Polishing thin sections for analysis of 3D printed and injection moulded polymers

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Thin section analysis is a new analysis method for 3D printing, though it has been done with polymers before to analyse material structure and processing defects in parts. The main aim of the thesis is to develop a polishing method by which thin sections of 3D printed and injection moulded parts can be obtained for studying by optical light microscope. Polishing of thin sections has been used in biology, metallurgy and geology, therefore knowledge from those field are utilized in this thesis for preparation of samples. In this work, thin sections of injection moulded and 3D printed parts are produced by the polishing method. The different preparation steps needed to produce thin sections are initially followed according to literature research, then reviewed after practical testing and experimenting. Limitations and benefits of the polishing method of producing thin sections are discussed and the preparation methods are developed to be more efficient and provide high quality samples. Thin section samples of polypropylene and polylactic acid processed by injection moulding and 3D printing, are analysed and interpret to justify the importance of thin sections for analysis of the internal structure on parts. The secondary aim of this thesis work is to develop the microscope at Arcada to support cross-polarized transmitted light illumination for analysing of polymer thin sections, and benefit future studies and teaching.

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Tunntvärsnittsanalys är en ny typ av analysmetod för 3D-utskrivna delar, samma analysmetod har använts tidigare för plastmaterial för att analysera materialstruktur och defekter i plastdelar. Huvudsyftet med examensbetet är att utveckla en poleringsmetod genom vilken tunna tvärsnitt av 3D-utskrivna och formsprutade delar kan erhållas för att analyseras med optiskt ljusmikroskop. Polering av tunna tvärsnitt har använts inom biologi, metallurgi och geologi, kunskapen på dessa områden utnyttjas i detta slutarbete för att framställa provbitar av polymera material. I detta arbete produceras tunna tvärsnitt av formsprutade och 3D-utskrivna delar med en poleringsmetod. De olika beredningsstegen som behövs för att producera tvärsnitten görs först enligt litteraturanvisningarna, sedan revideras metoderna i enlighet med praktiska prov och experiment. Begränsningar och fördelar med poleringsmetoden för att producera tunna tvärsnitt, diskuteras i arbetet och metoderna för att förbereda arbetet utvecklas för att bli effektivare och tillhandahålla högkvalitativa provbitar för analysering. Tunna tvärsnitt av plastdelar producerade av polypropen och polylaktid tillverkade genom formsprutning och 3D-utskrift analyseras med mikroskop och resultatet tolkas för att motivera fördelarna av tunna tvärsnitt för analys av den inre strukturen på plastdelar. Det andra syftet med detta examensarbete är att utveckla mikroskopet i Arcada för att stödja tvärpolariserad belysning som är väsentligt för analys av polymera tvärsnitt, och gynna framtida studier och undervisning i Arcada.

Nyckelord:	Tunna tvärsnitt, Polering, 3D-utskrift, Formsprutning, Polymer, Mikroskop
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FOREWORD

Firstly, I would like to thank my supervisor Stewart Makkonen-Craig for his guidance and support for this thesis work.

I would also like to thank my fellow student Petrik Blåfield for his support, and Erland Nyroth for his assistance and guidance in the machining work done for this thesis.

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ABBREVIATIONS

CNC – Computer Numerical Control

DIC – Differential Interference Contrast

DSLR Camera – Digital Single Lens Reflex Camera

FEPA – Federation of European Producers of Abrasives

IM – Injection Moulding

ISO – International Organisation for Standardization

kHz – Kilo Hertz

kW – Kilo watts

PC – Polycarbonate

PLA – Polylactic Acid

POM – Polyoxymethylene

PP – Polypropylene

PTFE – Polytetrafluoroethylene (Teflon)

R&D – Research and Development

RPM – Revolutions per Minute

UHMWPE – Ultra High Molecular Weight Polyethylene

1 INTRODUCTION

1.1 Thin sectioning

Thin section analysis is a common method in biology, metallurgy and geology, therefore knowledge from these fields can be implemented in the preparation of polymer thin section. The production of polished samples and microscopic analysis with cross-polarized light is very similar for both polymers and geology. In geology this method enable scientists to study crystal shapes and symmetry in rocks of different types. Biological thin sections enable studying of tissue and cells of organisms, though the samples are generally prepared by microtomy.

Thin sections have been used in analysis of plastics and polymers for studying failures, melt flow, weld-lines, molecular orientation and R&D of injection-moulded parts, but some online investigation shows that there has not been any publications regarding 3D printed applications. 3D printing is currently a relatively new manufacturing process, which is possibly one reason the two have not met.

1.2 Background

The idea behind this thesis work is to utilize methods of thin sectioning in geology and apply that to injection moulded and 3D printed polymers. Injection moulding and 3D printing of polymers, are processing methods being utilized on advanced levels in Arcada. Thin section analysis is not a necessity at this point, but a highly valuable method of polymer analysis for the school, since it provides structural information about parts and materials at microscopic level that is otherwise invisible to the naked eye, and may inspire new studies and experiments. Most of the tools and equipment for producing thin sections are available at Arcada, and with some studying and tailoring, the polymer analysing infrastructure of the school can be extended to thin section analysis.

The main aim of this thesis is to successfully produce thin section samples of plastic parts for optical microscopy analysis, by implementing methods from geology and other field where the method is currently used.

Other aims include:

- Studying details of methods to find factors affecting the end result, including time efficiency
- Microscopy study of polymer thin sections samples
- Planning and conducting a project

Since the thesis includes microscopy, the student aims to become more experienced in that field and develop Arcada's optical microscope for thin section analysis of polymers.

Lastly, this thesis work will provide an extension in the infrastructure of plastics analysis to an important field at Arcada, which can be utilized in future projects and in teaching of new students.

The structure of this thesis work is divided into the following four sections:

- Review of methods
- Methods used in this study
- Experimental results and analysis
- Conclusions

The review of methods concludes the preparation procedures of thin section sample according to literature studied for this work. The preparation procedures that have been used in this thesis work are explained in methods used in this study, while experimental results and analysis discusses the results of the methods with images of the sample produced with interpretation of them. The conclusion discusses the achievement of aims and recommendation for future studies and analysis of polymer thin sections.

2 REVIEW OF METHODS

2.1 Thin section – definition

A thin section is a cross section of an object, which is thin enough for light to transmit through, and analysed with optical light microscopy. There is no specific thickness the cross section needs to be, rather the thickness depends on the material and its transparency. The thickness of the thin section also regulates visual features seen in the sample through an optical microscope and is therefore specific to the sample. Figure 1 illustrates the main stages of transformation from original sample to thin section.

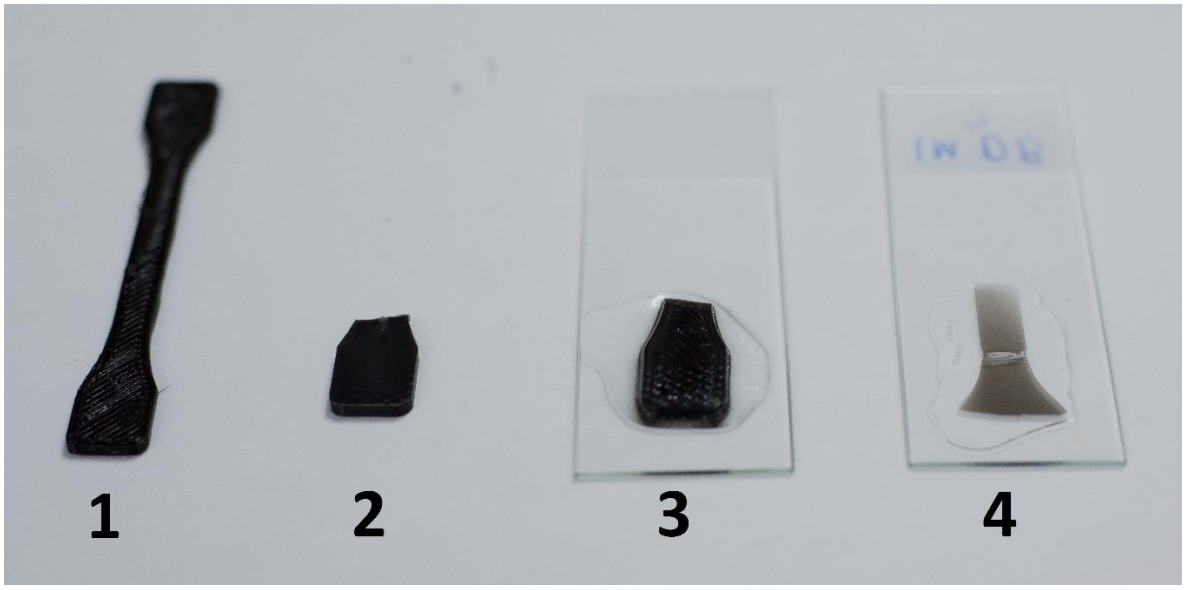


Figure 1: 1) Original sample 2) Cutting of sample and first polishing complete 3) Cemented sample prepared for second polishing 4) Finished thin section ready for microscopy analysis

2.2 Sample selection

When having selected the sample, the preparation begins by selecting a cross-section or plane through the sample that will provide useful information. Preparation steps such as embedding or cutting could potentially be skipped depending on the demand of the sample. Unlike cutting, polishing can produce thin sections in complicated planes or angles since the sample does not need to be secured for cutting, and can be embedded in a supporting resin for polishing. Limiting material properties are poor adhesion to embedding resin or cement, and toughness for wear. Polishing is a cutting method that removes small amounts of material at one time, which can require excessive amounts of time and resources if the material to be polished is resistant to wear.

2.3 Embedding

In the embedding stage, a casing is cast around the sample out of resin. The purpose of the casing is to provide the sample with a larger surface area for cementing and polishing, and also keep the sample upright if the selected surface to be polished is narrow.

Embedding of the specimen should be done in a resin with a strong mechanical structure and good adhesive bonding to the sample for creating a casing around the sample that will not detach during polishing. Epoxy and polyester are good general options but other resins with good bonding properties to the specific sample can also be selected (Böhme, 1990). The curing of resin is an exothermic reaction that can produce gas bubbles that become trapped within the resin. The resulting bubbles can cause the specimen to detach from its resin casing due to the mechanical forces produced during polishing. Since the volume of resin mixed for embedding is small, the amount of heat generated is relatively small and a fast curing resin is still preferred to make the process quicker. Resins with high rates of shrinkage are also prone to detach the specimen during polishing. When preparing hollow specimens the embedding process needs good planning to prevent cavities from forming, and a syringe can be used to inject resin into cavities of the sample before embedding (Böhme, 1990).

Embedding is not a necessity in all situations. If the thin section to be produced has a relatively large surface area on both the cementing and polishing surface, the embedding step can be skipped since the cement will have an adequate surface area for bonding, and the mechanical forces during polishing are distributed evenly over a large area. However, if the surface area is very small or the test piece needs to be polished in an upright position for desired cross section, a casing gives side support and distributes the force on a greater area while polishing. This also helps in controlling the material removal rate and thickness, to create a flush surface on the sample during polishing.

The embedding resin does not need to be visibly clear since the side of the sample to be cemented will be grinded until the specimen and embedding resin are flush with each other, hence the resin will not interfere with the optical path during microscopic analysis.

2.4 Cutting away excess resin

Depending on the mould shape for embedding, it could be desirable to remove some excess resin from the casting before polishing. Cutting is a task which has potential to damage the sample, in a scale not visible to the naked eye, e.g. by producing micro-cracks. Micro-cracks can be mistakenly observed as cracks or voids as features in the sample. Furthermore, vibrations from cutting the resin can cause the specimen to detach from its resin casting (Sawyer et al., 2008). In order to minimize the probability of such cracks, it is advised to make the rough cut at a good distance from the sample (Sawyer et al., 2008).

2.5 Polishing & cementing

2.5.1 Polishing

Since the surface cemented to the slide needs to have a good and clear finish to provide valid information during microscopy analysis, the polishing should be done in two steps regardless of embedding. The first polishing is to prepare the sample for cementing, while the sample is cut to its final thickness in the second polishing step.

All polishing and grinding of samples should be done wet. Water acts as a coolant and lubricant for the sample during grinding and also carries debris from the grinding away from the sandpaper (Sawyer et al., 2008).

2.5.2 First polishing

In the first polishing step, the sample should be grinded with a rough grain e.g. P320 sandpaper, to the bottom of the desired plane of observation. The sample surface is then polished with finer grain in incremental steps to P1200, to improve the surface quality for microscopic analysis (Böhme, 1990). Flushness of the sample surface is also of great importance at this stage, to enhance adhesion in the cementing stage (Böhme, 1990). The grain levels of the sandpapers used in this thesis are listed in Table 1 with their average particle diameter. Greater number refers to finer grain.

Table 1: FEPA/ISO Grain designations with their respective average particle diameter.

ISO/FEPA Grain Designation	Average Particle Diameter (μm)
P320	40.5
P600	25.8
P1000	18.3
P1200	15.3
P4000	2.5

2.5.3 Cementing

Before the second polishing, the sample is mounted to a glass microscopy slide as illustrated in Figure 2. The desired features in the adhesive for cementing are good bonding with glass and sample, and clear transparency. The transparency and clarity of the adhesive reduces interference when studying the sample with transmitted cross polarized light and is therefore of great importance. Ultrasonic washing of both the sample and the microscopy slide it is cemented to is advised to remove loose particles from the surfaces for a stronger uncontaminated bond (Böhme, 1990). The washing also removes small particles from the polishing, which during microscope analysis can be misinterpret as voids or other defects in the sample. Before cementing, the scratches from the first polishing should only be pointing in one direction if visible, to avoid confusion during analysis.

A mounting block can be used for applying a constant and centred force on the specimen during curing of the cement. This will result in layer with consistent thickness between the glass slide and sample. The weight should have a pin, through which the force of the weight is applied in the centre of the sample. (Böhme, 1990)

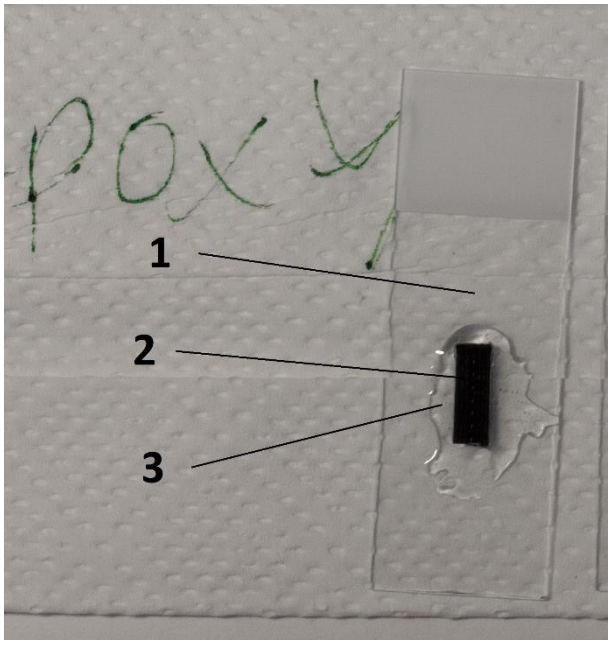


Figure 2: Non-embedded samples cemented to microscopy slides with epoxy after first polishing. 1) Microscopy slide 2) Sample 3) Cement

2.5.4 Second polishing

In the second polishing stage the top surface of the sample will be ground down to its final thickness. Like the first polishing, the grinding should start with a rough grain and incrementally transfer to finer grain, finishing off with no less than P1200 grain (Scheirs, 2000). A suction cup mounted to the opposite side of the glass slide suggested to aid holding the sample on the polisher (Böhme, 1990). When closing in on the target thickness, the additional thickness of the cement should be considered with a 10 μm section (Böhme, 1990).

2.5.5 Thickness of sample

When polishing, it is important to have control over the feed of the cutting, since too thin samples may not display any relevant information during microscopy analysis with transmitted light (Böhme, 1990).

2.6 Post-polishing treatment

2.6.1 Ultrasonic cleaning

After the second polishing of the samples they should be washed in an ultrasonic bath to remove the smallest of the grains adhering to the sample surface from the polishing paper (Sawyer et al., 2008). Ultrasonic cleaners send high frequency sound waves through water, which creates cavitation bubbles, which in turn agitates microscopic sized particles adhered to the sample surface. As a result, the debris is transferred from the sample to the water (Ensminger & Stulen, 2008). After cleaning, the sample can be dried off with Freon gas (Sawyer et al., 2008), or preferably less toxic but non-contaminating gas dusters. Compressed air or dryers that move air can contaminate the sample, with particles from the atmosphere. Wiping of the sample can cause damage in form of scratches, which can be misinterpret during analysis.

2.6.2 Immersion oil

To improve the image quality of microscopic inspection, immersion oil can be utilized, by applying a drop on the sample and covering it with a cover slip. If the sample is to be stored for a longer period of time, the immersion oil can be washed off with alcohol (Böhme, 1990). Immersion oil reduces the refraction of light, and focuses it into the objective of the microscope. More light through the objective results in a clearer and sharper image, which is more obvious when using high levels of magnification (Microscope World, n.d.).

2.7 Microscopy

2.7.1 Reflected light

Within optical microscopy, the two modes for analysing samples are reflected and transmitted light. Reflected light microscopy is generally used to analyse the upper surface of a sample, since the light from the lamp is directed to the top surface on the sample via the objective, from where reflections are captured by the objective that magnifies the image and transmits it to the ocular lenses or an image plane in the microscope. On semi-transparent samples, some features beneath the surface can be observed with reflected light.

2.7.2 Transmitted light

Transmitted light microscopy is used on thin, near transparent samples. A light source is placed underneath the sample and directed through a condenser which focuses the light on to the sample. After the light has travelled through the sample, it is focussed by an objective placed above up to the ocular lenses or imaging plane. By transmitted light microscopy, features within samples can be observed, with the perquisite of a semi-transparent sample (John Innes Centre, n.d.). Figure 3 illustrates the microscope used in this study, set up for transmitted light analysis.

2.7.3 White light filter

Light sources emit light with different wavelengths, and usually have a specific wavelength with grater intensity than the others. Therefore, light sources usually have a specific tint. For example, light emitted from a candle has a yellow colour since the light contains primarily wavelengths from the yellow range of the visible light spectrum (Rottenfusser, et al., n.d.).

In microscopy, tinted light is not commonly desirable, since it generally manipulates the colour of features observed in samples. Light transmitted through a white light filter has wavelengths with equal intensity, and as a result, the light can be referred to as neutral.

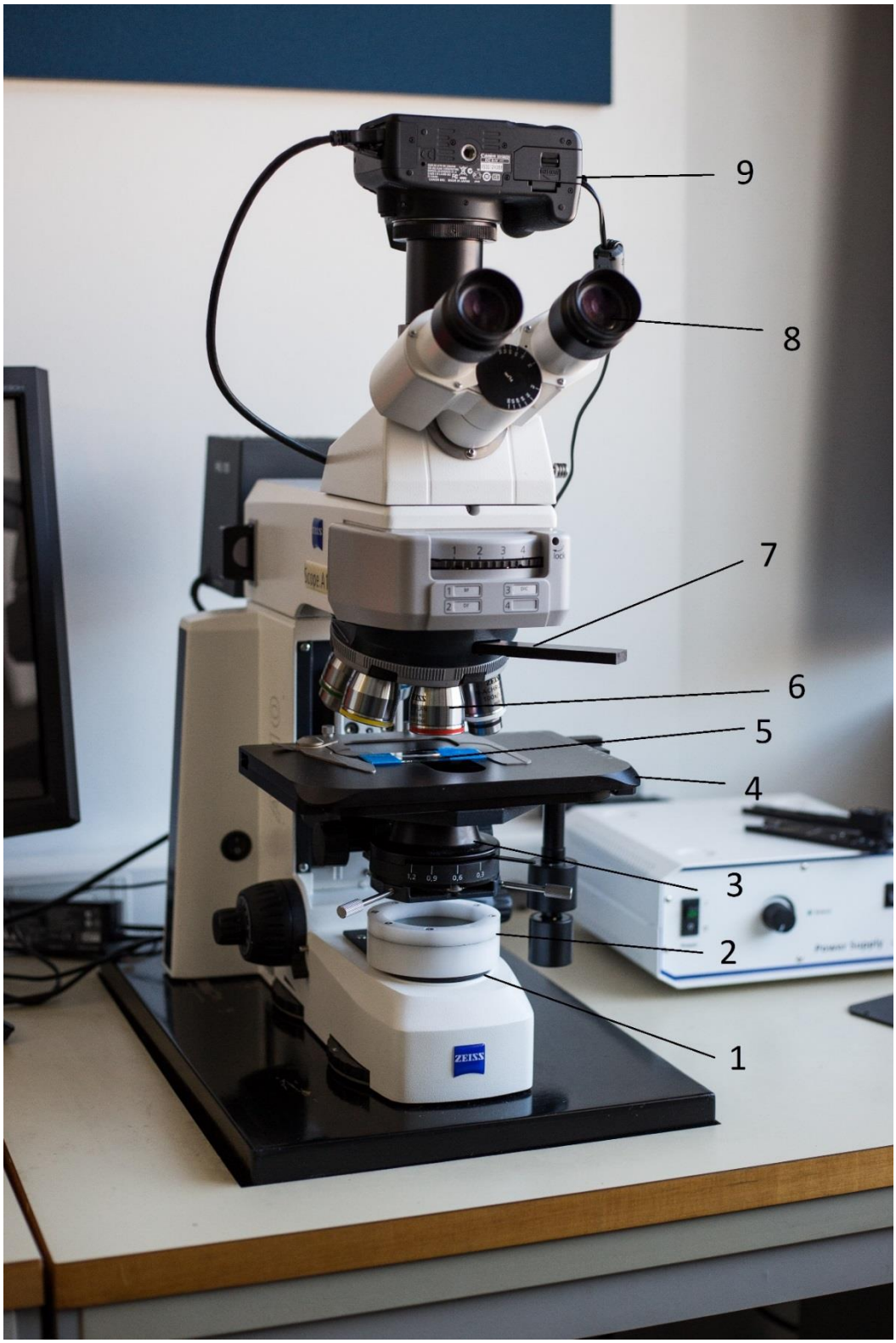


Figure 3: Zeiss Axio Scope. A1 optical light microscope, setup for transmitted light. 1) Illumination source 2) Custom polarizing filter 3) Condenser 4) Stage 5) Sample 6) Objective 7) Custom analyser 8) Ocular lenses 9) DSLR camera

2.7.4 Polarized light

Polarized light is a key factor for extracting information from thin sections in transmitted light microscopy, since thin sections of anisotropic polymers are nearly fully transparent. With unpolarised light, a thin section would only reveal features that disrupt the light transmission through the sample. Cross polarized light reveals more detail in anisotropic polymers due to the contrast it creates when transmitted through a sample, as presented in Figure 4.

Figure 4: A sample of 3D Printed PLA viewed with crossed linear polarizers (top) and un-polarized light (bottom) illustrating the difference in contrast.

2.7.5 Linear polarization

Visible light is electromagnetic radiation with a range in wavelength the human eye can detect. Light waves vibrate perpendicularly to the propagation of the light beam in all directions at different amplitudes.

When light passes through a linear polarizer, only vibrations in the electric field vector parallel to the polarizer are transmitted, and all other vibrations are absorbed or reflected. All light passing through the polarizer is vibrating in a parallel pattern and is called polarized light. As a result, polarizers also reduce the intensity of light (University of Cambridge, n.d.).

When placing two polarizer in a perpendicular orientation on top of each other, the upper polarizer (analyser) will absorb the polarized light, since it will strike with vibrations in perpendicular orientation to the upper filter. This orientation of the polarizers is illustrated in Figure 5 and called extinction, since no light will transmit through the second polarizer unless an anisotropic samples is placed between them.

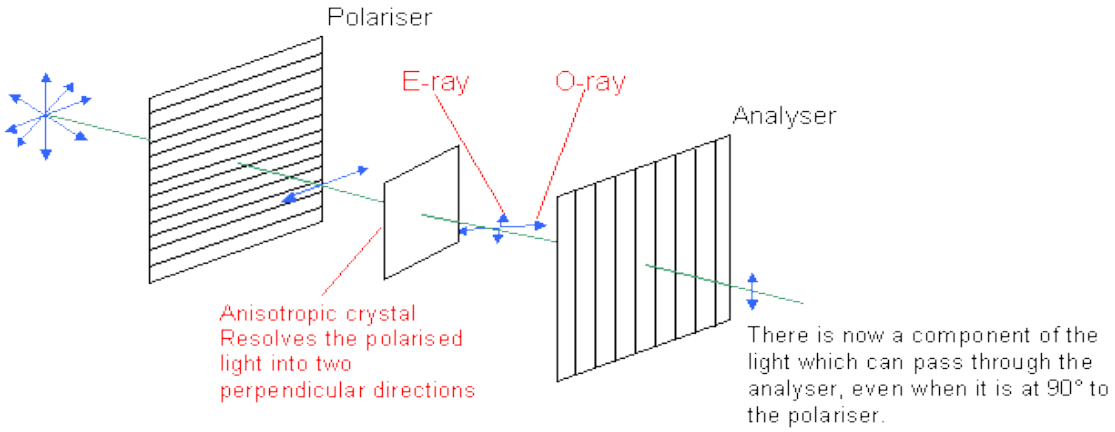


Figure 5: Diagram of crossed polarizers with sample in-between. (University of Cambridge, n.d.)

2.7.6 Anisotropy

When polarized light is transmitted through an anisotropic sample e.g. crystalline or semi-crystalline polymer, the beams split to ordinary and extraordinary rays that vibrate in fixed angles. When the light transmits through the second polarizer (referred to as the analyser), the vibrations perpendicular to the analyser will be absorbed, while parallel vibrations will transmit through (University of Cambridge, n.d.).

2.7.7 Isotropy

Isotropic or amorphous materials, e.g. amorphous polycarbonate, do not affect polarized light, since light can vibrate through it with equal effort in all directions (University of Cambridge, n.d.). Due to its transparency, an isotropic sample would appear dark when viewed through crossed polarizers.

2.7.8 Rotation of anisotropic sample

The brightness of the sample can be adjusted by changing the orientation in relation to the crossed polarizers. The brightest position is at 45º relation, which can be achieved at four different positions 90º apart from each other, during one rotation. By contrast, the darkest positions are at a 90º relation in 90º increments. The sample turns dark when the vibrations of light caused by the anisotropy become perpendicular to the direction of propagation. (Olympus, n.d.)

2.7.9 Circular polarization

Unlike linear polarized light, circular polarized light vibrates in two planes perpendicular to each other and the propagation, with equal amplitude and a 90º difference in phase. This creates a resultant oscillation, which rotates in a circular pattern around the axis of propagation as illustrated in Figure 6 (Nave, 2012). With crossed laminar polarisation, birefringent material appears bright if the optical axis of the material is oblique to the axes of the polarizers. As a result, the sample may appear as disconnected bright regions (Stain Technology, 1986).

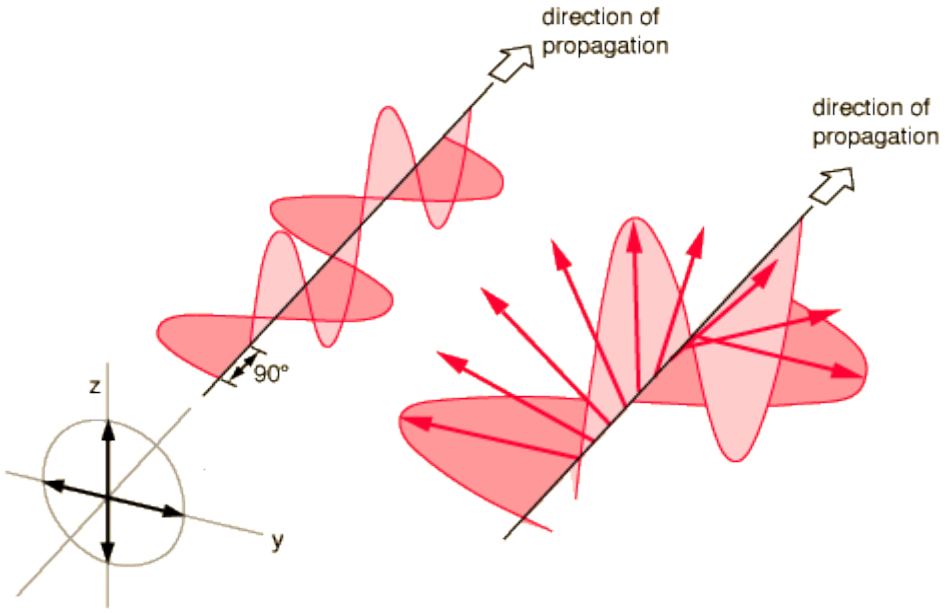


Figure 6: Illustration of electromagnetic waves in circular polarized light. (Nave. R, 2012)

Linear polarized light can be converted to circular polarized light by transmittance through a quarter-wave plate at a 45º angle. By contrast, circular polarized light can be converted to laminar polarized light with a quarter-wave plate at a zero degree angle. A quarter-wave plate retards oscillation of light by a quarter of a wavelength (Olympus, n.d.).

2.7.10 Differential interference contrast (DIC)

Differential interference contrast is a form of polarisation that can give samples 3-dimensional appearance with great resolution (John Innes Centre, n.d.).

DIC can be achieved for transmitted light microscopy with a similar setup to that of circular polarization, however, the result and features are different. For DIC linearly polarized light is transmitted through a Wollaston prism, which splits the linearly polarized light into two beams with polarized oscillation in a perpendicular relationship to each other. The result is similar to that of circularly polarized light, however, the two beams that oscillate in perpendicular planes have a different propagation rather than the same. Following the transmission of the Wollaston prism, the beams are aimed toward the sample by a condenser. After passing through the sample, the objective focus the split beams to a specific point in another Wollaston prism, which combines the beams which are then transmitted through an analyser for linear polarization (Lang, 1968). Figure 7 displays the optical path of a DIC setup for transmitted light.

The splitting of beams effectively results in having the sample illuminated by two different light sources, one with a 0º polarization and the other with 90º polarization. In microscopic imaging, this can give features in samples a three dimensional appearance.

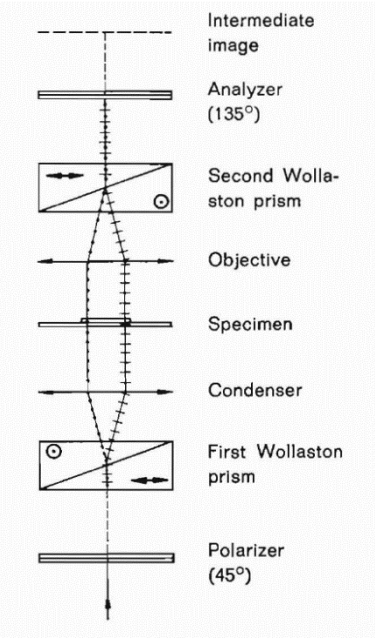


Figure 7: Scheme illustrating DIC setup for transmitted light (Lang, 1968)

2.7.11 Features visualized by thin section microscopy

Weld-lines

Weld-lines are apparent in injection-moulded parts where there are two or more gates for the melt to enter the mould, or if there are features in the mould that cause disruption in the melt flow. Weld-lines are created when two streams of melt collide and fuse together in the mould. The strength of the weld-lines may vary between 20-100% of the nominal strength depending on various factors, such as selection of material, injection pressure and temperature. The strength of a weld-line is dependent on how well polymer chains from the melt fronts are entangled to each other. Amorphous polymers commonly presents more homogeneous melt at weld-lines than semi-crystalline, because of greater flow rate. (Bozzelli, 2008)

Weld-lines should be accounted for at the stage of designing the mould, since the orientation and angle of the weld-line has also a great effect on its strength. Melt fronts that meet head-on are generally of the weaker type, while melt flow separations that later meet are stronger. Inserts in moulds are the most common cause for melt-flow separation, but also a cause for internal stresses. (Scheirs, 2000)

Visual, non-destructive inspections of weld-lines can help to adjust processing parameters to some extent, whereas thin-section analysis of weld-lines reveal information of the homogeneity of the weld. Weld-lines are also prone to trap bubbles of gas, which decrease strength and are not apparent in full context in visual inspection of the surface, but visible on cross-section perspectives. A thin section view of the melt homogeneity in a weld-line can be compared to nominal areas of the piece, to visually determine the strength of the weld to the rest of the part. This information is useful to prevent premature failure of polymer parts under nominal intended stresses.

Crystallinity

Crystalline polymers contain obtain molecular chains with orderly structures called crystallites, in a small scale which cannot be observed by optical microscopy. Crystallites easily form superstructures featuring spherulitic arrangement, which can be observed by optical microscopy of thin-sections at moderate levels of magnification. Hardness, strength and brittleness of plastics depend on the degree of crystallinity in the material. (Johannaber, 1994)

The molecular structure of a material is the main deciding factor in degree of crystallization, but it can also be altered during processing. In injection moulding, the mould temperature has the greatest effect, since high rate of cooling results in lower crystallinity. Parts cooled with a high rate generally have a skin nearly transparent skin. These areas are affected by post-crystallization, which can be a long lasting phenomenon that causes change in properties and dimensions. (Johannaber, 1994)

Shearing of the melt at low temperature can also affect crystallinity. When injecting moulding a low temperature melt in a cold mould, the result is going to be a product with very heterogeneous structure. A homogeneous crystalline structure is generally most desirable in all plastics parts, since a heterogeneous, un-even structure is the main cause of premature failure. (Johannaber, 1994)

In thin-sections of injection moulded parts, the crystalline structure and spherules can be visually observed under optical microscopy with crossed polarizers. Features in parts caused by rapid cooling can be analysed and consequently processing parameters can be adjusted for a desirable result. In essence, thin-section analysis can be beneficial for preventing premature failure of parts, prevent deformation and property change.

Crystallinity of polypropylene sample is illustrated in Figure A1 in the appendix and spherulites are illustrated in Figure A2.

Molecular orientation (Injection moulding)

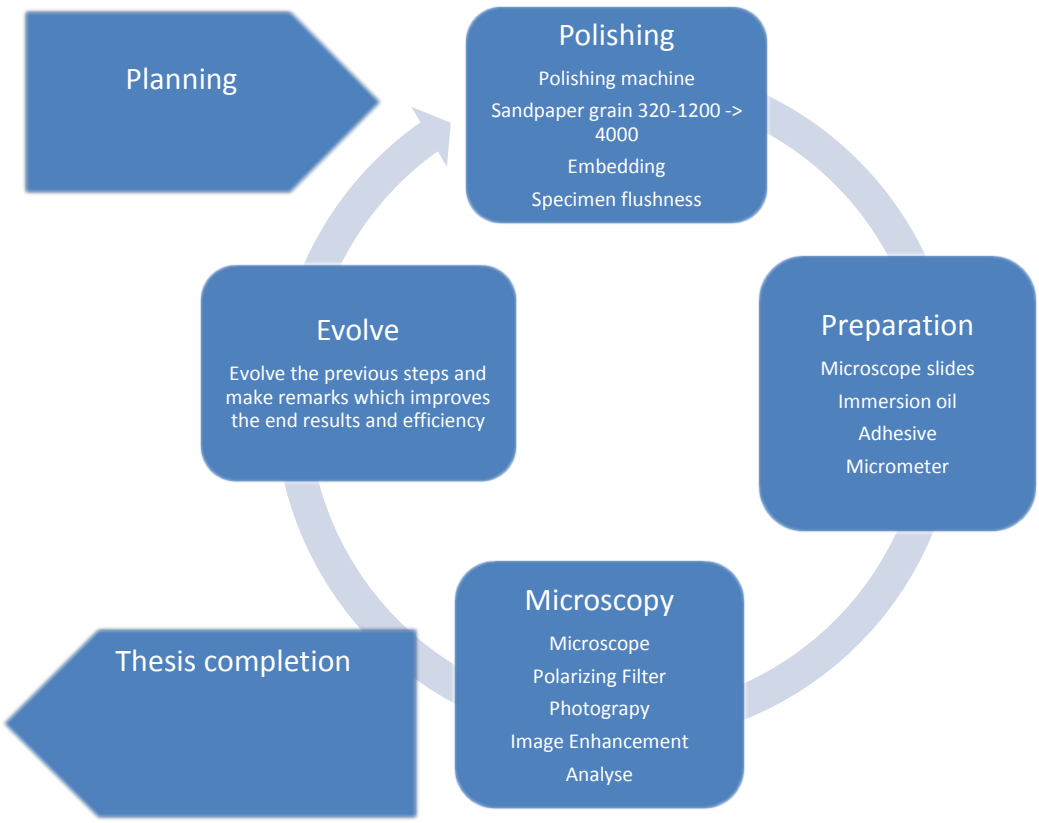
When a melt is injected into a mould in a preferred direction, the molecular chains within the material align in a particular orientation from their original irregular orientation. Shear velocity of the melt is high close to the cavity walls, where the melt is stretched, because the melt has a tendency to adhere to the walls. Due to the adhesion, the melt in the centre of the cavity has a more rapid flow velocity than areas close to the walls. Once the injection process is completed the melt comes to a standstill and regains most of its irregular orientations due to thermal motion. This is referred to as relaxation of the material. Relaxation usually occurs a few seconds after the injection in completed, but continues to some extent until the temperature drops below the material glass transition temperature. (Johannaber, 1994)

The magnitude on how many of the chains remain in their particular orientation depend on the cooling rate of the part post-injection. A faster cooling rate below the materials glass transition temperature, will cause more of the chains to "freeze" in their orientation. Generally, a warm mould with optimised cooling is more desired, since rapid cooling can decrease the toughness of some materials and cause brittleness. If a part is heated above the glass transition temperature after the moulding process, this could result in deformation of the part if cooled too rapidly, since the molecular chains may begin to recoil onto their original orientation again. (Scheirs, 2000)

3 METHODS USED IN THIS STUDY

3.1 Structure

With methods applied from other scientific fields, the procedure of this study is quite straight forward. Initially each individual procedure for producing thin section samples will be followed by guidelines of other fields, with some changes to make it more suitable for plastics. The three main steps are polishing, preparation for microscopy, and microscopy. Following each cycle, remarks will be made and questions arise. The next cycle will account for them by changing details in the methods to give answers and greater results. The cycle will go on until the results are at an adequate level, and methods are adjusted accordingly for polymers.



3.2 Samples

The experiments in this thesis work includes polymeric materials PLA, PC, PP, PTFE and UHMWPE. The samples include both pristine materials, recycled 3D printed materials from Arcada, and recycled waste material from external sources. The materials and processing methods are listed in Table 2.

Table 2: List of materials used in this study with their respective processing methods.

Material	**Processing method**
PLA	Injection moulded / 3D printed
PC	Extruded
PP	Injection moulded
PP waste	Injection moulded
PTFE	Extruded
UHMWPE	Extruded

The majority of samples are excess dog bones (illustrated in Figure 8) used for tensile testing in other experiments, and were selected due to their availability. Samples have been produced out of both pristine pieces which have not experienced stresses or wear, and tensile tested pieces, both intact and broken. Pristine samples are ideal for observing features in crystallinity and weld lines produced by the manufacturing method, while tested pieces are used for analysing damage and change in crystallinity due to testing.

Figure 8: Injection moulded "dog bone" out of PP used in this work.

3.3 Embedding

For experimenting, the resin chosen for sample embedding was West Systems 105 epoxy with 206 slow hardener due to its desirable features of resins available. The mixing ratio of the components was 5:1 (epoxy:hardener) with a gel time of 10-15 min, and curing time of 9-12 h. The moulds were disposable paper coffee cups. The inside of the cups featured a wax coated surface, which made them waterproof for their intended use. This feature was beneficial for moulding since it provided a surface from which the cured resin could easily be detached.

The mixing of resin was done in a separate cup from the samples and the mixing ratio was measured by weight.

3.4 Cutting

Post embedding, the samples were cut to fit on a microscopy slide using a band saw. The band saw was equipped with a blade for cutting of composites, featuring smaller teeth than a blade for wood.

Epoxy and resins in general, are hard materials relative to wood, which is why large teeth can cause damage on the sample or blade since they are design to cut large amounts of soft materials.

In cases where embedding was not utilized, thick samples were cut with a band saw, while thinner samples with side cutters or scissors. Embedded sample cut with band saw are illustrated in Figure 9.

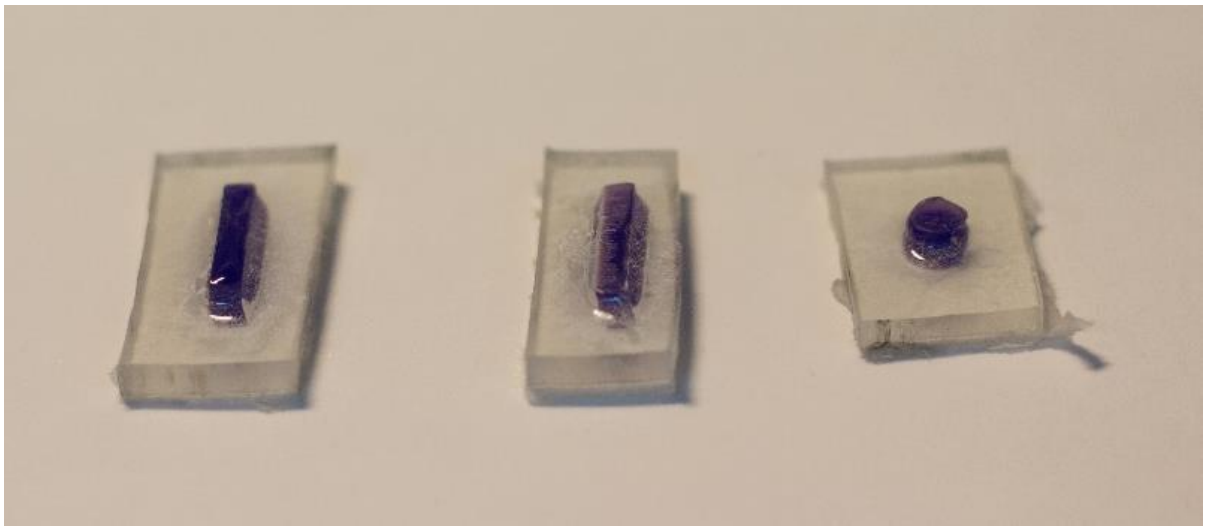


Figure 9: Injection moulded polypropylene samples cut with band saw after embedding in epoxy resin.

3.5 Cementing

Three adhesives were tested for cementing with varying results. The samples were cemented to microscope slides by spreading out the adhesive on the slide, and then pressing the sample on top. Mounting blocks or weights were not used in this study since a block was not available, and weights were problematic due to their tendency of shifting the samples of the glass slides due to improper distribution of pressure to the samples.

3.5.1 Cyanoacrylate

Cyanoacrylate adhesive branded as Loctite 435 was applied to glass slides for cementing both embedded and unembedded samples. Cyanoacrylate was a preferred adhesive for cementing by literature due to it being convenient application and strength and therefore tested in this work. Cyanoacrylate has a very short curing time, however, the samples were still left to cure overnight.

3.5.2 Quick epoxy

Quick epoxy by Biltema was utilized for cementing similar samples as for the cyanoacrylate, due to its strength and in testing for benefits over the cyanoacrylate. The epoxy featured a base and a hardener which were mixed in a 1:1 ratio. The resin began curing in approximately 5 minutes, and obtained full hardness in approximately 2 h. The quick epoxy was left to cure overnight after application.

3.5.3 Epoxy

The West Systems 105 epoxy resin with 206 hardener was also tested for cementing, since it could potentially provide an even clearer transmission of light than the quick epoxy. The main downside with the epoxy is its slow curing time, and after being let to cure overnight after application, it still showed some softness. After having cured for approximately 38 h, the resin was stable enough for polishing.

3.6 Polishing

All polishing for this thesis work was performed on a circular wet polishing machine, with grain ranging from P320 to P4000. The device was a Latzke Innovation 200 M featuring a polishing table diameter of 200 mm. The speed of rotation was 300 rpm and could not be adjusted. The maximum power output of the motor was 0.2 kW. The Latzke Innovation 200 M used in this work is illustrated in Figure 10.

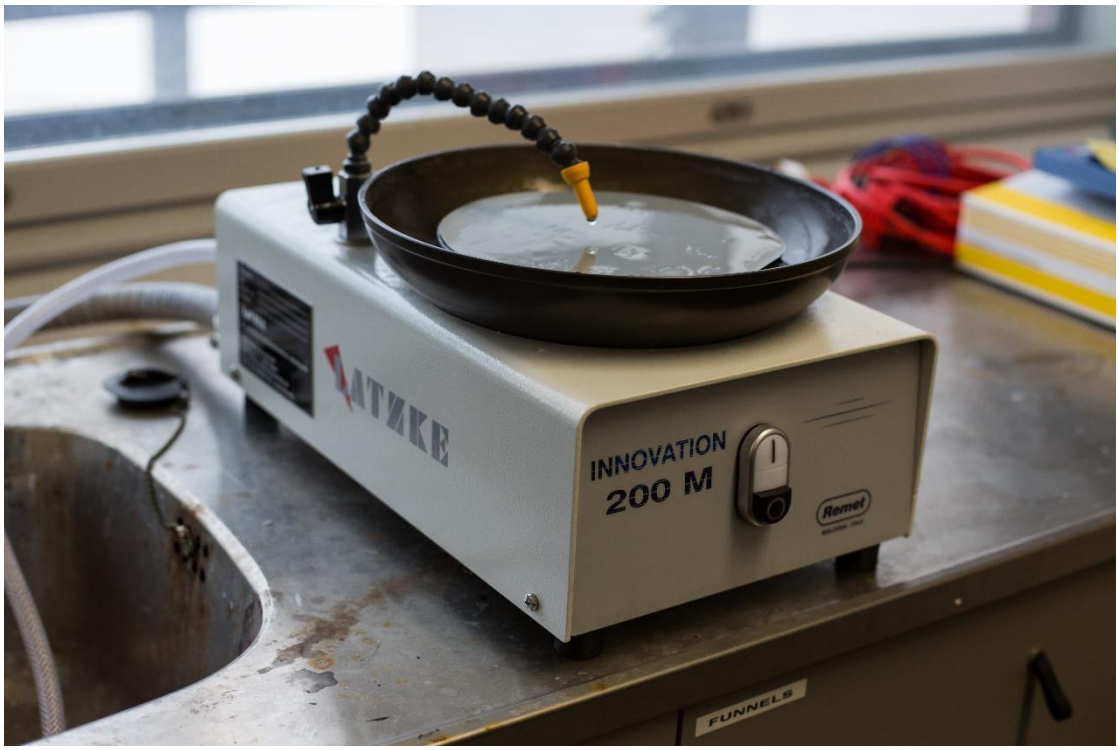


Figure 10: Latzke Innovation 200 M wet polishing machine

3.6.1 Grain

When polishing, rough grain sandpaper will remove more material at a faster rate, but also generates more stress on the sample than finer grain. However, when using fine grain it is possible for the person grinding to apply excessive pressure on the sample to increase the material removal rate. This lead to more stress on the specimen than a rough grit, and could cause the specimen to detach from its casing or the glass slide during the second polishing stage. The stresses also cause buckling of the specimen and glass slide, which can lead to water ingestion between the specimen and glass slide. Since fine grit only can remove little material, a rough grit was used until closing in on the final thickness. The finer grain papers were used to even out the surface and remove most of the deeper scratches from the rough grit.

3.6.2 Speed of rotation

The Latzke Innovation 200 M polishing machine used in this study, does not feature adjustability on the speed the polishing table rotates. However, the cutting speed was adjustable by moving the sample away or towards the centre of the table. When moving the sample towards the centre the cutting speed decreases, while moving it toward the edge the cutting speed increases. If the sample was moved to close to the centre, it was subjected to shear forces that could cause it to spin around the axis of rotation on the machine.

3.6.3 Cooling

Polishing generates heat due to friction produced between the sandpaper and sample, which is why wet polishing is critical for cooling the sample. The water holds the temperature of the sample at a low constant temperature, which prevents it from expanding and other damage. The water also acts as a lubricant and carries debris away, resulting in a finer finish and longer lifetime of the sandpaper.

3.6.4 First polishing

The first polishing is the step where the desired depth of observation for the sample is selected, and the surface is prepared for cementing. The samples was grinded down with a rough grain of P320 to the desired level and then polished with P600 for a fine finish for cementing. A rough grain removes a good amount of material and the sandpaper was not filled up with debris too quickly, however, a parallel surface was difficult to obtain by hand due to the rapid material removal rate.

When fine polishing the surface that will be cemented to the glass slide, it is important to focus on obtaining a parallel surface. Theoretically, a rough surface would be good for cementing since it gives a larger surface area for cement to adhere, however, a non-parallel cement layer would make it difficult to judge the thickness of the final thin section. Thickness variations of the sample during polishing were measured with a micrometre and pressure adjusted to thicker areas during fine polishing with P600 grain. The sample were turned 90° within short intervals during polishing to obtain a good quality and parallel surface.

3.6.5 Second polishing

In the second polishing stage, the samples was grinded down to its final thickness for microscopy analysis. Because the samples were at this point very thin, it is important not to subject them to great mechanical forces, and focus on obtaining a flush surface to have a greater depth of view during microscopy analysis. The second polishing begins with P320 grain and finished with no rougher than P1200 grain.

3.6.6 Water flow-rate

When the sample becomes thin, the glass slide with the specimen tended to stick to the sandpaper and fly out of hand. This was likely caused by having an excess amount of water running on the sandpaper. When there is too much water on the sandpaper, it might begin to flow on top of the slide rather than underneath. This causes a suction effect on the bottom of the slide, which makes it to stick to the sandpaper. When using only a minute amount of water during fine polishing the probability to this reduced, and on the other hand, the specimen was also less likely to aquaplane on the water.

3.6.7 Measuring thickness of polished samples

A Mitutoyo NO.2119-50 micrometre with a 1 µm accuracy, was used to measure the thickness of the polished specimen but since the pin on the gauge has a very small surface area, it left marks on soft samples, which can be misinterpret during analysis. After the final polishing all scratches should be pointing in the same direction and no measuring should be done to obtain a clean surface for analysis. When analysing samples, the precise thickness is not of great importance, unless two or more samples are compared to each other.

3.7 Preparation

3.7.1 Ultrasonic washing of sample

After polishing, the samples contained small debris of different size from the grinding process. Wiping the specimen with a wet tissue can result in damage on the fine polished surface, therefore the samples were given an ultrasonic bath in a baker of de-ionized water for 30 s or longer.

3.7.2 Ultrasonic washing of microscopy slides

Pristine microscope slides feature contamination which disturbs analysis of thin sections under microscope. Before cementing the slides were wiped with ethanol to remove grease and large particles of the surfaces, followed by a 30 s ultrasonic bath in a beaker containing de-ionized water, for removal of smaller debris. The procedure ensured the best circumstances for the cement to adhere.

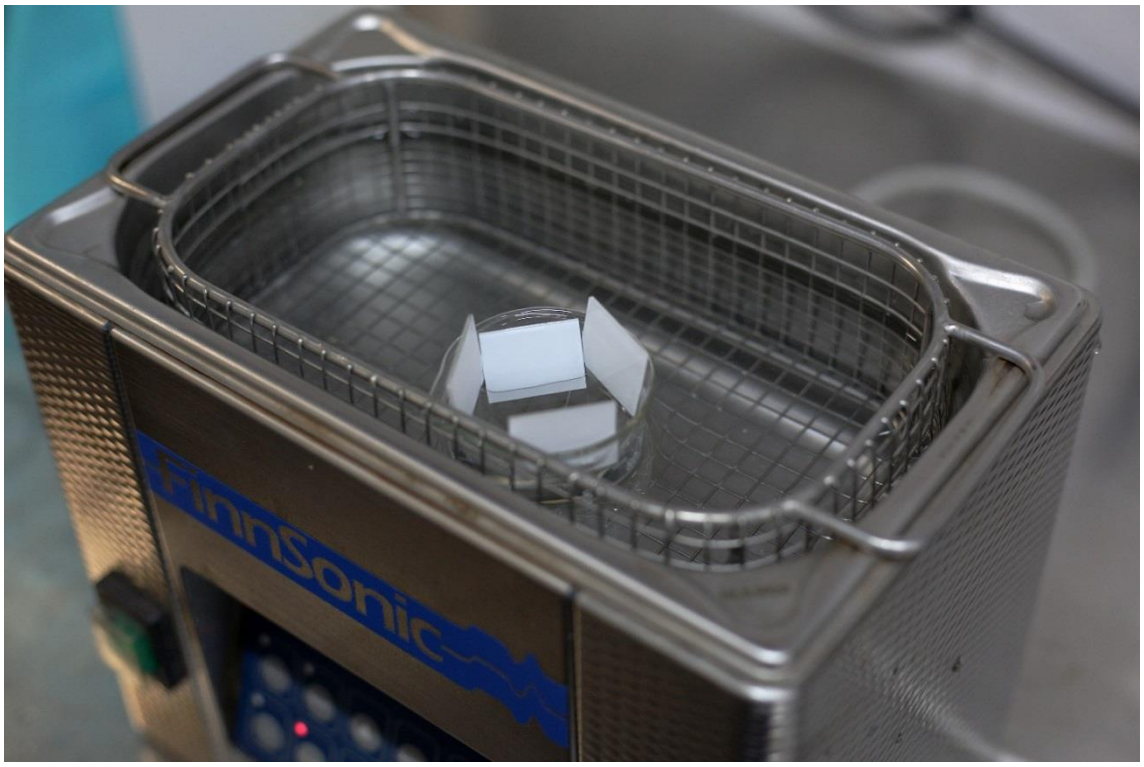


Figure 11: Ultrasonic washing of microscope slides in Finnsonic m03 washer at 40 kHz frequency, in baker containing de-ionized water.

3.7.3 Ultrasonic bath

The ultrasonic bath used for this work was a Finnsonic m3 cleaner, producing ultrasonic waves at 40 kHz frequency, illustrated in Figure 11.

3.7.4 Immersion oil

Zeiss Immersol® 518N immersion oil was applied for some samples for improving the image quality. A droplet of immersion oil was applied on top of the samples, and covered with a glass slip for even distribution, and for protecting the microscope objectives from contact with the oil. To ensure a clear view, the sample and covering slip should be ultrasonically washed before application of immersion oil.

3.8 Microscopy

3.8.1 Microscope

The microscope used in this study was a Zeiss Axio Scope. A1 optical light microscope, capable of transmitted and reflected light observation. The objectives provided magnification levels of 5x, 10x, 20x, 50x and 100x, with addition of a 100x magnification objective for immersion oil applications.

3.8.1.1 Reflective light

For reflected light observation, the microscope featured white light filter for illumination, aperture adjustment and a circular polarizer. Available illumination modes included bright light, dark field and DIC.

3.8.1.2 Transmitted light

In terms of filters for transmitted light, the only available was a white light filter and a circular DIC, however, there were adjustments for illumination intensity, aperture and condenser height. The condenser height could be adjusted with the aperture for focusing the illumination on a smaller area of the sample. This was very beneficial for high levels of magnifications, since higher magnification objectives reduce the amount of light passing through the objective to the eyepieces.

3.8.2 Polarizing filters

Since thin sections of polymers are effectively transparent in unpolarised light, the fundamentals of thin section analysis would require a polarizing filter below and above the sample in crossed relation to each other to create vitally important contrast for transmitted light observation.

Since polarizing filters were not available for the needed application, the decision was made to produce the filters for justifying the need of crossed polarization and potential future investments in high grade components for Arcada.

For this study only laminar polarizers were used for microscopy analysis of thin sections.

3.8.2.1 Experimental prototype

The first prototype was made out of excess polarizing film of unknown grade sourced from another project. The lower polarizer was constructed by adhering a piece of the film on a microscope slide, since the film possessed an adhesive layer on one side. The slide was then placed above the light source of the microscope, such that the film was on the top of the slide to reduce potential interference of the polarized light created by the microscopy slide.

The analyser (top polarizer) consisted of only a piece of the film being placed on top of the sample, with the adhesive layer removed with alcohol. Placing the filter on the sample, brings it close to the focal plane of the objective when observing the sample. This results in dirt, debris and scratches on the filter interfering with the analysis. The interference could be abolished if the filter was placed further away from the focal plane.

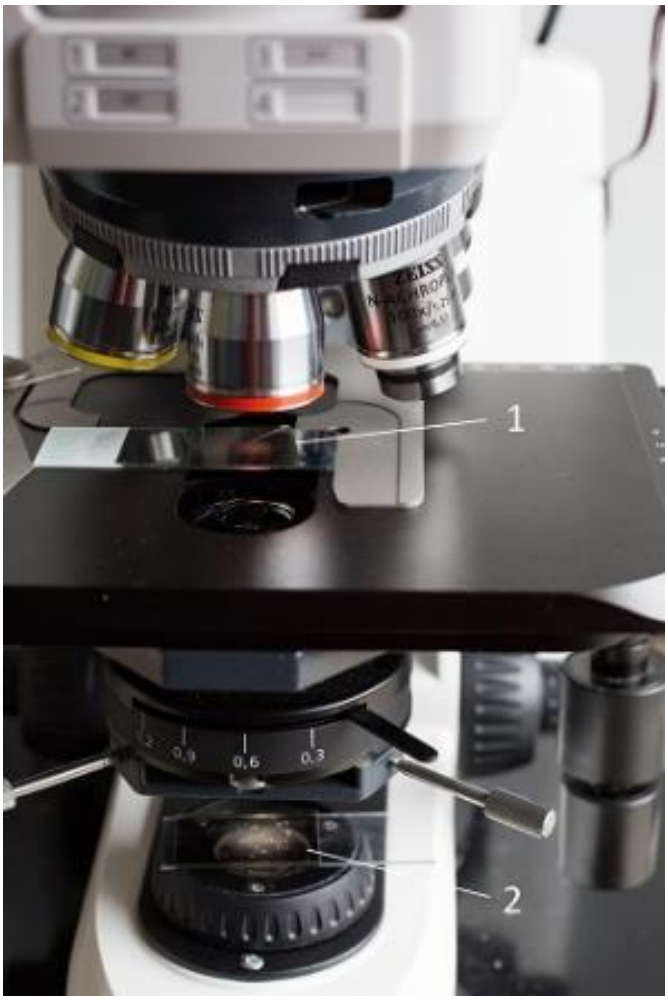


Figure 12: First prototype, featuring analyser (1) and lower polarizer (2)

In practical use, the analyser would remain stationary when placed on top of the sample, while the lower polarizer could be rotated by hand above the light source as illustrated in Figure 12.

The first prototype displayed the effect of the principle and its importance for polymer thin section analysis with great success, and inspired to develop the arrangement for polarizing films.

3.8.2.2 Polarizing filter holders

Polarizing films displayed adequate performance for analysis in this assessment, therefore thoughts on developing polarizing prisms and optics were discarded due to complexity.

The two main drawbacks of the first prototype was the interference of the analyser, and lack of ease in rotation of the lower polarizer. Having a fixed analyser and rotatable polarizer was seen as a manageable arrangement which could be integrated for the Zeiss Axio Scope A.1 in a more practical manner.

3.8.3 Lower polarizer

3.8.3.1 Design

The area around the light source of the microscope featured an edge on to which a wheel could be placed and rotated without interference. As a result, a wheel was designed on 3D software, consisting of two parts. A polarizing film was then to be placed in-between the two wheels which are then secured together with bolts. The design enables the film to be replaced or changed for another type of filter, and enables turning of the polarizer with ease. Figure 13 illustrates a 3D assembly of the polarizer drawn in Solidworks.

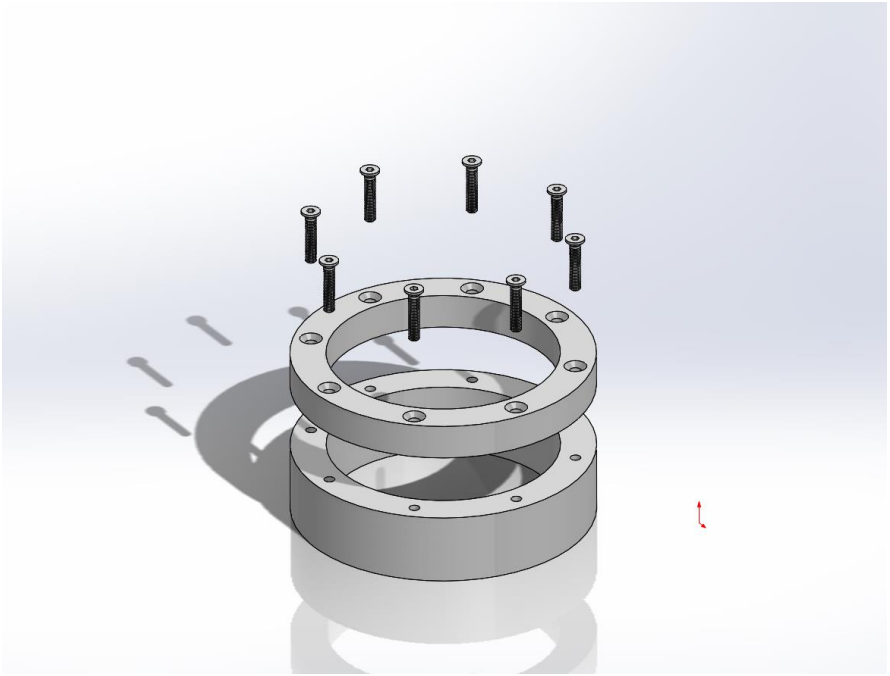


Figure 13: 3D rendering of the lower polarizer assembly in Solidworks, excluding the polarizing film

3.8.3.2 Manufacturing method

Due to the lack of complexity of the piece, it was decided to be CNC machined. The manufacturing method is time efficient for this item, and would produce high quality results out of a solid material to become durable. The filter was not to be manufactured for this thesis work only, but to be passed on for further work at Arcada. Therefore it needed to be tough and wear resistant.

3.8.3.3 Material

Material options for the part included mainly plastics and polywood. Metals were discarded since that could damage and scratch the microscope due to their hardness, when the filter is rotated around the illumination source. Due to their hardness, metals are also slightly more difficult to machine compared to polymers.

Polyoxymethylene (POM) was the material selected for this piece due to its availability and suitability for machining. POM is a material with low friction characteristics which is desirable for this application, since the filter will be rotated around the illumination source when resting on the metal edge.

3.8.3.4 Machining

The two wheels of the assembly were machined separately but in very much the same manner. For both parts, a plate of POM was face milled to the final thickness of the wheels into which the holes were then drilled in their planned positions. The plate was then secured to another thicker plate with temporary bolts through the holes, which was secured in a vice on the machining table. The wheels were then machined by their contours and ready after the bolt threads had been made. On the top wheel, the bolts were countersunk for the surface to remain flush. Figure 14 illustrates a top view of the complete assembly with polarizing film installed.

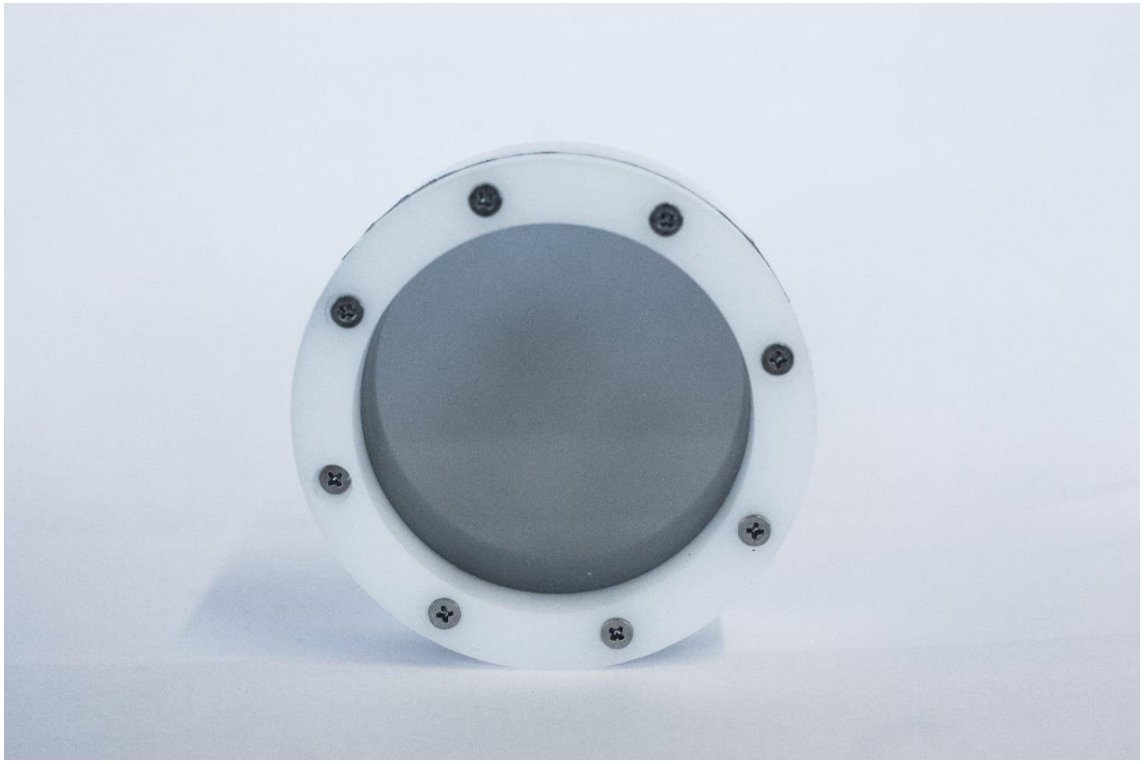


Figure 14: Complete lower polarizing filter assembly with film installed.

3.8.4 Analyser

The analyser (top polarizer) was not required to be rotatable since the filters relationships could be adjusted by turning the lower polarizer.

The microscope featured a rotatable turret, in which different illumination modules were installed for reflected light observation. The turret had one empty slot where an analyser could be installed, but that would result in difficulty changing from polarized to non-polarized observation, since the turret would need disassembly for removal of the filter.

Between the objective and the turret of the microscope, there was a slot for a circular DIC filter to be positioned in the optical path. The DIC filter is a narrow plate with a circular filter that can be inserted and removed in seconds. Due to the convenience of its function, it was decided that the analyser should be placed in the same slot for the modularity of the microscope to remain intact.

3.8.4.1 Design

The slot for the circular DIC filter featured a ridge to guide the plate to the correct position in the right orientation along with the precise dimensions of the part. The most reasonable way to proceed was to produce a plate with identical measurements and features, and produce a pocket and lid at the area of the optical path, for a piece of polarizing film to be inserted. Likewise to the lower polarizer, the analyser was designed for the filter to be interchangeable for utilization in future projects. A 3D rendering of the analyser plate assembly is illustrated in Figure 15.

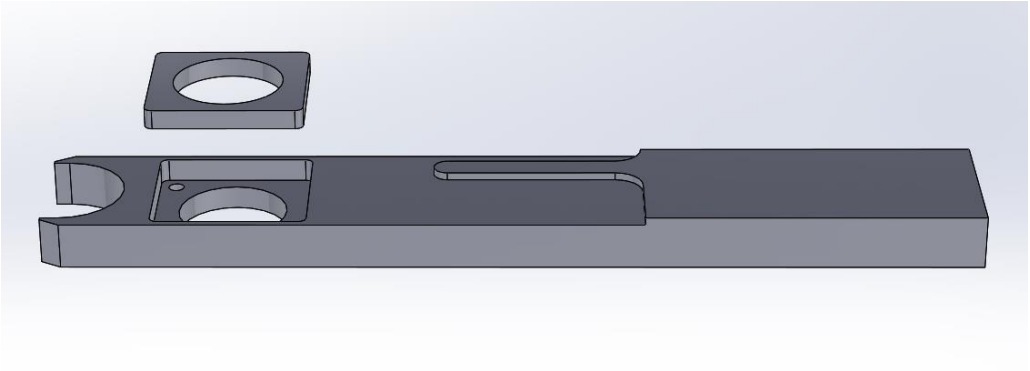


Figure 15: Analyser assembly rendered in Solidworks

3.8.4.2 Material

With successful material selection of the lower polarizer POM was also selected for the analyser, but in black colour to minimize the risk of interference by reflections, since it is placed above the objective. The toughness and low friction characteristics of POM are desirable for this application as well, since the plate will be slid in and out of the slot in the metal frame, otherwise resulting in wear over time. The machinability of the material is of great importance since the application require small details to be machined with great accuracy for the stick to be positioned correctly in its slot.

3.8.4.3 Machining of analyser

A suitable piece of excess POM was face milled flush by its surfaces and to the correct thickness. While the work piece was still clamped to the vice on the machining table after the face milling, the filter end with the half circle at the end of the stick was machined by following the outside contours.

The ridge on the analyser reaching a height of 1 mm was machined with a pocket program after the work piece was lifted 3 mm above the vice, measured from the highest surface area on the piece. This action left 2 mm of space from the milling tool to the top of the vice when machining the contours of the ridge, while leaving enough area for the work piece to be secured in the vice by its sides.

The pocket for the filter was machined halfway through the thickness of the stick to leave some thickness and rigidity for the lid.

The lid was manufactured by face milling a plate of POM to the desired thickness, and then drilling the hole for the optical path, followed by the outside contours of the lid.

According to initial plans, the lid was to be secured by bolts or tape, but the precision of the CNC machine enabled the lid to be milled 0.05 mm narrower than the pocket, which enabled it to be secured by the friction of the tight fit.

On the top side of the analyser, two holes were drilled through the pocket in the stick for removal of the lid. This enables replacement and cleaning of the film when in use.

3.8.5 Polarizing film

A standard grade polarizing film was selected for this application, due to its reasonable cost, availability and adequate performance. According to the retailer, the film is aimed for applications in experimenting and teaching, but can also be utilized in microscopy. Table 3 features the specification of the film, provided by the retailer.

Table 3: Specifications of polarizing film according to retailer.

Colour Distortion	None
Polarization efficiency	99%
Transmittance single	38% +/- 1% @ 550 nm
Transmittance crossed	<0.005 @ 430-670 nm
Haze	1%
Thickness	0.15 mm

3.8.5.1 Concerns

Main concern regarding the polarizing film was its resistance to heat over a longer time period, since the lower polarizing filter was placed right above the illumination source of the microscope. This created an air pocket between the film and the illumination source with little air circulation. Long-time observation of samples could cause the film to warm up, soften and buckle, causing interference in the image.

3.9 Microscopy photography

Photography is an important aspect of documentation during microscopy analysis, since it provides visual proof for explanation and clarification of observations. Photos are also used for comparison of samples.

3.9.1 Cameras used in this study

The Zeiss Axio Scope.A1 microscope used in this thesis work featured a mount for Canon EF and EF-S DSLR cameras, enabling the use of Canon cameras from both the consumer and professional range. For this thesis work Canon EOS 500D and EOS 5D Mark III DSLR cameras have been used for photography.

3.9.1.1 Image sensor scale

When comparing images side to side, it is important to acknowledge that the image sensor size has a direct effect on the field of view. For example, the Canon EOS 5D Mark III has a full frame sensor, while the EOS 500D has cropped sensor with a crop factor of 1.61 compared to the 5D Mark III (Rehm & Butler, 2009). As a result, the images of the EOS 500D will have a smaller field of view with greater magnification.

Images photographed with full frame and cropped sensors cannot directly be compared side to side, since features and formation on the sample will appear larger on the images taken with the cropped sensor.

3.9.2 Image merging

Taking multiple images of an area in the sample and merging them together into one large image using computer software, is a common method to give greater context to images in microscopy, medical and other science related fields. Single images can be used for documenting details, but if larger areas need to be covered into one single image, merging is a useful method even at low magnification.

When producing images for merging, it is important to follow a pattern where the images can overlap each other as shown in Figure 16. This enables the editing software to find reference point, by which the images are stitched together. If the images appear very flat and lack details, a merging might not be possible due to absence of reference points.

Figures A1 and A3-A6 in the appendices feature merged images of samples produced in this work.

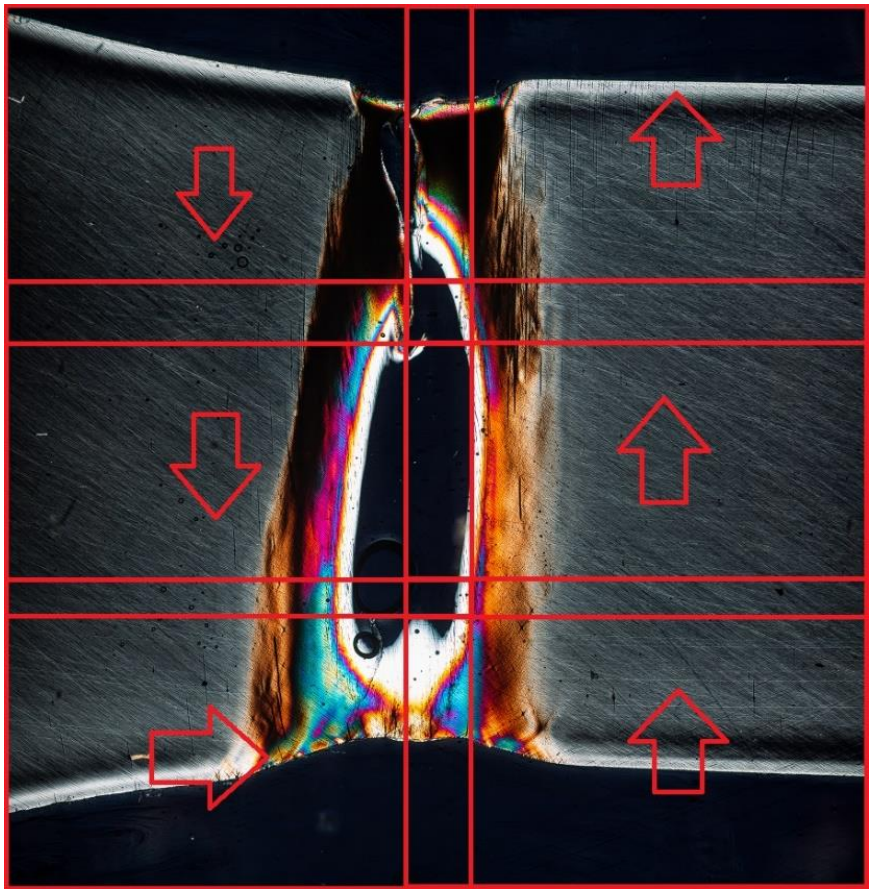


Figure 16: Merged image of tensile tested PLA sample, showing pattern and overlap of individual images.

4 EXPERIMENTAL RESULTS & ANALYSIS

4.1 Sample selection

Preparations of samples whether 3D printed or injection moulded was very similar. Challenges occurred generally in the embedding stage, since the sample shape made embedding difficult for the desired plane of observation to be obtained when polishing. Injection moulded samples were embedded in several different orientations for testing of both the preparation stages, and reveal of information during microscopy analysis.

Some broken tensile testing samples were also produced of both IM and 3D printed types. The samples were not embedded since the first attempt at preparation was successful, and showed no requirement of that for the specific samples. The samples had each obtained a major fracture, but were still partially connected as one piece.

4.2 Embedding

For embedding the West Systems 105 epoxy with 206 hardener was mixed in a 5:1 ratio by weight in a paper coffee cup, and then poured into separate cups containing the samples, placed in their desired position. Visually the resin featured a yellowish tint when cured. The tint was caused by the hardener, but a clear hardener could be purchased for a clear casting. The tint of the resin used for encasing was not of concern, since it would not be in the optical path during microscopy analysis.

4.2.1 Bubbles in resin

Since the curing of resin is an exothermic reaction, it may produce bubbles that get trapped within the resin. However, most bubbles were created during the mixing process of the epoxy and hardener. The bubbles generally managed to surface from within the resin as long as the viscosity was low, however that may only last for under 10-15 min after mixing, prior to the gel time.

When the bubbles in the resin transfer to the surface, the surface tension may prevent them from bursting. By heating up the resin surface with a gas torch the surface tension was broken and the bubbles were released to the atmosphere, leaving a bubble free casting for the sample.

4.2.2 Irregular and incomplete curing of epoxy resin

After examining the leftover resin in the mixing cup, the bottom of the cup often showed areas featuring incomplete curing of resin. That was likely the result of poor mixing of the epoxy and hardener in the area where the cups bottom and wall meets. To prevent similar events for castings including samples, no samples were embedded in the cup or container the mixing had taken place in.

In a particular case, PP samples were embedded and left to cure overnight, resulting in a curing time more sufficient than recommended by the manufacturer of the resin. After de-moulding, the castings showed softness which was not nominal for epoxy.

The components of the resin were mixed in the correct ratio by weight, but in a much smaller batch than in previous experiments. The West Systems resin is marketed for use in marine applications, therefore mixing of small volumes for experimental use may result in variating curing times. The encased samples were left to cure for another 24 hours and obtained their characteristic hardness.

4.2.3 Material bonding

Material embedded in this work include PP, UHMWPE, PC, PLA and PTFE. All materials, excluding PTFE, showed bonding with the epoxy that was more than sufficient for the intended use. The ease of using epoxy with its excellent properties and reasonable price, resulted in no other resin being used for this work.

PTFE showed poor results of bonding to epoxy, due to breaking out of the casting during the first polishing on three samples. The results were expected since the material has low friction characteristics and is softer than the other materials used, which leads to poor bonding and deformation during polishing. Due to bonding difficulties, PTFE was excluded because finding a solution was outside the scope of this study.

4.3 Cutting

The composite orientated blade for the band saw showed good performance for cutting the epoxy castings to fit the microscopy slides. Damage, in form of microscopic cracks resulted from cutting were not discovered on any samples during this study.

When cutting, the importance of producing chips was discovered. Cutting produces friction between the material and blade which results in heat being generated. The heat was removed from the area of cutting, by the chips produced from the cutting. If the cutting did not produce chips, it generally resulted in the material melting. If actions were not taken in time, the blade could have obtained damage due to overheating.

Chipping could be enhanced by feeding the material with greater speed, or switching for a rougher blade. As a general rule, hard and brittle materials should be cut with blades featuring small teeth with dense arrangement, while soft and tough materials require blades with large teeth and sparse arrangement.

When cutting samples with side cutters or scissors, the area on the sample where the cutting takes place may be subjected to some bending motion. This showed results in permanent re-orientation of molecules which were distinguished from the rest of the sample in microscopy analysis. The bottom of the sample illustrated in Figure 36 reveals a white area of such damage.

4.4 Cementing

The materials of the adhesives used in this study, showed little interference in microscopy analysis with cross polarisation due to their clarity and thin layer. The differences in their performance is more obvious in the polishing and cementing stages as explained below.

4.4.1 Cyanoacrylate

Favourable features of cyanoacrylate were quick curing time and clarity of material when cured. Cyanoacrylate has a low viscosity when applied and is not exothermic when curing. These features were key, regarding a bonding layer with minimal amount of trapped bubbles. Downsides of cyanoacrylate were apparent during polishing, since the cement often cracked around the samples, as illustrated in Figure 17, and let in water between the glass slide and specimen. During microscopic observation, the presence of water was highly disturbing and it also carried debris under the sample during polishing. The emerging of the cracks was most likely from the mechanical forces the polishing creates on the sample. The cyanoacrylate created a thinner bond than e.g. quick epoxy and seemed to be a brittle material for cementing samples.

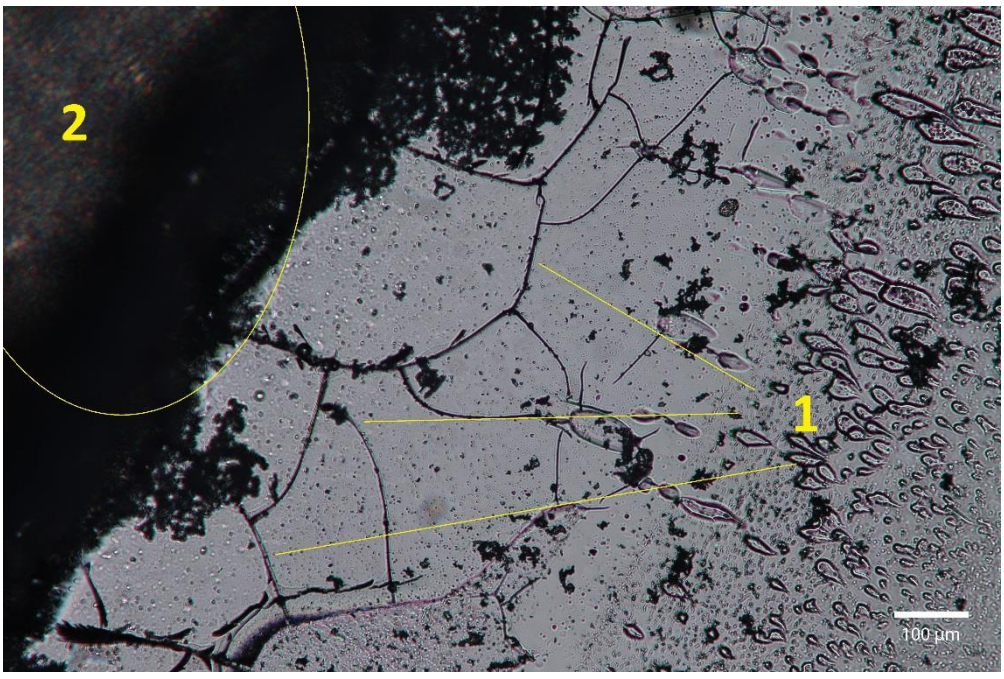


Figure 17: Microcracks in cyanoacrylate caused by polishing. 1) Microcracks 2) Sample, [10x magnification transmitted illumination]

4.4.2 Quick epoxy

Quick epoxy is a clear, strong and ductile material for cementing samples to glass slides. It showed to be resistant to cracking during polishing and isolated water from entering between the slide and sample as well. Quick epoxy is a two component adhesive containing a base and hardener. In comparison to cyanoacrylate, epoxy was a slower method since it required mixing in correct ratio before application, and had a longer curing time. More downsides were high viscosity and bubbles being trapped in the resin as a result of the mixing, which weakened the bond and caused minor disruption in the optical path. To reduce bubbles from building, the base was heated to 50 ºC which makes the resin less viscous for mixing. The base was heated in a baker filled with water for uniform and controlled heating as illustrated in Figure 18.

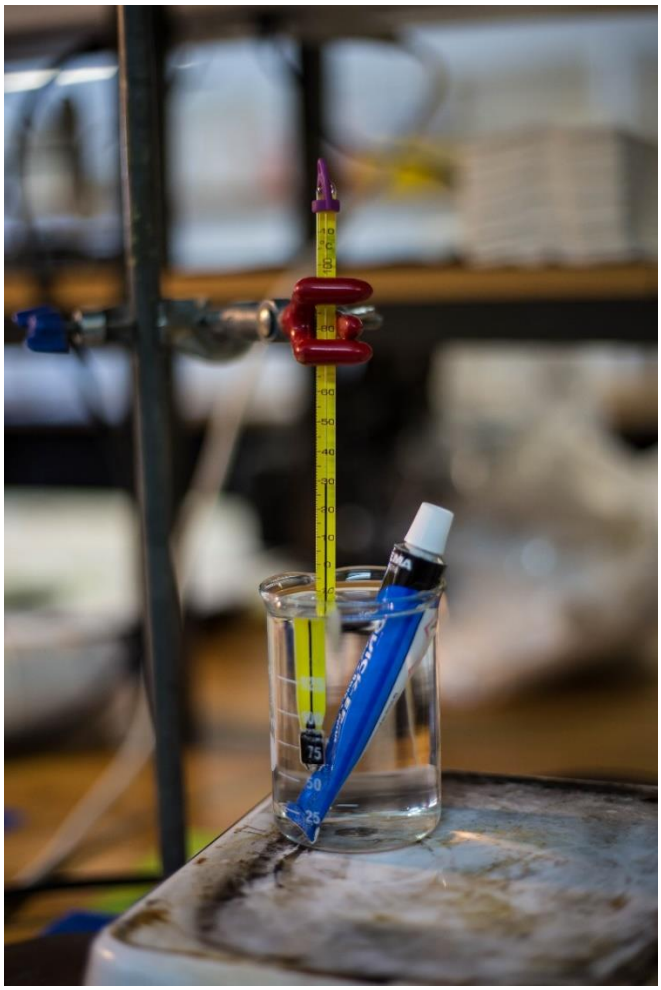


Figure 18: Heating of quick epoxy base in baker containing water, placed on hot plate.

Introducing the mixed epoxy to a vacuum removed bubbles resulted from the mixing, but also expanded smaller bubbles, which then became trapped when the resin began to cure. As a result, the vacuum was from there on excluded from the study.

The most effective method for reducing bubbles, besides the heating of the base, was to move the sample around in a small circular pattern on the epoxy, which had been applied to the glass slide, while pressing the sample down. The action pressed excess resin away from underneath the sample which created a thinner layer and carried bubbles away.

4.4.3 Epoxy

The West Systems epoxy did not display any meaningful benefit over the quick epoxy in microscopy analysis. Since the adhesive layer securing the sample to the slide was so thin, the slight difference in clarity between the two showed no visual difference during microscopy analysis.

Mixing of the components required more effort than the quick epoxy due to the unconventional mixing ratio, and the curing time of at least 9-12 h was excessively long for the cementing step.

4.4.4 Adhesive layer thickness

For the second polishing, it was important to know the thickness of the adhesive for compensation. If the sample was measured to be 50 µm after the second polishing, this value also included the adhesive used for cementing.
In an experiment, two glass slides measured to be 0.8–1.0 mm thick at three different points, were glued together and then measured at the same three points to calculate the thickness of the adhesive layer. The calculated average was 23.3 µm but the deviation in thickness between the measurement points were too great and therefore the result was deemed inaccurate. A side of the same sample was polished and viewed under a microscope. A picture was taken through the microscope and a photo of a scale taken with the same magnification in the same conditions, was placed over the photo with editing software, to determine the actual thickness of the adhesive layer. The result revealed the

thickness to be 30 µm consistently over the whole length of the layer as illustrated in Figure 19.

When cementing a handheld polished specimen the layer thickness will undoubtedly become thicker since the surface will not be as flush as a glass slide. A micrometre was used to observe the flushness of the surface of samples, which made it easier to estimate the amount to compensate. As a rule of thumb when polishing, a distance of 30-40 µm should be added to compensate for the adhesive.

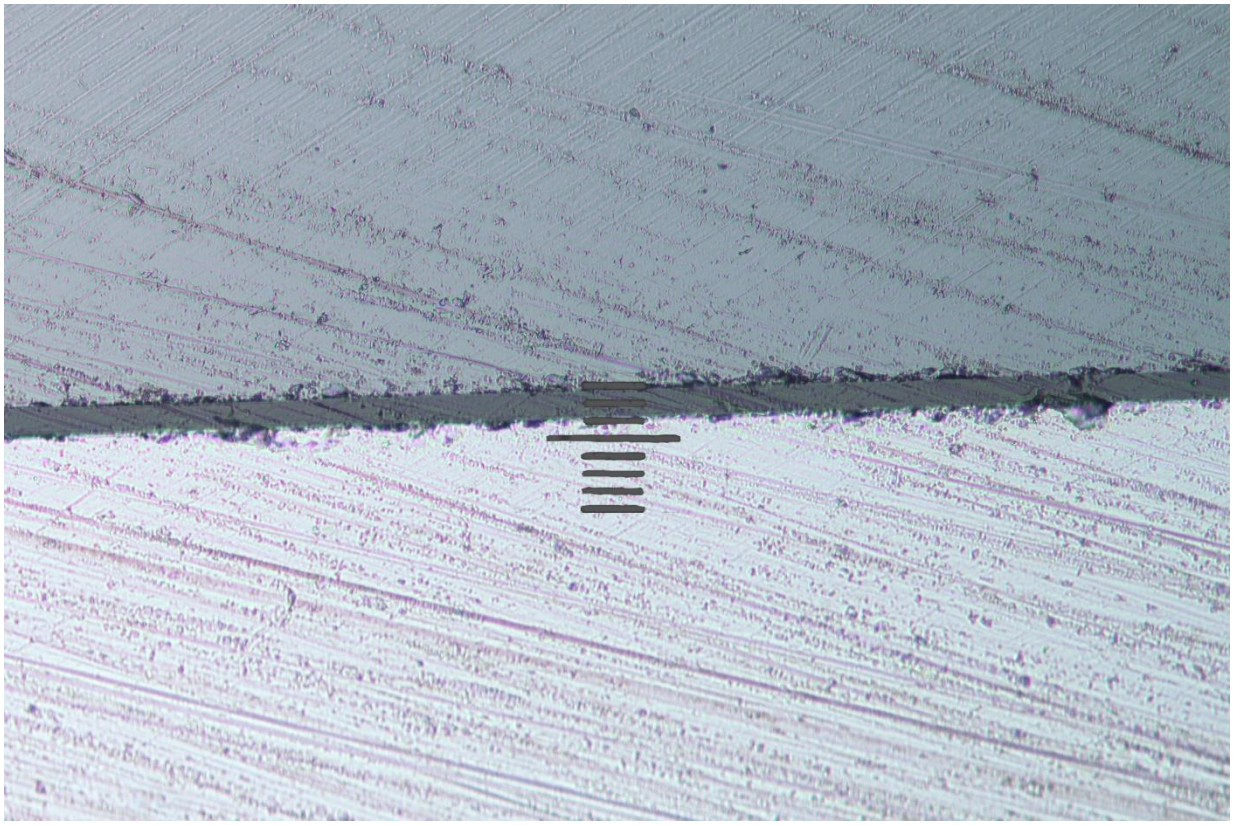


Figure 19: Two microscopy slides glued together with cyanoacrylate, resulting in a layer thickness of 30 µm. [20x magnification, reflected illumination]

4.5 Polishing

4.5.1 First polishing

For the first stage, the polishing was started with the roughest grain available (P320) for all samples, to plane the sample at the desired area for analysis in a short amount of time. Since the side polished in the first step will be cemented a microscopy slide, the surface was in early experiments fine polished with P600 grain to leave more area for the cement to adhere. It was also expected that the cement would fill in the small grooves left by the grain to erase any interference during analysis.

In later experiments, the first polishing was finished with P4000 silicon carbide grain, with expectation to a marginally clearer image for analysis. The results displayed great improvements in clarity compared to samples finished with P600 before cementing. Finer grain produces a finer finish since the grooves produced by the grains are smaller. When polishing with finer grit, flatness was easier to achieve since pressure can be adjusted to thicker areas without removing material too quickly.

4.5.2 Second Polishing

Like the first polishing, the second polishing started with P320 grain after which the polishing was gradually stepped up with finer grain. The second polishing was the most time consuming step in the process of producing thin sections, excluding the curing time of embedding resin and cement, since there are many factor to account for at one time, such as surface flushness, roughness, and sample thickness.

The second polishing of the first samples, took several days to successfully complete since fine grains were used to early, radically slowing down the removal of material from the sample. For later samples rough grain (P320-P600) was used until the thickness approached 300-400 µm. The grain was then switched for P1000, and pressure focused on thicker areas of the sample to obtain flushness of the surface. The method was similar to that of the first polishing, however, the thickness needed be cautioned or the sample could be destroyed. The flushness was checked by moving the sample under a micrometre in short intervals during polishing until the result was adequate.

On early samples the polishing was finished with P1200 grain since immersion oil was expected to fill in the tiny grooves left on the surface, leaving a clear optical path for observation. The expectation was accurate, but if the sample was finished with the fines grain available (P4000) there was no need for immersion oil, which simplified preparation and storage of samples. The P4000 grain finish displayed the clearest results, and was therefore used on all samples thereafter.

4.5.3 Grain

When switching from a worn out sandpaper for a fresh one, the difference in roughness could surprise the user, especially with rougher grain papers. A pristine P600 sandpaper may remove material quicker than a slightly worn P320 paper. When closing in on the final thickness during the second polishing, this needed to be accounted for.

4.5.4 Materials

The sample materials showed some differences during polishing. Soft materials, such as PTFE and PP produced large grains of debris during polishing, especially when using rougher grain paper.

Hard and brittle samples, such as PLA and PC produced much smaller grains and wore down more evenly, resulting in less effort to produce a flush surface. On the other hand, the harder materials wore down the grain on the paper quicker resulting in more waste.

UHMWPE is a very strong and ductile material with a great molecular weight. The strength is obtained by long polymer chains entangled around each other. Extruded samples of UHMWPE were polished, but showed very low wear and wore out polishing paper too quickly. Due to requiring too much time and resources, UHMWPE was excluded from the study.

4.6 Microscopy

4.6.1 Microscopy analysis of injection moulded samples

The injection moulded samples produced in this work, displayed information of good quality about the crystallinity which can be utilized to study material cooling in the mould.

The injection moulded samples were produced from tensile test pieces referred to as “dog bones”. Longitudinal and transverse cross sections were produced out of PP dog bones for microscopic observation as illustrated in Figure 21.

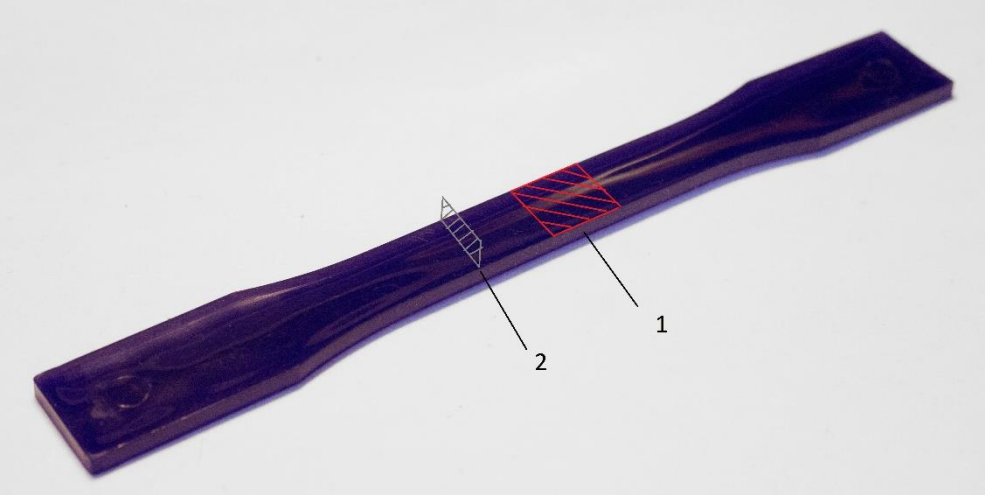


Figure 20: Injection moulded PP dog bone with the planes selected for samples. Depth of planes is not representative.

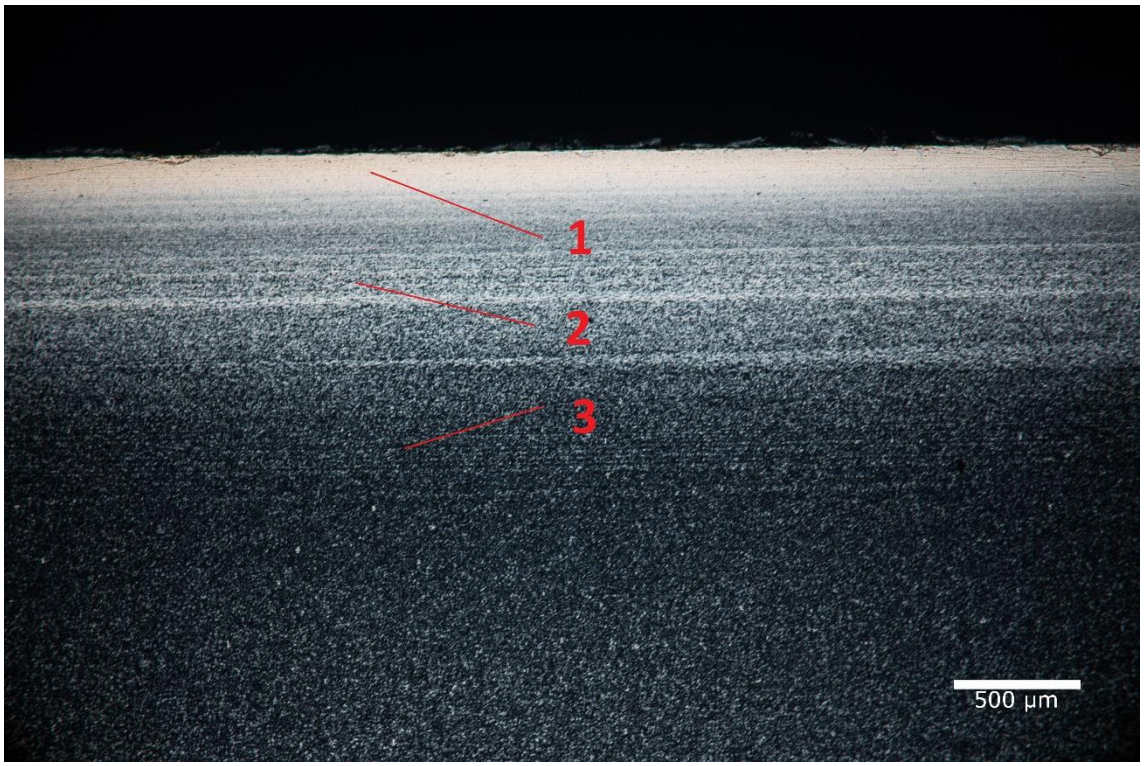


Figure 21: Thin section of plane 1 in Figure 21. 1) Rapidly cooled area 2) Moderately cooled area 3) Slowly cooled area [PP, 5x magnification, cross polarized transmitted illumination]

Figure 22 presents a longitudinal thin section from the top plane of an injection moulded PP dog bone, with a depth at approximately the middle of the sample. The bottom of the image shows a uniform melt of material which is nominal for good quality injection moulded parts. The line between the white and black area is the wall of the dog bone.

The white colour is caused by difference in crystallinity at the edge compared to the middle part. The crystallinity is the result of more rapid cooling at the edge than the middle due to contact with the mould wall.

Initial thoughts were that the contrast is caused by the sample being thinner at the edge due to uneven polishing, but since the contrast is uniform through the length of the sample the speculation was discarded.

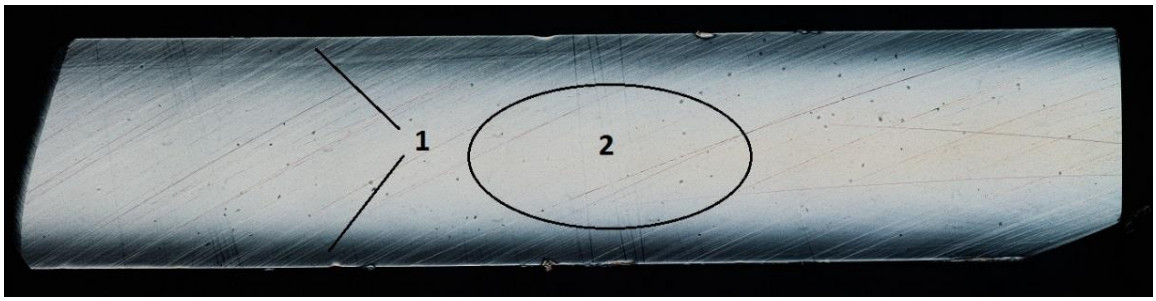


Figure 22: Thin section of plane 2 in Figure 21. 1) Amorphous structure 2) Semi-crystalline structure [PP, 5x magnification, cross polarized transmitted illumination, sample dimensions: 12.6 x 3.2 mm]

A merged image of the transverse plane 2 is presented in Figure 23. The cross section reveals more clearly the difference in crystallinity compared to Figure 22. Judging by the refraction of light caused by the sample, the core has obtained a semi-crystalline structure while the edges have an amorphous structure.

The darkness on the edges are produced by the lack of refraction the material causes on the light. Since the polarizers are placed in their extinction position, the more transparent areas appear darker.

Rapid cooling of injection moulded plastics results in a more amorphous molecular structure. The edges of the part are areas subjected to cooling first and as a result obtain a more amorphous structure than the crystalline or semi-crystalline core.

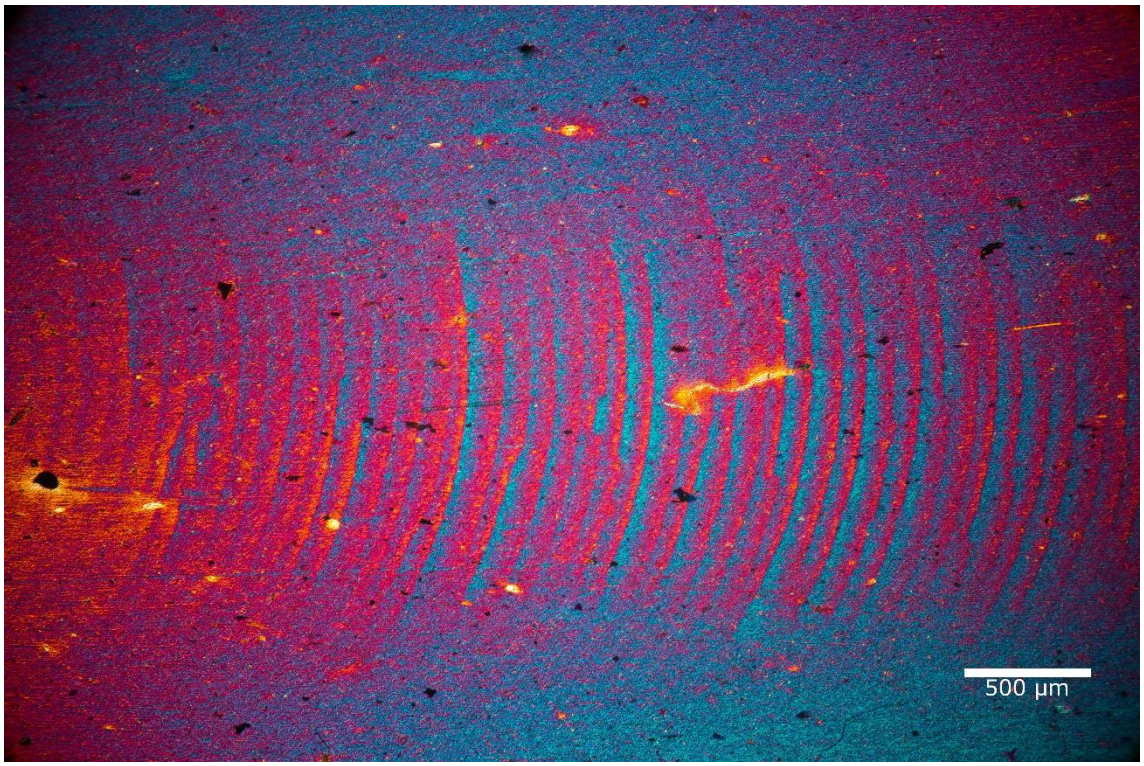


Figure 23: Thin-section of injection moulded polypropylene waste showing patterns of flow, due to molecular orientation. [PP waste, 5x magnification, cross polarized transmitted illumination]

In thin-sections of injection moulded parts, some of the orientations frozen into a part can be observed, such as melt-flow patterns shown in Figure 20.

4.6.2 Microscopy analysis of 3D printed samples

For initial testing of producing 3D printed samples, a block was 3D printed out of PLA and cut into two different cross sections shown in Figure 24. When reflecting on the literature research and results from the injection moulded samples, the 3D printed samples were expected to reveal distinguishable contrast where the print layers melt together.

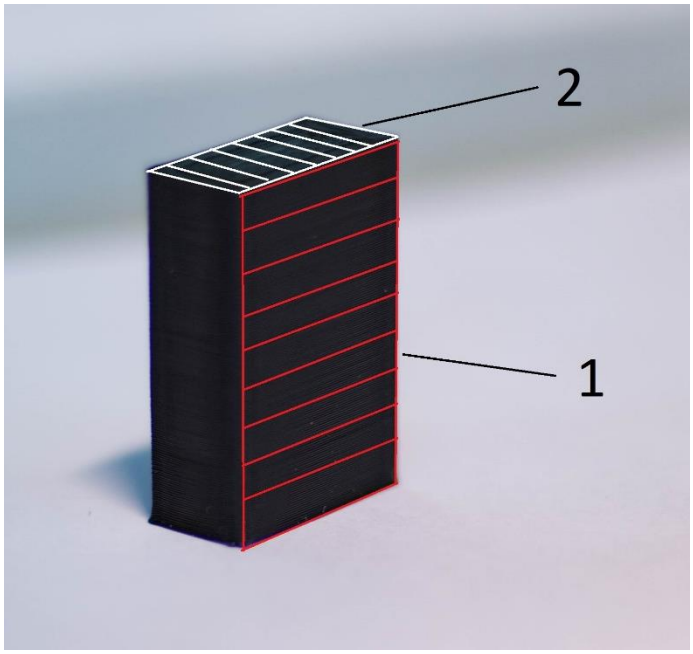


Figure 24: 3D printed block with planes of cross sections selected for samples. Depth of planes are not representative.

The block was printed in a concentric pattern from the bottom up. If viewed from a perspective perpendicular to plane 2, the printing nozzle started the pattern from the middle of the sample and move out towards the edges. Layer height was set as 0.2 mm on the printer with 100% fill.

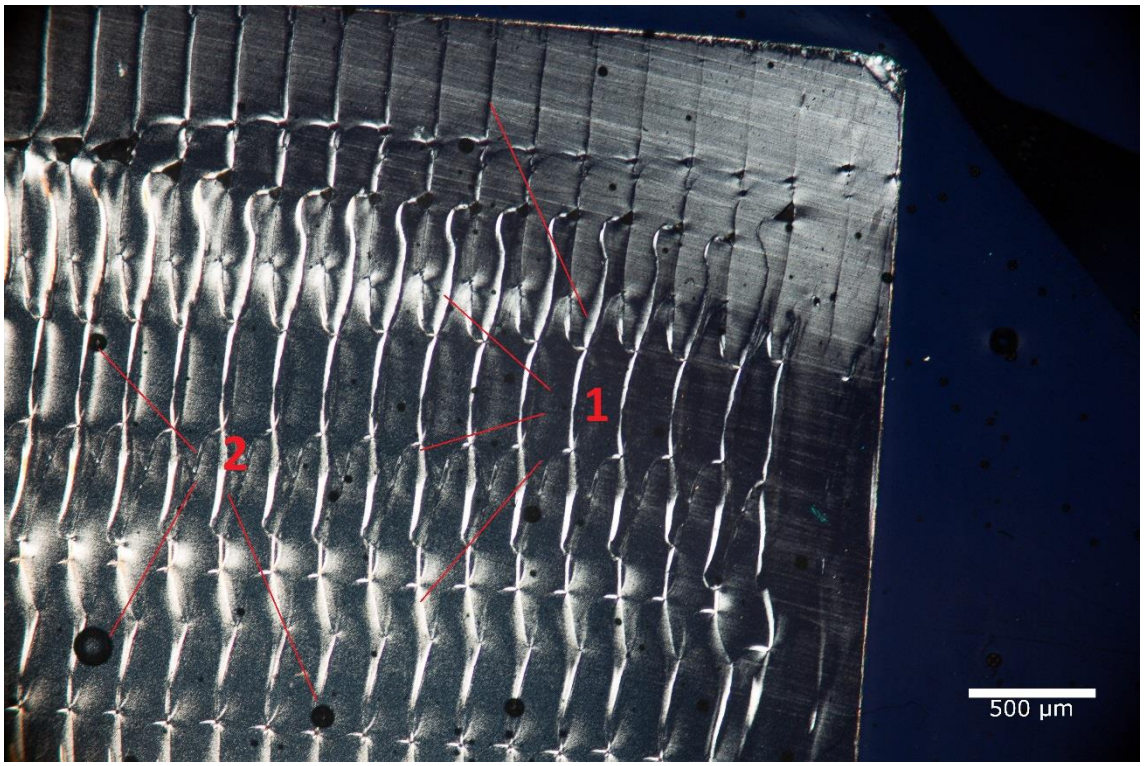


Figure 25: 1) Weld lines between layers 2) Air bubbles [PLA, 5x magnification, cross polarized transmitted illumination]

When observing the samples under crossed polarized light, the filament layers are clearly distinguishable and shows disruption and differences in crystallinity between the printed layers.

Figure 25 represents a thin section of plane 1 in Figure 24. The print layers can be easily distinguished since they feature refractions of light between each other. The refraction is likely caused from cooling of the layer before the next one has been applied. The pattern is essentially a form of weld line since two melts of material are fused together.

The round circular patterns in the image represent bubbles in the cementing resin and are not features in the sample.

Figure 26: Merged image of 3D printed PLA [PLA, 5x magnification, cross polarized transmitted illumination, sample height: 9.9 mm]

Figure 26 represents a cross section of plane 2 in Figure 24. The image has been merged together from multiple images to give greater context. The image reveals the concentric pattern by which the block has been printed including a void in the middle, which was not visible from the outside surfaces of the original part. The layers appear to have an even melt when moving in straight lines, but the turns feature disruptions of light which may be the result of internal stresses or inhomogeneous bonding of layers.

4.6.3 Polycarbonate samples

The samples produced out of extruded PC displayed no information under cross-polarized light. The result is due to PC being an amorphous material, which does not refract the polarized light. When observed through the microscope, the image displays a dark sample due to the extinction of the crossed polarizers.

4.6.4 Broken samples

The polishing method of producing thin section enables preparation of broken samples, which reveal information about stress concentrations and part failure. Stress concentrations are visible in form of cracks and by differences in the molecular orientation of the material, which can be detected with the aid of cross-polarized light. Broken tensile tested samples out of PLA prior to thin sectioning are presented in Figure 27.

Figure 27: 3D printed dog bone (left) and injection-moulded dog bone (right)

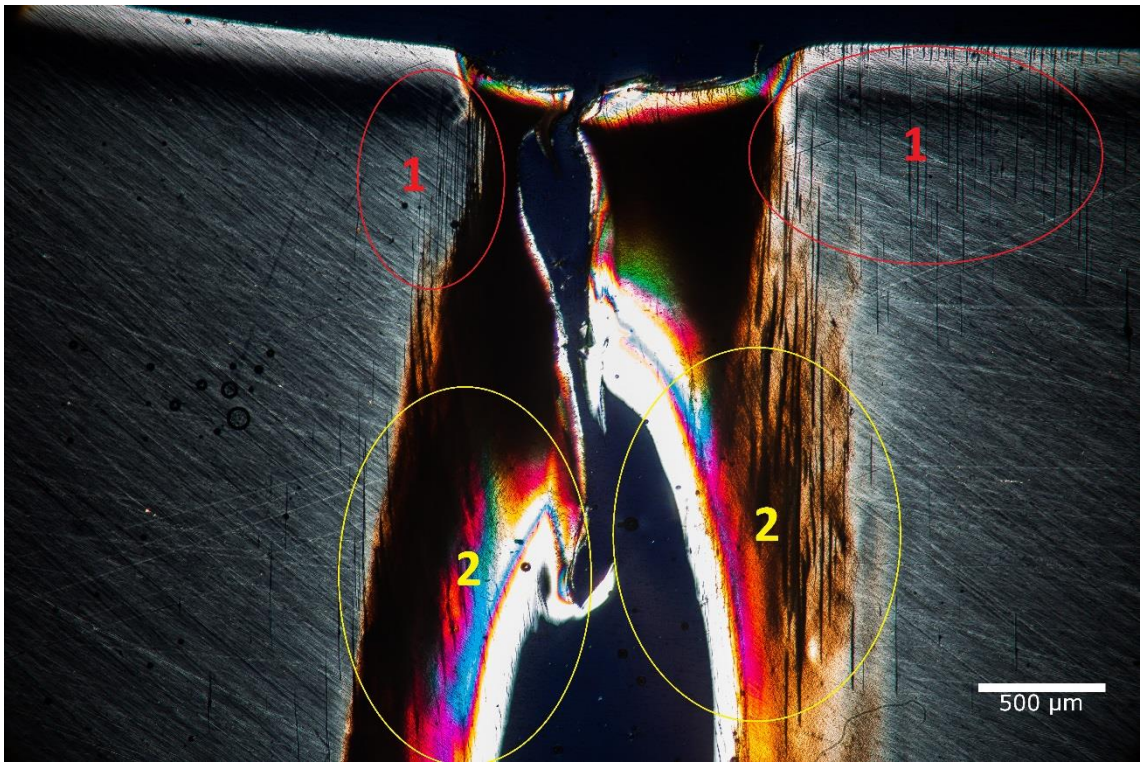


Figure 28: Tensile tested IM PLA. 1) Microcracks 2) Elongation damage [PLA, 5x magnification, cross polarized transmitted illumination]

Figure 28 presents a longitudinal thin section of an injection moulded dog bone out of PLA that has fractured because of tensile testing. The areas around the fracture displays different colours and shades. The different colours and shades are likely caused by differences in the molecular orientation and differences in thickness of the material, both results from the elongation during testing. Microscopic cracks can also be observed in the material further away from the fracture area.

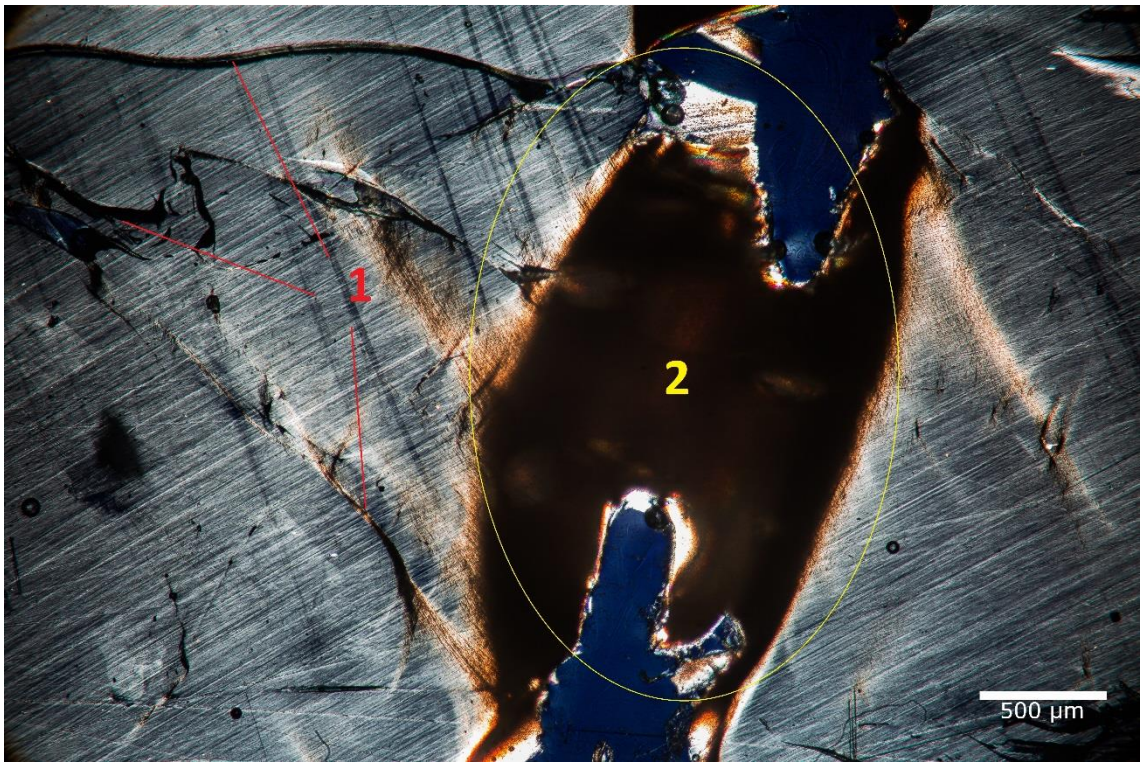


Figure 29: 3D printed, tensile tested PLA dog bone. 1) Weld lines from printing 2) Sample fracture area [PLA, 5x magnification, cross polarized transmitted illumination]

Figure 29 features a sample of the same material and background as the sample in Figure 28, with the exception of being 3D printed rather than injection moulded. The formation of the fracture seems to be more uneven than in the injection moulded sample, which could be due to weld lines created by filament layers of the printing. The weld lines may cause irregular distribution of the mechanical stresses in the sample during testing. This does not occur in injection moulded samples since the material is uniform and distributes stress more evenly. The weld lines in the sample can be seen as dark lines in Figure 29.

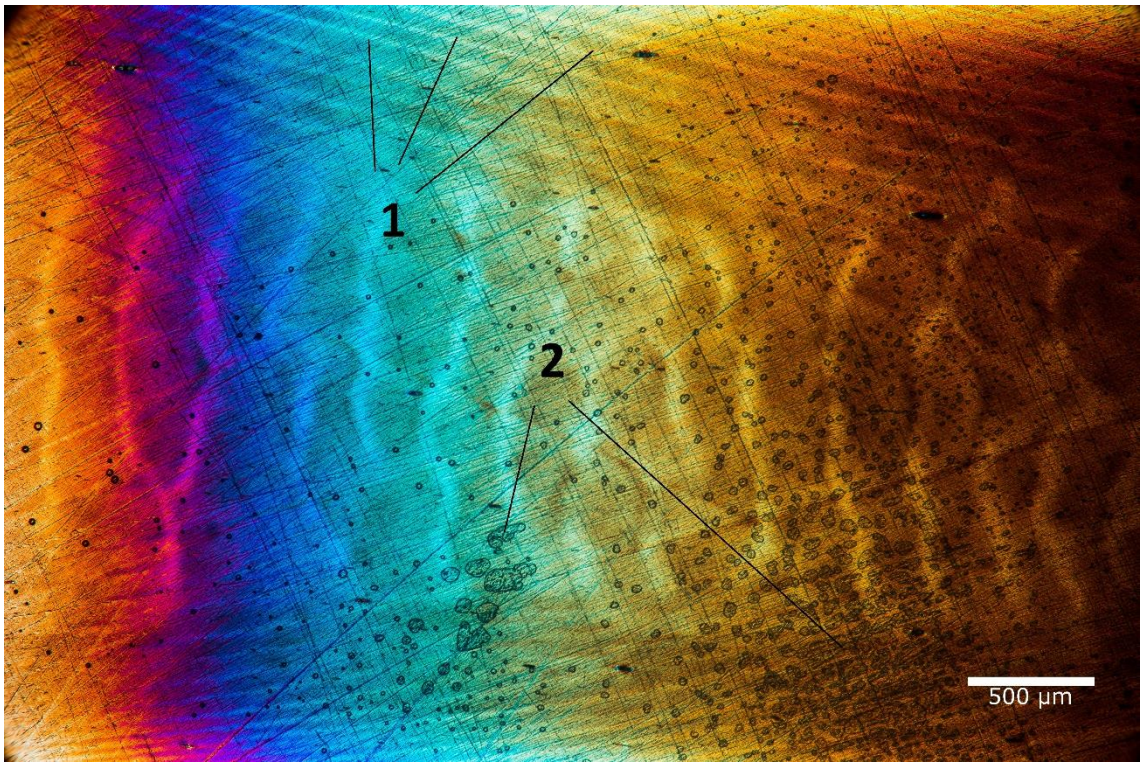


Figure 30: Elongated injection moulded dog bone 1) Patterns formed by tensile test 2) Contamination or bubbles in cement [PLA, 5x magnification, cross polarized transmitted illumination]

Figure 30 presents an injection moulded, tensile tested PLA dog bone very similar to the sample presented in Figure 28, however, the sample has stretched rather than fractured. Nominally, PLA is a brittle material, which fractures rather than stretches in tensile tests but since the sample featured in Figure 30 was produced out of PLA waste, the material probably obtained additives that gives it flexible properties. When thin sectioned and observed with microscope, the sample refracts the cross-polarized light into different colours and reveals patterns most likely form by the mechanical stresses of the stretching, since similar patterns were not discovered on samples that fractured. The resulting colours are particularly striking, since the original material was coloured black. The semi-transparent dots on the sample are likely bubbles or contamination embedded in the cementing resin.

5 CONCLUSIONS

The structure in this study has been a very straightforward process of following steps provided by literature. Since polishing of thin sections is a well-known method in geology science, it was expected that most of the procedures needed to be implemented from that field. However, literature review revealed that there are guides available on how to prepare specifically polymer thin sections. As a result, methods of the guides were utilized in this study rather than preparation steps of geology studies. Experiments conducted in this thesis showed varying results compared to the steps in literature, with some suggested methods providing poor results and other being unnecessary in common situations. For example, cyanoacrylate was suggested for cementing by some sources but in the experiments, all samples cemented with the cement in question partially detached from their microscopy slides during polishing and obtained water between the sample and slide. Embedding of samples in a resin was also suggested by several sources, but was deemed not necessary for samples with great surface area for cementing and polishing, therefore reducing the sample preparation time by 9-12 h.

Experiments with the sample cutting and preparation steps did not show any relevant contradiction with the procedures in literature, and neither did the polishing to some extent. The literature suggested that P1200 grain sandpaper was fine enough to finish the polishing, but when samples were finished polishing with P4000 grain in experiments, the surface finish was so great that application of immersion oil to the sample was not needed for microscopy analysis. As a result, the preparation time was reduced and sample storage and preservation time were lengthened. When polishing with fine grain sandpaper the water caused severe difficulties in controlling the sample which was not discussed in the literature. However, a solution was found during the experiments by reducing the water flow on to the sandpaper when polishing thin samples with fine grains.

The crossed polarizing filters are essential for studying polymer thin sections due to the contrast they produce. The polarizing filter used in this work are of the most basic grade, but provide adequate information. Developing of the filter holders for the microscope is of great importance for utilization of polymer thin section analysis at Arcada, due to their simple usability without sacrificing the modularity of the microscope. The filter holders are produced using only resources available at Arcada and can therefore be reproduced in the future if needed. The holders also provide possibility of using other than polarizing filters and feature room for future development.

The samples produced in this work successfully displayed a new perspective on polymer analysis on both injection moulded and 3D printed parts. This purpose of this study was mainly to develop a polishing method of producing thin sections at Arcada, rather than analysing them. As a result, the sample analysis was brief but will hopefully spark interest for further studies, especially concluding 3D printed parts.

5.1 Alternative methods for thin sectioning

In geology thin section are produces by the polishing method, since rocks cannot be cut through due to their hardness. In biology however, where the samples are of softer material a cutting device known as a microtome is used. A microtome is a device that can cut thin sections of samples with a specific thickness as thin as a few dozen microns. Microtoming can also be applied for polymers and produce samples for analysis in a short amount of time, but with some limiting factors. Material properties of the sample is a common limitation since hard materials can prove difficult to cut. A sample to be microtomed needs be secured in some way, and that could potentially deform the sample. The securing also limits planes that can be cut.

Polishing is a more time consuming methods since it includes many preparation steps in other for a successful result. However, because of more detailed preparation, and polishing with fine grains the resulting sample is generally of higher quality and reveal greater detail during microscopy.

5.2 Achievement of aims

The main aim in this thesis work was to successfully produce thin sections of polymeric samples of injection moulding and 3D printing background for optical microscopy analysis. Successful production of polymer thin sections was achieved in the early stage of experimenting, but discovery and development of cross polarized microscopy was more demanding but essential to complete the aim. During the several sample preparations conducted in this thesis, methods suggested by literature were tested but also successfully tailored to specific samples, improving time efficiency of the preparation and clarity of sample during analysis. Injection moulded and 3D printed samples produced in this thesis work were studied by microscopy, and interpret by the aid of literature, displaying differences in crystallinity, weld lines and other features resulting for from the respective production methods.

The final aim set for this thesis was to plan and conduct a project. The work has reflected the planning very well and successfully completed the aims. There have been several minor situations causing risk of failure of deviation of the original plans but solutions have been successfully engineered to overcome the problems. Discovering the need for crossed polarizers during analysis and developing a setup for the Arcada microscope was one important step, since analysis of thin sections with unpolarised light did not reveal the information needed.

5.3 Future recommendations

Once an experiment is completed, it answers questions but also creates more questions to be answered and more experiments to be conducted. That was certainly the case with this work and due to limited time, there are some still areas and methods that should be developed and tested in the future.

5.3.1 Circular polarization

In terms of sample illumination during microscopy, circular polarized light can produced with little effort by placing one-quarter wave retardation plate on the polarizer and another one below the analyser. The circular polarized light may illuminate some regions on the sample that become extinct with plane polarized light, therefore circularly polarized light may display more details of samples.

5.3.2 Polarizers

The polarizers used for this work are filters of educational grade and do not reflect the microscopes level of quality. Polarization can also be achieved by the use of prisms and potentially result in greater degree of polarization than that of filters. Higher degrees of polarisation could be useful to investigate further for improving results in the final image, and can be improved by simply upgrading for higher quality filters.

5.3.3 Mounting block for cementing

A device pushing the sample firmly to the slide during curing of the cement, would likely result in a bonding layer with more uniform thickness than having no weight on the sample at all. A uniform cement layer would aid in determining the sample thickness if relevant, and prevent possible interference of cement during analysis due to uneven thickness. The block may also aid in pushing bubbles away from underneath the sample, resulting in images with less interference and a stronger bond between the sample and slide.

5.3.4 Handle for polishing

For improving the polishing, a handle or mechanism for holding the sample could be developed to prevent the person polishing from cutting his/her fingernails or puncture the skin on the sandpaper. A handle with a firm grip could also aid in controlling the cutting of the sample and result in a flatter surface.

5.3.5 Analysis of fibre reinforced polymers and composites

Thin sectioning has been applied to fibre-reinforced polymer and composites before to some extent. In fibre reinforced polymers the alignment of fibres can be observed and give a visual interpretation of the strength and stiffness of the material. Similar features can also be observed on composites. Generally, the textile weave of composites are studied by reflected light but benefits of thin sectioning should be analysed in future studies.

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7 APPENDICES

This section feature microscopy images produced in this work that are mainly merged together to produce images with greater context. The images are placed in the appendix since placing them in the text would require cropping and missing of details. This section also contains the drawings for the polarizers produced in this work.

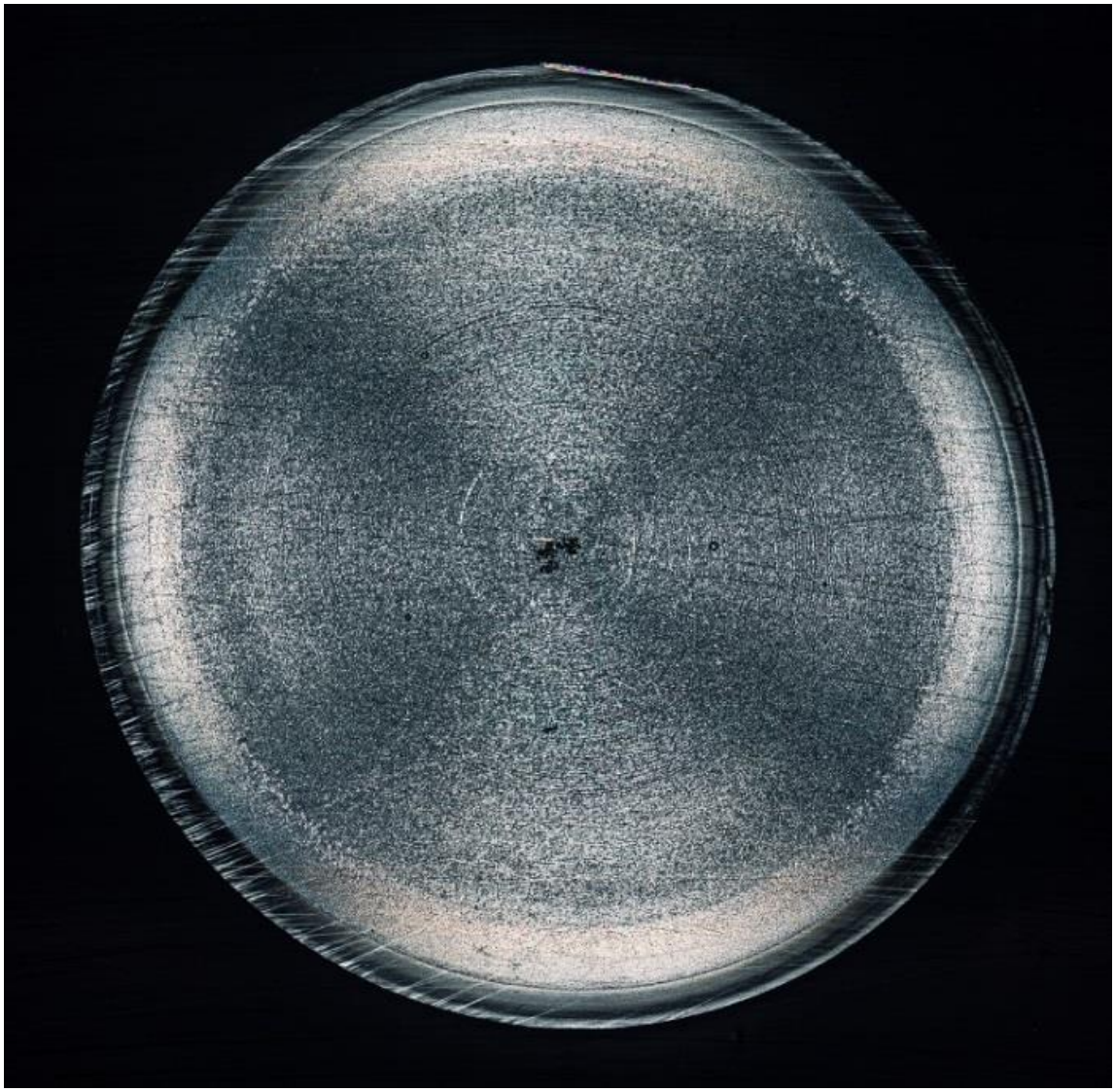


Figure A1: Merged image of injection moulded polypropylene thin section, of runner section in mould, showing differences in crystallisation. [PP, 5x magnification, cross polarized transmitted illumination, sample diameter: 5.5 mm]

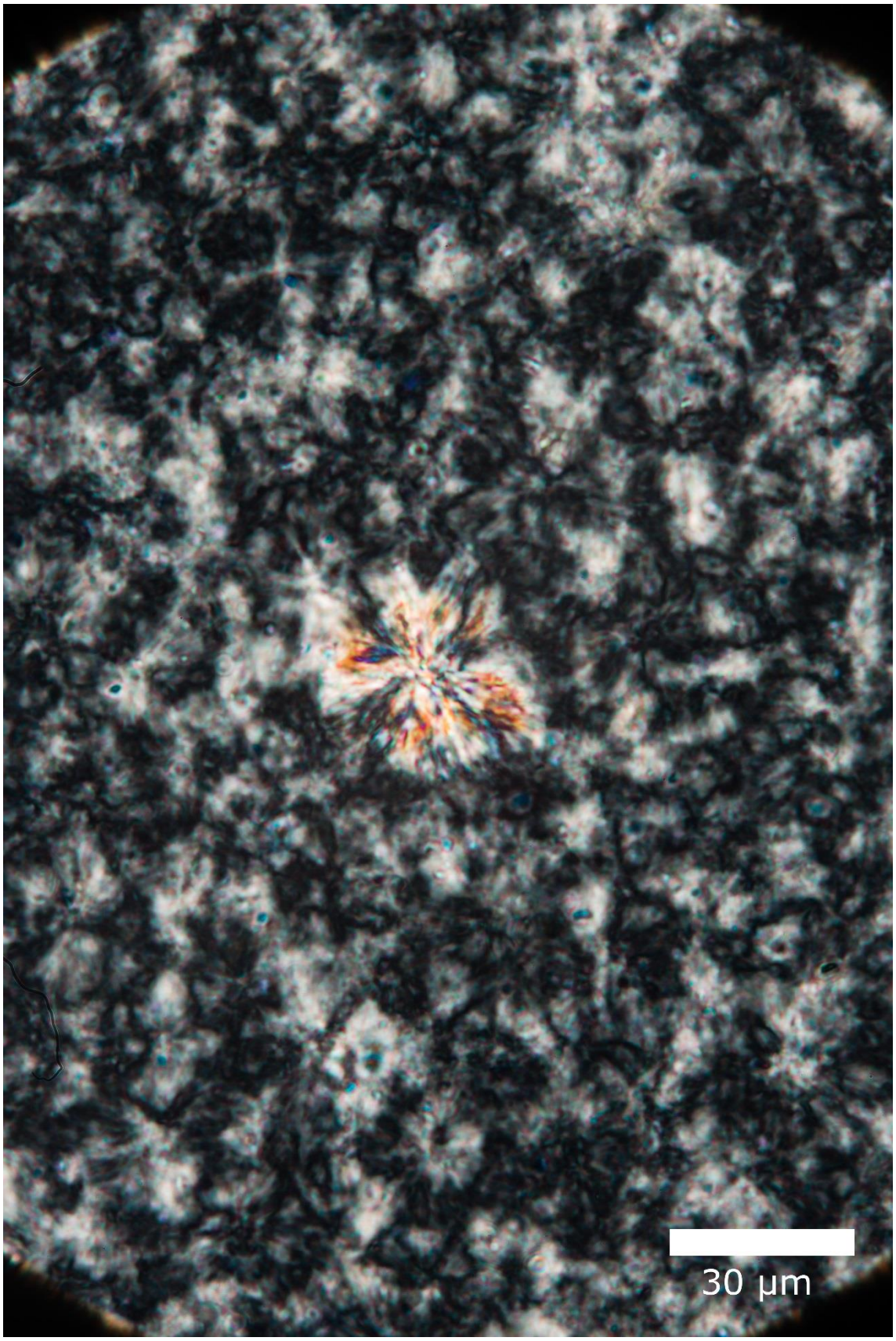


Figure A2: Spherulites in polypropylene sample. [PP, 100x magnification, cross polarized transmitted illumination].

Figure A3: Merged image of fracture area on tensile tested piece out of 3D printed PLA. [PLA, 5x magnification, cross polarized transmitted illumination, sample height: 5.2 mm]

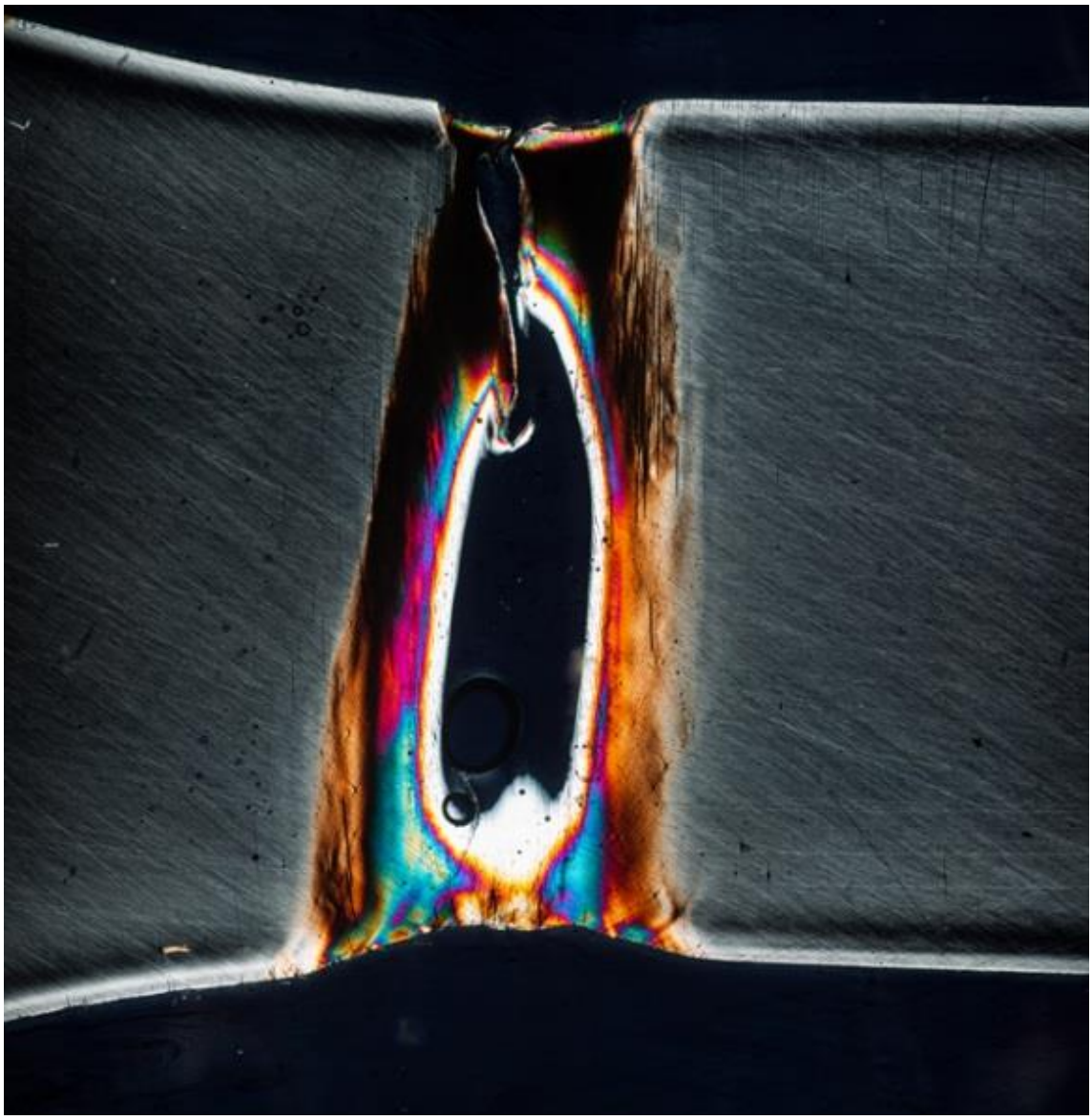


Figure A4: Merged image of fracture in tensile tested piece of injection moulded PLA. [PLA, 5x magnification, cross polarized transmitted illumination, sample height: 5.7 mm]

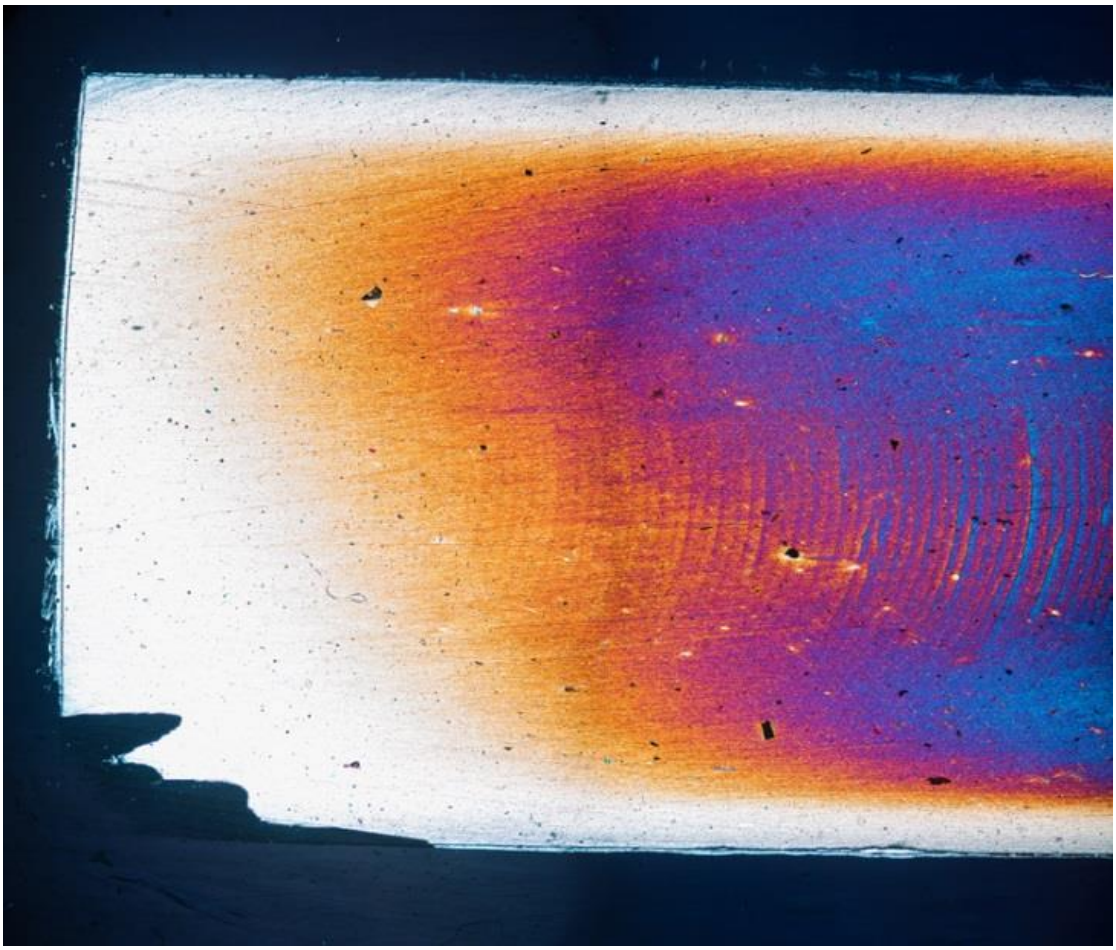


Figure A5: Merged image of injection moulded PP waste sample. [PP, 5x magnification, cross polarized transmitted illumination, sample height: 5.5 mm]

Figure A6: Merged image of tensile tested, 3D printed PLA sample. [PLA, 5x magnification, cross polarized transmitted illumination, sample dimensions: 13.7 x 5.4 mm]

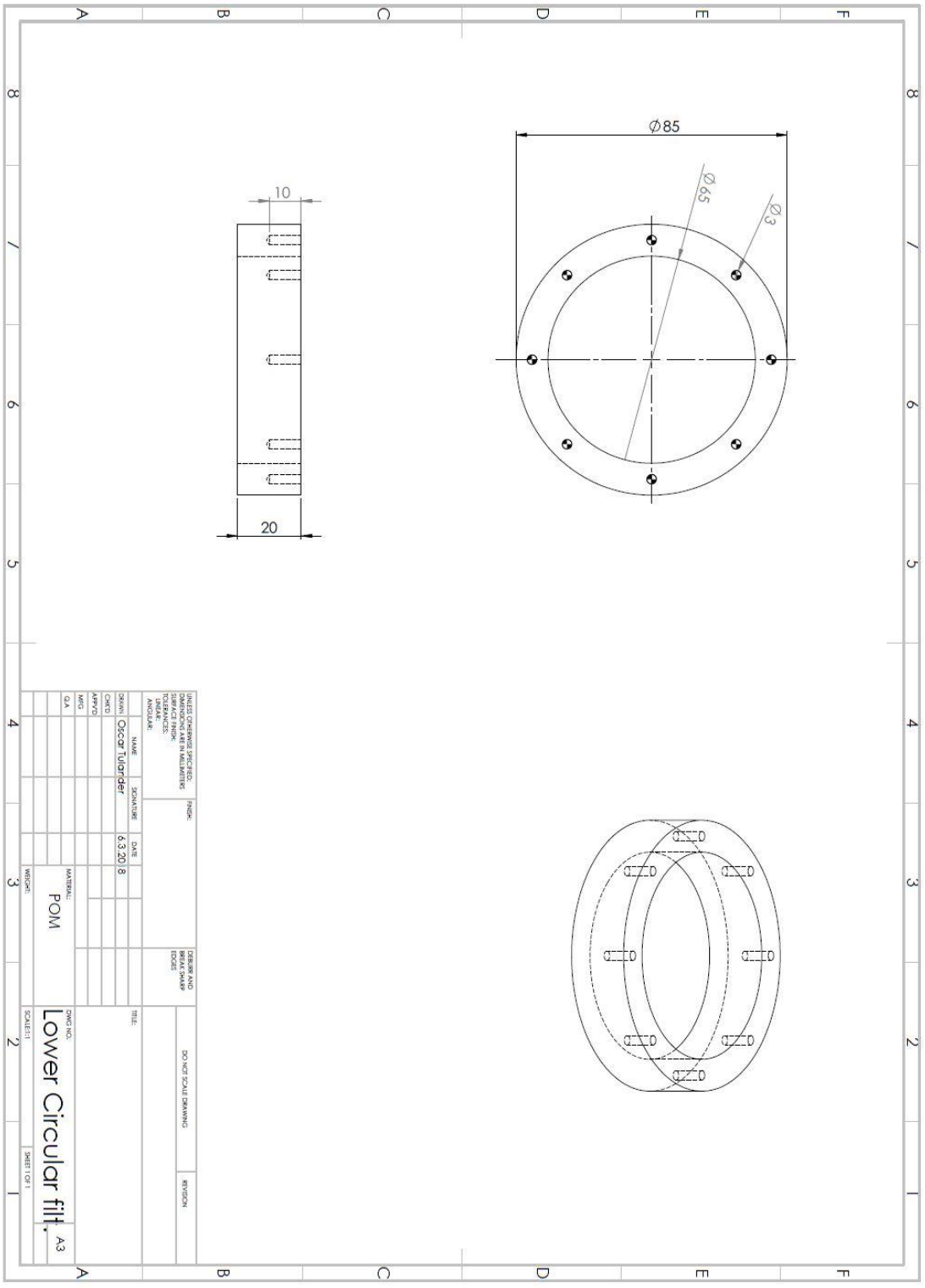


Figure A7: Polarizer lower part drawing

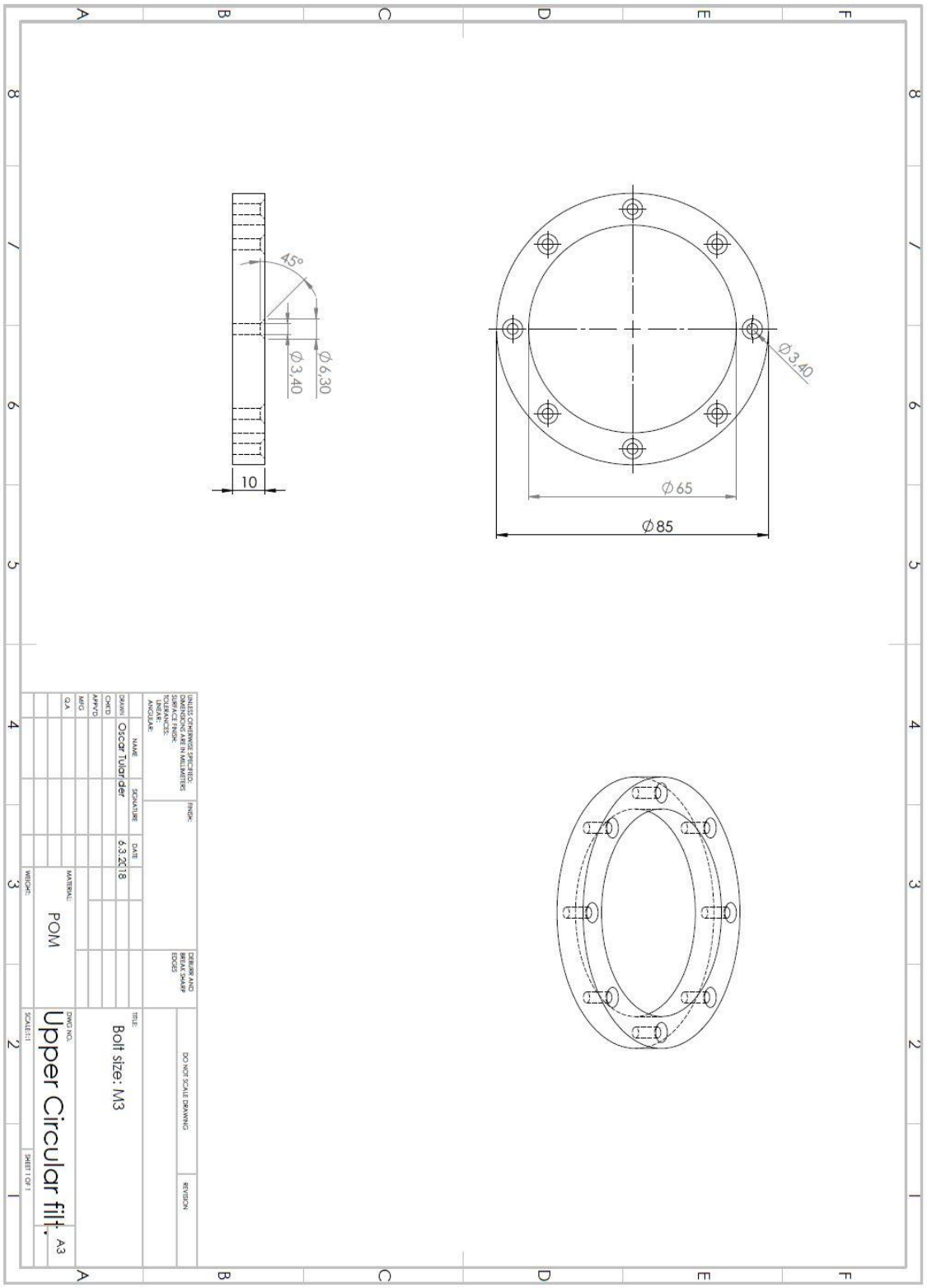


Figure A8: Polarizer upper part drawing

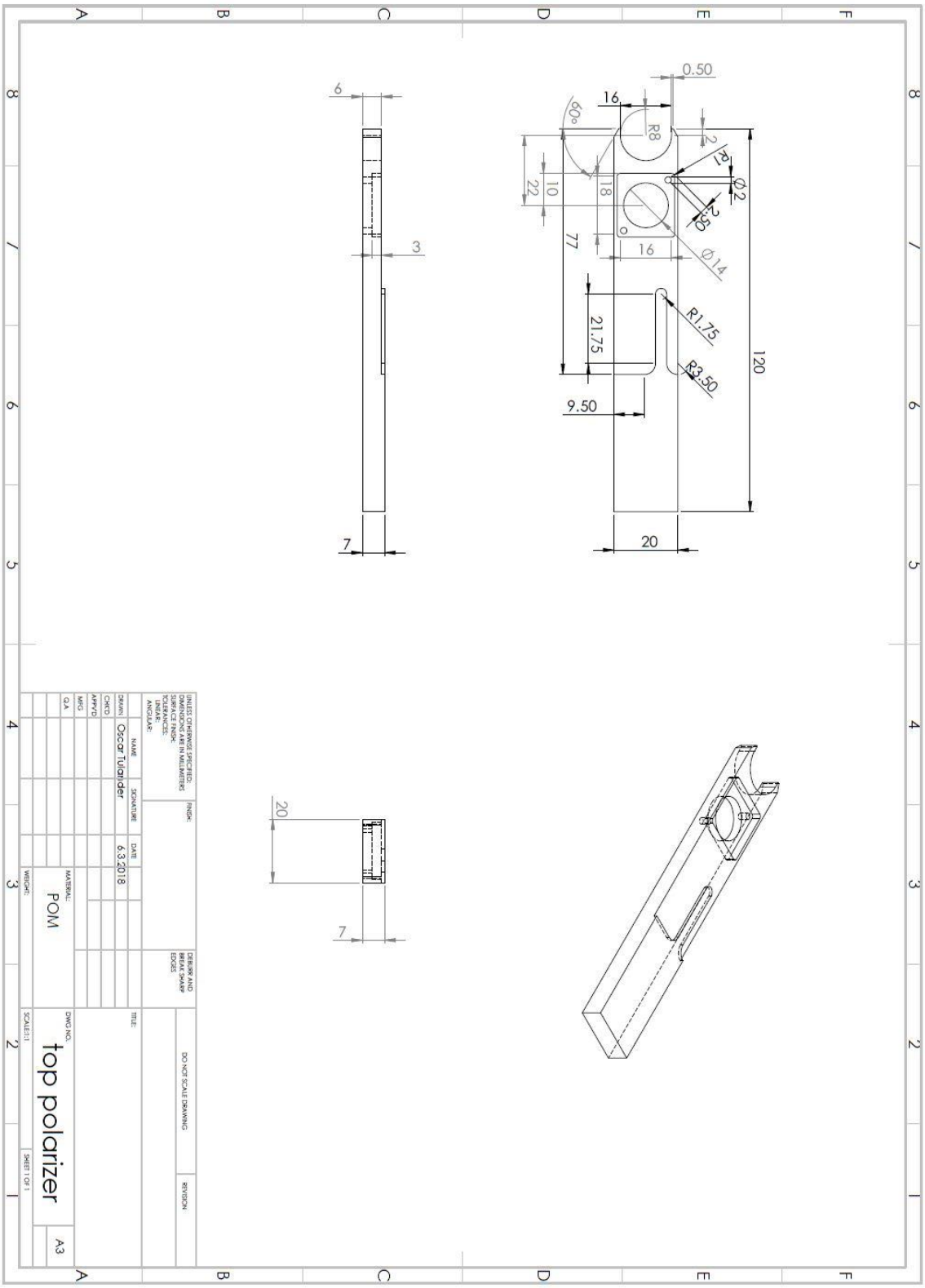


Figure A9: Analyser drawing

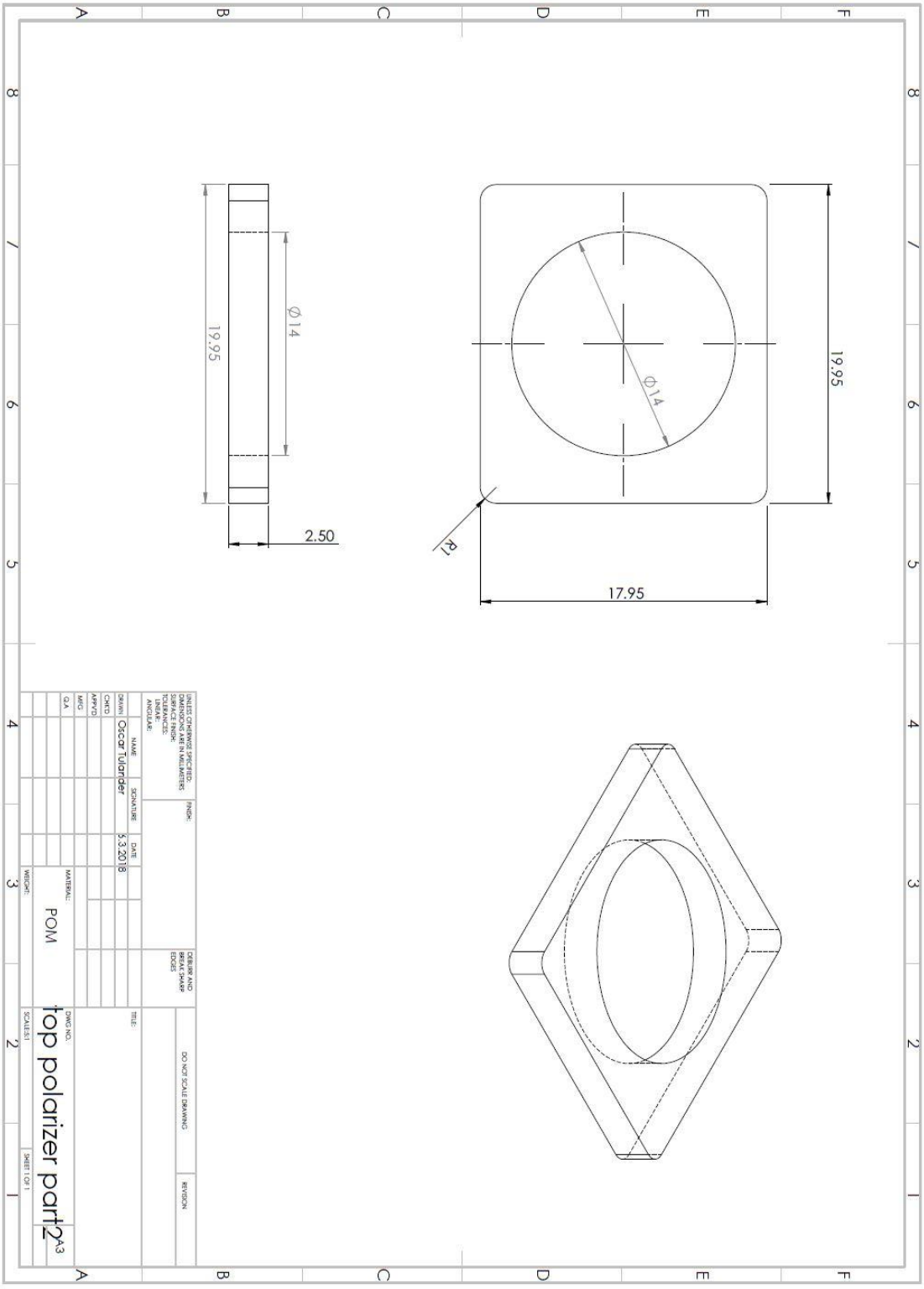


Figure A10: Analyser lid drawing

8 SAMMANDRAG

8.1 Introduktion

Tunn tvärsnittsanalys är en vanlig metod inom biologi, metallurgi och geologi, därför kan kunskap från dessa områden utnyttjas vid framställning av polymera tvärsnitt. Produktionen av polerade provbitar och mikroskopisk analys med tvärpolariserat ljus är mycket likartat inom plastteknik och geologi. Inom geologin möjliggör denna metod att forskare kan studera kristallformer och symmetri i stenar av olika slag.

Tunna tvärsnitt har använts i analys av plaster och polymerer för att studera fel, smältflöde, svetslinjer och molekylär struktur av formsprutade delar, men litteraturgenomgången visar att det inte har publicerats litteratur om 3D-utskrivna applikationer. 3D-utskrift är för närvarande en relativt ny tillverkningsprocess, vilket möjligen är en orsak till att de två inte har mötts.

Syftet med detta examensarbete är att utnyttja metoder för tunn tvärsnittning från geologi och tillämpa det på formsprutade och 3D-tryckta polymerer. Formsprutning och 3D-utskrift av polymerer är bearbetningsmetoder som utnyttjas på avancerad nivå i Arcada. Tunn sektionsanalys är en mycket värdefull metod av polymeranalys för skolan eftersom den ger strukturell information om delar och material på mikroskopisk nivå som annars är osynlig för blotta ögat, och denna metod kan inspirera till nya studier och experiment. Största delen av verktygen och utrustningen som behövs för att producera tunna tvärsnitt är redan tillgängliga på Arcada, och med lite utredning kan skolans beredskap för analysering utvidgas mot tunn tvärsnittsanalys.

Huvudsyftet med detta examensarbete är att framgångsrikt producera provbitar av plastdelar för optisk mikroskopianalys genom att implementera metoder från geologi och annat område där metoden redan används.

8.2 Definition

Ett tunt tvärsnitt är ett tvärsnitt av ett föremål vilket är tillräckligt tunt för ljus att tränga sig igenom och kan därför analyseras med optiskt ljusmikroskop. Det finns ingen specifik tjocklek som tvärsnittet bör vara, snarare beror tjockleken på materialet och dess genomskinlighet. Tjockleken på tvärsnittet reglerar också visuella egenskaper som ses genom ett optiskt mikroskop och är därför specifikt för varje provbit.

8.3 Förberedning av provbitar

När provbiten blivit vald börjar förberedelsen genom att välja ett tvärsnitt eller ett plan där provbiten kommer att avslöja nyttig information. Förberedningssteg som t.ex. inbäddning eller skärning kan eventuellt hoppas över beroende på provbiten. Till skillnad från skärning av tunna tvärsnitt kan polering producera provbitar i komplicerade plan eftersom provbiten inte behöver spännas fast för skärning, men kan istället inbäddas i stödjande harts för polering. Begränsande materialegenskaper i provbitar för polering är dålig vidhäftning till inbäddningsharts eller lim och seghet för slitage. Polering är en skärningsmetod som avför små mängder av material på en gång vilket kan kräva överdrivet mycket tid och resurser om materialet som ska poleras är slitstarkt.

Förberedningsstegen efter att provbiten är vald är de följande:

- Inbäddning
- Bortskärning av överskridande harts
- Första poleringen
- Limning till glasskiva
- Andra poleringen
- Rengöring och förberedning för analys

8.4 Inbäddning

Syftet med inbäddningsskedet är att ge provbiten en större yta för limning och polering, och även hålla provbiten upprätt om den valda ytan är komplicerad att polera.

Inbäddning av provbiten bör göras i harts med stark och god vidhäftning till provbiten för att skapa ett hölje som inte kommer att lossna under polering. Epoxi och polyester är bra allmänna alternativ men andra hartser med bra bindningsegenskaper för den specifika provbiten kan också väljas. Härdningen av harts är en exotermisk reaktion som kan producera gasbubblor som kan fastna i hartset då det härdar. Bubblorna kan leda till att provbiten lossnar från sitt hartshölje på grund av de mekaniska krafter som produceras under polering. Harts med hög krymphastighet är också benägna att lösa provbiten från sitt hölje under polering. Vid förberedelse av ihåliga provbitar behöver inbäddningsprocessen bra planering för att förhindra kaviteter från att bildas. En spruta kan användas för att spruta in harts i provbitens kaviteter före inbäddning.

Inbäddning är inte en nödvändighet i alla situationer. Om provbiten som skall framställas har en relativt stor yta på både limnings- och poleringsytan kan inbäddningssteget hoppas över eftersom limmet kommer att ha en tillräcklig yta för bindning och de mekaniska krafterna under polering fördelas jämt över ett stort område. Om ytan är mycket liten eller provstycket behöver poleras i upprättningsposition för önskat tvärsnitt, ger ett hölje sidostöd och fördelar kraften på ett större område under polering. Detta bidrar också till att kontrollera skärningshastighet och tjocklek genom att skapa en flat yta på provstycket under polering.

8.5 Bortsågning av överskridande harts

Beroende på gjutformen för inbäddning kan det vara önskvärt att avlägsna överskott av harts från gjutningen före polering och limning. Sågandet kan möjligtvis skada provstycket i en skala som inte är synlig för blotta ögat, t.ex. genom att producera mikroskopiska sprickor. Sprickorna kan felaktigt observeras som sprickor eller tomrum som egenskaper i provstycket. Vidare kan vibrationer från sågandet av hartset få provstycket att lossna från sitt hartshölje. För att minimera sannolikheten för sådana sprickor, rekommenderas det att såga med gått avstånd från provstycket. Under experimenten uppkom inga ifrågavarande sprickor eller annan skada på provbitarna p.g.a. sågande.

8.6 Polering och limning

Ytan som är limmad till glasskivan bör ha en bra och klar yta för att ge giltig information under mikroskopet, därför bör poleringen ske i två steg, oavsett om inbäddning är nödvändigt eller inte. Den första poleringen förbereder provstycket för limning, medan provet skärs till sin slutliga tjocklek i det andra poleringssteget.

All polering och slipning av provstycken bör göras vått. Vatten fungerar som kylvätska och smörjmedel för provstycken under slipning och bär även skräp efter slipningen bort från sandpappret.

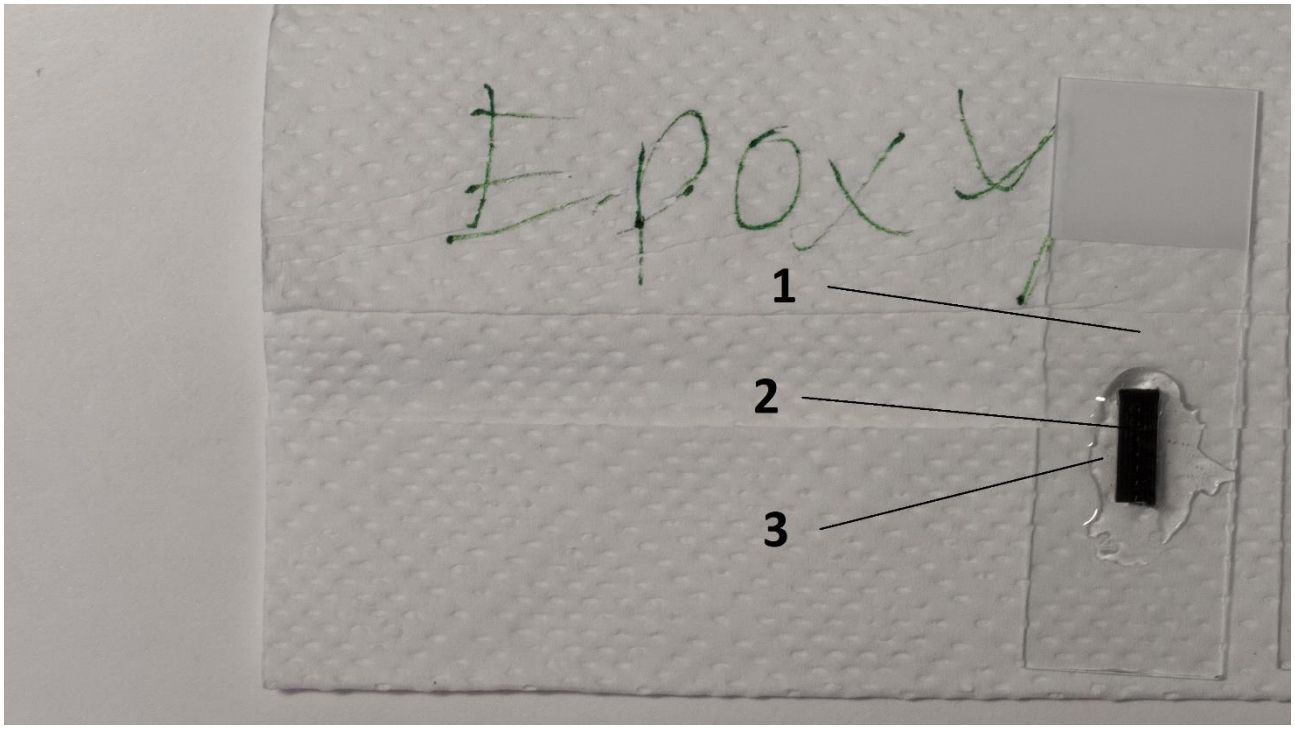
8.7 Första poleringen

För det första steget startades poleringen med det grovaste sandpappret (P320) för alla provbitar, för att plana ut bitarna på den yta som skall analyseras. Eftersom sidan av bitarna som poleras först, kommer att limmas till en glasskiva, var ytan i tidiga experiment finpolerade med P600 sandpapper för att lämna en relativt grov yta åt limmet att vidhäfta. Det förväntades också att limmet skulle fylla i de små skråmorna som sandpappret lämnat efter och radera eventuella störningar som kan uppkomma under analyseringen.

I senare experiment avslutades den första poleringen med P4000 kiselkarbid sandpapper med förväntan på en marginellt tydligare bild för analys. Resultaten visade stora förbättringar i klarhet jämfört med stycken som finslipats med P600 sandpapper före limning. Finare korn ger en finare yta eftersom spåren som produceras av kornen är mindre. Vid polering med finare grovhet var planheten av ytan lättare att uppnå eftersom tryck kan anpassas till tjockare områden med liten skärningshastighet.

8.8 Limning

Innan den andra poleringen monteras provstycket på en glasskiva som visas i Figur 1. De önskade egenskaperna i limmet är bra bindning med glas och materialet av provstycket och dessutom genomskinlighet. Genomskinlighet och klarhet minskar störningar när man analyserar stycket med tvärpolariserat ljus och är därför av stor betydelse. Ultraljudstvätt av både provstycket och den glasskivan rekommenderas för att avlägsna lösa partiklar från ytorna för en starkare bindning. Tvättningen avlägsnar också små partiklar från poleringen som under mikroskopanalys kan misstolkas som defekter i provstycket. Före limningen bör reporna från den första poleringen peka i samma riktning ifall de är synliga, för att undvika misstolkning under analysen.



Figur 1: Provstycket limmat till glasskiva med snabbepoxi. 1) Glasskiva 2) Provstycke 3) Epoxi

Tre olika lim provades med olika resultat. Cyanoacrylat (snabblim) hade en kort härdningstid och klar genomskinlighet efter härdning. Cyanoakrylat har låg viskositet och är inte exoterm vid härdning. Dessa egenskaper var viktiga när det gällde ett skapa bindningsskikt med minimal mängd bubblor. Nackdelen med cyanoakrylat var uppenbar under polering, eftersom limmet ofta sprack runt provstyckena och släppte in vatten mellan glasskenan och provstycket.

Snabb epoxi är ett klart och starkt material för limning av provstycken till glasskivor. Det visade sig vara motståndskraftigt mot sprickbildning under polering och isolerade vatten från att tränga in mellan glasskivan och provstyckena. Snabb epoxi är ett lim som består av två komponenter, en bas och härdare. Jämfört med cyanoakrylat var epoxi en långsammare metod eftersom komponenterna krävde blandning i korrekt förhållande före limmandet och hade en längre härdningstid. Flera nackdelar var hög viskositet och bubblor i hartset som skapades av blandningen av komponenterna. För att minska bildningen av bubblor uppvärmdes basenkomponenten till 50 ºC vilket sänkte viskositeten.

West Systems epoxin som användes för inbäddning visade inte någon fördel gentemot snabb epoxi eftersom limskiktet var så tunt var det ingen skillnad i klarhet mellan de två under mikroskopianalysen.

Blandning av komponenterna krävde mer ansträngning än snabb epoxi på grund av det okonventionella blandningsförhållandet, och härdningstiden av åtminstone 9-12 timmar var alltför lång för limningssteget.

8.9 Andra poleringen

Likasom den första poleringen började den andra poleringen med grovt P320 sandpapper, varefter poleringen gradvis ökades till finare papper. De första styckena var finslipade med P1200 sandpapper eftersom immersionsoljan som senare skitades på stycket förväntades fylla i de små reporna efter poleringen. Förväntan var korrekt, men när stycken senare finslipades med det finaste tillgängliga sandpappret (P4000) var det inte nödvändigt att använda immersionsolja, vilket förenklade förberedelse och lagring av provbitar.

8.10 Rengöring och förberedning för analys

Efter polering finns det små skräp av mikroskopisk storlek på provstycken. Torkandet av stycken med ett blött papper kan leda till repor på den finpolerade ytan, därför bör styckena sättas i ett ultraljudsbad i en bägare med destillerat vatten för 30 s eller längre.

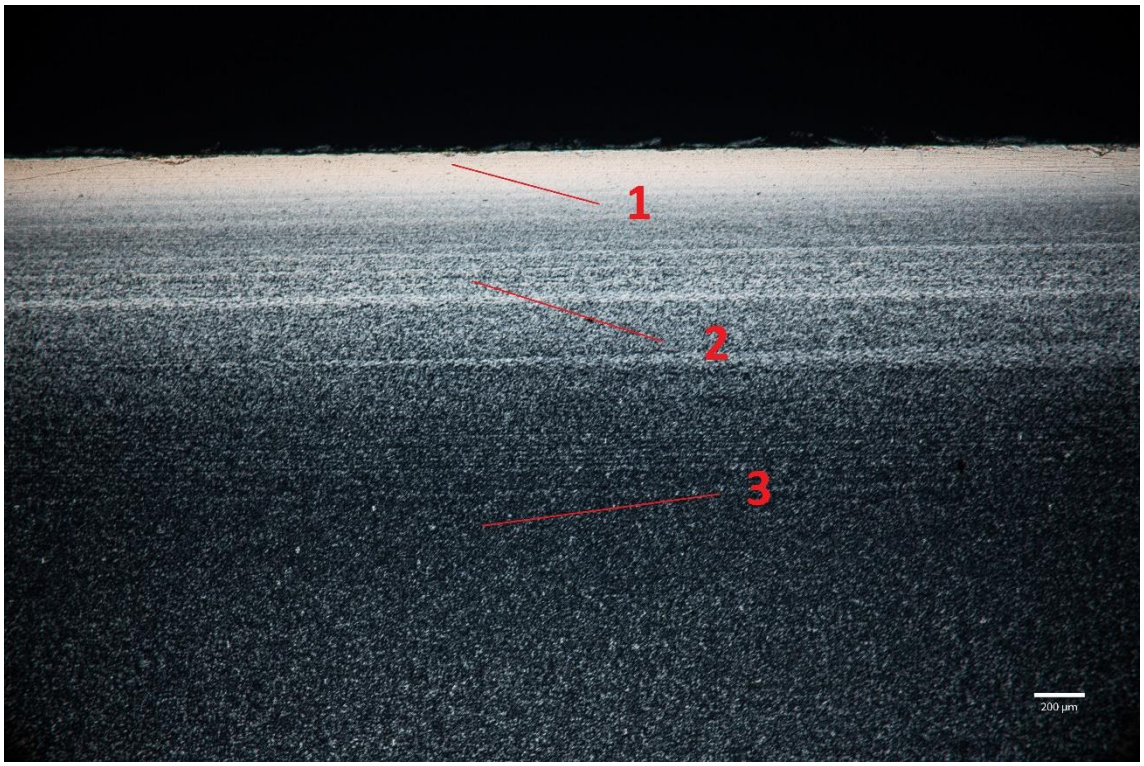
8.11 Analys av provstycken

Formsprutade provstycken

De formsprutade provbitarna som produceras i detta arbete visade information om kristalliniteten som kan utnyttjas för att studera hur materialet kyls i formen.

De formsprutade proverna framställdes av dragprovstycken utav polypropen. Längsgående och tvärgående tvärsnitt framställdes för mikroskopisk observation som illustreras i Figur 2.

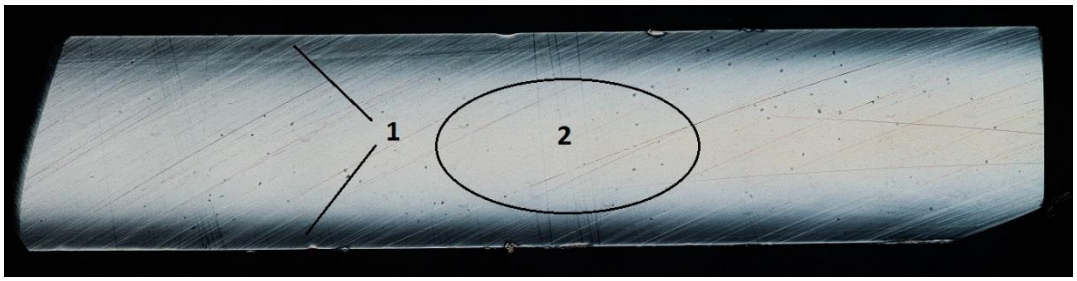
Figur 2: Formsprutat dragprovstycke utav polypropen med planerna valt för provstycken. Planens djup är inte representativa.



Figur 3: Tvärsnitt av plan 1 i Figur 2 1) Snabbt nedkylt område 2) Medelsnabbt nedkylt område 3) Långsamt nedkylt område [PP, 5x förstorning, tvärpolariserad belysning]

Figur 3 presenterar ett längsgående tvärsnitt från topp planet av ett formsprutat dragprovstycke vid ett djup på ungefär mitten av stycket. Nedre kanten av produkten på bilden visar att materialet smält jämnt vilket är utmärkande för formsprutade produkter av jämn kvalitet.

Den vita färgen orsakas av skillnad i kristallinitet vid kanten av provstycket jämfört med mittområdet. Kristalliniteten är resultatet av snabbare kylning vid kanten jämfört med mitten, på grund av styckets kontakt med formens vägg.

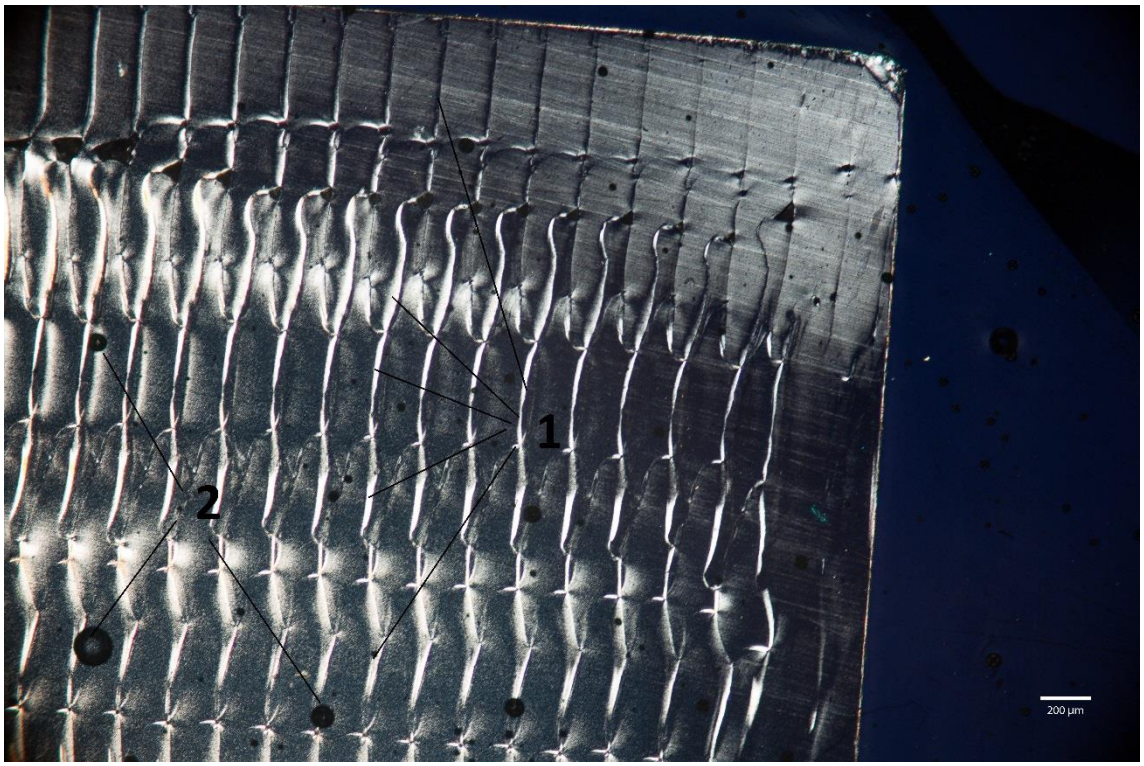


Figur 4: Tvärsnitt av planet 2 i Figur 2, Sammansatt bild, tvärpolariserad belysning. 1) Amorf struktur 2) Halvkristallin struktur [PP, 5x förstorning, tvärpolariserad belysning]

En sammansatt bild av tvärplanet 2 presenteras i Figur 4. Tvärsnittet visar tydligare skillnaden i kristallinitet jämfört med Figur 3. Bedömning av det tvärpolariserade ljusets brytning orsakad av stycket, avslöjar att centrumet erhållit en halvkristallin struktur medan kanterna har en amorf struktur.

Snabb kylning av formsprutad plast resulterar i en mer amorf molekylstruktur. Kanterna på stycket är områden som kyls först och erhåller därför en mer amorf struktur än det kristallina eller halvkristallina centrumet.

3D-utskrivna provstycken



Figur 5: 3D-utskriven PLA 1) Svetslinjer mellan skikt 2) Luftbubblor i harts [PLA, 5x förstoring, tvärpolariserad belysning]

Figur 5 representerar ett tvärsnitt av ett 3D-utskrivet PLA provstycke. Utskriftsskikten kan lätt särskiljas eftersom de uppvisar skiljbar ljusbrytning mellan varandra. Brytningen är sannolikt orsakad av kylning av skikten innan nästa skikt har skrivits på. Brytningen är en form av svetslinje eftersom två smält frontar svetsas ihop.

De runda cirkulära mönstren i bilden är bubblor i hartset som binder stycket till glasskivan, och är inte fenomen i själva provstycket.

8.12 Slutledning

Strukturen i detta examensarbete har varit en mycket enkel då processen baserat sig på att följa litteraturanvisningar steg för steg. Eftersom polering av tunna tvärsnitt är en välkänd metod inom geologi, förväntades att de flesta metoderna implementeras från detta område. Litteraturrecensionen visade emellertid också att det finns guider tillgängliga om hur man förbereder polymera tvärsnitt. Experiment som gjorts i detta examensarbete visade varierande resultat jämfört med metoder föreslagna i litteraturen som givit dåliga resultat och andra metoder som ansätts onödiga.

Styckena som producerades i detta examensarbete visade framgångsrikt ett nytt perspektiv av polymeranalys för både formsprutade och 3D-utskrivna delar. Syftet med denna studie var främst att utveckla en poleringsmetod för att producera tunna tvärsnitt i Arcada, snarare än att analysera dem. Analyseringen av provstyckena var kortfattad men kommer förhoppningsvis att gnista intressen för vidare studier, speciellt för 3D-utskrivna delar.