using $0.1w\%NaOH$

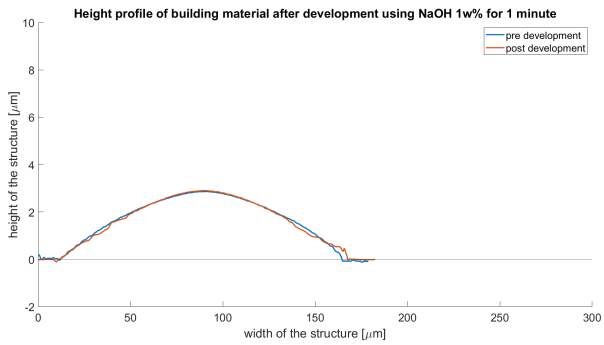

Figure 5.26: Height profile of the building material before and after development using $1w\%NaOH$

5.7. Summary

All the information of the previous sections is combined in table 5.8 and used in the next chapter to create overhanging structures.

Property	Sacrificial material	Building material
Name	KL5315	mr-UVCur26SF
Drop volume $[pL]$	14.2	12.3
In-scan resolution $[dpi]$	600	700
Cross-scan resolution $[dpi]$	4800	700
Method for height creation	high resolution	multiple layers
Measurable height	3 pixel wide	1 layer
Thermal curing $[^{\circ}C]$	90	-
UV curing $[mJ/cm^2]$	300	$1620 \cdot 10^3$
Removal	$1w\%NaOH$ for 1 minute	-
Substrate temperature $[^{\circ}C]$	40	40
Print speed $[mm/s]$	25	25
Print direction	uni-directional	uni-directional

Table 5.8: Summary of material properties used during printing of the materials

6

Overhanging structure

This chapter explains the procedure followed to create overhanging structures. First, the building process is described, followed by the development of the structure. In the end, the measurements and some images of the created structures are shown.

6.1. Building process

The knowledge gained in the previous chapter is used during the production of an overhanging structure. In the previous chapter the materials were used separately, when creating the overhanging structure they are used together and influence each other.

First proposed process

The proposed overhanging structure to be printed is a bridge. The bridge is supported on both sides and is connected by an overhanging part. A simple method to produce an overhanging structure is first tried, see figure 6.1 and consists of the following steps:

1. A five pixel wide line of sacrificial material is printed on a glass substrate ($T = 40[°C]$) with a cross-scan resolution of $4800dpi$ and an in-scan resolution of $600[dpi]$. This will create a layer that is a couple of micrometer high and will support the building material during the production. The substrate is heated till $90[°C]$, exposed by UV light with a dose of $300[mJ/cm^2]$, and cooled down to $40[°C]$ afterward.

2. A three pixel wide line of building material is printed on both sides of the sacrificial material with an in-scan and cross-scan resolution of $700[dpi]$ cured for 10 minutes at 90% intensity. These two lines will function as pillars for the overhanging part. The distance between both lines of building material is varied creating enough space for the sacrificial material.

3. A new layer of building material is printed using the same resolution on top of the existing three lines (two building material and one sacrificial) to connect both building material lines. And cured for 10 minutes at 90% intensity resulting in a dose of $1620 \cdot 10^3[mJ/cm^2]$

4. The whole structure is developed in a $1w\%NaOH$ solution removing the sacrificial material and creating a microbridge.

Performing this process caused some issues which can be seen in figures 6.2 and 6.3. In figure 6.2 it can be seen that the building material gets attracted to the sacrificial material. This happens when the building material is printed too close to the sacrificial material. This is undesired because the shape of the building material is lost.

In figure 6.3 the opposite is shown, the building material is printed too far away from the sacrificial material leaving a gap between both materials. If a new layer of building material is printed on top of these lines the material spreads again and the shape is lost. Because of this issue, it was not possible to create well defined overhanging microbridges using this method.

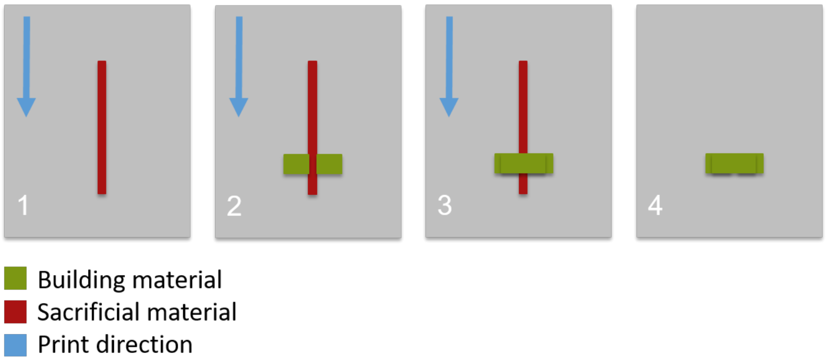

Figure 6.1: Schematics of the first proposed process

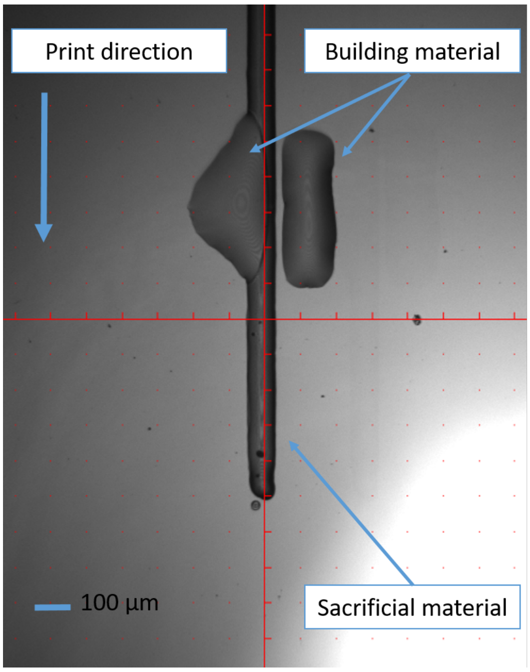

Figure 6.2: The building material got attracted to the sacrificial material, because it was printed to close to the sacrificial material

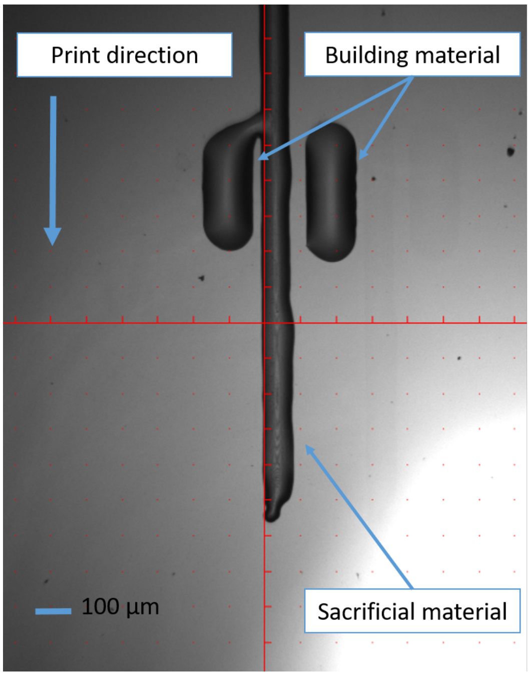

Figure 6.3: The building material is printed too far away from the sacrificial material

Second proposed process

The main issue during the first proposed process is that the building material can not be printed close enough to the sacrificial material without losing its shape. To keep the shape the spreading must be stopped which can be done in multiple ways. A paper by Derby et al. [7] used a channel in the substrate to stop the spreading of material. This gave the inspiration to create a physical barrier (dikes) using the sacrificial material to prevent the building material from spreading. Because the dikes are built using the sacrificial material they are removed during the development step and not present in the final microstructure.

A second process is proposed to manufacture a microbridge and is shown in figure 6.5. Steps 1 and 2 are only shown schematically, all other steps also show an example of a step during the actual production of a microbridge. All steps are explained below.

1. Three lines (in this case all five pixels wide) of sacrificial material are printed in the y direction. The in-scan resolution is $600[dpi]$ and the cross-scan resolution is $4800[dpi]$. The substrate temperature is $40[°C]$.
2. Two lines of sacrificial material are printed in the x direction on top of the already existing lines.

The in-scan resolution, cross-scan resolution, and substrate temperature are the same as in step 1. These lines are also five pixels wide.

3. The substrate is heated till $90[°C]$ exposed to a UV dose of $300[mJ/cm^2]$ and cooled down.

4. Now the space surrounded by dikes on the left side of the centre line is filled with building material. The material is printed using an in-scan and cross-scan resolution of $700[dpi]$ and in this case the image is a $4x4$ pixel block.

5. The right space is filled separately because the width of the sacrificial material differs because of inaccuracies in the high-resolution method. The alignment of both the left and the right block is more precise when printed individually. When the alignment is off it can happen that the building material does not will the whole space as can be seen in figure 6.4

6. The material is cured for 10 minutes at 90% intensity using the UV light giving the material a dose of $1620 \cdot 10^3[mJ/cm^2]$.

7. A top layer is printed on top of the already existing lines. This layer consists of $4x8$ pixels and is printed in the middle of the line created by the sacrificial material. When the layer is larger, the material might flow to the other sides of the outside dikes which is unwanted.

8. The material is cured again for 10 minutes at 90% intensity using the UV light giving the material a dose of $1620 \cdot 10^3[mJ/cm^2]$.

9. The substrate is developed, removing the sacrificial material and creating a microbridge. This development step is explained in the next section of this chapter.

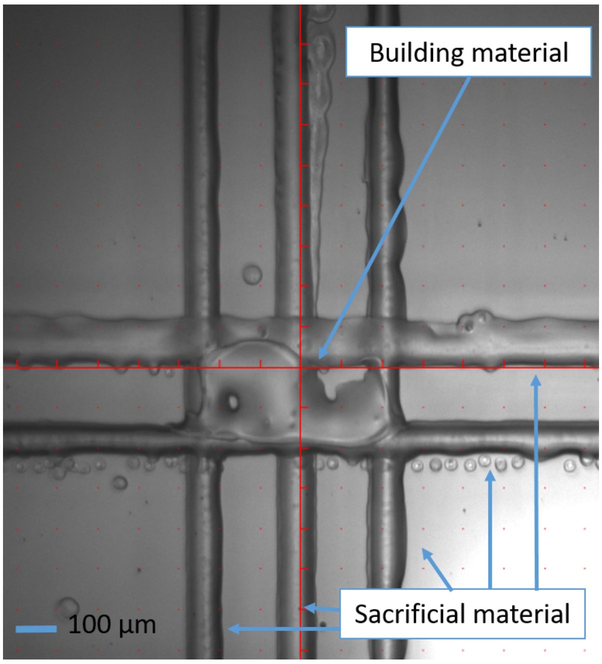

Figure 6.4: Error in filling the space in between the dikes created by sacrificial material with building material

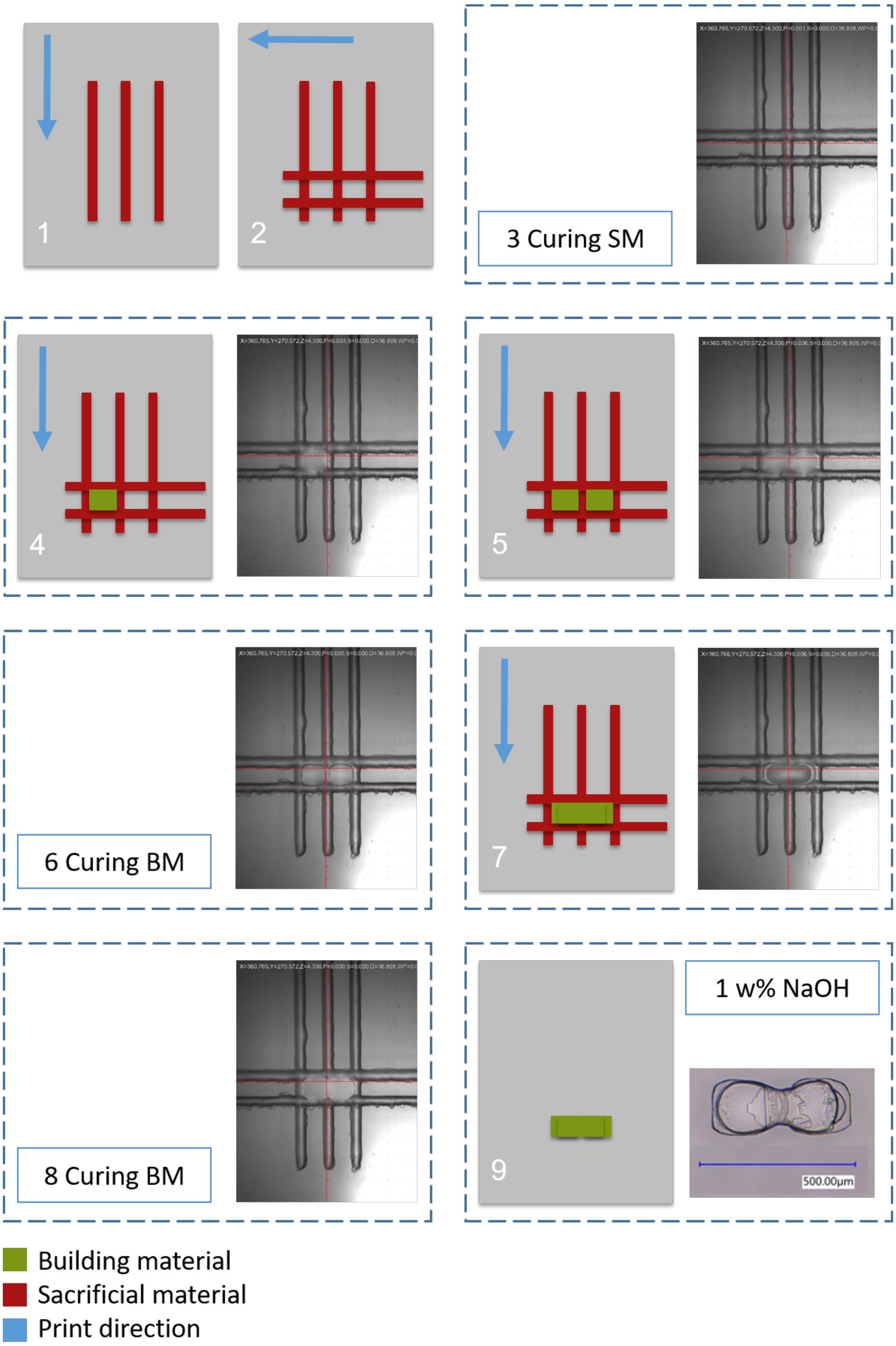

Figure 6.5: Process of creating an overhanging structure using MR-UVCur26SF (building material) and KL5315 (sacrificial material)

6.2. Development

Once all the material is printed and cured the sacrificial material can be removed. This is done in a solution of $1w\%$ NaOH for one minute at a time and is shown in figure 6.6. The photos are taken from the top of the substrate. It can be seen that all the sacrificial material which was not covered by building material is removed after one minute which corresponds to the previous experiment. However, the material covered by the building material is not fully removed after one minute. When the structure is developed for an additional minute it can be seen that the light-coloured areas on top and bottom of the structure get bigger. After four minutes both light-coloured areas connect and the development was stopped.

Not all the sacrificial material gets properly removed by the developer. The remaining material clusters on the substrate and/or the structure as can be seen in the top left of the substrate after four minutes shown in figure 6.6. The remaining sacrificial material can also be seen at the edges of the structure from the bottom, but one can also see gaps in this edge of sacrificial material. A larger image of the bottom photo can be found in appendix A.

During the development, a lot of structures got loose from the substrate and got lost. A better adhesion between the building material and the substrate might prevent this and the use of adhesion promoters could be considered for further research. When the adhesion of structures to the substrate is better, development can be done for a longer period of time with less risk of losing structures. This could result in better-defined shapes because more sacrificial material is removed.

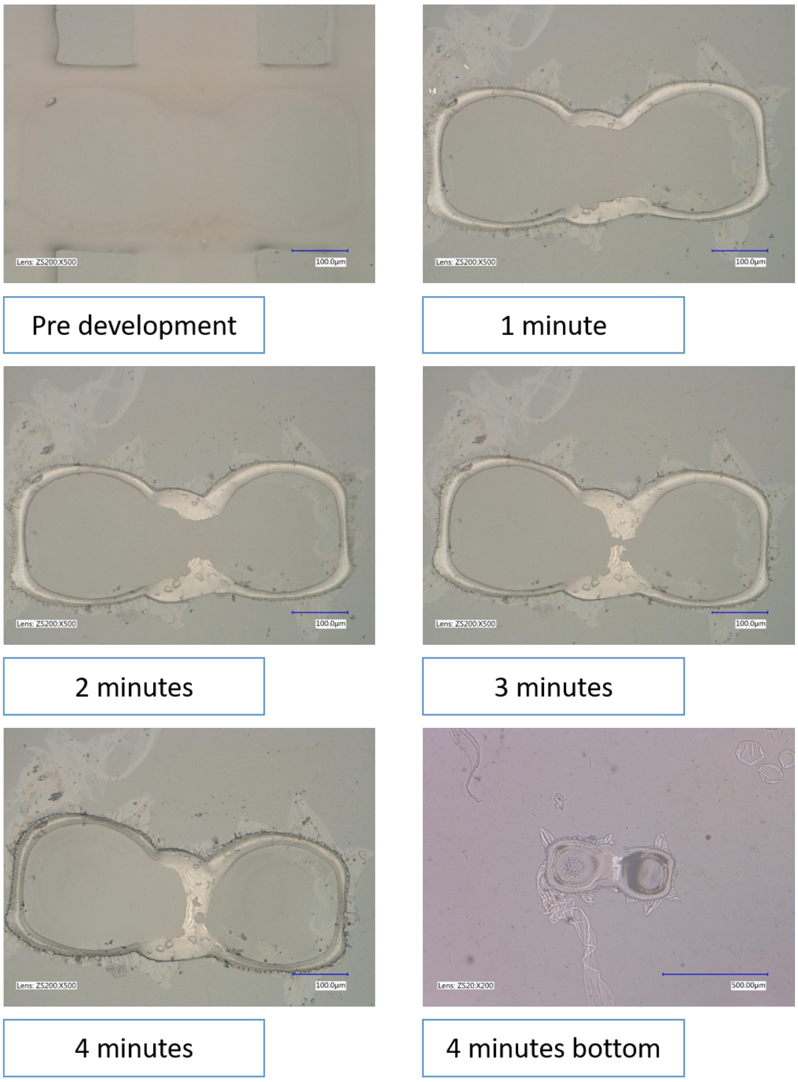

Figure 6.6: Development of a structure over time, the sacrificial material is more and more removed when development takes longer

6.3. Measurements

In figure 6.7 the height profile of the structure developed in the previous section is displayed. Besides the structure before and after the development, the height profiles of the dikes just above the structure are shown in the same figure. The centre dike consists in this case of 15 pixels printed in groups of five with five white pixels between them. It can be seen that the height of the structure before and after the development is the same. This would suggest that the structure has not collapsed. This means that the light coloured areas are caused by the removal of sacrificial material resulting in an overhanging structure.

Looking at the edges of the height profile a clear difference at the edge of the structure before and after the development can be seen. The building material is printed on one side of the dikes and the other side is uncovered. After development the substrate is measured at the position of the uncovered part of the dike. The building material remains at the same position and is measured again.

The space between the dikes is around $140[\mu m]$ wide when printing a $4x4$ square the centre to centre distance between the first and last drop is $108[\mu m]$. Taking the spreading of the drop into account this will fill the dike with one layer of material. The width of the centre dike is approximately $230\mu m$ when looking at the profile.

To check the overhanging part of the microbridge, SEM images are taken from the sides of the structure at an angle of $45[°]$ which can be seen in figure 6.8. At the side with a clear gap in the remaining sacrificial material (bottom) an overhanging part is seen in the SEM photo. The length of the overhanging part is around $50\mu m$, this corresponds to the light-coloured area in the microscope photo. On the top, only small gaps can be seen on the left side of the structure. It looks like a layer of not removed sacrificial material blocks most of the area where centre dike was printed. In appendix A another example of a structure after development is shown.

Figure 6.7: Height profile of the structure before and after development and the dikes created using sacrificial material

6.4. Discussion

Sacrificial dikes can prevent building material from spreading and help to create overhanging microstructures. However, the freedom in design is limited because the dikes determine the shape one can make. For now, all dikes are printed parallel and perpendicular printing one line in one swath. Printing them at different angles width respect to each other might increases the freedom of the shapes. Now a single line is printed in one swath when changing the angle a single line is printed using multiple swaths which might lead to inaccuracies

One thing to consider is the importance of the printing resolution of the building material if spreading is prevented by the dikes. The material is printed in the space between the dike and spread in this area. So if the resolution is lower the material spreads until the dikes stop it.

The removal of the sacrificial material takes longer if it is covered, maybe an increase in the curing dose might speed up the development. Also, the difference between the width of the printed dike and the width of the overhanging part of the microbridge suggests that not all the material is removed. This lack of removal also results in contamination of other places of the substrate (e.g. covering the gap of the overhanging parts).

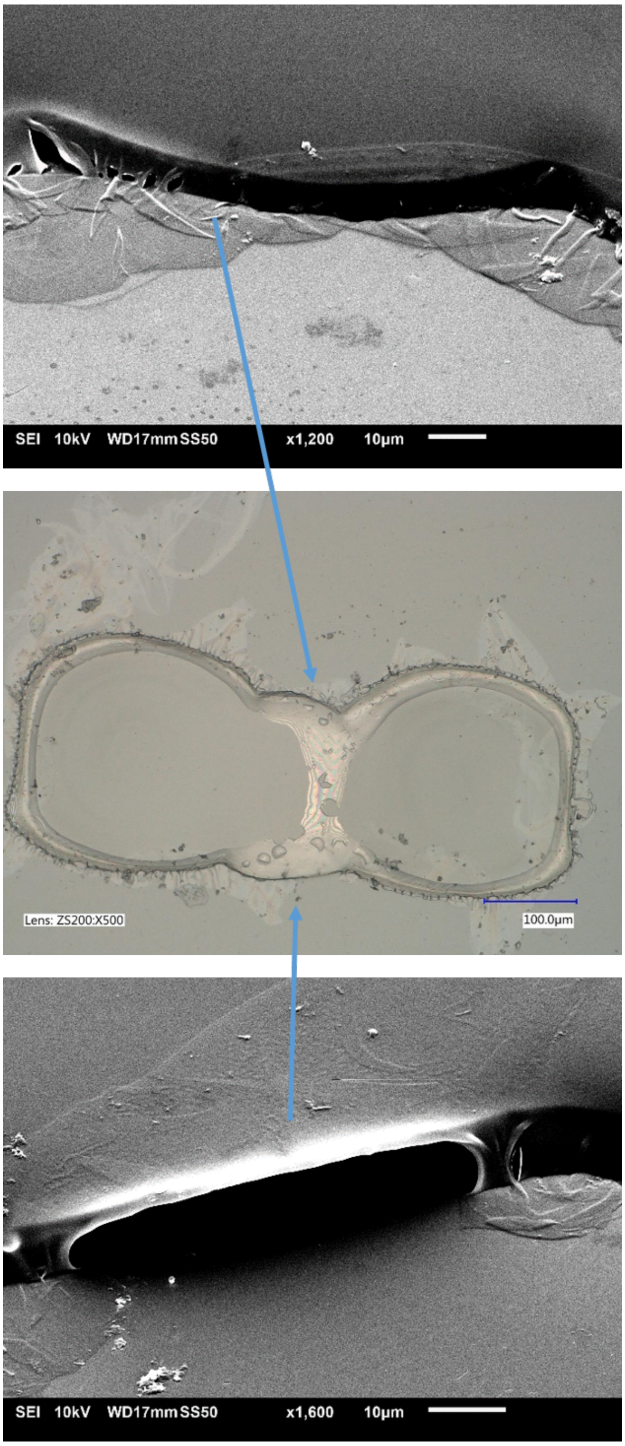

Figure 6.8: Photo of the top of the structure and the corresponding SEM photos

7

Conclusion and recommendations

Sacrificial support material is needed to print overhanging microstructures using inkjet printing. The criteria used to select the materials were based on the printer properties and the curing method. Most candidate materials were not suitable because they cannot be printed at the maximum printhead temperature or needed curing at temperatures outside the range of the printer.

The building material that was selected is mr-UVCur26SF and the sacrificial material is KL5315. Stable drops were created using both materials and the sacrificial material was removed using a NaOH (1w%) solution. While these materials were suitable for this project the following properties should be kept in mind. The building material was affected during the removal of the sacrificial material. Moreover, the curing of one layer of building material takes $10[min]$ which makes it not suitable for high throughput.

To increase the height of the structures two methods are used. The first method is placing layers of material on top of each other with intermediate curing building the structure from the bottom up. While this method is used to print the building material it was not possible to print the sacrificial material using this method due to poor alignment between the layers. Therefore a second method is used to print the sacrificial material. By printing multiple lines close to each other using a high cross-scan resolution ($4800[dpi]$) it was possible to create higher structures. However, the structure had bulging lines because the sacrificial material spreads too much. The height of the sacrificial material was difficult to predict due to the spreading of the material and the unstable printing.

Two methods to create overhanging structures were tested during this research. The first method consists of first printing the sacrificial material followed by printing the building material. This method could not be used due to the loss of the building material's shape because it got attracted to the sacrificial material. To solve this problem a second method uses the sacrificial material not only as support but also as dikes to prevent the material from spreading over the substrate. A microbridge with an overhanging part of approximately $50[\mu m]$ was created using the sacrificial dike method. For now, only straight dikes were used using this method which limits the design freedom of the overhanging parts that can be created.

The following limitations were found during the research. The instability of the sacrificial material while printing leads to height control by changing the cross-scan resolution. The building material is affected during the removal of the sacrificial material which leads to a loss of shape or even losing the whole structure.

During this research the removal of sacrificial material is stopped because of the risk of losing the whole structure. Therefore not all the sacrificial material covered by the building material was removed. If the adhesion between the building material and the substrate is increased more sacrificial material could be removed resulting in a larger overhanging part.

Recommendations

Some interesting topics for further research:

- Explore different material to build overhanging structures

  For now, two materials have been chosen each having limitations in printing and curing. Maybe the printing techniques that did not work using these combinations might work with different materials. Possibly resulting in better precision and more freedom in the structure design.

- What kind of shapes can be made using sacrificial dikes?

  Only straight dikes have been printed to prevent the spreading of the building material. Changing the orientation and shape of the dikes creates possibilities to form different shapes.

- Can primers be used in the process, keeping the building material attached to the substrate but making sure the sacrificial material can be removed?

  Using a primer helps the adhesion between the building material and the substrate but how does it affect the adhesion between the sacrificial material and the substrate? How is the primer affected by the developer of the sacrificial material and can the sacrificial material be removed when the primer is used?

- Try to decrease the drop volume to create a smaller structure

  If the drops are smaller structure can be made and the current structures can be made more precise. Smaller drops lead to thinner layers and this might lead to shorter curing times.

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A

Additional experimental results

Development

Below the bottom view of the structure discussed in section 6.2

Figure A.1: bottom view through the glass substrate of structure discussed in section 6.2

Extra SEM images

Below photos of a structure made using the SEM and microscope

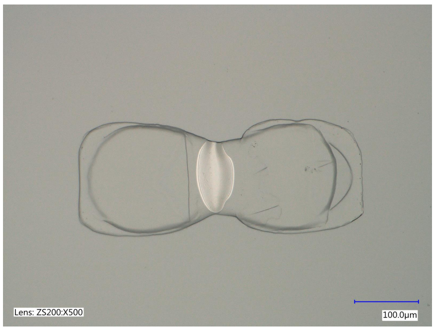

Figure A.2: Same structure as the SEM images but image taken using the Keyence

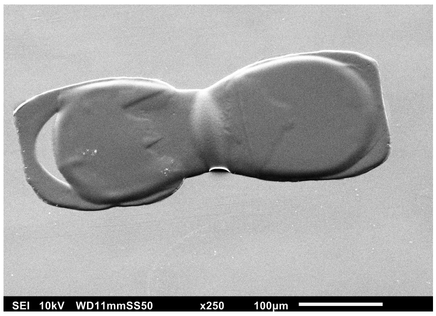

Figure A.3: SEM image of the top edge of the structure seen in figure A.2 at a $30°$ tilt

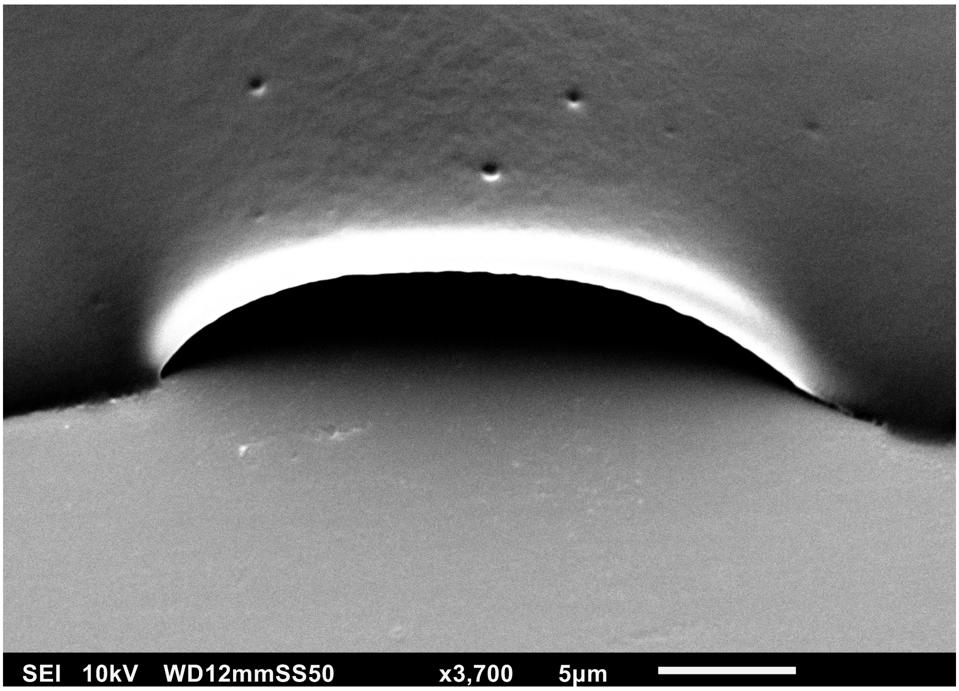

Figure A.4: Close up of the overhanging part of the structure