Testing mold design for investigation of powder-binder separation during powder injection molding

Bc. Lukáš Jiránek

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Zásady pro vypracování:

The aim of this diploma thesis is step by step description of the major quality influencing factor for metal injection moulding (MIM) — phase separation. Phase separation might cause visual defects, porosity, warpage and cracks. The theoretical part of this diploma thesis generally describes MIM technology, its advantages and disadvantages, MIM process and its main stages -- feedstock preparations, injection moulding, debinding and sintering. This part also focuses on a theoretical definition of the phase separation. Following practical part consists design proposal of testing injection mould, software simulations, optimization of injection moulding for various commercially available feedstock compositions. Keywords: phase separation, metal injection moulding, mould design, software simulations, debinding, sintering.

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doc. Ing. Petr Hlaváček, CSc.
děkan

UNIVERZITA TOMÁŠE BATI VE ZLÍNĚ
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ABSTRAKT

Cílem této diplomové práce je konstrukce testovací vstřikovací formy. Tato forma by měla sloužit k lepšímu pochopení jevu separace fází, jenž ovlivňuje kvalitu produktů vyrobených technologií vstřikování prášků (PIM technologií). Separace fází zapříčiňuje vizuální vady, pórovitost, zborcení a tvorbu prasklin. Teoretická část diplomové práce obecně popisuje technologii vstřikování prášků, její výhody a nevýhody, proces výroby a jeho hlavní fáze – příprava směsi, injekční vstřikování, odstranění pojiva a slinování. Tato část je také zaměřena na teoretickou studii separace fází. Následující praktická část obsahuje návrh testovacího tělesa, konstrukci vstřikovací formy včetně modelu a výkresové dokumentace. Dále jsou uvedeny výsledky ze zkušebního vstřikování různých komerčně dostupných PIM kompoundů.

Klíčová slova: separace fází, návrh testovací formy, injekční vstřikování kovových a keramických prášků.

ABSTRACT

The aim of this master thesis is to design a testing injection mold, which would help us to understand a quality influencing factor in powder injection molding (PIM) – a phase separation. Phase separation might cause visual defects, porosity, warpage and cracks. There are two parts of this master thesis. The theoretical part describes PIM technology in general, its advantages and disadvantages, PIM process and its main stages – feedstock preparations, injection molding, debinding and sintering. This part also focuses on a theoretical study of the phase separation. The second part is practical and consists of a design proposal of the testing specimen, a description of a construction of the injection mold, its drawing documentation and any results from injection molding of various commercially available feedstock compositions.

Keywords: phase separation, mold design, powder injection molding, metal and ceramic powders.

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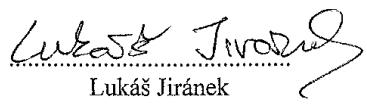
" Try not to become a man of success but rather to become a man of value. "

Albert Einstein (*1879 - †1955)

I hereby declare that the print version of my Master's thesis and the electronic version of my thesis deposited in the IS/STAG system are identical.

Zlín, 7th May 2010



Lukáš Jiránek

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INTRODUCTION

Powder injection molding (PIM) is a production process, which eliminates shape and material restrictions inherent in conventional metalworking technologies and in production of ceramic components. PIM technology was developed as a branch of powder metallurgy industry. However, also more complex shape compared to usual powder metallurgical sintered part is gained. This feature is reached by combining the plastic injection molding process and powder metallurgy, while using a mixture of fine metal/ceramic powder and binder. Such a feedstock allows us to produce a powder rich part by injection molding, from which the binder is extracted, and subsequently the part is sintered. Therefore, products made by PIM technology can take an advantage of the material flexibility of the powder metallurgy and the design flexibility of plastic injection molding. These advantages of PIM allowed the technology to spread all over the world and this trend is still continuing. The parts manufactured by either metal injection molding (MIM) or ceramic injection molding (CIM) process are found in numerous industrial applications, including aerospace, automotive, medical, jewellery, IT, telecommunications and electronic.

The theoretical part of this master thesis includes a general description of metal/ceramic injection molding process, advantages and disadvantages of PIM technology. Also a literature study of the different existing theories [1, 2] and practical knowledge of the phase separation are presented. The phase separation is a major quality issue during injection molding of a PIM feedstock. The practical part focuses on a design of a testing mold, which would help us to quantify the phase separation. Therefore, the most critical geometrical elements for the phase separation had to be implemented into the cavity of the testing mold. The drawing documentation is also included. The desired outcome is captured on the pictures enclosed in the experimental part of this master thesis.

I. THEORETICAL PART

1 POWDER INJECTION MOLDING

1.1 Characteristics of a PIM product

As previously mentioned in the introduction, products made by PIM technology can take advantage of material flexibility of powder metallurgy and the design flexibility of plastic injection molding. Before a detailed description of a PIM process is given, it is necessary to explain how these features could evoke such a rapid growing market share of this technology in metalworking industry and in production of ceramic components. As in any other technology there is a group of products, which benefit from using PIM and another group which is not suitable for being produced by PIM. To have a better idea of products suitable and those being currently produced by PIM technology, some cases - awarded and published by Metal Powder Industries Federation (MPIF) [3] - are displayed as application examples on the following pages.

Articulation gear for a surgical stapling device

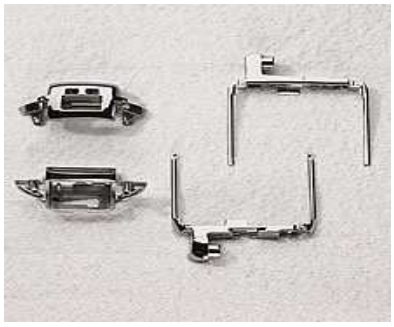
Parmatech Corporation, Petaluma, California, USA won the MPIF 2008 grand prize in the medical/dental category for a 17-4PH stainless steel MIM articulation gear used in a surgical stapling unit. Produced to a density of more than 7.65 g/cm^3, the part has an ultimate tensile strength of 900 MPa, yield strength of 730 MPa, and 25 HRC hardness. MIM was selected as a performance improvement over plastic and for dramatic cost savings over a machined aluminum solution. A challenging design of this part included an interrupted gear tooth with a window, overmolding with plastic and assuring a seal in the over mould. This complex MIM design is formed to a net shape and requires no finishing operations. MIM production provided a 70 % cost saving, compared to machining the gear from bar stock. Approximately 20,000 pieces are produced annually.

Orthodontic bracket, slide and removable drop-in hook

Flomet LLC, DeLand, Florida, USA won the MPIF Grand Prize in 2007 for three parts - bracket, slide, and drop-in hook - used in the Damon 3MX self-ligation orthodontic tooth positioning system. The very tiny, intricate parts are made by MIM from 17-4PH stainless steel powder to a density of 7.5 g/cm^3. They have impressive physical properties: a tensile strength of 1,180 MPa and a yield strength of 1,090 MPa. All of the parts are made to a net shape. The customer tumble polishes them and performs a brazing operation before assembly.

Trigger guard for hunting rifle

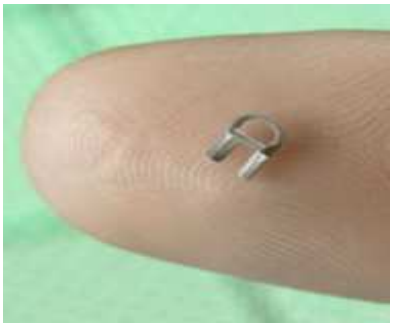
Megamet Solid Metals Inc., Earth City, Missouri, USA, won the 'Hand Tools/Recreation' category in 2007 for a rifle trigger guard made by MIM for Modern Muzzleloading, Inc., Alabama, USA. The MIM guard supports the trigger group and hammer in the "quick detachable trigger" mechanism in a 50 caliber muzzle-loading hunting rifle. Produced to a density of 7.4 g/cm^3, the 88 g MIM steel part has an as-sintered tensile strength of 650 MPa and 400 MPa yield strength. The part is held to critical dimensions of +/- 0.1 mm. Secondary operations include reaming three holes, tapping two screw holes, and deburring. The customer applies the black oxide surface finish and drills one hole because of a design change. It is claimed that the MIM process offered substantial cost savings.



Mobile phone component

Mobile phone component won the grand prize in the electronic/electrical components category of the 2006 MPIF awards. The parts are made by Advanced Materials Technologies Pte Ltd, Singapore. The MIM parts make up the dual-hinge opening mechanism in Motorola's PEBL mobile phone. The MIM process allowed a very complex thin wall, overhanging structures and three-dimensional design. Properties include: tensile strength of 1,185 MPa, yield

strength of 1,090 MPa and 30 HRC hardness by using 17-4PH stainless steel sintered to a density of 7.6 g/cm^3. Both parts are coined, machined, polished and plated. Machining these parts, an alternative fabrication method, would have cost five times more than MIM.



Medical pin shroud implant device

Kinetics, a Climax Engineered Materials Company, Wilsonville, Oregon, USA, has earned the Award of Distinction in 2007 for a 316L stainless steel metal injection molded pin shroud. The critical part is used in the device for arthroscopic surgical repair of torn rotator cuffs. The implant device secures a sutured tendon to the shoulder bone. Made close to a net shape, the MIM pin has a typical density of 7.85 g/cm^3, a tensile strength of 540 MPa, and yield strength of 200 MPa. Metal injection molding replaced an assembly made of three parts and assembled together by laser welding. Choosing the single MIM part reduced the customer's final assembly time from 15 minutes to just 5 minutes per unit.

Above shown examples can be generalized into guidelines for choosing a part suitable for a PIM production. Characteristics of a PIM production can be divided into its advantages and disadvantages.

Advantages of using PIM process [4, 5, 6]:

- ✓ PIM is outstanding at reducing fabrication costs for components where large material scrap would occur when using machining or grinding.
- ✓ PIM is favorable for components that are difficult to machine due to their design.
- ✓ Benefits arise when multiple parts can be consolidated by using PIM into a single piece to save on inventory and assembly costs.
- ✓ PIM can form novel material combinations that are difficult via traditional processes, for example laminated or two-material structures or mixed metal-ceramic materials.

Disadvantages of using PIM process [4, 5, 6]:

- Size of the component and wall thickness is limited by the price of fine powders and by the technologies being used in PIM process.
- PIM as an injection molding technology creates defects that must be located in noncritical positions or removed after fabrication; examples include gates marks, ejector pin marks, or parting lines.
- Tooling and set-up costs are difficult to justify for low production quantities, thus PIM works best when annual production quantities of an expensive parts exceed 20,000. For automotive industry and other lot productions, PIM is usually attractive for annual production of more than 200,000 parts [7].
- PIM tends to have a relatively high cost from secondary operations if tight tolerances are required; the as-sintered tolerances are generally around ± 0.3 %.

1.2 PIM process

Powder injection molding process is using a feedstock containing fine metal or ceramic powder and binder. After homogenizing metal/ceramic powder and binder, the feedstock is granulated into pellets. Pelletized feedstock can be molded on an injection molding machine commonly used in the plastics industry. After molding the binder is extracted and the part is sintered in the furnace to a nearly theoretical density.

Therefore, resulting elemental stages of the PIM process are:

- Mixing
- Injection molding
- Debinding
- Sintering

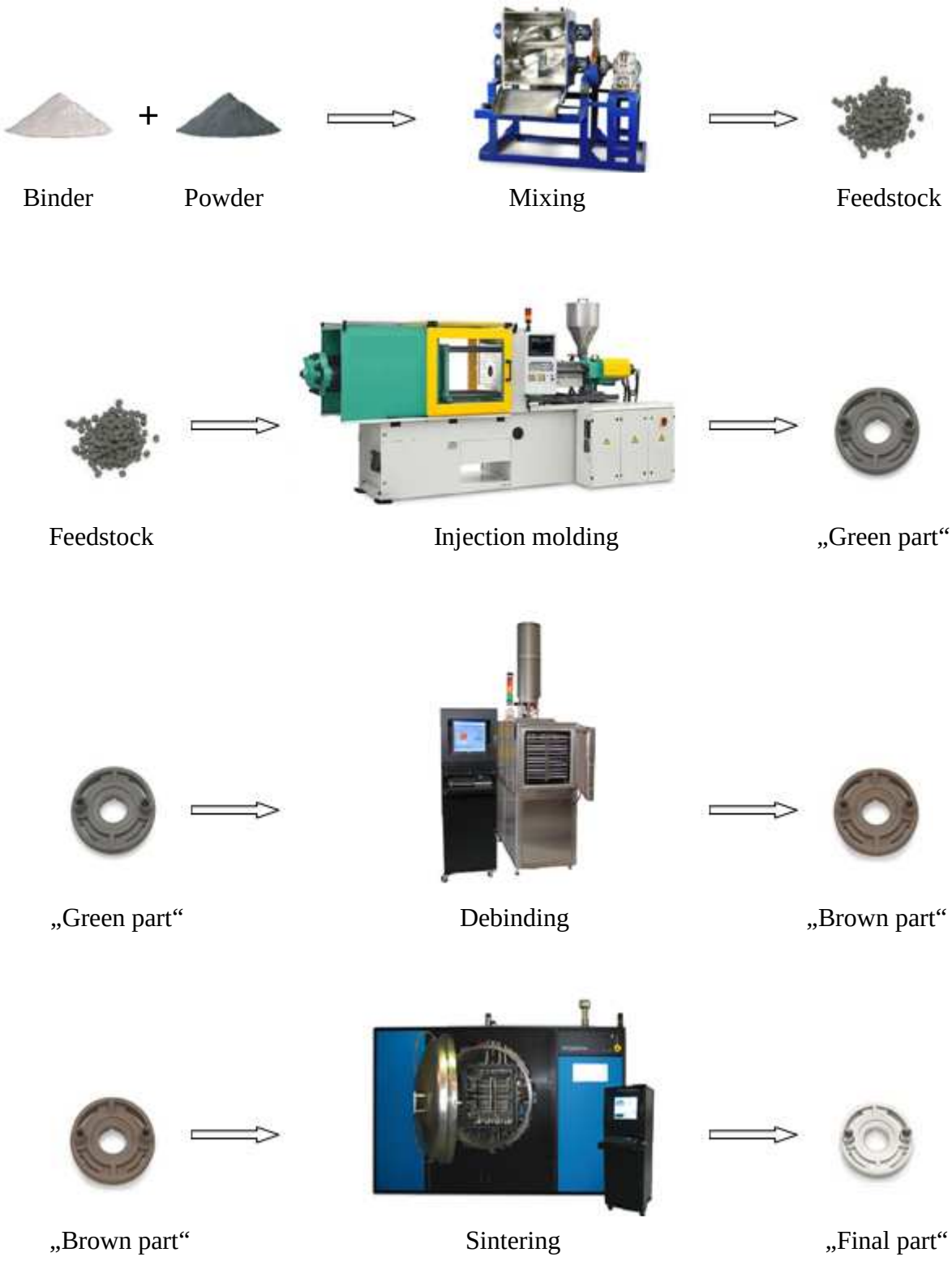


Fig. 1: PIM process

Following pages will give a closer look at the fine powders and binders being used in PIM and also each stage of the PIM process will be described in full details.

1.2.1 Metal and Ceramic powders

The listing of metals that can be used includes many common and several less common metals and their alloys - plain and low alloy steels, high speed steels, stainless steels, super alloys, magnetic alloys and hard metals. Simply said any metal can be processed by using MIM except pure aluminum due to its oxide film on the surface, which inhibits sintering. As for ceramics, any material processed by conventional ceramic forming techniques can be processed by CIM. Examples include Al_2O_3, TiO_2, ZrO_2, silica graphite, and boron nitride. From an economic point of view the most suitable materials for PIM are the most expensive ones. As a closed-cycle technology there is no scrap, therefore the saving of costs grows along with a price of the material being used [4, 8].

No matter which kind of powder is used, there are two main characteristics – a particle shape and a particle size distribution. Apart from these main factors, several others have to be considered when planning PIM production, for example non-toxic particles surface for a predictable interaction with the binder, average particle size, availability of the powder on the market and cost. Let us look at some requirements, for fine metal and ceramic powders used for PIM processing [4, 9, 10]:

- Spherical or near spherical shape is preferred due to its more predictable flow behavior in the binder during mixing and injection molding, but the risk of the skeleton going out of shape during the debinding process is increased;
- Finer powders sinter more readily than coarser powders, but fine powders particles tend to agglomerate and this is undesirable;
- Finer powders also improve surface finish as compared to coarser powders;
- Ideal powder should combine large and small particles in tailored particle size distribution, which provides high packing density and suitable viscosity along with low cost;
- Particle size of industrially used powders is between 0.1 and 20 µm, which is ideal for rapid sintering;
- Dense particles free of internal voids are desired as well.

In reality, the choice is restricted to what is available on the market, but increasing demand motivates powder manufacturers to meet the special requirements of PIM. Also new technologies in powder production are implemented to extend the range of fine metal and ceramic powders for PIM producers [4, 5].

1.2.2 Binders

The other component of PIM feedstock is a binder. The binder composition represents one of the most critical factors in PIM. It is the binder that allows us to shape metal/ceramic powders with injection molding. Therefore, it is necessary to lubricate particle sliding during injection molding. It means that the powder particles are wet by the binder system during mixing. The least possible amount of binder should be used, but the viscosity that allows us to use injection molding technology has to be reached. Easy binder removal has to be considered as well. Typical binder consists of two or three components with at least one non-reactive during debinding process, keeping the shape of the part prior to sintering. There are various kinds of binder used practically. Each of these binder systems has its own debinding method which is the most important difference for PIM producers. Debinding methods are described and compared in the later stage of this thesis. First of all, let us see what are the components and characteristics of each binder system [4, 11, 12]:

Binder	Main ingredients	Polymer backbone	Additives
Wax-based	paraffin, carnauba, microcrystalline, bees wax	PE, PP, PS, PA, PMMA, EVA	stearic acid
Oil-based	peanut, vegetable, palm oil	PE, PP, PS, PA, PMMA, EVA	oleic acid, phtalic acid esters
Polyacetal	POM	Non-catalytical polymer immiscible with POM	PEO oligomers, stearic acid, fatty alcohols
Water-soluble	PEO, PVA	PE, PP, PS, PA, PVA, PMMA	surfactants, aliphatic monocarboxylic acids
Water-based	water	agar, methyl cellulose	glycerin, boric acid
Thermosetting	epoxy resin, aniline	paraffin	butyl stearate

Tab. 1: Composition of binder systems

Wax-based binder systems could be called traditional ones. As an example such a wax-based binder can consist of 65 vol. % paraffin wax, 30 vol. % polypropylene and 5 vol. % stearic acid. The usual content of this binder system in the PIM feedstock is around 40 vol. %, but the binder content is variable and strongly depends on the characteristics of the fine powder. The thermoplastic binders can be debinded by several methods – by solvent, thermal, catalytic or supercritical debinding [11].

Oil-based binders are not widely used due to their low toughness after molding. More often, a combination of wax and oil as main ingredients is used [9, 12].

Polyacetal binders provide good moldability and excellent shape retention. Binder removal is done in highly concentrated nitric or oxalic acid. That brings some concerns for the producers about health and safety regulations in their production and manipulation. This type of binder can be easily used for a continuous binder removal and sintering process, because the part is not as brittle as the one produced with the thermoplastic binder. The polyacetal binder as developed by BASF company as Catamold® - being sold as ready to use formulation with different powders such as stainless, heat resistant and tool steels, titanium, tungsten and oxide ceramics. This patented binder system and its debinding technology is the most reliable one in the production and hence it is widely used [5, 13].

Water-soluble binders use polyethylene oxide or polyvinyl alcohol, which are hydrophilic. Therefore, these binders can be debinded by using water as a solvent [9, 12].

Water-based binders offer the ability to produce larger and thicker parts than previously mentioned binders. It is possible to debind parts weighing up to 2 kg. But the main limitation of this binder system is its low toughness after molding, which can cause a distortion or warpage of the part. There is no need for an extra debinding step as the debinding is usually performed during the first hour of sintering. The water-based binders are based on agar, a water soluble polysaccharide (derived from seaweed), and a gel-forming material solvent (water addition around 55 % by volume, which is an amount sufficient to dissolve the gel-forming material – agar, at the melting temperature of the binder system). This system is environmentally friendly and hence the research and development of binder systems is focused on this area [9, 10].

Binder systems containing thermosets, which crosslink when injected molded, may be advantageous for the reason that a crosslinked polymer transforms from solid to gas during debinding. This process is avoiding melting, which can cause the deformation during

debinding. However, thermosets are impractical because they do not allow reusing the feedstock in scrapped parts after injection molding. This is an important economical aspect for producers because of the high cost of fine powder in feedstocks [12].

1.2.3 Mixing

As mentioned previously, each particle surface has to be covered with the binder. To ensure a precise homogenization and no particle agglomeration, sufficient shear stress is necessary. For this reason planetary mixers or Z-blade mixers would be the preferred option. The temperature of a pre-heated mixing barrel depends on the type of the binder system. Generally, mixing temperatures for thermoplastic binders are above 100 °C and for gellation binders around 80 °C. Mixing can take up to couple of hours. This mode of mixing is called a batch mixing. It means that all components are loaded into the mixer and mixed until the feedstock is homogenized. Then the feedstock is discharged from the barrel and the mixer is ready for the next cycle. Planetary or Z-blade mixers are often used as a laboratory equipment to produce feedstocks [4, 11].

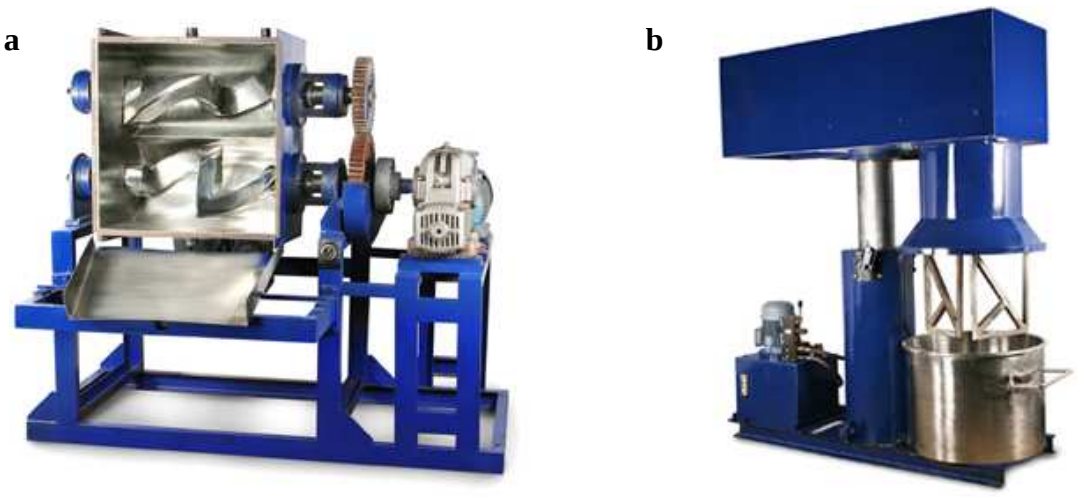


Fig. 2: Batch mixers. Z-blade mixer (a); planetary mixer (b)

In a lot production, where a high volume of feedstock is needed, twin screw extruders or shear rolls are used for the feedstock homogenization. Feedstock is usually mixed twice on these machines or premixed by planetary or Z-blade mixers first and afterwards homogenized with twin screw extruders or shear rolls. This extra homogenizing step for

the high volume of feedstock brings absolute certainty to the producer that the feedstock is homogenized properly. After homogenizing, the feedstock is converted into pellets by a granulation process. The granulation process is usually carried away right behind an extrusion head or at the end of a shear roll [4, 11].

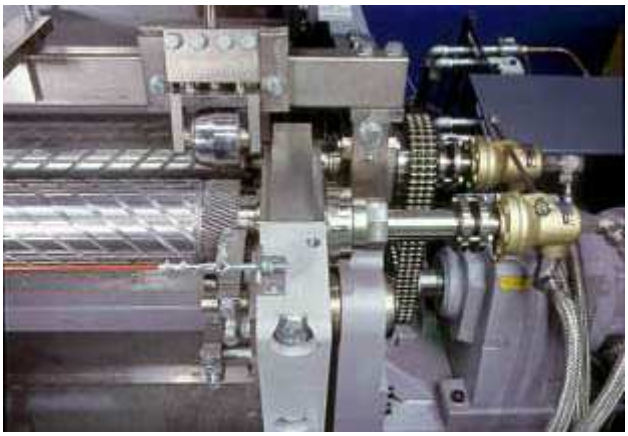


Fig. 3: Granulation unit at the end of a shear roll

1.2.4 Injection molding

Injection molding is a technique well known in the plastic industry. It is a process that transforms the feedstock into a shape of the part. For this transformation an injection molding machine and a mold are used. Although there is no fundamental difference in the injection molding in PIM process and the one in plastics industry, there are injection molding machines specially offered to MIM/CIM producers. These machines are optimized for the processing of powder materials and feature highly wear-resistant cylinder and screw geometry, which is especially adapted to powder injection molding. Such geometry benefits from lower compression rate and extended compression zone compared to standard screws for thermoplastics. These modifications allow lower shear heating, and therefore reduce wear. Commonly used injection molding machines are also suitable for PIM, however higher abrasion arising from the feedstock has to be considered. Injection molding machine generally consists of a clamping and an injection unit [14, 15].

The clamping unit secures the closure of the injection mold before injecting the melted feedstock. The hydraulically powered clamping unit allows one side of the mold to slide,

when the other side is attached to the injection molding machine. Injection molding machines are usually categorized by its maximum clamping force [16, 17].

The injection unit consists of four main parts – hopper, reciprocating screw, heated barrel and nozzle. The injection unit is heating and injecting the material into the mold. At first, the hopper is filled with feedstock pellets and this material is fed into the barrel. In the barrel the feedstock is melted by pressure, friction, and additional heaters and is moved towards the mold as the screw rotates and slides axially. Melted feedstock is injected into the mold through the nozzle at the end of the barrel by a forward action of the screw [16].

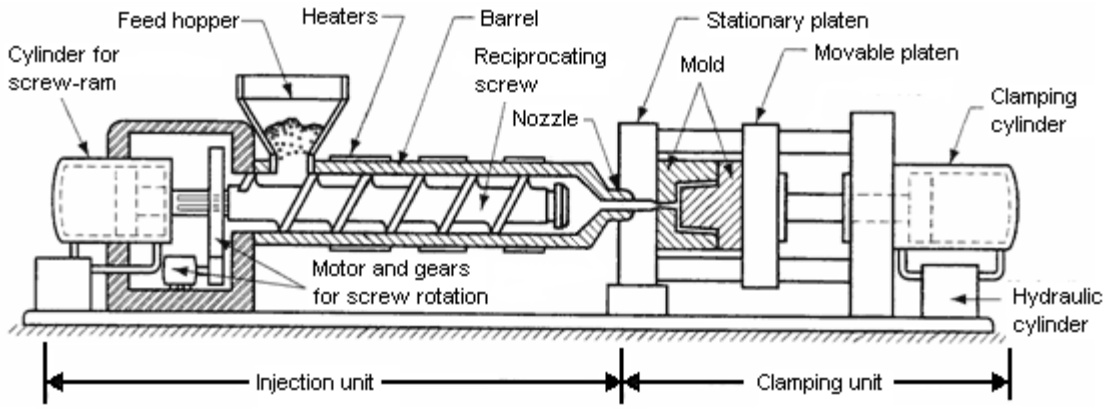


Fig. 4: Injection molding machine

The injection mold is a tool, typically made of steel, aluminum or metal alloy, which can be opened and closed along the mold's parting line. The injection mold consists of several components, but can be divided in two main areas – the mold core and the mold cavity. After the molten feedstock is injected into the mold and the cavity is filled, material is cooled down and after the mold opens, the part is ejected. This molded part is called a „green part“ in the PIM technology. The mold design is the main topic of the practical part of this thesis, therefore the description of injection mold components is presented there in more detail [16, 17, 18].

The mold design is one of the most important steps to realize a successful PIM process. The mold cavity has to be enlarged by approximately 20 % - value strongly depends on the binder and a packing ratio of the feedstock. The reason is that the molded part will shrink later during the sintering process. When a feedstock is being totally homogenized, shrinkage during sintering is uniform and therefore the enlargement of the cavity is

constant in every direction. The part design restrictions are the same as for injection molding of polymers. Simply said, any part made of thermoplastic that can be produced with injection molding, can also be produced by PIM. The list of major guidelines for designing a PIM part includes [4, 16, 19]:

- The wall thickness should be minimized and kept uniform throughout the part. It is worth noting that in the PIM process, minimizing wall thickness not only reduces material volume and cycle time, but also reduces debinding and sintering times;
- An additional ribs for structural support are implemented, rather than wall thickness increased;
- The uniform wall thickness and gradual section thickness changes will ensure uniform cooling and reduce defects;
- The round corners should be implemented to reduce stress concentrations and fracture and inner radius should be at least the thickness of the walls (at least 0.05 mm). For micro PIM parts there is a limitation for a corner radius due to a spherical shape of a fine powder particle;
- PIM parts do not require any draft (except long parts). The polymer binder used in the powder material releases more easily from the mold than most injection molded polymers. Also, PIM parts are ejected before they fully cool and shrink around the mold features because the metal or ceramic powder in the mixture takes much more time to cool;
- During sintering, PIM parts must be properly supported or they may distort as they shrink. By designing parts with flat surfaces on the same plane, standard flat support trays can be used. Otherwise, more expensive custom supports may be required during sintering;
- No undercuts on internal bores;
- Minimize the number of external and internal undercuts, because external/internal undercuts require side-cores/internal core lifters which add to the tooling cost;
- The features with external threads should be oriented perpendicular to the parting direction. Threaded features, which are parallel to the parting direction, will require an unscrewing device, strongly enhancing a tooling cost.

1.2.5 Debinding

In this step the binder is removed from the molded part. This can be done by several methods. Applicability of each method is defined by the chosen binder system. The strength of the debinded part is greatly decreased after any of the debinding processes. Therefore, this so called „brown part“ has to be handled very carefully.

Techniques to extract binder systems are:

- Solvent debinding
- Thermal debinding
- Combined debinding

Solvent debinding

During solvent debinding a solvent is dissolving all the binder components except the polymer backbone used in the binder system. It is necessary that the dissolved agents and the polymer are insoluble in each other. Immersion is practiced by placing the parts in a solvent (heated to the appropriate temperature – usually between 40 °C an 80 °C) for several hours. The alternative is debinding by a solvent vapour. The solvent vapour invokes condensation on the part, and subsequently extracts the binder components that drop away with the solvent. This method can be also combined with subsequent immersion. The solvent choice depends on a binder system, but widely used solvents for immersion are ethylene dichloride, heptane or trichloroethane. For debinding by solvent vapour carbon tetrachloride, methyl ethyl keton, hexane, heptane, or another high vapour pressure species are being used. Every debinding reactor includes a distillation attachment, which removes the binder from the solvent before recycling the solvent [5, 9].

Debinding of water-soluble polymeric binders

Solvent debinding method also involves a debinding of water-soluble polymeric binders. These binders contain polyethylenglycols, polyethylene oxide, polyvinyl alcohol or polyacrylamide. All named polymers, which contain oxygen or nitrogen atoms in their monomers, are hydrophilic. For this reason the binder system can be easily debinded by

using water as a solvent. This relieves a debinding process of using flammable, toxic or even carcinogenic solvents. Debinding of water-soluble binder is also much faster than the thermal extraction of a binder system [9].

Catalytic debinding

Catalytic debinding is using the characteristic of polyacetal polymer included in the binder system. Polyacetal polymer can be easily depolymerised significantly below its melting point by an acid. This allows the binder, consisting mainly of modified polyoxymethylene, to be dissolved out by an acid. Nitric acid is the most suitable one for the catalytic debinding, because the anhydrous acid vapour does not react with most of the commonly used powders in PIM. The shape of the part during debinding remains the same as the debinding temperature is below a melting temperature of the binder [5, 9].

Supercritical debinding

Supercritical debinding is very time effective way compared to the thermal debinding method due to a usage of lower temperatures. A supercritical fluid is a state between liquid and gas. Such a physical state occurs, when the chemical substance is over a critical temperature and pressure. Then the supercritical agent such as CO_2, Freon, propane and other, extracts all the components of the binder system but it does not dissolve out the polymer backbone [9].

Thermal debinding

During thermal debinding the binder is removed through an applied heat. Debinding is carried out in a debinding reactor, often with a forced atmosphere circulation, at gently elevated temperatures. Thermal debinding can be performed, depending on selected powder, under air, hydrogen, nitrogen or argon atmospheres at various temperatures. Slight and uniform heating is required in order to avoid disruption of the part. Thermal debinding normally takes many hours or even couple of days. The time required is defined mainly by the wall thickness of the molded part. The binder can be extracted either by degradation, evaporation or liquid extraction using a wicking material (porous substrate) in contact with the part [5, 11].

Debinding of a gellation binder

Debinding of a gellation (water-based) binder is usually incorporated in the sintering process. This water-based agar feedstock molded into the „green part“ is totally dried in the sintering furnace and the small amount of binder that remains in the part can be quickly evaporated due to a porosity of the matrix created by the eliminated water. Thus there is no real limitation in the thickness of the part [9].

Combined debinding

As an alternative, combined debinding can be used. In that case, binder is dissolved out with the low molecular weight species, such as acetone, hexane, methane or water and evaporated by heating the part in the next step.

Binder system	Debinding technique	Conditions	Duration
Wax - polypropylene	Oxidation	Slow heat to 150 °C, hold, heat to 600 °C in air	60 hours
Wax - polyethylene	Vacuum extraction	Slow heat while passing low pressure gas over compacts, heat to sintering temperature	36 hours
Wax - polyethylene	Wicking	Slow heat to 250 °C, hold, heat to 750 °C in hydrogen	4 hours
Oil - polymer	Solvent immersion	Hold in ethylene dichloride at 50 °C	6 hours
Wax - polymer	Supercritical	Heat in freon vapour at 10 °C/min to 600 °C under 10 MPa pressure	6 hours
Polyacetal - polyethylene	Catalytic debinding	Heat in nitric acid vapour at 150 °C	6 hours
Water - gel	Air drying	Hold at 60 °C	10 hours

Tab. 2: Comparison of debinding techniques and times for samples with a section thickness of 10 mm, an average particle size of 5 µm, and solid loading of 60 vol. % [4]

1.2.6 Sintering

Sintering is a technology generally used in the powder metallurgical industry. It uses a high temperature furnace, where the separate metal/ceramic powder particles weld together to create the final product. It is carried out at a temperature below the melting point of the powder (usually between 0.6 and 0.9 of the melting temperature). Metal powders are sintered in protective atmospheres or in the vacuum. The atmospheres used in the sintering furnace are reducing in general. That avoids oxidation of the metal and reduces the oxides on the powder particles surface. The atmosphere composition and the temperature during the sintering depend on the particular material. For CIM, any protective atmosphere is not needed due to a non-oxidizing behavior of a ceramic powder. The duration of sintering action is increasing with the size of each component. As the debinded part is very porous, large shrinkage occurs during sintering. Sintering shrinkage is usually isotropic and uniform. The final density of the part is almost theoretical, generally greater than 97 % and the mechanical properties are not significantly, if at all, below those of wrought material [5, 7, 8].

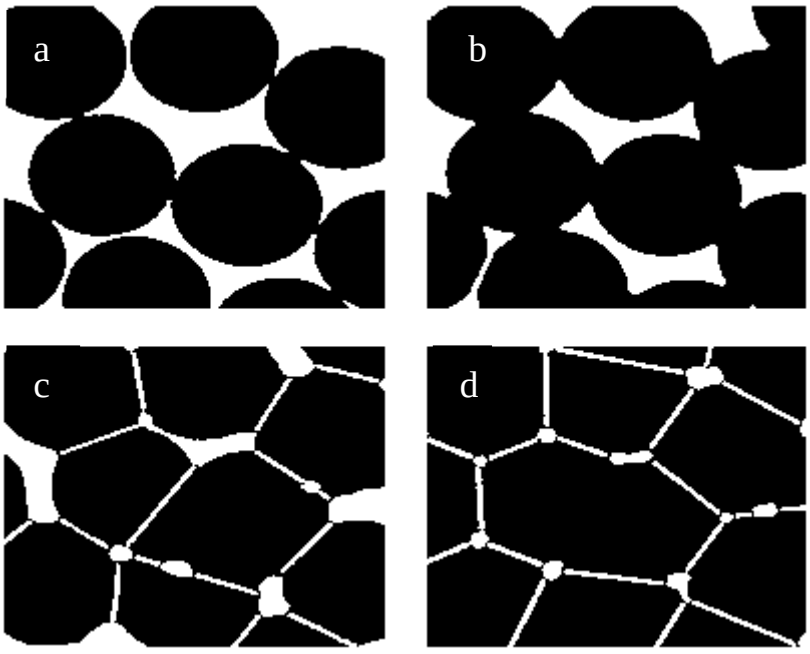


Fig. 5: Procedure of sintering: start of a bond growth (a); pore volume shrinks (b); grain boundaries form at the necking (c); pores become smaller (d)

1.2.7 Post-sintering operations

After sintering the part is fully densified, therefore no post treatments are necessary. Any standard process that is applicable to wrought material can be used to add features, improve material properties, improve tolerances and surface finish, or assemble other components. For example, a PIM part can be machined, heat treated, or welded [5].

2 PHASE SEPARATION

2.1 Quality issues of PIM process

Being still widely considered as a brand new technology, PIM constitutes an ongoing topic of both commercial and scientific interests. As an evolving technology, PIM has many issues relating to quality of a final product. As described in the previous part of this thesis, PIM process consists of four main stages - feedstock preparation, injection molding, debinding and sintering; therefore the possibility of an unpredictable defect is larger than by using a conventional technology.

Till lately most researchers focused mainly on debinding and sintering stage of a PIM process in connection with the quality of a final product. This interest was caused by unpredictability of these steps from the very beginning of the PIM production history. Shape retention and satisfactory binder extraction during debinding, and consequently unpredictability of shrinkage and warpage of the component during sintering, were the main quality challenges for the PIM producers. Even nowadays debinding and sintering are still considered as the most critical stages in reference to final tolerances, surface finish, visual defects, porosity, warpage, non uniform shrinkage, cracks and other quality characters. But development of new debinding methods, such as catalytic debinding, eliminated majority of the quality issues to a great extent [4, 11].

Even when high quality debinding and sintering of the parts are performed, above named defects are occasionally observed on the final parts. Consequently, origin of these quality defects must have been in a molded part before a debinding step. This revelation led to detailed quality analysis of the injection molding step in PIM process. This is because, if the quality defects on the part could be recognized after injection molding, feedstock would be recycled. Also no energy and time would be spent for debinding and sintering of such a part. There are no official figures for scrap rates in PIM, but in some instances up to 25 % of sintered parts are not reaching the quality requirements [20]. Therefore, advanced in-mold quality management would help to lower the production costs notably.

In-mold quality management is usually performed by temperature and pressure sensors placed inside the mold cavity and simulation software adjusted particularly for powder injection molding. These precautions are sufficient to secure a trouble-free injection

molding, where defects such as air traps, dead zones, or welding lines can be predicted. However, we are not able to predict a major quality issue during injection molding, which is a phase separation [17, 20].

2.2 Characteristics of phase separation

Phase separation is a term commonly used for an adhesion failure of powder and binder during injection molding step in PIM. As it was previously mentioned, a basic requirement for a high quality production is a homogenous mixing of powder and binder, adhesion and mutual interaction between them. Therefore, a separation of these two ingredients is causing quality issues such as visual defects, mechanical weak points, warpage and local hollowness. Phase separation is a defect that can be easily recognized in some feedstocks. The areas with phase separation are marked with „black lines“ on the surface of the molded part. These „black lines“ indicate elevated binder concentration in the area. Although we are able to identify the critical areas of the phase separation on a molded part, its influence over above named quality defects is not quantified. It has still not been fully understood by researchers when the „black lines“ are truly critical for the quality of final part. Also driving forces behind the phase separation of a feedstock that is totally homogenized before injection molding are not discovered yet. It is necessary to understand the mechanism of phase separation phenomenon in order to develop a simulation software module, which would provide a reliable prediction of the phase separation during injection molding and would also be able to quantify if such a separation would have a detrimental influence on the quality of a sintered part or not. Two recent studies [1, 2], one focused on a virtual simulation of the phase separation and another one on an experimental evaluation of phase separation in PIM components are mentioned in the following paragraphs [1, 20].

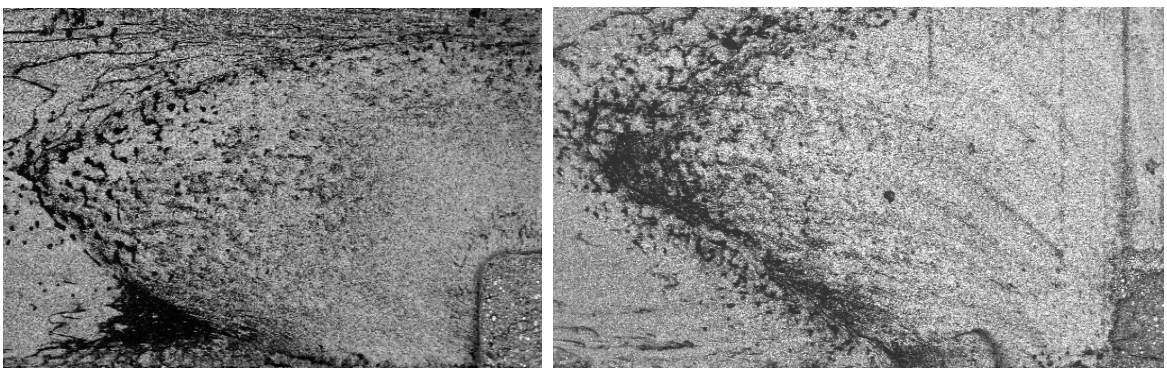


Fig. 6: Various phase separation on the surface of molded part

2.2.1 Theoretical study of phase separation

According to a theoretical study of phase separation, presented by Thornagel (SIGMA Engineering GmbH) [1], local shear rate gradients are the forces initiating phase separation. Local shear rate gradients should force powder particles to leave areas of high gradients. The effect behind this approach is visualized below.

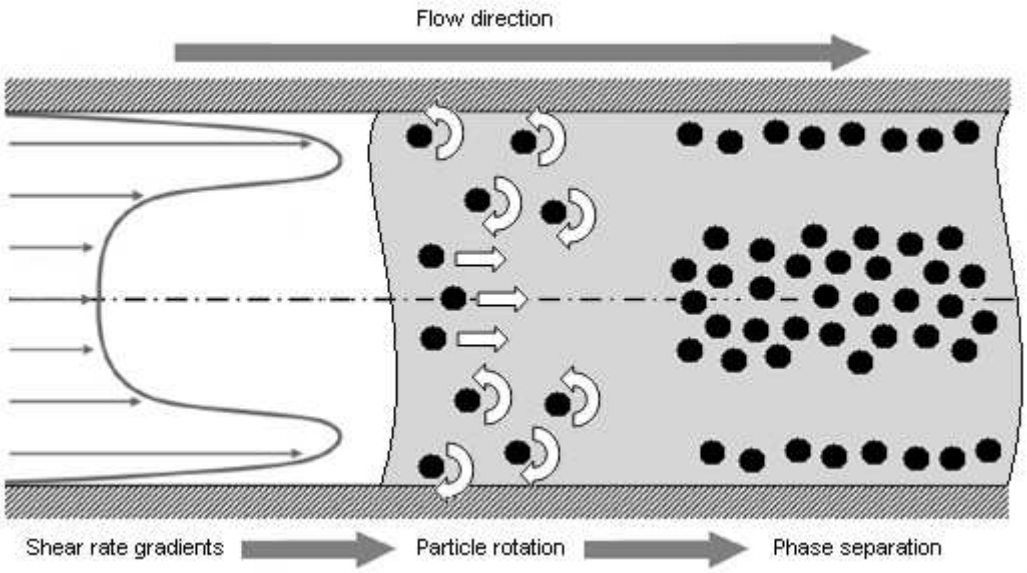


Fig. 7: Local shear stress gradients cause powder - binder separation

Fig. 7 shows a typical shear rate pattern across the channel during the feedstock flow. Due to adhesion of the feedstock at the wall of the channel, a significant shear rate peak occurs close to the wall and a plateau at a much lower shear rate level is observed in the middle of the flow domain. Focusing on the powder particles, reaction forces are depending on their current position in the shear rate field and on their size. Particles flowing in the middle of the channel within the plateau of the lower shear rate experience uniform reaction forces and continue their way without changing the direction. Other particles not flowing within the lower shear plateau experience a non uniform shear rate influence resulting in a rotation of these particles. The rotation speed grows along with the shear stress gradients. Such rotating particles try to leave areas of high shear gradients. Therefore the area of the highest shear rate is characterized by high binder content in the channel while the plateau of the lower shear rate accommodates a powder rich material [1].

According to this study, local shear rate gradients cause the separation of powder and binder during injection molding. Unfortunately due to the feedstock flow in the mold cavity, the location of the final effect - caused by these driving forces - changes. The feedstock flow moves the separation pattern and changes the pattern continuously. Simplified example of such a behavior is shown in Fig. 8 [1].

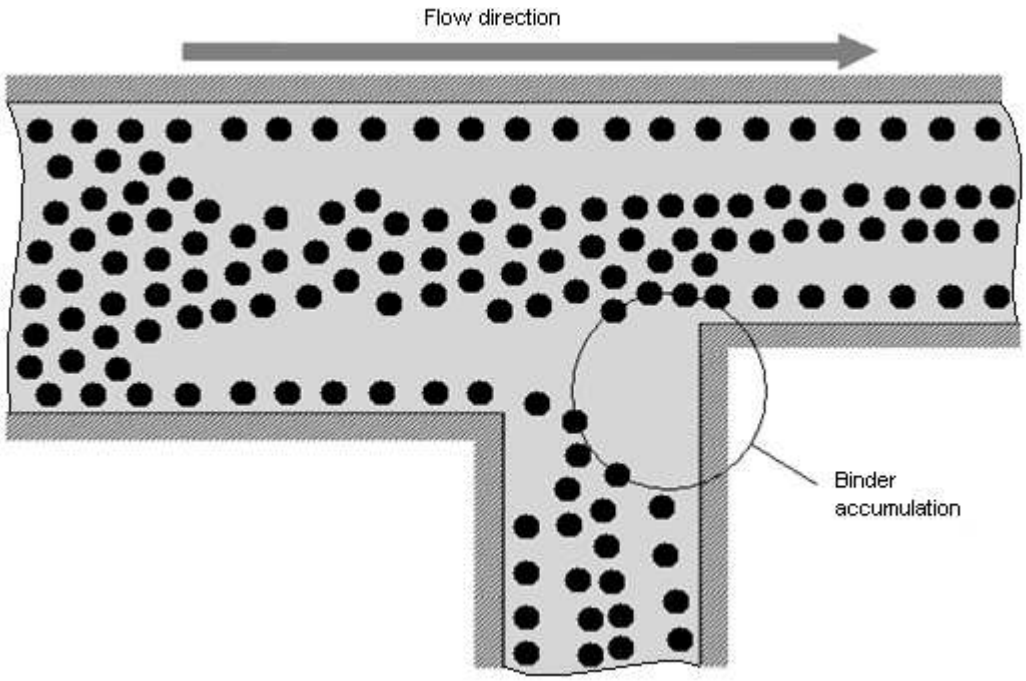


Fig. 8: Feedstock flow changes the separation pattern

In this case, the history of the feedstock, which is travelling through injection machine, nozzle, runner system and cavity, has to be implemented in the software simulation to predict the phase separation area on the molded part. All available software simulations for injection molding using PIM feedstock are considering feedstock as a bulk. Therefore it is necessary to model a flow of each component that is contained in the feedstock. Because PIM feedstock consists of binder, which is a mixture of several ingredients, and at least one type of fine powder, a multi-phase simulation is needed. However, a multi-phase simulation is extremely time and hardware consuming, and thus not practicable for use in a production at the moment. For this reason the above formulated theory has not yet been practically verified [1].

Modeling the feedstock as a bulk and extending currently used 3D Navier Stokes equations in a way that powder and binder concentrations and their variations can be predicted depending on the shear rate history is currently developed by SIGMASOFT. The particle flow model is based on a diffusive flux model. Such a simplified simulation model would be practicable for mold producers, if it is reliable [1].

2.2.2 Empirical study of powder binder separation of feedstocks

In this approach Jenni et al. [2] studied the influence of injection parameters for different materials on the process, resulting separation and its software simulation. Software simulation uses calculations with the balance model for separation compared to the diffusive flux model used in the previous study. The balance model is developed for the flow of rigid, spherical particles in a Newtonian fluid. Simulation output was compared to the real parts to check the quality of the results [2].

Because of the large number of samples, a simple and fast procedure to quantify the local powder content had to be found. Differential scanning calorimeter (DSC) was considered as the best option after several different approaches. To compare different materials and processing parameters used for injection molding, three testing molds shown in Fig. 9 were produced [2].

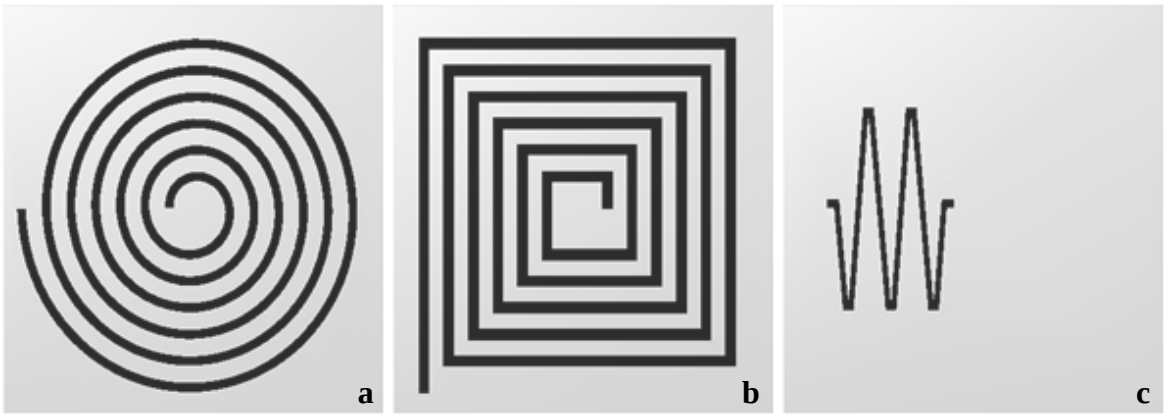


Fig. 9: Three different cavity geometries of the testing mold.

Spiral (a); Square spiral (b); Zigzag (c)

These testing moulds were particularly designed to force and exaggerate phase separation during filling. Design of the cavities is similar to those used for investigating the moldability of plastics. Moldability is practically characterized as the length of the mold cavity filled under defined conditions. Because the testing mold is cold, a frozen layer continuously forms along the cavity walls and the mold is filled by the flow through the partially frozen channel. The center of a channel progressively closes along with the length of the mold cavity and eventually halts the flow. This process is commonly known as a fountain flow [2].

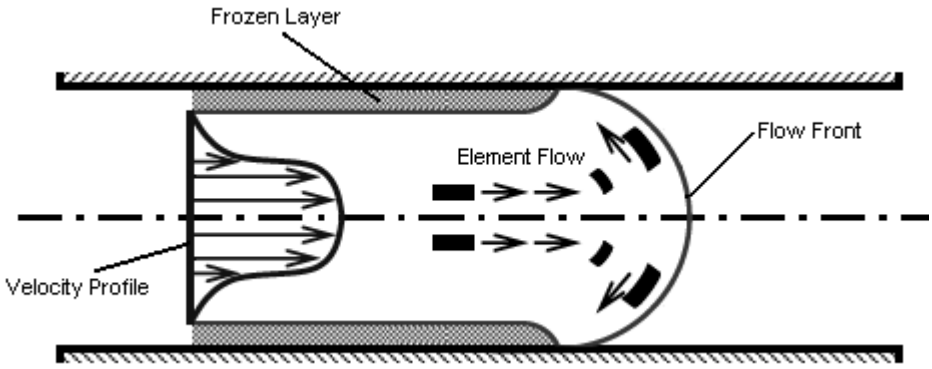


Fig. 10: A fountain flow phenomenon

The experimental runs were performed with the dependent variable for the moldability being the flow length and the input variable being nozzle temperature, mould temperature and injection speed. After finding the best conditions for the process, the variables were shifted up and down. Resulting feedstock behavior was as expected. Better flowability of the feedstock comes with increased temperature and injection speed. Also the flow length depends significantly on a solids loading. The mean difference for the flow length of the used tungsten with 60 vol. % and 50 vol. % solids loading in the square spiral geometry was about 108 mm resp. 68 % of the filled length. Also the powder content was measured for each sample at a defined location. After analyzing these results and comparing them with other materials with different powder loadings, the development of a fountain flow becomes more significant with higher temperature and injection speed. The binder flows more easily due to its low viscosity, while the powder particles follow behind. Therefore, like it was observed in the square spiral testing mold, general powder content will decline to some extent with increasing number of corners in cavity geometry. It was also

confirmed that reduction of the nozzle and mold temperature decreases phase separation of the feedstock. The same applies to injection speed [2].

To get a qualitative comparison between the results from the software simulation using the balance model and experimental samples, two and three dimensional pictures of the powder distribution were produced. Molded part had to be divided into pieces to measure the powder content. The most suitable locations to observe phase separation are in the corners of the square spiral geometry. For this reason a corner was cut horizontally into 3 pieces to take out 1 mm high plate out from the middle. Such a thin plate was cut into 64 pieces with 1 mm side length at later stage. After cutting operations, a picture of the powder distribution over the corner can be taken. Radiography, computer tomography and DSC measurements were used. Unfortunately, tungsten was very complicated to measure because of its high density and absorption. And therefore the resolution was too small and the results were not satisfying [2].

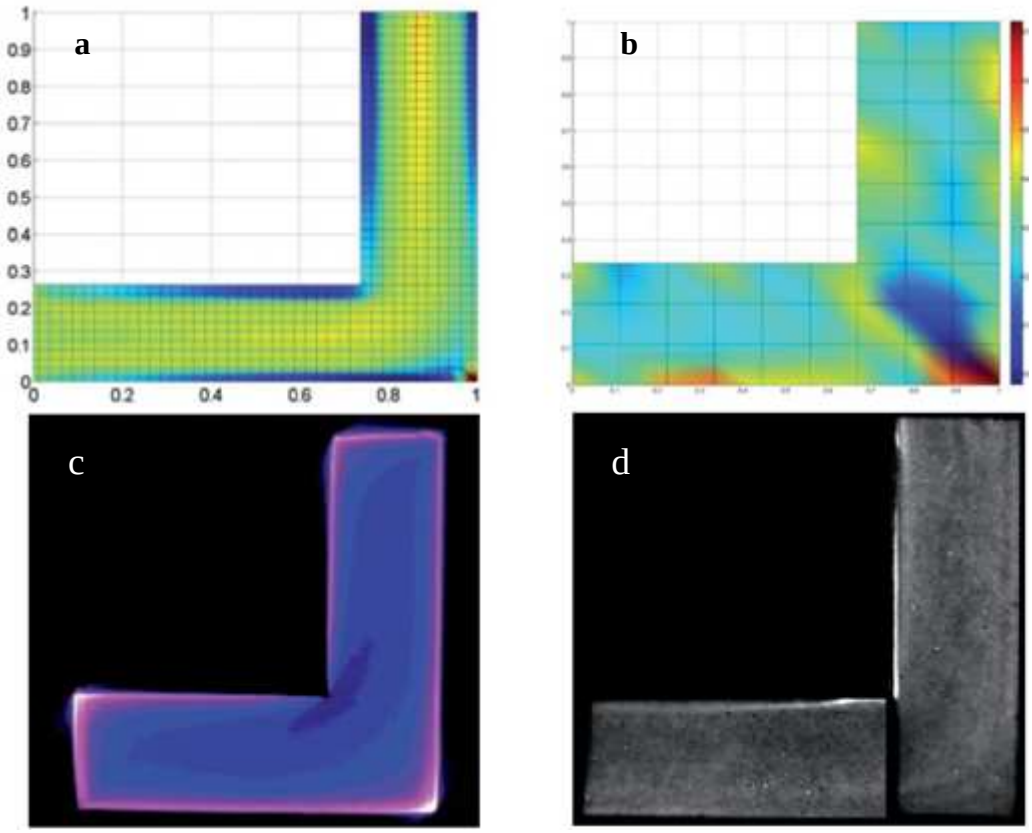


Fig. 11: Powder distribution along a corner of the sample (tungsten feedstock with 60 vol. % solids loading). Balance model (a); measured by DSC (b); measured by computer tomography (c); measured by radiography (d) [2]

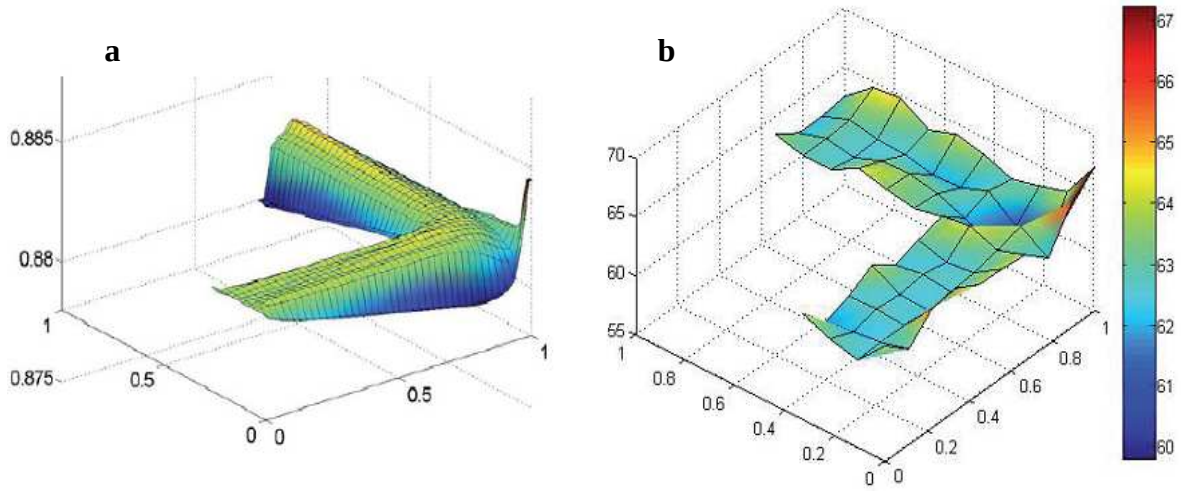


Fig. 12: Powder distribution along a corner of the sample (tungsten feedstock with 60 vol. % solids loading). Balance model (a) - powder content is shown as a fraction of maximum flowable solid content (68 vol. %); measured by DSC (b) [2]

The balance model gives a clear picture of migration of particles for simple geometries, but the irregularities of the feedstock flow such as slip effects at the wall and the fountain flow are not intercepted by this model. Therefore, further investigations of feedstock flow behavior and its wall effects as slip, rolling of particles and flowing layers with increased binder content are necessary. These additional physical phenomena during the flow have to be implemented into software simulation to reflect the reality in more detail. Also further research will be needed to quantitatively relate the phase separation effect to shrinkage and distortion effects occurring during debinding and sintering [2].

Currently none of these studies [1, 2] can reliably predict factors or characteristics causing phase separation. It means that further research is needed to explain the effect of phase separation and to predict the quality of final product by implementing gained knowledge into existing simulation software. In order to understand the mechanism of phase separation and its influence on the shape retention during debinding and sintering, a testing mold including critical geometry elements has to be developed. Design of such a mold is the aim of this thesis.

II. EXPERIMENTAL PART

3 AIMS OF THE MASTER THESIS

For this master thesis following aims were defined:

- Literature study of phase separation
- Evaluation of the most critical areas on various molded samples where phase separation occurred
- Design of a specimen containing all the geometric elements, which appear to be crucial for phase separation
- Construction of an injection mold to produce designed specimen
- Processing of a drawing documentation for a mold producer
- Injection molding and visual evaluation of commercially available feedstocks

Phase separation and two existing theories [1, 2] focusing on this phenomenon were described in the previous theoretical part.

In the experimental part of this master thesis, design proposals of the testing specimen and the mold are presented. Images of injected feedstocks are also included. The complete drawing documentation of the mold is presented as an appendix.

4 TESTING SPECIMEN DESIGN

As previously mentioned, testing specimen should contain critical geometry elements. After a visual study of various molded parts, the most critical areas for phase separation were defined as:

- Inner and outer corners
- Radical thickness changes including gates
- Weld lines
- Thin films or flashes

Apart from these geometrical requirements, other PIM process demands on the specimen had to be considered:

- Moldability of a designed geometry
- Simple ejecting apparatus due to a low cost requirement on the mold
- Predictable sintering behavior – minimum warpage, uniform sintering
- Specimen with a large flat surface that allows the use of standard debinding and sintering trays to eliminate the need for custom made supports

Satisfactory testing specimen design is shown on the pictures below:

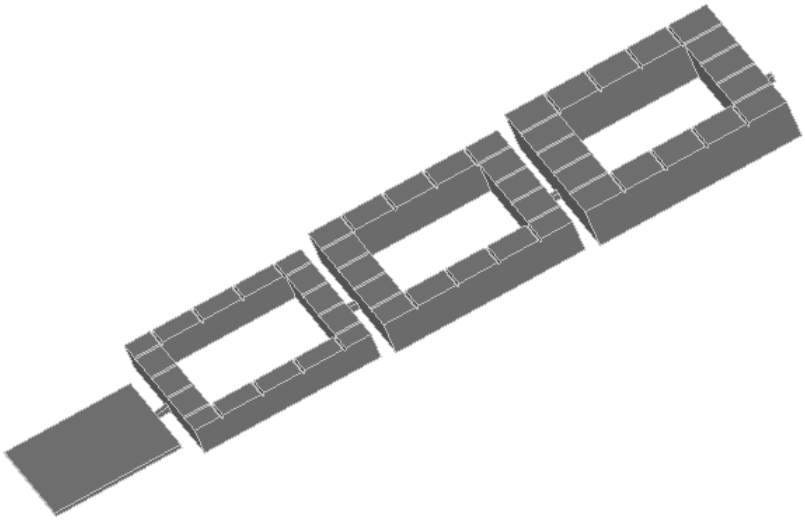


Fig. 13: Testing specimen design – top side

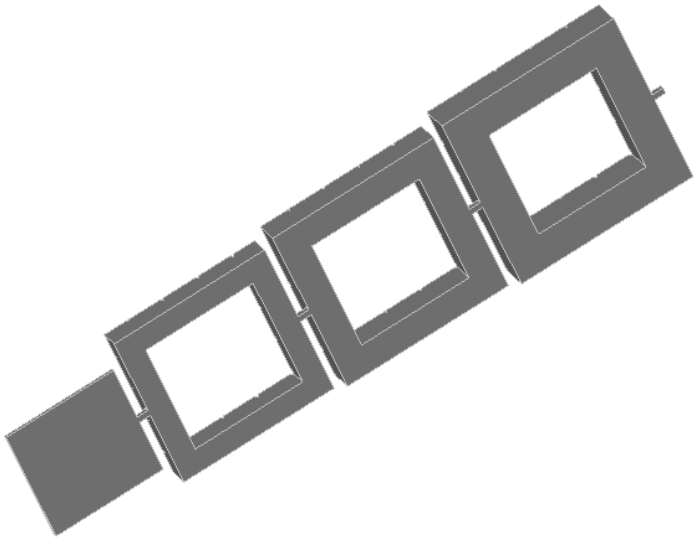


Fig. 14: Testing specimen design – bottom side

Testing specimen consist of four elements separated from each other by a thin link, which could be described as a gate. First three elements (in the direction of the flow) contain both inner and outer corners, gates and weldlines. All three elements have a square shape with the same inner side length of 10 mm. These elements were designed to compare the alterations in phase separation by gradually decreased section size. The side length of a square section is 3 mm for the first, 2.5 mm for the second and 2 mm for the third element. Each of them are equipped with 16 slots (4 on each side of the square) to make future measurement easier. The inlet and connecting gates are 1 mm long and have square section of 0.5 mm side length. The final element represents a 0.3 mm high flash and also works as an evacuation zone for the feedstock flowing out of the third element. This thin film has also square geometry with a side length of 10 mm. All four elements have a common plane on the bottom side allowing this testing specimen to be debinded and sintered on commonly used plates or trays. The square geometry of each element also provides uniform sintering and minimizes the risk of warpage. Designed geometry can be injection molded and subsequently ejected easily. Therefore, this specimen design covers all originally defined requirements and critical geometries.

5 MOLD DESIGN

Mold design and drawing documentation was performed in a standalone licensed Education Suite version of the Autodesk® Inventor® 2010 software. This CAD (Computer Aided Design) software application allows 3D mechanical design of the injection mold components, their assembly and consequently 2D drawings can be generated and dimensioned.

It was decided that the mold components will be provided by HASCO Company to lower the mold production cost. HASCO offers standard mold units and their 3D models can be found in the HASCO Digital Catalogue. These 3D components can be imported to various CAD softwares, modified and assembled into a mold. This procedure was used for the testing mold construction and is described on the following pages.

5.1 Parting line

Before designing the left and right cavity plates, the parting line of the mold had to be set. In our case the parting line could be straight and placed in the same plane as the bottom side of the testing specimen as shown in Fig. 15.

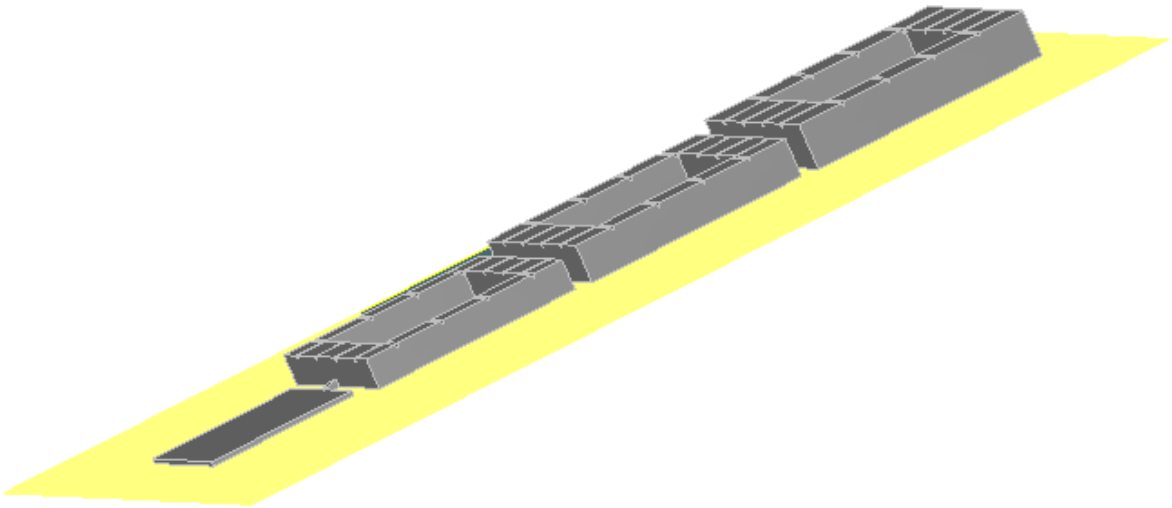


Fig. 15: Parting line of the mold

5.2 Ejecting apparatus

Ejecting system was designed after the parting line had been set. Using ejection pins were the best solution for this testing specimen. Because of a brittle PIM feedstock, pins with 1.8 mm diameter were placed in each corner of the squares to ensure that the square elements would not break during an ejection. One more ejection pin with 7.5 mm diameter was used to eject the sprue puller, which pulls the sprue out of the sprue bushing. Another two 10 mm diameter pins were used as safety pins for a reverse movement of the ejector plates and to prevent ejectors from any contact with the cavity surface. The total of 19 ejection pins was embedded in the ejector assembly of an ejector plate and an ejector retaining plate as shown in Fig 16.

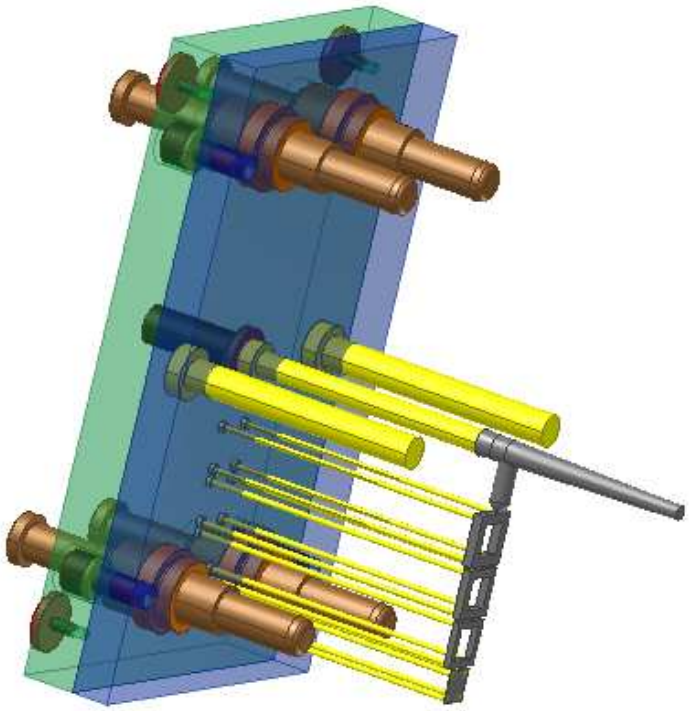


Fig. 16: Ejector assembly

The ejector assembly is also equipped with four ball guide bushes and pillars to guarantee fluent ejecting of the molded specimen. The ejecting movement of the assembly is actuated by a bar mounted on the ejector plate. This ejector bar provides connection between the clamping unit of the injection machine and the ejector assembly. In our case hexagon socket head cap screw was used to make the ejector assembly fit with any commonly used ejector bars.

5.3 Runner system

Runner system was designed to meet specification of the sprue bushing by HASCO. Standardized sprue bushing was shortened to the shortest extent possible, making the runner system reasonably short. Modified sprue bushing (Fig. 17) and runner system used (Fig. 18) are shown below.

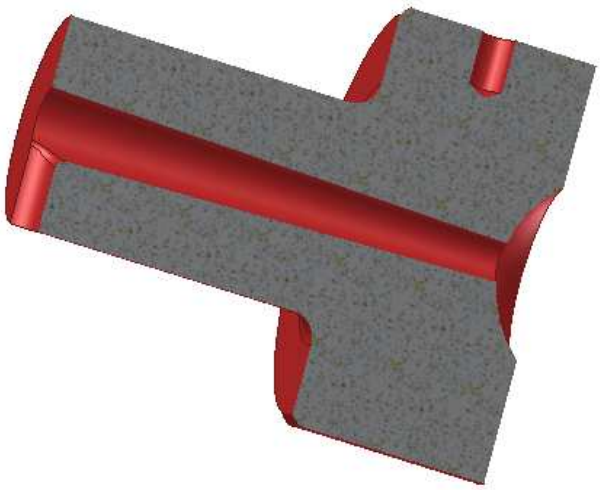


Fig. 17: Modified sprue bushing

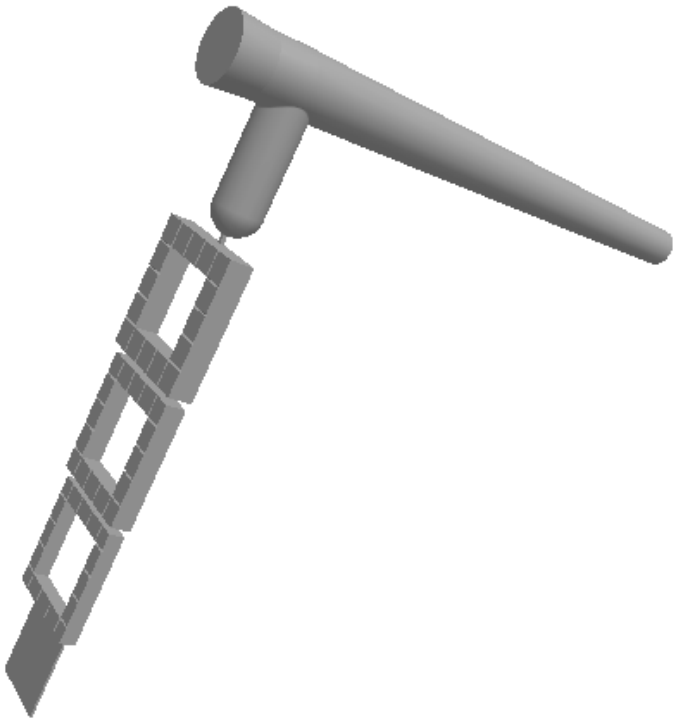


Fig. 18: Runner system

5.4 Cavity plates

Both left and right cavity plates have the same size of 196 x 196 x 36 mm and are made of DIN 1.2767 - X 45 NiCrMo 4 (ISO 4957 standard tool steel). These steel cavity plates were hardened to 56 HRC by further heat treatment. The chemical composition of DIN 1.2767 steel enables it to be hardened with minimum distortion and also possesses good thermal properties for injection molding. It also displays excellent polish ability and toughness in the hardened condition making it suited to high pressure injection molding applications. Contact surfaces of the cavity plates and the cavity itself were polished to Ra 0.2 µm. Cavity of the mold was made as an exact negative of the specimen design. Unlike thermoplastic injection molding, PIM parts usually do not require any draft. PIM feedstock is highly loaded with fine powder that remains heated long after the molding cycle has been completed. Therefore, post molding shrinkage that occurs for thermoplastic parts while they are still in the mold, occurs for PIM parts after they have been removed from the mold. This allows the part to be ejected before it can cool and shrink around cores or other mold cavity features. Also the binder used in PIM feedstock acts as a lubricant to assist in the ejection of the part from the mold cavity.

Left cavity plate (movable) includes the cavity with holes for ejection pins and the sprue puller shown in Fig. 19 in the middle. Proper venting of the mold cavity is performed by the groove of 0.02 mm depth, grinded in the parting plane.

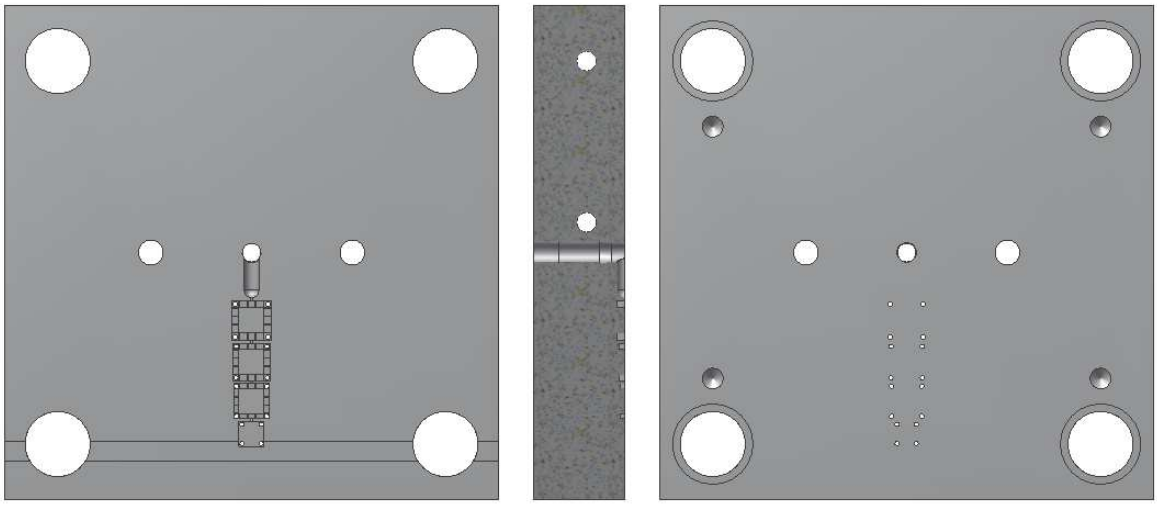


Fig. 19: Left cavity plate

Right cavity plate (stationary) was modified to accommodate two in-mold cavity sensors with front diameter of 2.5 mm for combined measuring of mold cavity pressure up to 2,000 bar and contact temperature on the flat front up to 450 °C (Fig. 20).

Fig. 20: KISTLER p-T-sensor type 6189A

Right cavity plate represents the bottom side of the specimen and hence there is no need for machining the cavity.

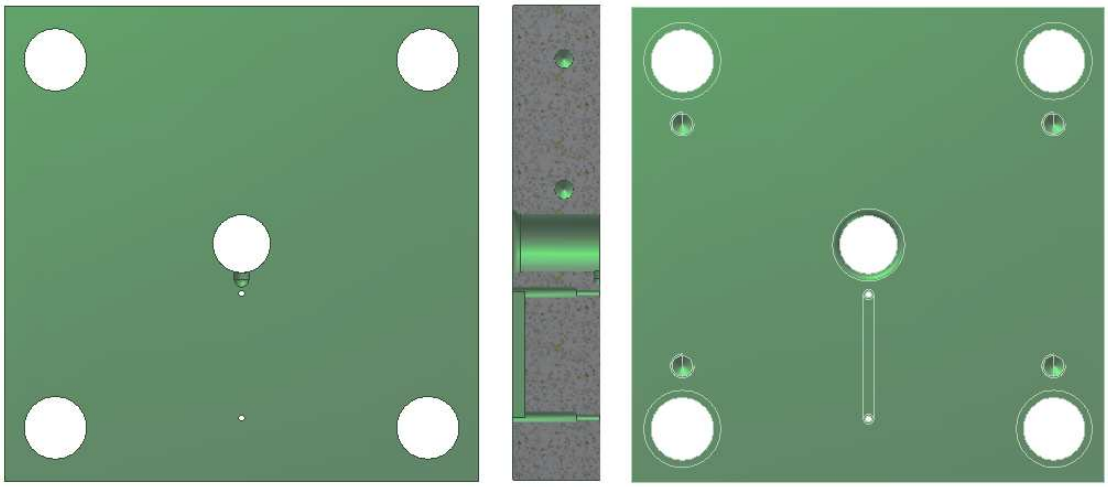


Fig. 21: Right cavity plate

5.5 Cooling system

Channels with a diameter of 8 mm were used for the design of the mold cooling system. To ensure uniform cooling, channels were implemented in both cavity plates. Four shut-off nipples provide the connection between the mold cooling system and the inlet/outlet hose for a cooling medium. The open-ended channels were sealed by shut-off screws. Extra cooling channels in the top sections of both cavity plates were designed for a potential need, if another cavity is added.

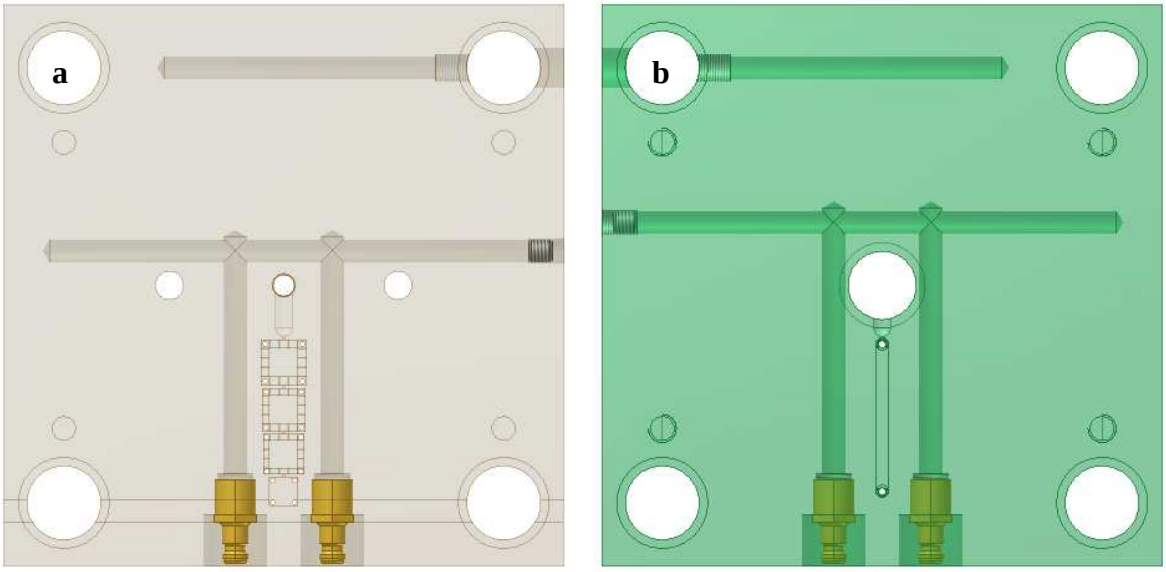


Fig. 22: Cooling system. Left cavity plate (a); Right cavity plate (b)

5.6 Clamping plates

Left and right clamping plates were also modified from standardized plates provided by HASCO. Both having the same size of 196 x 246 x 27 mm and made of DIN 1.1730 (ISO 4957 standard tool steel) that provides hard surface and tough core of the plates.

Left clamping plate (movable) was modified in the centre by a 22 mm diameter borehole for the ejector bar and four countersunk holes for guiding pillars of the ejector assembly.

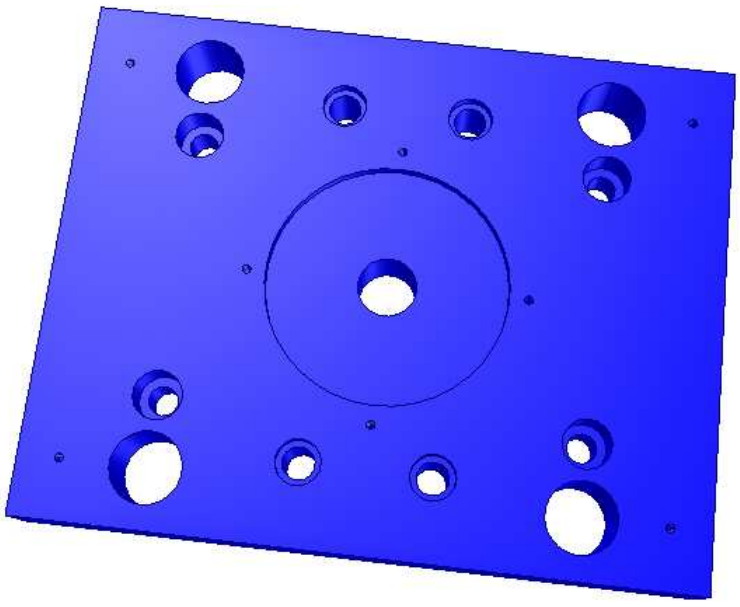


Fig. 23: Left clamping plate

Right clamping plate (stationary) was modified to embed in four connectors (two for each sensor) and cables for pressure and temperature signal. Machining a cavity into the clamping plate was preferred, as there was not enough space to use standard mounting plates. Also a groove for the dowel pin securing the right position of the sprue bushing was machined into the central prebored hole.

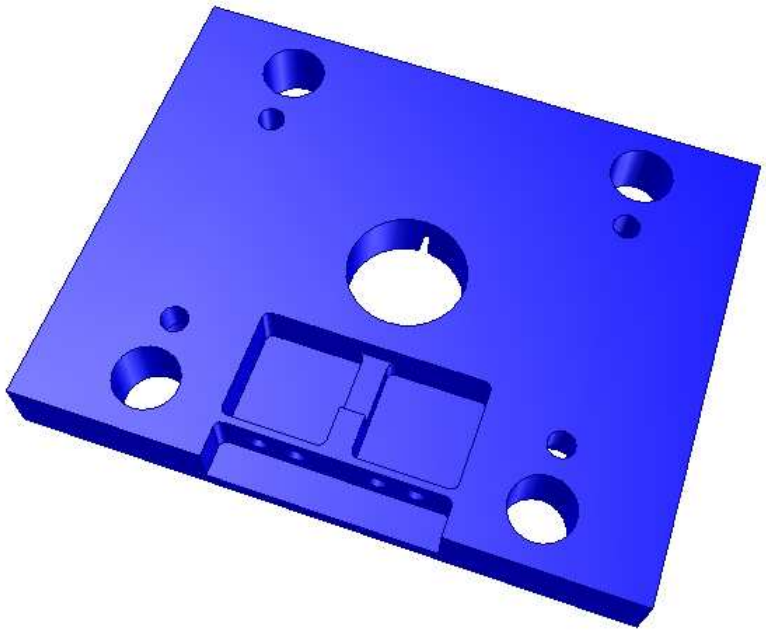


Fig. 24: Right clamping plate

In addition to above mentioned modifications, threaded holes for mounting thermal insulating sheets, locating rings and lifting device were machined into both plates.

5.7 Risers and guiding elements

Standardized risers with the length of 76 mm were used. These risers meet the ejector assembly requirements for its ejecting movement and provide sufficient size of the mold that allows it to fit in injection molding machines selected for testing. The risers also accommodate main guiding elements in each quarter of the mold. The mold is equipped with a centring sleeve and a guiding bush in the left (movable) half and a guiding pillar in the right (stationary) half. Plates of each half of the mold are fixed together by four hexagon socket head cap screws as shown in Fig. 25.

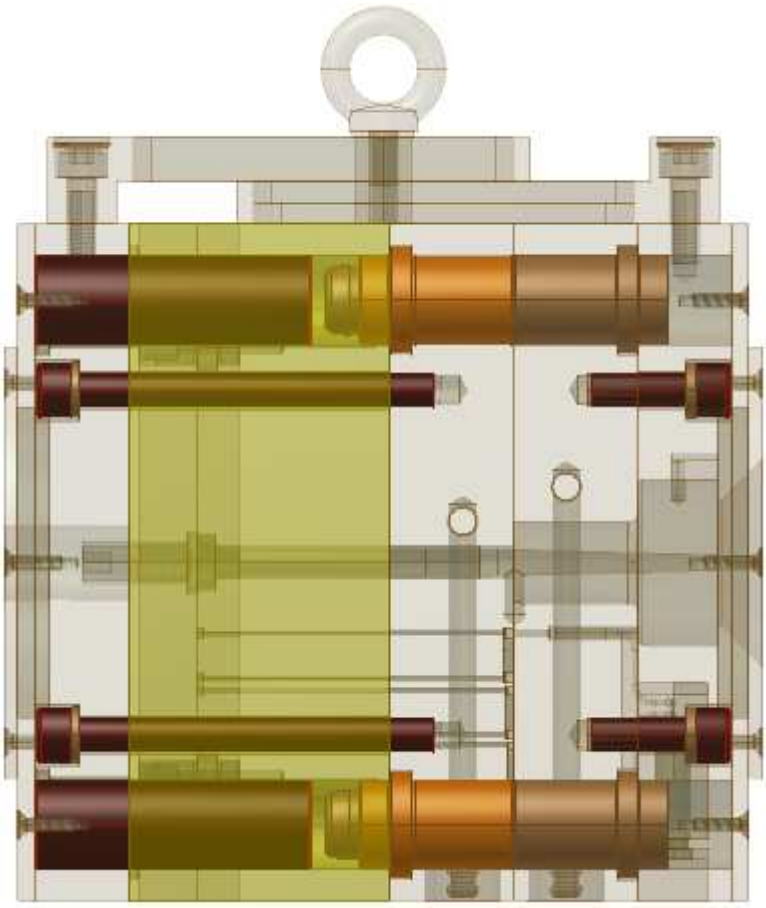


Fig. 25: Risers, guiding and mounting elements

5.8 Locating rings and thermal insulating sheets

Four countersunk holes were machined in both standardized locating rings to attach them to clamping plates.

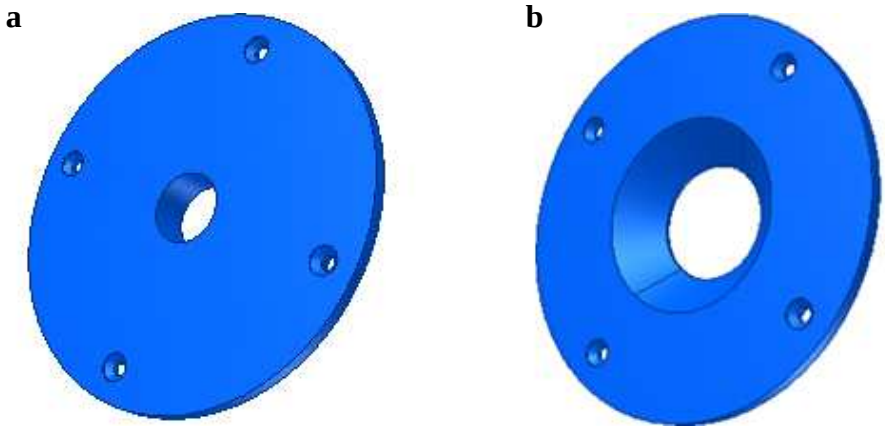


Fig. 26: Left locating ring (a); Right locating ring (b)

In addition to four countersunk holes, the left locating ring (movable) was modified by a 22 mm diameter borehole for the ejector bar. The central borehole in the right locating ring is prebored in the standard component.

Thermal insulating sheets made of glass fiber reinforced synthetic resin were placed on both sides of the mold. Therefore, countersunk holes in each corner were machined to mount insulating sheets on the clamping plates.

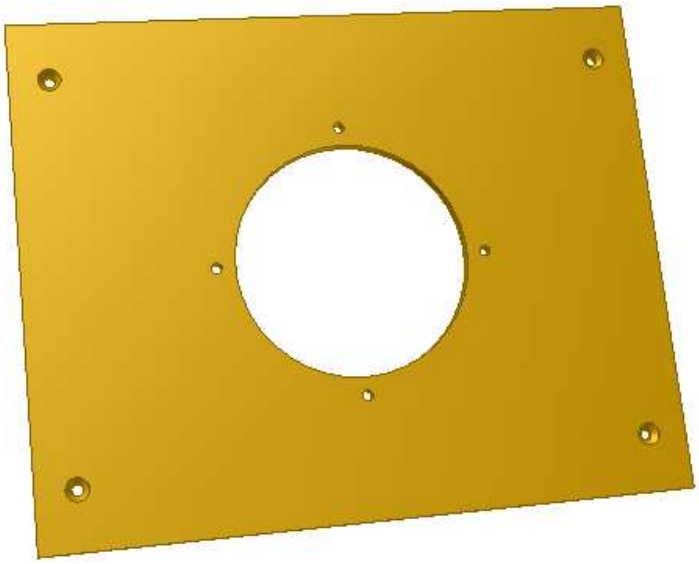


Fig. 27: Thermal insulating sheet

5.9 Lifting device

Standardized lifting device by HASCO was used to make manipulation with mold easier.

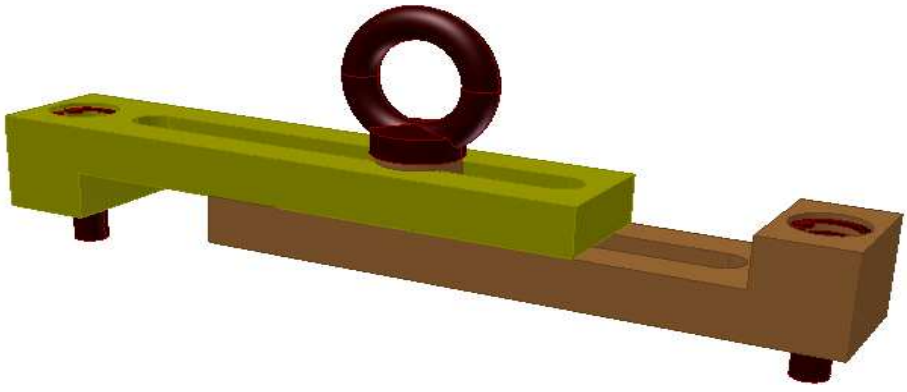


Fig. 28: Lifting device

5.10 Mold assembly

Final mold assembly of above described components is shown below.

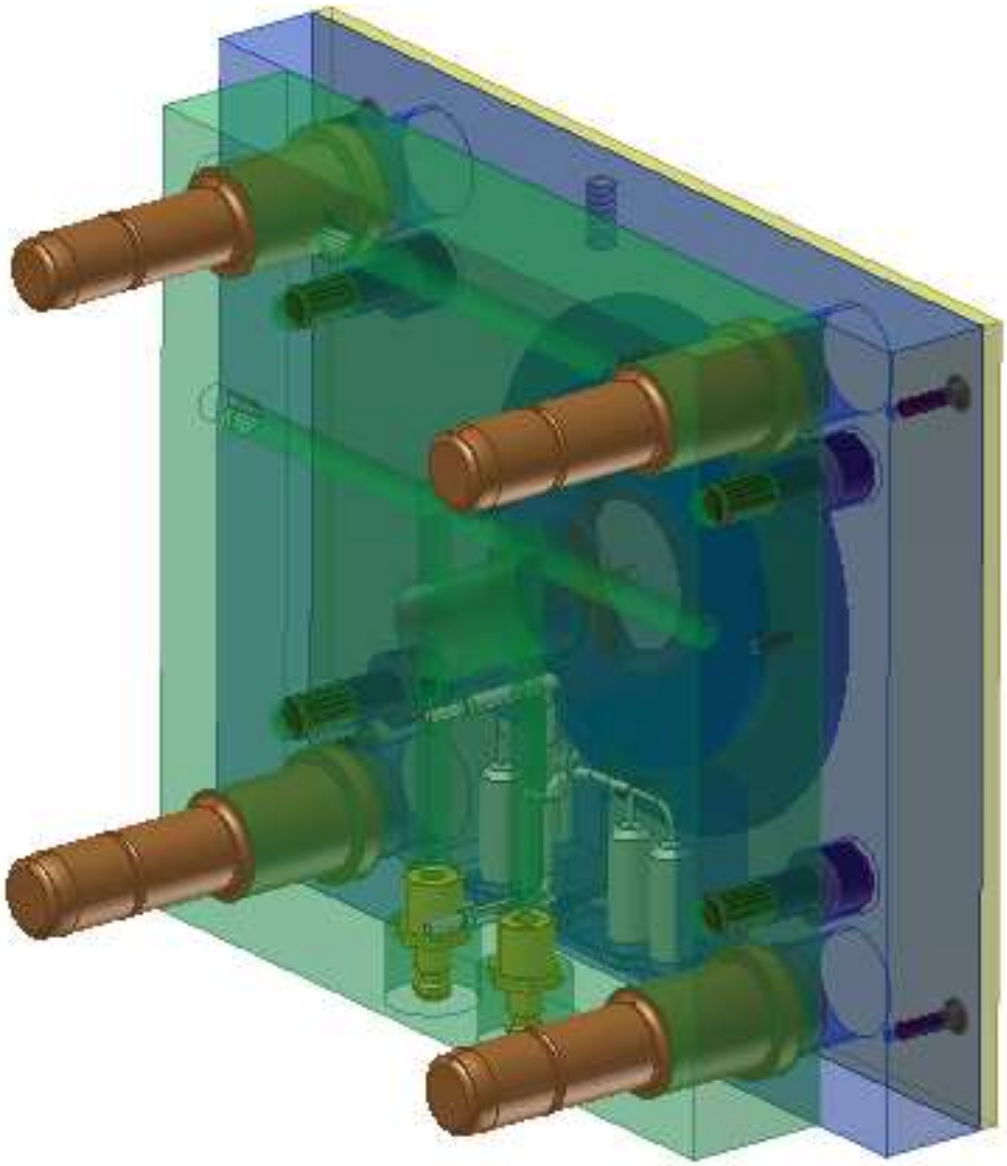


Fig. 29: Right (stationary) half of the mold

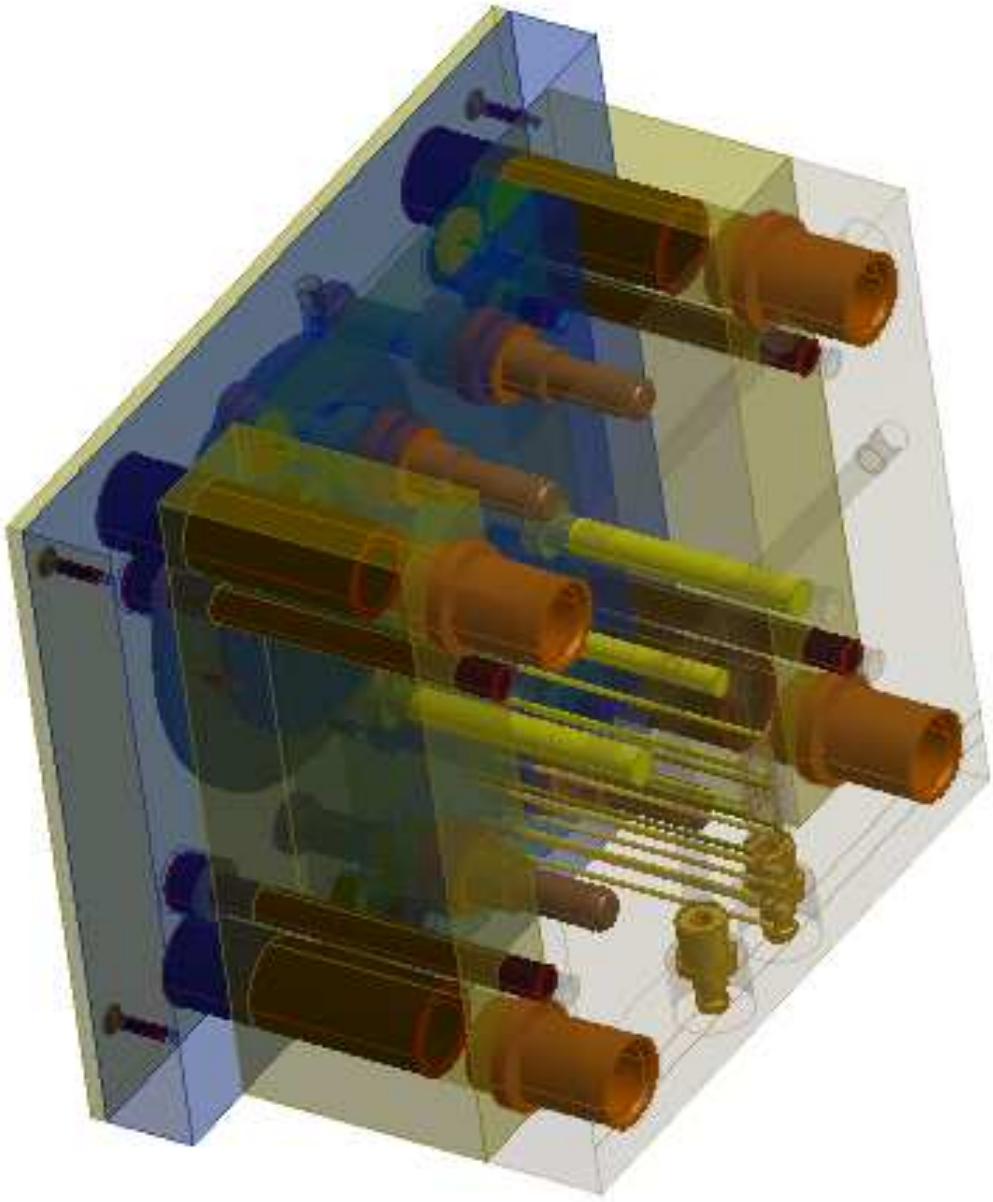


Fig. 30: Left (movable) half of the mold

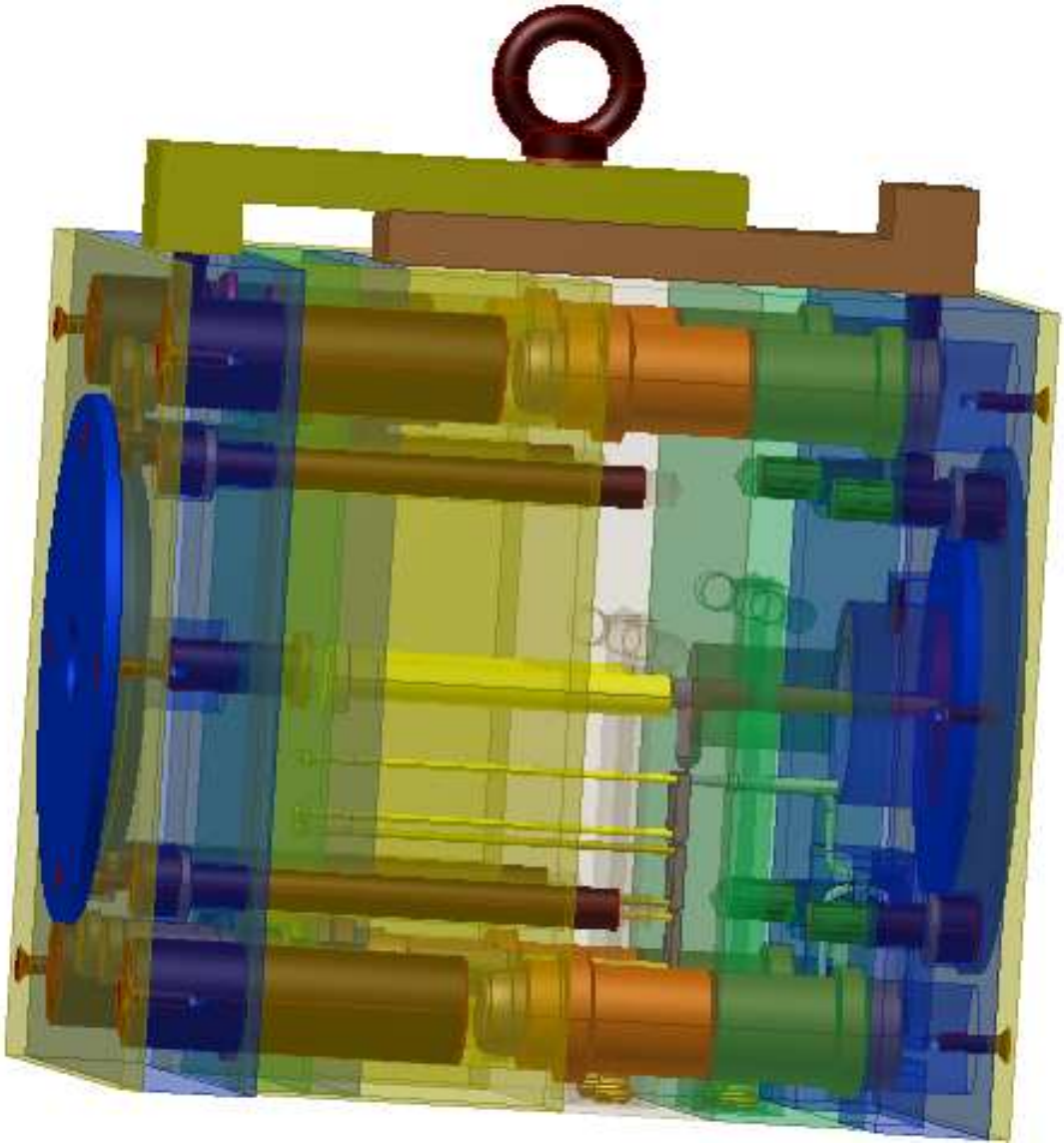
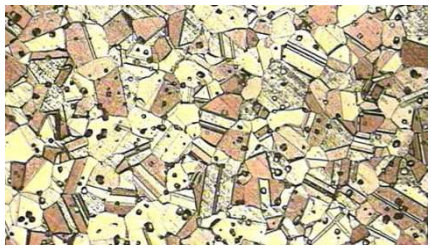


Fig. 31: Mold assembly

6 TESTING CONDITIONS AND RESULTS

6.1 Feedstock characteristics

Two different stainless steel feedstocks were used for injection molding tests. Both feedstocks, polyMIM® 316L and polyMIM® 17-4PH, were supplied by Polymer-Chemie GmbH. Properties of these MIM feedstocks are described in Tab. 3 and Tab. 4.

<table>
<tr><td colspan="6">polyMIM® 316L D 120E</td><td colspan="5" rowspan="3">  </td></tr>
<tr><td colspan="2" rowspan="2">Oversize factor - dimensions must be multiplied by this rate.</td><td>min.</td><td>average</td><td>max.</td></tr>
<tr><td>1.1627</td><td>1.1669</td><td>1.1711</td></tr>
<tr><td colspan="2" rowspan="2">MVR melt index - DIN EN ISO 1133 (210 °C / 5 kg)</td><td>min.</td><td>average</td><td>max.</td></tr>
<tr><td>15</td><td>40</td><td>65</td></tr>
<tr><td rowspan="2">Typical composition as sintered; weight %</td><td>Fe</td><td>C</td><td>Ni</td><td>Cr</td><td>Mo</td><td>Mn</td><td>Si</td><td>S</td><td>P</td></tr>
<tr><td>Balance</td><td>< 0.03</td><td>10.0-14.0</td><td>16.0-18.5</td><td>2.0-3.0</td><td>< 2.0</td><td>< 1.0</td><td>< 0.03</td><td>< 0.045</td></tr>
<tr><td colspan="3">Typical properties (as sintered)</td><td colspan="7">Injection molding process settings</td></tr>
<tr><td colspan="2" rowspan="2">Density</td><td rowspan="2">≥ 7.90 g/cm^3</td><td rowspan="2">Cylinder temperature</td><td>Zone1</td><td>Zone 2</td><td>Zone 3</td><td>Zone 4</td><td>Nozzle</td></tr>
<tr><td>170 °C</td><td>175 °C</td><td>180 °C</td><td>183 °C</td><td>185 °C</td></tr>
<tr><td colspan="2">Yield strength Rp02</td><td>≥ 140 MPa</td><td>Tool temperature</td><td colspan="2">40-60 °C</td><td>Injection pressure</td><td colspan="2">550-700 bar</td></tr>
<tr><td colspan="2">Tensile strength Rm</td><td>≥ 450 MPa</td><td>Injection speed</td><td colspan="2">3-20 cm^3/s</td><td>Post pressure</td><td colspan="2">400-650 bar</td></tr>
<tr><td colspan="2">Elongation A10</td><td>≥ 40 %</td><td rowspan="2">Peripheral screw speed</td><td colspan="2" rowspan="2">5-20 m/min</td><td rowspan="2">Back pressure</td><td colspan="2" rowspan="2">20-30 bar (hydraul.)</td></tr>
<tr><td colspan="2">Hardness</td><td>≥ 150 HV1</td></tr>
<tr><td colspan="3">Debinding process</td><td colspan="7">Sintering process</td></tr>
<tr><td colspan="2">Solvent</td><td>water</td><td>Sintering atmosphere</td><td colspan="6">100 % pure hydrogen</td></tr>
<tr><td colspan="2">Debinding temperature</td><td>40-60 °C</td><td rowspan="4">Sintering cycle</td><td colspan="6" rowspan="4">3 K/min → 600 °C,
2 h hold 600 °C,
5 K/min → 1320 °C,
2 h hold 1320 °C,
15 K/min → 80 °C,
cooling</td></tr>
<tr><td colspan="2">Debinding time</td><td>Depending on part thickness (e.g. 4 mm part, approx. 10h at 60 °C)</td></tr>
<tr><td colspan="2">Weight loss</td><td>> 3.8 weight %</td></tr>
<tr><td colspan="2">Drying</td><td>to constant weight approx. 2 hours, 100 °C</td></tr>
</table>

Tab. 3: Properties of polyMIM® 316L D 120E

<table>
<tr><td colspan="6">polyMIM® 17-4PH D 222E</td><td colspan="4" rowspan="3">  </td></tr>
<tr><td colspan="2" rowspan="2">Oversize factor - dimensions must be multiplied by this rate.</td><td>min.</td><td>average</td><td>max.</td></tr>
<tr><td>1.1627</td><td>1.1669</td><td>1.1711</td></tr>
<tr><td colspan="2" rowspan="2">MVR melt index - DIN EN ISO 1133 (210 °C / 5 kg)</td><td>min.</td><td>average</td><td>max.</td><td colspan="4"></td></tr>
<tr><td>75</td><td>100</td><td>125</td><td colspan="4"></td></tr>
<tr><td rowspan="2">Typical composition as sintered; weight %</td><td>Fe</td><td>C</td><td>Ni</td><td>Cr</td><td>Mn</td><td>Si</td><td>Cu</td><td>other</td></tr>
<tr><td>Balance</td><td>< 0.07</td><td>3.0-5.0</td><td>15.0-17.5</td><td>< 1.0</td><td>< 1.0</td><td>3.0-5.0</td><td>< 0.45</td></tr>
<tr><td colspan="3">Typical properties (as sintered)</td><td colspan="6">Injection molding process settings</td></tr>
<tr><td colspan="2" rowspan="2">Density</td><td rowspan="2">≥ 7.65 g/cm^3</td><td rowspan="2">Cylinder temperature</td><td>Zone1</td><td>Zone 2</td><td>Zone 3</td><td>Zone 4</td><td>Nozzle</td></tr>
<tr><td>170 °C</td><td>175 °C</td><td>180 °C</td><td>185 °C</td><td>190 °C</td></tr>
<tr><td colspan="2">Yield strength Rp02</td><td>≥ 660 MPa</td><td>Tool temperature</td><td colspan="2">45-65 °C</td><td>Injection pressure</td><td colspan="2">800-1100 bar</td></tr>
<tr><td colspan="2">Tensile strength Rm</td><td>≥ 800 MPa</td><td>Injection speed</td><td colspan="2">3-20 cm^3/s</td><td>Post pressure</td><td colspan="2">600-950 bar</td></tr>
<tr><td colspan="2">Elongation A10</td><td>≥ 3 %</td><td rowspan="2">Peripheral screw speed</td><td colspan="2" rowspan="2">5-20 m/min</td><td rowspan="2">Back pressure</td><td colspan="2" rowspan="2">20-30 bar (hydraul.)</td></tr>
<tr><td colspan="2">Hardness</td><td>≥ 250 HV10</td></tr>
<tr><td colspan="3">Debinding process</td><td colspan="6">Sintering process</td></tr>
<tr><td colspan="2">Solvent</td><td>water</td><td colspan="2">Sintering atmosphere</td><td colspan="4">100% pure hydrogen</td></tr>
<tr><td colspan="2">Debinding temperature</td><td>40-60 °C</td><td colspan="2" rowspan="4">Sintering cycle</td><td colspan="4" rowspan="4">3 K/min → 600 °C,
2.5 h hold 600 °C,
5 K/min → 1350 °C,
3 h hold 1350 °C,
15 K/min → 80 °C,
cooling</td></tr>
<tr><td colspan="2">Debinding time</td><td>Depending on part thickness (e.g. 4 mm part, approx. 10h at 60 °C)</td></tr>
<tr><td colspan="2">Weight loss</td><td>> 3.7 weight %</td></tr>
<tr><td colspan="2">Drying</td><td>to constant weight approx. 2 hours, 100 °C</td></tr>
</table>

Tab. 4: Properties of polyMIM® 17-4PH D 222E

6.2 Injection molding machine

The injection molding machine ARBURG Allrounder 320C (EUROMAP size 600-100) was used for the testing. This machine is used for an injection molding of thermoplastics on a daily basis. Therefore, there are no additional features for PIM on this machine.

Fig. 32: ARBURG Allrounder 320C

Clamping unit		Injection unit	
Clamping force *(max.)*	600 kN	Screw diameter	25 mm
Closing force *(max.)*	35 kN	Effective screw length	20 L/D
Opening force *(max.)*	25 kN	Screw stroke *(max.)*	100 mm
Opening stroke *(max.)*	350 mm	Calculated injection Volume *(max.)*	49 cm^3
Mould height *(min.)*	200 mm	Shot weight *(max.)*	45 g (PS)
Daylight *(max.)*	550 mm	Injection pressure *(max.)*	2240 bar
Distance between tie bars	320 x 320 mm	Injection flow *(max.)*	124 cm^3/s
Platen size	446 x 446 mm	Peripheral screw speed *(max.)*	52 m/min
Weight of movable mold half *(max.)*	180 kg	Screw torque *(max.)*	150 Nm
Ejector force *(max.)*	30 kN	Back pressure positive / negative *(max.)*	350 / 200 bar
Ejector stroke *(max.)*	125 mm	Material hopper capacity	50 l

Tab. 5: Technical data for ARBURG Allrounder 320C

6.3 Testing conditions

First of all, functionality of the mold was proved by an injection molding of a thermoplastic material prior to the testing of polyMIM® feedstocks. Testing specimen made of polypropylene is shown in Fig. 33.

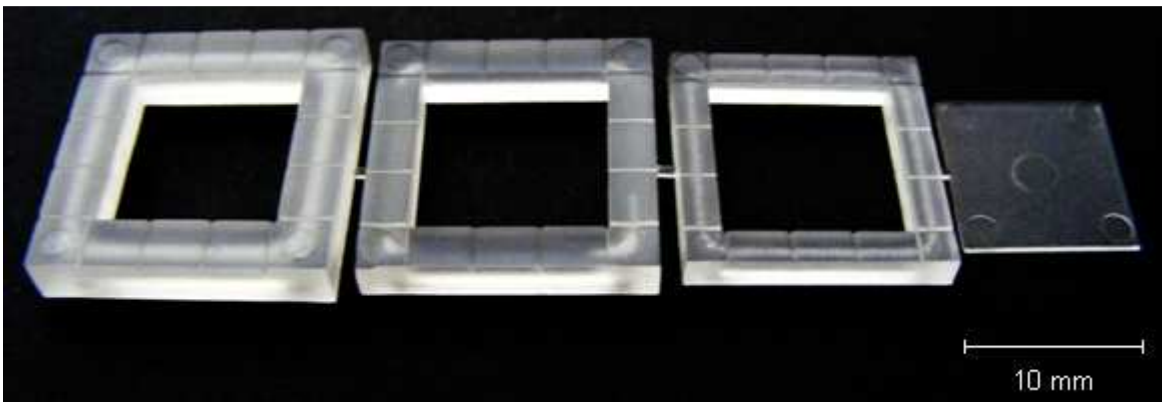


Fig. 33: Testing specimen made of polypropylene

After this trial polyMIM® 316L and polyMIM® 17-4PH were injection molded under the conditions shown in the following table.

	polyMIM® 316L				polyMIM® 17-4PH			
Cylinder temperature	Zone 1	Zone 2	Zone 3	Zone 4	Zone 1	Zone 2	Zone 3	Zone 4
	180 °C	185 °C	200 °C	208 °C	175 °C	180 °C	180 °C	185 °C
Nozzle temperature	210 °C				190 °C			
Mold temperature	60 °C				65 °C			
Injection speed	160 mm/s				188 mm/s			
Injection pressure	193 bar				193 bar			
Injection time	0.28 s				0.26 s			
Hold pressure	193 bar				193 bar			
Hold pressure time	0.3 s				0.3 s			

Tab. 6: Testing conditions for polyMIM® 316L and polyMIM® 17-4PH

Example of the testing specimen made of polyMIM® 316L feedstock is shown in Fig. 34.

Fig. 34: Testing specimen made of polyMIM® 316L feedstock

6.4 Results

The most visible area of the phase separation, occurring on a surface of the testing specimen, was located around the gates entering to each square element. Therefore, the scans of these areas were taken with a scanning electron microscope (SEM) to observe the phase separation. The locations of the scanned areas are marked in Fig. 35.

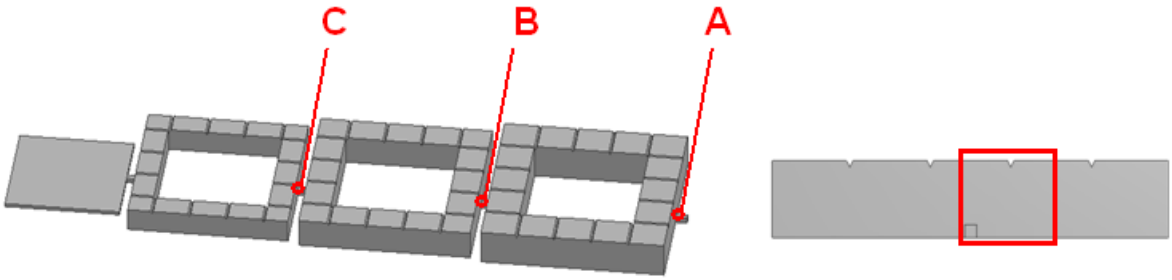


Fig. 35: Areas marked for scanning electron microscope analysis

SEM pictures displayed on the next pages demonstrate the development of phase separation in each area (A, B and C) of the testing specimen made of polyMIM® 316L (Fig. 36) and polyMIM® 17-4PH (Fig. 37). For each area several scans were taken. The resolutions 60x and 200x were used for the polyMIM® 316L testing specimen and the 60x and 400x resolutions were selected for the testing specimen made of polyMIM® 17-4PH.

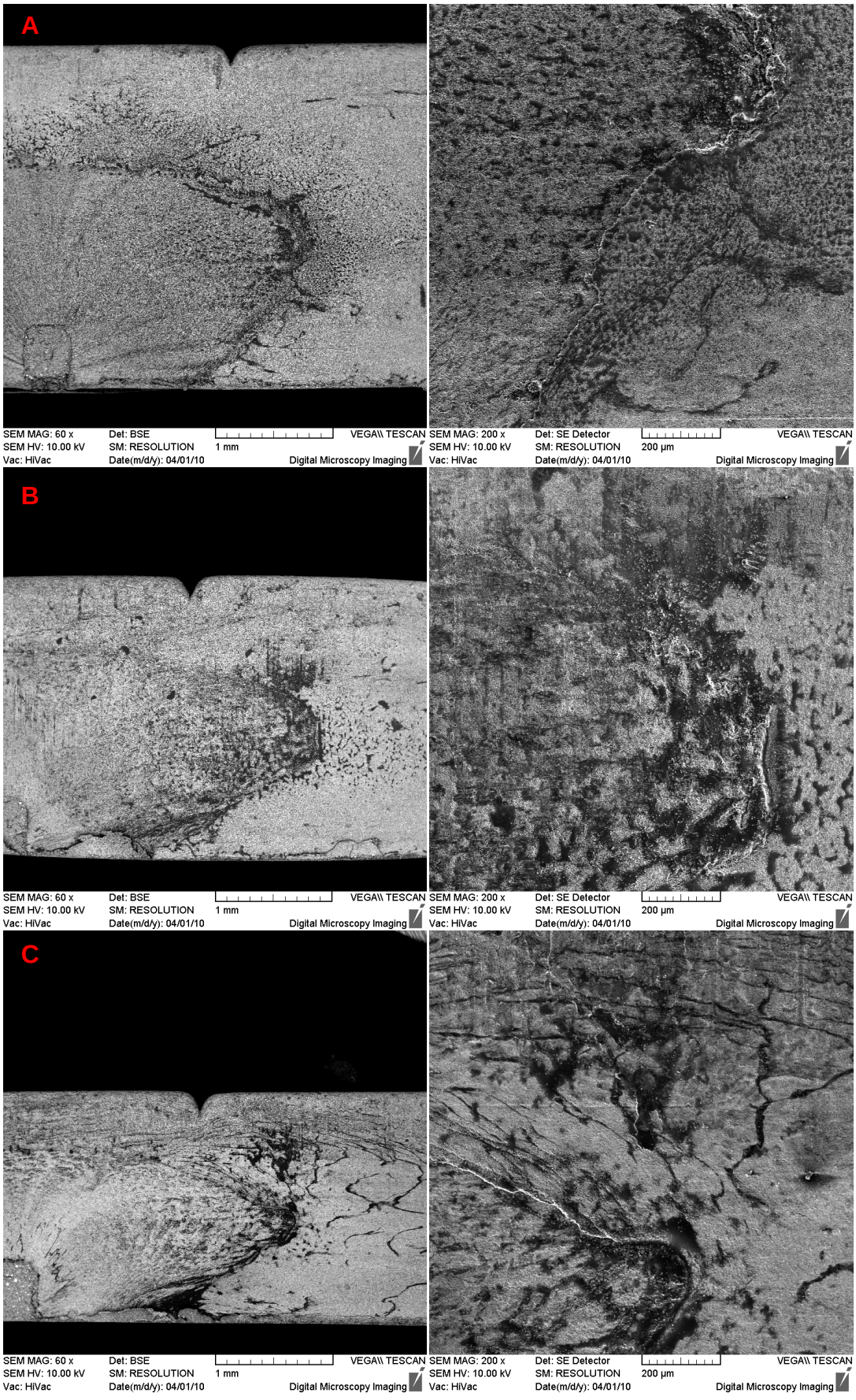


Fig. 36: SEM of phase separation areas of polyMIM® 316L

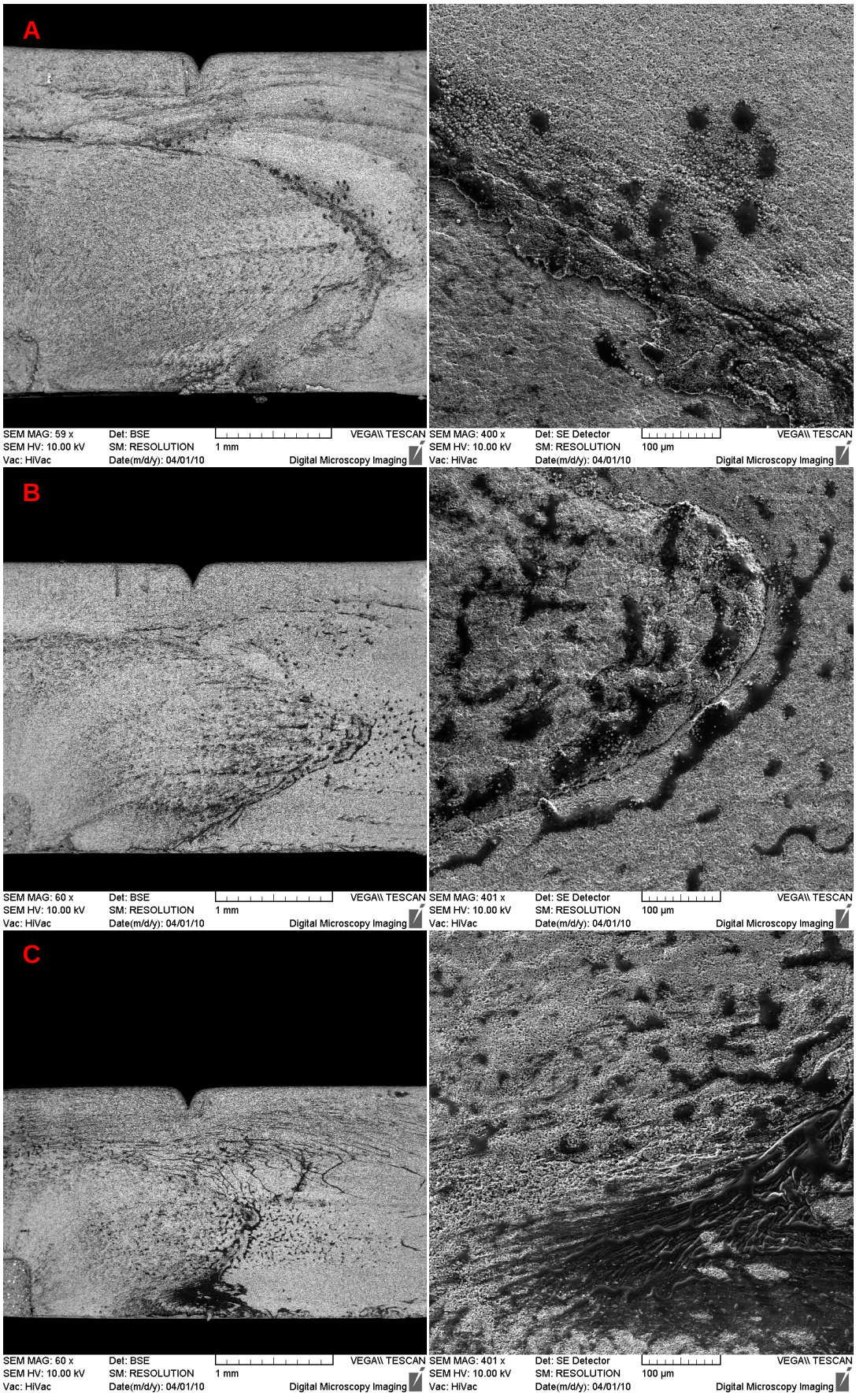


Fig. 37: SEM of phase separation areas of polyMIM® 17-4PH

The SEM analysis clearly shows the development of phase separation during the filling of the mold. The size of binder-rich area significantly progresses as the flow front moves from the position A to the place marked C. It can be considered as a result of a decreasing cross section as well as increasing flow length.

Futher, the difference between the phase separation of polyMIM® 316L feedstock and polyMIM® 17-4PH feedstock can be seen on the SEM pictures when these two materials are compared. The polyMIM® 316L shows slightly larger phase separation than the polyMIM® 17-4PH despite of their different melt flow indexes and molding conditions used. Thus, it seems that the testing mold can be utilized to compare various feedstocks from the point of view of their disposition to powder-binder separation.

However, to quantify the phase separation and understand the mechanism of its origin, a broad range of feedstocks, having various rheological properties, should be tested as well as the influence of different molding conditions has to be evaluated. By the help of such an experimental approach, this most critical feedstock feature could be identified.

CONCLUSION

Powder injection molding (PIM) is considered as an effective alternative to machining in production of metallic and ceramic components. PIM process combines the productivity of injection molding technology with the ability to fabricate metals and ceramics. Even though this technology offers a great potential, its worldwide recognition still remains rather low. It is mainly due to a fact that PIM production has a complicated process chain, where each step has its own quality issues. Some of these quality issues cannot be identified until the final stage of the PIM process is reached, and thus resulting in material, time and energy losses. A quality influencing factor of major importance is phase separation, which occurs during injection molding step. The phase separation might cause visual defects, porosity, warpage or even cracks. The mechanism of phase separation has still not been fully understood. The challenging idea of this work consists in linking the properties of injection molded parts to the quality defects appearing on final sintered products via determination of factors responsible for powder-binder separation effect.

In the theoretical part of the thesis successful PIM application examples are given, advantages and disadvantages of this technique are summarized, and completed with a detailed description of particular stages of the process - mixing, injection molding, debinding and sintering. PIM feedstock composition and material characteristics are also presented. The remaining pages of the theoretical part describe phase separation as a quality influencing factor in PIM technology, interpreting results of a study focused on a virtual simulation of the phase separation, as well as investigation based on an experimental evaluation of the phase separation.

In the practical part of the thesis the design of testing specimen is created for a better understanding of the phase separation phenomenon. The geometry of this testing specimen was developed to involve critical elements causing the phase separation as observed on various molded PIM components. Testing specimen contains inner and outer corners, radical thickness changes, weld lines and a thin film part. The designed geometry also allows us to observe the phase separation progression by including elements with different cross sections. This testing specimen, particularly designed to evaluate PIM feedstock disposition to the phase separation, can be used in the future instead of the currently used molds, designed mainly for investigating the moldability of plastics.

To produce the testing specimen, an injection mold was designed by using the Autodesk® Inventor® 2010 software. The injection mold assembly was constructed by modifying standardized parts supplied by HASCO. A brief description of the modifications made on each mold component is given in the practical part of this thesis. The drawing documentation of the injection mold assembly and all modified components is enclosed as an appendix. The final chapter presents the testing conditions and the properties of commercially available feedstocks used for testing. To demonstrate the results, phase separation areas on the surface of injection molded specimen were analyzed with a scanning electron microscope.

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LIST OF ABBREVIATIONS

2D	Two dimensional
3D	Three dimensional
CAD	Computer-aided design
CIM	Ceramic injection molding
DIN	Deutsches Institut für Normung
DSC	Differential scanning calorimetry
EVA	Ethylene vinyl acetate
ISO	International Organization for Standardization
IT	Information technology
MIM	Metal injection molding
MPIF	Metal Powder Industries Federation
PA	Polyamide
PE	Polyethylene
PEO	Polyethylene oxide
PIM	Powder injection molding
PMMA	Polymethyl methacrylate
POM	Polyoxymethylene
PP	Polypropylene
PS	Polystyrene
PVA	Polyvinyl alcohol
®	Registered trademark
Ra	Roughness (arithmetic average of absolute values)
SEM	Scanning electron microscope

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