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Study of microstructures replication for self-cleaning applications

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STATEMENT OF INTEGRITY

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Resumo

A presente dissertação consiste no desenvolvimento de uma *protective optical window* com a propriedade auto-limpante, para a protecção de um sensor *LiDAR* aplicado em condução autónoma. A *optical window* deve ter uma excelente transmissão ótica no comprimento de onda do sensor.

A primeira fase do trabalho consistiu na revisão bibliográfica do estado de arte relativo às superfícies auto-limpantes na natureza e também de casos de biomimetismo destas superfícies em peças poliméricas. A pesquisa também incidiu sobre os métodos de replicação de microestruturas bem como nas propriedades adequadas para obter substratos microestruturados.

A funcionalidade da *protective optical window* pode ser assegurada pela configuração das microestruturas a replicar e do polímero usado. Por esta razão, uma configuração pré-definida foi testada para avaliar a sua influência na hidrofobicidade da superfície. Como o PMMA possui melhores propriedades óticas, foi realizado um teste preliminar para estudar as condições de processamento que favorecem a replicação, ao longo de um substrato. Por fim, o protótipo da *protective optical window* foi produzido por moldação por injeção, com o PMMA e a configuração previamente estudada com o inserto de cobre. Contudo, foi desenvolvido um novo inserto, num material diferente do usado no teste preliminar, para ser integrado num molde de injeção, com propriedades mecânicas adequadas para as altas pressões características desta técnica.

A replicação por moldação por injeção resultou em microestruturas não completamente preenchidas, com 1 μm de altura. Estes resultados correspondem a um aumento do ângulo de contacto de 13 °, apesar de não ter sido alcançado o regime hidrofóbico. Antes da moldação por injeção, adaptando a técnica de hot embossing, com a configuração de microestruturas previamente estudada, foi possível obter microestruturas com 8 μm de altura com um aumento do ângulo de contacto da água de 44 °. Caso a moldação por injeção não permita replicar a altura pretendida, foi desenvolvida uma proposta de molde de injeção-compressão, baseado na revisão bibliográfica, que combinado com um sistema variotérmico, é vantajoso para a replicação das microestruturas.

Os resultados sugerem que a presente configuração pode levar à propriedade auto-limpante, aumentando as propriedades hidrofóbicas da superfície, apesar de vários fatores precisarem de ser estudados.

Palavras-chave: Auto-limpante, Microestruturas, Replicação, *Protective optical window*, Ângulo de Contacto.

Abstract

The present dissertation comprises the development of a protective optical window, with self-cleaning ability, to cover a LiDAR sensor applied to autonomous driving. The optical window must have excellent optical transmittance concerning the operational wavelength of the sensor.

The first stage of this work consisted of a literature review on the state of the art concerning the self-cleaning surfaces in nature, as well the attempts to biomimetic these surfaces in polymer parts. The research also focused in the replication methods of microstructures as well the proper conditions to successfully obtain microstructured substrates.

The functionality of the protective optical window can be assured by the configuration of the microstructured pattern and the used polymer. For this reason, a pre-defined configuration was evaluated through its influence in the surface hydrophobicity. Since the PMMA presents better optical properties, it was performed a preliminary study to achieve the proper processing conditions which favour the microreplication along the substrate. Finally, the protective optical window prototype was produced by injection moulding, using PMMA and the configuration previous tested with the copper insert. However, a new insert of a different material was developed so it can be integrated in an injection mould, with accurate mechanical properties for the higher pressures characteristic of this technique.

The replication by injection moulding resulted in incomplete filled microstructures, with 1 μm of height. These results corresponded to an increase in the water contact angle of 13 ° although the hydrophobic regime was not achieved. Before injection moulding, improvising hot embossing allowed to obtain, with the same microstructured pattern, microstructures with 8 μm with an increment of water contact angle of 44 °. In case of the injection moulding will not allow to achieve the desired replicated height, a proposal for an injection-compression mould was developed, which is pointed in literature, combined with a variotherm system, as advantageous to replicate microstructures.

The results suggested that the present configuration can lead to the self-cleaning ability, by enhancing the surface hydrophobicity, although several factors need attention.

Keywords: Self-cleaning, Microstructures, Replication, Protective Optical Window, Water Contact Angle.

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List of Acronyms and Abbreviations

2D – Two Dimensions

3D – Three Dimensions

ACNT – Aligned Carbon Nanotube

CA – Contact Angle

CAD – Computer-aided Design

CAE – Computer-aided Engineering

CAH – Contact Angle Hysteresis

CENTIMFE – *Centro Tecnológico da Indústria de Moldes, Ferramentas Especiais e Plásticos*

CNC – Computer Numerical Control

Cu – Copper

DEP – *Departamento de Engenharia de Polímeros*

DOE – Design of Experiments

HE – Hot Embossing

ICM – Injection-Compression Moulding

IM – Injection Moulding

INL - International Iberian Nanotechnology Laboratory

LiDAR – Light Detection and Ranging

LIGA – *Lithographie, Galvanoformung, Abformung*

Ni - Nickel

PAN – Polyacrylonitrile

PC – Polycarbonate

Phold – Holding Pressure

PIEP – *Pólo de Investigação em Engenharia de Polímeros*

PMMA – Polymethyl methacrylate

PP – Polypropylene

PTFE – Polytetrafluoroethylene

Si – Silicon

Sinj – Injection Speed

Std Dev – Standard Deviation

Tmould – Mould temperature

UV – Ultraviolet

WCA – Water Contact Angle

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1 Introduction

The recent advances in technology aim for comfort and safety of people, sustainability and innovation. The automotive industry follows this development, investing to improve and develop technologies for providing an autonomous driving. Among the several advantages, the strongest is the reduction of human failure, due to the capability of avoiding crashes. It improves the road safety and optimizes the traffic flow and, consequently, reduces the gas emission. Another sustainable advantage for environment purposes is the reduction of vehicle weight and, proportionally, the fuel consumption [1,2].

The present dissertation is in the context of *Innovative Car HMI*, a project in the scope of *Bosch Car Multimedia* and *University of Minho* cooperation, which one of the objectives is the development of a LiDAR (*Light Detection and Ranging*) Technology based sensor, to allow autonomous driving.

LiDAR is a system which provides topographic information from environmental background and it has been adapted to autonomous driving [3]. LiDAR principle consists in determine distances, between the transmitter and the reflector. The transmitter emits a laser, with a specific wavelength and the distance is calculated by using the time elapsed since the transmission until the receptor capture the energy of reflector [4].

To preserve and assure the performance of the sensor technology, emerge the need of to develop a protective optical window for the sensor, with excellent optical transmittance and with a surface free of particles, which can interfere with the light behaviour, compromising the technology functioning. The employment of a self-cleaning surface can be a solution for the protective optical window.

One of the ways to obtain self-cleaning surfaces is through replication methods, by replicating the characteristic hierarchical structure of self-cleaning surface, present in natural surfaces. Polymers are a strong material to apply, due to their optical properties and the capability of to reproduce the hierarchical structure, which is crucial to obtain super hydrophobicity and, consequently, the self-cleaning ability.

1.1 Motivation

Scientific community have been studying the phenomenon of natural self-cleaning surfaces. Some plants, being the most known the Lotus flower leaves, presents superhydrophobic properties. Water droplets, when in contact with these surfaces, acquires an almost spherical shape and easily roll-off from small inclinations. It was found that this behaviour is possible due to the hierarchical roughness of the surfaces in micro- and nanoscale.

In the past years, investigators worked to create functional products by biomimetics, which can be a solution to optimize the lifetime and performance of a product. The attempts pass through changing a surface free energy by applying coatings or mimicking the hierarchical roughness by replication.

In the scope of this work, there are three main questions which motivated it. The first is if it is possible to obtain the required microstructured pattern in productive tools, namely microstructured inserts. The chosen insert material must provide a replication process quality, namely in the demoulding after the replication on polymeric parts. Then, if it is possible to combine the microstructures replication accuracy with the replication process reproducibility. For this, there is a need to study the process conditions which favours the microstructures replication as well other factors. Finally, it is necessary to understand if it is possible to find a commitment between the self-cleaning ability and the device optical performance.

1.2 Organization of work

Figure 1 shows a flow chart representing the tasks follow-up to fulfil the defined goals of this dissertation.

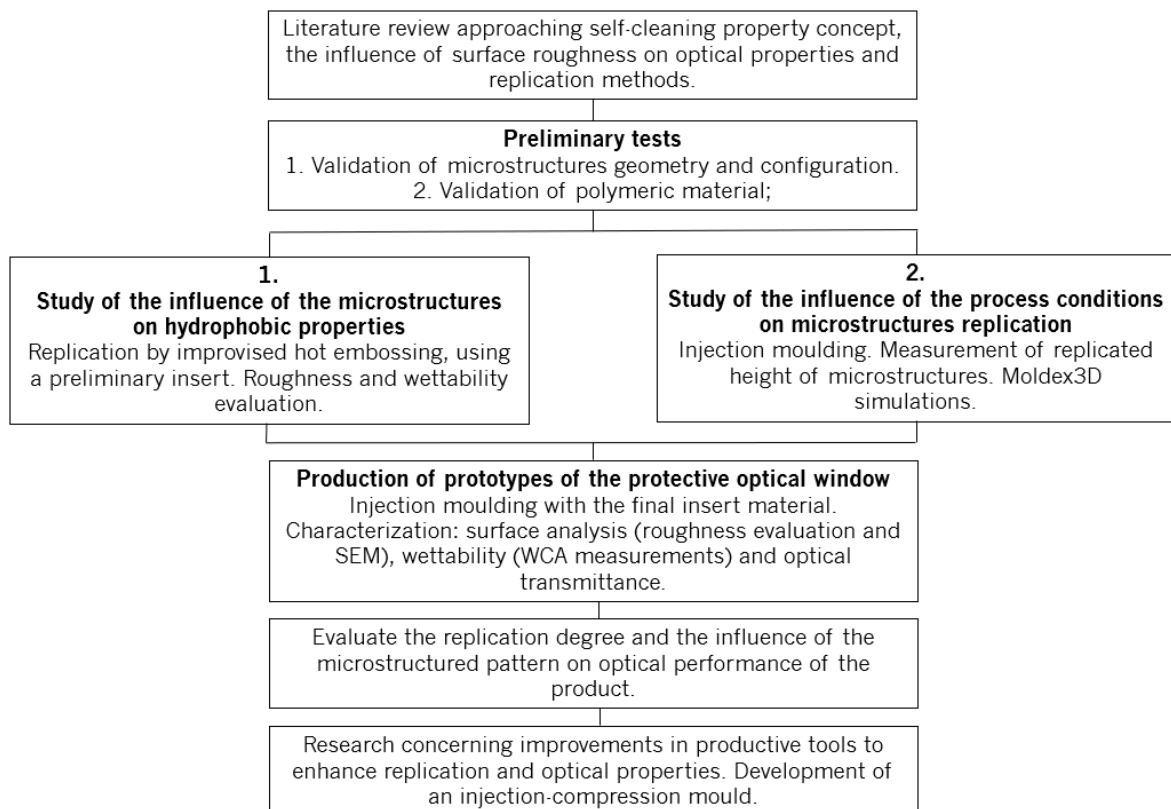


Figure 1 - Organization of master thesis work.

1.3 Master thesis structure

The present chapter gives an approach of master thesis goals, a definition of work motivation and it also provides an overview of tasks and its organization.

The second chapter comprise the research of concepts and the state-of-art concerning the self-cleaning property, the influence of surface roughness on optical properties and an overview of replication process, namely the usual techniques and how its process conditions influence the replication.

A description of the experimental techniques and methods is given on third chapter. It is described the used processing techniques and the used polymers. Also, it is described the methodology to characterize the produced samples as well the procedure applied to Moldex3D simulations.

The fourth chapter concerns the productive tools used in the experimental work, where it is detailed their production methods and the techniques applied. The structure of the used injection moulds is detailed, as well the modifications performed to adapt the moulds at the injection moulding machine of the experimental work. The production method of the microstructure mould inserts is also provided on this chapter.

The results obtained on experimental work are shown on fifth chapter.

The sixth chapter comprises the design of an injection-compression mould, a proposal of future work.

The seventh chapter corresponds to a summary of the results as well the main conclusions and on eighth chapter is given suggestions for further work in the scope of the project where this master thesis is inserted.

2 Literature review

2.1 Self-cleaning surfaces

Self-cleaning ability can be described as the way how dust particles and water droplets interact with a surface, being related with their hydrophobicity or super hydrophobicity. In nature, the most popular example is the Lotus flower leaves, the reason for the phenomenon being known as “Lotus effect.”

The investigation of these natural surfaces has been increasing due to their several characteristics with potential for engineering applications, with practical and sustainable contribution. Self-cleaning surfaces can contribute in several fields, such as: biomedical [5–8], enabling fabrication of surfaces which prevent bacteria contamination or precise dosage drug delivery devices; automotive, to replace windows windshield [9] and optical field [10–12], for example in solar panels, where dirt can compromise the cells efficiency. The characteristics of self-cleaning surfaces allow the full performance of devices and permits self-maintenance actions, important to reverse environmental effects.

This subchapter intends to provide to the self-cleaning ability a physical explanation, based on wettability phenomenon. It will be introduced the concepts of Contact Angle (CA), Contact Angle Hysteresis (CAH) and Sliding Angle (SA). Next, it will be explained the relevant wettability models. Finally, after a brief summary of the conditions for the self-cleaning ability, the Lotus effect will be explained, based on the state-of-art and it will be presented some attempts of mimicking its leaves structures in polymers surfaces.

2.1.1 Wettability

Wettability is a common interface phenomenon, characterized by the contact angle formed when a liquid meet with a solid surface. In general terms, and considering the water as the liquid, if the formed contact angle has a value inferior to 90° , the surface is classified as hydrophilic. On the other hand, a contact angle value above 90° indicates a hydrophobic surface. If this value is between 150° and 180° , the surface is classified as superhydrophobic and the water droplet acquires an almost spherical shape.

In contact angle formation during wetting process (Figure 2), there are involved three interfacial boundary surfaces tensions which will influence the contact angle value [13–15]: γ_{SV} , γ_{SL} and γ_{LV} , representing solid/gas, solid/liquid and liquid/gas interface tensions, respectively. The equilibrium among the referred surface tensions, providing the lower state of energy, leading to the stability of the drop, forming a contact angle (θ).

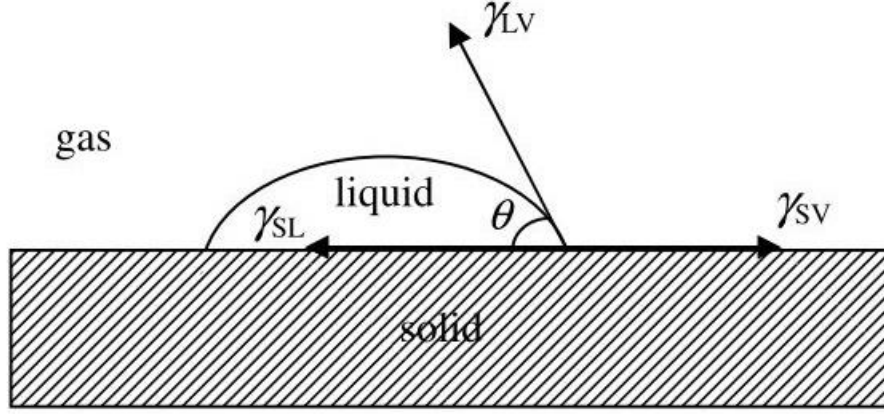


Figure 2 - Representation of the interfacial boundary surfaces tension and contact angle formation. Source: [16].

For an ideal and smooth surface, Young's equation correlates the formed contact angle with the interaction of the interfacial tensions:

$$\cos \theta = \frac{\gamma_{SV} - \gamma_{SL}}{\gamma_{LV}} \quad (2.1)$$

However, the measured contact angles of a droplet on the same surface can change, due to temperature variations, water adsorption or even irregularities on the solid surface, such as roughness [17,18]. The Contact Angle Hysteresis (CAH) quantifies this difference and it is given by Equation 2.2.

$$CAH = \theta_A - \theta_R, \quad (2.2)$$

where θ_R is the Receding Contact Angle (Figure 3a) and θ_A is the Advancing Contact Angle (Figure 3b), being the minimum and maximum contact angles values observed on a surface, respectively. The sliding angle (α), represented in Figure 3c, corresponds to the necessary angle to a droplet moves in a tilted surface and it is related with the Contact Angle Hysteresis. In other words, when the droplet moves, it will advance of the downhill side and recede on the uphill side. This hysteresis must be minimal to maintain the droplet spherical shape and allow the rolling movement which is crucial for self-cleaning ability [16,19], as seen in Figure 4.

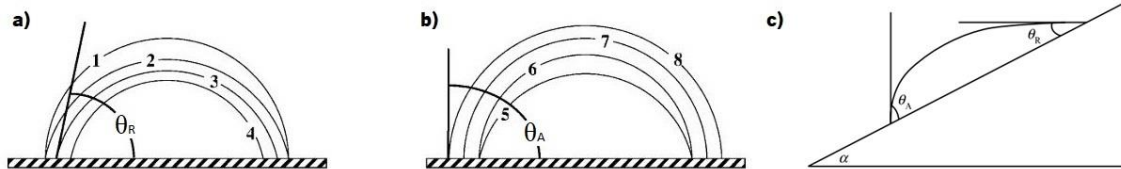


Figure 3 - Representation of a) Receding Contact Angle (θ_R), b) Advancing Contact Angle (θ_A) and c) Maximum and Minimum Contact Angle related to the Sliding angle (α). Adapted from [16,17].

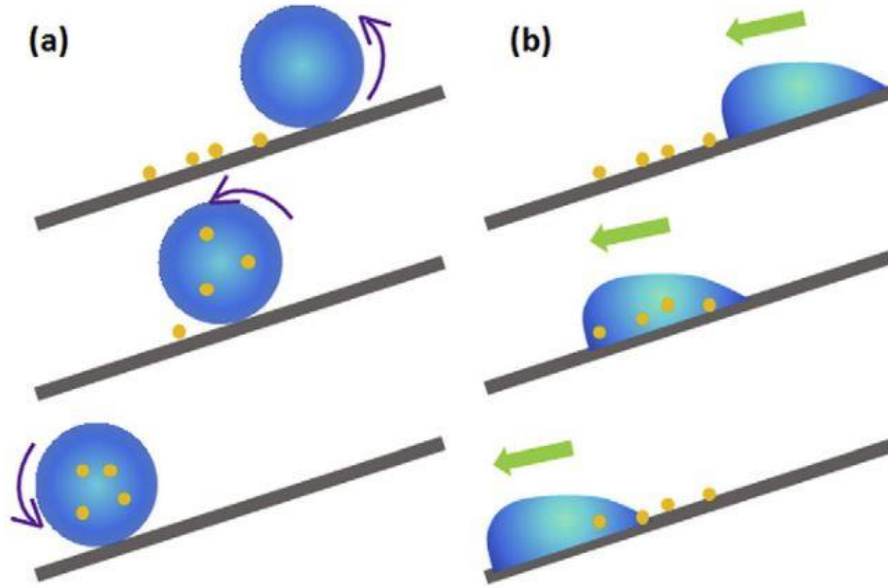


Figure 4 - Difference between a surface with a) self-cleaning ability and b) without self-cleaning ability. Source: [20].

For rough surfaces it is only experimentally possible to measure the apparent contact angle (θ'), since the apparent contact area is different from the measured on a smooth surface. The existing models [19,21–23] for wettability on rough surfaces are based in Young's model, deducing a relation between θ and θ' . Below, it will be given a brief description of the classic states that describe the droplets behaviour on rough surfaces: Wenzel's state and Cassie-Baxter's state.

Wenzel's state

Wenzel described the relation between contact angle and the apparent contact angle (Equation 2.3) as dependent of an increase in surface area caused by the roughness increasing. In this model, the liquid wets all entire surface roughness (peaks and valleys), as represented in Figure 5a.

$$\cos \theta' = r \cdot \cos \theta, \quad (2.3)$$

where r corresponds to the roughness factor, the ratio between the solid-liquid area (corresponds to the area under the drop) to its projection area in a plan (geometric area) [19]. Polymers have low surface energy, due to its chemical composition, so it presents a certain resistance to wet. Inducing roughness will increase this resistance, accordingly to Wenzel's model.

Cassie-Baxter's state

Cassie and Baxter modelled the wetting of porous surfaces, where it is formed a heterogeneous interface of liquid and air. It happens due to the formation of air pockets when the liquid contacts with the solid surface and does not get in the roughness valleys, as represented in Figure 5b. This relation can be described by the Equation 2.4.

$$\cos \theta' = f \cdot \cos \theta + (f-1), \quad (2.4)$$

where f corresponds to the fraction of contact area between solid and liquid, and $(f-1)$ to the area of solid-air interface.

A droplet in Wenzel's state presents a higher CAH and high surface adhesion, being unable to move. On the other hand, the droplet movement is promoted in Cassie-Baxter's state, due to the non-wet contact mode and the low surface adhesion force. Besides, the droplet state can also be defined by a transition between Wenzel's and Cassie-Baxter's states (Figure 5c). In this case, if the surface is submitted to a sliding angle, the droplet movement is promoted.

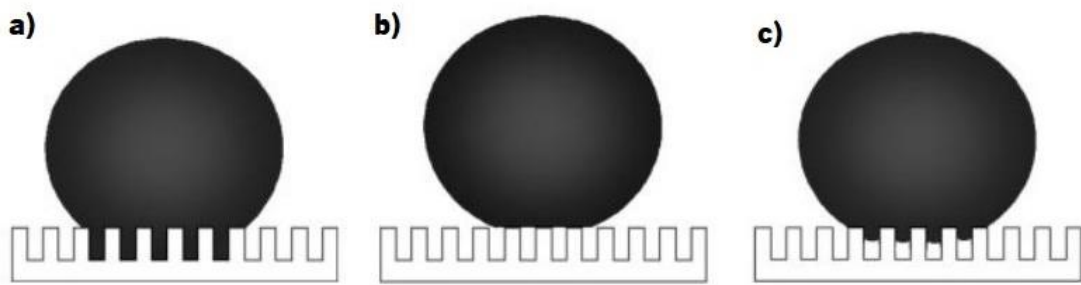


Figure 5 - Schematic representation of wetting states on rough surfaces. a) Wenzel's state, b) Cassie-Baxter's state, c) Transition between Wenzel's and Cassie-Baxter's states. Source: [21].

2.1.2 Biomimetics of self-cleaning effect: Lotus effect

Due to the potential of self-cleaning effect in several fields, namely in engineering applications, the research focused in mimicking this natural phenomenon have grown exponentially. The most famous is called the "Lotus effect", noticed in Lotus flower leaves surfaces. As stated in the previous subchapter, on self-cleaning surfaces the non-wetting property should be enhanced, to enable the flow of the fluid and to allow the dust particles removal by a rolling water droplet at a small sliding angle [16,24]. In general, the main conditions for self-cleaning ability, stated below, are satisfied with a surface with the characteristics of Lotus leaf [25,26]:

- i. Contact angle superior to 90° (hydrophobic surface);
- ii. Required low contact angle hysteresis (typically between 2° and 5°);
- iii. Low adhesion force between the particle and the surface;
- iv. Dust particle size.

The water droplets, when in contact with Lotus flower leaves surface acquires an almost spherical geometry (Figure 6a), with a water contact angle usually between 150° and 160° (Figure 6b). A small inclination, with a low contact angle hysteresis, between 2° and 5° , should be enough to allow an easily

roll of the water droplets, collecting dust and other particles during the process [27,28]. The Lotus leaves surfaces are characterized by a hierarchical roughness of the surface structure: a level composed by micro-scale pillar structures similar to peaks, between 5 and 10 μm of height (Figure 6c), and a nanostructured level, covering the leaf surface including the microstructures top, with dimensions in a range of 150 nm (Figure 6d). The surface is also covered by a low surface energy epicuticular wax with regular thickness [27,29].

A special case of wettability is observed in Lotus flower leaves (Figure 6e), which it is similar to Cassie-Baxter's state. Due to the hierarchical roughness, a high CA and low CAH are promoted, leading to self-cleaning ability.

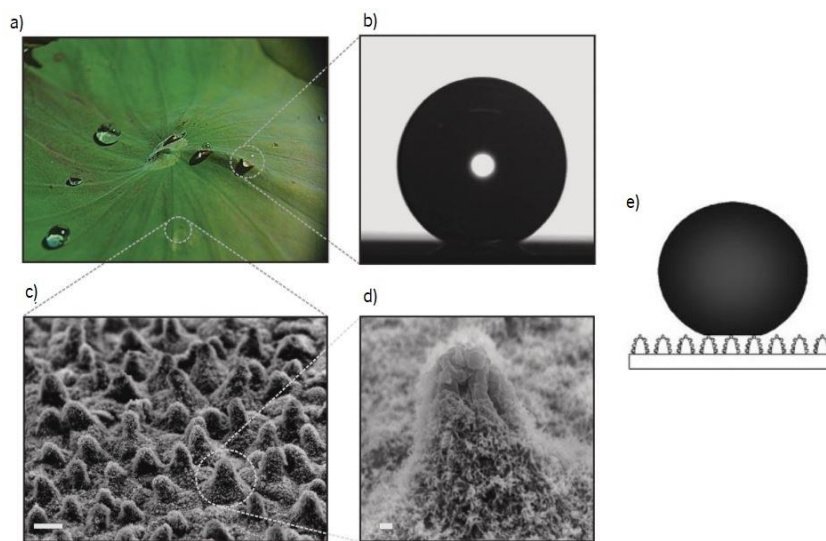


Figure 6 - a) Water droplets laying in a Lotus leaf surface, b) droplet with a CA of 153° , c) microstructured pattern of the leaf structure, d) magnification of a microstructure, where is seen the characteristic nanostructure e) schematic representation of lotus leaf surface wetting state. Adapted from [21] and [29].

To achieve self-cleaning in engineering applications, researchers have been studying the role of the coating wax and the nano- and microstructure observed in these natural surfaces. Cheng et al. [30] compared the contact angles of dried lotus leaves with and without the nanostructure present, obtaining a contact angle of 142° and 126° , respectively. They concluded that a nanostructured surface is essential to achieve higher contact angles and, consequently, super hydrophobicity. In biomimetic field, Feng et al. [31] summarized the individual influence of nanostructured surfaces as well the effects of combined micro- and nanostructures. Their measurements were made on surfaces with similar nanostructures but with different density of structures. The highest contact angle was measured in the less dense, because it allows to air to get stuck under water droplets, reaching to Cassie-Baxter's state [32].

As mentioned above, hydrophobicity or super hydrophobicity are not enough to achieve self-cleaning. It is required a low CAH and several studies [31,33,34] point the hierarchical roughness responsible for influence the contact angle hysteresis, related with the capability of droplets roll off of the surface. Zheng and Lü [35], based on state-of-art, explain that the sliding angle decrease is a result of an increase, in length and energy, of the triple phase contact line, provoked by the reduction of the roughness scale combined by the trapped air pockets characteristic of the Cassie-Baxter's state.

Scientific community have been working to mimic the Lotus effect, in polymer substrates. Zhou et al. [36] produced a hierarchical structure (Figure 7) by injection moulding, with PP and PC, to achieve hydrophobic properties. For a smooth surface samples, the WCA measured on PP and PC samples were 100.0 ° and 82.3 °, respectively. For PC, the highest CA measured was 150.6 ° on samples produced with 178 °C of mould temperature with a combined structure (hierarchical), as seen in Figure 8.

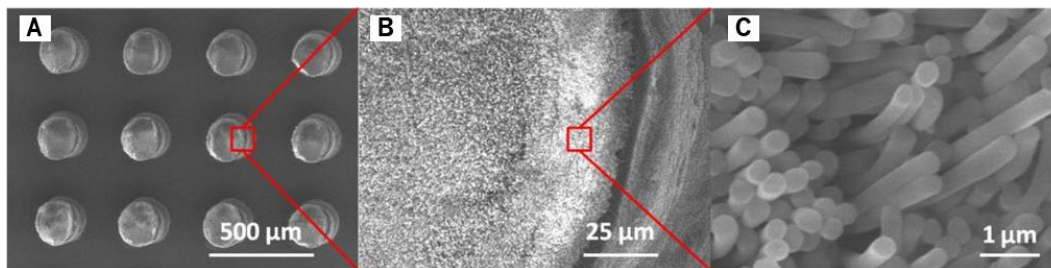


Figure 7 - SEM images of replicated hierarchical structures on a PC surface, through different magnification. Adapted from [36].

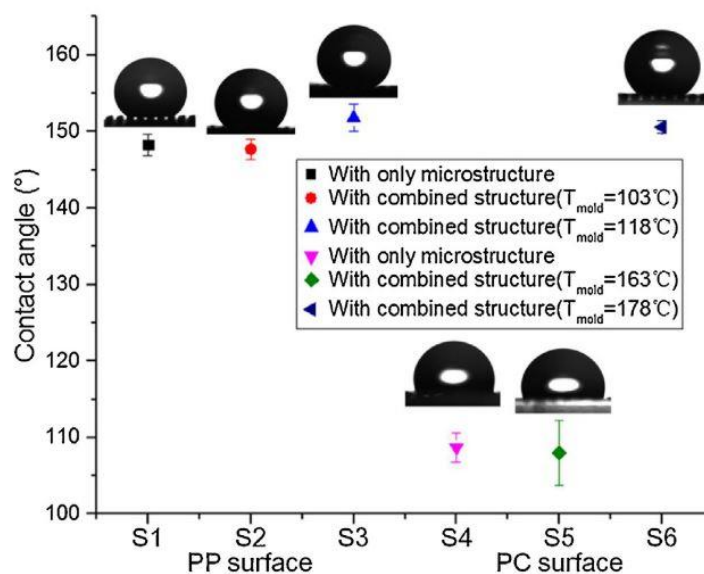


Figure 8 - Measured contact angles in PP and PC surfaces, with different surface structures. Source: [36].

Also by injection moulding, Saarikoski et al. [37] modified PC samples surfaces, creating nano-, micro- and hierarchical structure, obtaining for the hierarchical structure an increment of 57° for CA, face to the one measured on smooth surface. For the surfaces with only micro- or nanostructure, the CA increases between 29° and 41° . In Figure 9, it is possible to see the microfeatures pattern, etched in mould insert (a), as well this pattern replicated in PC samples (B). The microfeatures has a characteristic nanoroughness, which forms the hierarchical structure. The authors increased the magnification to capture a SEM image of the bottom of one microfeature in mould insert (C) and of its replica, in PC sample (D).

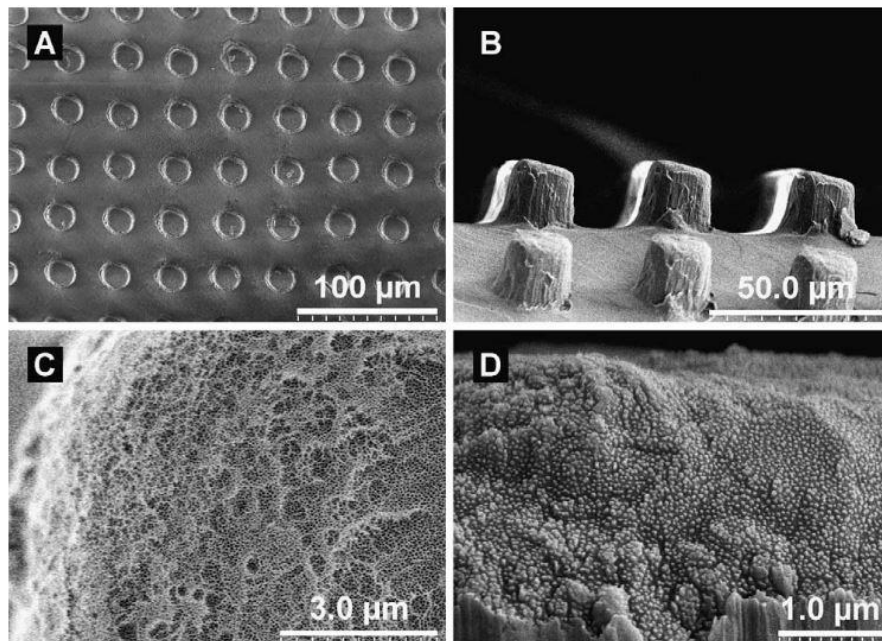


Figure 9 - SEM images of hierarchical roughness on mould insert and replicated PC samples: A) Mould insert microcavities, B) Replicated microstructures on PC sample, C) nanoroughness on bottom of a microstructure in mould insert, D) Replicated nanostructure on PC sample. Source: [37].

These progresses in replication by injection moulding bring advances in reproducibility and mass production of hierarchical structures for enhance hydrophobicity. The development of mould inserts which can provide this functionality to polymers is essential to reach products with self-cleaning ability.

2.2 Optical components

Plastic optical devices have become economic competitive in the market, replacing glass components. Due to the high reproducibility of plastics, it is possible to achieve complex parts with high optical quality and functionality. The requirements to produce optical components are centred in dimensional stability of the component and in its internal properties which influence the performance of an optical system. The polymers, specially PMMA and PC, are suitable for optical applications, due to their excellent transparency at a specific wavelength resulting in good optical transmittance. The process

should be highly controlled, since it can induce alterations in moulded components which deteriorate the polymer optical properties [38].

Individually, the manufacturing of optical components is a challenge. It is required a proper machine technology, the development of precise productive tools and optimal process conditions.

Injection-compression moulding is pointed as the most accurate replication technique to optical components [39]. It combines the uniform pressure distribution of compression moulding and the automation of injection moulding, resulting parts with excellent mechanical properties, low residual stress and tight tolerances. Injection moulding to produce optical lenses is also used. Lu and Khim [40] focused in how process parameters influence the lens functionality. Their main conclusions include mould temperature as the most influent parameter in dimensional stability of lenses and that a holding pressure stage divided into two steps enhances the surface replication of the lens. Also, they conclude that the injection speed contributes to modify the stress distribution: by increasing the injection speed, the high stress centre region tends to enlarge.

As mentioned, the optical devices performance is influenced by the internal properties of the optical components used in its protection, as with the LiDAR. However, external factors also have its influence, such as the dust, ice or other particles which, when accumulated, interfere in the device optical performance. A solution to be implemented can be the self-cleaning ability, characterized for rough surfaces which provides characteristics such as superhydrophobicity. However, it is difficult to find a commitment between the optical transmittance and the surface characteristics, since the microstructures leads to changes in light direction.

For smooth surfaces, transmitted and reflected light behave differently compared to a rough surface. While in the first one, the light is composed by a specular part in a particular direction, in the second, a phenomenon named as light scattering is responsible for light diffusion [41].

Optical transmittance can decrease due to the interaction of photons with the matter: by light absorption or light scattering (Figure 10). Light scattering consists in a change of direction of photons, when it finds a surface irregularity (such as roughness or scratches) or other element, such as dust particles. It can be observed reflection, refraction or diffraction of light, resulting in a decrease of the light transmittance, when a beam is subjected to scattering [42].

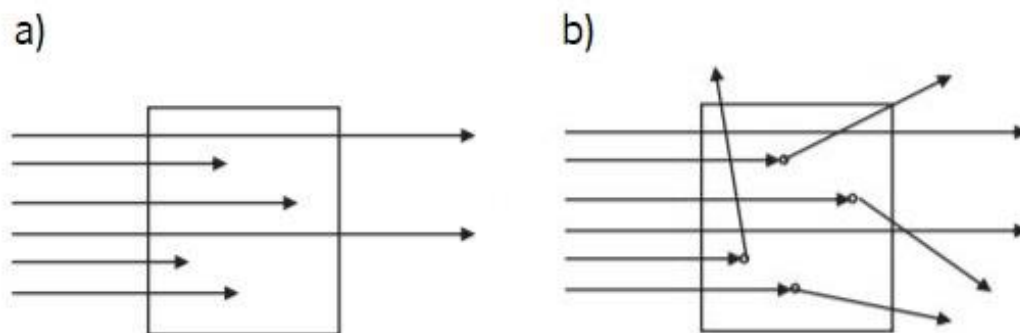


Figure 10 - Interaction of the light beam with matter: a) light absorption and b) light scattering [42].

Nakajima et al. [43] prepared superhydrophobic films by a separation phase of a sol-gel and the addition of an acrylic polymer, at different concentrations (Figure 11). To finalize, the films were coated with fluoroalkylsilane. Highest acrylic polymer concentration results in different arrangement and height of microstructures, influencing the CA and the SA (Figure 12), reaching to the required conditions to self-cleaning behaviour.

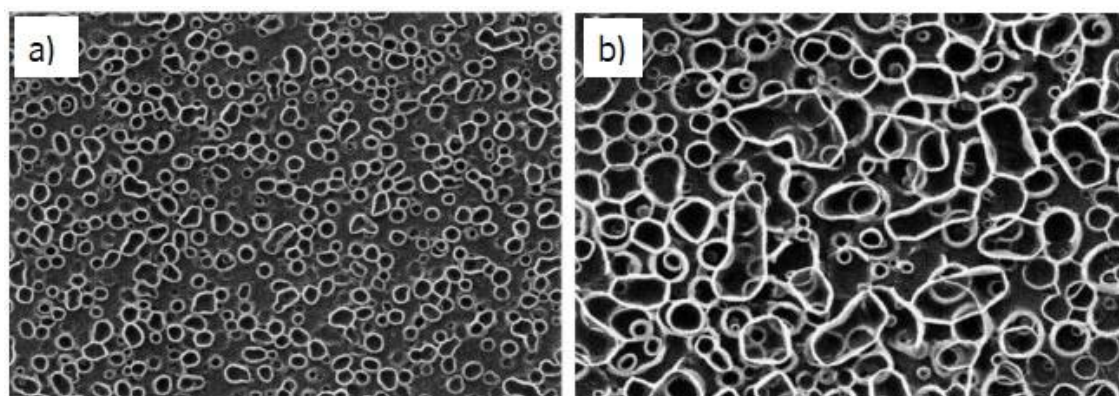


Figure 11 - Acrylic polymer concentration in the film preparation: a) 1% and b) 4% [43].

In relation to contact angle and the sliding angle necessary to the droplets roll off, these parameters were favoured by the highest polymer concentration. As seen in Figure 12, it was obtained a CA higher than 150° and a SA lower than 10° , which can result in self-cleaning ability. However, it was not possible to find a commitment with the film optical transmittance, since this parameter decreases about 40 %. The authors emphasize the measured values for 1 % concentration. Despite of a superhydrophobic state, the obtained optical transmittance is 90 % (Figure 13).

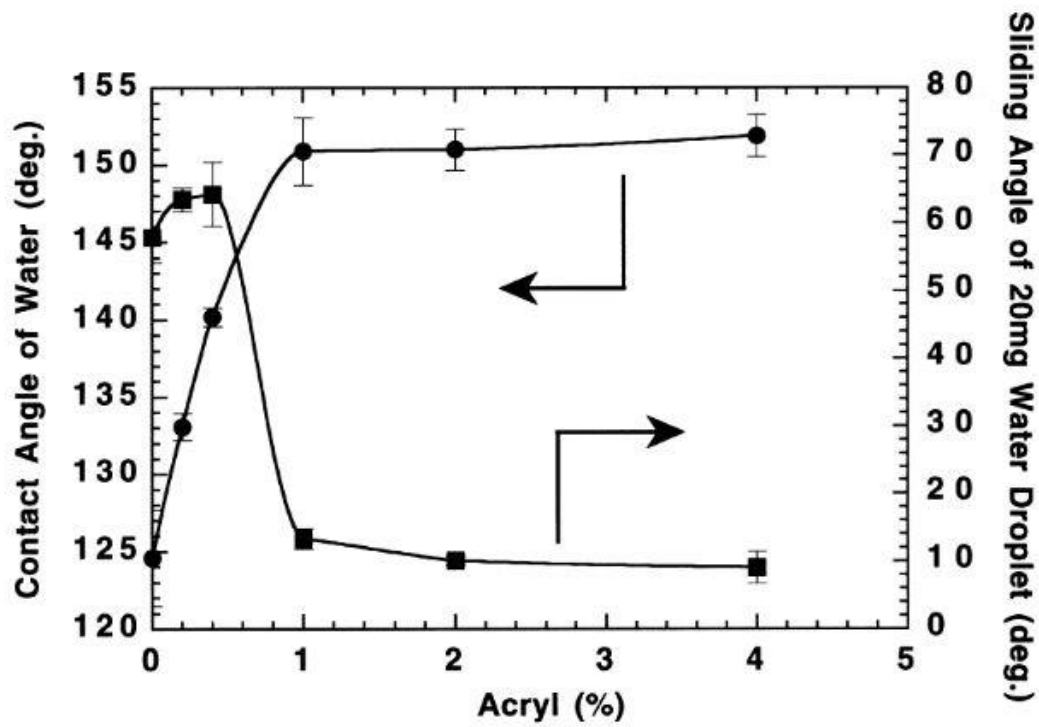


Figure 12 - Influence of polymer concentration (level of induced roughness) in CA and SA. Results from [43].

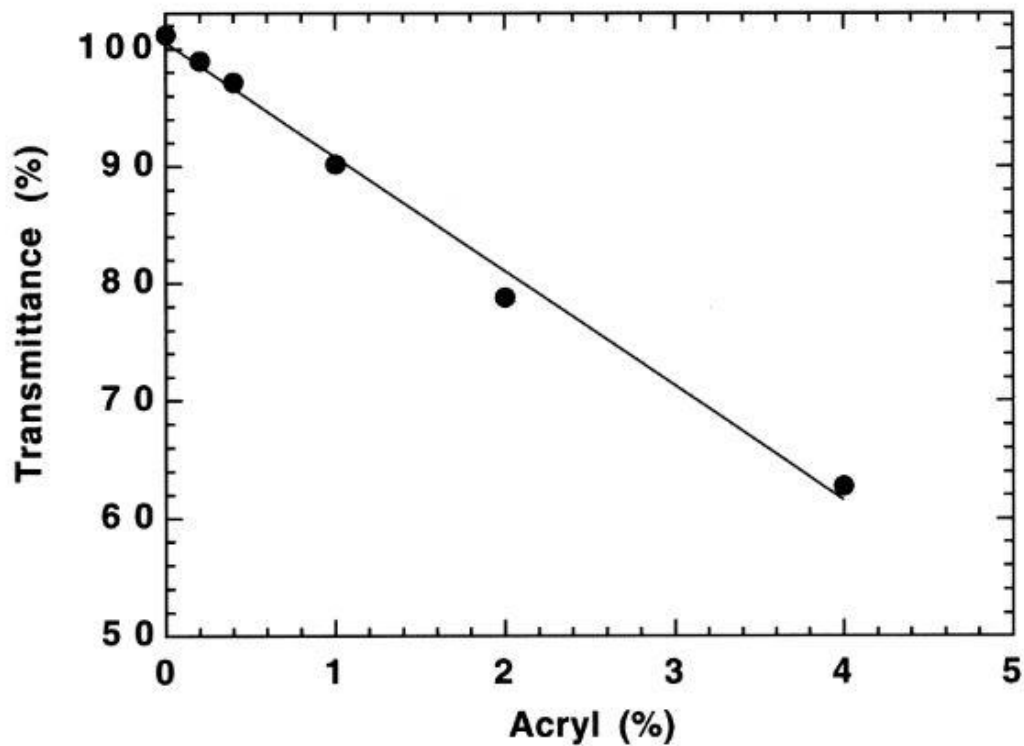


Figure 13 - Light transmittance at 500 nm wavelength in function of acrylic polymer concentration. Results from [43].

Since surface roughness induces alterations in light behaviour, it is important to study solutions which provides a commitment between optical transmittance and micro- and nanostructured surfaces. For example, by modifying the surface roughness of PC samples, Saarikoski et al. [37] compares a

smooth surface and nanostructured surfaces. The part with smooth surface is thicker than the nanostructured parts. With this specification, the presence of nanostructures increased the optical transmittance in the wavelength range of 380 to 780 nm (Figure 14). This increase is most notorious when the nanostructure diameter decreases. These results suggest that commitment between surface roughness and optical transmittance can be achieved by substrate thickness reduction, combined with adequate diameter of structures.

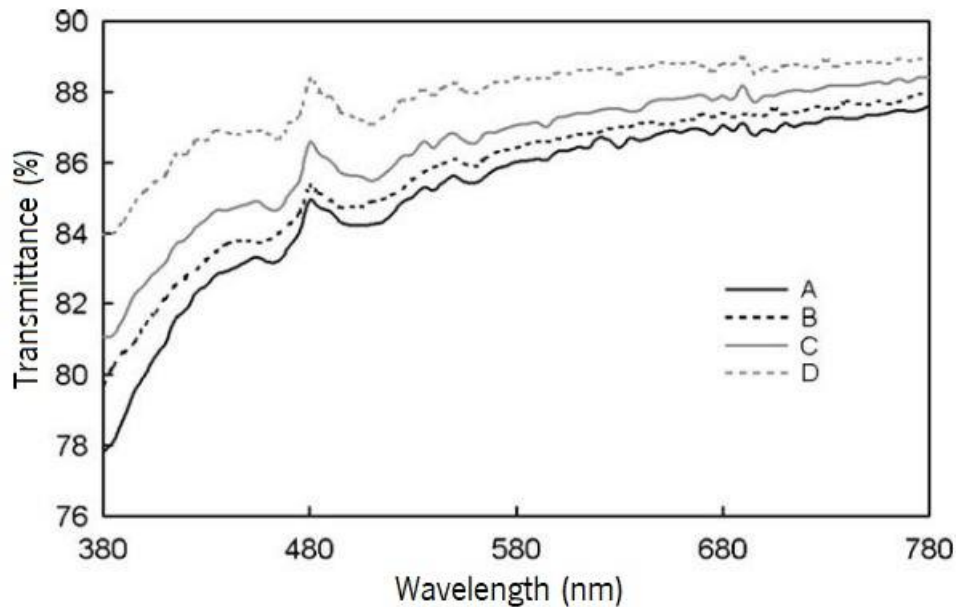


Figure 14 - Optical transmittance comparison between A) smooth PC surface and nanostructured surfaces with diameter of B) 59 nm, C) 49 nm and D) 41 nm. Source: [37].

2.3 Replication of microstructures

Another challenge in the biomimetic field is to replicate the microstructured patterns using polymers. The replication of micro- and nanostructures consists in the reproduction of a micro- or nanofeature geometry from a mould to a specific material workpiece subtract. It depends on the accuracy of the chosen technology, combining the replication fidelity with the reproducibility of the process. Besides this, process conditions of each technology have highly influence in replication success. The tool and subtract material behaviour as well the microfeatures configuration and geometry also influence the replication process [44].

The quality of replication is strongly related with the technology involved. In addition, it is important to find a commitment between quality and process automation, fulfilling the following criteria [45]:

- Low cycle times and large series production;

- Flexibility in exchange the microstructured insert;
- Low flow paths for microcavities filling;
- Reduction of internal stress and warpage;
- Cost effectiveness (the number of produced parts offsets the initial investment).

Injection moulding was the primary chosen technology to produce the protective optical window prototype, due to the process automation and knowledge associated. This technique will be described, as well the hot embossing, the most accurate technology to obtain quality parts. Finally, some studies where it is reported the influence of the process conditions on replication are presented.

2.3.1 Hot Embossing

Hot embossing process is often used when the replication requires high precision and quality, as in the optical components. This technique consists in pressure application into a semi-finished polymer subtract, under vacuum, to assure microcavities filling. The use of a semi-finished part guarantees a cavities filling with the lowest flow path, since there are a homogeneous distribution of the melt over all the tool surface area [44,46,47].

There are three concepts of hot embossing, represented in Figure 15: flat area against flat area (a), rotating cylinder against flat area (b) and two rotating cylinders (c). The microstructures geometry and the flexibility of the workpiece to be replicated have to be taken into account [48]. For high aspect ratio microstructures, the most accurate is flat area against flat area, due to the uniform applied pressure and the integrated extraction system, normal to the microstructures, which prevent its damage in demoulding.

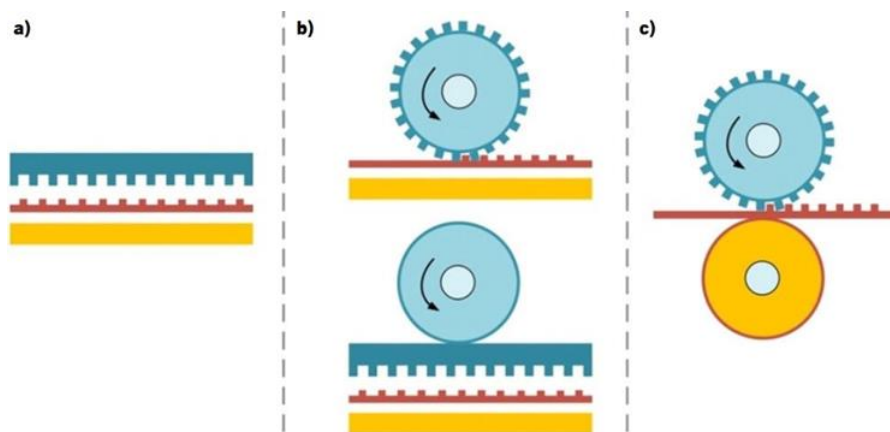


Figure 15 - Schematic of the three concepts of hot embossing: a) flat area against flat area, b) rotating cylinder against flat area and c) two rotating cylinders. Adapted from [48].

The process can be divided in three main steps [46,49] represented in Figure 16:

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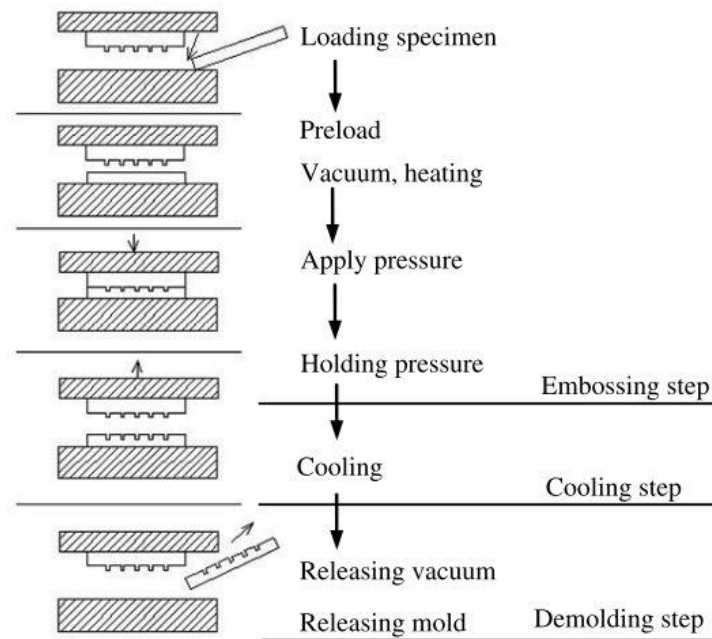


Figure 16 - Schematic representation of hot embossing process. Source: [50].

- **Heating** the polymer substrate until it acquires molecular mobility and **embossing**, controlling the force and velocity. The polymer is heated above its glass transition temperature, to allow the microcavities filling and to avoid large distortions.

The crucial process parameters associated to this step are embossing temperature, embossing pressure and holding time. Yong He et al. [50] concludes, through process simulation, that cycle time increases if the embossing pressure is not enough, due to the necessity of higher holding time, to fill the microcavities. Higher pressure results in more efficiency of filling, thereby it damages the mould insert, specially the most fragile, like silicon.

- **Cooling**, while the embossing pressure is still applied. Additionally, there is a difference between the thermal coefficient of the polymer and the mould, giving an additional stress which influence the demoulding [51] . Also, not only the cooling but also the heating of the subtract should be as homogeneous as possible, to avoid premature solidification of some areas.
- **Demoulding**. The involved demoulding forces can damage the replicated structures, due to the friction forces enhanced by the thermal stress caused by the shrinkage between the mould and the workpiece. It is in demoulding that occurs the main defects present in the embossed parts [47,52]:
 - microstructures distortion, caused by a non-vertical demoulding collectively with the stress involved;

- pulled out microstructures because of the high adhesion between the polymer and the metal insert;
- neck formation, due to the joint action of frictional force and high demoulding temperatures.

2.3.2 Injection moulding

The technology of injection moulding allows high production cadence, mouldings of several dimensions and geometries, dimensional precision and high reproducibility.

The moulding cycle of IM technique is represented in Figure 17. For all non-conventional techniques derivate from IM, the cycle is similar. Next, is given a brief description of the cycle [53,54]:

- **Mould closing**, triggered by clamping unit advance until the two mould halves meet and the mould is closed completely, followed by **injection unit advance**.
- **Injection** of polymer melt, filling the mould cavity by screw axial movement;
- **Cooling**, which starts when melt contacts with mould walls. It is the longest process step and it dictates the time cycle.
- To compensate material shrinkage during cooling, additional material is forced to enter the mould cavity while it is applied a **holding pressure**. This step is controlled hydraulically, to adjust the pressure at the gate.
- After holding pressure, **injection unit step back** and it is initiated the **plasticisation** for the next cycle. The screw rotation and the heating barrel provides the shearing to plasticize the polymer pellets. The resulting melt quality is important to avoid material degradation, non-homogeneous melt and non-plasticized pellets can disturb the cycle, resulting in high material waste.
- When moulded part has enough stiffness, after cooling, the mould ejection system **ejects** it from the cavity, after **mould opening**.

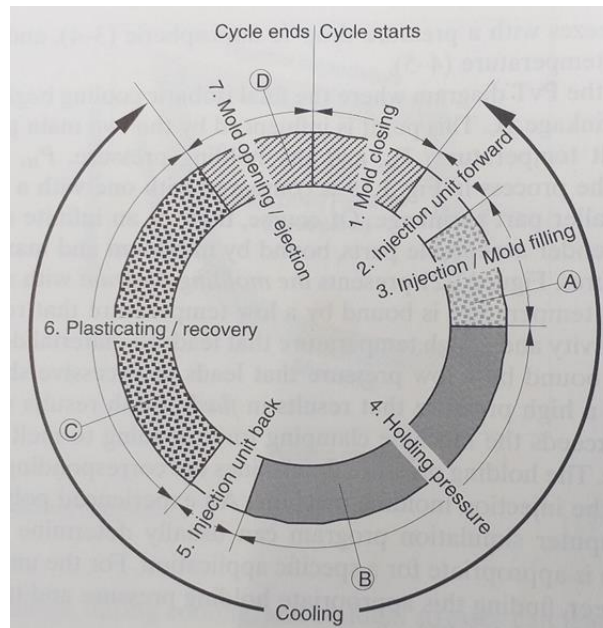


Figure 17 – Injection moulding cycle. Source: [55].

If the parts have microscale precision (as micro thin-wall parts), weights in order of a few milligrams, or if they have micro featured surfaces, then the process is classified as microinjection moulding (μ IM) [55,56]. The process cycle is similar to the conventional injection moulding, although there are some differences, especially in tools [57]:

- The μ IM technique requires machines with high accuracy with reduced time of melt residence [58]. So, it was developed screws with smaller diameter, allowing a homogeneous melt with minimal volume. Thereby, the melt residence time is reduced, decreasing the probability of material degradation.
- The μ IM machines are capable of more accurate metering, highly required in the replication of small features, avoiding overfilling.
- Injection speed must be higher in μ IM, due to the highest cooling rates which causes premature polymer solidification. The μ IM machines must allow lower injection times.
- The holding pressure stage has less influence in μ IM, since the cavity fills during injection step, though it is not clear the influence of this parameter in the micro- and nanostructures filling [59].

Another derivate from injection moulding is the injection-compression moulding technique. It combines two concepts and its advantages: injection moulding and compression moulding (same principle of hot embossing), providing the automated production of high accuracy with uniform inner properties parts [39]. In the ICM cycle (Figure 18), a compression stage is added during or after injection of polymer melt into cavity, while the mould is slightly open, resulting a compression gap which

determines the compression stroke. The compression stage can be performed by the machine or for some element present in the mould. This process allows to obtain excellent results in both optical parts and replication of microstructures.

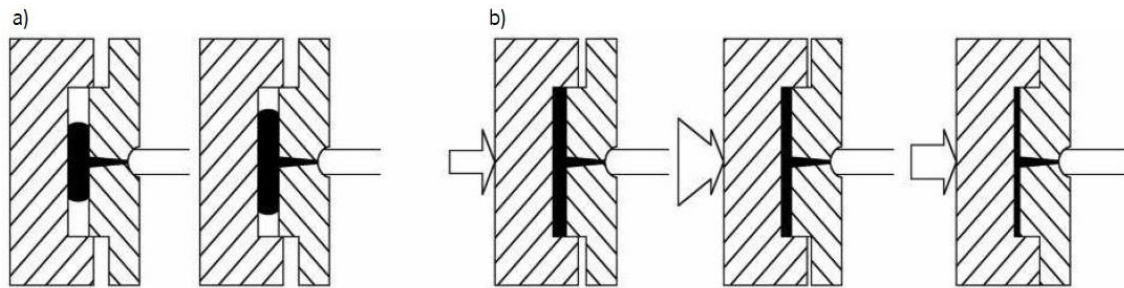


Figure 18 - Injection compression moulding scheme. a) Injection step followed by b) Compression step.

2.3.3 Balance between the available processes

Hot embossing presents several advantages to obtain quality microstructured parts, with the disadvantage of too long cycle times, being impossible to meet market needs. Nevertheless, hot embossing is suitable for product development in laboratories, allowing to determine the mould insert characteristics that provide a better replication, to adjust some process conditions and to select the best configuration for the microstructured substrate.

The automation of injection moulding provides a competitive product, at economical and energetic levels. The quality problems associated, such as parts residual internal stress, can be solved by injection compression moulding technique, characterized by a reduction of flow paths due to the additional pressure applied while or after the polymer injection. The pressure applied is more homogeneous, reducing warpage defects and helping the filling of microfeatures.

Table 1 - Resumed balance between Hot embossing, Injection moulding and Injection-Compression Moulding.

Requisites	HE	IM	ICM
Automatization and low cycle times		x	x
Lowest residual stress	x		x
Lowest flow paths	x		x
Easiest exchange of mould inserts	x		
Cost effectiveness		x	x

In Table 1, a resumed balance between the replication techniques is provided. In literature, Sortino [60] compares IM and ICM techniques for replication of a micro-optical device, with six types of microfeatures geometry. The study allows to conclude ICM as the process with most repeatability and

reproducibility of the structures, where it was obtained the highest replication degrees and the lower results variation.

2.3.4 Factors influencing microreplication in microinjection moulding

Since the protective optical window consists in a microfeatured substrate with a thickness in order of millimetres, the process is classified as microinjection moulding, whose replication has its limitations based on process conditions, material properties and microfeatures geometry.

The resistance to flow verified by the melt at the entrance of microcavities will increase, due to the thickness variation along the flow path. This phenomenon is known as “hesitation effect”. The polymer tends to fill the thicker and less resistant areas, which is enhanced by the increase of viscosity due to premature freezing. There is a demand for a pressure increase, usually achieved at end of filling, combined with the polymer flow to allow a complete filling of the microstructures [61–64].

The influence of process conditions affecting the microcavities filling and quality finish is being studied, namely for high aspect ratio microstructures. The studies are focused on the conditions/parameters which most influence the filling and post filling in μ IM [65], expressed in Table 2.

Table 2 - Process conditions with most influence in cavities filling.

Filling parameters	Post-filling parameters
Melt temperature	Packing time and packing pressure
Mould temperature	Cooling time
Injection speed	Ejection forces

Influence of filling parameters

During the filling phase, the replication of microstructures is strongly related with the polymer viscosity, consequently influenced by temperature and shear rate. A lower viscosity facilitates the flow of polymer melt into microfeatures, and it can be achieved at higher values for all filling parameters [66–71]. It should be established a range where not only quality is not compromised but also the process efficiency is assured.

High barrel temperatures facilitate the polymer melt flow and provides lower pressure drop, assuring the filling of the cavity. The limit temperature must consider a value which avoid polymer degradation. Sorgato and Lucchetta [72] reported that highest melt temperatures improve significantly the replication, as seen in Figure 19a. Also, their results suggested an increment in the replicated height

by combining the effects of highest melt temperature and air evacuation, particularly for high viscosity polymers, such as COC (Figure 19b).

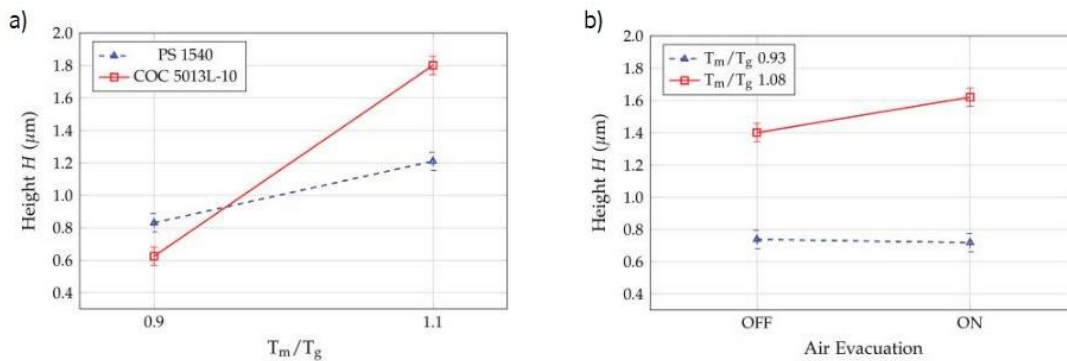


Figure 19 - a) Effect of highest melt temperature (highest T_m/T_g ratio) in PS and COC. b) Effects of T_m/T_g ratio combined or not with air evacuation on replication degree. Results reported in [72].

Conversely to IM, the cooling rate in μ IM is high, because of the low volume/surface ratio causing the earlier formation of a frozen layer along the surface. For this reason, it is important to keep the mould at higher temperatures, to avoid the increase of viscosity. The ideal range of temperatures is above the glass transition temperature of amorphous polymers [73]. Figure 20 shows that an increase of aspect ratio requires higher mould temperatures to achieve a good degree of replication.

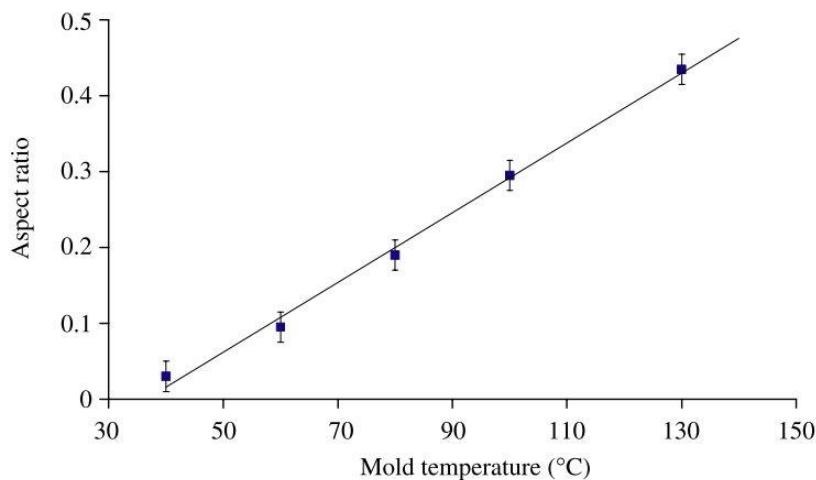


Figure 20 – Aspect ratio results achieved by replication in function of mould temperature. Source: [73].

To avoid an increment of cycle time due to the highest mould temperatures, a variotherm system is an excellent solution to implement in μ IM, allowing high mould temperatures during the filling stage, to allow the filling of microfeatures. Then, it provides a rapid cooling without cycle time increase [74,75]. To reach higher mould temperatures, it is usual the implementation of an electrical heating system. Huang and Tai [76] tested a variotherm system, where the heating was performed until 80 °C by oil heater and until 110 °C by induction. The use of the heating system achieved 95 % of replication degree

of the microstructures height, while the result for a simple oil heating was 89 %. Another study, conducted by Rytka [77], compares the filling of microstructures using isothermal IM (IIM), isothermal ICM (IICM), variothermal IM (VIM) and variothermal ICM (VICM). Analysing the PMMA degree of replication results (Figure 21), despite of a minor improvement noticed between using IICM instead of IIM, the degree of replication rounds 100 % when the variothermal system is used.

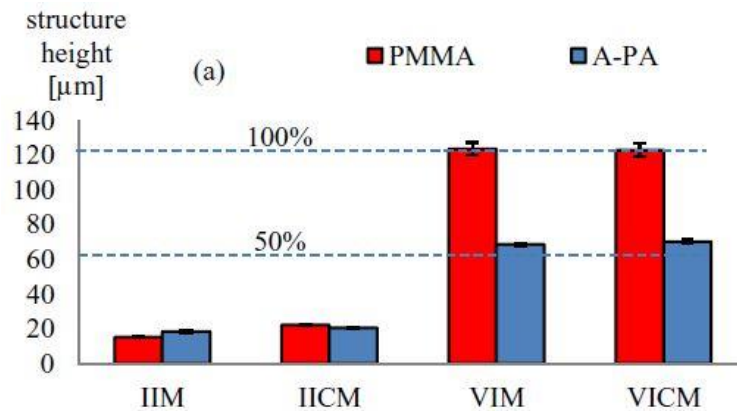


Figure 21 - Replication degree of microstructures of different processes: IIM (isothermal IM), IICM (Isothermal ICM), VIM (variothermal IM) and VICM (variothermal ICM). Source: [77].

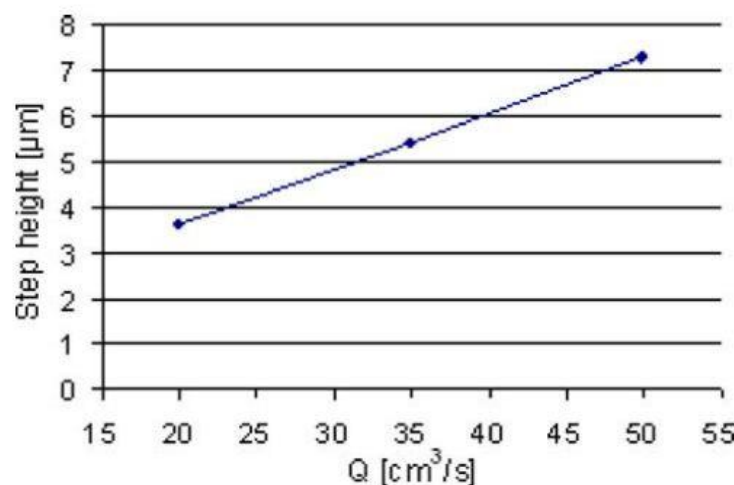


Figure 22 – Microfeature step height in function of the injection flow rate. Source: [69].

The injection speed will determine the filling time of the part and the injection pressure on cavity. High injection speeds lead to high shear rates and, consequently, to shear heating, which allow a decrease of viscosity, preventing premature solidification. It is also important to refer that high injection speeds originates high injection pressures and can result in high internal stresses, not desirable in optical applications. Theilade and Hansen [69] attained higher degree of replication (higher step height) rising the injection flow rate (Figure 22). Song et al. [78] conclude from their design of experiments that the replication of accuracy is uniform in both directions (against and along flow direction) at higher injection

speeds, in contrast with lower injection speeds. Sorgato et al. [79] studied the influence of injection speed and mould temperature on the melt flow front temperature (T_{peak}). In general, higher injection speeds leads to a high melt flow temperature, assuring a low viscosity which improves the filling. The effect is accentuated when combined with higher mould temperatures (Figure 23).

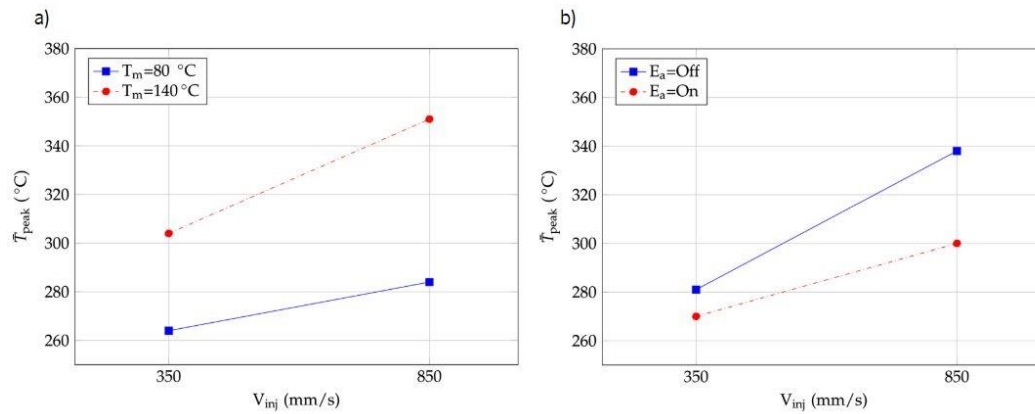


Figure 23 - Influence of the injection speed (V_{inj}) in the Melt Flow Temperature (T_{peak}), interacting with Mould temperature. Source: [79].

In sum, the filling parameters values must concern the highest value to assure the melt flow at the lowest viscosity possible, but in a range to avoid material degradation and other process defects. Also, to improve replication, technologies such as vacuum, to avoid incomplete filling of microstructures and a variotherm system, to avoid cycle time increases, should be employed.

Influence of post-filling parameters

The post-filling conditions has also influence in microstructures replication. After filling stage, the packing stage and demoulding as the main responsible for incomplete replication or damage of the replicated features, respectively.

The packing stage in μIM has less influence comparing to IM, since the part is filled completely with injection stage. However, its influence in microstructures filling is not complete understood and needs some attention.

Park et al. [80] compares the replication degree of microfeatures with 2, 3 and 4 μm of width, in function of packing time and packing pressure. The results (Figure 24) shows that degree of replication is directly proportional to the increase of packing time, achieving better results when the feature has higher thickness.

About packing pressure, in general the replication degree reached to the 0.9 or 1.0 by an increase from 5 to 10 MPa, maintaining the results at higher packing pressures. Only for the microfeature with 2 μm of width, only with 40 MPa of packing pressure provides a replication degree about 1.0.

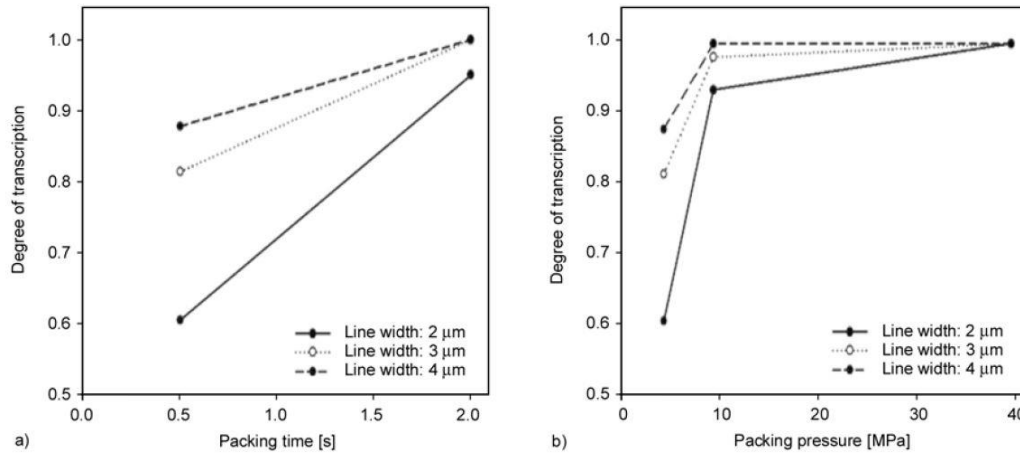


Figure 24 - Replication degree in function of a) Packing time and b) Packing pressure. Source: [80].

Another important factor concerns the demoulding of moulded microstructures, due to its fragility. It is important to assure the correct operation of the ejection system and, also, to minimize the adhesion between the substrate. The importance of selecting the adequate mould insert material or cavities coating is connected with the influence of ejection forces on replicated workpiece [81].

For high aspect ratio structures, a coated mould is helpful to improve surface replication, assuring a fidelity between microfeature insert depth and replicated microfeature. Besides reducing the adhesion effect between the mould and the subtract, coated cavities helps to maintain the accuracy of replication along the cycles.

Using microreplication by hot embossing, Guo et al. [82] identified the major cause of demoulding forces as the friction force, caused by interface adhesion. The adhesion force is defined by the surface energy of the polymer and the insert, so the main objective consisted in the surface energy of the insert reduction and, also, reduction of the coefficient of friction between them.

The authors tested a Ni insert and the replicas showed some defects due to extraction. So, to optimize the process, the authors produced a Ni-PTFE compound material mould insert. The surface energy of PTFE and Ni are, respectively, 0.018 J/m² and 1.7 J/m². Regarding the coefficient of friction between PTFE/polymer and Ni/polymer, the values are, respectively, 0.073 and 0.45. The fabrication method used was an UV-LIGA process, electroforming in nickel galvanic bath, adding PTFE particles later. The authors compare the performance of Ni-PTFE compound mould insert with a Ni mould insert. The

results confirmed an easy demoulding, due to its lower coefficient of friction and an average surface energy. Besides that, the lifetime of the compound increases in comparison with the Ni insert [83].

3 Experimental work methodology

This chapter compiles all the techniques used in the experimental work defined for this dissertation. First, a brief description of samples production techniques will be provided, followed by an overview of samples characterization, namely the evaluation of surface roughness, wettability and optical performance. The protective optical window prototype produced samples were submitted to the characterization tests described in section 3.3.

The experimental procedure of the present dissertation is divided in three studies:

1. Preliminary study of the influence of microstructures configuration and geometry, using improvised hot embossing technique;
2. Preliminary study of the processability of PMMA, by injection moulding;
3. Production of protective optical window prototype samples by injection moulding, using PMMA and the configuration studied in study 1.

3.1 Processing techniques

As pointed on section 2.3.3, hot embossing is the most appropriate technique to obtain quality parts, but it is not profitable in terms of reproducibility. For its reproducibility and ability to produce complex parts, injection moulding was the selected process to produce the prototype of the protective optical window. Nevertheless, hot embossing is advantageous for product development, namely, to test the microstructures geometry and configuration as well the quality of the insert.

For this reason, hot embossing was the technique applied in the first study, while injection moulding was adopted to the studies 2 and 3.

3.1.1 Hot embossing

Since there was not available any hot embossing machine, the process was simulated with an improvised station using a hydraulic press (Figure 25), in the laboratories of *DEP* at University of Minho. The hot embossing station is composed by an upper and a lower plate. The lower plate was the one where the microstructured insert and the sample was placed, so it was heated up above the glass transition temperature. The upper plate was the one which applied the defined pressure and it was heated a little below of the glass transition temperature of the polymer material. The process is similar to the hot embossing, except during demoulding, since this press is not equipped with extraction system, so the part demoulding had to be manual.



Figure 25 - Improvised hot embossing station from a hydraulic press.

3.1.2 Injection moulding

The injection moulding machine used for the studies 2 and 3 was a *FANUC Roboshot α -s15iA* (Figure 26), at PIEP facilities. This machine has a maximum injection speed of 800 mm/s, allowing to reach at lower injection times required for microinjection moulding; a screw diameter of 14 mm, enabling a homogeneous melt with low residence time; a maximum clamping force of 15 tons and is 100 % electrical, so besides the advantages of lowest energy consumptions, it is assured a higher precision due to the electrical driven axis. Due to the characteristics of the protective optical window, a microstructured surface, a higher precision is extremely required to the microstructures filling. In Appendix A, the mechanical specifications of the injection moulding machine are provided.



Figure 26 - Injection moulding machine: FANUC Roboshot α-S151A.



Figure 27 – Mould temperature controller THP from Piovan.

During the process, it was also used a mould temperature controller by water, model THP from PIOVAN (Figure 27). Thus, it was assured the water flow in the mould temperature control system with high precision. During the part production, the mould cavities temperature is maintained high and constant, which is important for microstructures replication, since it promotes the filling of microcavities. The equipment allows a maximum fluid temperature of 140 °C.

3.2 Processing polymers

The polymeric material chosen to produce the protective optical window must fulfil the following specifications:

- Adequate mechanical and thermal properties, given the environment of product operation;
 - Excellent optical transmittance, to not affect LiDAR system performance;
 - Processability, namely a good flow property to facilitate microstructures filling.
- Mechanical properties, namely strain at break, are also relevant, while the part ejection.

Between the materials established for this project, it was chosen two materials to perform the experimental work of the present dissertation:

1. Polycarbonate *Makrolon* (PC) 2407 by *Covestro*;
2. Polymethyl methacrylate (PMMA) *Pexiglas Optical POQ62* by *Evonik Industries*.

Table 3 – Main properties of the materials used in the experimental work.

Main properties	PC <i>Makrolon</i> 2407	PMMA <i>Pexiglas Optical POQ62</i>
Melt volume rate (cm ³ /10min)	19	21
Glass transition temperature (° C)	143	110
Strain at break (%)	120 (50 mm/min)	2.8 (5 mm/min)
Optical Transmittance (%)	89	92

In Table 3 is shown the main and relevant properties of the material used in the experimental work. The PC was only used for the preliminary study 1. To produce the samples of the protective optical window, it was chosen the PMMA, since it has higher optical transmittance in comparison with PC. For this reason, arose the need to study the PMMA processability considering the microstructures filling.

3.3 Samples Characterization

3.3.1 Water Contact Angle measurement

Water contact angle is a direct form to understand if the modification of surface roughness had influenced in the surface hydrophobicity, an important property for self-cleaning purposes.

It was determined the static contact angle, for samples with and without microstructures. The equipment used was an *OCA model 20 (Optical contact angle measuring and contour analysis system)* by *Data Physics* (Figure 28), available at DEP laboratories. The equipment is constituted by a movable platform, where the sample is placed, a syringe and a camera with an associated software.

The sessile drop technique was used. The syringe released the water droplet, with a defined volume and rate of 1 μL and 2 $\mu\text{L/s}$, respectively, and the platform was regulated so that the droplet was caught by the polymer sample. The associated camera captioned the sideways of the drop and its shape was determined by the Young-Laplace method. This method recognizes the influence of liquid surface tension in the drop's shape, being consider the elongation caused by gravity during the release which can influence its shape [84]. The software determined the baseline, allowing its adjustment in case of need, and its contour. This way, it was presented the value of the static WCA.

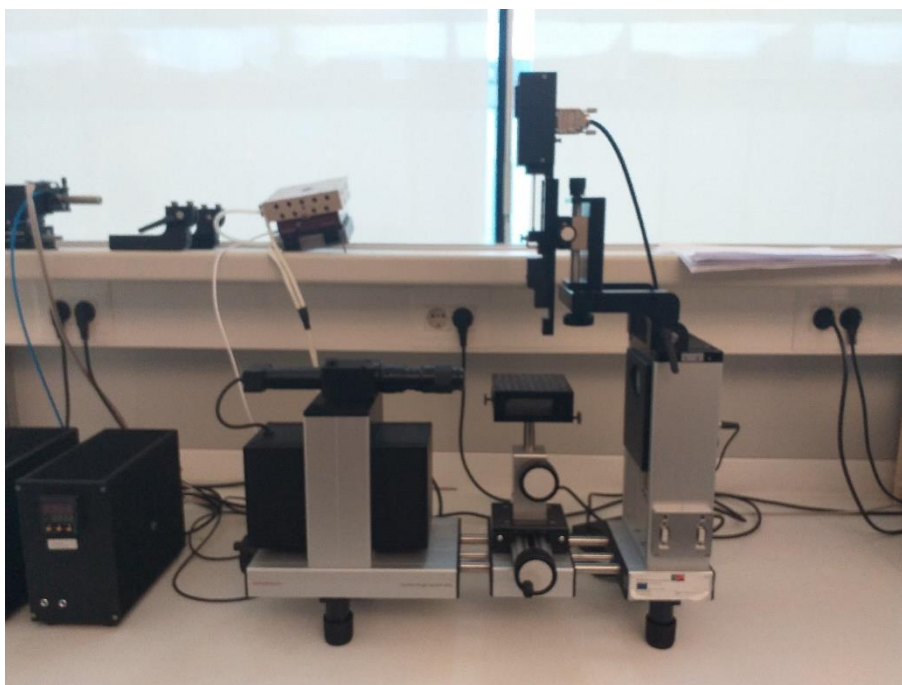


Figure 28 – OCA 20 by Data physics.

3.3.2 Roughness Characterization

To evaluate roughness, the equipment used was an *Infinite Focus SL* by *Bruker Alicona* (Figure 29), available at PIEP facilities. The equipment consists in an optical 3D measurement of surface topography.

The equipment has a measurement field of 50 x 50 mm. The output values are given by tracing a line, with a specific width, and the software calculates the amplitude parameters through the profile roughness over the line length. The most relevant parameters are described below [85]:

- Ra (Arithmetic average height) is the arithmetic absolute average value of the profile heights variation relative to the mean line. A_i corresponds to the area of each peak or valley.

$$Ra = \frac{1}{n} \sum_{i=1}^n |A_i| \quad (3.1)$$

- Rq (Root mean square roughness) corresponds to the standard deviation of height distribution, being more precise than Ra when occurs a large deviation from the mean line.

$$Rq = \sqrt{\frac{1}{n} \sum_{i=1}^n A_i^2} \quad (3.2)$$

- Rz (Ten-point height): it is selected the five highest peaks and the five lowest valleys along the profile length. Rz corresponds to the difference between the average of the selected values, in agreement with ISO system. This parameter is more sensitive than Ra when the profile has more height variations.

$$Rz = \frac{1}{n} \left(\sum_{i=1}^n p_i - \sum_{i=1}^n v_i \right) \quad (3.3)$$

In Figure 30, it is represented an example of output profile from Alicona software, where it was also represented the definition of Ra and Rz. The arrows indicate the five highest peaks and the five lowest valleys, separated by the mean line (in red), used to calculate Rz. The filled peaks/valleys represent the areas which enter in the calculation of Ra.

Besides roughness parameters, the Alicona software allows a profile form measurement, being possible to measure directly the height or depth of peaks or valleys, as seen in Figure 31. This functionality was often used to characterize the samples of the study 2. It was possible to export the data for profiles comparison.



Figure 29 - Infinite Focus SL by Bruker Alicona.

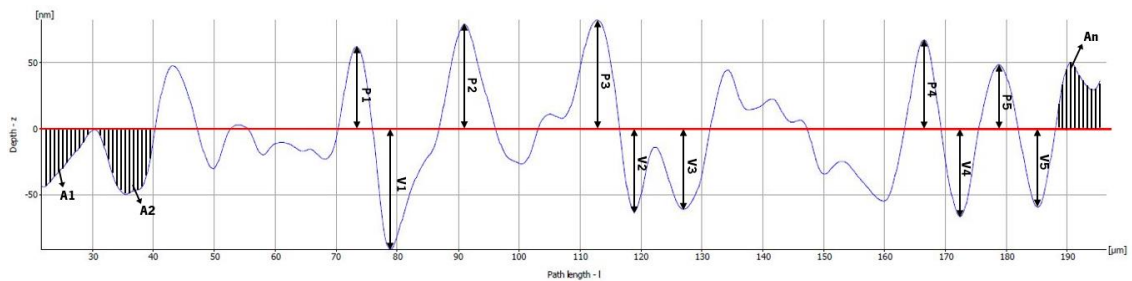


Figure 30 - Example of an output profile from the Alicona software and representation of the definition of R_a and R_z .

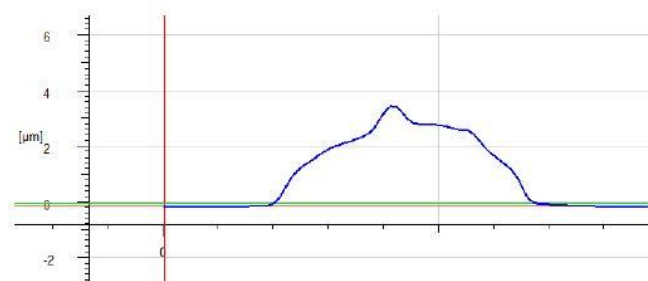


Figure 31 - Example of an output profile from the Alicona profile form measurement functionality.

3.3.3 Scanning Electron Microscope (SEM)

Scanning Electron Microscope (SEM) is a technique used to obtain a three-dimensional image, scanning the surface through an electron beam. When the electron beam is focused in the sample, some electrons are reflected as high energy electrons and others are absorbed, emitting secondary low energy electrons. The electrons which leave the sample are captured by a collector, showing the image in display. The luminosity intensity depends on the electronic emission in an area [86]. Through this technique, it was possible to observe the replicated pattern on samples as well the measurement of microstructures height.

The SEM characterization was performed at SEMAT, University of Minho, and the equipment used was a *FEI Nova 200* (Figure 32). In addition, a chemical analysis was performed, with the associated equipment *EDAX Pegasus X4M*, to clarify if the samples have contaminations, such as metal particles from the inserts, which can induce errors in WCA measurement. The polymer samples had to be coated with gold, to avoid the charging effect, because of polymers are not conductive.



Figure 32 – SEM characterization station at SEMAT: FEI Nova 200 and EDAX Pegasus X4M equipment.

3.3.4 Optical Transmittance

The most relevant property of a protective optical window consists in the efficiency of light transmittance, assuring the proper functioning of LiDAR device.

Transmittance is defined as the fraction of incident light of a specific wavelength which passes through a sample. It is a numerical value with a wide range between 0 % (no light is transmitted) and 100

% (all light is transmitted) [87]. The equipment used was a spectrophotometer from *Shimadzu*, model UV-2401PC (Figure 33).



Figure 33 – Spectrophotometer model UV-2401PC by Shimadzu.

3.4 Moldex3D simulations

CAE software products are used in plastic injection moulding industry to validate plastic parts project, optimize part design and process conditions. *Moldex3D* was the CAE tool used during experimental work, to aid in engineering tasks analysis. It offers solutions in injection moulding and non-conventional techniques, such as injection-compression moulding, bi-injection moulding, between others.

The simulation process starts in *Moldex3D Designer*, where it is possible to import the part geometry to create the feed system or to specify the melt inlet point. Next, it is specified the mould base and the cooling system and, finally, it is possible to generate a mesh. After this initial work, the mesh is imported to *Moldex3D* software, followed by the material and process conditions selection. The software has a database for several polymeric materials, which include their thermal, mechanical, and rheological properties. There is also a database for injection moulding machines, with information of their injection and clamp units as well the screw dimensions. It is expected to obtain results closer to the reality using these databases. Despite the model *Roboshot α-s15iA* was not available on machines database, the software allowed the addition of the main characteristics (such as screw diameter, maximum injection pressure, maximum clamping force and maximum shot weight) to the *FANUC* group machines.

The process method set was “*Machine mode 1 (by profile)*”, where the injection time is determined by the injection flow rate profile and packing settings. This method allows the interaction with

machine interface (Figure 34) of *FANUC*, where it is possible to input the same parameter values used in the experiences.

The image shows a screenshot of the FANUC machine interface, which is a software control system for injection molding machines. The interface is divided into several sections, each with a title and a set of input fields and buttons.

- INJECT Section:** This section is at the top left. It has a title "INJECT" and a "step" indicator set to "2". Below the title, there are five columns labeled "5", "4", "3", "2", and "1". The "2" column has a value of "110" and the "1" column has a value of "90". The units are "mm/s". Below these, there are fields for "MAX INJ PRS" (2500 kgf/cm²), "INJECT SW" (20 mm), and "TRANS MODE" (POS TRANSF, 10 mm). There is also a "MAX INJ PRS" field set to "MLTI".
- PACK Section:** This section is in the middle left. It has a title "PACK" and a "step" indicator set to "1". Below the title, there are three columns labeled "1", "2", and "3". The "1" column has a value of "815" and the "2" column has a value of "3". The units are "kgf/cm²" and "sec". Below these, there are fields for "HR MODE" (1), "STD" (2), and "BEF EXT" (0, 3).
- TRANS MODE Section:** This section is in the middle right. It has a title "TRANS MODE" and a "step" indicator set to "1". Below the title, there are fields for "MAX INJ TIME" (sec), "MAX PACK VEL" (mm/), "DWELL BEF INJ" (sec), "MAN. EXT." (mm), "BACK PRS" (kgf/cm²), and "ROTATION" (RPM). There is also a "MAX INJ PRS" field set to "MLTI".
- EXTRD Section:** This section is at the bottom left. It has a title "EXTRD" and a "step" indicator set to "1". Below the title, there are fields for "BACK PRS" (kgf/cm²), "ROTATION" (RPM), "SWITCH P." (25 sec), and "COOL TIME" (25 sec). There is also a "SHOT" field set to "35 mm" and a "DECOMP." field set to "4 mm".

The interface is designed with a blue background and white text. The input fields are rectangular boxes, and the buttons are labeled with their respective functions. The overall layout is organized and easy to use.

Figure 34 - Machine interface of *FANUC* available on "Machine mode 1 (by profile)" process method.

After verifying the computation run data, the model is ready for analysis. *Moldex3D* permits the simulation of filling, packing, warpage and stress analysis, providing several information which can help to explain experimental data results or in the part design validation.

4 Experimental techniques and methods

This chapter comprises the description of the techniques used in the experimental work, concerning the productive tools. It was used two different injection moulds, which had to be adapted to the injection moulding machine. Also, it will be described the production method of the microstructured inserts, used in studies 1 and 3 of the experimental work.

4.1 Injection moulds

This subchapter concerns to the injection moulds used during experimental work. Besides a brief description of their structure, it will be detailed the required modifications to adapt these moulds to *FANUC Roboshot α -s15iA* (Figure 26). All parts used to modify the moulds were produced or rectified using subtractive manufacturing, at DONE Lab facilities, University of Minho. It was used the *CNC* technology aided by *SolidCAM* extension from *SOLIDWORKS* software. This subchapter is divided into two sections, to describe the two different moulds used in studies 2 and 3:

- Micromould: used to study the processability of PMMA, in scope of the study 2 of the experimental methodology.
- IM Prototype mould: used in scope of study 3, to produce samples of the protective optical window prototype.

4.1.1 Micromould

The micromould (Figure 35), used to study the processability of PMMA, is stored in microinjection laboratory at DEP processing laboratory. This mould has a standard base structure and it was projected to adapt different mould inserts. A CAD design, with identification of the main components of the micro mould are available in Appendix B (Figure 104 and Figure 105).

The micromould has the versatility to produce with hot runners or cold runners (section view available in Figure 106, Appendix B). It had been used with hot runners, but the experiments to perform require cold runners, so there were some alterations and acquisitions to make in order to not having problems during processing, that will be listed below.

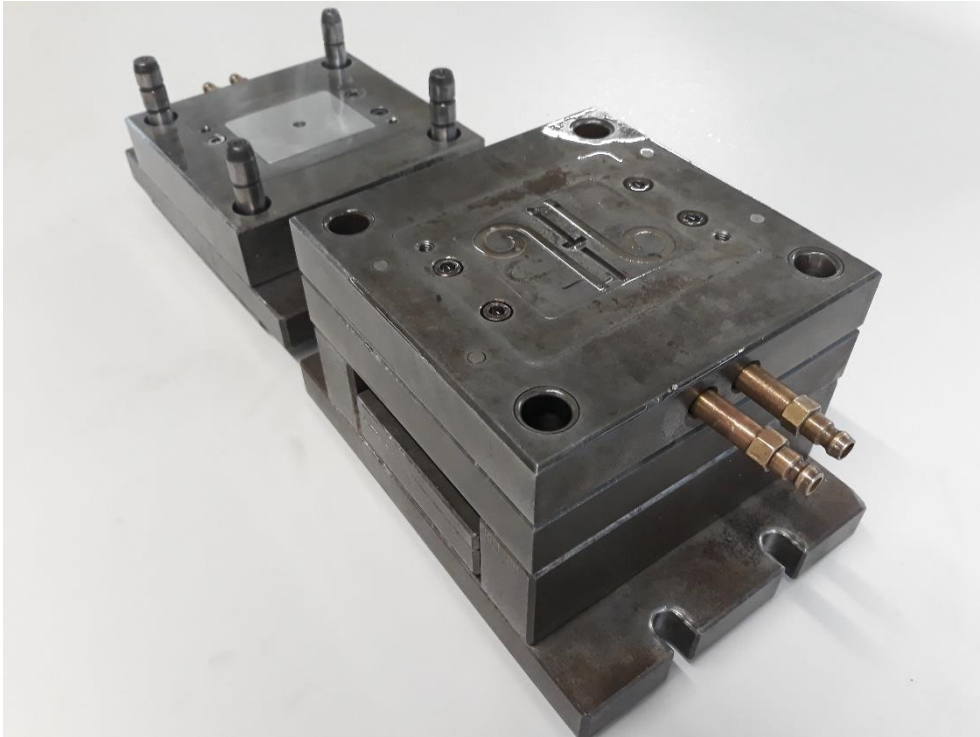


Figure 35 – Micro mould afterwards maintenance and modifications.

First, the mould original sprue bushing was not available, so it was purchased a standard one by *HASCO*. In accordance to the holes machined in the tightening plate and moulding plate, whose dimensions were according to the nozzle of the machine for which the mould was projected, it was chosen the sprue bushing referenced by *Z511/24x76x3x15,5* (Figure 36).

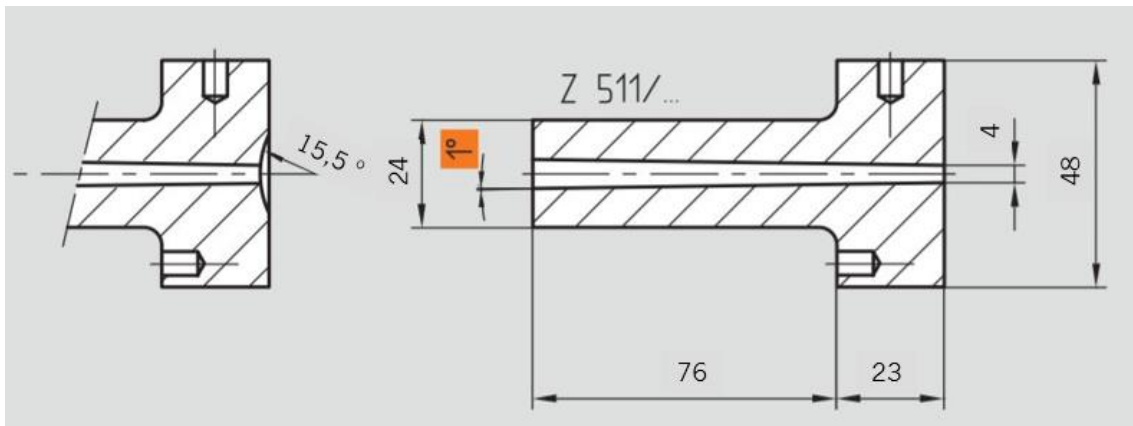


Figure 36 – Dimensions of selected sprue bushing. Original scheme by *HASCO*.

The micromould had a locating ring of 90 mm diameter, while the FANUC locating ring diameter is only 60 mm. So, a new locating ring was produced conform to the dimensions of the machine platen (upper side, Figure 37a) and the hole machined of the tightening plate from the injection side of the micro mould (ring base, Figure 37b). In ring base, it was machined a tab to accommodate the sprue bushing.

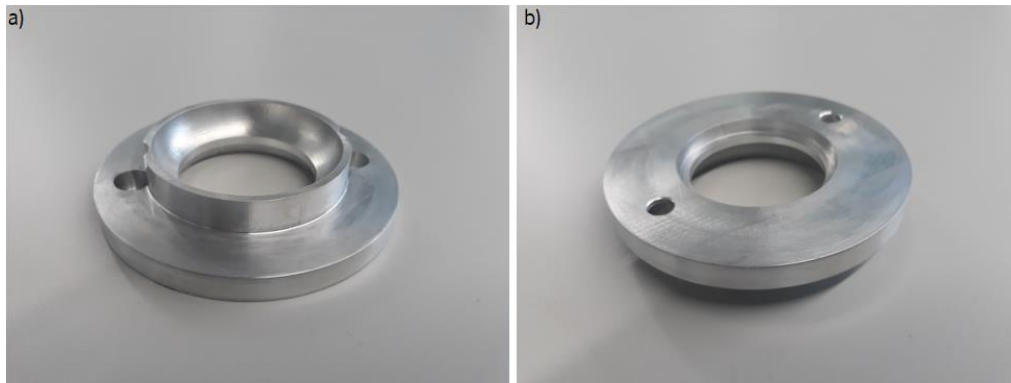


Figure 37 - Locating ring in aluminium. a) Upper side and b) ring base.

At last, it was produced a new flat insert for the injection side (Figure 105, Appendix B), also in aluminium. The original insert has a locking tab, useful for the machine nozzle lean against it when the mould was used with hot runners, which will impede the ejection of the sprue when used with cold runners. In the new insert, the locking tab was eliminated, and it was machined a hole with an exit angle, which is an extension of the sprue. In Figure 38, it is possible to see both inserts.



Figure 38 - Flat inserts. On the left, the original insert with the locking tab. On the right, the new insert.

In Figure 39, it is presented the final part design of the micromoulding and respective feed system.

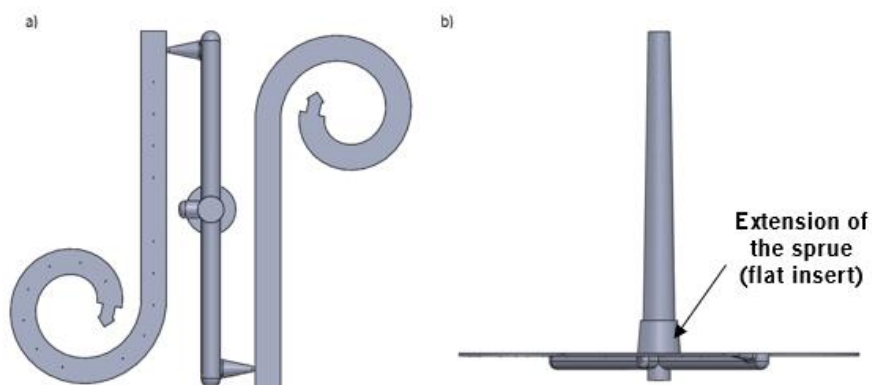


Figure 39 - Final micromoulding afterwards the alterations: a) microparts, runners and gates, b) sprue and sprue extension (from the flat part).

4.1.2 Prototype mould

The prototype mould was projected for development of a *protective optical window*, in a previous dissertation inserted in a previous project [88]. It has the capability of to produce two parts per cycle with dimensions of 70 x 50 x 2 mm (Figure 40). Bellow, a summary description of the relevant components and systems will be given. The CAD designs of the structure and some of the components are available in Appendix C.

The mould has cold runners and is composed by the sprue, two main runners with parabolic shape which connects the sprue to the mouldings through fan gates (Figure 40). The runners shape allowed the machining only into one of the moulding inserts plates, on the ejection side. On the injection side, the plate was polished in moulding areas to provide adequate surface finishing (Figure 41). However, before to perform injection moulding, the surface has to be clean, due to the marks in the polished area.

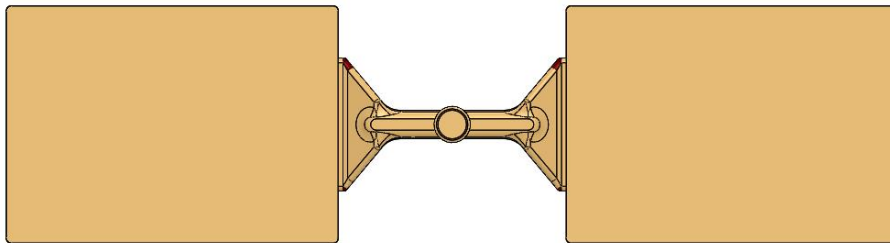


Figure 40 - Projected moulding of prototype mould, with two parts connected by the main runners.



Figure 41 - Polished surface on the injection side.

Since the mould is particularly to develop a microfeatured component, a vacuum system is indispensable, to avoid defects or incomplete filling of microfeatures, caused by trapped air. Thus, it was

installed the *Vacuumjet* + device, by *CUMSA*, to avoid problems caused by the presence of gases, when the melt flows into the cavities.

This system required the machining of a closed circuit around the cavity, to allow the gas escaping through a vacuum hole, where the device is connected. The thinner ribs, located where the flow ends, facilitates the air escaping from the cavities. Its depth concerns the viscosity of the processed polymers as well the probability reduction of defects occurrence. Another crucial element to ensure the correct performance of the device is the sealant O-ring, placed around the escaping gas circuit. Its function is to isolate the cavities from air entrance after the mould closes. This way, the device eliminates the air which could be trapped after mould closure.

However, the mould was developed for the injection moulding machine *ENGEL victory 50*, whose maximum injection weight and clamping force values are superior comparing with *FANUC Roboshot α -s15iA*. For this reason, the mould had to be adapted to the selected machine, making two modifications:

1. One piece per cycle instead of two.
2. Reduction of part size from 70 x 50 x 2 mm to 50 x 50 x 2 mm.

The first change was made by blocking out the flow path to one of the cavities. It was produced, in aluminium, a workpiece with the shape of the runner which was glued into the channel.

To reduce the part length, an aluminium block with dimensions of 20,57 x 50 x 10 mm (Figure 42) was manufactured. Since this workpiece was placed in the location of the end of filling, it was necessary to make the ribs for gas escaping.

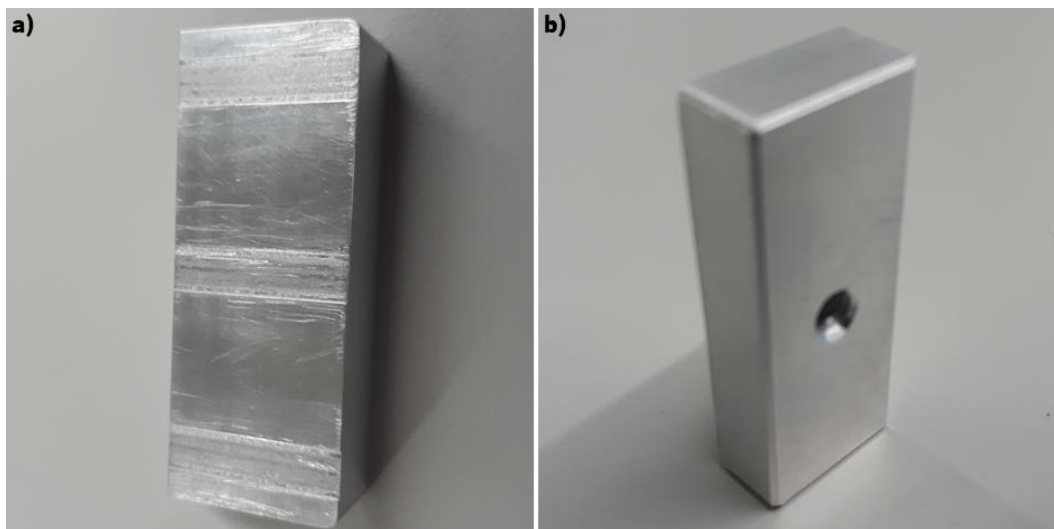


Figure 42 - Aluminium block produced to reduce cavity length: a) front surface, with craved ribs for air escaping and b) back surface, with a thread to fixation in the mould.

Also, it was cut two types of steel inserts: one to produce flat samples and another to nickel deposition to form the microstructured surface (Subchapter 4.2.2). In Figure 43, there is a picture from middle of the process, after machining the thread holes to insert fixing in the mould.

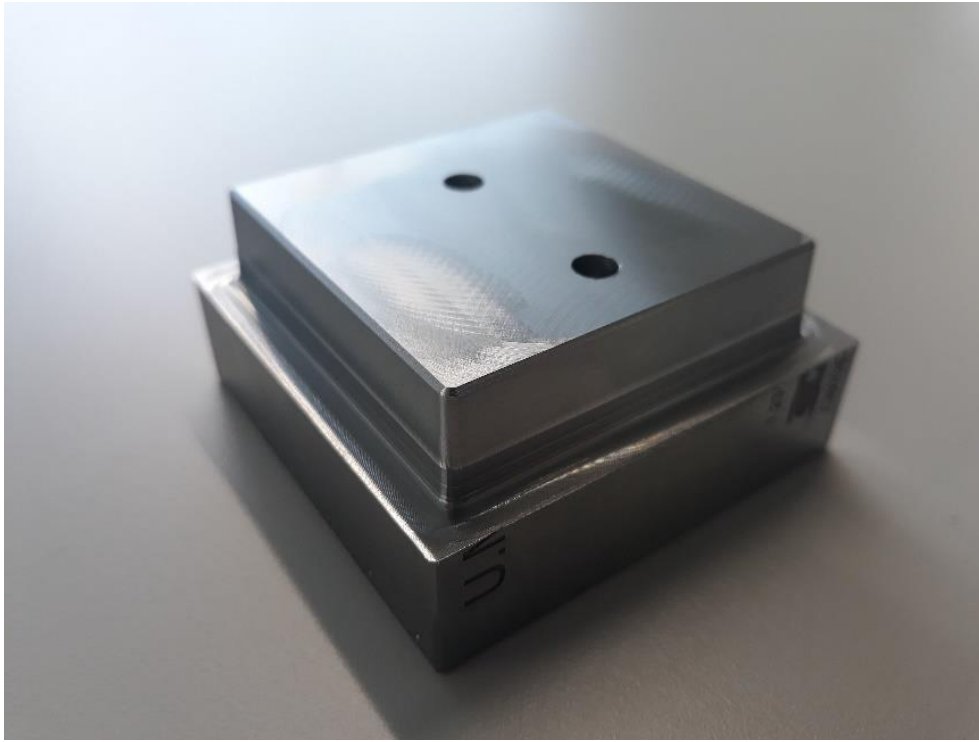


Figure 43 – Steel insert after machining the thread holes.

The final rectifications of aluminium and steel blocks was handmade, to adjust the blocks dimensions to the mould cavity where they will be fixed, whose dimensions differ from CAD design. The assembly of these new components with the mould are possible to see in Figure 44.



Figure 44 – Assembly of the new components with the prototype mould.

In conclusion, the changed moulding consists in a flat plate part with approximated dimensions of 50 x 50 x 2 mm, as seen in Figure 45, plus the feeding system.

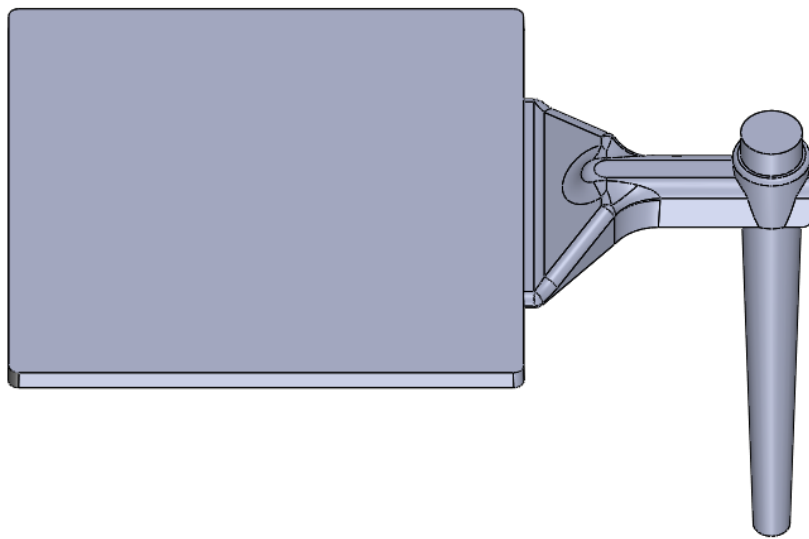


Figure 45 - Final moulding, part and feed system, after prototype mould modifications.

4.2 Microstructured inserts

During the development of a product, it is important to have flexibility in the used tools. Using a mould insert, a cavity which can be easily replaced and adapted to a tool, such as an injection mould, is advantageous. In this case, since one of the goals is the possibility to test various structures with different configurations, a mould insert is advantageous to avoid the replacement of the entire plate.

The mould insert material should be easy to manufacture, facilitating the creation of micro- and nanopatterns and should have excellent mechanical properties to support the pressures involved in the process techniques used for microreplication. Among several metals, the most used to fabricate microstructured mould inserts are silicon, nickel, aluminium, between others, mainly because of the easy manufacturing [70].

Pereira et al. [89] tested the configurations summarized in Table 4, using Silicon inserts. Due to its fragile nature, several problems arose in the attempts of microstructures replication. Using injection moulding, the insert broke while the injection phase due to the higher pressures characteristics of the IM process. When hot embossing was used, the Si inserts broke during demoulding, which was manual.

At this point, it was decided to test another material insert, less brittle, in order to understand the process conditions to obtain samples with higher hydrophobicity. The defined specifications for this new insert are represented in Table 5. It was chosen a circular base for the microstructures, since the edges of a squared base could complicate the demoulding.

Table 4 - Summary of previously tested configurations and geometries for microstructures.

Shape and size	Depth (μm)	Pitch (μm)
Squared Base (Side = 5 μm)	10	15
	10	25
	15	15
Circular Base (Diameter = 5 μm)	15	15

Table 5 - Dimensions and geometry of microstructures selected to the new insert.

Shape	Diameter (μm)	Depth (μm)	Pitch (μm)
Circular base	5	10	15

In this subchapter, it will be provided a description of the production method of the copper mould insert, used in study 1, and the nickel mould insert, used in study 3.

4.2.1 Copper insert

The first insert material tested was Copper and it was only used for the preliminary study of structures configuration and geometry, by hot embossing. This material was selected due to its availability and knowledge in create microstructured patterns with Cu. The insert production process was performed at INL facilities.

It was produced a Cu shim, with four sections (inserts) with dimensions of 70 x 50 mm. The method of production, schematized in Figure 46, consists in replication of micromachined substrates of silicon to copper, by deposition of copper particles. The process began with imprinting lithography, in a Si substrate, a negative of the microstructures selected pattern, reproducing its diameter and pitch. Next, to achieve the desired depth of 10 μm for microcavities, the Si substrate was submitted to DRIE (deep reactive ion etching), typically used for high-aspect ratio, followed by the elimination of the remains of lithography process by using plasma. The process of replication to copper was initiated by sputtering, when a 300 nm thickness of Cu seed layer was deposited, followed by an electroplating process until it was achieved a shim with 1 mm thickness. The process was finalized by the dissolution of the Si substrate with an aqueous solution.

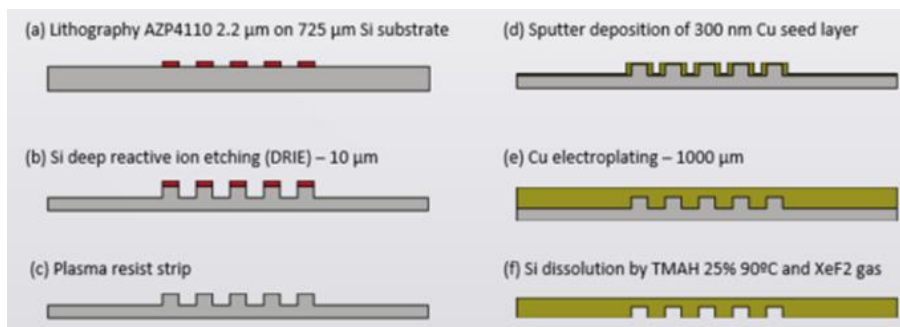


Figure 46 – Scheme of process method applied to copper shims production.

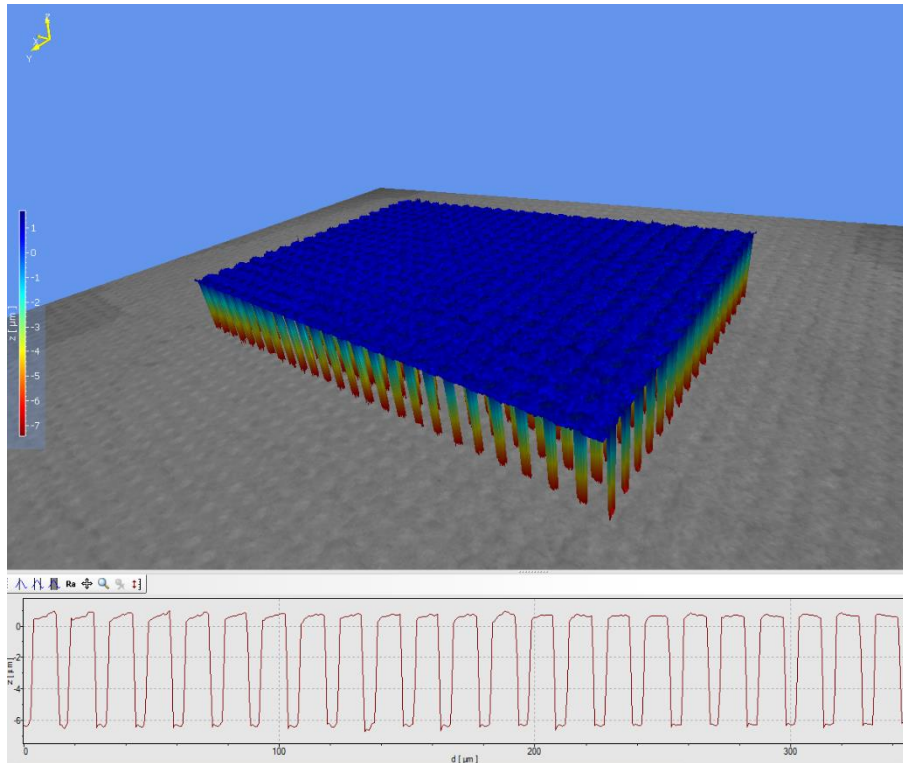


Figure 47 – Surface analysis of Cu insert, performed by INL.

In Figure 47 are presented the results of a surface analysis of the Cu insert, performed by INL, where seems to have a difference between the measured cavities depth, around 6 μm , and the desired depth of 10 μm . These results suggest that the desired depth was not achieved during the process or it occurred measurement errors, caused by double reflections in microcavities walls, as represented in Figure 48.

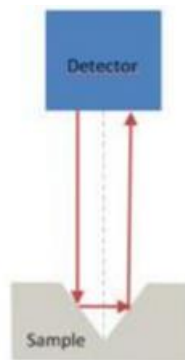


Figure 48 – Schematic of double reflections which cause measurement errors [90].

The Cu insert was only used only for preliminary tests due to its ductibility, amplified by its 1 mm of thickness. In addition to the easy distortion, it also does not allow the integration in the available tool for injection moulding (Subchapter 4.1.2).

A SEM analysis of the Cu insert surface was also performed. The results (Figure 49) shows that the surface, besides the required micropattern, presents a nanostructure, including inside the microcavities. This characteristic can influence the demoulding after replication.

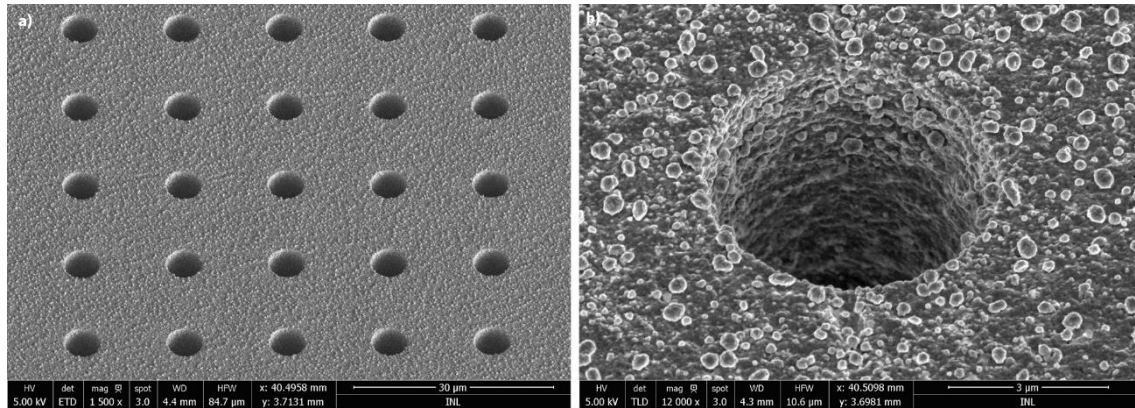


Figure 49 – SEM images of Cu insert, with different magnifications: a) small area of the micropattern and b) microcavity.

4.2.2 Nickel inserts

After the preliminary tests, where the microstructures configuration leads to satisfactory results, the next step were the study of microstructures filling by injection moulding, the preferential technique to produce the protective optical window. Therefore, the mechanical properties of the insert must be accurate to support the higher pressures used in the injection moulding process. Thereby, the aforementioned steel inserts (Subchapter 4.1.2) was used as nickel deposition substrate to reproduce the required micropattern.

The nickel deposition was performed by *Thunder NIL*, an Italian company specialized in the microtechnology domain. Before sending the substrates to the company, their surface has to be polished to correspond the roughness parameters requirements ($R_a = 0.03 \mu\text{m}$ and $R_z = 0.12 \mu\text{m}$) requested by *Thunder NIL* to allow the nickel deposition. The polishing process was performed by *CENTIMFE*.

The method to produce the micropattern was UV lithography. In Figure 50 is shown the principle followed by the lithography processes [91]. The process begins with the placement of the photoresist, a light-sensitive material, to create the desired pattern on the subtract. Next, the photomask with the pattern is placed, whereas the substrate is exposed to radiation to change the solubility of photoresist material. After to create the desired pattern, where is defined the shape and size of the features, it is performed an etching process, to define the height or depth of the feature, by removing the surface layers not covered by the photomask. The procedure ends with the dissolution of the photoresist exposed to the UV light, in a solution named as the developer.

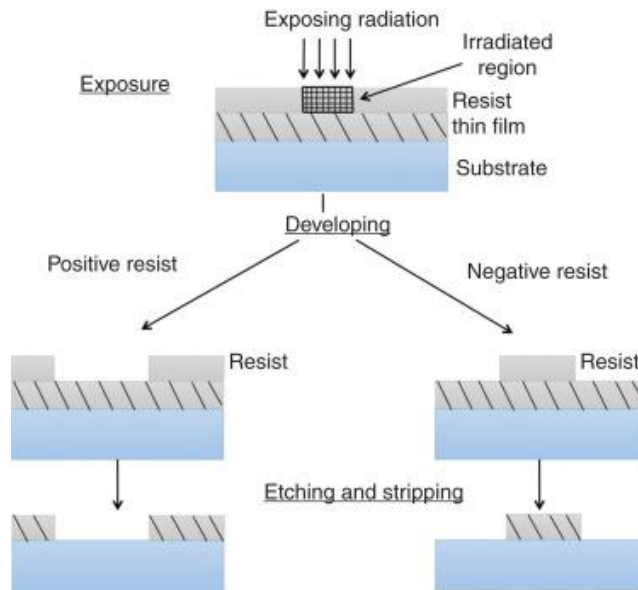


Figure 50 – Process method for fabrication by UV lithography [92].

The photomask can be a positive resist or a negative resist. When a positive resist is used, the mask contains a copy of the pattern to be fabricated. When exposed to the UV light, the photomask will suffer chemical changes in their structure, becoming into more soluble when the substrate is submerged into the developer, replicating the photomask pattern. On the case of negative resist, the photomask contains a negative duplicate of the pattern to be fabricated. The effects of light will initiate a crosslinking process, so the mask becomes more chemical resistant to the developer, resulting in the removal of the areas which are not exposed to the mask [93].

The initial plan of the company to reproduce the required microstructured pattern (Figure 51a) was the use of a positive resist, but it was not possible to achieve almost vertical side walls, wherefore the tone of the photomask had to be reverted to use a negative resist (Figure 51b). After optimize the UV lithography process to obtain pillars with enough height, it was started the nickel deposition, controlled step by step through SEM analysis, to reproduce the requested height (Figure 51c) and to avoid the deposition over the mask. With the negative resist, it was achieved pillars with an upper side larger than the base of the microstructure, which is favourable in demoulding step. Ended the deposition, it was used ultrasonic frequency vibrations to clean organic residual on the formed holes (Figure 51d), and it is obtained a microfabricated insert.

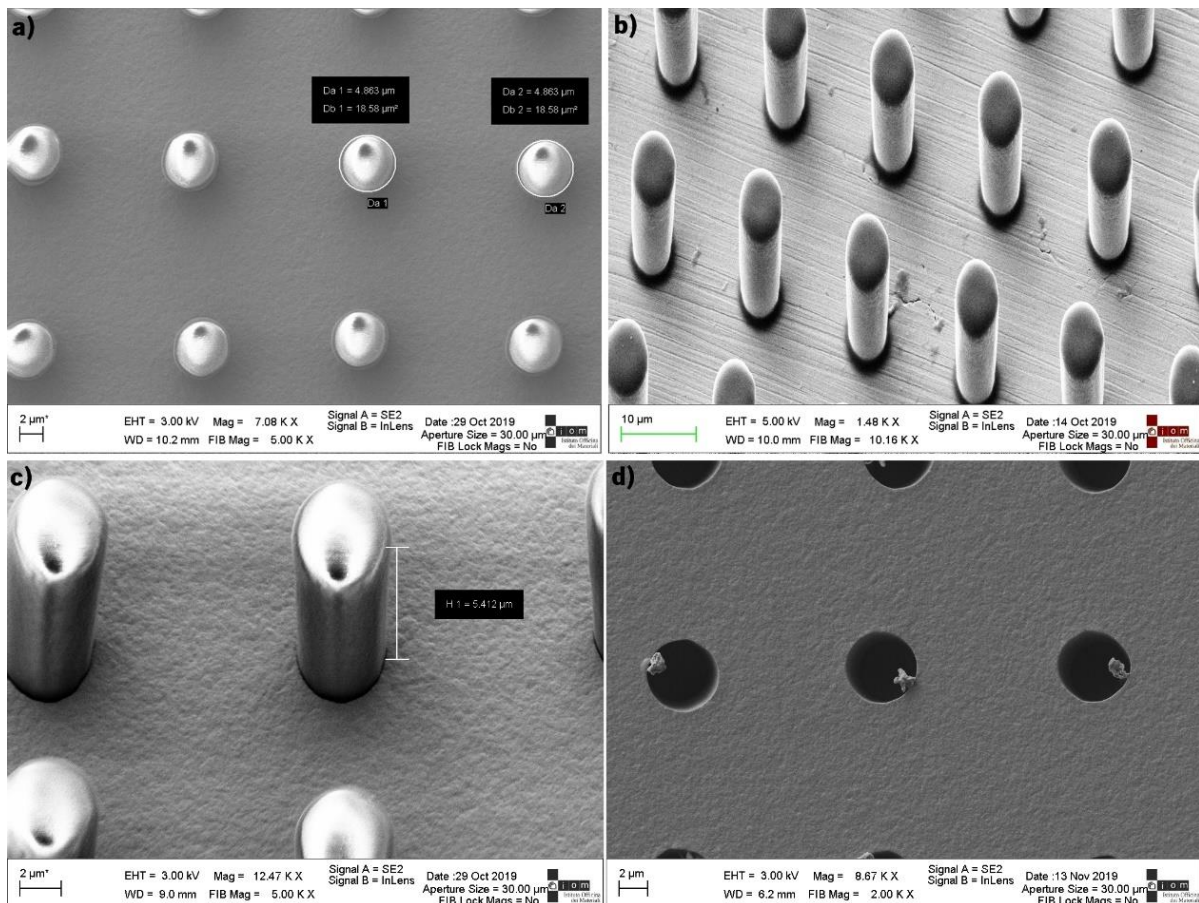


Figure 51 - SEM images of some process steps: a) Upper view of the pattern, b) photomask with negative resist, after finished, c) nickel deposition process and d) after nickel deposition, before cleaning the organic residuals. Provided by Thunder NIL.

5 Results and discussion

The results concerning the experimental work are shown on this chapter, which is divided in three subchapters corresponding to the three studies of the experimental work.

The results obtained in study 1, by improvised hot embossing, showed that the studied configuration influenced the surface hydrophobicity (subchapter 5.1). Then, the PMMA processing was studied, due to its excellent optical transmittance (subchapter 5.2). Finally, the protective optical window prototype samples were produced by injection moulding, using the validated configuration and PMMA material (subchapter 5.3).

5.1 Preliminary study of structures configuration and geometry for self-cleaning surfaces

The hydrophobicity is strongly related with the surface characteristics, namely its roughness. Morphological studies point the hierarchical structure of lotus leaf surface as the responsible for water repellence. For this reason, biomimetic surfaces have been developed, to reproduce the leaves structure.

In this first study, the goal consists into reproduce a microstructured pattern composed by pillars, to evaluate the microstructures height, pitch and base dimension influence in the surface hydrophobicity. As described in chapter 3, the processing technique and processing polymer applied were improvised hot embossing and PC, respectively. The process conditions are available in Table 6. It was also evaluated the effect of the embossing pressure in replication quality.

Table 6 - Process conditions applied to PC samples.

Process Conditions		
Embossing temperature (° C)	Upper plate	85
	Lower plate	165
Time (min)	Pre-heating	5
	Embossing	15
Embossing pressure (ton)	Condition 1	4
	Condition 2	6
Demoulding temperature (° C)		85

Another goal for this study consists in to test the Cu as material to produce the negative of the pattern and the quality replication in polymer samples. A similar configuration, which distinguish from the tested in this phase because of having squared base, was tested using Si inserts, with the same technique

and polymer [89]. The fragile nature of the Si caused problems during demoulding, breaking and contaminating the samples. Despite of this difficulties, it was possible to obtain well defined microstructures in small areas, with around 80 % of replicated height. Also, the samples showed evidences of pulled out microstructures, caused by demoulding. For this reason, the microstructures shape changed to circular in the copper insert.

After the improvised embossing, the first test was the profile roughness measurement, to understand if the structures were replicated in the semi-finished samples. Comparing the profile of a sample without microstructure with those with microstructures (Figure 52), there were possible to observe changes at the surface, namely the microstructured pattern replication, an indication that the polymer flowed into the microcavities. The profile roughness values of the samples (Table 7), measured in the microstructured zones of the replicated area indicate an increase of roughness, specially noticed in the samples produced with higher pressure.

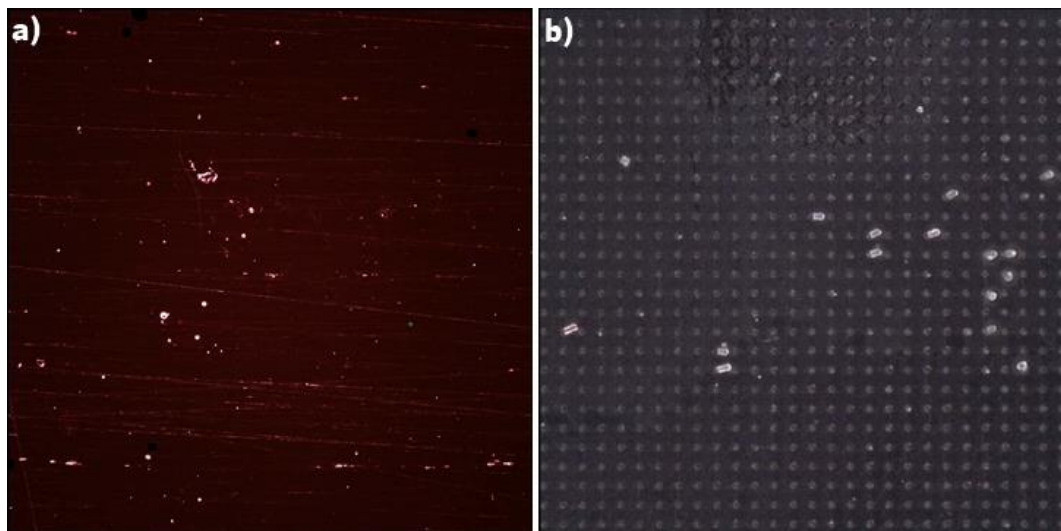


Figure 52 - Surfaces images observed with Alicona equipment. a) before replication b) after replication.

Table 7 - Values obtained through profile roughness measurement.

	Ra (nm)	Rq (nm)	Rz (nm)
Without microstructures	22.05	28.58	78.94
With microstructures (4ton)	891.06	1097.13	1976.42
With microstructures (6ton)	70.21	1135.26	2698.66

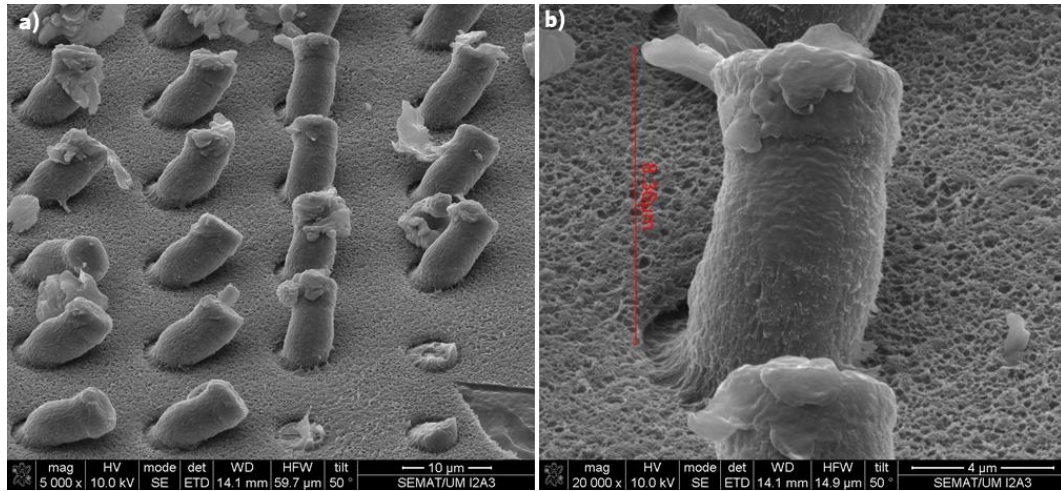


Figure 53 – SEM images of PC replicated sample, with (a) 5000x and (b) 20000x of magnification.

Scanning the surface through SEM, a clearest microstructures image was provided to determine its dimension. As shown in Figure 53b, micropillars with 8.36 μm of height were observed, although the height can be lower, due to the neck formation and the tilt of the plate where the sample was placed. The insert nanoscale roughness was also replicated in samples, creating a hierarchical roughness.

It was also possible to identify some defects associated to demoulding (Figure 54). The adhesion between the polymer and the mould insert, accentuated by the replicated nanoroughness, can justify these problems.

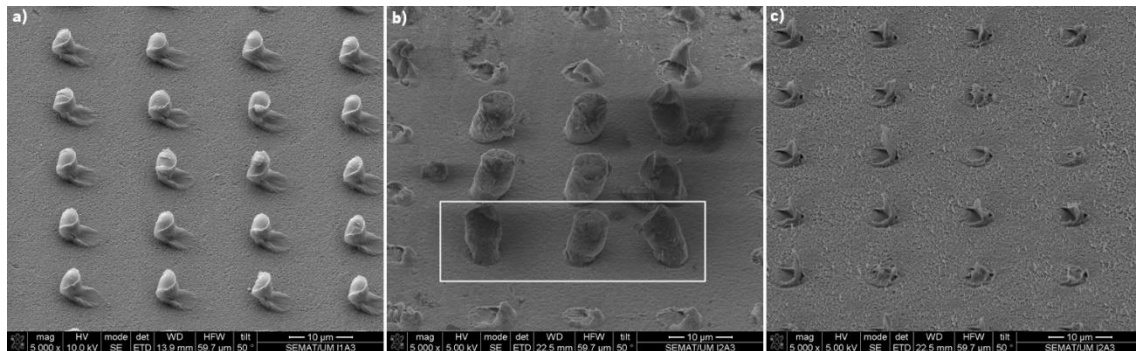


Figure 54 – Defects associated to demoulding: a) distorted microstructures, b) neck formation and c) pulled out microstructures.

The insert was also scanned using SEM, after four hot embossing cycles. The microstructures valleys were filled with polymer (Figure 55a), which was confirmed by the chemical analysis (Figure 109, Appendix D), confirming the presence of carbon (C) and oxygen (O) in Z1 area, identified in the figure. This result was considered, since the microcavities could be filled at first hot embossing cycle and affect the replication in the next production cycles.

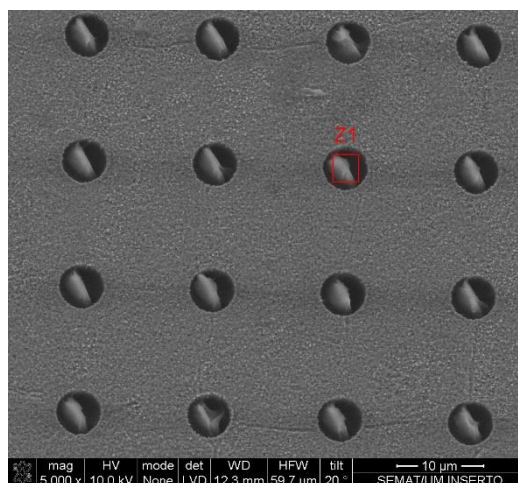


Figure 55 – SEM image, with 5000x of magnification of copper insert microcavities filled with polymer.

Next, to understand if the increase of roughness had effect in hydrophobicity, the water contact angle was measured. The WCA increases, in average, 34 %, independently of the pressure applied by the hot embossing station, as it is shown in Figure 56. The results suggested an enhancement of hydrophobic property, although the microstructures were not replicated in an entire area and, even so, the water contact angle increased.

The maximum measured WCA value was 133 ° (Figure 57). To exclude the hypothesis of surface contamination, it was performed a surface chemical analysis (Figure 110, Appendix D), which confirm that the surface was free of copper particles that could influence the measured WCA.

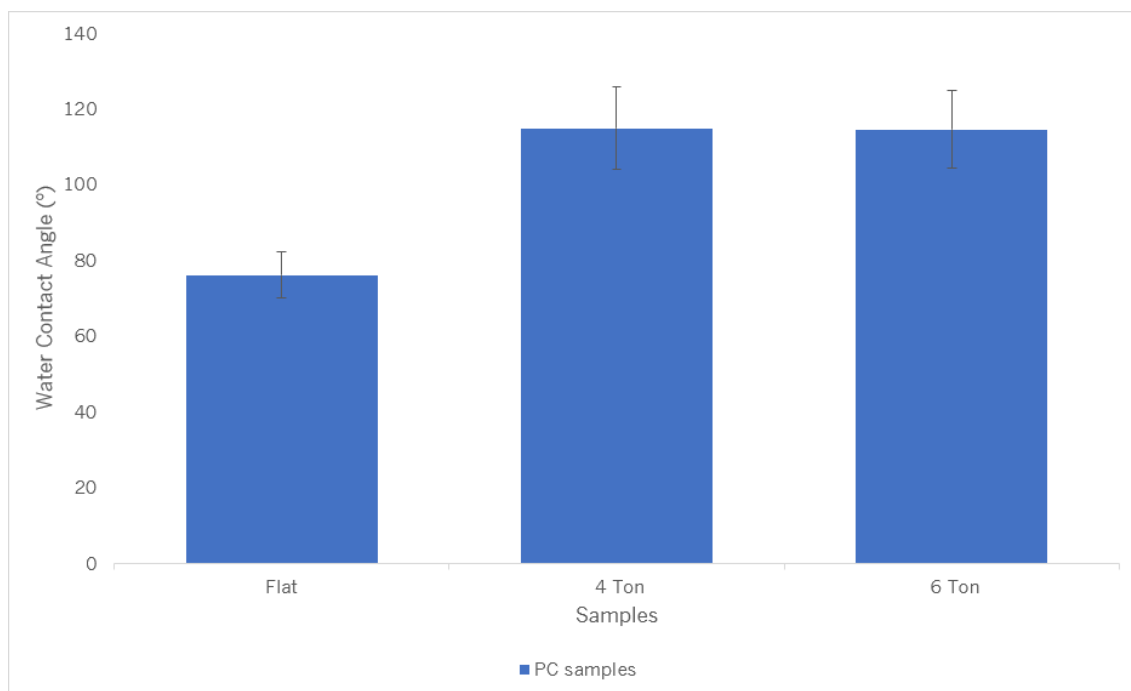


Figure 56 – WCA values measured on PC samples.

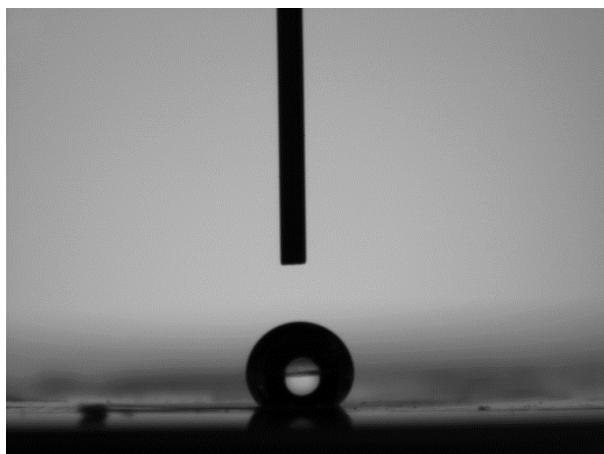


Figure 57 – Surface area with a measured WCA of 133 °.

Since it was obtained a microstructured surface, with a satisfactory replicated height resulting in changes in surface hydrophobicity, the next step was to evaluate if the microstructures affected the optical transmittance performance. As seen in Figure 58, the optical transmittance significantly decreased comparing the sample without microstructures with the microstructured samples. The transmitted light values decreased about 23 %, also influenced by the roughness of the plates where the sample was placed during the embossing process.

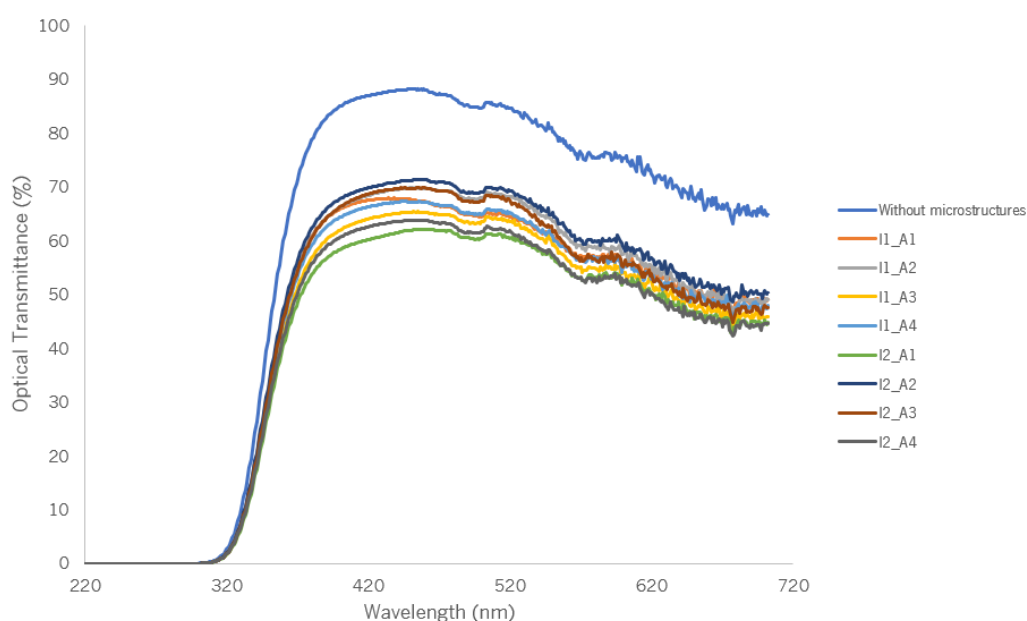


Figure 58 - Optical transmittance in function of wavelength for PC samples.

These preliminary results allowed to validate the studied microstructured pattern to be replicated polymer samples and change the surface hydrophobicity. The next step goal is to achieve a good replication degree by injection moulding. However, the used copper insert is not suitable for injection moulding, due to its fragility and the impossibility to adapt it to the available injection mould. So, a new

insert had to be produced and it was performed injection moulds whose replication results are represented in the subchapter 5.3.

5.2 Preliminary study of PMMA processability by injection moulding

The optical performance of optical devices such as LiDAR is strongly related with the intrinsic properties of the processing material. Among the selected polymers, the optical transmittance of PMMA is superior compared with the PC but there are more problems while processing. For this reason, a preliminary study focused on PMMA processability during microstructures filling was performed.

As for the replication of microstructured substrates, the filling of microstructures is a complex phenomenon, since the melt flow is affected not only by the process conditions but also by the substrate geometry. There is a need to study the influence of the process conditions on the replicated microstructures height along the substrate flow path and relate it with the distance from the gate.

Based on the literature review of the most influent parameters in the replication degree of microstructures (Section 2.3.4), mould temperature, injection speed and holding pressure were chosen to be studied. These parameters were studied through a DOE, whose values for each level are represented in Table 8. In Table 9, there are represented the fixed values of the experiments. Both group of values was set based on manufacturer recommendations of the polymer, suggestions of experienced workers and by some preliminary mouldings.

Table 8 – Studied parameters and respective values of the design experiments.

Level	Tmould (°C)	Sinj (mm/s)	Phold (bar)
-	60	110	200
+	90	150	800

Table 9 - Values of fixed parameters of the design of experiments.

Fixed parameters	
Melt temperature profile (°C)	255 – 260 – 255 – 250 – 50
Cooling time (sec)	25
Holding pressure time (sec)	3

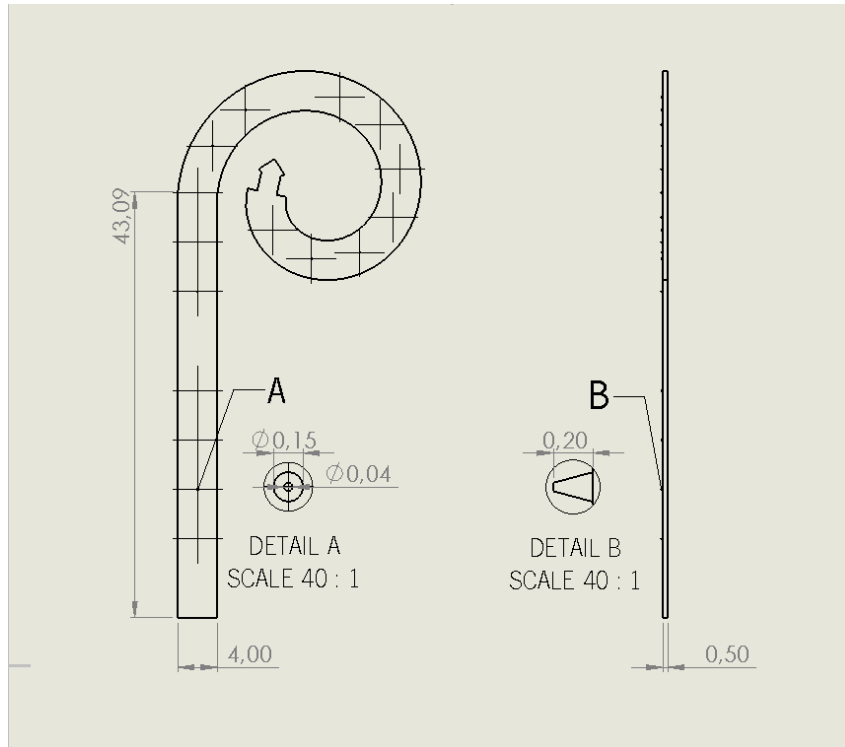


Figure 59 - 2D drawing of the case of study. Detail A: microstructure front view magnification and Detail B: microstructure side view magnification. Dimensions in mm.

A 2D drawing of the case of study are represented in Figure 59, a substrate of 0.5 mm of thickness with sixteen microstructures placed along the part surface. In details A and B, the microstructures dimensions are specified.

DOE execution and data treatment

It was executed a regular two level full factorial experimental design, with three factors and eight experiences (runs). For each run, it was collected twelve samples, being selected five of them to be analysed.

Before defining the studied parameters, the production of samples at lower melt temperature profiles was attempted, which resulted in incomplete fillings due to the low melt viscosity. Since for lower melt temperatures the required injection pressure is higher, it leads to sprue overpacking and, consequently, the sprue got stuck. During the processing at higher melt temperatures, this problem was verified again due to material flow from the nozzle, because of agglomerated polymer in the barrel. This problem was solved through screw decompression and by purging to remove degraded material.

The DOE response variable is the replicated height of microstructures, which was measured using the functionality of profile form measurement available in *Infinite Focus SL* (Section 3.3.2). The microstructures selected to study are encircled and identified according to its position along the flow path

in Figure 111 (Appendix E). Some microstructures were not included because it was not possible to accurately measure their height, due to some defects associated to sample ejection, such as distortion, or incomplete filling due to insert contamination, observed at the end of processing.

Alicona software allows to export the data to *.txt* files. For the considered microstructures, the data was exported to *Excel*, to be treated and to be possible to collect the points for comparison of replicated height. Because of the uneven substrate, due to warpage defects, the profile must be adjusted to the plot axes, to be possible to compare their height in microstructures centre. Figure 60 indicates the reference points used to align and to overlay the profiles, pointed by the arrows. The blue line corresponds to the insert profile form and the orange line to the replicated microstructures profile form. After profiles alignment, the profiles height was collected through the reference points marked in Figure 60 (below).

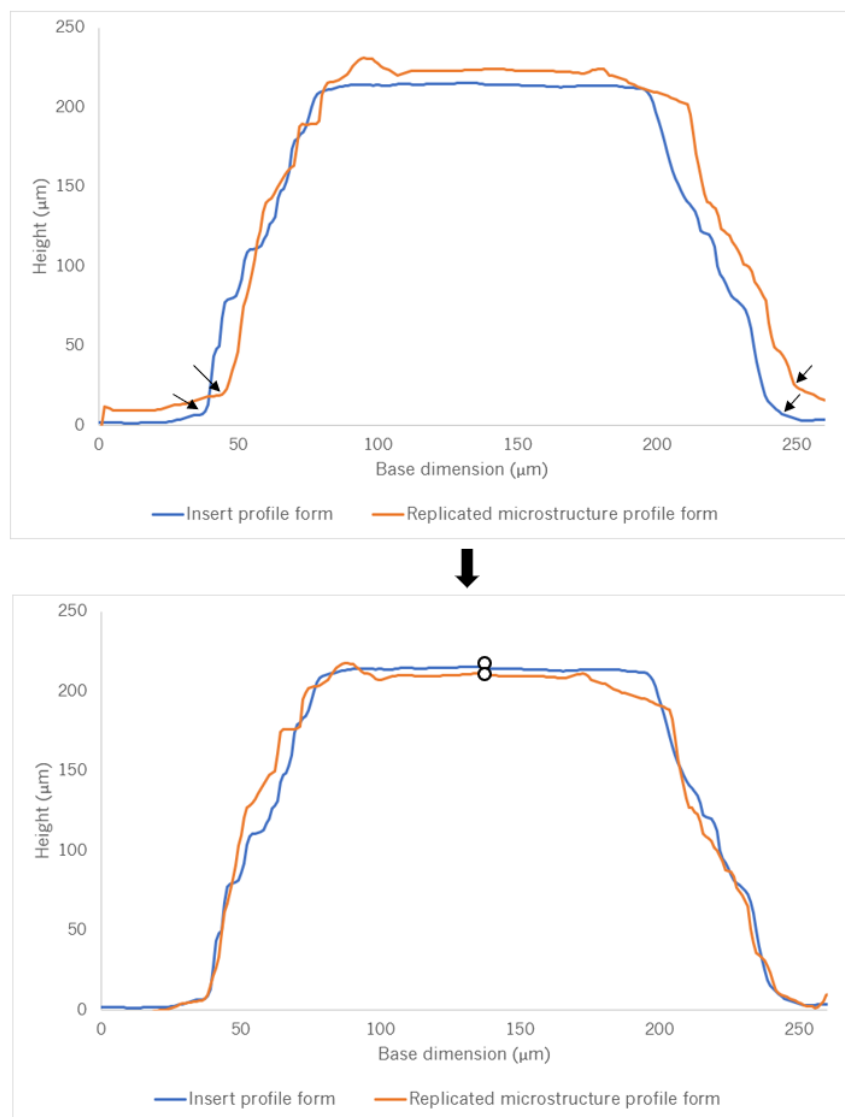


Figure 60 - Schematic of the data treatment for microstructure height measurement.

Through a qualitative analysis of the produced samples, it was observed the main defects occurring in the process [94], represented in Table 10. Warpage phenomenon is verified for the highest mould temperatures, an indication that the applied cooling time was not enough, and the ejection temperature was too high. The existence of flash defect can have two explanations, since it is verified in all experiments: or the melt temperature is too high, being necessary to find a balance to avoid incomplete fillings, or the moulding plates are not correctly adjusted in the parting line. About air pockets, since it occurred when the mould temperature is high, a possible justification is the incomplete filling of the cavity, due to low dosage. Finally, for Runs 6, 7 and 8 the parts were too brittle, which leads to moulding breaks next to the ejector closer to the gate, was caused by incorrect operation of the ejection system. In Figure 61 it is possible to see these defects in some of the produced samples.

Table 10 - Summary of the defects found in the produced samples for each run.

Run	Flash	Warpage	Air pockets	Brittle parts
1	x	x	X	
2	x	x		
3	x			
4	x	x	x	
5	x	x	x	
6	x			x
7	x			x
8	x			x

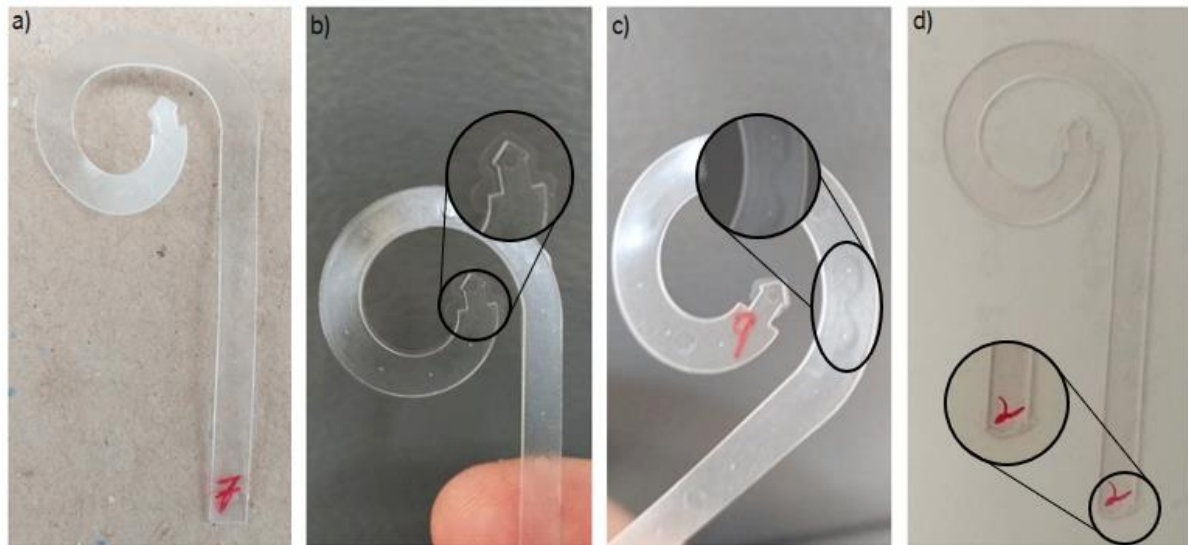


Figure 61 - Identification of the main and frequent defects: a) warpage, b) flash, c) air pockets and d) moulding breaks due to brittleness.

5.2.1 Experimental results: replicated height analysis

The results were analysed with *Design-Expert 12* software, which automatically plots the main factors effects and their interactions. The input replicated height on software, represented in Table 11, is an average value of the replicated height of the five samples. For each sample, the value corresponds to an average of the height measured of the studied microstructures. In Appendix G, the representation of the measured microstructures height and the respective standard deviation of each sample for the 8 experiments are represented (Figure 118 to Figure 125).

Table 11 – Parameters values by run and results obtained through DOE.

	T_{mould}	S_{inj}	P_{hold}	Replicated height	Std. Dev.
Run	(°C)	(mm/s)	(bar)	(µm)	(µm)
1	90	110	800	188.04	13.59
2	90	150	200	203.40	2.36
3	60	150	800	207.08	5.26
4	90	150	800	205.38	3.18
5	90	110	200	204.92	3.07
6	60	110	200	209.15	2.10
7	60	110	800	200.81	2.53
8	60	150	200	209.32	2.53

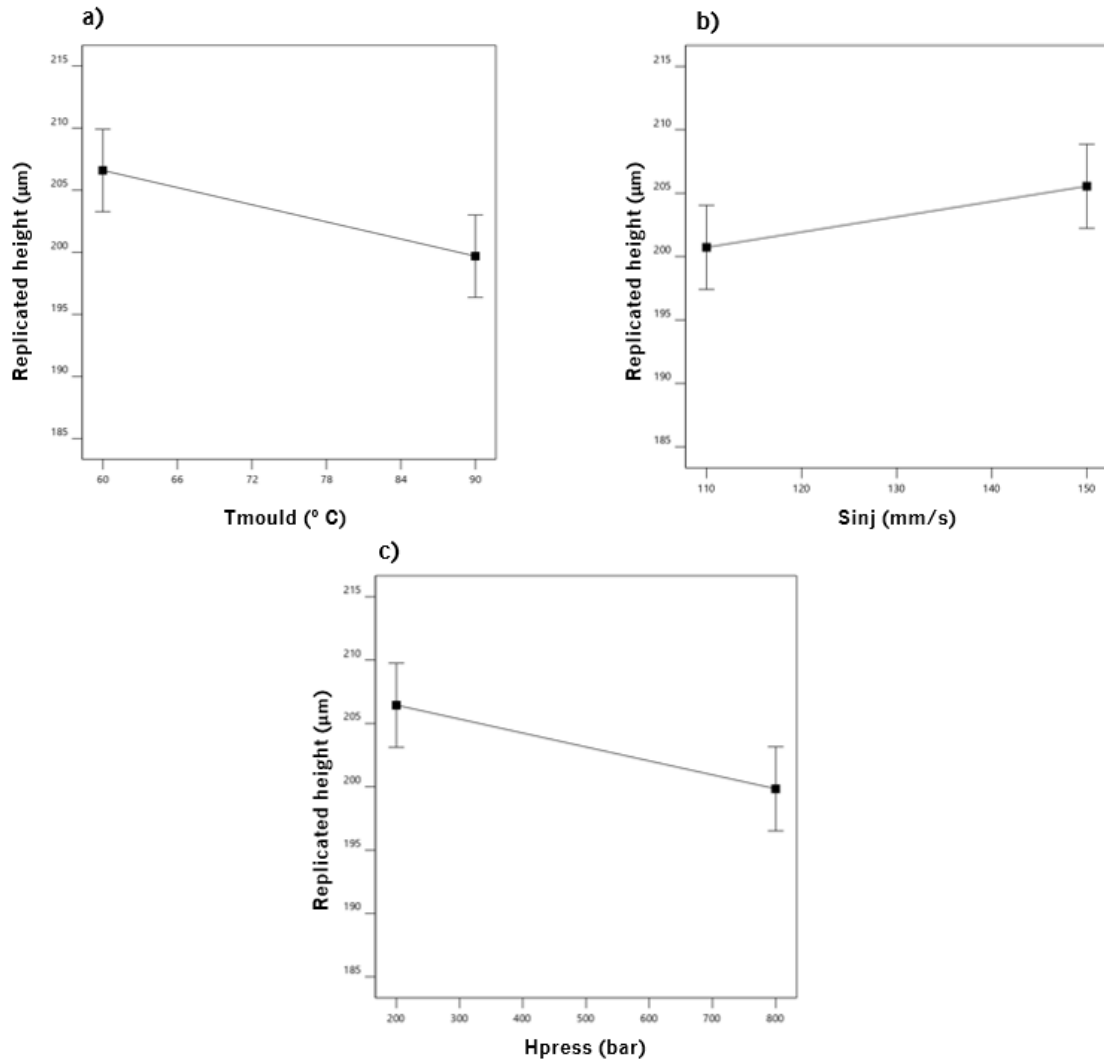


Figure 62 – Main effects plots for a) Mould temperature, b) Injection speed and c) Holding pressure.

Figure 62 shows the main effects plots. According to the obtained data, the replication is favoured by lowest values of mould temperature and holding pressure and highest values of injection speeds. The results associated to mould temperature were contradictory, since literature indicates mould temperature as a main factor for replication: high mould temperatures avoid the increase of viscosity, preventing the premature freezing and, consequently, an improvement of replication degree. Nevertheless, it is possible to explain these results looking upon the average values of linear shrinkage for each run.

Although it was not used as a response of the DOE, the thickness of the microstructured substrate was measured. With this data, it was determined the linear shrinkage (S) for each run:

$$S (\%) = \frac{l_w - l_f}{l_w} \times 100, \quad (5.1)$$

where l_w corresponds to the dimension of mould cavity (depth) and l_f to the dimension of the moulded part (thickness). In Figure 63 is represented the linear shrinkage for each run, whose values are

abnormal to PMMA, usually estimated between 0.3 and 0.6 % [95]. The shrinkage values are higher for mould temperatures of 90 °C, an indication that mould temperature is elevated or not balanced. This data also justifies the DOE effects results, since the samples present high shrinkage values which influence the measurement of microstructures height. Also, the shrinkage can be justified with non-uniform cooling, since the moulding cavities are from different materials, steel and aluminium, with different thermal conductivity.

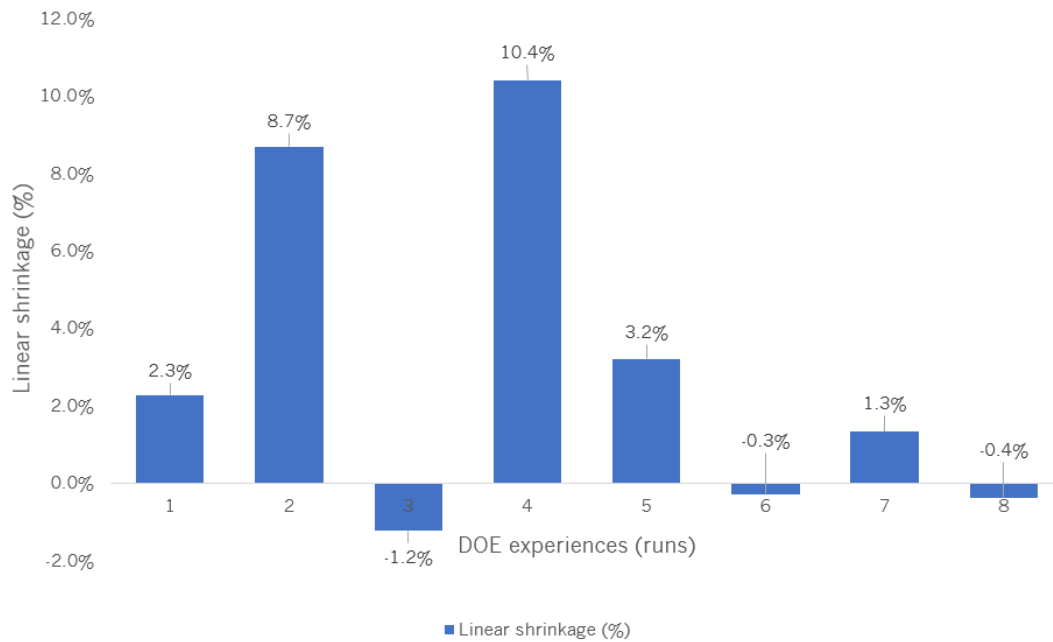


Figure 63 - Results of linear shrinkage for each experiments of DOE.

As seen in the graphic of Figure 63, for Runs 3, 6 and 8 the shrinkage has negative value, an indication that the samples expanded instead of shrinking, another phenomenon influencing the microstructures height measurement. The reason for samples expansion came from excessive packing pressure or packing time, since the substrate only has 0.5 mm of thickness. However, the study purpose intended the influence of holding pressure on microstructures replication.

Attending the moulding shrinkage, the effects were not completely clear. The process should be repeated with a lower range between the experience levels, preferentially after a micromould maintenance, to correct the problems detected during this preliminary test. However, it allows to acquire experience and sensibility to the used machine and material, useful for the experimental work whose results are specified in the subchapter 5.3.

5.2.2 *Moldex3D* simulation results

The design of experiments was also executed by simulation, using *Moldex3D* software, to understand the software fidelity in predicting the microstructures filling and, if possible, to compare with

the experimental results. In Figure 64 and Figure 65 are represented the filling results for Run 3 and 4, with mould temperature of 60 °C and 90 °C, respectively. The filling results for the other runs are available in Appendix F, Figure 112 to Figure 117. Conversely to experimental results, the simulation results indicates incomplete filling when the lowest mould temperature is used.

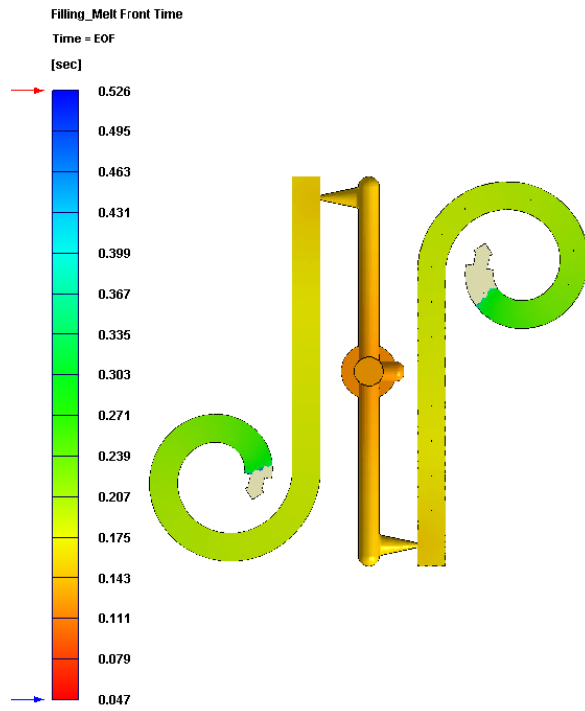
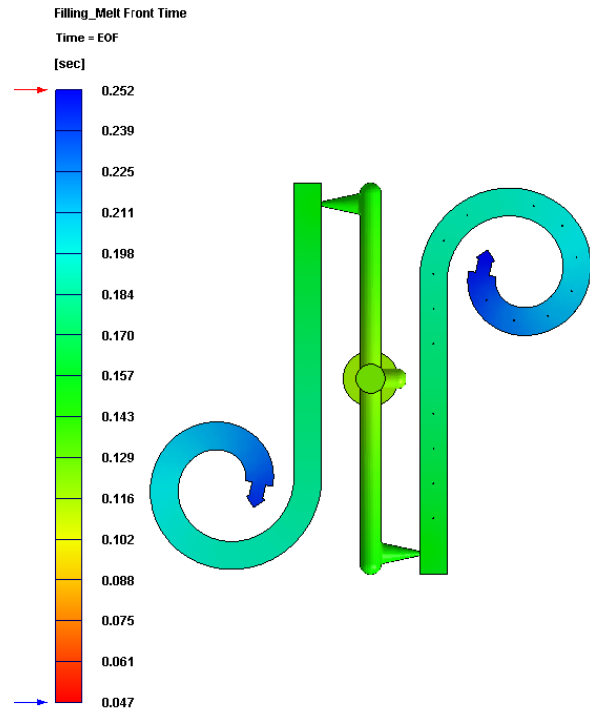


Figure 64 - Simulation filling results for the conditions of Run 3.

Figure 65 - Simulation filling results for the conditions of Run 4.

Simulation results shows the microstructures completely filled, although the simulation data can help to predict and withdraw some conclusions about the filling during the real process. Looking at the flow in the microstructures (Figure 66), it is possible to observe that the melt front advances along the substrate and the complete microstructure filling starts after the melt passes through the microstructure base. This concerns to the hesitation effects, caused by the difference of thickness between the substrate and the microstructure. In practice, due to the frozen layer formed when the melt meets the mould walls, can result into an incomplete filling of microstructures, due to the high viscosity.



Figure 66 – Flow behaviour in microstructures filling (Molded3D simulation).

In relation to the influence on microstructures replication considering the distance from the gate, the melt behaviour at the microstructure base allows to corroborate some literature results [96,97]. Considering the filling results of the Run 5, it was analysed along the sixteen microstructures, the evolution of shear rate, which is related with the generated viscous heating which facilitates the microstructures filling. In Figure 67 it is represented the shear rate at the time of the microstructures filling as a function of the flow length corresponded in each microstructure. As seen, the shear rate correspondent at the microstructures filling, manifested as the maximum shear rate at the selected point on the microstructures, decreases in function of the distance from the gate, associated to pressure drops. Theoretically, viscous heating decreases like the replication degree.

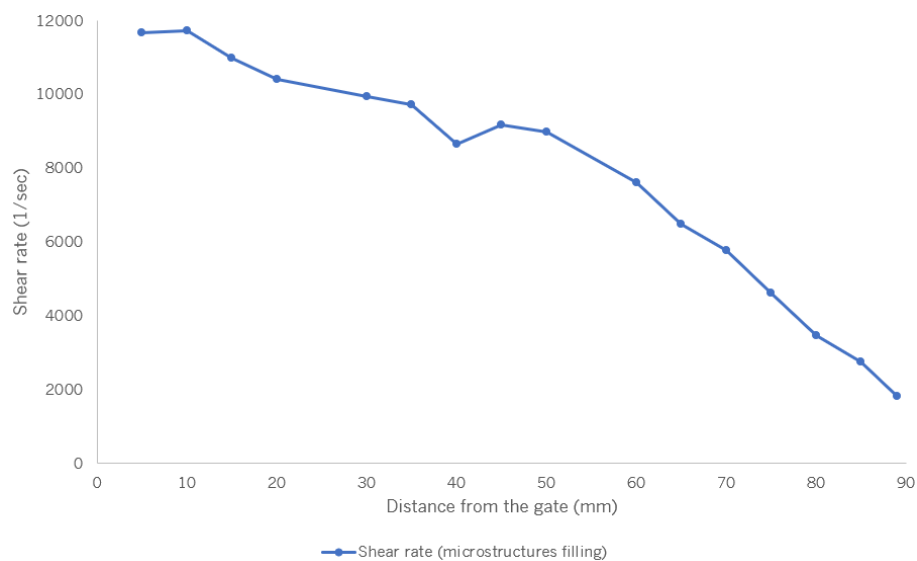


Figure 67 - Shear rate during microstructures filling in function of their distance from the gate.

The filling times of experimental and simulation results were also compared, another parameter to compare the process method chosen and described in section 3.4. Concerning the mould temperature of 60 °C, the filling time varies between 0.526 and 0.642 seconds, depending on the injection speed. These values correspond to the double of the experimental filling time, which was complete, between 0.252 and 0.331 seconds, despite of the incomplete filling verified in simulation. On the other hand, to a mould temperature of 90 °C, the filling time is quite similar in the simulation and in the experimental

results, being verified a difference in a range of 0.001 and 0.150 seconds, concerning the experiences with the highest mould temperature.

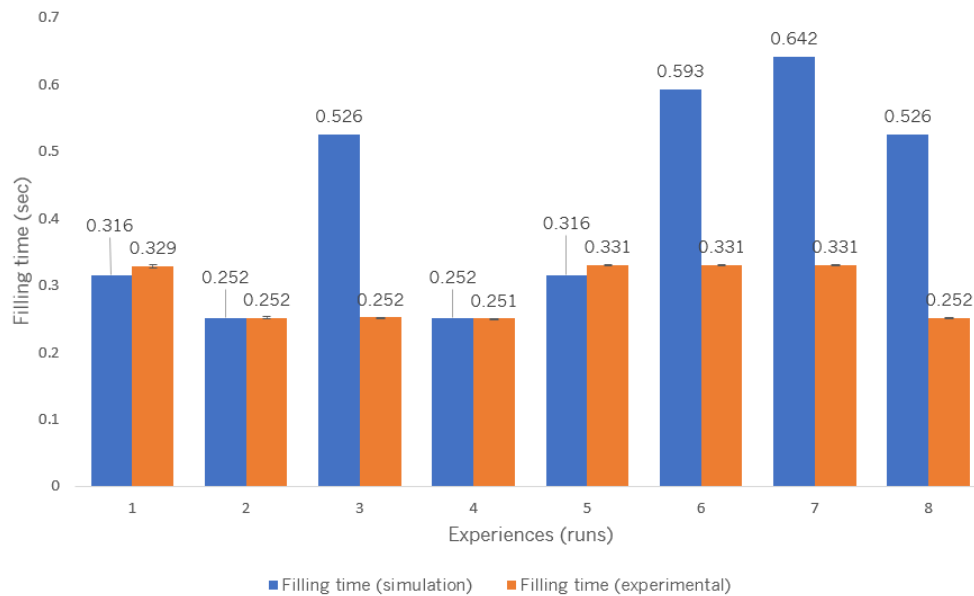


Figure 68 - Comparison between Moldex3D filling time and experimental filling time.

5.3 Production of protective optical window prototype samples by injection moulding

The results concerning the injection moulded samples of the protective optical window prototype are presented in this subchapter. As mentioned in chapter 3, the samples were moulded using the PMMA material and the microstructured insert described in section 4.2.2. After the process stabilization, it was possible to collect eight samples. The production cycle was interrupted by the displacement of the aluminium insert used to block the melt flow to the other cavity (Figure 44). For this reason, it was not possible to test different process conditions.

The preliminary study of the PMMA process conditions (subchapter 5.2) allowed to perform some alterations to minimize the defects associated to the process. The melt temperature was reduced to avoid the degradation problems which arose previously and to be possible to obtain quality samples to measure the WCA and evaluate the replication degree of the microstructures. In Table 12 are presented the process conditions used. The moulding filling was simulated in Moldex3D (Appendix H) and the results was the start point to define the process conditions. During the process stabilization, some samples presented air pockets (Figure 69) and the problem was solved by increasing the holding pressure and the holding time.

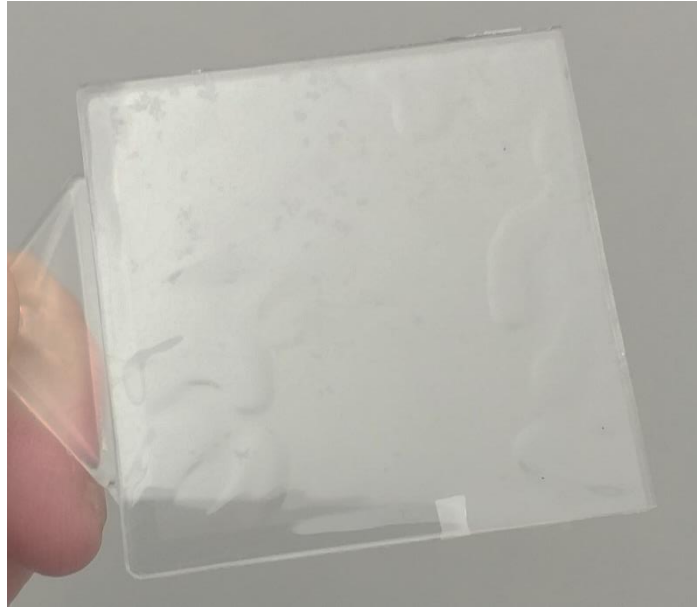


Figure 69 – PMMA microstructured sample with air pockets, collected during process conditions stabilization.

Table 12 – Process conditions to produce the protective optical window samples using PMMA.

Process conditions	
Melt temperature profile (° C)	250 – 255 – 250 – 245 – 50
Mould temperature (° C)	90
Injection speed (mm/s)	400
Holding pressure (MPa)	200
Holding time (sec)	5
Cooling time (sec)	45
Vacuum	ON

Before the injection cycle was interrupted, were collected eight samples. It was not possible to clean the polished area before the injection moulding, so the samples were marked, as seen in Figure 70. For this reason, the optical transmittance was not evaluated, and the characterization was centred in the microstructures replication and its influence in surface hydrophobicity.

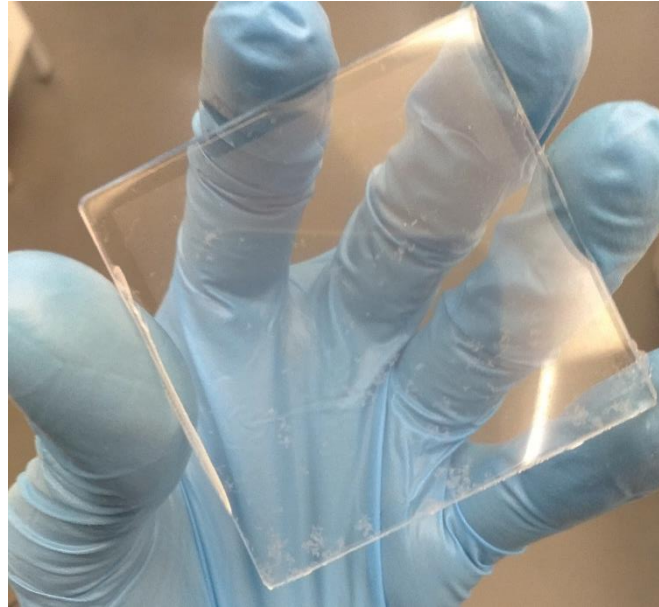


Figure 70 - PMMA microstructured sample, with the marks on the injection side.

First, the samples were analysed by optical 3D measurement, but it was not possible to find the proper conditions to achieve a suitable profile to measure the roughness parameters. The samples transparency difficulted to adjust the light contrast and brightness of the microscope.

To evaluate the replication quality, the samples were submitted to SEM analysis. It was measured the microstructures height, whose values round $1.00\ \mu\text{m}$. Since the microcavities have around $4\ \mu\text{m}$ of depth (Figure 71), the replication degree rounds 25 %. Figure 72 shows the SEM images of a microstructured PMMA sample, where the microstructure has $1.02\ \mu\text{m}$. In fact, the polymer could flow into the microcavities, but it results in incomplete filling. The main reason can be insufficient dosage or low injection speed, that should be adjusted in further work.

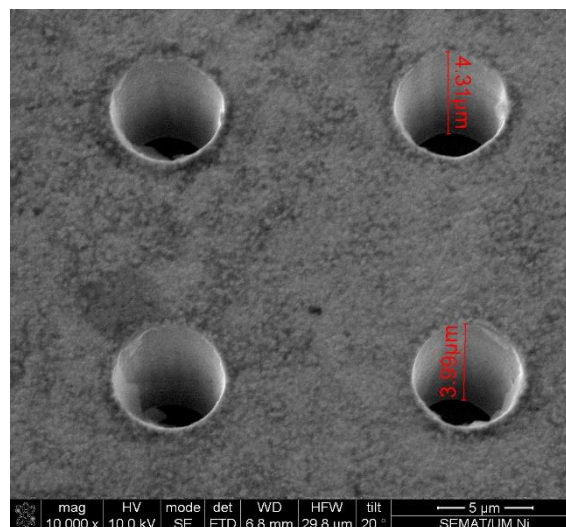


Figure 71 - SEM image of Nickel mould insert.

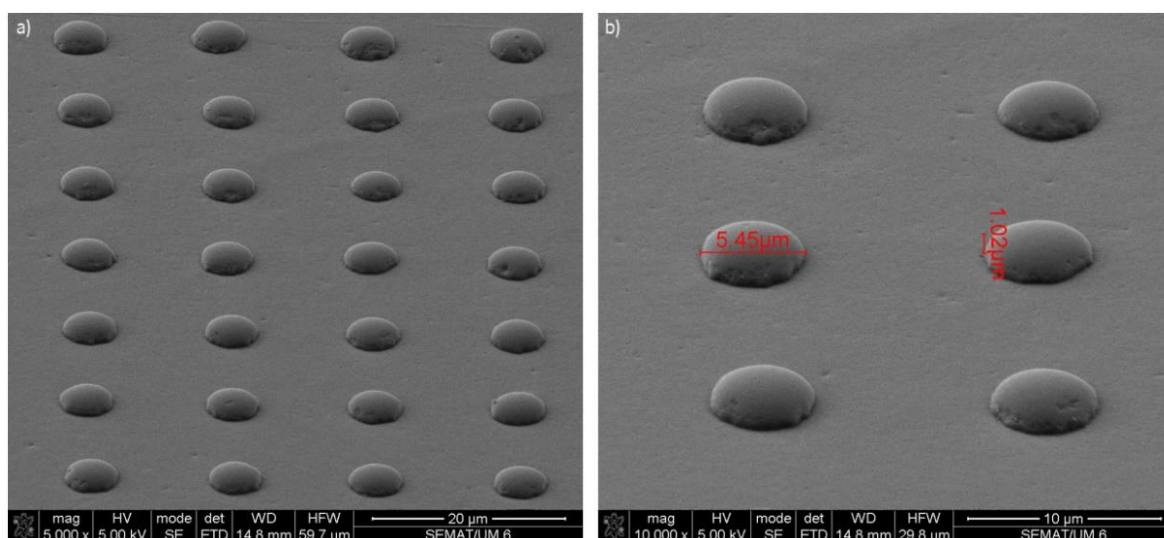


Figure 72 - SEM images of PMMA microstructured sample: a) 5000x magnification and b) 10000x magnification.

Before to activate the vacuum system, it was moulded a sample to compare the filling with and without applying vacuum. Although the replicated height does not have significant differences, the sample without applied vacuum shows small holes on top, as also verified by Rodgers et al. [98]. The quality of replicated structures is improved when vacuum is applied, as seen in Figure 73.

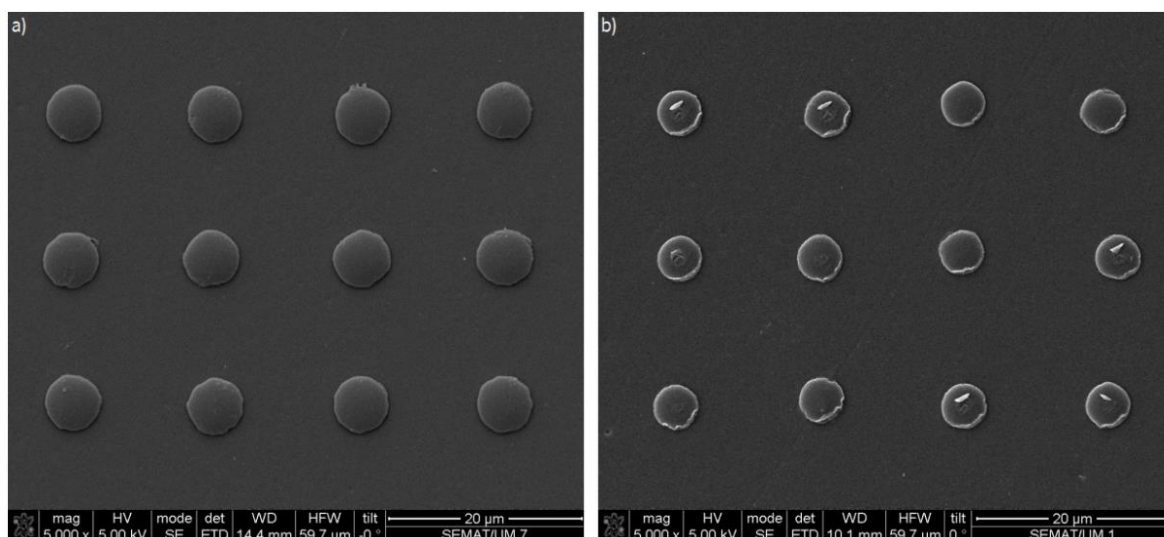


Figure 73 – Top view of moulded microstructures with 5000x of magnification a) with vacuum and b) without vacuum.

To evaluate if the replicated structure influenced the surface hydrophobicity, it was determined the water contact angle. The measurements were made in nine points of the surface, for both microstructured and flat samples, for comparison purposes. The measured areas were near the gate, in sample centre and in the gate opposite side and it was noticed any differences on replicated height along the substrate. Figure 74 shows the water contact angle measured for the eight samples and for the non-

microstructured sample. The results correspond to the average of the nine measurement points for each sample, as well the standard deviation.

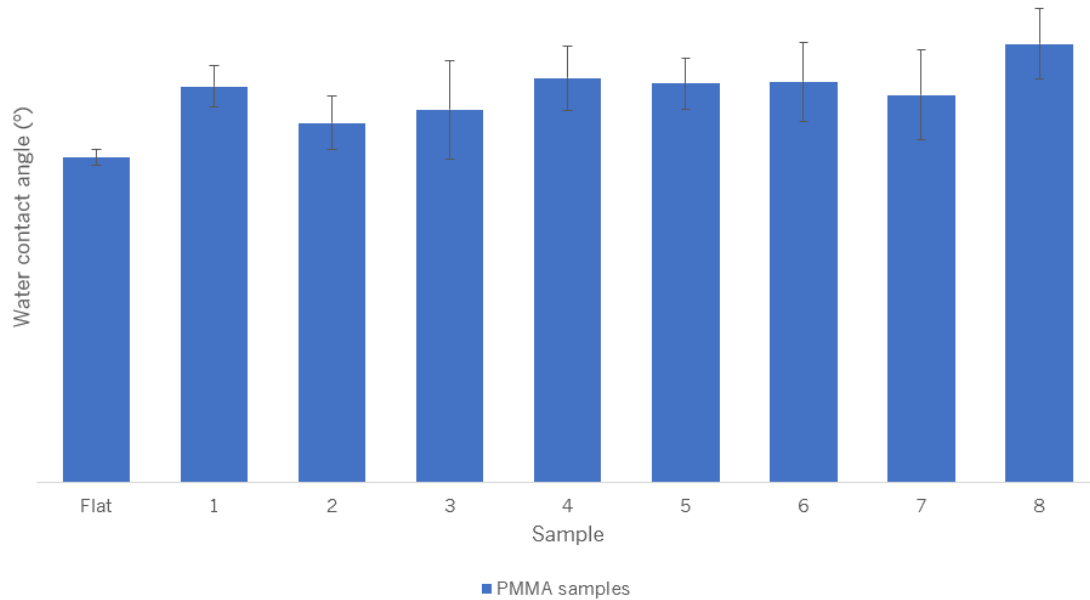


Figure 74 - PMMA samples water contact angle values.

For the non-microstructured sample (flat), the WCA value is 61.1 °, in average. For the microstructured surfaces, the obtained WCA values increases to an average of 74.2 °, considering the average value for the eight samples. Water droplets laying on the flat surface, with a WCA of 68 °, and on a microstructured surface, with a WCA of 79 °, are shown in Figure 75.

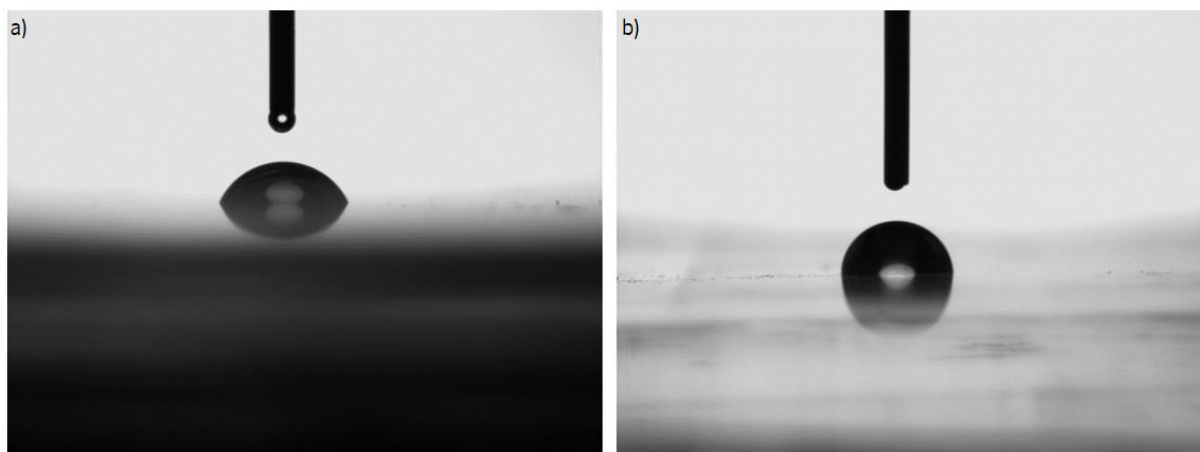


Figure 75 - Water droplets a) on a non-microstructured surface and b) on a microstructured surface.

The presence of the microstructures, even though they are only 25 % of the desired height, allowed an increment of the water contact angle in 13 °. The defined configuration produces alterations in surface hydrophobicity and provides good expectations for a complete filling of the microstructures.

As mentioned, the process conditions should be adjusted to improve the replicated height. However, some authors performed experiences where the injection moulding and injection-compression moulding were compared [60] as well the integration of a variotherm system [74,77]. It is believed the compression stage, characteristic from injection-compression moulding, and the possibility to process with mould temperature superior to glass transition temperature will improve the microstructures replication. For these reasons, it was designed an injection-compression mould, described in chapter 6.

6 Design of an injection-compression mould

The use of replication methods to reproduce hydrophobic surfaces as several advantages comparing to coatings application. The replication methods allow greater process repeatability and the hydrophobic property can be obtained during the part moulding, conversely to the coatings application. Besides, when a coating is applied, and additional barrier is added to the light pathway, influencing the optical transmittance of the product.

Based on this reason, it was previously developed an injection mould, described in Section 4.1.2, capable of produce two parts per cycle. Meanwhile, it was acquired a precision injection moulding machine, whose maximum injection weight of material is lower than the weight of the moulding, so the mould had to be adapted based on the specifications of the machine. However, this solution was not a long-term option, due to the wear along cycles and material leaks, since there is a minimum gap between the insert and the cavity where it is placed. So, there is a need to develop a new productive tool.

The microreplication using injection moulding technique have some limitations. The hesitation phenomenon, due to the thickness variation along the substrate allied to the premature freezing, inhibit the filling of the microstructures. These two problems can be solved by the implementation of a variotherm system. Besides, since the requirements for the final product comprise the replication of microstructures to allow the self-cleaning ability and the optical performance, which is highly influenced by the geometrical precision of the part and its inner properties, an injection-compression mould can meet a commitment between replication degree and optical quality.

In this chapter, will be described the design and selection of components to an injection-compression mould, a future work proposal.

6.1 Mould structure

The design of mould structure, namely the constituent plates and its dimensions, is influenced by the components of each mould system as well the dimensions of part and feed system.

HASCO provides an application, *HASCO Uni*, which enables the download of CAD files of components and standard structures. Next, the files can be converted to *Solidworks* extensions and modified, posteriorly.

It was selected a proper structure for a cold runners system with only two moulding plates, since the desired feed system does not require additional plates. The structure and identification of the constituent plates are shown in Figure 76. The mould dimensions are limited by the machine

specifications. The height and length of mould plates should concern the machine platens size and the total mould height must be between the range specified in datasheet (Figure 103, Appendix A). Considering the space needed for the moulding and ejection elements, it was chosen a structure with dimensions of 196 mm x 156 mm, with exception of the both tightening plates, whose dimensions are 216 mm x 196 mm. The total mould height is 208 mm.

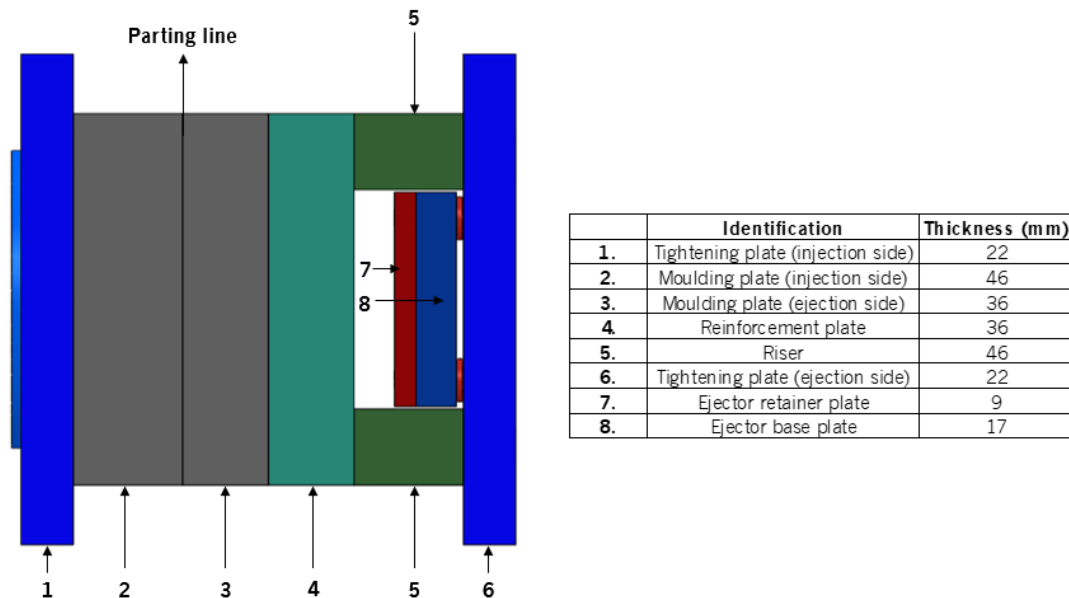


Figure 76 - Downloaded HASCO mould structure, plates identification and respective dimensions.

6.2 Feed system design

The feed system is responsible to guide the polymer melt from the machine injection nozzle to the cavities. Usually, is composed by the sprue, the runners and the gate.

To fulfil its function, the feed system must be correctly dimensioned, to fill the cavities and to obtain quality parts. The geometry and positioning of the feed system elements are also a crucial parameter, since it can minimize the defects and problems during the process. The prototype of the protective optical window corresponds to a plate with dimensions of 50 x 50 x 2 mm. For ejection purposes, the part was designed with a draft angle of 2 °, for an easier demoulding.

The feed system should be located laterally, to avoid marks in the part which can interfere with the desired optical performance of the protective window. The selected gate type must assure an uniform flow front and reduce substantially the probability of warpage and residual stress [99]. In *Manual do Projetista* [100], there are presented three gates which fits in these specifications: side gate, film gate or fan gate (Figure 77).

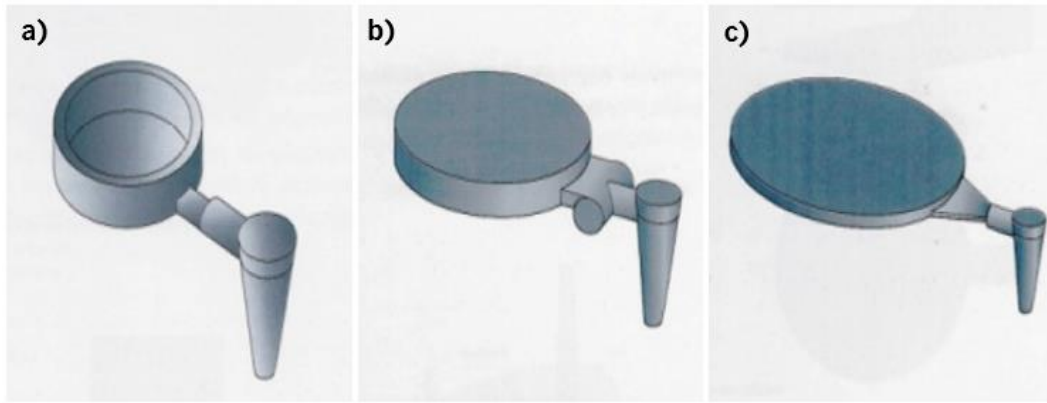


Figure 77 – Available lateral gates: a) side gate, b) film gate or c) fan gate. Retired from [100].

The side gate allows higher control of the injection speed, which is crucial in the filling of microstructures, but the film and fan gates provide a uniform flow front, being the effect more significant in the film gate, due to its position along the almost entire width of the part.

For this motive, it was selected and dimensioned a film gate variation, with a composite structure (Figure 78). The gate 1, connected with the sprue, has lower sectional area. When the melt enters in gate 1, due to its lower sectional area compared with the sprue, the injection speed and the shear rate will increase, causing a homogeneous melt mixing. Since the melt passes to the second gate, with greater sectional area, the melt will expand due to a decrease in velocity. This effect implies that the melt enters in the cavity with a laminar regime, parallel to the gate [101]. The gate 2 has a sectional area reduction in its edges to facilitate its separation from the part.

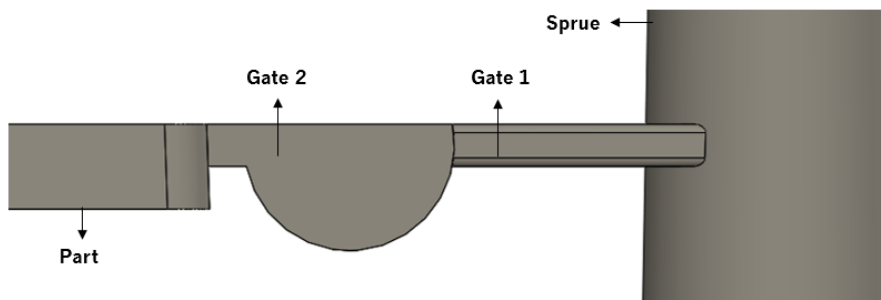


Figure 78 - Film gate variation, with a composite structure.

The rules for sizing the film gate are represented in Figure 79. It was opted for the left version, that requires less material and allows the machining of only one of the two moulding plates.

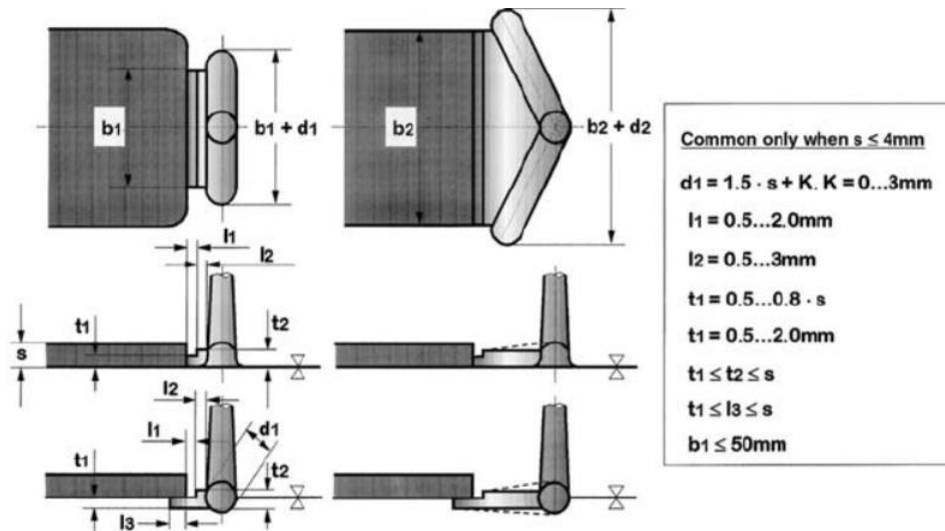


Figure 79 – Dimensioning rules to apply to film gates. Source: [99].

The sprue is placed on the injection side of the mould and it is machined in an external tool, known as sprue bushing, to facilitate the replacement in case of wear. HASCO offers standard sprue bushings, schematized in Figure 80, which can be easily selected taking in the account the characteristics of the machine nozzle. The diameter $d1$ should be 1 mm larger than the machine nozzle diameter, to seal the melt polymer and avoid leaks. The dimension Sr should be greater than the radial concavity profile of the nozzle, in order to fit perfectly to initiate the injection step. The exit angle, α , promotes the ejection of the moulded sprue from the sprue bushing and it should be extended to the moulding plate of the ejection side, to force the sprue ejection during the mould opening. This way, it is created a cold slug which retain the first material which is injected and avoid its entrance in the cavity, avoiding premature freezing and obstruction of the feed system [100].

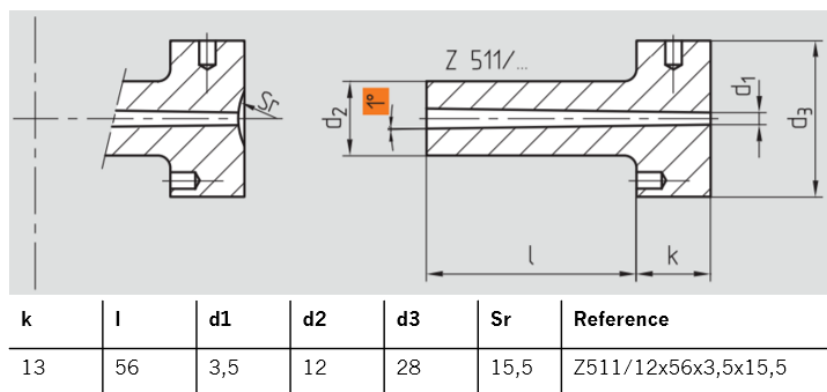


Figure 80 - Scheme of a sectional view of a sprue bushing, with the selection parameters represented. Dimensions in mm. Source: HASCO.

The final design of the moulding, part, and feed system are represented in Figure 81. The design of the feed system was a validation process, where was performed the simulation in *Moldex3D* until it

was finding a commitment between a uniform cavity filling and a minimum volume of material. Other factors, such as the possibility of machining only one of the two moulding plates was taking in account.

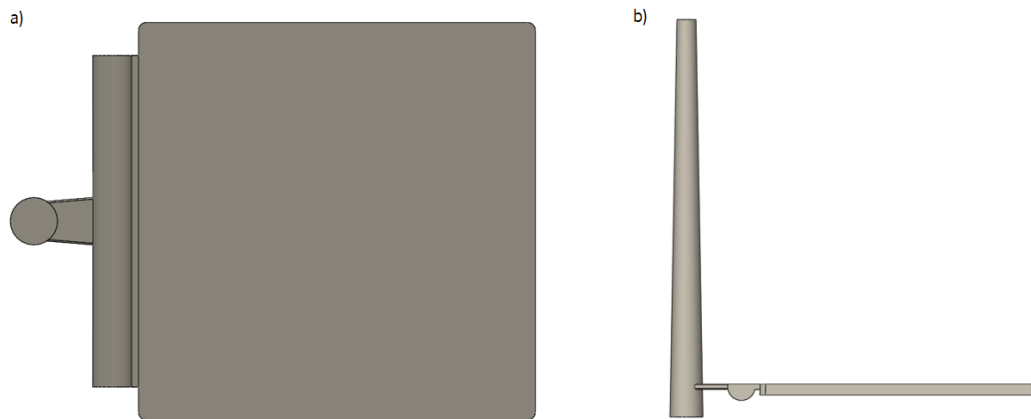


Figure 81 – Final moulding, part and feed system: a) front view and b) side view.

Figure 82 shows, by percentage of time, that the feed system allows a uniform flow front, parallel to the gate since it enters in the cavity. Another relevant result consists in the warpage displacement, a functionality of *Moldex3D*, in warpage analysis section, which indicates the effect of warpage along the part surface, considering the injection and the cooling down to the room temperature, initially inputted. This result is compared with the warpage displacement of the moulding of the prototype IM mould (Figure 45), where it is possible to see a reduction of about 50 % of warpage along the part length (Figure 83).

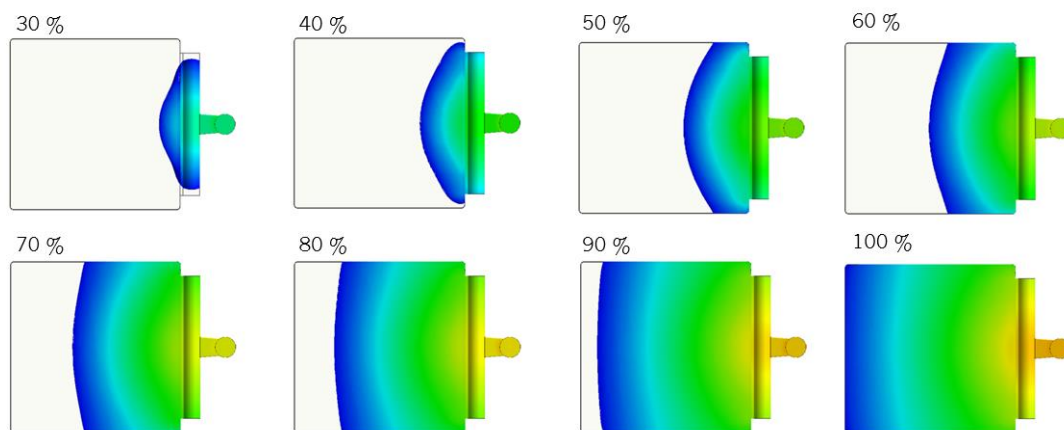


Figure 82 - Results from *Moldex3D* simulation. Melt front flow by percentage of filling time of the developed feed system for the injection-compression mould.

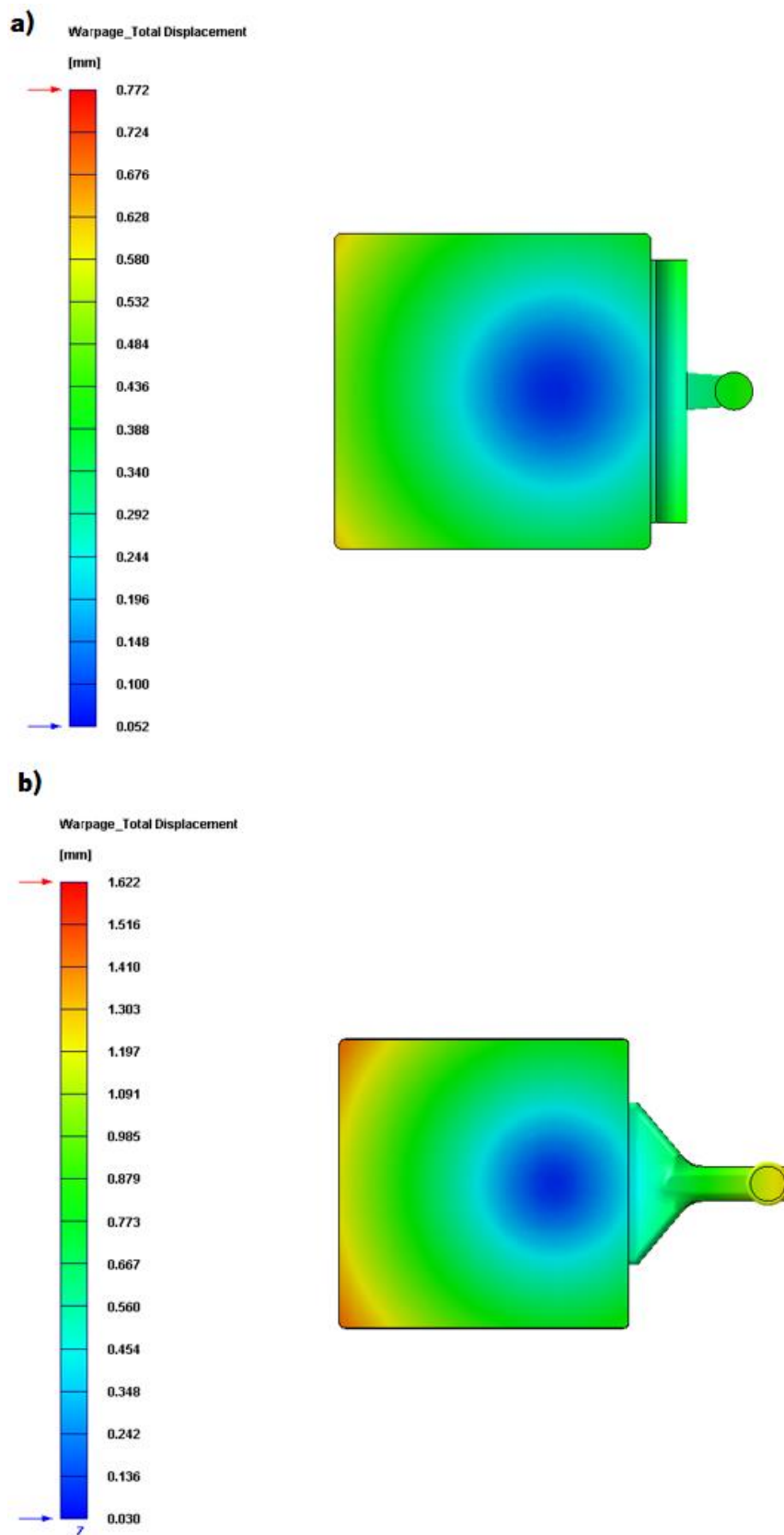


Figure 83 - Comparison of warpage displacement of a) developed feed system and b) feed system of the prototype mould.

6.3 Vacuum system

Similar to the prototype IM mould, the presence of a vacuum system is essential to eliminate the air after the mould closing. Otherwise, the air or other gases will occupy space destined to the melt, causing defects in the final part, named voids, weld lines or irregular shrinkage. Besides this, since the part corresponds to a micro featured surface, the presence of air will result in incomplete filling of microstructures and the desirable hydrophobicity of the surface, to self-cleaning purposes, may not be achieved.

To a proper gas escaping, the parting line is not enough to remove the air. The most efficient way is to perform vacuum trough the cavity by placing small ribs around the cavity. The depth of these ribs is influenced by the processed polymer. Its location should consider the end of flow areas and the results of filling simulation. In Figure 84, it is represented the results of filling simulation of a non microstructured part. It is shown that the areas with highest chance of air trapping in the part are in its edge corners.

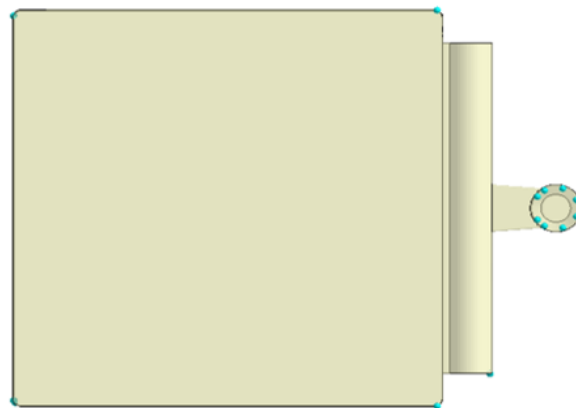


Figure 84 - Filling simulation in Moldex3D. Air traps location results.

Taking the results in consideration, the ribs were placed near the edges and spread along the part. It was defined a depth of 0.040 mm, the minimum value inserted in the range of values for PMMA and PC, suggested in *Manual do Projetista* [100], to avoid material leaks. The ribs are connected to a circuit where the air or other gases will flow until the vacuum hole, which is connected to a device whose function is to extract the air. In Figure 85 is represented the schematic of the vacuum system design.

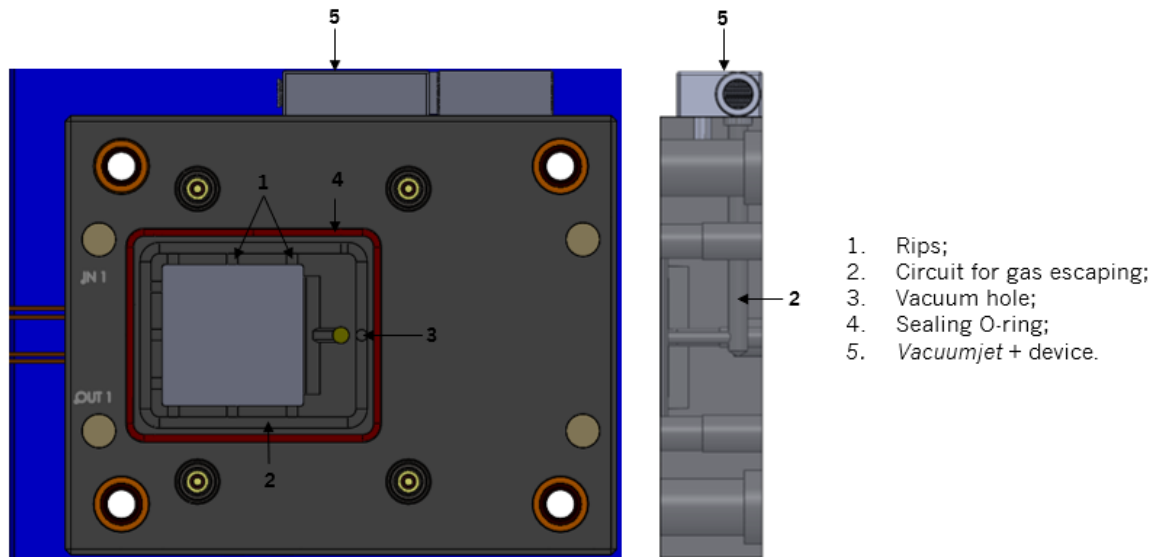


Figure 85 - Front view of the mould movable side: moulding plate, where is placed the vacuum system.

The selected device is the *Vacuumjet +* (Figure 86a), selected from *CUMSA* catalogue. It works according to Venturi principle, where the vacuum is generated through the entrance of compressed air (Figure 86b).

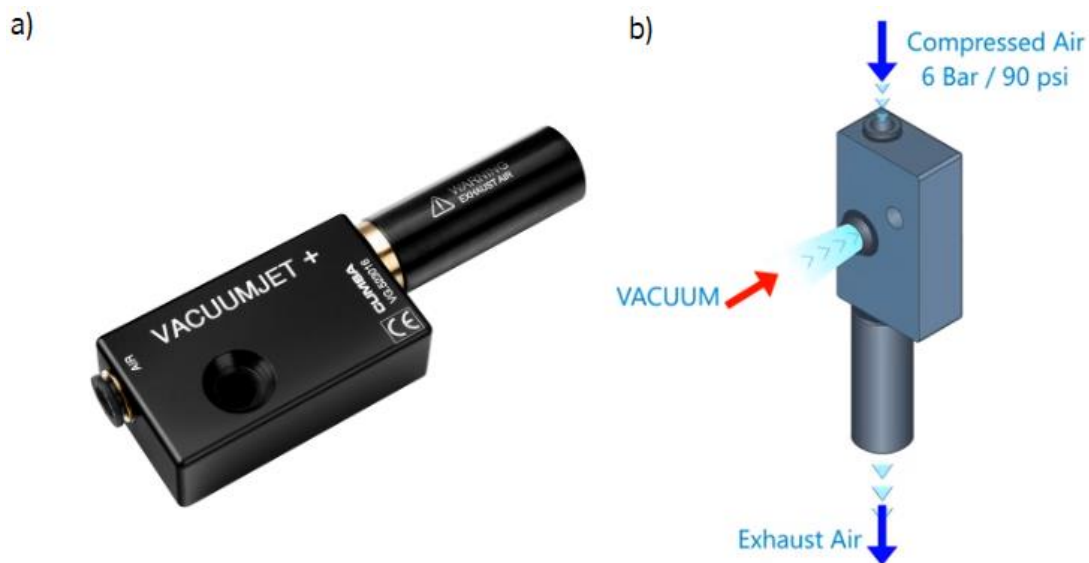


Figure 86 – *Vacuumjet +* by *CUMSA*: a) device and b) operating principle. Reproduced from *CUMSA* website.

Like the prototype IM mould, the cavity must be isolated with a sealing O-ring to avoid the air entrance across the parting line, after the moment of the mould closing. In the moulding plate of the movable side, it was machined an O-ring cavity with dimensions according to *CUMSA* norms (Figure 87). The O-ring has a diameter (A) of 3 mm.

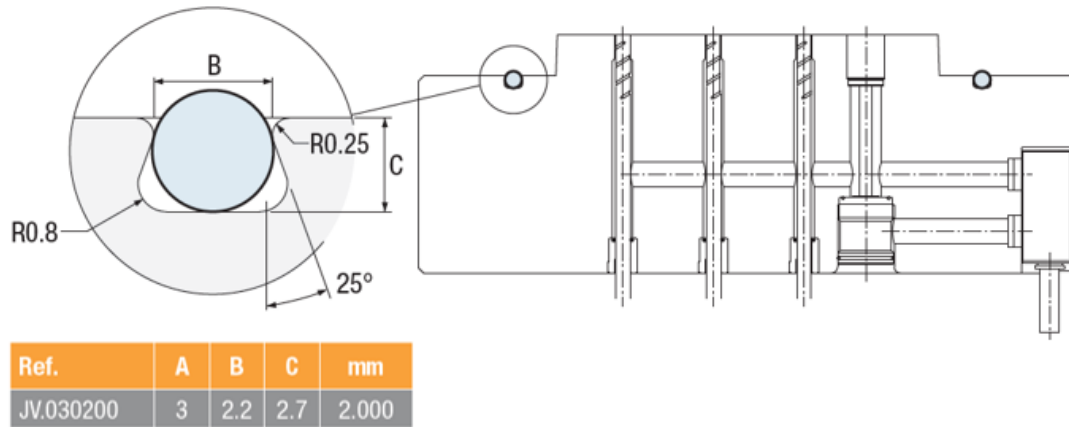


Figure 87 - CUMSA's norm to machine cavity for sealant O-ring of 3 mm (A). Source: CUMSA website.

6.4 Variotherm system

As seen in literature review, the implementation of a variotherm system leads to an improvement of replication degree of microstructures. It is defended, between scientific community, that a cavity surface temperature superior to the polymer glass transition temperature in amorphous polymers improves replication [74,77,102]. However, if a conventional temperature control system is used, such as water channels, the cycle time increases significantly.

Using a variotherm system, the cavity surface is heated up before the polymer melt injection, until the defined temperature. This temperature, preferentially above polymer glass transition temperature of amorphous polymers, is kept during filling. The next step is the moulding cooling, until the ejection temperature.

The variotherm system selected for this mould combines the electrical heating, through cartridge heaters, with conventional cooling, through water channels.

The cooling channels were dimensioned according to the rules expressed in *Manual do Projetista* [103], as seen in Figure 88.

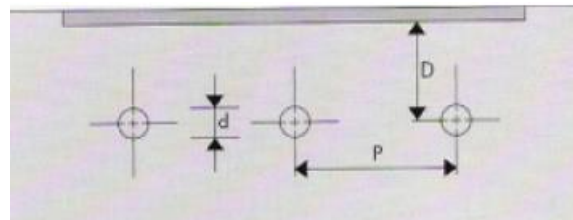


Figure 88 – Dimensioning rules for cooling channels [103].

It is recommended a constant distance between the cooling channels (P) as well the distance from the cavity (D), which are related with the channels diameter (d), expressed below:

$$P = 2.5 \text{ to } 3.5 \text{ d}$$

$$D = 0.8 \text{ to } 1.5 \text{ P}$$

It was defined a 9 mm of diameter to the cooling channels, spaced between them with 35 mm. The channels were placed a 17 mm from the cavities, due to the available space in the movable core (subchapter 6.5), so it was not possible to comply with the expression. The circuit and the dimensions are equal in both sides of the mould, to promote a uniform and controlled cooling.

The cavities heating will be performed by cartridge heaters, available in *HASCO* catalogue. The first step to select a cartridge heater corresponds to the determination of the needed power to heat up the cavity [104]. Considering the weight of the microstructured steel insert, the following equation allows the determination of the needed power to raise the cavity temperature to the desired value in an established time, according to the process efficiency:

$$P \text{ (kW)} = \frac{W_{\text{steel}} \times c \times \Delta T}{860 \times h \times \eta} \quad (6.1)$$

In Table 13, the terms used in Equation 6.1 are specified. Usually, it was set a target time to heat up, determining the power needed. However, the available space after to machine the cooling channels and holes for other elements, such as the return pins, must be considered. This way, each moulding plate will have two cartridge heaters. HASCO offers a minimum cartridge diameter of 6.5 mm, with a maximum power of 200 W. This way, the minimum time to raise a maximum temperature of 60 °C is about 60 seconds (1/60 hours). The efficiency used is a middle value between the range given by the manufacturers.

Table 13 – Terms used to determinate the cartridge heaters power.

Equation term		
<i>W_{steel}</i>	Weight of steel insert (kg)	0.300
<i>c</i>	Specific heat capacity of steel (kcal/kg °C)	0.113
<i>ΔT</i>	Increased temperature (°C)	60
<i>h</i>	Heating time (hours)	1/60
<i>η</i>	Efficiency	0.3

The variotherm system scheme, similar in both moulding plates, is shown in Figure 89. The holes for the heaters have to be machined with a clearance of 0.05 mm, so the cartridge can be placed smoothly. To impede the cartridges displacement, it must be fixed, using wire connectors.

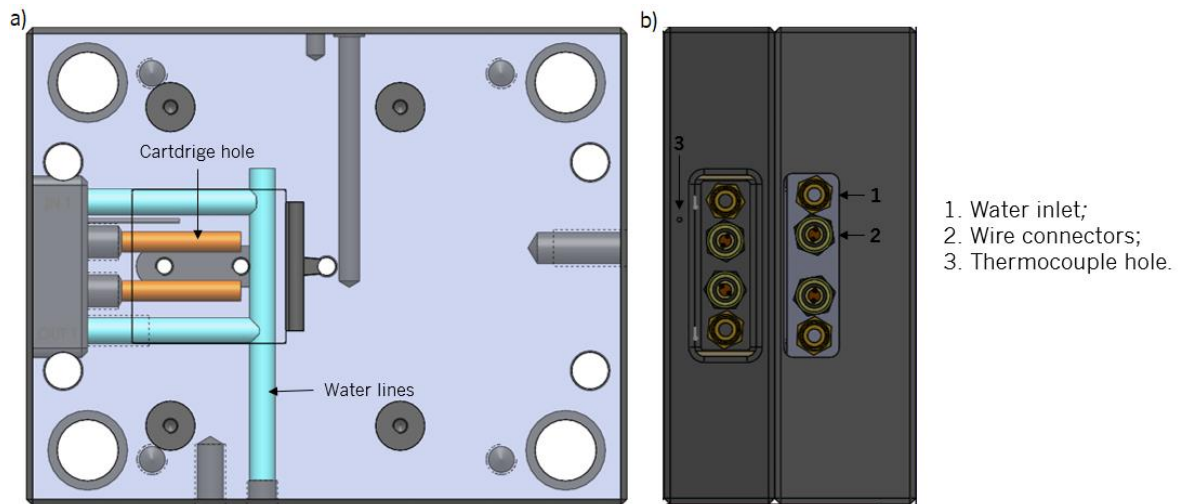


Figure 89 - Variotherm system: a) front view of the mould movable side and b) down view of two moulding plates.

To operate the variotherm system, two control technologies are required: a thermoregulator, to maintain the water flow in cooling channels, such as the used in experimental work, model THP from PIOVAN (Figure 27); and a programmable temperature controller with dual set point, able to operate between the desired mould temperatures. The temperature controller has to be connected to thermocouples, which measure the temperature in the heating area and allow the temperature control of the cartridges. It is recommended by several manufacturers the placement of the thermocouples as near as possible from the cartridges, to prevent cartridge overheating.

The components needed to the heating stage of the variotherm system are represented in Figure 90. The components were selected from catalogues of *HASCO* and *A.P.U. Enterprises*:

- Cartridge heater by *HASCO*, reference Z110/6,5 x 40 x 200 (Figure 90a);
- Thermocouple by *HASCO*, reference H1295/5/1,5 x 71 (Figure 90b);
- Temperature controller by *A.P.U. Enterprises*, model TC-06 (Figure 90c).



Figure 90 – Components for heating in variotherm system: a) cartridge heater, b) thermocouple and c) temperature controller.

6.5 Compression mechanism

Injection-compression moulding differs from the conventional injection moulding by the introduction of a compression stage during the polymer melt injection. The compression movement can be performed by the IM machine clamping unit movement or by the compression of the cores, actuated by integrated hydraulic cylinders in the mould. The compression via cores is more precise since the weight to move is lower, resulting in a uniform compression. To move the core, a hydraulic cylinder should be employed.

The injection moulding machine *FANUC Roboshot α -s15iA* has a function called “pre-injection”, whose functioning principle corresponds to ICM concept. Using “pre-injection”, the polymer melt is injected before the mould closes completely. It was not possible to test this functionality during the experimental work (subchapter 5.3), but it should be evaluated to understand if it improves the microstructures replicated height. In this way, the hydraulic cylinder, mentioned below, can be excluded, simplifying the mould design.

For now, it was opted to integrate a compact hydraulic cylinder, located on the plates of the fixed side. Although the space available is greater on the movable side, it was decided to perform the

compression by the injection side to maintain fixed the microstructured insert (subchapter 6.7), to minimize the wear of an expensive tool.

In Figure 91, a section view of the compression system is shown. The compact cylinder is fixed in both tightening plate and moulding plate.

When the injection cycle begins, the core is backtracked, leaving a compression gap, defined as 2 mm, which can vary to study the effects of the compression gap. During the injection phase, the hydraulic cylinder piston moves forward to perform compression. To activate the movement, the cylinder has to be connected to servo valves, which in turn communicate with the IM machine. The connection between the compression core and the cylinder is established with a set screw, to assure the core retreat after compression, when the mould opens to eject the part.

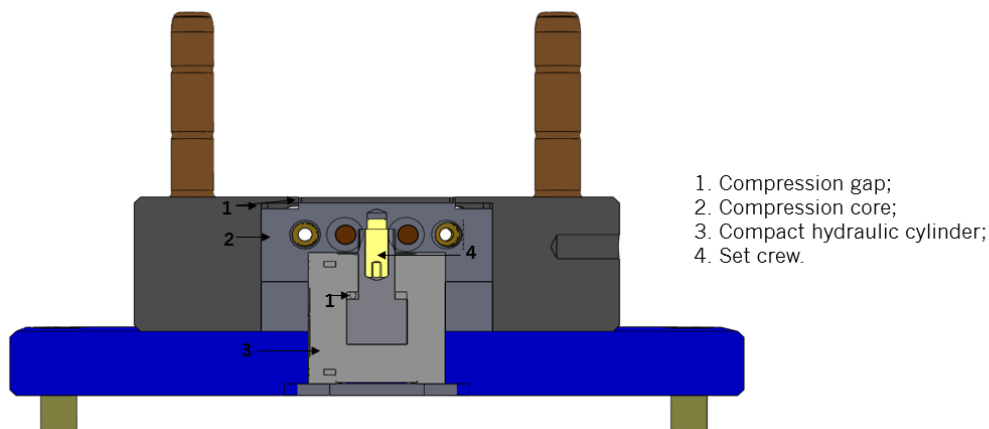


Figure 91 - Solidworks sectional view of mould fixed side, with the compression system.

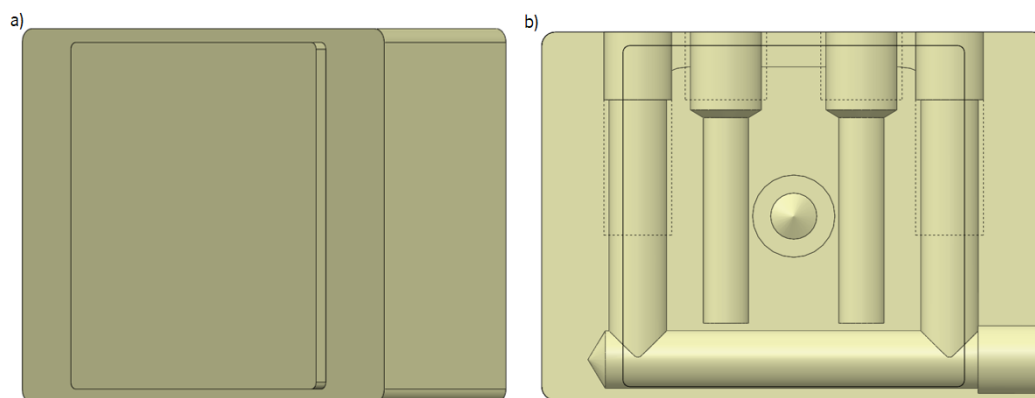


Figure 92 - Compression core: a) overall view and b) front view with transparent surface.

The CAD design of the compression core is shown in Figure 92. The core has a protruding area, which acts in the flow to perform compression. The larger lateral area of the core will limit the core movement, entering in contact with the stopper machined in the moulding plate. In Figure 92b, it is shown

the machined holes for cartridge heaters and water flow, which has to be machined in the core since it is a movable element.

The CAD design of the selected compact hydraulic cylinder, by *Parker Hannifin*, is shown in Figure 93.

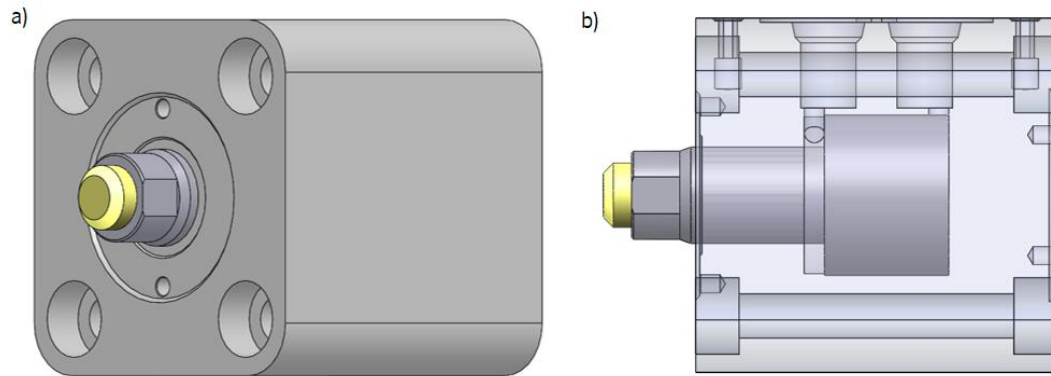


Figure 93 – Compact hydraulic cylinder by Parker Hannifin: a) overall view and b) lateral view, with transparent surface.

6.6 Ejection system

The IM cycle finished with the ejection of the moulded part, after its cooling until achieve enough stiffness to be retired from the mould without damages. Usually, this task is difficult due to the part shrinkage and consequent adhesion to the cavity, requiring mechanical forces to eject the part, which must contemplate the part functionality and avoid damages. Also, for this reason, while the part and feed system design, it must be considered an exit angle in the part and include a cold slug in the movable mould side, to an easier removal of the sprue.

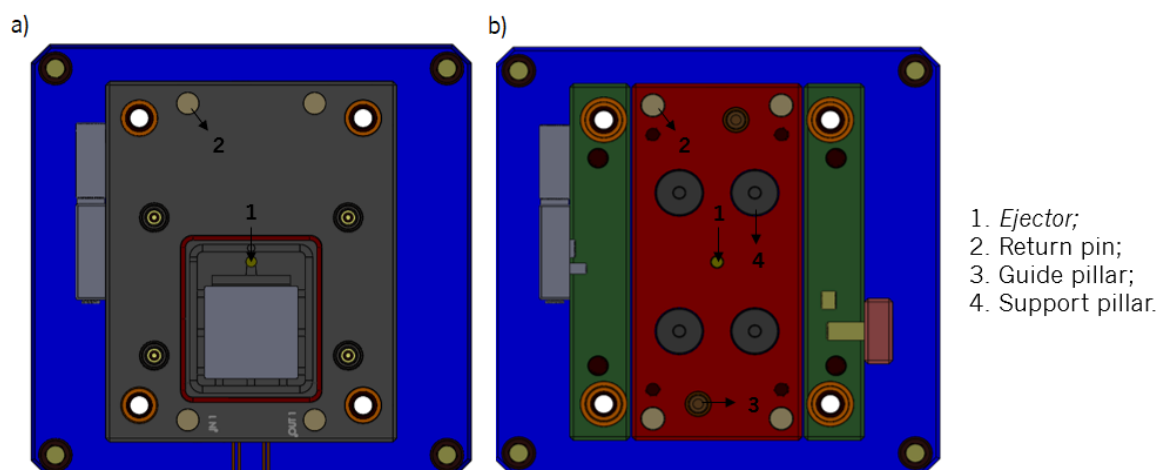


Figure 94 - Front view of the mould movable side and identification of main components of ejection system: a) in moulding plate and b) in ejector retainer plate.

The developed mould is composed by a simple ejection system, represented in Figure 94. It was only used one ejector, placed in cold slug area, to remove the part from the cavity. Although it is desirable

to place ejectors in the part extremities, to uniformize the ejection and the correct microstructures demoulding and, also, to prevent damages associated to bending stress, this option was excluded since it would mark the surface, which is not desirable, given the optical functionality of the protective window. To select the ejector, it was considering a diameter of 6 mm, tight to the cold slug to avoid material leaks.

The ejection stroke should be enough for a partial ejection only to separate the part from the cavity, for it be subsequently removed with the aid of a robotic arm. This solution will prevent the part drop by gravity, preventing damages of the microstructures, and it provides a level of automation not possible with manual removal, by the operator.

Usually, the ejection system is controlled by the IM machine, by the movement of the ejectors plates. This movement is forced by an ejector coupling rod, which acts in the ejectors base plate, hydraulically controlled. In Figure 95, it is represented a sectional view of the left side of the mould, where it is shown the coupling rod placement, connected to the ejector base plate by mean of a set screw.

The return pins, with the double diameter of the ejector, will ensure that ejector plates are pulled back before the beginning of the next injection cycle, to position the ejector without damaging it. For a proper guidance of the ejector plates, two guide pillars were placed in the edges, with lowest diameter than the guides of the parting line. In the Figure 95 it is also identified the support pillars, which are installed in the ejector retainer plate, but their function is to reinforce the movable side, reducing the bending stress in moulding plate and supporting the pressure during injection stage [105].

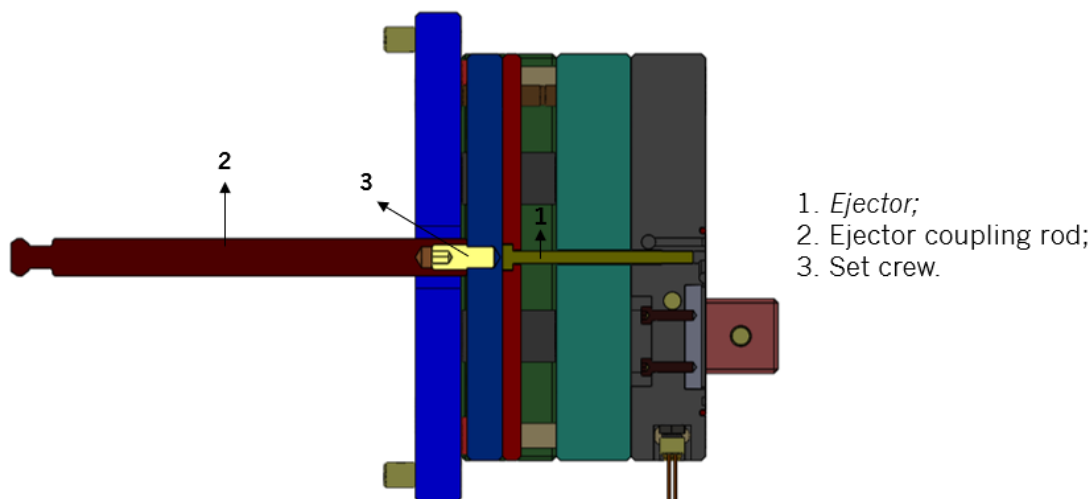


Figure 95 - Sectional view of the mould movable side.

All these components were selected and downloaded from *HASCO* catalogue, to be assembled to the mould structure.

6.7 Final mould

The final mould is shown in Figure 96, an overall view of the movable side (a) and the injection side (b) of the mould, where is possible to see the components referred along this chapter.

In Figure 97, it is shown a down view of the mould, where it is placed the water inlets and the wire connectors which fix the electric cartridges, since it is the most favourable mould side to have access to the thermoregulator and the temperature controller.

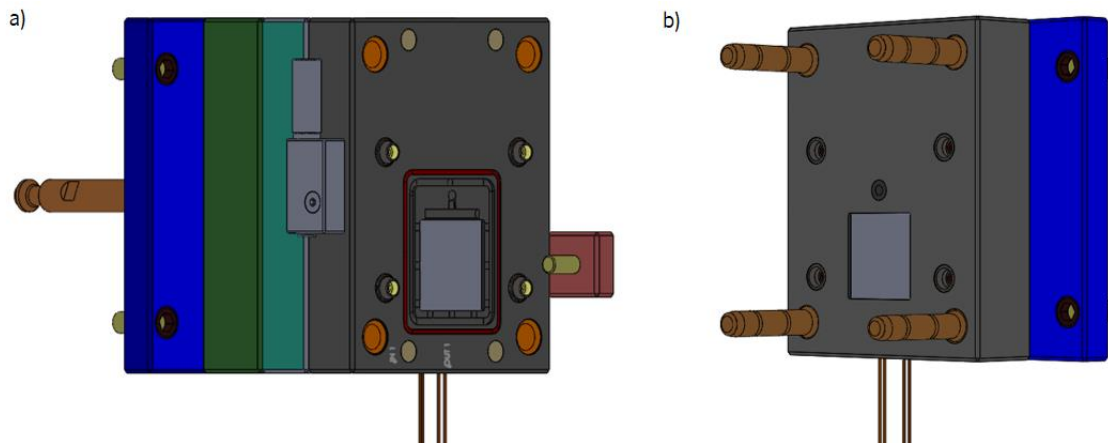


Figure 96 - Final ICM mould: SolidWorks overall view of a) movable side and b) fixed side.

The microstructured insert will be fixed in the mould movable side, considering the existing insert (Section 4.2.2). It was designed a cavity with the insert dimensions, including the thread holes, as seen in Figure 98.

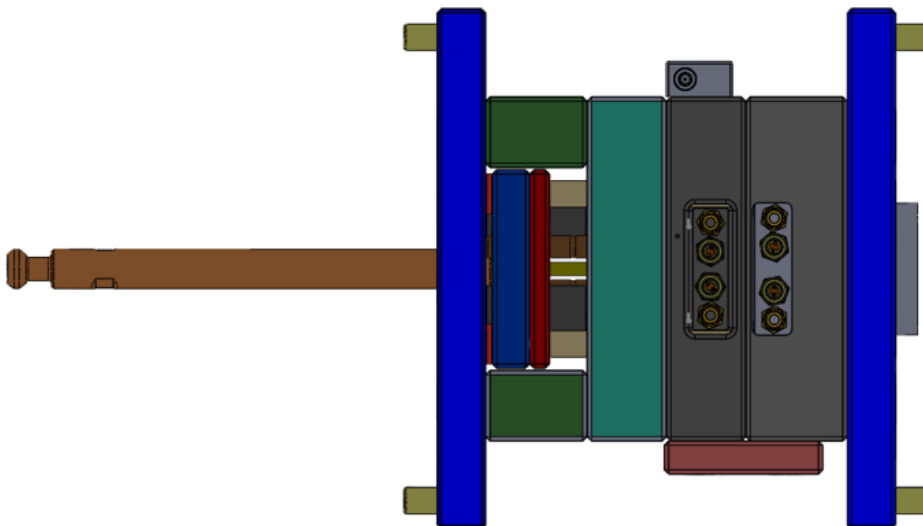


Figure 97 - Down view of ICM Mould.

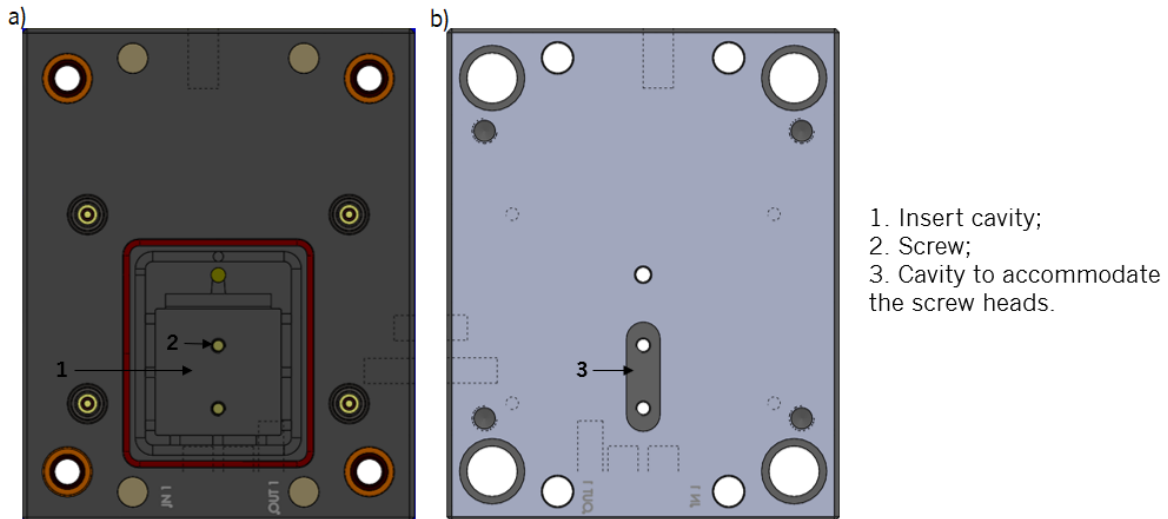


Figure 98 - Front view of moulding plate of the movable side: a) front view and b) back view.

Comparative to the original structure, the tightening plates dimensions were increased from 216 mm x 196 mm to 236 mm x 196 mm. Although it implies an increase of mould weight, it was necessary so it can be possible to fix the mould in the machine by means of screws. In this way, it is possible to fix the mould using the holes of the platens machine whose dimensions are 210 mm x 140 mm. Otherwise, the mould fixation would be performed by power clamps, which interferes with robotic arm operations due to the occupied lateral space. Also, from the fixed side, the original centring ring, with 90 mm of inner diameter, was replaced for a suitable ring for the machine, with 60 mm of inner diameter, capable of accommodate the sprue bushing. These components are displayed in Figure 99, from an overall view from the side of operator.

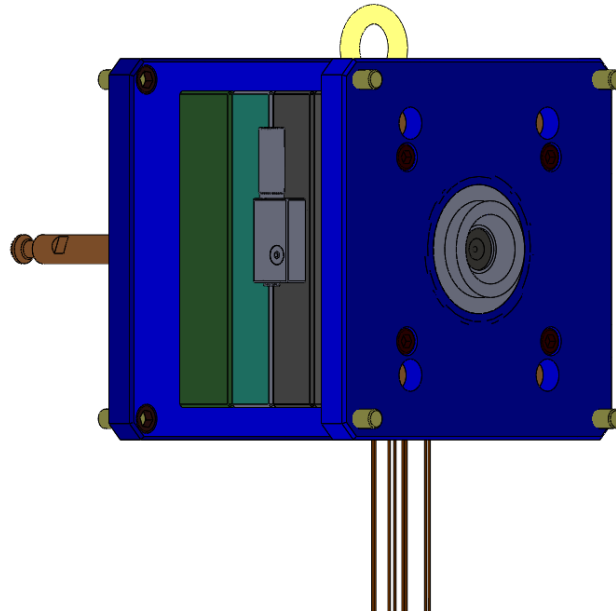


Figure 99 - SolidWorks overall view of Mould, from operator side.

To an accurate precision, it was included interlocks (Figure 100), a solution to precise alignment of mould cavities. This solution prevents minor lateral movements of the movable side related to the fixed side, that could result in wall section or weight variation of moulding.

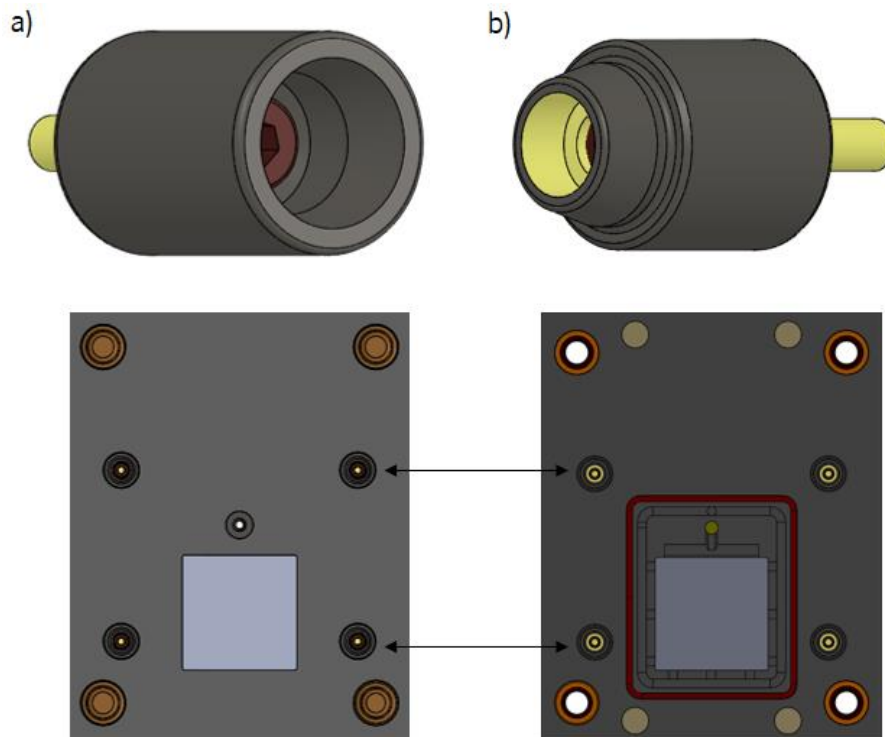


Figure 100 – Overall view of the tapered interlocks: a) female interlock, located in fixed side and b) male interlock, located in movable side.

There are two main types of interlocks: side interlocks, with squared base and tapered interlocks, with circular base. Due to the highest contact area, the ideal interlocks are the squared base, that should

be placed on the middle of the edges. However, the available space in the mould does not allow the use of ideal conditions, so it was placed four tapered interlocks with conic alignment, closest as possible from the cavities.

Finally, to the mould CAD design were added the elements that assure that mould transportation and its mounting in machine platens are safe: the lifting eye bolt (in yellow), to allow the transportation by a crane, and the closing bar (in red), to the mould does not open during this operation, both visible in Figure 101.

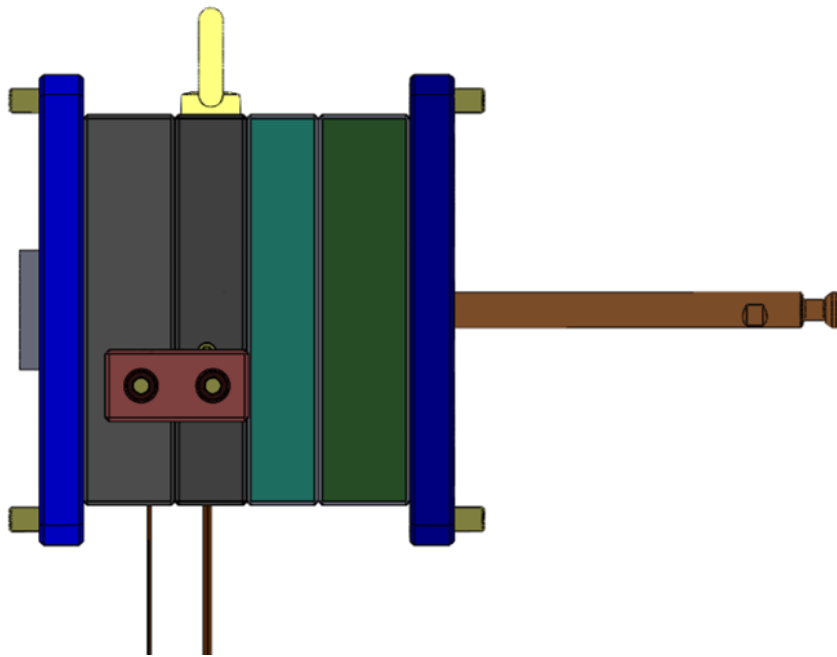


Figure 101 - Solidworks side view of Mould, from operator opposite side.

7 Conclusions

Several researchers have been focusing in to study how the self-cleaning behaviour can be achieved by biomimetic replication, evaluating the influence of the wettability and the microstructured configuration. The studies carried out for this work have as main goal the development of optical protective window prototype, with self-cleaning ability and excellent optical performance.

It was performed two preliminary tests before to produce the protective optical window prototype. The first, using improvised hot embossing, allowed to verify that the configuration defined in the previous project is favourable to enhance the samples hydrophobicity. Although the pattern was not replicated in entire sample, it was observed microstructures around 8 μm of height and an increase in 44 ° of WCA. The second preliminary study concerns to evaluate the processability of PMMA, with higher optical transmittance than PC. It was studied the influence of the process conditions in replicated height as well the flow behaviour along the flow path. Besides the results was influenced by the samples shrinkage, this preliminary test allows to adjust the processing of the protective optical window using PMMA.

After adapting the existent injection mould, the protective optical window production was performed by injection moulding, using PMMA. The microstructures were not completed filled, being measured 1.00 μm of height, which provided an increase of 13 ° in the WCA values. However, the hydrophobic regime was not reached, so there is a demand in to obtain higher replicated height.

To comprise the defined goals until this stage, the process conditions have to be adjusted. In case of the injection mould technique would not be enough to achieve the desired height, a new technique should be employed. For this reason, it was design of an injection-compression mould with a variotherm system, based on suggestions of literature review, which usually improves the replication significantly.

The goal was not completely achieved, although advances were made. Adopting the injection moulding process for microstructures replication requires several experiments until to achieve the proper conditions which provides the microstructures complete replication. However, it can be assumed the studied configuration as favourable to achieve enough hydrophobicity for self-cleaning ability.

8 Future Work

The future work should focus in to improve the microstructures replication, to study other materials concerning their optical and mechanical properties and to properly characterize the produced parts.

First, to improve replication, some process parameters must be adjusted, namely the dosage, to understand if the microstructures did not fill completely due to insufficient material. Concerning the productive tools, it should be considered the production of the developed injection-compression mould, described in Chapter 6, or to evaluate the integration of a variotherm system in the existent productive tool. Also, there is a need to develop microstructured inserts which facilitates the produced part demoulding, by reducing the interfacial adhesion between the insert and the part. A valuable suggestion can be the fabrication of a compound microstructured insert, made of Nickel and PTFE particles.

The replication using the PMMA material should be continuing to be studied, due to its excellent optical transmittance. However, the PC material, with good optical transmittance as well, presents better mechanical properties, that can be an advantage during the demoulding step of the microstructured part, preventing against the microstructures damages during this step.

After obtaining a well-defined microstructured part by injection moulding, there is a need to understand if it was achieved the self-cleaning ability, the influence of the microstructures in optical transmittance and the mechanical resistance of the microstructures. Hence, the hydrophobicity and the self-cleaning ability should be tested, and, besides the static CA measurement, it should be performed a dynamic CA measurement, performed in tilted platforms, to evaluate the droplets movement and if it was achieved the self-cleaning ability. To complement this evaluation, pollution tests, by inducing dirt on the parts to test the self-cleaning ability, must be performed. The optical properties of the replicated parts should be measured, at different incident angles, concerning the LiDAR device functioning. The goal is to understand the effects of the microstructures geometry and configuration in optical transmittance. Finally, a mechanical characterization, namely abrasion tests, should be performed, to understand the mechanical resistance of the replicated microstructures.

9 Bibliography

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10 Appendices

A. FANUC Roboshot α -s15iA technical file

FANUC ROBOSHOT α -S15iA

Mechanical specifications

Item		Unit	Data		
Clamping unit	Clamping mechanism	---	Double toggle		
	Tonnage	kN	Standard 150 (15tonf)		
	Maximum and minimum mold height	mm	Standard 260-130		
	Clamping stroke	mm	160		
	Locating ring diameter	mm	$\phi 60$		
	Tie bar spacing (H×V)	mm	260×235		
	Platen size (H×V)	mm	355×340		
	Minimum mold size (H×V)	*1) mm	150×135		
	Ejector stroke	mm	50		
Injection unit	Maximum ejector force	kN	7(0.7tonf)		
	Screw diameter	mm	14	16	18
	Injection stroke	mm	56	56	75
	Maximum injection volume	cm ³	9	11	19
	Inj. speed 525mm/s	Maximum injection pressure	*2) MPa	250	250
		Maximum pack pressure	*2) MPa	250	250
		Maximum injection rate	*3) cm ³ /s	80	105
		Maximum injection speed	*3) mm/s	525	525
		Maximum screw rotation speed	min ⁻¹	450	450

Figure 102 - Specifications of clamping and injection unit of FANUC Roboshot α -s15iA.

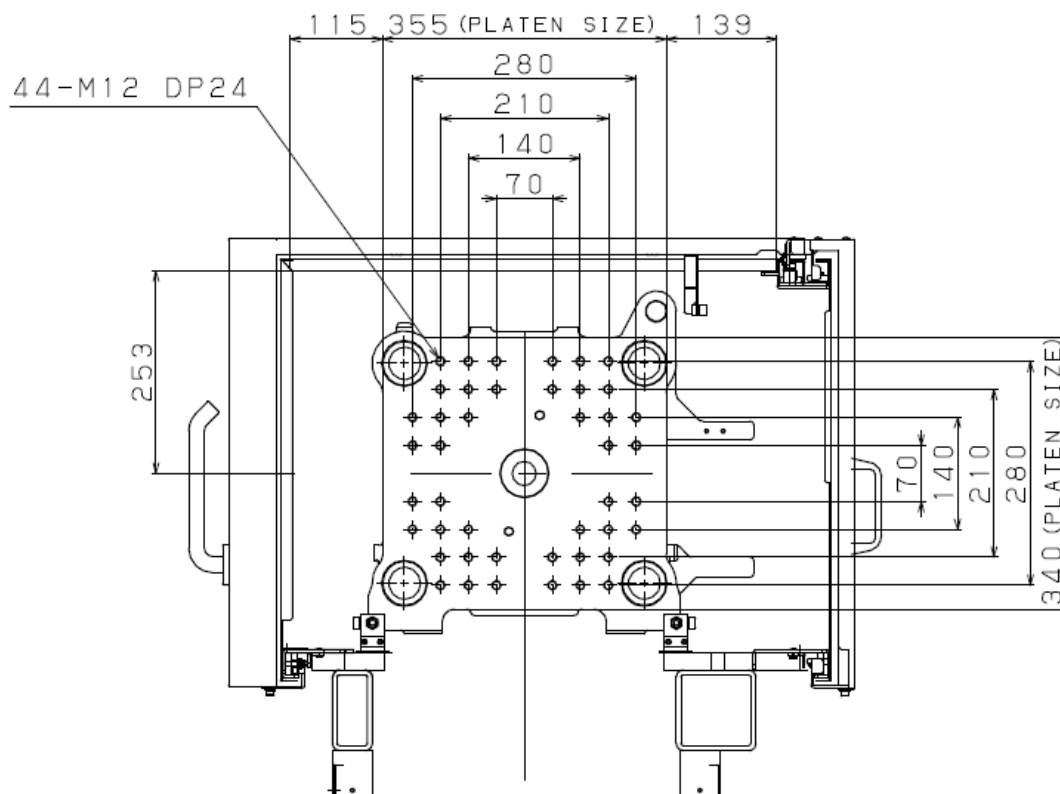


Figure 103 - Dimensions of FANUC Roboshot α -s15iA platen.

B. Micro mould CAD designs

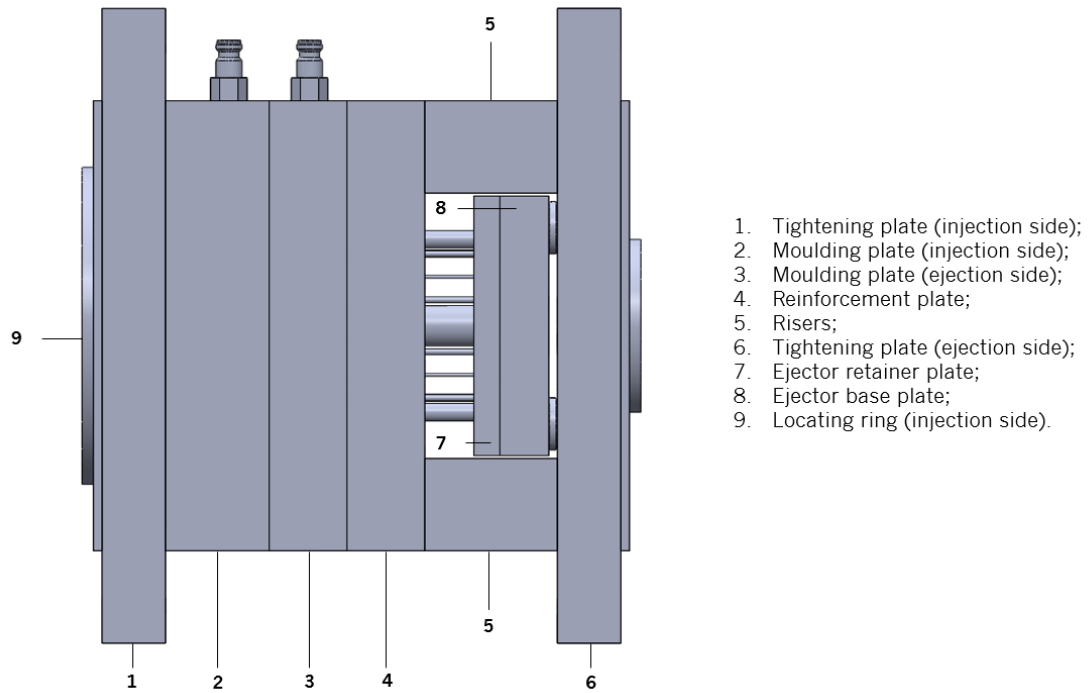


Figure 104 – Identification of micromould plates.

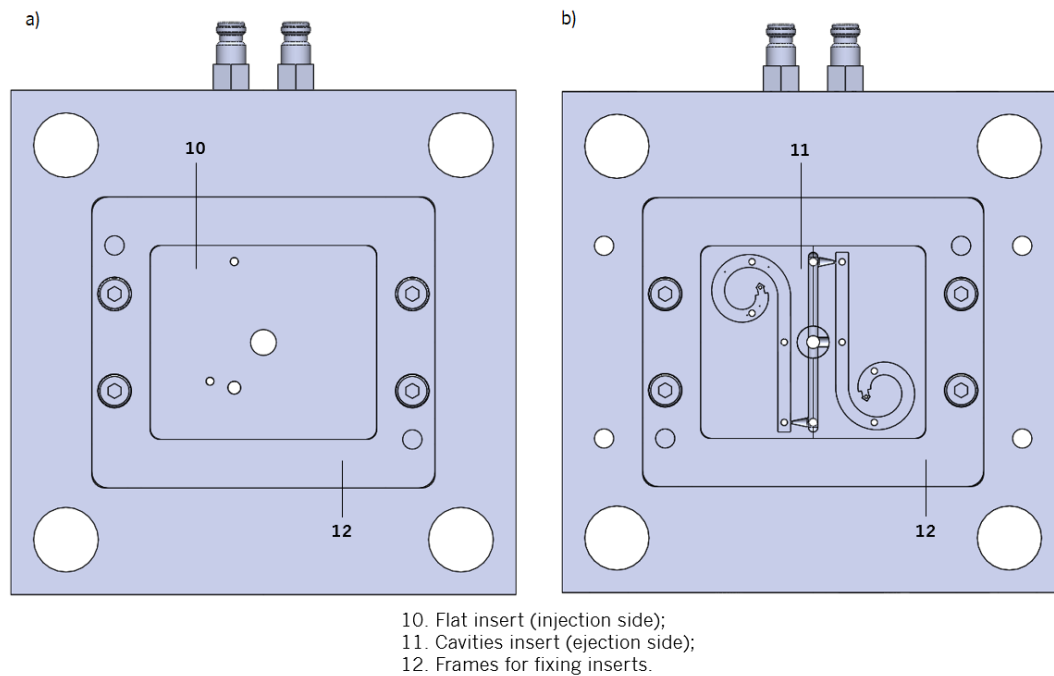


Figure 105 – Front view of moulding plates and respective moulding inserts: a) injection side and b) ejection side.

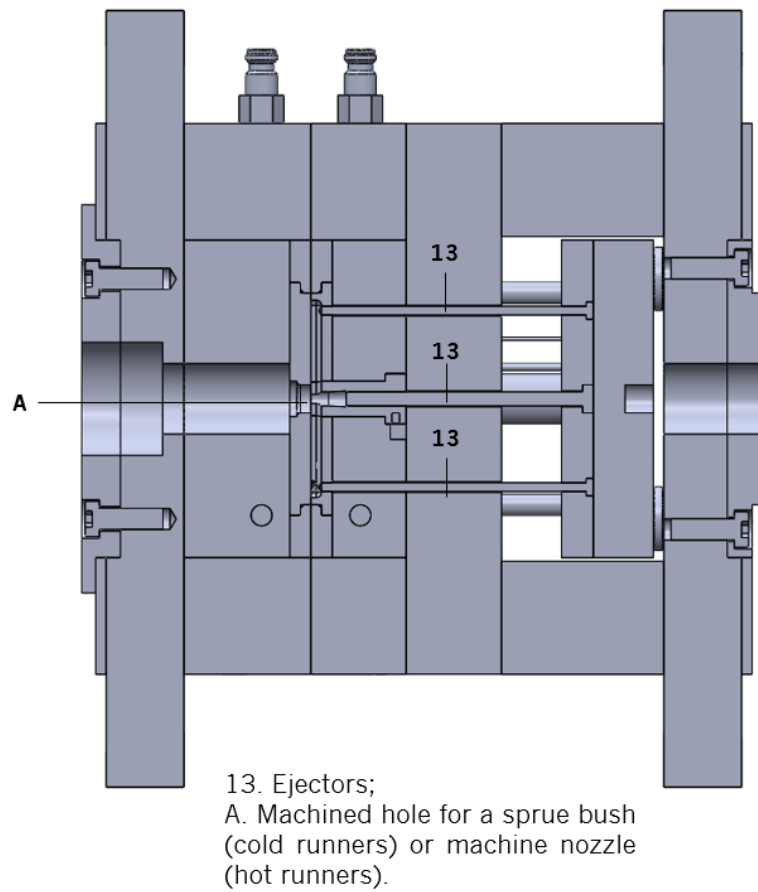


Figure 106 - Sectional view of micromould.

C. Prototype mould CAD designs

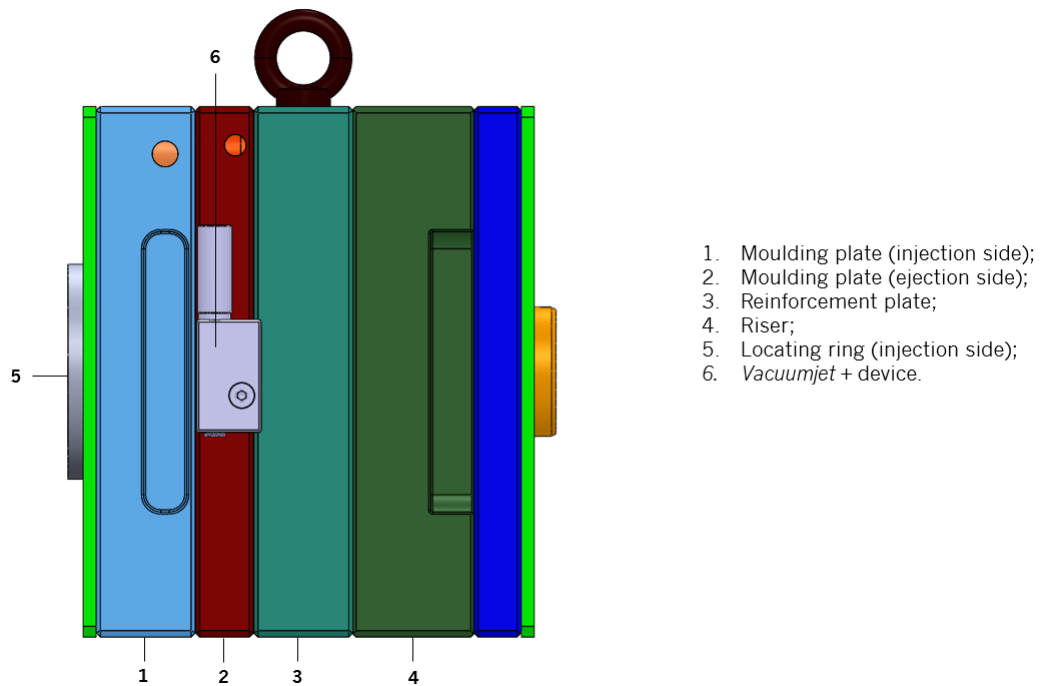


Figure 107 – Identification of main components of prototype mould (side view).

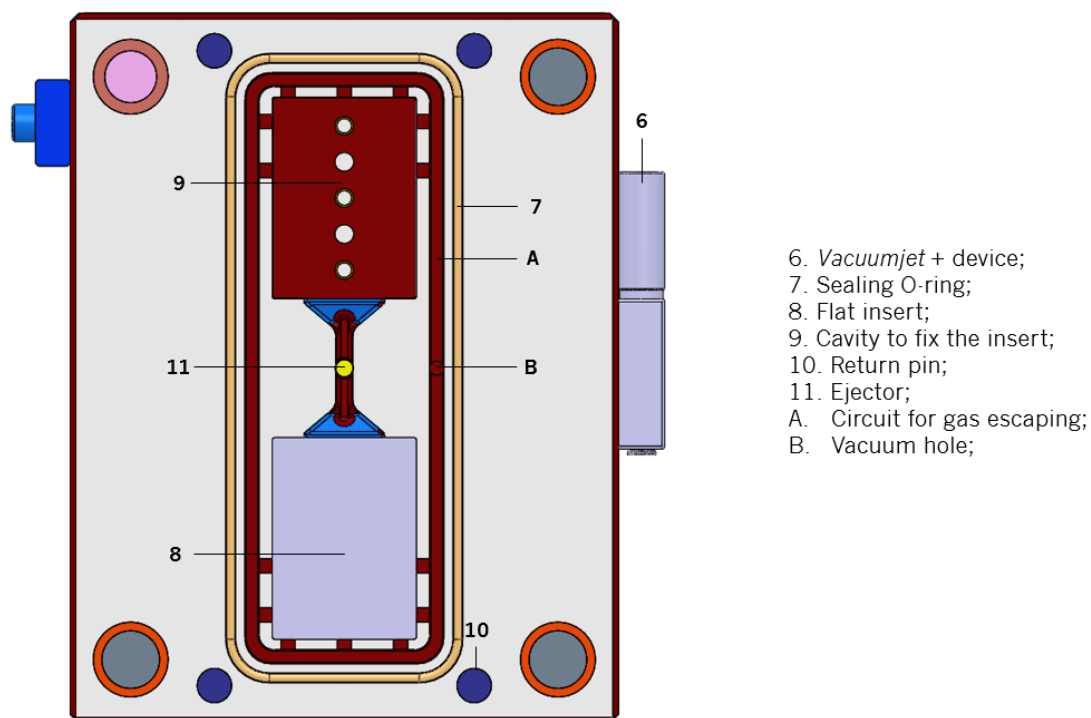


Figure 108 - Front view of ejection side moulding plate and main elements of vacuum system (6, 7, A and B) and ejection system (10 and 11).

D. Chemical analysis performed in preliminary study 1

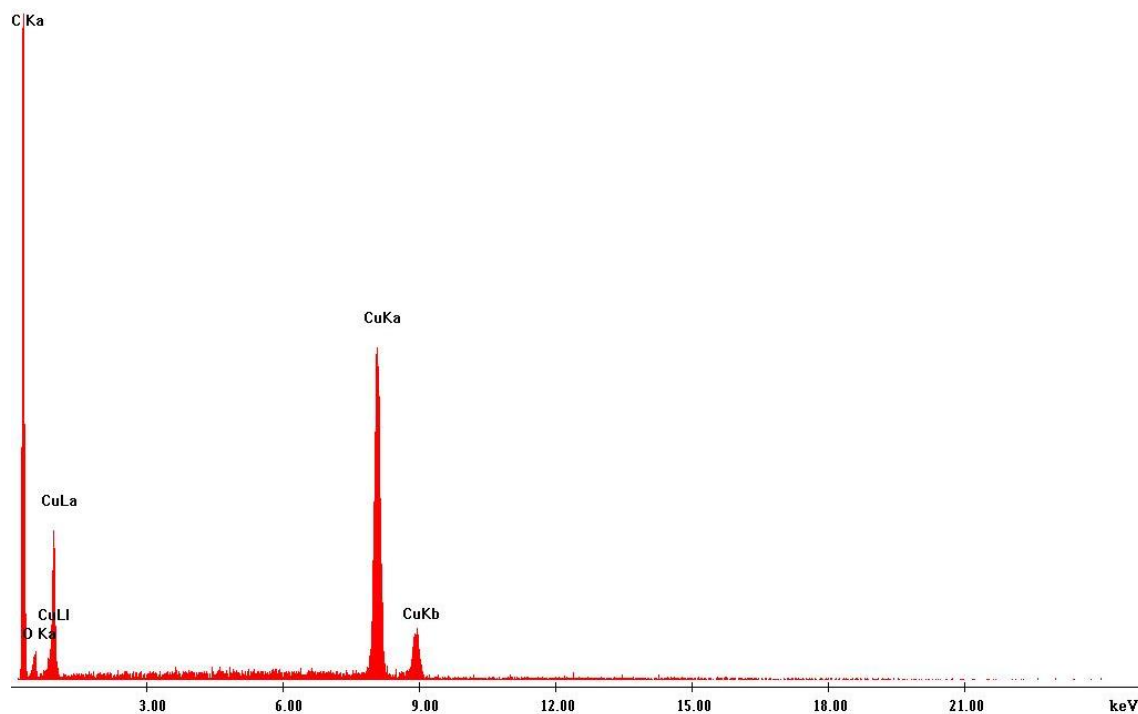


Figure 109 - Chemical analysis performed in Z1 area of copper mould insert.

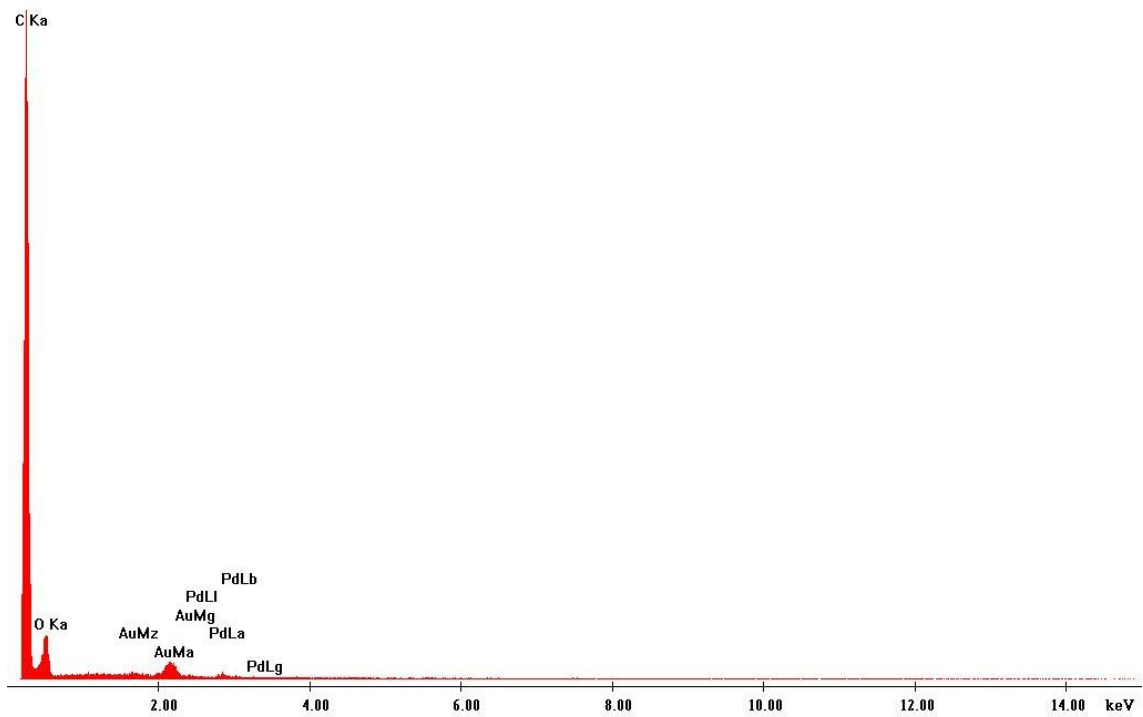


Figure 110 - Chemical analysis performed in a PC sample.

E. Case of study (Preliminary study 2)

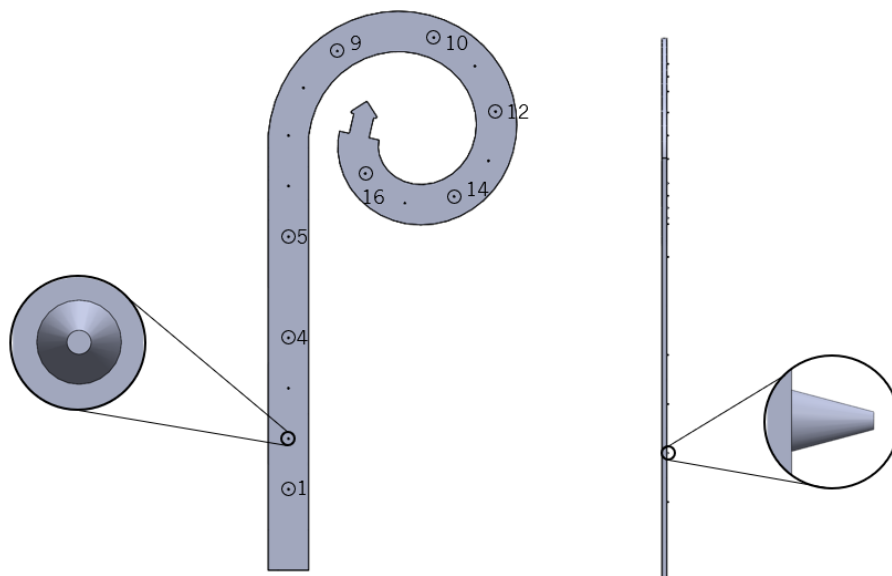


Figure 111 - Case of study: detail of microstructures. Identification of the studied microstructures.

F. Filling results of Design of Experiments from Moldex3D simulations

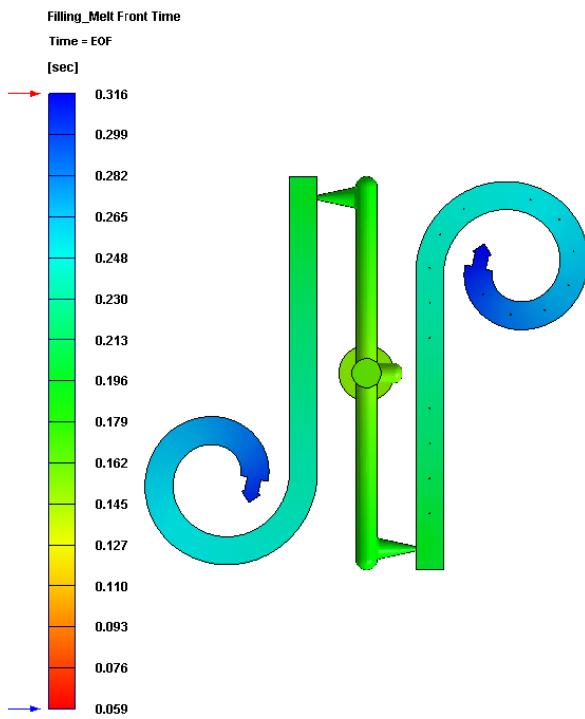


Figure 112 – Simulation filling results for the conditions of Run 1.

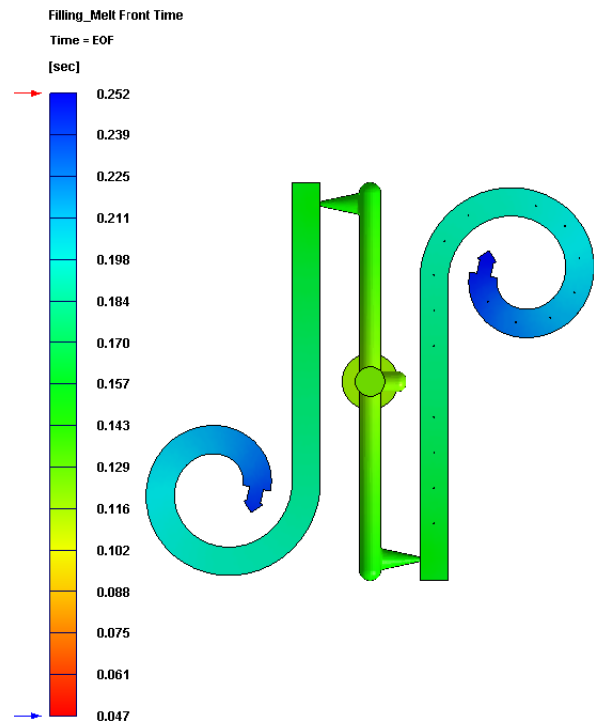


Figure 113 - Simulation filling results for the conditions of Run 2.

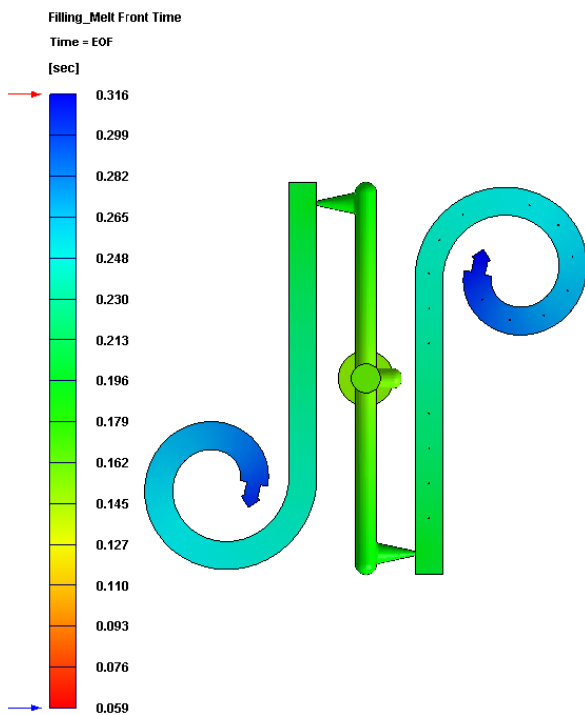


Figure 114 - Simulation filling results for the conditions of Run 5.

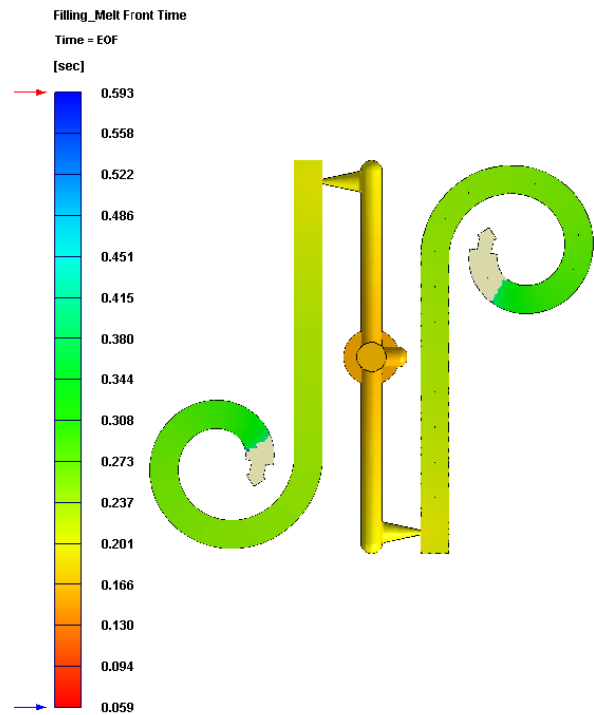


Figure 115 - Simulation filling results for the conditions of Run 6.

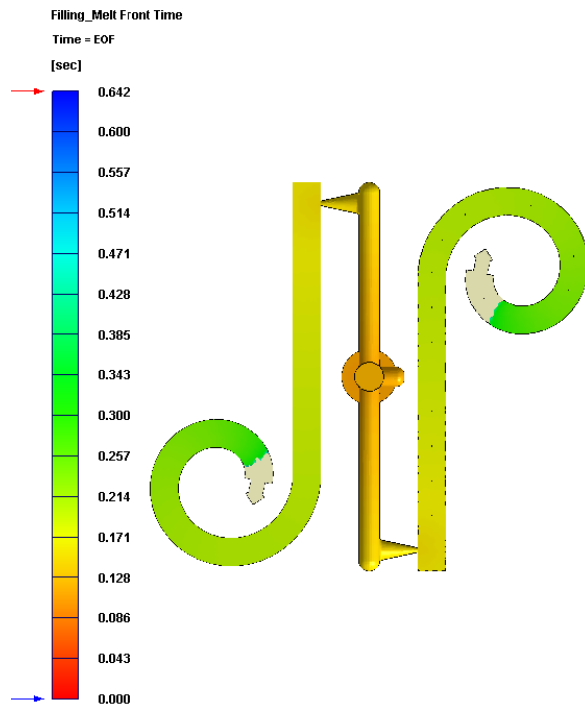


Figure 116 - Simulation filling results for the conditions of Run 7.

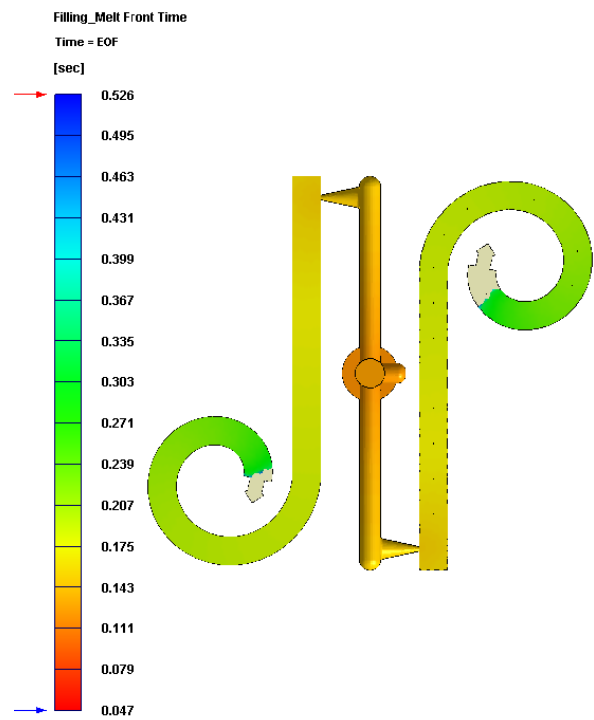


Figure 117 - Simulation filling results for the conditions of Run 8.

G. Results of replicated height of each sample per run (study 2)

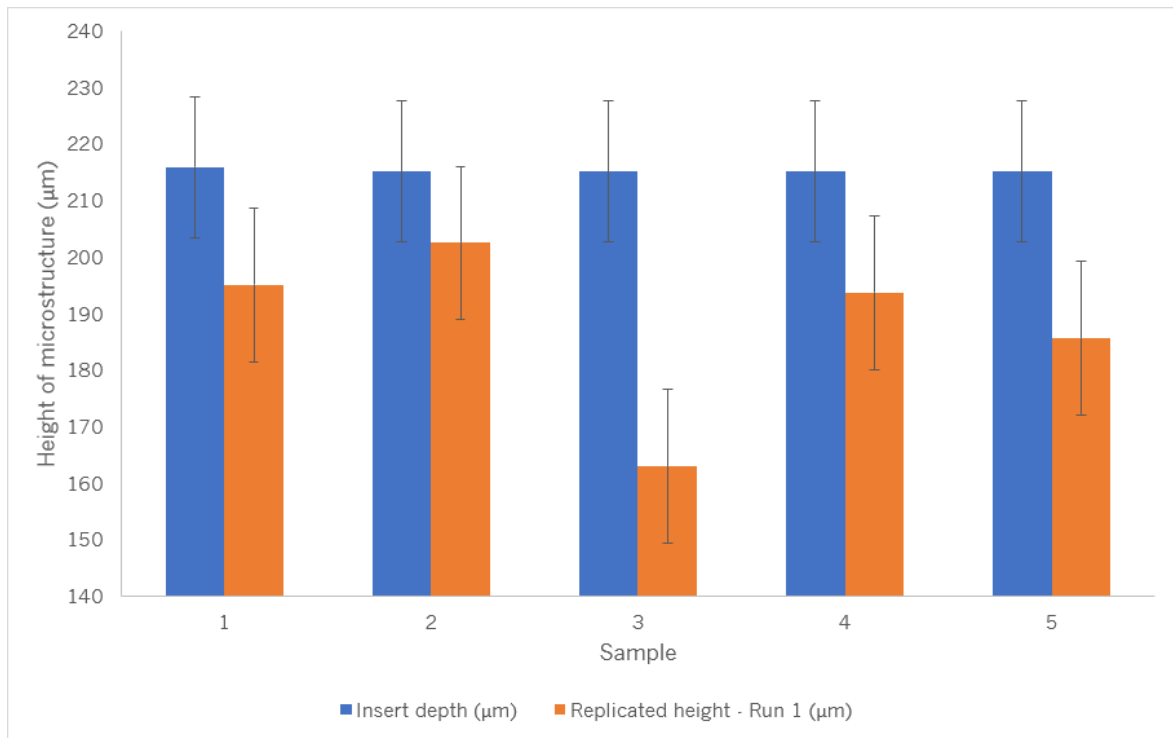


Figure 118 - Comparison between insert depth and replicated height (per sample) for run 1.

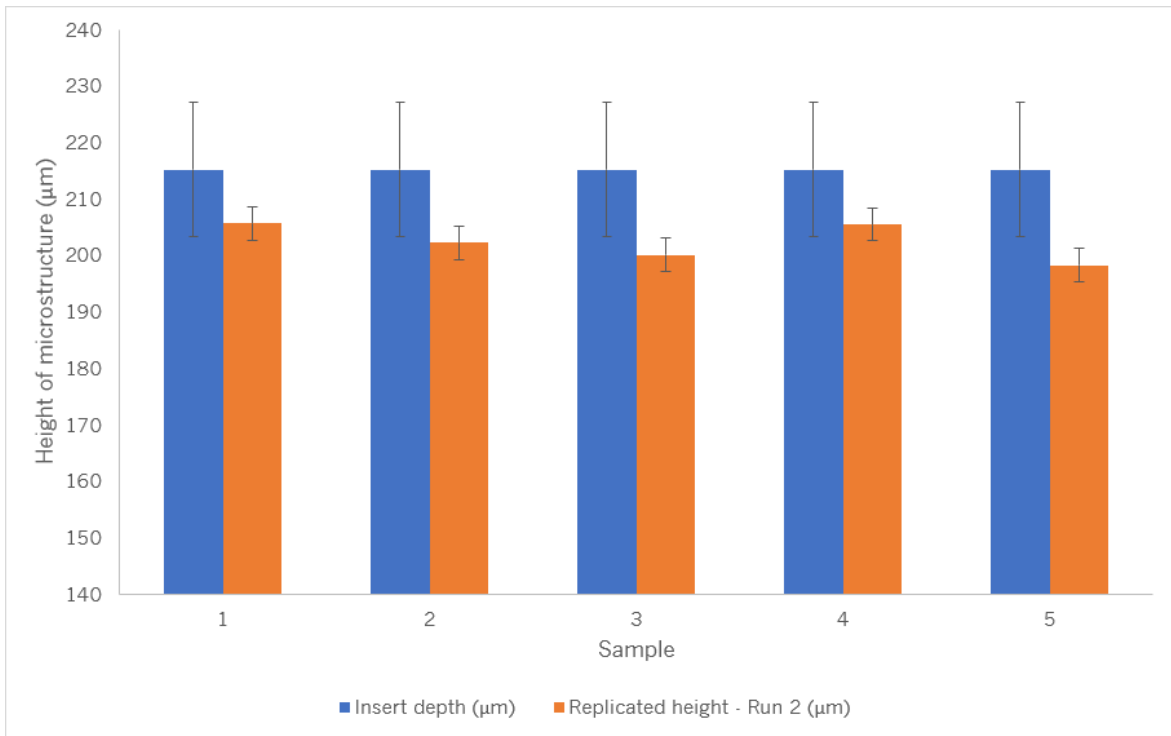


Figure 119 – Comparison between insert depth and replicated height (per sample) for run 2.

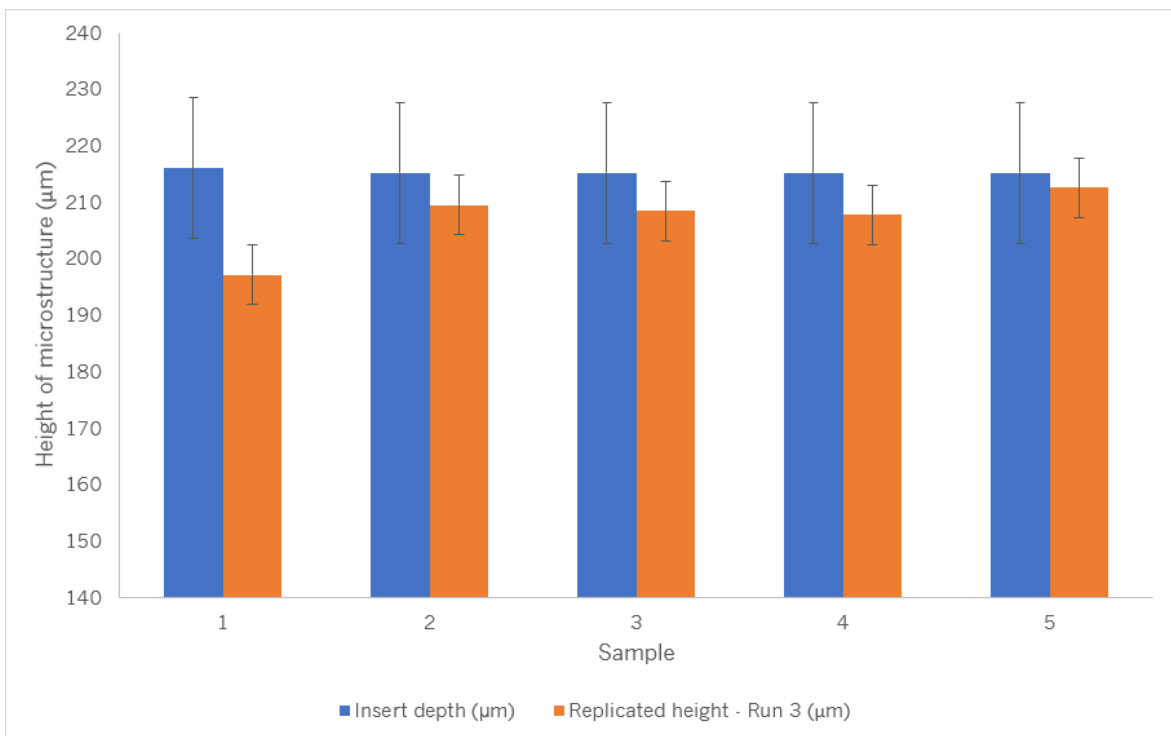


Figure 120 - Comparison between insert depth and replicated height (per sample) for run 3.

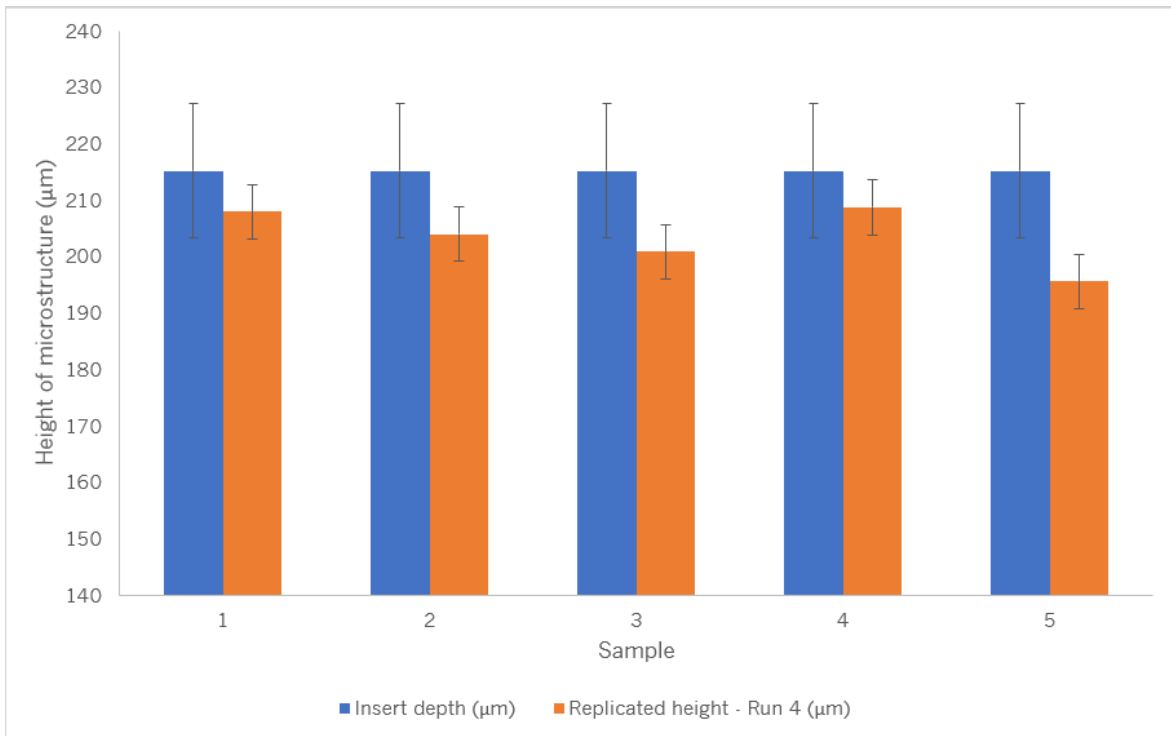


Figure 121 - Comparison between insert depth and replicated height (per sample) for run 4.

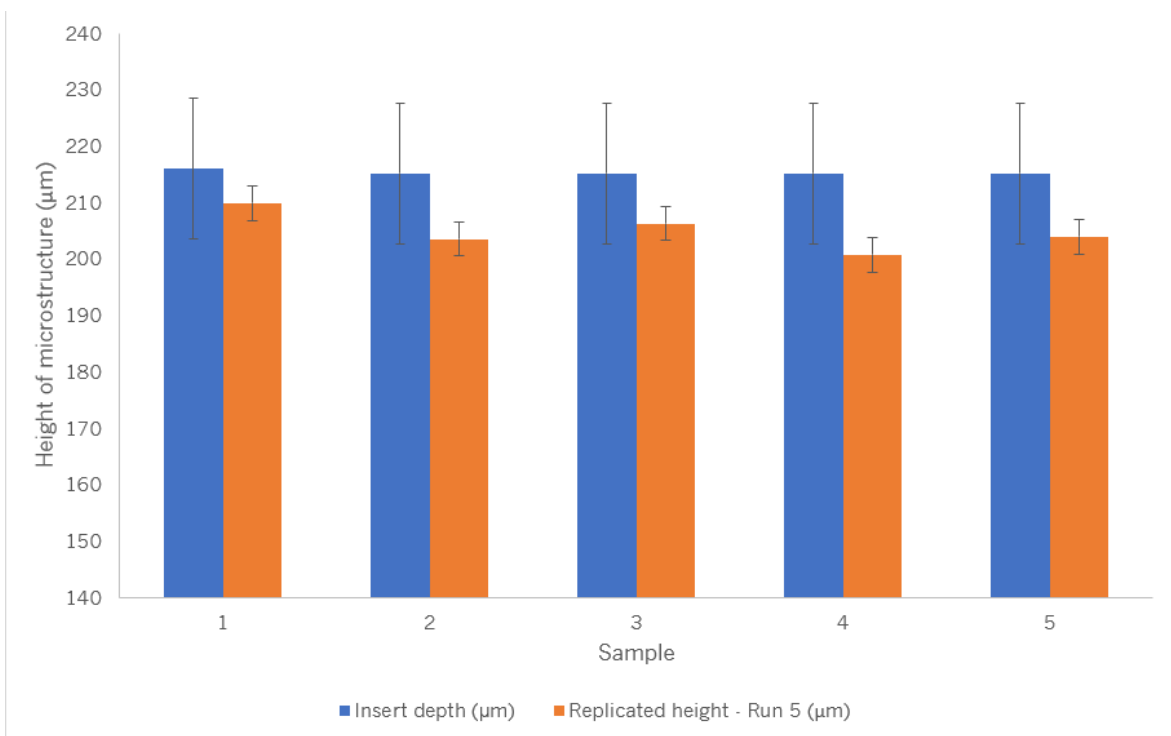


Figure 122 - Comparison between insert depth and replicated height (per sample) for run 5.

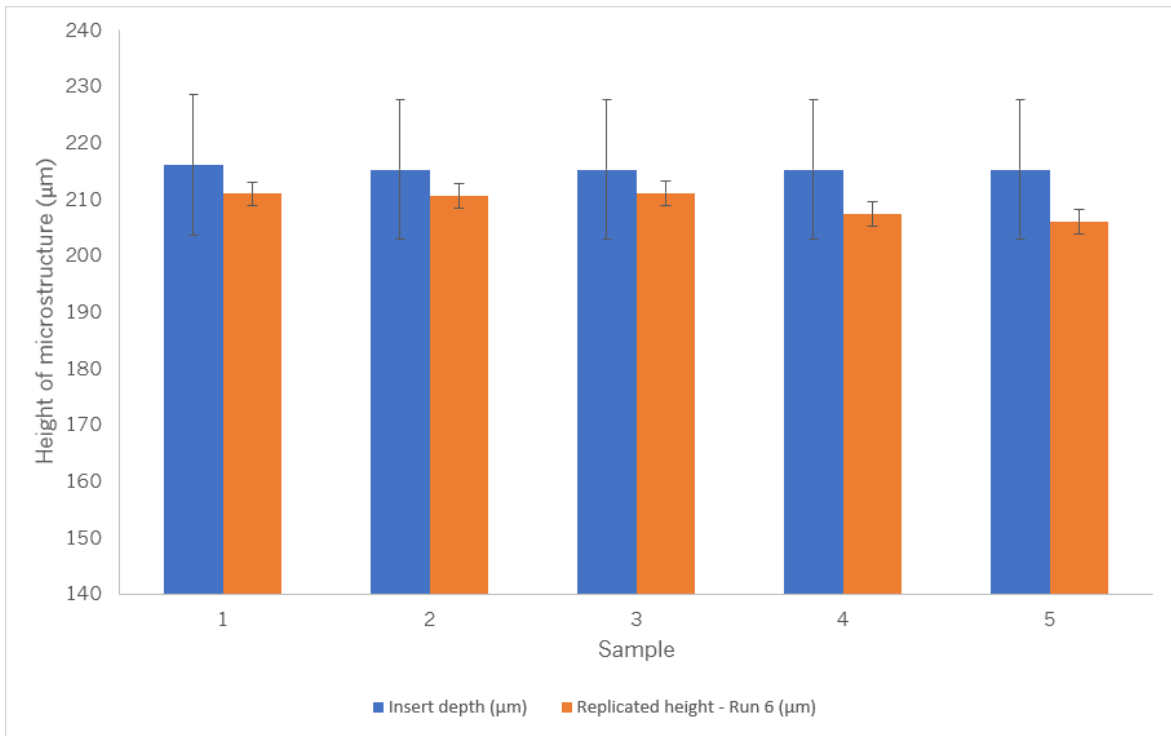


Figure 123 - Comparison between insert depth and replicated height (per sample) for run 6.

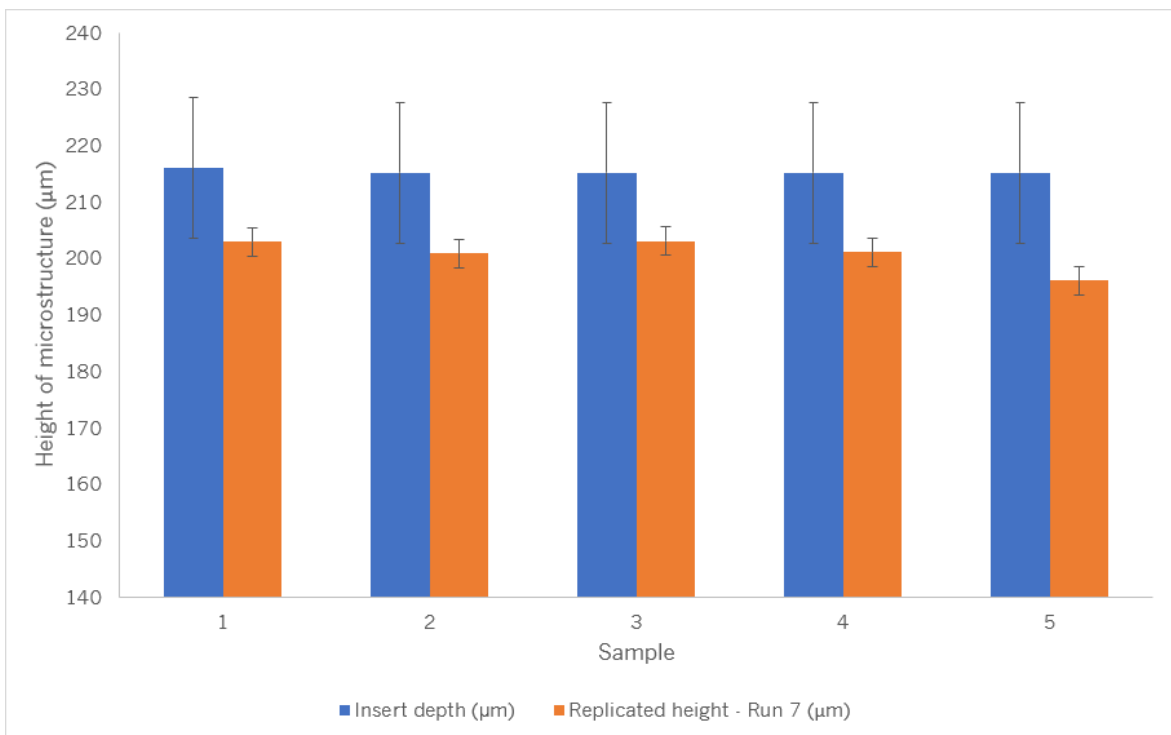


Figure 124 - Comparison between insert depth and replicated height (per sample) for run 7.

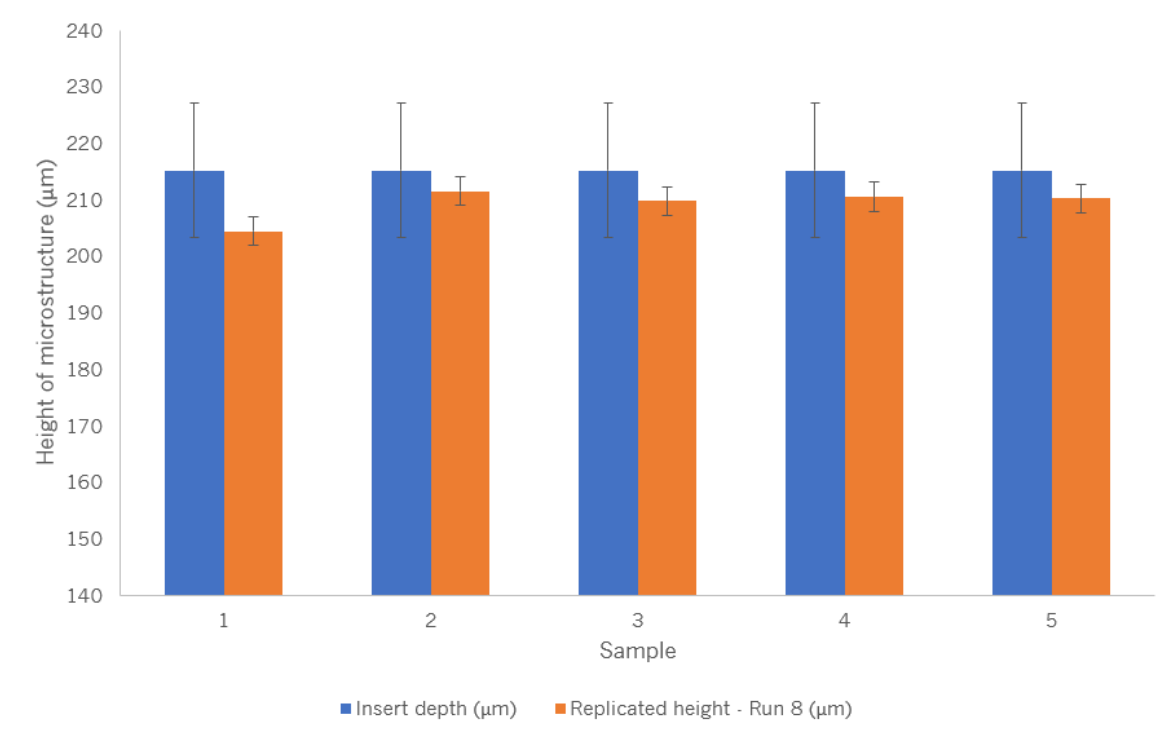


Figure 125 - Comparison between insert depth and replicated height (per sample) for run 8.

H. *Moldex3D* simulation of protective optical window prototype filling conditions

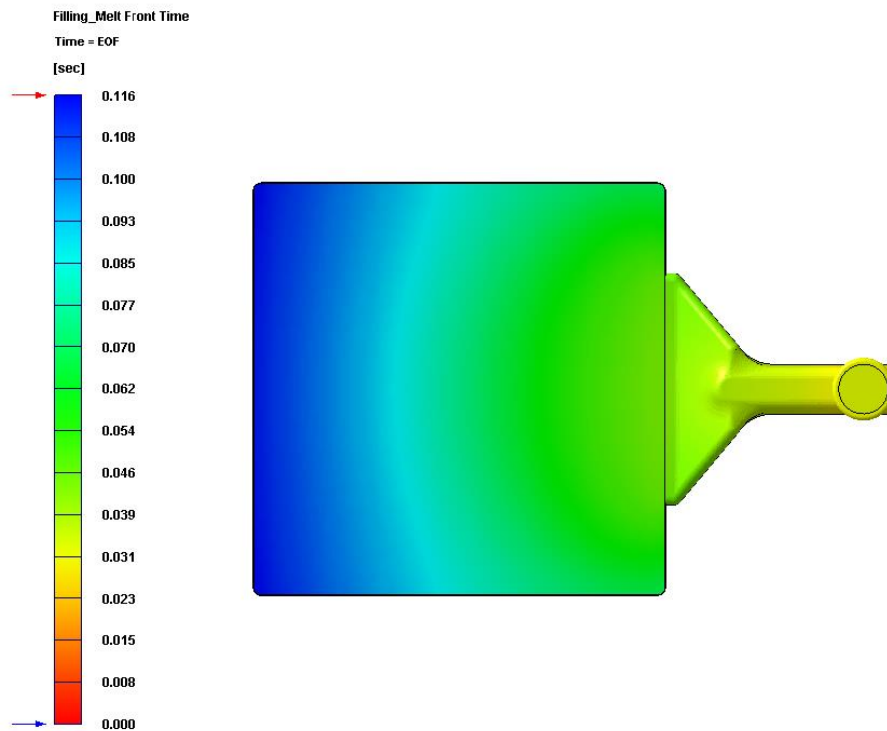


Figure 126 - *Moldex3D* simulation result of protective optical window filling time.

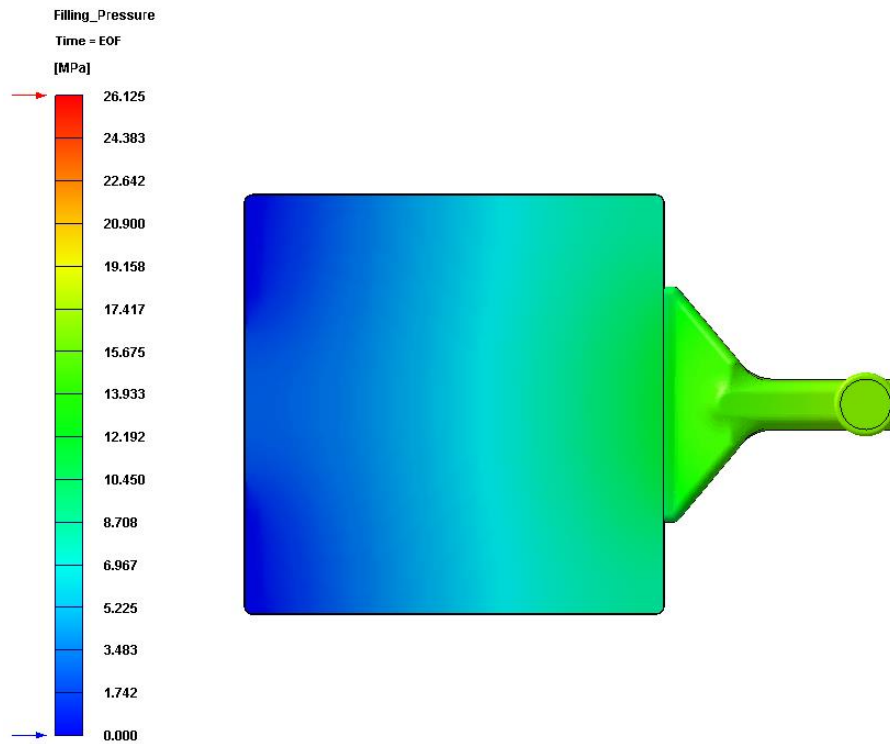


Figure 127 - Moldex3D simulation result of protective optical window filling pressure at end of filling (EOF).

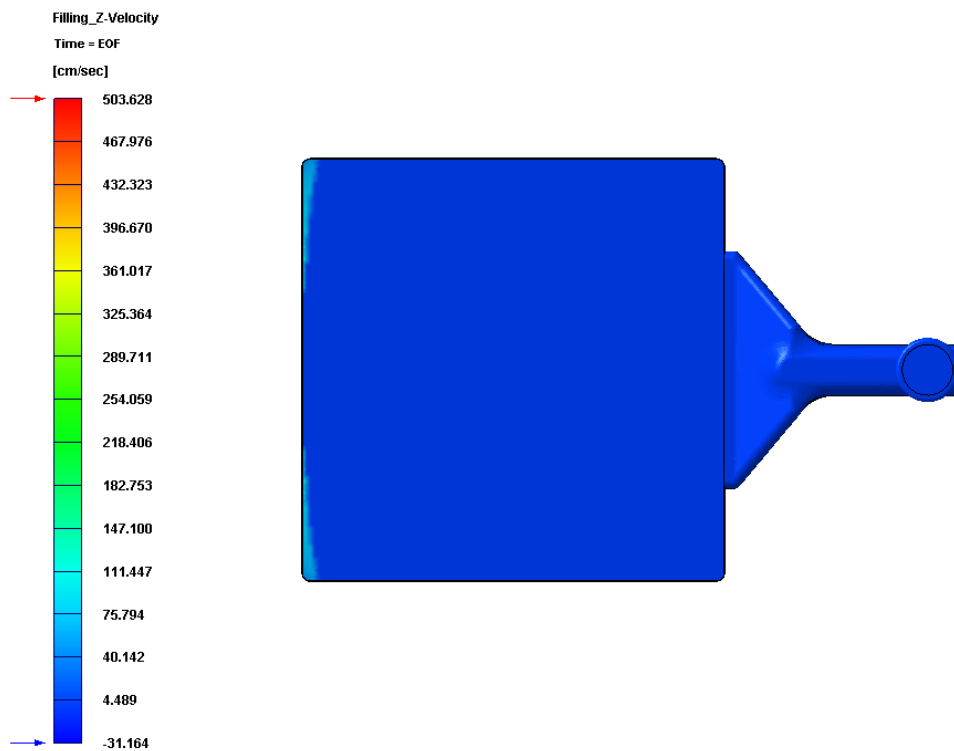


Figure 128 - Moldex3D simulation result of protective optical window injection speed during filling.