

Metal Injection Molding

A review of the MIM process and its optimization

Sebastian Engström

EXAMENSARBETE	
Arcada	
Utbildningsprogram:	Plastteknik
Identifikationsnummer:	
Författare:	Sebastian Engström
Arbetets namn:	Metallformsprutning: En redogörelse av MIM processen och dess optimering
Handledare (Arcada):	Erland Nyroth
Uppdragsgivare:	Plastoco Oy Ab
<p>Sammandrag:</p> <p>Metallformsprutning är en metod där man producerar metallkomponenter genom plastproduktionstekniken formsprutning. Denna tillverkningsmetod möjliggör produktion av metallkomponenter med komplicerad design och låga toleranser tack vare formsprutningsmaskinens funktionalitet och automation. Processen kräver dock några extra steg för att uppnå den slutliga komponenten. Syftet med detta examensarbete är att redogöra för hela metallformsprutningsprocessen och alla dess delmoment. En fördjupad insyn i varje enskild process och underliggande mekanismer presenteras. Redogörelsen fokuserar på optimeringen av processparametrar och på de komplikationer som kan uppstå i samband med den. Forskningen genomförs främst genom litterära studier av böcker, skrifter och tidigare forskningsarbeten. En ytterligare informationskälla är konsultering med experter och personer som jobbar inom metallformsprutningsproduktion. Förutom detta baseras sig informationen också på personligt åskådande av och deltagande i tillverkningsprocessen. Det huvudsakliga målet med forskningen är att undersöka hur man inleder och verkställer optimeringen av parametrar i en metallformsprutningsproduktion. Forskningens resultat tyder på att en analys och en prövningsinriktad metod är mest givande. Arbetet avslutas med ett diagram över arbetsflödet i optimeringsprocessen.</p>	
Nyckelord:	Metal, Injection molding, MIM, Debinding, Sintering
Sidantal:	67
Språk:	Engelska
Datum för godkännande:	

DEGREE THESIS	
Arcada	
Degree Programme:	Plastteknik
Identification number:	
Author:	Sebastian Engström
Title:	Metal Injection Molding: A review of the MIM process and its optimization
Supervisor (Arcada):	Erland Nyroth
Commissioned by:	Plastoco Oy Ab
<p>Abstract:</p> <p>Metal injection molding is a manufacturing method for producing metal parts using the plastics injection molding technique. This manufacturing method allows to produce metal parts with complicated shapes and small tolerances, with the ease and automation of the injection molding machine. The process however requires some extra steps to accomplish this. The aim of this thesis is to cover the whole metal injection molding process and all the stages involved. An in-depth look at each individual stage and the mechanisms involved are presented. Throughout the review the focus is centered on the potential for process optimization as well as the complications involved. The research is approached primarily through the studies of literature on the subject of metal injection molding, this includes comprehensive books as well as research papers. Another source for information is through the consultation of experts and people who work with this manufacturing method. In an addition to this, personally witnessing and partaking in production is also used as a source for firsthand information. The main goal of the optimization-centered research is to explore how to approach an optimization of parameters in a metal injection molding production. The research results indicate an analysis heavy, trial and error approach being most feasible. The thesis concludes on a workflow diagram of the optimization process.</p>	
Keywords:	Metal, Injection molding, MIM, Debinding, Sintering
Number of pages:	67
Language:	English
Date of acceptance:	

Table of contents

1	Introduction.....	6
1.1	Aim	6
1.2	Background	6
1.3	Method.....	7
2	METAL INJECTION MOLDING.....	8
2.1	Injection molding.....	9
2.1.1	<i>Injection molding machine</i>	10
2.2	Feedstock	12
2.2.1	<i>Powder</i>	14
2.2.2	<i>Binder</i>	17
2.2.3	<i>Powder Binder mix</i>	20
2.3	Debinding	21
2.3.1	<i>Primary debinding</i>	21
2.3.2	<i>Solvent debinding systems</i>	23
2.3.3	<i>Catalytic debinding system</i>	25
2.4	Secondary debinding.....	29
2.5	Sintering	31
2.5.1	<i>Sintering mechanisms</i>	31
2.5.2	<i>Shrinkage and densification</i>	37
2.5.3	<i>Sintering equipment and atmosphere</i>	39
2.5.4	<i>Equipment</i>	42
2.5.5	<i>Cycle</i>	44
3	PROBLEMS AND OPTIMIZATION	46
3.1	Molding problems	47
3.1.1	<i>Molding defects</i>	47
3.2	Debinding problems.....	50
3.2.1	<i>Debinding defects</i>	50
3.3	Sintering problems.....	52
3.3.1	<i>Sintering defects</i>	52
3.4	Monitoring.....	55
3.4.1	<i>Molding</i>	55
3.4.2	<i>Debinding</i>	56

3.4.3	<i>Sintering</i>	57
3.4.4	<i>Inspection / Final testing</i>	57
4	Optimization of parameters	58
4.1.1	<i>Molding</i>	59
4.1.2	<i>Debinding</i>	60
4.1.3	<i>Sintering</i>	61
5	RESULTS	64
6	DISCUSSION AND CONCLUSION	66
7	SUGGESTION FOR FURTHER WORK	67
8	References	Error! Bookmark not defined.

1 INTRODUCTION

1.1 Aim

The aim of this research is to provide an in-depth look at the metal injection molding process. It will examine the mechanisms behind the process and the parameters involved. Throughout the review the focus will be centered on the potential for process improvements as well as the complications involved. The concluding task is to formulate a concise and general model for the optimization process.

1.2 Background

The background and idea for this thesis came about as a commission to optimize a commercial metal injection molding production process. While doing research it became evident that there aren't any rules or perfect formulas for calculating the optimal parameters. This led the research to be more focused on figuring out the approach for an optimization process.

There is somewhat of a lack of information on the subject so any new research is valuable. The metal injection molding manufacturing method is quite complex and time consuming so by optimizing the process the benefits will be vast. For a producer of metal injection molded components the economical benefits will stem from a shorter process duration, smaller resource consumption and more reliable production. Improved mechanical properties of the produced parts can also be achieved by optimizing the process parameters.

Another reason for this research subject is to raise awareness about the production method. Metal injection molding is a production method which might get easily overlooked or not know about at all. There's value in simply shedding light and informing engineers on the technique of producing metal parts with a plastics molding machine.

1.3 Method

The approach for the research will be primarily through studying literature on the subject of metal injection molding. This will include comprehensive books as well as research papers. Another source for information will be consulting with experts and people who work with the manufacturing method. In an addition to this personally witnessing and partaking in MIM production will also be conducted in order to gather firsthand information. The collected material will function as the basis for a reasoned conclusion.

2 METAL INJECTION MOLDING

Metal injection molding is a manufacturing method for producing parts made of metal. The Metal Injection Molding process is usually called by its short acronym MIM, as it will do in this work. MIM is a subcategory of the Powder Injection Molding (PIM) manufacturing technique. PIM utilizes the plastics-production technology of injection molding with its feedstock consisting of a polymer and an inorganic material. The inorganic component of the feedstock can be a metal or a ceramic. (German & Bose, 1997, pp. 11-13)

The fundamental idea behind the MIM manufacturing method is to combine the shaping benefits of injection molding with the resilient mechanical properties of metals. This “best of both world” approach allows the production of complex and detailed metal parts with high strength and stiffness. Traditional casting methods requires the metal to be in a molten state during the casting, the metal powder and polymer feedstock used in MIM enables the molding process to be performed at much lower temperatures. (Heaney, 2012, pp. 1, 109)

Due to the multiple material consisting MIM raw material and the sub melting point temperature of the molding process some extra steps are required in order to produce the finished part. The molding stage is quite a simple and straight forward process. It is done in the exact same way as regular injection molding of plastic parts: feedstock is loaded into the injection molding machine and the machine drives the screw filling the mold. Unlike injection molding of plastics, the molded part still needs to go through a few more steps to be complete. (German & Bose, 1997, pp. 133-135)

The first stage following the injection molding is debinding. The debinding stage is designed to get rid of the polymer binder material from the molded part, making it purely metal. After the binder material has been removed follows the final stage, sintering. At the sintering stage the now pure metal powder part is heated up to near melting tempera-

tures. The high temperature makes the particles of the metal powder fuse together, increasing the density and strength of the part. A well performed sintering provides the MIM-produced part with properties similar to that of a die casted. (German & Bose, 1997, pp. 11-15)

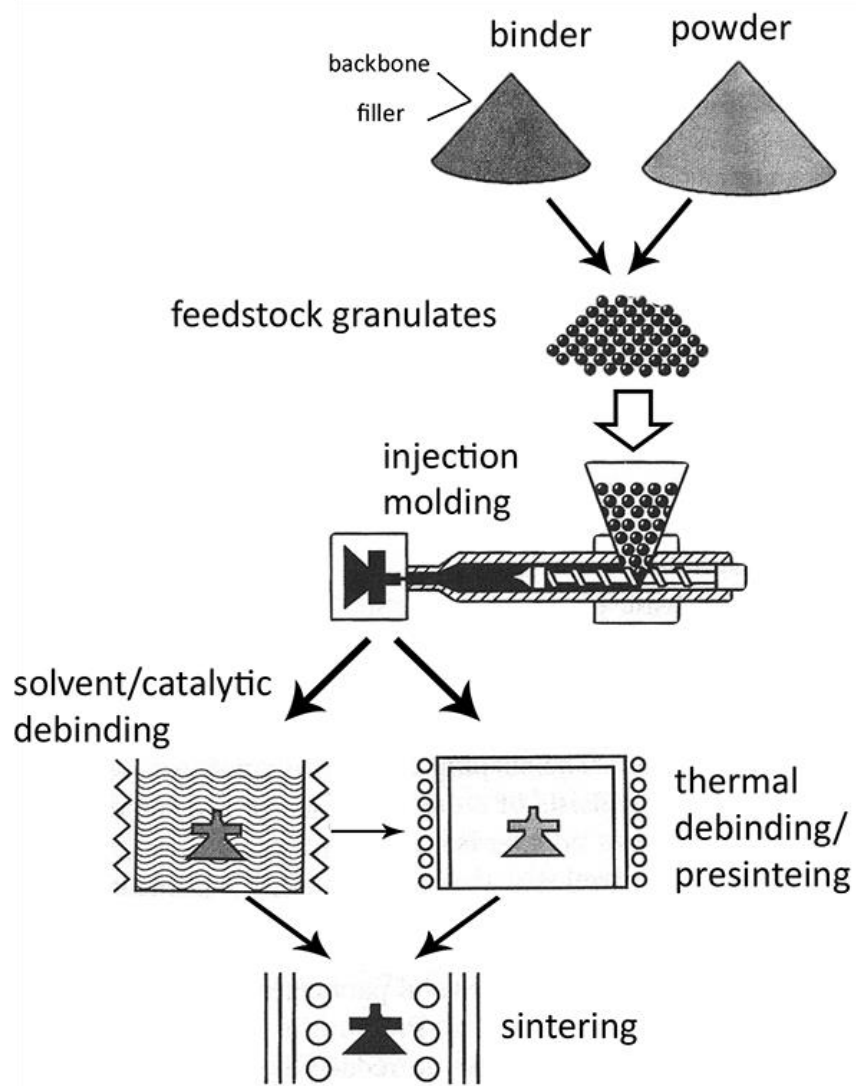


Figure 1. Metal injection molding process stages. (German & Bose, 1997, p. 15)

2.1 Injection molding

Injection molding is the principal manufacturing method used in plastics production. Since the MIM process starts out with a feedstock consisting of a substantial amount of polymer (40 vol.%) the injection molding stage is very similar to plastics molding. As only the binder component of the MIM feedstock will be molten during the injection the

process can essentially be viewed as a normal thermoplastics injection molding. (Heaney, 2012, pp. 110-112)

The basic work principle of injection molding is fairly simple, heat up a material till it's molten, then inject the liquid material into a mold with the desired shape, the material will adopt the shape of the mold and stay that way after it's cooled back down into solid state.

Metal injection molding is performed with the same injection molding machines as used in plastics production. Molding machines using a reciprocating screw is by far the most common in both plastics and MIM production.

2.1.1 Injection molding machine

An injection molding machine is the apparatus that helps shape and produce parts. The machine is made up of two main units, an injection unit and a clamping unit. The two units are focused on performing two different tasks which when combined will produce the parts. (German & Bose, 1997, pp. 133-136)

Injection unit

The main task of the injection unit is to heat up the feedstock and then force it into the mold. The injection unit controls features from feedstock viscosity to shot size. The unit consists of a few central components:

Hopper: Is the container that sits above the machine and feeds granulates to the process

Reciprocating screw: is the heart of the injection unit. It not only provides the push that forces the molten raw material into the mold it's also the prime producer of heat.

Barrel: is the casing around the screw, the barrel is covered in adjustable heating bands that keep the barrel at constant temperature.

Screw motor: Provides the rotation for the screw.

Injection cylinder: Moves the screw forwards and backwards inside the barrel. Also provides the thrust for the injection.

Nozzle: Connects the barrel to the mold. Provides the final heating to the shot before it enters the cavity. (German & Bose, 1997, pp. 136-145)

Clamping unit

The main task of the clamping unit is to control the mold throughout the cycle. With the help of a hydraulic motor the clamping unit can open and close the mold. The most demanding aspect of the clamping unit is keeping the mold halves tightly together during injection and packing. The clamping unit also controls the part release through moving the ejector rod and pins. (German & Bose, 1997, pp. 136-145)

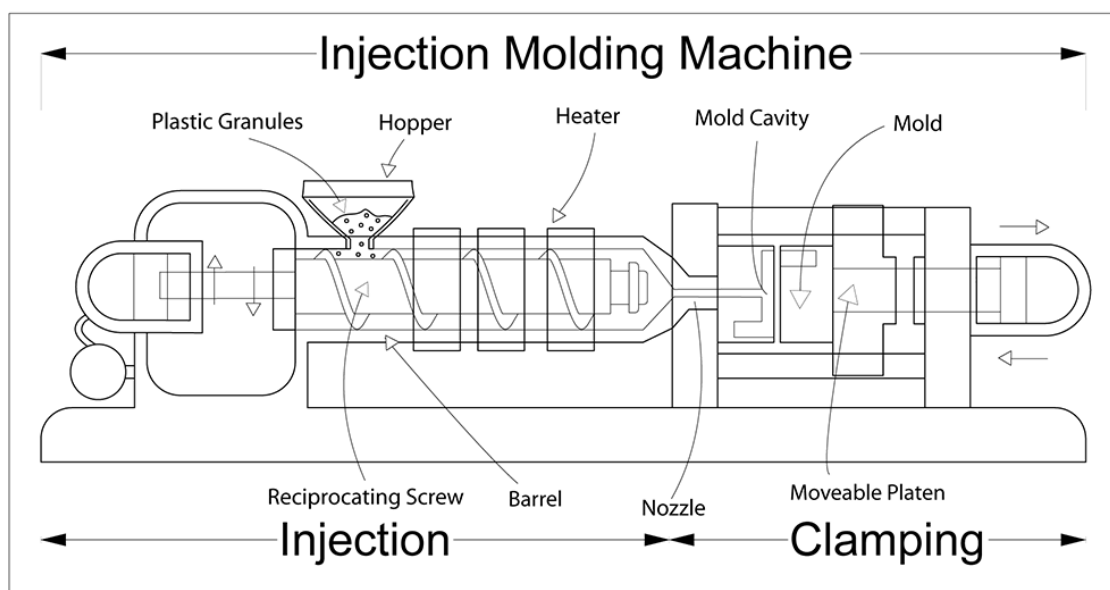


Figure 2. Injection molding machine (B.Rocky,2009)

Molding cycle

The molding cycle can be divided into different phases:

1. Feedstock granulates are fed from the hopper into the heated barrel of the injection molding machine.
2. A rotating screw inside the barrel crushes and melts the granulates. The rotating screw forces the molten material towards the end of the barrel right up to the mold, meanwhile the screw is moving back.
3. A hydraulic ram forces the collected liquid-state material into the mold cavity. Over 90- 99% of the cavity will get filled.

switch over (the second stage injection commences)

4. The remaining < 10% of material is injected with high pressure. The high pressure forces the molten material into every corner and hard to fill place. Following the second stage injection a holding pressure is introduced to insure the molded melt doesn't lose shape or shrink during solidification.

5. Mold opens, ejector pins push the solidified part out from the mold cavity.

6. Mold closes and cycle repeats. (Heaney, 2012, pp. 116-118) (Polyplastics, 2016)

The above-mentioned cycle is roughly the same for both traditional plastics injection molding as well as metal injection molding. One difference though is that the MIM feedstock isn't completely molten during molding. Only the binder part of the feedstock will be molten while the powder stays in a solid pulverized form. (Heaney, 2012, pp. 116-118)

2.2 Feedstock

Feedstock refers to the raw material used in metal injection molding. The whole MIM process is essentially based upon the properties and constituency of the feedstock. The feedstock consists of two main components: a binder and a metal powder. Both components provide their own unique function in the production process. (German & Bose, 1997, p. 25)

The feedstock is usually in the form of pellets similar to the granules used in plastics production. Since the feedstock itself plays such a central role in the MIM process, the characteristics of the feedstock will influence every step from start to finish. The influence that the feedstock will have on the molding process can be traced back to five main factors:

- Metal powder characteristics
- Binder composition
- Powder/binder ratio
- Mixing method
- Pelletizing technique

(German & Bose, 1997, p. 43)

Metal powder characteristics

Powder is a term describing a collection of fine particles. The properties of a powder are a cumulative product of the particles characteristics. The particle characteristics will influence every step of the MIM process. Particle size and shape for instance will affect the interparticle friction, packing density, rheology and so on. The injection molding process will be affected by the above-mentioned characteristics by the ease of molding and tool wear. The metal type and grade will also play its part on the molding since it will influence the particle weight. (Heaney, 2012, pp. 50-55)

Binder composition

The second component to the feedstock is the binder. Although the binder is a disposable element during the MIM process its characteristics will have a fundamental impact on the whole production. The binder is composed of at least two different material. The material which makes up the binder will influence the binders melting point and rheology. The injection flow and operation temperature are directly influenced by the binder constituents as well. For a successful molding the melt needs to have the correct viscosity to fill the mold cavities fully yet not produce flash. The ratios of the different binder components will also affect the binder characteristics. Some ready-made feedstocks contain additional additives in the binder to help with mold release and injection. (Heaney, 2012, p. 70)

Powder/binder ratio

The ratio between how much powder and binder are in feedstock will influence its performance. In order to have a successful molding process the two components need to have suitable proportions. Too much powder will cause problems with shape retention as well as being abrasive on the injection molding equipment. (German & Bose, 1997, p. 26)



Figure 3. MIM Catamold feedstock granulates, 316L stainless steel (author,2017)

2.2.1 Powder

The metal powder portion of the feedstock is what provides the mechanical properties to the finished product. A produced MIM-part displays similar properties as the metal that the powder is made from. In principle any metal powder can be used in MIM production as long as it satisfies the following criteria; the powder particle size needs to be small, mix well with polymers, sinter to a sufficiently high density and have high enough melting and sintering temperature to not interfere with the debinding process. As an example, the widely used metals magnesium and aluminum are not common due to their low melting temperature and strong oxidation during sintering. Most commonly used alloys consist of stainless steel, tool steel, copper, cemented carbides, titanium and other refractory metals. (Heaney, 2012, pp. 50-56)

The characteristics of a powder are usually presented by describing the individual particles rather than a collective powder lot. The properties of powder can be boiled down to a few main characteristics: particle size, size distribution and particle shape. The sum of these characteristics result in the powder its packing density.

Packing density describes how tightly particles can be packed together. Using a powder with high packing density is essential to the MIM process. A powder with high packing density will result in better sintering with less shrinkage. Using particles that can obtain a high packing density has also the benefit of increased interparticle friction. The friction between the powder particles will help keep the molded components intact during debinding. (German & Bose, 1997, pp. 55-57)

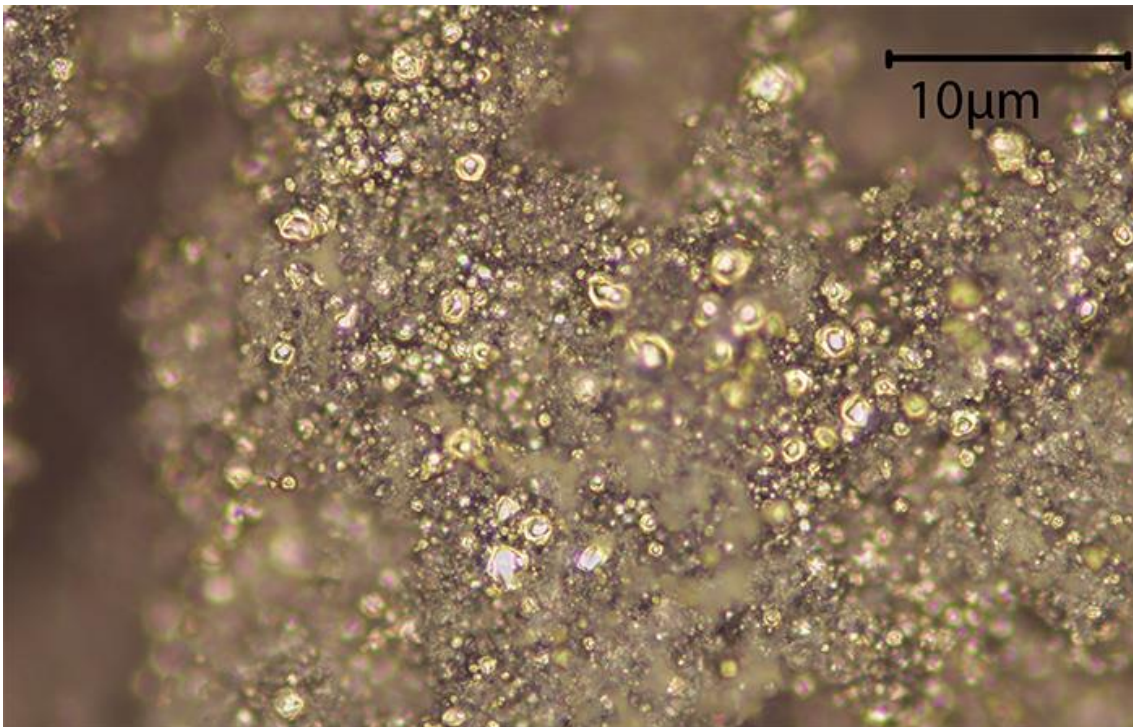


Figure 4. MIM Catamold feedstock (316L) under microscope. The shining orbs are metal particles. (author,2017)

Particle size

In theory, the particle size of a powder can be as big as 45 μm but the ideal particle size for most alloys is below 22 μm in diameter. A small particle is desired since it will increase the packing density and improve homogeneity in the feedstock. Some metals such as cemented carbides can have particles less than 5 μm . A powder with a small particle size also provides a smoother surface finish as well as being less abrasive to the injection molding machine. The only reason for choosing a powder with a larger particle size would be the lower initial cost. But in practice the production cost might not be much lower since larger particles have a tendency to cause difficulties during the injection molding.

Smaller particles also have a greater surface area and consequently higher surface energy. High surface energy is favorable since it's what drives the sintering mechanism. (German & Bose, 1997, p. 58) (Heaney, 2012, pp. 51-52)

Particle shape

The shape of the particles have a huge impact on the success of the MIM process. The particles get their shapes from their manufacturing method. A spherical shape is desired for numerous reasons. A round shape will help the feedstock flow better during injection. Spherical particles also mixes well with the binder material as well as distributes evenly, resulting in good homogeneity. (German & Bose, 1997, pp. 58-60)

Feedstock composed of powders with irregular particle shapes are nevertheless used regularly in MIM productions. The irregular shaped particles are shown to provide slight structural benefits to the molded part, helping the part keep its shape during debinding. A sphere shape is still preferred since it has descent shape retaining properties. The large surface area of a round particle increases the friction between particles in all directions, keeping it in shape. The inter-particle friction can be enhanced furthermore by reducing the particle size and thereby increasing the particle to particle contact. Spherical shapes can obtain quite high packing density amidst each other as well as obtain high solids loading when mixed with the binder material. (German & Bose, 1997, pp. 58-60) (Heaney, 2012, pp. 54-55)

Size distribution

In a batch of MIM powder, every particle won't be the exact same size. This variance in size can be quite substantial and wort paying attention to. The size distribution in MIM powders are described by their D10, D50 and D90 value. The D10 value states the size that only 10% of the particles of a powder are below (e.g. D10 @ 4,6 μ m). D50 describes the mean particle size, in other word 50% of the particles are below this size (e.g. D50 @ 11 μ m). D90 value is the size which covers 90% of the particles, only 10% of the particles in this powder batch will measure above this size (e.g. D90 @ 18 μ m). (Heaney, 2012, pp. 53-54)

Table 1. Example of MIM powder characteristics (German & Bose, 1997, p. 77)

manuf. tech	material	type/grade	vendor	composition	D10 [μm]	D50 [μm]	D90 [μm]
gas atomized	stainless steel	316L	Ultrafine	Fe/Cr/Ni/Mo	4,6	11	18
water atomized	stainless steel	17 -4 PH	Mitsubishi	Fe/Cr/Ni/Cu	4,8	20,7	50,9

2.2.2 Binder

The second component to make up the feedstock is the binder. While the metal powder is what provides the structural properties to the finished component the binder material won't provide anything and in fact won't be present in the end at all. The role of the binder is purely to provide the feedstock mix with moldability and shape retaining properties during manufacturing. The binder material plays a very decisive role and the success of the whole MIM operation is often dependent on the performance of the binder.

The binder is made up of a combination of a few different materials, all with their own unique function. Typically a MIM binder consists of three main components; a polymer, a filler material and a surfactant. Of these three components the polymer and the filler material make up the vast majority of the binder. The filler material is usually some type of wax or wax-like material. The polymer and wax usually make up similar proportions of the binder. By having both components roughly the same quantity a good interconnection within each material is achieved throughout the molded part. Typically one of these major components will make up between 20 to 80% of the binder. Although even proportions are desired complete miscibility isn't. The differences in molecular weight and chemical structure keeps the polymer and wax from completely mixing together, which helps the binder components extract separately during debinding. A difference in melting point temperature between the components is the main attribute to facilitates the selective removal of separate components throughout the MIM process. Waxes tend to have low melting temperatures (< 100°C) while polymers tend to have higher (> 100°C). This implies that the wax phase is the first one of the major components to be removed, this

happens during the debinding. The conditions of the debinding aren't harsh enough for the polymer to degrade. The polymer extraction takes place after the filler is removed and coincides with presintering. (German & Bose, 1997, p. 83) (Heaney, 2012, pp. 64-66)

Although there's an almost endless selection of polymers and filler materials to choose from there's only a limited number of feasible combinations. The binder components need to have properties compatible with one another as well as function as a whole. The binder configuration also need to work with the powder and display good wetting. Approaching the binder composition from a practical angle, the foremost criteria of selection is based on the available debinding methods and the desired powder material. (German & Bose, 1997, p. 83) (Heaney, 2012, pp. 64-66)

Secondary binder

The Secondary binders function is to provide structural strength to the molded MIM part during debinding and early sintering. Due to its crucial role as structural aid trough out the MIM process the secondary binder component is often referred to as "backbone". The secondary binder is almost always made from a polymer. It's usually the backbone polymer that has the highest melting temperature of all the binder components. The polymer part of the binder is the trickiest and most time-consuming constituent to remove. (German & Bose, 1997, pp. 83-90)

Polyethylene and polypropylene are the most frequently used binder polymers. They bear the ideal properties since they can provide the necessary structural strength while still being relatively effortless to remove. They have low melting temperatures and low viscosity which provides for efficient molding. They also have good wetting so they mix well with the other binder-materials as well as the powder. Their short molecular chain lengths allow them to decompose with relatively small volume changes minimizing the chance of complications during debinding and sintering. PE and PP are also two of the most commonly produced plastics making them very inexpensive as binder material components. They also create very little carbon contamination from the final burnout in the secondary debinding step. (Heaney, 2012, pp. 69, 144-146)

Primary binder

The primary binder or “filler phase” is usually a wax or wax-like material. Its purpose is to provide moldability and structural strength while still being easy to remove. Unlike the polymer which also serves the same structural purpose the wax is completely removed during debinding. The filler phase along with the surfactant are often referred to as the primary binder. Waxes have melting temperatures much lower and degrade easier than polymers, this provides them to be removed separately. The low molecular weight of waxes makes them evaporate easily, this is a desired aspect since it helps during debinding. Most commonly used waxes as fillers are paraffin wax, carnauba wax and bees wax. (Heaney, 2012, pp. 69, 136-141) (German & Bose, 1997, pp. 86-92)

Surfactant

As mentioned prior a typical binder consists of three main elements, of which the backbone polymer and the filler are the essential ones. The third and remaining component is the surfactant. A surfactant is a compound which lowers the surface tension between two liquids or between a liquid and a solid. In the case of metal injection molding the surfactants function in the binder is to improve the wetting between the metal powder and the binder itself. A binder can be composed of only two materials as long as the filler material has properties that resemble that of a surfactant. A secondary function of the surfactant is to aid with tool release during molding. (German & Bose, 1997, pp. 86-92) (Heaney, 2012, p. 69)

Table 2. Example binder constituents (German & Bose, 1997, p. 91) (Heaney, 2012, p. 81)

316L		316L		316L	
starch	41 %	LDPE	45 %	paraffin wax	30 %
glycerol	23 %	paraffin wax	45 %	carnauba wax	10 %
LDPE	29 %	stearic acid	10 %	bees wax	10 %
citric acid	2 %			PP	45 %
stearic acid	5 %			stearic acid	5 %
17-4 ph		17-4 ph		copper	
paraffin wax	63 %	paraffin wax	55 %	paraffin wax	65 %
microcrystalline p.w	16 %	microcrystalline p.w	16 %	PE	30 %
EVA	15 %	EVA	15 %	stearic acid	5 %

HDPE	5 %	HDPE	5 %
stearic acid	1 %	stearic acid	9 %

iron-nickel		iron-nickel		iron-nickel	
paraffin wax	79 %	paraffin wax	79 %	paraffin wax	55 %
EVA	20 %	HDPE	20 %	PP	25 %
stearic acid	1 %	stearic acid	1 %	stearic acid	5 %
				carnauba wax	15 %
HS12-5-5 HS steel		316L		316L	
HDPE	50 %	paraffin wax	65 %	paraffin wax	75 %
paraffin wax	50 %	EVA	35 %	PE	20 %
				EVA	5 %

2.2.3 Powder Binder mix

The ratio between metal powder and binder material is highly critical to the success of the whole MIM process. An unbalanced ratio between the two components will result in defects and improper shrinkage. Too little powder and excessive amounts of binder will lead to complications during molding. With excess binder in the feedstock the powder and binder will separate from each other during injection causing an uneven spread of metal particles. The excess binder will also result in low viscosity making flashing likely. A large enough binder excess can cause the parts to slump or completely collapse during debinding once the binder is getting extracted. Conversely if the ratio is off due to excess powder and insufficient binder empty voids inside the parts will occur. A part containing voids will easily crack during debinding. Other problems caused by excess powder are related to molding difficulties. The large amount of powder won't flow properly causing a multitude of defects. The optimal mixture ratio is at something called critical solids loading. Critical solids loading is the point where powder particles are packed as tightly as possible together and binder material fills all the space between the particles. In other words all the particles are chained together by contact and the binder allocates all unfilled space leaving no room for voids. (German & Bose, 1997, pp. 26-28) (Heaney, 2012, p. 73)

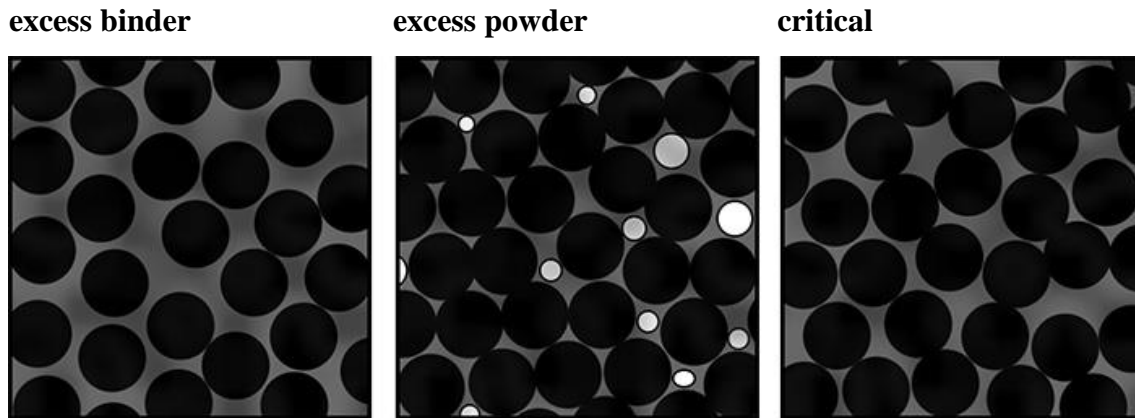


Figure 5. Illustration of different powder/binder ratios. Black= metal particles, Gray=binder, White= voids

2.3 Debinding

Debinding is the process of removing the binder-material from the molded part. At the end of the debinding process the produced parts will be consisting purely of metal. The debinding process is perhaps the most critical step in MIM production. The success relies heavily on how carefully the binder is removed. During debinding the molded part must endure the stresses produced by the binder being extracted from within the part, while still maintaining its shape. The debinding process requires two steps: primary debinding and secondary debinding. The function of the primary debinding is to get rid of the filler phase and surfactants. The secondary debinding step is where the backbone binder is removed and the sintering process sets in. It's during debinding that the binder portion of the feedstock goes from being the sole structural component to becoming fully disposable. (Heaney, 2012, pp. 133-134) (German & Bose, 1997, pp. 175-180)

2.3.1 Primary debinding

Primary debinding is the process of getting rid of the more easily removable binder components. This part of the binder is referred to as: primary binder or soluble binder. The idea is to remove the primary binder efficiently and without damaging the part while concurrently creating a tunnel system for the backbone polymer to exit through during secondary debinding.

The debinding is performed either by thermal debinding or solvent based debinding. In the case of thermal debinding the molded part is introduced to an environment of temperature high enough to make the primary binder evaporate away. In practice this is a very slow and time-consuming process since the temperature has to be low enough to not affect the backbone polymer. The evaporation also has to occur gradually, moving from the edges inwards in order not to crack or distort the molded part. Due to the time consuming nature thermal debinding is rarely used in commercial production. (German & Bose, 1997, pp. 202-206) (Heaney, 2012, pp. 136-137)

Solvent based debinding is the more modern approach for removing the primary binder. Rather than thermally evaporating the primary-binder solvent debinding relies on the chemical characteristics of the binder components. Solvent debinding works on the principal of using a primary and secondary binder that aren't soluble by the same solvent. By introducing the molded part to an atmosphere consisting of a solvent the primary binder will react by losing its solidity and flowing out of the compact, creating a pore structure required for the final secondary debinding. The type of solvent used and the binder materials only work in certain combinations since they need to be completely compatible for the debinding to work. There are a number of different methods of introducing the molded part to the solvent.

Since the binder-materials, solvents and the work method are so codependent and exclusive to each other they are often referred as a collective system. In contrast to thermal debinding where the binder is removed as a vapor, solvent based extraction usually happens through liquid flow. (Heaney, 2012, pp. 136-137) (German & Bose, 1997, pp. 202-206)

By the end of primary debinding all of the primary binder will have been removed and the part is now referred to as being "brown". With all the filler phase removed the part is now full of pores through out the part and the only thing keeping the part from falling apart is the backbone polymer. A part in the brown state is fragile and needs to be handled with care. In contrast to when the part was just molded (green) the brown part has a rougher surface and now feels metallic to the touch. (German & Bose, 1997, pp. 202-206) (Heaney, 2012, pp. 142-144)

2.3.2 Solvent debinding systems:

Wax based systems are perhaps the most common in commercial MIM production and is especially frequent in North America. As the name suggests this method is intended for feedstock consisting of a binder with a wax filler phase. This system is based around the waxes ability to dissolve easily. The procedure is quite simple and straightforward: the molded parts are immersed in a bath of solvent until the parts are completely primary binder free. The liquid solvent is usually kept at a temperature above the waxes melting temperature to accelerate the process. When the binder is completely removed the parts are taken out of the solvent and dried. (Heaney, 2012, pp. 137-138)

To increase automation and reduce adverse environmental impact modern wax based debinding systems are closed-loop systems where the whole process takes place in a closed chamber. Trichloroethylene, tetrachloroethylene and 1-propyl bromide are some typical solvents that have been used in wax based debinding systems. These chemicals are excellent solvents for organic materials such as waxes. However due to their halogenated chemistry they are considered a hazard to both the ozone layer as well as humans. The industry has almost entirely moved over to using less harmful organic solvents instead. Acetone, heptane and isopropyl alcohol are some of the more prevalent organic solvents used in modern wax based systems. (Heaney, 2012, pp. 137-138) (German & Bose, 1997, pp. 192-197)

A Water-soluble system works on the same principle as all solvent based systems, but the solvent in this case is just normal water. For this debinding method to work the primary binder needs to easily be soluble in water yet also function as a filler material. A typical primary binder for a water-based system is the polymer polyethylene glycol or PEG for short. (Heaney, 2012, pp. 141-142)

The work process of a water-soluble system is quite straightforward: the molded green parts are submerged into a water bath until the primary binder has solved away, after the debinding is complete the parts are moved into an air oven to dry. Since the process

doesn't involve any hazardous chemicals nor precarious elements its common practice to use "home-made" equipment although commercial automated systems are available. (Heaney, 2012, pp. 141-142) (German & Bose, 1997, pp. 192-197)

Supercritical solvent debinding is a highly technical and modern method of debinding. The idea is to use a supercritical fluid as solvent for removing the primary binder. Supercritical fluid is a state that a gas obtains when its subjected to high temperature and pressure at the correct ratio. Under normal conditions when a gas is pressurized it transitions into a liquid. But if the temperature is increased as well as the pressure the gas will become supercritical. Supercritical fluid displays properties of both a liquid and a gas. They can bear the properties of a liquid solvent yet are able to permeate into an objects as if a gas. This is a desired property since it will accelerate the debinding process. (German & Bose, 1997, pp. 197, 215)

The biggest downside of this method of debinding is the specialized equipment needed. The high pressure needed for the process also restricts the size of the debinding chamber and hence only small productions are feasible. (German & Bose, 1997, p. 198)

Common solvents used in supercritical debinding are carbon dioxide, propane and Freon, all of which are gases at room temperature. Carbon dioxide is the most frequently used due to its environmental friendly nature. After the molded parts have been subjected to the supercritical liquid carbon dioxide and the primary binder is extracted from the parts the debinding chamber is depressurized back to atmospheric pressure and the carbon dioxide evaporates away.

The soluble binder of supercritical debinding systems are usually made of wax. At the end of the debinding the wax can easily be cleaned from the debinding chamber. (German & Bose, 1997, pp. 197-198)

2.3.3 Catalytic debinding system

Catalytic debinding systems utilizes a combination of thermal and solvent debinding mechanisms. The system works by breaking down the polymer chains of the primary binder with the help of a catalyst. The catalyst is a chemical designed to lower the temperature at which a polymer is broken back into its monomer form. (Heaney, 2012, pp. 134, 139-140, 453) (German & Bose, 1997, pp. 197, 205, 216)

The temperature during the debinding process is lower than the softening temperature of the binder, for most polymers it's around 120°C. The amount of catalyst used is relatively low and is in the form of vapor inside the debinding chamber. Since the debinding occurs from contact between the binder and the catalyst-vapor the progression of debinding is moving uniformly from the outsides of the parts towards the core. This uniformed progression avoids structural strength differences within the parts, reducing the chance of distortion. Since the temperature on its own won't radically alter the binders structural integrity it can provide support longer into the debinding process. Another attractive attribute of not letting the debinding occur purely from thermal exposure is the lack of vapor-pocket buildup, vapor building up from inside of a part can lead to the part cracking. The catalytic debinding process resembles more a solvent debinding systems than a thermal one. The mechanism for removing the binder is based on catalyst-vapor entering the pore it has created and allowing the permeation of decompositions to exit through them. In contrast to other solvent systems the debinding-time and progression isn't restricted by the permeation of the solvent but rather the depolymerization rate. (Heaney, 2012, pp. 134, 139-140, 453) (German & Bose, 1997, pp. 197, 205, 216)

Catalytic debinding oven

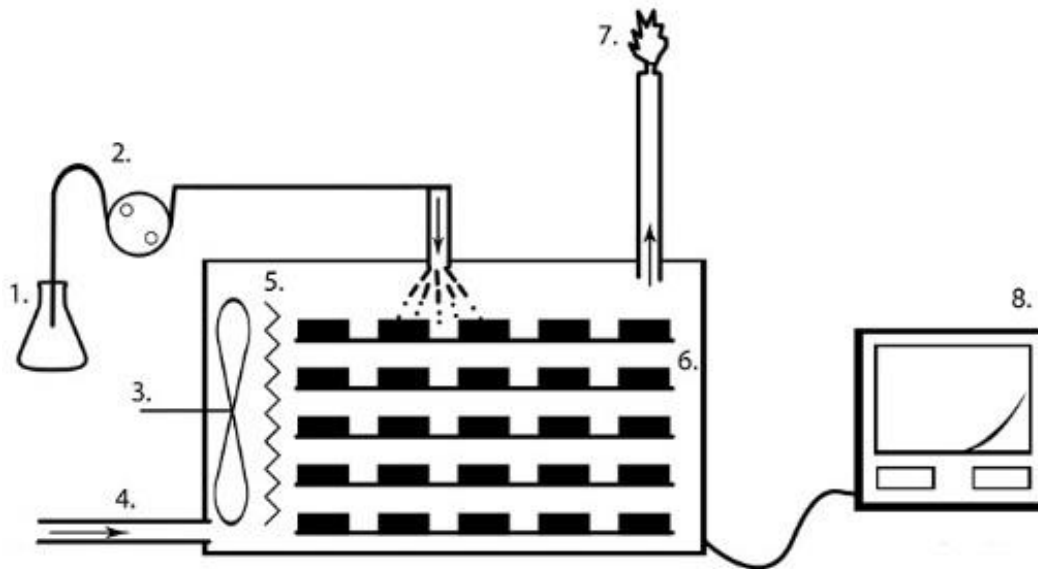


Figure 6. Catalytic debinding oven. (author,2016)

1. Acid: contains the acid catalyst. Nitric acid commonly used as the catalyst.
2. Pump: transports the acid catalyst from its container to the oven chamber. The pump also controls the catalyst concentration.
3. Fan: helps to spread the temperature uniformly throughout the chamber, helps the catalyst vapor spread evenly, creates an air flow that helps the vapor escape from the components pores.
4. Nitrogen inlet: lets in inert nitrogen gas replacing all the oxygen, eliminating the chance of explosions.
5. Heater: heats up the oven chamber.
6. Support trays: batch debinding ovens are usually equipped with a tray system for easier loading. The parts usually sit on a ceramic base which is custom design to provide better support.

7. Burner: the hazardous vapors formed from the debinding are lead out through a channel. At the top of the channel sits a burner with a pilot flame. The flame ignites the gas, burning it away.

8. Control unit: a computer containing the process parameters controls the gas valves, acid pump, fan and heater.

Catamold feedstock system

Catamold is a catalytic feedstock system produced by the chemical giant BASF. It is the most widely used feedstock system in MIM production. The Catamold feedstock is comprised of a metal powder and a binder just like most other systems. The binder consists of Polyoxymethylene (POM) as binder. (Heaney, 2012, pp. 134, 139-140, 453) (German & Bose, 1997, pp. 197, 205, 216)

Polyoxymethylene, acetal or polyformaldehyde is a thermoplastic with high strength and stiffness as well as good shape retaining properties. Although POM is a tough engineering plastic its polymer chain is vulnerable to degradation catalyzed by acids. This vulnerability is what the debinding and feedstock is designed around. Nitric acid is the catalyst Catamold systems use for the debinding. (PlasticsEurope, 2016) (BASF, 2009)

The debinding process of Catamold feedstock follows the same technique as most other catalytic system. The molded parts are introduced to a nitric acid atmosphere of a temperature between 100°C and 140°C. The combination of the nitric acid and the temperature depolymerizes the POM by breaking up the polymer-chain into its monomer. The monomer of POM is called formaldehyde and is in a gas state at temperatures above -19°C. As the primary binder turns into formaldehyde gas it evaporates out from the pores of the molded part, leaving only the polyethylene secondary binder and metal powder behind. The produced formaldehyde is a toxic and explosive gas, to avoid explosions the debinding chamber has inert nitrogen flowing through it during the debinding. To reduce the risk of toxic exposure the debinding ovens have an exhaust system through which the produced formaldehyde gas is cleanly burnt out. (Heaney, 2012, pp. 134, 139-140, 453) (German & Bose, 1997, pp. 197, 205, 216)

Due to the nature of the formed byproducts Catamold feedstock require ovens intended specifically for the system. The debinding-ovens tend to be batch ovens with at least some degree of computer automation. The automation controls parameters such as: chamber temperature, process times, catalyst feed rate, nitrogen flow and the exhaust burner. (BASF, 2009)

Once the POM primary binder has been removed the now “brown” part accommodates an interconnected pore system similar to an anthill. The Catamold system does not have two separate primary/secondary binder materials. (Heaney, 2012, pp. 134, 139-140, 453) (German & Bose, 1997, pp. 197, 205)

Depolymerization process

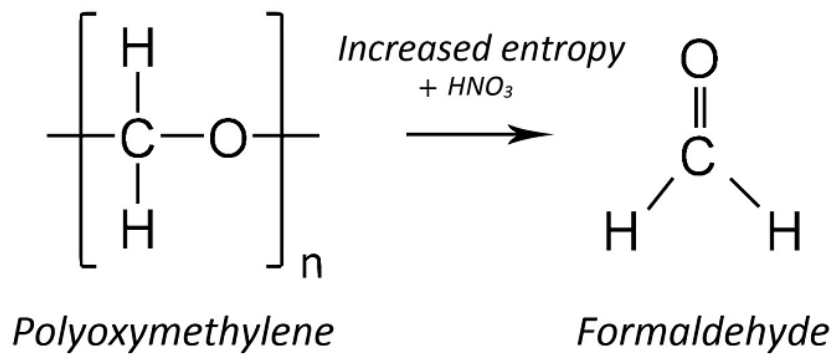


Figure 7. The solid POM polymer with a melting point of 180°C is turned into Formaldehyde monomer with boiling point of -19°C by a nitric acid catalyst. (author,2016)

Table 3. Example debinding systems (Heaney, 2012, p. 144)

Primary binder	Secondary binder	Primary debind method	Debind Temperature	Debind rate (mm/h)
Wax-based		Solvent		
Paraffin wax	Polypropylene	Heptane	50°C	1,5
Synthetic wax	Polypropylene	Perchloroethylene	70°C	2

Water-soluble

Polyethylenglycol	Polypropylene	Water	40°C	0,3
Polyethylenglycol	Polyoxymethylene	Water	60°C	0,5

Catamold

Polyoxymethylene	Polyethylene	Nitric acid	120°C	1,5
------------------	--------------	-------------	-------	-----

2.4 Secondary debinding

Once the primary binder has been removed from the components the following step is secondary debinding. The aim of the secondary debinding process is to remove the secondary “backbone” binder from the components. Following the primary debinding the parts have become brittle as they are only held together by the backbone polymer and friction between the powder particles. The secondary debinding is done by thermal degradation in conjunction with the sintering process.

The secondary debinding takes place in the sintering furnace during the early stages of sintering. Since the backbone polymer is the main thing keeping the parts intact the process requires a few things to occur. The secondary debinding can be viewed as a transition phase. In this phase the structural responsibilities of the binder is shifted over to the metal powder itself. This transition needs to happen with as little impact on the components as possible. As the binder is being removed the sintering process needs to set in motion in order for the parts not to crumble. (German & Bose, 1997, pp. 2012-206) (Heaney, 2012, pp. 144-145)

The temperature inside the sintering furnace is not held at a constant throughout the debinding/sintering process. The temperature is held at a few different steps with each their own goal. The debinding starts with a slow up-ramping of the temperature inside the chamber. Once the evaporation temperature of the primary-binder is achieved it's customary to hold for a while to ensure complete removal. As the temperature reaches the evaporation point of the secondary binder the temperature ramping is put on hold once again. As the secondary binder is subjected to the energy rich atmosphere its polymer chain stars breaking up, evaporating the material. As mentioned prior the primary debinding process has left the produced parts filled with an interconnected pore system. These tunnels help

the vapor to escape and prevents vapor-pockets from being formed. The temperature at this step is high enough for the powder particles to start producing diffusion-bonds between each other. By the time the binder has completely evaporated the diffusion bonds will be strong enough to support the component. (Heaney, 2012, pp. 144-145)

Following the removal of the secondary binder and the formation of diffusion-bonds the process continues by increasing the temperature inside the furnace once again. This time temperature will be high enough to start the sintering process.

Table 5. Secondary debinding (Heaney, 2012, p. 145)

Secondary debinding temperatures of common backbone polymers:

<u>Secondary binder</u>	<u>Thermal debinding temperature</u>
Polypropylene	450-500°C
Polyethylene	500-600°C
Polyacetal	300-450°C

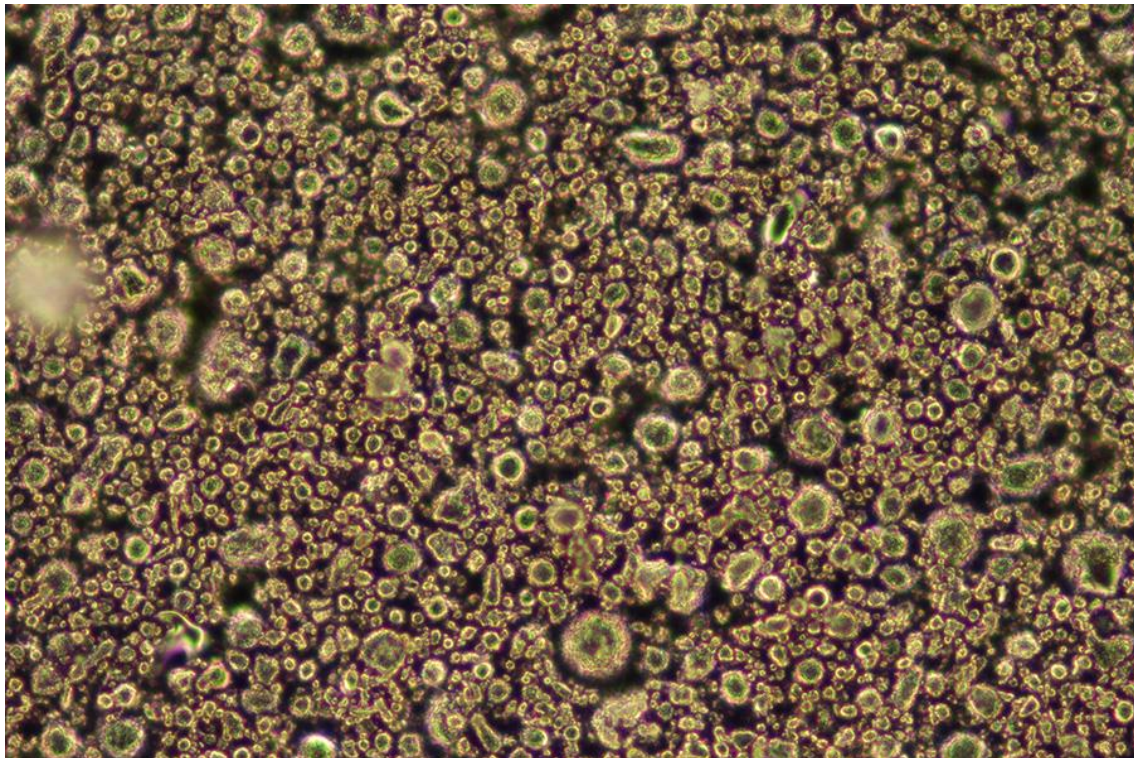


Figure 8. Closeup microscope image of the surface of a debound (green) MIM component. (Catamold 17-4ph) (author, 2016)

2.5 Sintering

Sintering is the process that gives MIM parts their strength. Up until now the parts have only been held together by particle friction and binder-material. The sintering process will transform the rigid powder compacts into proper solid metal objects. After sintering the produced parts will exhibit strength, hardness, ductility, wear resistance, conductivity and even visual resemblance similar to parts produced by conventional metalworking methods.

Sintering is a thermal treatment that makes particles bond together. By subjecting the debound components to high temperatures, bonds between the metal powder particles will start to form. As the process goes on the particles fuse tighter and tighter together eliminating the pores created in the primary debinding. At some point all the pores will be gone and all the particles will be interfused into one dense and solid mass. As a consequence the components will undergo rather drastic shrinkage. Although the sintering process will produce a solid metal object, the temperature during the process will never actually exceed the melting point of the material. The sintering temperature is a bit below that of the melting temperature. During sintering the compacts will be subjected to immense amounts of stress, but since the particle-bonds at the same time become stronger the impact won't be as substantial as during debinding. (German & Bose, 1997, pp. 219-222) (Heaney, 2012, pp. 147-148)

Although sintering plays a big role in the MIM process, it's not to be considered a forming method. Sintering is a primarily a thermal treatment method for densification. The mechanism which makes the particles fuse together is not exclusive to metals, ceramics and polymer can also be sintered. And the sintering process is part of all powder injection molding productions.

2.5.1 Sintering mechanisms

Nature strives for a state of minimal energy. This strive for reaching a lower energy state is also the driving force behind sintering. All surfaces have surface energy. By reducing the surface area the energy will also decrease. As two particles fuse together their sum

surface area will decrease, lowering the energy. The particle shape also helps drive the sintering process. The high stress held within a curved surface provides additional surface energy to spherical particles, therefore spherical particles are an advantage. (German & Bose, 1997, pp. 222-223)

Mass transport mechanisms

The natural vibration of atoms combined with nature's pursuit for minimal energy result in mechanisms which make the particles fuse together. These mechanisms have been broken down and isolated into six unique mass transport mechanisms. The mass transport mechanisms describe the movement patterns and flow of atoms that occurs between two particles. (Heaney, 2012, pp. 148-149) (German & Bose, 1997, pp. 222-226)

During the sintering process the initial inter-particle activity is achieved by surface transport mechanisms. As the name suggests the particle fusion starts from the surface of the particles. As the sintering progresses mechanisms inside the particles begin to activate as well. The mechanisms that occur from inside the particles are known as bulk transport mechanisms. (Heaney, 2012, pp. 148-149)

Progression of particle diffusion:

#1. Assuming that the feedstock has provided sufficient packing density of metal powder the particles will still make contact with each other following the debinding. This initial contact area functions as the foundation for fusion. (Heaney, 2012, pp. 148-149)

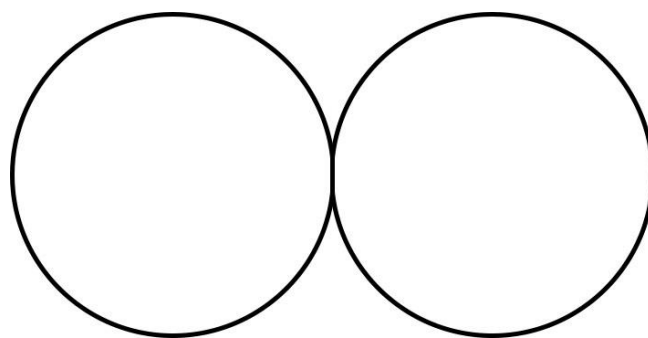


Figure 9. Initial particle contact (author, 2017)

#2. Neck formation and early neck growth: during the first stages of sintering atoms from the surface of the particles begin to move towards the contact area between the particles. Eventually enough atoms will have cumulated for a “bridge” to have formed between the particles. The atom at the contact area will start to form bonds with atoms from adjacent particles. During this necking process, the mass transport flow of atoms is only derived from the surface of the particles, hence the center of the particles will still have their original distance to each other. (Heaney, 2012, pp. 148-149)

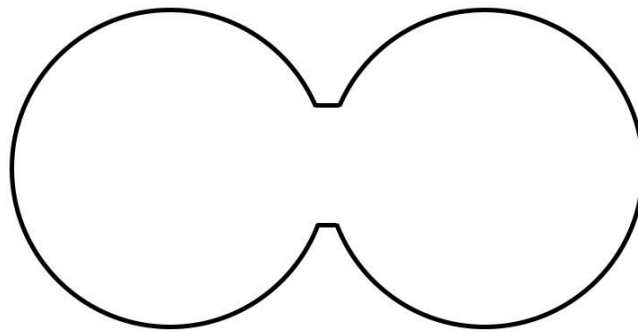


Figure 10. Necking (author, 2017)

The mechanisms that are behind the sintering at this stage are collectively known as Surface transport mechanisms. The surface transport mechanisms are devised by three main processes: evaporation/condensation, surface diffusion and volume diffusion. (Heaney, 2012, pp. 148-149)

#3. Neck growth: As the sintering has gone on for a while and “necks” between the particles have been formed the process will enter its later neck growth stage. At this point the surface transport mechanisms will no longer be the predominant driving force. The process is now primarily done by bulk transport mechanisms. The bulk transport mechanism moves atoms from inside the particles rather than the surface. The flow of atoms is still in the direction of the necks, making the necks grow yet more. (Heaney, 2012, pp. 148-149)

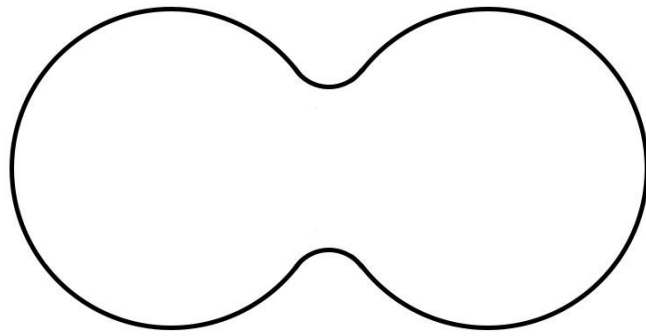


Figure 11. Neck growth (author,2017)

The bulk transport mechanisms that take place during the late stage neck growth are: plastic flow, viscous flow, grain boundary diffusion and volume diffusion. In contrast to the last stage the bulk transport mechanisms will cause the centers of the particles to move closer to each other. The activation energy for the bulk mechanisms are higher than for surface mechanisms, therefore initiate later into the sintering. In addition to the neck growth, pores will also get eliminated. As a consequence of the neck growth and pore elimination shrinkage will be observed. (Heaney, 2012, pp. 148-149)

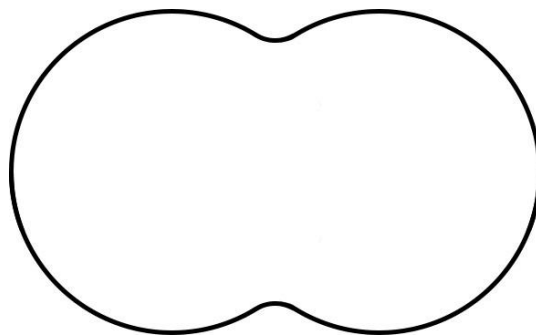


Figure 12. Late stage neck growth (author,2017)

The mentioned mass transport mechanisms are present to a varying degree, dependent on the material and the sintering conditions. The different mechanisms work with their own particular methodology although they are driven by more or less the same force. By understanding which mass transport mechanisms are affiliated with what material one can design an optimal sintering atmosphere for the material in question. (Heaney, 2012, pp. 148-149)

A rundown of the mass transport mechanism, where they are prevalent and their significance:

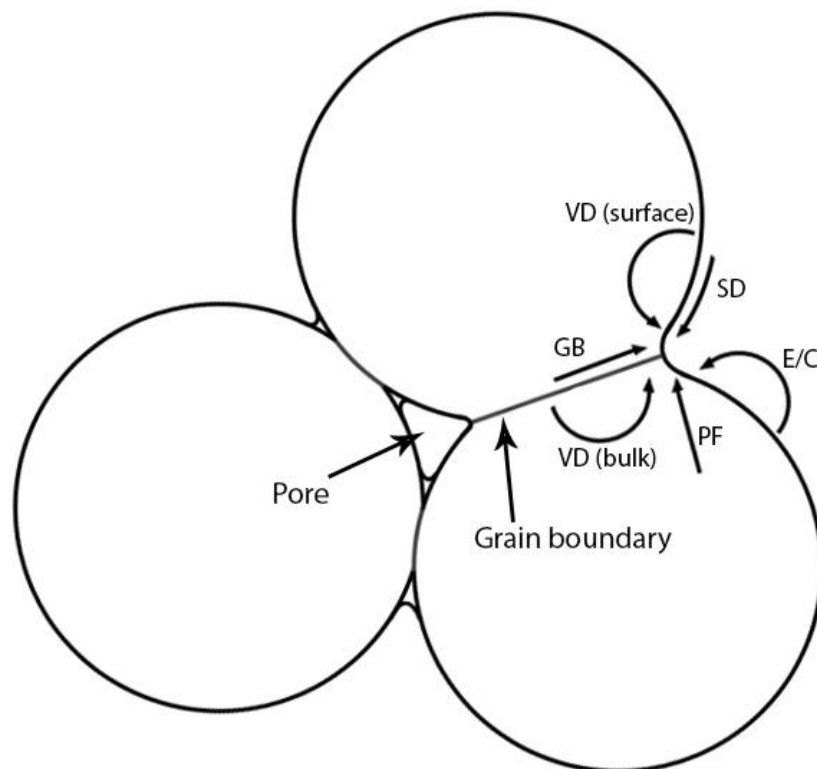


Figure 13. Three particles display of all the possible transport mechanisms (German & Bose, 1997, p. 225)

Evaporation and condensation (E/C): this mechanism is most prominent in volatile materials, meaning materials which will easily evaporate. The evaporation and condensation mechanisms impact is practically nonexistent in typical MIM production sintering. Water, sodium chloride and titanium oxide however do display this behavior, especially pressure atmospheres. High performance ceramics such as silicon nitrate also displays the

property of the evaporation and condensation mechanism and can therefore be sintered through an evaporation/condensation based process. (Heaney, 2012, p. 149)

Surface diffusion (SD): As the name suggests this mechanism takes place on the surface of the particles. This mechanism is of much significance in MIM sintering since it primarily applies to crystalline solids. In the strive for lower surface energy atoms on the surface of the particles bounce along imperfections and eventually end up at the contact area between two particles. The atoms will settle at the contact area, and as more and more atoms cumulate a neck between the particles will start to form. Surface diffusion occurs already at low temperatures which is highly beneficial. By forming necks between the particles early it will help keep the debound powder intact minimizing the chance of shape defects and collapse. (Heaney, 2012, p. 150)

Volume diffusion (VD): This mass transport mechanism applies to crystalline material. It works by using crystallographic defects as means of atom migration. Vacancies and dislocations in the crystalline structure motivates “free atoms” to move around. Volume diffusion occurs both on the surface of particles and from within. The mechanism contributes to neck formation as well as densification. (Ehrhart, 1991, p. 88) (Schilling, 1978, pp. 69-70) (Heaney, 2012, p. 150)

Plastic flow (PF): This form of mass transport occurs due to imperfections in the crystalline structure. Vacancies of atoms in the crystal lattice will allow atoms to “leap” over from adjacent rows filling the vacancy. This leap will result in the adjacent row now having a vacancy, which will set off a chain reaction of moving vacancy around. This vacancy-moving combined with the natural vibration of atoms help the particles fuse together. Plastic flow has shown to become the dominant mass transport mechanism in the presence of external pressure. (Heaney, 2012, p. 150)

Viscous flow (VF): this mass transport mechanism applies foremostly to amorphous materials, hence it plays little significance in metal sintering. Amorphous material lack any sort of structure in its atom arrangement. An amorphous material can be thought of as a slow flowing liquid even though it assumes the shape of a solid object. Subjected to a

temperature increase the viscosity of an amorphous material will decrease. As the viscosity decreases the material will start to fuse together. (Heaney, 2012, p. 149)

Grain boundary diffusion (GB): the lattice structure of a crystalline material isn't perfect throughout the whole material. The material is comprised of crystalline chunks known as grains. The area between these grains is a grain boundary. The grains won't fit perfectly together thus producing a channel. This channel allows atoms to move around with relatively low effort. The grain boundary accommodates the elimination of pores by helping atoms flow to them as well as providing passage to the surface for vacancies. The mechanism is somewhat similar to volume diffusion but due to the particulars of the grain boundary it has a lower activation energy. (Heaney, 2012, p. 150)

2.5.2 Shrinkage and densification

The process of sintering fuses the powder particles together and eliminates the pores created during debinding. Naturally these occurrences will have an impact on the dimensions of the parts. The process will result in significant dimensional shrinkage. This shrinkage is not to be considered as a problem or concern but rather a side effect of increased density, which ultimately is the end goal. The success of a sintering operation is often actually measured by the change in density. (Heaney, 2012, p. 158) (German & Bose, 1997, pp. 232-238)

After the debinding process and prior to being sintered the density of the MIM components are at around 60- 70% of theoretical. Once the components have undergone sintering their density will have increased to near 100%. (German & Bose, 1997, pp. 220-221)

Although the amount of shrinkage caused by sintering is substantial it occurs uniformly throughout the whole component. The sintering will retain the shape and proportions of the component and only makes it smaller. The sintering shrinkage must be taken into account already when planning the mold tool. MIM tools are designed to be oversized in order to compensate for the shrinkage that will take place during sintering. (Heaney, 2012,

p. 158) (German & Bose, 1997, pp. 232-238). The amount of shrinkage is feedstock specific, and is primarily determined by solids loading. With a typical feedstock consisting of around 60% solid powder and 40% binder by volume, shrinkage around 15% can be expected. A feedstock with lower powder volume for example 40%, shrinkage closer to 25% is more realistic. (German & Bose, 1997, pp. 234-235)

Feedstock vendors present the feedstock shrinkage on their material data sheet. But to acquire truly accurate and reliable shrinkage amounts is by empirically testing. The parameters used and the equipment itself will influence the shrinkage. Dimensional tolerances are extremely accurate and repeatable once a reliable shrinkage factor is known. Small screw threads and other low tolerance features are regularly produced with metal injection molding. (Heaney, 2012, p. 158) (German & Bose, 1997, pp. 232-238)

The dimensional impact from sintering can be foreseen by taking into account the shrinkage factor (Y). The shrinkage factor is based on the feedstock solids loading and the final density as well as theoretical density of the material. The shrinkage factor can be calculated using a simple formula: (German & Bose, 1997, p. 101)

Y =Shrinkage factor (%), Φ =feedstock solids loading, ρ =final density, ρ_t =theoretical density

$$Y = 1 - \left(\frac{\Phi}{\rho/\rho_t} \right)^{1/3}$$

(German & Bose, 1997, p. 101)

As mentioned sintering shrinkage is not seen as a problem nor an error, but rather a direct effect of density increase. This signifies the direct relation between amount of shrinkage and sintering performance. By simply measuring the dimensional change caused by sintering a good idea of the successfulness is gained. The final density can be calculated using the dimensional change and density of the debound component. (German & Bose, 1997, p. 221)

δL = change in length, L_o =original length, ρ_G =green density, ρ_S = sintered density.

$$\rho_S = \frac{\rho_G}{\left(1 - \frac{\delta L}{L_0}\right)^3}$$

(German & Bose, 1997, p. 221)

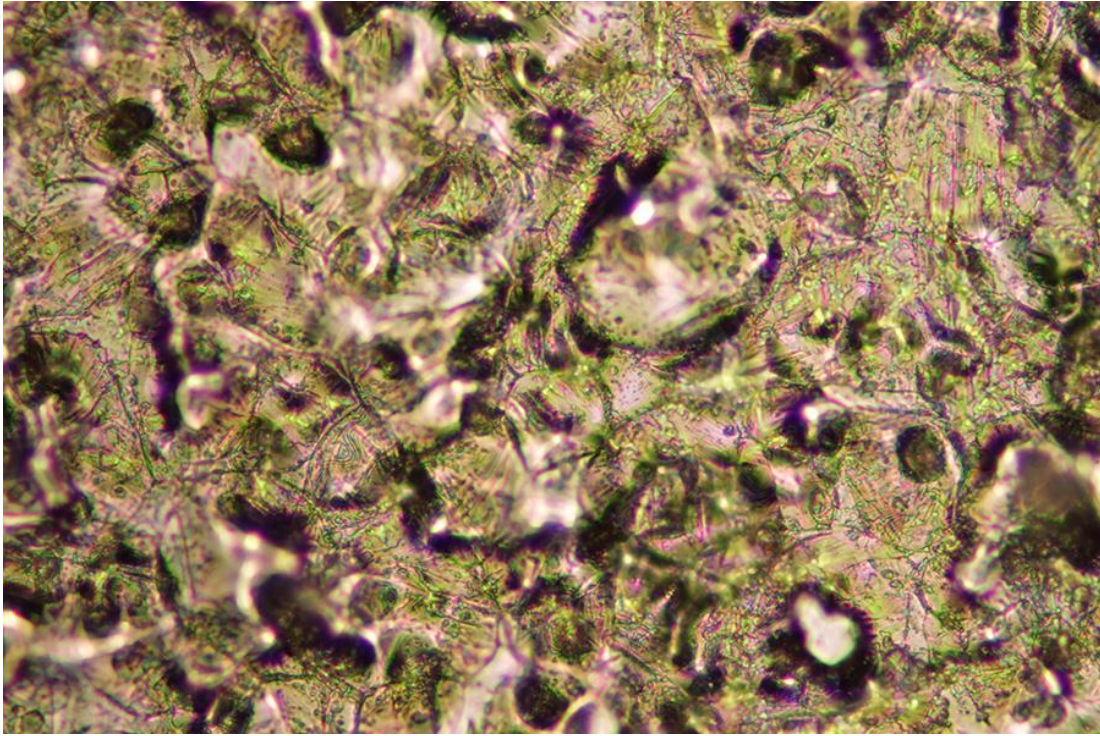


Figure 14. Closeup microscope image of the same surface as (fig.8) after sintering.

2.5.3 Sintering equipment and atmosphere

All metals are to some degree vulnerable to oxidation. When a metal object oxidizes its structural properties are affected adversely, hence oxidation should be avoided. MIM parts are very sensitive to oxidation during production, especially during sintering. Oxidation is a chemical reaction between the surface of a material and the oxygen in the air. The reaction takes place only at the exposed surface of an object which for a typical metallic object implies the outer surfaces. Following the debinding and prior to being sintered the MIM parts are essentially loosely packed powder structures filled with pores. The actual surface area is incredibly high as well as reaches through out the whole part all the way down into the core. The sintering process also requires high temperatures which accommodates and drives an oxidation reaction. (Heaney, 2012, pp. 157-158) (German & Bose, 1997, pp. 242-244)

In order to protect the MIM parts from oxidation during sintering the process is done in an oxygen free atmosphere. By flushing out all the oxygen from inside the components and the sintering furnaces oxidation can be avoided. The oxygen filled air is replaced with a less reactive gas such as nitrogen or hydrogen. The type of gas used depends on the metals reactivity. For highly reactive metals such as titanium which will even react with nitrogen the sintering is performed in a vacuum or halogen atmosphere. (Heaney, 2012, pp. 157-158) (German & Bose, 1997, pp. 242-244)

The gases used for the sintering atmosphere can chemically differ quite drastically from each other. They work by different principles and are metal specific. The most commonly used atmospheres are nitrogen, hydrogen, carbon gases, argon, air and vacuum. (Heaney, 2012, pp. 157-158) (German & Bose, 1997, pp. 242-244)

Nitrogen

Nitrogen is an inert gas, meaning it doesn't easily react chemically. The working principle of using nitrogen as sintering atmosphere is based around replacement of oxygen. The idea is to fill the furnace with inert nitrogen gas in order to avoid a redox reaction between the metal powder and the surrounding atmosphere. Although nitrogen doesn't react with the metal it won't completely prevent oxidation from occurring. The elements found in the metal will start oxidation-reduction reactions with each other. For example, the carbon in carbon-steel will be consumed in a reaction with the oxygen found in the metal powder. Carbon containing steel can be nevertheless sintered in a nitrogen atmosphere by adding a calculated surplus of carbon to the powder mix. Typical metals sintered in nitrogen atmosphere are carbon steels and high alloyed carbon containing steels. (Heaney, 2012, pp. 161-166) (German & Bose, 1997, pp. 242-248)

Hydrogen

Hydrogen is regularly used as a sintering atmosphere. The principle behind a hydrogen atmosphere follows nothing like that of nitrogen, and in fact is quite the opposite. While nitrogen aims to stop reactions from taking place hydrogen relies on reactions occurring. The idea is to use hydrogen as a reductant and letting it get oxidized instead of the metal

parts. By filling the sintering furnace with hydrogen gas any reaction between the different metal components are circumvented. (Heaney, 2012, pp. 157,161-166) (German & Bose, 1997, pp. 242-248)

For example, when sintering stainless steel a redox-reaction between the chromium and carbon would take place if hydrogen wasn't present. When sintering low carbon stainless steel and other low carbon alloys hydrogen is the preferred atmosphere. With that said stainless steel is regularly sintered commercially using nitrogen, resulting in decreased properties. (Heaney, 2012, pp. 161-166) (German & Bose, 1997, pp. 242-248)

Argon

The inert noble gas argon has a similar working principle as nitrogen, it relies on replacing oxygen. Argon is usually used for the most reactive metals. These metals contain alloys which won't get reduced by hydrogen and will react with nitrogen. Another reason for using argon instead of nitrogen is to avoid nitrogen pick up. Titanium and some super alloys require an argon atmosphere to be sintered properly. (Heaney, 2012, pp. 161-166) (German & Bose, 1997, pp. 242-248)

Air/Vacuum

Some rare metals do not require a special atmosphere and can be sintered with normal air in the furnace. This is primarily only the case for noble metals like gold and platinum.

Removing the atmosphere altogether is also a way to protect the metal from oxidation. By producing a vacuum inside the furnace the oxygen will get removed. This method is used for the most reactive metals. Vacuum and argon atmosphere share the same material base. Expensive vacuum pumps and some additional equipment are need for this method. A more significant downside with sintering in a vacuum is the lack of proper heat distribution. Without any atmosphere, heat is transferred purely by radiation, so the distance to heating elements etc. will have bigger impact. No convection or other flow is present

either to help spread the heat. (Heaney, 2012, pp. 157, 161-166) (German & Bose, 1997, pp. 242-248)

2.5.4 Equipment

The sintering process is done with one main piece of equipment, the sintering furnace. The furnaces job is to provide and contain the sintering atmosphere. By providing a stable and repeatable sintering environment a successful MIM production becomes likely. Heating, atmosphere control and timing are the main contributions of the furnace. (German & Bose, 1997, pp. 239-241)

The fundamental driving force behind sintering is heat. This is the primary objective of the furnace. Heating elements inside the furnace chamber supply heat for the process. The furnace walls provide an isolating enclosure, keeping the heat contained and constant. The heating elements are controlled by predetermined values fed by a computer. Temperature measurements are collected from inside the chamber and processed by the control computer which reacts accordingly. (German & Bose, 1997, pp. 239-241)

The atmospheres main purpose is to protect the metal parts from oxidation during sintering. Atmosphere control refers to the regulation of gases inside the furnace chamber. The furnace chamber separates the MIM parts from the outside air and acts as a container for a more suitable atmosphere. The furnace has gas lines connected to it. The furnace computer unit controls gas valves while measuring units inside the chamber collect data on oxygen, hydrogen, nitrogen, dew content, chamber pressure etc. The computer acts according to its cycle program and regulates based on gathered measurements. Aside from the furnace chamber itself another atmosphere related piece of equipment is a vacuum pump. It is essential for vacuum sintering but is also used as part of oxygen removal with other atmospheres. (German & Bose, 1997, pp. 240-242)

Sintering furnaces come in two different types; batch and continuous. Although both have the same work principal and objective they look very different to each other. A batch furnace is a sealed (usually cylindrical) chamber with a robust door at one end. And a

continuous furnace is a long construction with a conveyor running through it. A continuous furnace resembles more a production line than a single piece of equipment. The dividing factors for choosing type of furnace are: production size, metal reactivity and production flexibility. (German & Bose, 1997, p. 240)

A continuous furnace is constructed of a few different chambers with a conveyor mechanism between them. Typical chamber layout could be: pre-heating zone, sintering zone, cool down zone. The idea is to maintain constant automated production while minimizing down time. The foremost reason to use a continuous furnace is production size. If a large production of the same component and material is expected continuous is usually the way to go. The different chambers won't be perfectly sealed to the outside, so gas consumption is relatively high as well oxygen contamination. Vacuum atmosphere is seemingly also not possible. The large size of the furnace and heat-spill will also set certain requirements. (German & Bose, 1997, pp. 240-241) (Heaney, 2012, pp. 171-173)

The batch furnace consists of one single chamber. It is less automated than a continuous furnace and requires manual restocking and unloading after each batch. The batch furnace doesn't utilize any sort of conveyor mechanism but simply has a tray rack in the center of the chamber. The furnace chamber is completely airtight and sealed off by a vault style door. This allows for better atmosphere control, and there for less oxidation. Most batch furnaces are capable of withstanding high pressure as well as vacuum. This entails pressure assisted sintering and vacuum sintering are viable options. Although a reducing or inert gas is used, batch furnace cycles often include a vacuum stage to lower oxygen content. The batch furnace is best suited for a small production size of varying components. Each type of product and material can have its own tailor made sintering cycle, optimizing results and run time. (Heaney, 2012, pp. 174-175) (German & Bose, 1997, p. 239)

It's not only a suitable atmosphere that is required for successful sintering, the furnace material itself has to be compatible. Atmosphere, compact material, heating element material and furnace material all have to be a suitable combination. If the atmosphere reacts with the chamber wall or heating elements they might get damaged. A reaction between

the furnace and atmosphere can also produce chemistry which can affect the MIM components adversely. Randall M. and Animesh Bose (1997:242) present in the book *Injecting Molding of Metals and Ceramics* a PIM example where AlN sintered in nitrogen with molybdenum heating elements resulted in 98% density and only 78% when using graphite. The two materials regularly used for furnaces are graphite and refractory metal. In general, a refractory metal furnace tends to be more tolerant but also more expensive to acquire. A refractory batch furnace is therefore the most flexible and optimizable type of sintering furnace.

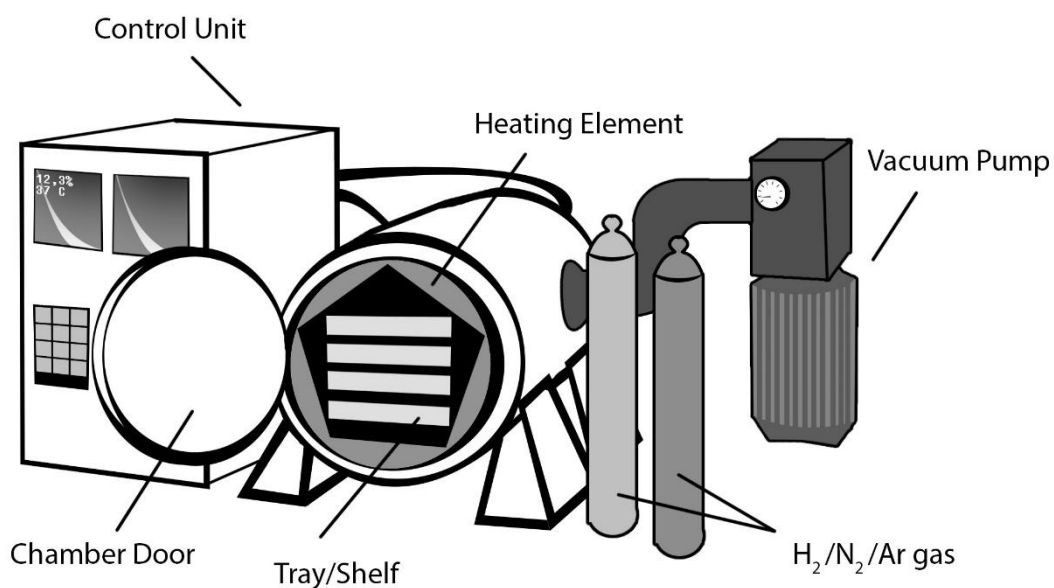


Figure 15. Batch sintering furnace (author,2017)

2.5.5 Cycle

The sintering cycle refers to the heating pattern a furnace follows during sintering. Temperature ramping, holds and cooldown are all part of a sintering cycle. The cycle essentially conveys a temperature over time relationship. It describes the rate of heating, cooling and duration of holds as well as the temperature. Each material has its own sintering cycle. The temperature holds in the sintering cycle have specific goals. For example the early holds can be to eliminate leftover binder-material or burnout of carbon residue. The later holds tend to correspond with some mass transport mechanism. The holds will also insure that the whole MIM part has reached the same temperature. (German & Bose, 1997, pp. 254-255)

Example of sinter heating cycle:

3°C/min	→	300°C	+	1h hold
3°C/min	→	500°C	+	1h hold
3°C/min	→	600°C	+	1h hold
5°C/min	→	700°C	+	2h hold
5°C/min	→	800°C	+	2h hold
5°C/min	→	900°C	+	2h hold
5°C/min	→	1050°C	+	1h hold

(Heaney, 2012, p. 456)

The cycle is primarily based on the type of material that's being sintered. Although to a lesser extent other factors such as particle size, part size and part geometry will influence sintering behavior as well. To truly have an optimized production all factors need to be taken into consideration. By using personalized cycles for each product, the best properties and shortest run time can be achieved. (German & Bose, 1997, pp. 254-255)

Empirically testing out different cycle adjustments is the foremost way to find optimal parameters. Although the cycle usually refers to temperature and timing, atmosphere control may also be included. The cycle can for example start with producing a vacuum inside the furnace chamber. The concentration of protective gas and pressure inside the furnace is included in the cycle instructions. The cooldown and ventilation at the end are also all part of the sintering cycle. (German & Bose, 1997, pp. 254-255)

The whole sintering process is automated and controlled by a computer unit. The computer follows a set of instructions fed by the operator. The instructions often are in the form of a table not much different from a Microsoft Excel sheet. The table consists of a parameter and its corresponding value and a time period. Measuring units inside the furnace provides info to the computer which in order can respond by sending more current to heating elements or by opening gas valves. (German & Bose, 1997, pp. 254-255)

3 PROBLEMS AND OPTIMIZATION

The whole metal injection molding process is long and consists of several different elements. There's a lot which must go right and little that can go wrong in order to produce good parts. The extensiveness of the process and the little room for error makes problems likely. It's crucial to be able to identify the source of the problem and know how to fix/circumvent it. By ironing out the causes of problems the production should run smoothly and produce consistent identical parts. One shouldn't just settle for a working MIM production but try to get it to be as optimal as possible. An optimal MIM process could for example entail: optimal product properties, consistency, production time or a combination/compromise of them all.

Whether searching for usable MIM parameters or improving on old ones it's important to inspect the result after each step. Errors caused during molding may only show up later at sintering, and it gets harder to pin point the cause the further the process moves along. A problem can be categorized either by its cause or how it manifests itself. (Heaney, 2012, p. 235)

Problems associated with MIM and the optimization of the process are closely linked together. The best way to find better process parameters is by empirically testing out adjustments. As one starts fiddling around with the parameters of a working process problems are likely to arise. The following scenarios could for example occur:

Cut down on debinding time → leaves binder behind → cause compacts to crack during sintering

Cut down on debinding time + increase temperature → remove all binder + causing heat distortion to compacts during debinding

As some aspects improve others might suffer, for example if the mechanical properties improve the success rate might suffer. In other words there's a difference between optimization of product properties and optimization of the process. Ultimately it's a combination of both aspects, where the product quality is the priority. Optimizing the process essentially entails not wasting time and resources as well as not compromising quality.

3.1 Molding problems

Molding is the first step of the MIM process and problems can occur here already. Some of these problems take the form of typical molding defect while others can appear at a later stage in the process or only at final quality control. The molded components will serve as the foundation for the remaining steps, and just like a house with a poorly made foundation the whole thing might come crumbling down. The sooner defects are detected the less parameters are involved making it easier to fix the problem. There are of course also more immediate defects which can be observed prior to debinding and sintering. (Heaney, 2012, p. 235)

3.1.1 Molding defects

MIM molding defects are largely the same as the ones encountered in traditional plastic injection molding. The use of improper parameters such as temperature, pressure and shot size are all common causes of problems in traditional injection molding and they apply to MIM as well. The mold tool itself can cause defects by consisting of complex geometry or lacking adequate air channels. Most minor defect can be resolved by altering the temperature-time-pressure relationship. (Heaney, 2012, pp. 129, 238)

Incomplete fill

As the name suggest this defect is related to the mold cavity not filling up completely and there by a defected part is produced. This defect is caused by the molten feedstock not reaching everywhere inside the cavity. The reason for this could be that the gate gets solidified before the cavity is completely filled or that the shot size is simply too small. Increasing the shot size, packing pressure, injection speed or molt temperature can fix the incomplete fill. Improving the molds air channels or increasing mold temperature might

also be solutions. If the problems stem from material feeding, the issue might lie in the with feedstock or recovery backpressure. (German & Bose, 1997, pp. 155, 162-164) (Heaney, 2012, pp. 130, 238)

Flash

Flash is the buildup of excess material around the molded part. This is caused by material leaking in between the two mold halves. This usually happen when the pressure inside the mold is greater than the force keeping the mold together. By increasing the clamping force or decreasing injection speed and pressure, the mold should stay sealed better. The tool quality will also influence flashing. An uneven or dirty mold surface prevents a proper seal from forming, so using high quality clean tools are necessary. Using higher viscosity feedstock can also help flash from forming. Flash can also be cut off or ground away, this should be done prior to debinding. (Heaney, 2012, pp. 130, 242)

Voids

The formation of voids in the molded parts is usually caused by gas being trapped in the cavity. The source of this gas can be the binder-material itself or just air trapped on mold closing. Possible remedies could be: increasing hold pressure or decreasing injection speed. Both work by aiding the ventilation of trapped gas. Increasing the mold temperature might also help. If tool redesign is an option moving the gates or increasing their size can also remove this defect. (Heaney, 2012, pp. 131, 242)

Weld lines

This defect occurs when molten feedstock inside the mold is split up into different directions and comes together thereafter. At the point where the molten feedstock fronts collide a welding line defect can form. If the fronts aren't hot and runny enough the material won't bond back together properly. This will create a weak point in the molded part. The feedstock being too cold is essentially the cause of this defect. By making sure the feedstock is hot enough at the point when the fronts collide this defect can be avoided. Increasing mold and/or feedstock temperature can be a solution. Another approach is to decrease the fill time and thereby avert the feedstock from cooling down and becoming viscous. This

can be done by increasing injection speed or enlarging the gates. (Heaney, 2012, pp. 131, 240-243)

Sinks

Sinks are defects that taken the shape of indents on a molded part. They are not caused by incomplete fill but uneven shrinkage during cooling. By increasing the packing pressure and/ or packing time the component is more likely to keep its correct shape while solidifying. Reducing mold temperature can also provide for a more uniformed shrinkage. (Heaney, 2012, pp. 131, 142)

Cracking

If the parts come out of the mold as cracked pieces it's most likely an ejection issue. MIM parts are more brittle than pure polymer ones so the ejection is more critical. Increasing the mold heating or using less packing pressure will help with releasing the parts from the mold, lowering stress from ejector pins. The brittle nature and heavy weight of the metallic components will put them at risk of cracking after injection too. As the mold opens and the parts are ejected they should land as gently as possible. This might require construction of additional ramps and cushioning for the part collector system. (Heaney, 2012, p. 130)

Warping

Warping in the molded part is either caused by an ejection issue or by uneven pressure in the component. Since the feedstock is partially metal powder the parts will accumulate heat well, which is problematic during ejection. Not only can ejection cause the hot molded part to crack but also warp. By increasing cool time or reducing mold heating, warping should stop. A pressure gradient within the part might also cause the part to warp, this can be corrected by decreasing the hold pressure. (Heaney, 2012, p. 131)

Powder/Binder separation

This defect entails that the powder and binder of the feedstock get separated from each other during injection. The low viscosity binder and the heavy powder particles behave differently when passing a narrow point in the mold. The binder flows easily through while the powder moves slowly. This will cause a higher concentration of binder to collect at far corners of the component while the powder will collect near gates and narrow point. This is primarily a cosmetic issue, but in extreme cases it can influence the final strength of the MIM part. By reducing the injection speed the feedstock components won't separate as easily. (Heaney, 2012, p. 239)

3.2 Debinding problems

It's during debinding that the parts experience the largest amount of stress of the MIM process. Removing the binder will expose any molding defect by amplifying them, potentially removing all structural integrity, resulting in collapse. The heavy weight of the metal powder makes the components prone to deformation, but by having a well-molded part and using the correct parameters the debound part should come out as intended. Towards the end of the debinding cycle the pre-sintering starts to set in, strengthening the brittle compacts. Debinding is a long and time-consuming process, and it should be done slowly to avoid defects. Although the process should progress slowly the long duration will also signify a room for improvements, speeding up the process by just a few percent a lot of time can be saved. (Heaney, 2012, pp. 243-244) (German & Bose, 1997, p. 210)

3.2.1 Debinding defects

Defects that occur during debinding are caused by the stresses involved. It's not only gravity and the decreasing amount of binder that makes the components vulnerable to defects during debinding. The binder extraction itself causes stress inside the parts. And the harsh energy-rich atmosphere accommodates for uneven thermal expansion and other problems. In general the most common debinding defects are cracking and distortion. The type of defect depends on the debinding method. Thermal and solvent debinding tend to have different defects. A common debinding method is to utilize both thermal and solvent debinding in a two-stage debinding process. With a two-stage debinding process

the problems of both methods are faced. Debinding is in the middle of the molding-debinding-sintering chain and therefore any minor defect can cause problems during the following sintering process. Defects will also of course have been inherited from previous molding step and may only show up now. (Heaney, 2012, pp. 243-244) (German & Bose, 1997, p. 210)

Thermal debinding

The number one cause of thermal debinding defect is too rapid heating. The process needs to be very slow in order to avoid defects, even several days long debinding times are not unheard of. Thermal debinding works by degradation of binder components into gas. If the gas buildup is big and proper pores channels have not formed yet pressure inside the parts will create stress. The pressure buildup can lead to cracks and blisters. By decreasing the heating rate the gas buildup won't be as rapid, solving the problem. If the parts end up distorted or bent the solution is likely to be the same. By utilizing some sort of ceramic support for the parts, risk of distortions occurring is reduced. A good air flow around the parts will help remove the formed gas faster thereby reducing the pressure inside. (Heaney, 2012, pp. 245-248, 259) (German & Bose, 1997, p. 211)

Solvent debinding

Most defects that occur in solvent debinding aren't directly related to the debinding parameters. Feedstock constituency and molding are primarily the cause of the defects. The binder components may not be perfectly compatible with each other or the solvent. If some binder component gets extracted at an ill-fitting time the part can lose its structural integrity. An improper combination of backbone polymer and solvent is also a cause of defects. Solvent entering the binder causes the polymer to swell, the larger the expansion the greater the stress. A large enough expansion may lead to permanent defects. The most commonly encountered defects in solvent debinding are slumping and cracking. It's likely that these defects can't be mended by adjusting process parameters since they are likely caused by binder chemistry or high molding pressure. Adjustments to the molding cycle or switching feedstock/solvent will sort these problems. (Heaney, 2012, pp. 244-245, 249) (German & Bose, 1997, p. 211)

Catalytic debinding

The catalytic debinding method is more advanced and more gentle than thermal or solvent debinding. Catalytic debinding has a higher tolerance for the parameters and is less likely to produce defects. The catalytic process is a combination of both thermal and solvent debinding. But it doesn't directly suffer from the problems of each. In contrast to thermal debinding the catalytic method creates gas from the surface inwards, meaning trapped gas won't be a problem. The temperature during the process isn't very high as well so thermal distortion is not likely. The swelling problem will also not be as prominent in catalytic debinding since all the action takes place on the surface. The problems that do occur are basically a combination of both thermal and solvent although they are more rare. (Heaney, 2012, p. 244)

3.3 Sintering problems

The sintering process is the last step in the MIM cycle. It's now that the components get their final strength and the results are revealed. Since this is the final process this is also where minor defects introduced during previous processes will show up. The sintering working principle is based on densification and shrinkage. Accordingly the most common sintering defects will also be related to dimensional control. Another typical cause of sintering problems is the atmosphere. For a successful sintering atmosphere control is essential. This requires the gas chemistry and concentration to be correct. In addition to dimensional and atmosphere related defects the familiar molding and debinding defects: cracking, distortion and blisters etc. are all common during sintering as well. (German & Bose, 1997, pp. 249-250)

3.3.1 Sintering defects

Dimensional defects

If the sintered part comes out as too large or small it's usually viewed as a tool design error. By not foreseeing exactly how much larger the mold should be designed the produced part will have incorrect dimensions. The size can be adjusted to some degree by increasing or reducing the sintering temperature. If the part doesn't shrink uniformly throughout, it's likely to be caused by uneven heat exposure. One area of a part might be

in the “shadow” of another part. This can be resolved by simply moving the piece. Increasing the sintering time can also be a cure. This allows the temperature to spread evenly throughout the part. If the shrinkage isn't uniformed in height and width it's probably due to gravity. By adding extra support, this problem can be avoided. (German & Bose, 1997, pp. 249-252)

Atmosphere related defects

Since the MIM parts are so chemically vulnerable during sintering, it important to use a suitable atmosphere. A lot of sintering defects stem from having improper chemical reactions between the metal parts and the atmosphere. The redox reaction that will take place should be accounted for. If the concentrations aren't correct defects will occur. If a stainless steel loses too much of its carbon it indicates that a reaction with oxygen has taken place. This can be corrected by either using a surplus of carbon or decreasing the oxygen in the atmosphere. Or if the oxidation isn't uniformed the gas flow has to be increased or the parts moved around. If the parts get oxidized, oxygen might be leaking into the sintering furnace or the atmosphere doesn't contain enough reducing agent. (German & Bose, 1997, pp. 241, 245) (Heaney, 2012, pp. 262-263)

Low density

If the density after sintering isn't as high as desired, adjusting the heating rate can alter the densification. By increasing the peak sintering temperature there should also an increase of density, or alternatively increasing the length of the peak temperatures duration. If a sintered part has variations in density it's likely caused by uneven heating of the furnace. (German & Bose, 1997, p. 252) (Heaney, 2012, p. 262)

Low properties

Low properties refers primarily to poor mechanical strength of the produced parts. This could be caused by incomplete densification. Increasing the sintering temperature should remedy this. Conversely too intense heating produces poor properties too due to grain growth. Grain segregation during cooling can also occur, this has a negative impact on the properties. Adjustments to the cooling parameters should fix it. Finding a suitable sintering cycle will help with most strength related defects. (German & Bose, 1997, pp. 252, 254)

Large pores

If the sintered parts have large pores or hole in them it could either be due to a feedstock mixing problem or oversintering. If oversintering is the cause it's related to grain growth. Reducing the heating rate should ease grain growth. (German & Bose, 1997, p. 252)

Cracking

Too rapid heating is a common cause of crack formation. By decreasing the heating rate at early sintering the components will have the time to build up strength before they get subjected to great stress. The formation of oxides can cause the MIM part to crack as well. Using a suitable atmosphere will minimize oxidation. (Heaney, 2012, p. 259) (German & Bose, 1997, p. 251)

It's likely also that the cracks that appear following sintering may have been caused already during molding, the sintering stress just makes the defects more pronounced.

Distortion

Various reasons can cause the parts to get distorted during sintering. A common cause is related to gravity and the component support. By orientating the parts in a way that provides the most amount of support for the heaviest areas the risk of distortion is lower. A product-specific support tray minimizes distortion the most. A support that follows the part shape grants support for the whole part. Although a large support surface is generally a good thing it can be the cause of distortion as well. The shrinkage that occurs during sintering means that the part will have to "move" in relation to the surface. If the friction between the support surface and part surface is too large it will cause distortion to the component shape. This can be fixed by using a smoother support surface. (German & Bose, 1997, p. 251)

If the sintering cycle goes through too high temperatures and too much of the metal enters a liquid phase the structural integrity of the part is lost. This means distortion is very likely. An uneven heating can cause distortion as well. By making sure that temperature is as uniformed as possible thermal gradients are less likely.

3.4 Monitoring

Monitoring refers to the practice of analyzing the MIM parts progression and final result. The primary aim is to observe and diagnose defects. It also provides a numerical value on the success rate of the MIM process. Since metal injection molding is such a long process it's important to keep a close watch on how the process progresses. After each step a set of analysis methods can be applied to evaluate the success of the previous process. If a problem or defects arises a good monitoring practice will provide essential information resulting in a quick fix. Constant monitoring is crucial when changes to process parameters are being done. In other words optimization goes hand in hand with good monitoring practice. Analysis methods are often labor intensive as well as time consuming this can make them quite costly. Once a good and reliable process setup is found monitoring becomes less important. This is especially the case for simple products with low consequences from failure.

Although the end result is what actually only matters each individual process has its own set of criteria and analysis methods. This helps identify the source of a defect rather than just a defected end-product. The analysis techniques aim to answer two main questions: how well did the process accomplish its task and did any defects get formed. For example, debinding analysis seeks to answer the amount of binder removed as well as the formation of cracks and weld lines. If both are within their tolerances it can be assumed that the debinding process will not be the reason for subsequent problems or poor end results. (Heaney, 2012, pp. 254, 258)

3.4.1 Molding

The molding process objective is to produce the shape and foundation for the end-product. The degree of success is measured by how well the molded part has accomplished the desired shape and the dimensional accuracy. By visually looking at the molded part and measuring its dimensions one can conclude how well the molding went. A better and more effective way is by simply measure the parts mass with a scale. A variation in mass will indicate an abnormality in volume. If the volume is incorrect it is caused by a molding

defect or a dimensional error. For a more precise measure of density a pycnometer instrument can be used. (Heaney, 2012, pp. 260-263, 259)

The injection molding machine itself functions as a type of monitoring instrument. The machine will monitor parameter values and changes throughout its cycles. If a value doesn't stay within its given tolerance the parts will be disqualified. Typical parameters that should be paid extra attention to are variations in shot size, screw torque and switch-over pressure. (Heaney, 2012, pp. 260-263, 259)

A visual inspection is sufficient enough to detect most molding defects. Cracking, blisters, weld lines, powder/binder separation and voids all have a visual element to them which can easily be spotted if they are severe enough. If the products are intended for high consequence use such as medical or aerospace x-ray analysis could be implemented. (Heaney, 2012, pp. 261-263, 259, 260)

3.4.2 Debinding

The debinding process is designed to remove the binder from the molded part. So logically the tests associated with debinding are aimed to measure the extent to which binder material has been removed. The amount of binder removed can be measured using a scale. By measuring the weight loss caused during debinding and comparing it with the feedstock powder/binder weight ratio the amount of binder remaining can be calculated.

The most common debinding defects are blisters and cracks. A visual inspection is sufficient method for detecting them. Thermal debinding as well as a pre-sintering step will both cause some shrinkage to occur. To make sure that the magnitude of the shrinkage is according to what's intended a linear measurement can be done. A linear measurement can also determine if any distortion or warping has happened. (Heaney, 2012, pp. 262, 259, 260)

3.4.3 Sintering

The goal of the sintering process is to give the MIM parts their strength. It accomplishes this by removing pores and fusing powder particles together. This will result in an increased density. By measuring the density of the part and comparing it with the theoretical density of the material the extent of the sintering can be determined. A pycnometric test is a common way to study the density. A visual inspection of the parts will expose the common defects such as voids and cracks. By looking at the microstructure one can determine if the parts are getting over- or undersintered. A microscope will show how well the grains sit together as well as any grain boundary growth. Pores, voids and other small defects can also be detected using a microscope.

Although the shrinkage associated with sintering isn't a defect in itself, improper dimensions are. Sintering is the last step in the process and it provides the final dimensions to the part. A linear measurement of critical dimensions can be done to make sure the parts satisfy their required tolerances. (Heaney, 2012, pp. 262-263,259,260)

3.4.4 Inspection / Final testing

A final inspection of the end result is common practice. The extent of the inspection depends on how critical the parts end use is. For high consequence parts, analysis methods such as x-ray can be used to make sure they are defect free. For most components a quick visual inspection and a linear measurement of the part size is sufficient. Application specific test such as surface finish or corrosion resistance are also done as part of the final inspection.

Destructive tests are the best way to makes sure the produced parts satisfy the mechanical properties required. Tensile testing and three-point bending are good example of this type of testing. These tests won't identify defect, but will answer the ultimate and most important question: are the produced part strong enough. By pushing the parts beyond their limit we will learn how strong they truly are. The strength of a MIM part comes from how well it has been produced, in other words the weakness of a MIM part is the sum of all the defects combined. Tensile testing and three-point bending tests are highly valuable

whenever parameters have been changed or a new part is put into production. (Heaney, 2012, pp. 259, 262-263)

4 OPTIMIZATION OF PARAMETERS

With a grasp on the MIM defects and monitoring methods the next step is optimization. Optimization of the MIM process means finding the most efficient parameters. The premier goal is usually to minimize the process run-time while not compromising quality. This can be accomplished by cutting away excessive run-times or by increasing the intensity of the process. Another optimization goal could be merely focused on improving part properties.

Finding the optimal process parameters is a highly empirical task. Knowledge about the feedstock characteristics and behavior will only provide basic guidance for setting up a debinding process. Same goes for singeing and the mass transport mechanisms involved. Since an empirical trial and error approach is how the optimization needs to be done a good understanding of defects and monitoring techniques outweigh the knowledge of the exact mechanics behind the process.

The best way to approach the optimization is by focusing on one process step at a time. If changes are done to both singeing and the debinding process at the same it's harder to evaluate the effect of the adjustment. Also, if parameters of the debinding process are changed the monitoring should be focused on debinding related defects. By focusing on a low number of parameters at a time the result are going to be more valuable and the variables less overwhelming.

The best indicator for how the optimization is progressing are defects. By analyzing defects when they appear and measuring mechanical strength of the finished parts an indication of which direction the process is moving can be concluded. Hence the great importance of monitoring techniques and ability to recognize defects and their potential causes.

4.1.1 Molding

Optimization of the molding parameters usually aims to improve properties of end product or to make the injection molding as reliable and defect free as possible. Cycle-time reduction is usually not a high priority since it's quite insignificant compared to the overall duration of the whole MIM process. By finding a reliable set of parameters which produce defect free part a more significant time-save is achieved. It is critical to have a molding setup that's defect free since it's the first step in the MIM process and everything will be built on this foundation. The same goes for part properties, the molding step has surprisingly large influence on the end products mechanical properties.

An example that shows how properties may be optimized by molding parameters is the research paper: Optimization of Injection Moulding Process Parameters in MIM for Impact Toughness of Sintered Parts by P. Pachauri and Md. Hamiuddin. The paper highlights how the impact strength of a MIM part can be improved just by optimizing molding parameters. The research reveals that the biggest factors on impact strength are injection pressure, mold temperature and powder loading.

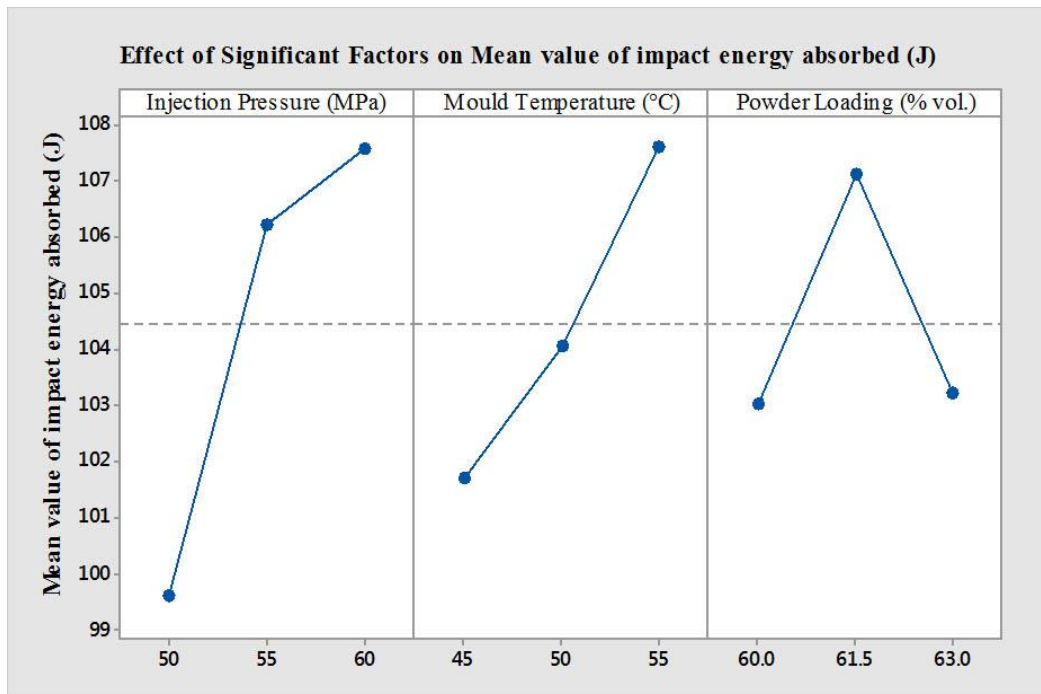


Figure 16. Mean impact energy absorbed (Pachauri & Hamiuddin, 2015)

Each product will have its own optimal set of molding parameters. Part size, geometry and feedstock are only some of the factors that will have influence on parameters. The parameters found in the research paper might also only specifically improve impact strength and not the other mechanical properties.

4.1.2 Debinding

Debinding is a long process that subjects the molded parts to high internal stress. The main focus of an optimization would be on minimizing occurrence of defects and cutting down process duration. As there are many different debinding methods the parameters involved can be vastly different. One thing they all have in common is the long run-time. They're all also prone to producing defects because of the high stress involved with extracting binder. The high likelihood of defects becomes more evident as parameters are being readjusted and optimized. A common contradiction that occurs when optimizing any debinding method is the conflict between minimizing process duration vs. minimizing defects. In a sense, each goal is a cause against the other.

In the case of minimizing process duration, the first step is to cut off any excess run-time, and the second step is to increase debinding intensity and thereby cutting down time even further. An increase in intensity will create greater stress resulting in increased risk of defects.

cut down time → binder left → increase intensity

increase intensity → cracking, distortion, other defects

The idea is to find an optimal “middle ground” which doesn't produce defects while getting rid of the binder in the shortest time possible. It's not just as simple as increasing intensity until defects start to appear. The optimal intensity of a debinding cycle won't follow a linear increase path. Factors such as binder constituency and powder characteristics will shape the optimal path into a specific way. A cycle with a customized set of parameters will be optimal. The optimal heating pattern for thermal debinding avoids all the problem areas while still cuts as close as possible to save time. (German & Bose, 1997, pp. 198-200)

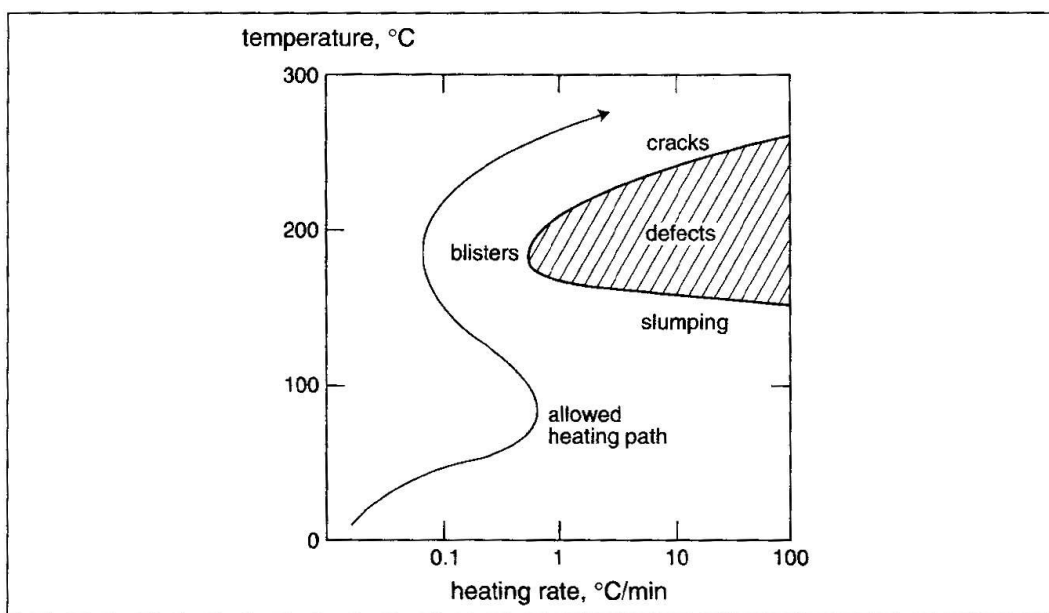


Figure 17. Thermal debinding heating path. (German & Bose, 1997, p. 199)

The main influencer on the debinding process is the exposure. It's made up of the parameters intensity and duration. Intensity entails temperature or solvent concentration, depending on the debinding method. The best way to find out the optimal parameters for a debinding process is through empirical test and monitoring. If a short process duration is a low priority the best approach is to use an excessively long debinding time since over-debinding doesn't produce defects.

4.1.3 Sintering

The sintering process has perhaps the biggest impact on the products properties after feed-stock characteristics. The goal of a sintering process optimization is usually to achieve the best properties possible in shortest amount of time. Of the two properties are the first priority. Since the sintering procedure is so long there's going to be potential for cutting off excess run-time. Just as with debinding the process duration can be sped up by increasing intensity.

The goal with sintering is to increase the components density. The higher the density is the stronger the properties of the sintered part will be. The following graph shows the relation between density, time and temperature. An increased temperature will allow for

better density. The restricting factor on the temperature are defects. If the heating rate is too rapid defects such as cracking and distribution are likely to occur. If the peak temperature isn't high enough the metal particles won't fuse together properly no matter how long the sintering goes on, leaving the density too low resulting in poor properties.

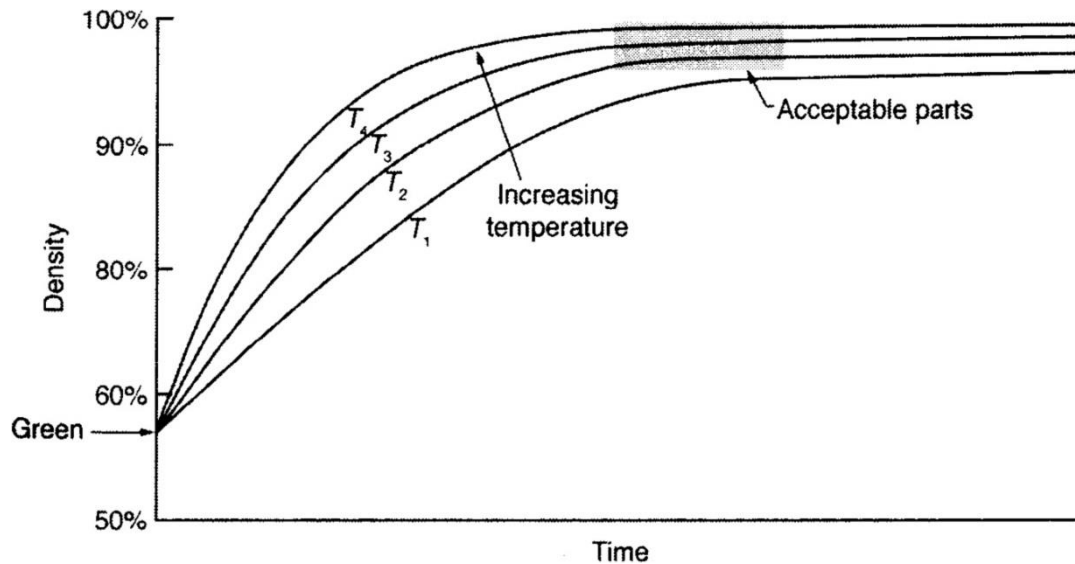


Figure 18. Density over sintering time. (Heaney, 2012, p. 159)

Once the parts have reached their peak density for the given temperature the densification stops and any further sintering won't increase the density. For an optimal process duration, this is when the sintering should stop and the cooldown stage commence. The graph above highlights a theoretical area where the sintering should stop in order to achieve good parts. If the sintering process continues for too long it will result in “oversintering”. Oversintering is a defect caused by grain growth in the microstructure. Grain growth causes reduced mechanical properties in the produced parts. (Heaney, 2012, pp. 158-159)

The optimal strength requires the grain size to be as small as possible and the density to be as high as possible. A density between 95- 100% of theoretical is typical for a well-produced MIM part with good properties. The graph below shows the correlation between density and grain growth inside the “acceptable parts zone” near the end of a sintering process. (Heaney, 2012, pp. 230-231)

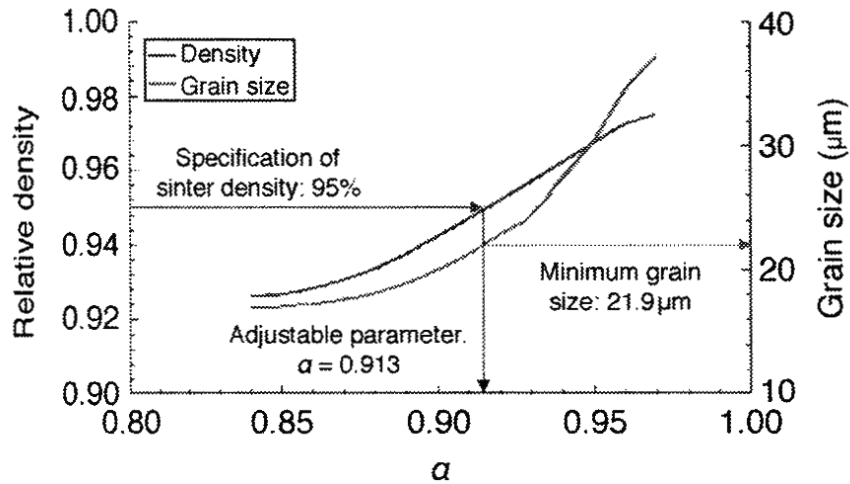


Figure 19. Plot of minimum grain size for specific density of 17-4ph stainless steel. (Heaney, 2012, p. 231)

The graph below shows a rough example of what a typical heating pattern would look like. It highlights the scale and ratio of the heating rate, peak temperature duration and cooldown rate. This sample cycle could be optimized by cutting the peak time as short as possible and increasing heating rate just up to the point where defects start to form for instance.

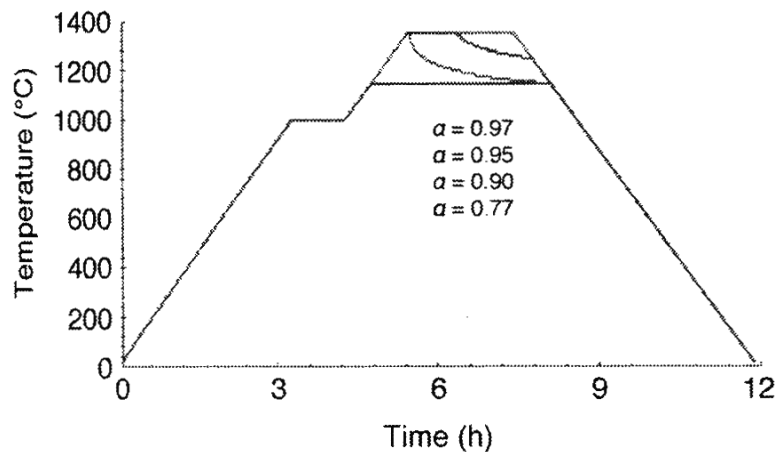


Figure 20. Sintering heating pattern for high density low grain size 17-4ph. (Heaney, 2012, p. 231)

Empirical tensile tests are the best way of determining how well the components have been sintered. Microscopy can also be a useful tool for looking and measuring grain

growth. Finding the optimal parameters for a sintering cycle requires multiple series of test runs and analysis.

5 RESULTS

The optimization of a metal injection molding process turns out to be a quite labor intense and long process. There are no simple and concise calculations which can provide the optimal parameters, but merely function as guide lines. Finding the best parameters is a process in itself. The task of optimization is highly empirical and relies heavily on analysis and defect recognition. The research concludes that optimization of MIM parameters follows a certain work flow. This work flow is best illustrated in the form of a logic diagram.

The following diagram show a rough idea of how to approach an optimization of MIM process parameters:

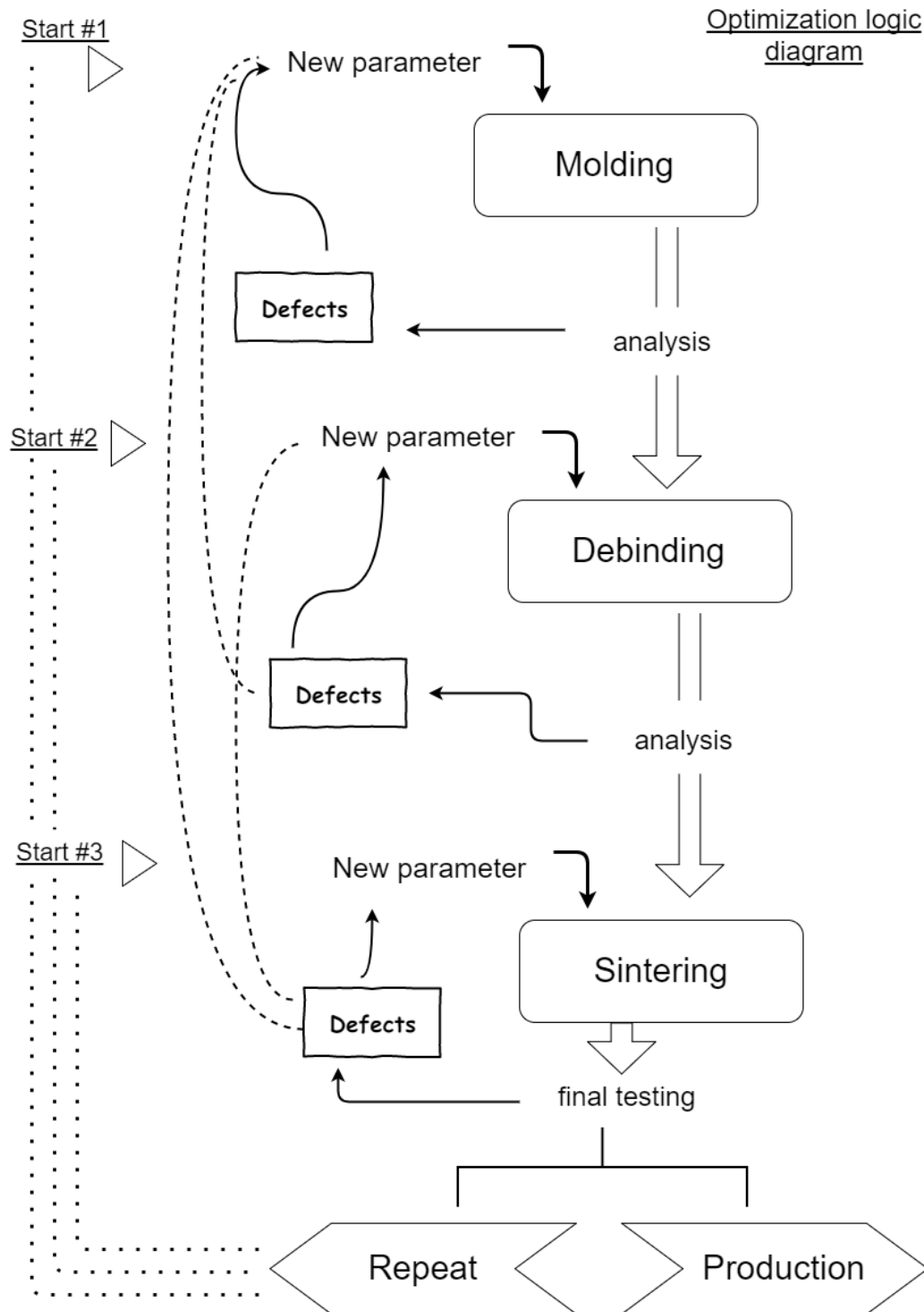


Figure 21. Optimization logic diagram (author,2017)

- New parameter: represent that a change has been done or needs to be done to a process parameter. This is also where the optimization begins.
- Analysis: means the parts a being studied in order to find defects and to evaluate the success of the previous process. Common analysis methods for each process are mentioned under the Monitoring chapter.
- Defects: distortion cracking, voids etc. Defects also include incomplete process such as binder leftover or insufficient densification.
- Repeat: the process should be repeated until satisfactory results are found.
- Production: once it's believed optimal parameters have been found or it's not worth continuing searching further the new parameters are to be used in production.
- Start # 1 / 2 / 3: corresponds to the start of a specific process stage. If the optimization is only directed at a specific process the steps above its corresponding start point can be ignored.

6 DISCUSSION AND CONCLUSION

The aim of the research was to find a way to optimize the parameters of a metal injection molding process. I was expecting to come across a quick and easy method for figuring out what parameters should be used. But it didn't turn out to be that simple, no formula and calculation could take all the variables into account. It became evident that optimization of the MIM parameters should be approached as a trial and error exercise.

The end result displays how to go about doing an optimization or adjustment of MIM parameters. The result is very general and applies to any debinding and sintering method. Each debinding method could have its own more specific guide. The diagram shouldn't be followed too rigorously, it should only be a tool for conveying the logic and mindset behind starting optimizing an already functioning production.

With so many parameters and variables, achieving the perfectly optimal process is extremely demanding if not impossible. And the whole point of optimizing the MIM process is to be more resourceful and to cut down on process time. One could continue trying out changes to the process forever but at some point more time will have been spent on the

optimization than what the improved process will save. The risk of analysis paralysis should be kept in mind whenever changes are being done to a functioning production.

7 SUGGESTION FOR FURTHER WORK

A more specific guide for each debinding method and sintering furnace type should be researched. Also, a more in-depth look at a specific parameter would be valuable. Computer simulations and optimization should also be looked into further.

The feedstock is what makes the whole MIM process possible and alternative applications for it should be looked into. The ease of shaping and good mechanical properties of the end-product provided by the feedstock could possibly be used in other manufacturing methods. The most topical would be to see if MIM feedstock could be used for metal 3D printing.

8 REFERENCES

BASF, 2009. 1. [Online]
Available at:
http://www.catamold.de/cm/internet/en_GB/function/conversions:/publish/content/Microsite/Catamold/Technische_Informationen_/Verfahrensanweisungen/GeneralProcessing_Instructions_Catamold_CIM.pdf
[Accessed 2 12 2016].

Ehrhart, P., 1991. *Properties and interactions of atomic defects in metals and alloys*. 3 ed. Berlin: Springer.

German, R. M. & Bose, A., 1997. *Injection Molding of Metals and Ceramics*. New Jersey: Metal Powder Industries Federation.

Heaney, D. F., 2012. *Handbook of metal injection molding*. Cambridge: Woodhead Publishing Limited.

Pachauri, P. & Hamiuddin, M., 2015. *Optimization of Injection Moulding Process Parameters in MIM for Impact Toughness of Sintered Parts* , Aligarh: International Journal of Advanced Materials and Metallurgical Engineering .

PlasticsEurope, 2016. 1. [Online]
Available at: <http://www.plasticseurope.org/what-is-plastic/types-of-plastics-11148/engineering-plastics/pom.aspx>
[Accessed 1 12 2016].

Polyplastics, 2016. *Polyplastics*. [Online]
Available at: <https://www.polyplastics.com/en/support/mold/outline/>
[Accessed 5 11 2016].

Schilling, W., 1978. *Self-interstitial atoms in metals*. s.l.:Institut für Festkörperforschung der Kernforschungsanlage.