Development and analysis of micro powder injection molding

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Development and Analysis of Micro Powder Injection Molding

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ABSTRACT

Micro powder injection molding (μ PIM) is a potential process for the mass fabrication of metal or ceramic micro-structures. It has many advantages such as low cost, high productivity, suitable for fabricating micro-structures with complex geometry. This thesis deals with the molding and demolding of high aspect ratio 316L stainless steel micro-structures.

μ PIM was first used to fabricate 316L stainless steel micro-structures using existing conventional mold and silicon mold insert with micro-cavities etched by deep reactive ion etching (DRIE). It was found that incomplete filling and demolding problem were two main hindrances to the successful fabrication of micro-structures, especially in the case of micro-structures with high aspect ratio. To mold high aspect ratio micro-structures, a variotherm mold was designed and fabricated (rapid mold temperature variation technology is termed as variotherm).

The new variotherm mold is equipped with rapid mold heating/cooling system, hot sprue system, cavity vacuuming system to prevent air entrapment and combined pressure and temperature sensor. The variotherm mold facilitates the injection molding of high aspect ratio micro-structures because it improves the complete filling of the micro-cavity at the elevated mold temperature. Variotherm mold heating /cooling system enables the rapid adjustment of demolding temperature. Thus,

fabrication of high aspect ratio micro-structure was possible due to complete filling of the micro-cavity and successful demolding. $\phi 40\text{ }\mu\text{m} \times \text{height } 174\text{ }\mu\text{m}$ micro-structures with aspect ratio of 4.4 and $\phi 20\text{ }\mu\text{m} \times \text{height } 160\text{ }\mu\text{m}$ micro-structures with aspect ratio of 8 were successfully injection molded using the variotherm mold.

The demolding of the micro-structures was analyzed both theoretically and experimentally. Finite element method (FEM) software ABAQUS was used to analyze the demolding of an array of 24×24 (total of 576) micro-structures. It was found that there exists a “critical temperature” at which the demolding force for the micro-structures array is a minimum. The stress distribution of the micro-structures and demolding force for the micro-structures during the course of demolding were analyzed for demolding temperatures both higher and lower than the critical temperature. Packing pressure and demolding temperature have apparent impact on the demolding force. A series of demolding force measuring experiments at different packing pressures and demolding temperatures were conducted to verify the theoretical results.

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LIST OF SYMBOLS

b_{1m}	data-fitted coefficient of PVT curve of the feedstock
b_{2m}	data-fitted coefficient of PVT curve of the feedstock
b_{3m}	data-fitted coefficient of PVT curve of the feedstock
b_{4m}	data-fitted coefficient of PVT curve of the feedstock
b_{1s}	data-fitted coefficient of PVT curve of the feedstock
b_{2s}	data-fitted coefficient of PVT curve of the feedstock
b_{3s}	data-fitted coefficient of PVT curve of the feedstock
b_{4s}	data-fitted coefficient of PVT curve of the feedstock
b_5	data-fitted coefficient of PVT curve of the feedstock
b_6	data-fitted coefficient of PVT curve of the feedstock
b_7	data-fitted coefficient of PVT curve of the feedstock
b_8	data-fitted coefficient of PVT curve of the feedstock
b_9	data-fitted coefficient of PVT curve of the feedstock
$B(T)$	a variable in modified double-domain Tait PVT model that accounts for the pressure sensitivity of the feedstock
C	universal constant 0.0894 in modified double-domain Tait PVT model
CTE	coefficient of thermal expansion
d	the diameter of the micro-cavity
d_d	the diameter of the micro-structure at demolding temperature
D_c	the diameter of mold cavity
D_d	the diameter of green part at demolding temperature T_d
F	frictional force between two frictional surfaces
F_D	demolding force for the green part
F_{Fb}	frictional force between cavity wall and the circumferential surface of the base of green part
F_{Fm}	frictional force between micro-structure and the inner surface of the micro-cavity
F_{Sb}	sticking force between top surface of the base of green part and cavity inner surface
F_{Sm}	sticking force between top surface of the micro-structure and the bottom surface of the micro-cavity
F_N	normal force
F_v	additional release force due to vacuum effect for the case where contact between the micro-structure and the micro-cavity exists
H	the thickness of mold cavity
h	the depth of the micro-cavity
H_d	the thickness of the base of green part at demolding temperature T_d
h_d	the height of the micro-structure at demolding temperature T_d
k_c	consistency of the feedstock
M	mass of the feedstock packed into mold cavity
N	normal force between two frictional surfaces

List of symbols

N_m	the number of micro-structures
p_s	cavity pressure at the moment of gate solidification
p_a	atmospheric pressure
T_{cr}	critical temperature
T_d	demolding temperature
T_s	temperature of micro-structure at the moment of gate solidification
T_i	transition temperature of the feedstock at pressure p
UTS	ultimate tensile strength
V_c	the volume of the mold cavity
V_d	the corresponding volume of green part after demolding
γ	linear shrinkage ratio
Δd	the diametric variation of the micro-structure at the demolding temperature
η	viscosity
η_a	apparent viscosity
μ	coefficient of friction
μ_s	coefficient of static friction
μ_d	coefficient of sliding friction
$v_0(T)$	the specific volume at zero gauge pressure
$v(T, p)$	the specific volume at temperature T and pressure P
$v(T_d, p_a)$	the specific volume of green part at atmospheric pressure after demolding
$v(T_s, p_s)$	the specific volume of the feedstock packed in the mold cavity at the moment of gate solidification

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CHAPTER 1

INTRODUCTION

1.1 Background

The fabrication of components in the micron-scale range is one of the leading technologies for the present century and is expected to have a high market potential [1]. In the past ten years, there has been an upsurge in the mass fabrication of complex micro-components including micro-structures and microparts etc. that allow consistent reproduction of features down to sub-micrometer precision. Such micro-structures and microparts hold enormous potential for the important area of microsystem applications, such as miniature multiconnectors, microfluidic pumps, chemical mixers, acceleration-sensors, micro heat exchanger, microtiter-plate and micromedical tools [2-7]. Microparts are generally understood to be single or separated components with outer dimensions in the range of a few millimeters, but having details in the sub-millimeter range [8]. Microstructured parts have outer dimensions of several millimeters up to a few centimeters with three-dimensional micro-structures being located on one or several surface areas [8].

High aspect ratio micro-structures are demanded in various applications. The aspect ratio of a micro-structure is the ratio of the height/length of the micro-structure to the minimum micro-feature of the micro-structure [9]. In general, an aspect ratio of between 5 and 10 is considered high, while above 10 it is considered very high. The

applications of high aspect ratio micro-structures include micro electromechanical systems (MEMS), microfluidic and biomedical applications. For example, the demand for high aspect ratio micro-structures for sensor and actuator applications has increased, as they offer a number of advantages including increased structural rigidity, low driving voltage, higher actuation force, and larger displacement in actuator systems, higher sensitivity in sensor applications by virtue of large mass, and large magnetic force for magnetic MEMS [10]. A typical type of high aspect ratio micro-structures – an array of micro/nano pillars has been exploited for applications ranging from microfluidic chips for separation of long DNA molecules [11, 12] and adhesion substrates for cell growth [13] to the investigation of hydrophobicity and hydrophilicity on nanostructured surfaces [14]. Introducing high aspect ratio micro-structures to a sheet of material can dramatically change the surface properties of the sheet, such as its surface energy, radiation absorptivity and emissivity, ability to absorb mechanical and acoustic energy, catalytic activity etc. For example, the rate of heat transfer between an object and the surrounding medium can be dramatically changed by covering the surface of the object with high aspect ratio micro-structures, without significantly changing the size or weight of the object [15].

The fabrication of micro-structures is dominated by materials based on silicon or polymers [16]. However, metal makes it more attractive as it has superior mechanical properties and thermal stability. For example, metals inherently have higher fracture toughness than silicon and other semiconductor materials which are brittle and are therefore less durable and have lower shock-resistance. High aspect ratio metal micro-

structures are elements of many innovative devices including nickel micromold with maximum aspect ratio of 15 [10], micro electromechanical systems (MEMS) such as titanium/nickel comb drive microactuator [17, 18], free-standing copper anti-scatter grids for mammography [19] and drug delivery [20]. There are also a variety of applications for high resolution metal micro-structures with high aspect ratios. These include electron beam sources, which make possible the fabrication of high power microwave devices, free electron lasers, projection electron beam lithographic sources and flat panel displays [21].

Processes such as diamond microcutting, lithography and electroplating, and MARIO (metal anisotropic reactive ion etching with oxidation) have been used to fabricate high aspect ratio metal micro-structures [18, 22, 23]. But they suffered from low productivity, high fabrication cost or limited suitable materials [24]. They are also not suitable for mass fabrication. Micro powder injection molding (μ PIM) has the potential to fabricate micro-components such as micro-structures or microparts in large quantity with low fabrication cost [24-29]. It also has advantages such as shape complexity and applicability to many materials [24].

μ PIM is adapted from powder injection molding (PIM) but some special requirements (as compared to PIM) are necessary. They include smaller powder size, feedstock with higher strength and use of a mold insert with micro-scale features. As shown in Figure 1.1, μ PIM also comprises the same four main processing steps as PIM: mixing,

injection molding, debinding and sintering. The powder is mixed with a binder to form the feedstock. The feedstock is then fed into the injection barrel of the injection molding machine. In the barrel the feedstock is heated up and melted. The feedstock melt is injection molded (similar to plastic) into the required shape to form green (molded) micro-structures or microparts. The binder is removed via thermal or other means such as catalytic debinding. The final micro-structures or microparts are obtained by sintering the debound micro-structures or microparts in a controlled environment giving a small porosity and significant increase in hardness, strength and wear resistance.

Figure 1.1 Processing steps of μ PIM (for the fabrication of micro-structures)

1.2 Challenges & issues

In the injection molding step, μ PIM faces two challenges for it to be a viable method for the fabrication of metal micro-structures. The first is the complete filling of the

micro-cavities of the mold insert with feedstock (to form the micro-structures). The second is the successful demolding (i.e. ejection) from the micro-cavities without causing breakage of green micro-structures.

The first issue deals with the heat loss and rheological properties of the feedstock melt during injection molding. The feedstock melt solidifies fairly rapidly when it encounters the inner surface of the cold micro-cavity. The situation is worsened when molding micro-structures with high aspect ratio as they have high surface area to volume ratio which makes it more sensitive to heat loss, thus incomplete filling is highly possible.

The second deals with the friction and adhesion forces between the green micro-structures and the micro-cavities, and the green strength (strength of the green micro-structures). Again, the high surface area to volume ratio makes demolding difficult.

The above issues can be overcome by using a variotherm mold and a feedstock with high green strength. The “variotherm” mold mentioned has been used for micro plastic injection molding [9] and it should be possible to use the variotherm mold for micro powder injection molding. When using a variotherm mold, the mold is heated to temperatures near the melting point of the feedstock (prior to injection), where the feedstock melt has good flowability that assists in the complete filling of the micro-cavities. This is very crucial especially for high aspect ratio micro-structures. After filling, the mold is cooled until the green strength is sufficient to ensure successful

demolding. The use of commercial BASF Catamold feedstock which has high green strength will facilitate successful demolding.

1.3 Research objectives

The first objective of this research is to empirically investigate the feasibility of the variotherm process for μ PIM, and then determine suitable operating conditions for fabricating high aspect ratio 316L stainless steel micro-structures.

The second objective of this research is to study the demolding mechanism of the micro-structures theoretically and experimentally so as to establish suitable processing conditions for the successful demolding of the micro-structures. The theoretical analysis is conducted using ABAQUS finite element method (FEM) software and the pressure-volume-temperature (PVT) properties of the BASF Catamold 316L stainless steel feedstock. The effects of two main injection molding parameters, namely packing pressure and demolding temperature, on the demolding force are studied.

1.4 Dissertation organization

The dissertation organization and the main topics are shown in the following flow chart:

Chapter 2 Literature review

1. State-of-the-art of μ PIM
2. Area of research in this dissertation

Chapter 3 Material characterization and experimental equipment

1. Characterization of the feedstock including thermal, rheological, mechanical and frictional properties for selecting suitable injection molding conditions and subsequent use in the theoretical analysis
2. Introduction of main experimental equipment

Chapter 4 μ PIM without variotherm mold

1. Experimental results using conventional mold
2. Limitations of the conventional mold

Chapter 5 μ PIM with variotherm mold

1. Variotherm mold design for the fabrication of high aspect ratio micro-structures
2. Experimental results using variotherm mold

Chapter 6 Analysis of micro-structure demolding

1. Theoretical analysis of the demolding of micro-structures based on PVT properties of the feedstock
2. Finite element method (FEM) analysis of the demolding of micro-structures to select suitable processing parameters for successful demolding
3. Experimental verification

Chapter 7 Conclusions and future work

CHAPTER 2

LITERATURE REVIEW

2.1 PIM

Considering that μ PIM evolves from PIM, a literature review of PIM is presented. Powder injection molding (PIM) is a process used to fabricate net-shape/near net-shape parts. It combines the benefits of plastic injection molding (complex shapes and high productivity) with the advantages of powder metallurgy (nearly unlimited choice in alloy-composition and isotropic properties) [30]. PIM can be classified into ceramic injection molding (CIM) and metal injection molding (MIM).

A typical PIM process includes the following four processing steps: mixing, molding, debinding and sintering. The physics of PIM steps are shown in Figure 2.1. PIM begins by mixing selected powder and binder, the mixture of powder and binder is termed “feedstock”. The purpose of mixing is to break up powder agglomerates using high shear and coat the powders with the binder to get a homogeneous feedstock. The powders are small to aid sintering and usually have sizes between 0.1 and 20 μm with near spherical shapes [31]. The binder ensures the flowability of the feedstock during injection molding and contributes to the strength of the green part (molded part) and the debound part (part after debinding). It is usually based on a common thermoplastic, but food-grade polymers, cellulose, gels, silanes, water, and various inorganic substances are also in use. Usually, the binder consists of several components.

Figure 2.1 Physics of PIM steps (adapted from [31])

The second step is injection molding. The pelletized feedstock is heated to a sufficiently high temperature so that it melts in the plasticating barrel of the injection molding machine. The feedstock melt is forced into the mold cavity by the reciprocating screw or plunger. There, the feedstock melt cools and solidifies assuming the shape of the mold cavity. The molded part is termed the “green part”. Finally, the green part is ejected when the molding cycle ends.

After injection molding, the binder has to be removed from the green part. Depending on the binder used, a wide array of options exists for binder extraction, but mainly consists of three methods: thermal, solvent and catalytic [31]. In most commonly used thermal debinding, the binder is thermally decomposed into low molecular weight components such as water, methane, carbon oxide etc and subsequently removed by diffusion or permeation, as shown in Figure 2.1.

The last step is sintering in which the powders in the debound part are bonded into a coherent, solid mass whose density may reach near theoretical density. Consequently, the void space remaining after removal of the binder is eliminated with appreciable shrinkage. Sintering often occurs in protective atmosphere or vacuum at a specific temperature. In sintering, the initial open pore structure and high porosity are consumed by inter-particle neck growth, grain growth and pore shrinkage, with eventual formation of closed spherical pores in the final stage.

PIM markets are found in small complex parts [32]. Being a net-shape/near net-shape process, it is cost effective since secondary operations are reduced or eliminated. In addition, PIM is able to fabricate complex parts with high productivity. As described in Figure 2.2, PIM has three key attributes – shape complexity, high performance and low cost [31].

Figure 2.2 Venn diagram of PIM three key attributes [31]

Despite the numerous advantages, PIM also has disadvantages and there are hurdles to growth: (a) the raw materials, especially metal powders are expensive [32], (b) for shapes with simple or axial-symmetric geometries, it is not competitive in contrast with other shaping technologies [31] and (c) it's difficult to achieve tight dimensional tolerance in sintered part so that in some cases a secondary operation is a must, thus increasing the fabrication costs [32].

As the demand for complicated-shape metal and ceramic components has expanded rapidly in recent years, PIM has become a popular method to meet the requirements. It is the preferred process for mass fabrication over other slurry techniques like dry pressing, cold isostatic pressing, slip casting and tape casting etc [31]. PIM has been

widely used in various industries such as automobile, defense, aerospace, electronics, household appliances and medical/dental appliances.

2.2 Applications of μ PIM

As mentioned in Section 1.1, μ PIM is adapted from PIM. Some applications of μ PIM are discussed below:

a) Biomedical applications

Many medical devices, especially instruments and implants, are fabricated from difficult-to-machine materials such as stainless steels, cobalt-chromium alloys, and titanium alloys. New applications for biomedical components are trending toward smaller, more-complex devices for minimally invasive surgery (MIS), such as laparoscopic instruments for grasping tissue, cutting and suturing. μ PIM is particularly well suited for their fabrication and 316L stainless steel has been widely used for many implants [33, 34].

b) Microfluidics application

A typical application is nozzle micro-structures with smallest detail of 20 μm as shown in Figure 2.3 [35]. Another example is an annular gear pump shown in Figure 2.4 [36]. Ceramics are used in both applications.

Figure 2.3 Spin nozzle [35]

Figure 2.4 Annular gear pump [36]

c) Microreactors, sensors and microrods array

Microreactors made by μ PIM is shown in Figure 2.5 [37]. In Figure 2.6 and Figure 2.7, PZT transducer and microrods array are presented respectively [38].

Figure 2.5 Microreactor [37]

Figure 2.6 Transducer for fingerprint scanner [38]

Figure 2.7 Microrods array [38]

d) Micro optical parts

Low thermal expansion ceramic micro optical devices such as micro optical benches that can be used for communication systems in outdoor environment. These benches have to carry functional units like micro lenses or micro beam splitters [8, 39].

e) Mold inserts

A metal mold insert fabricated by μ PIM for the structuring of plastics, metals and ceramics is shown in Figure 2.8 [40].

Figure 2.8 Metal mold insert [40]

2.3 Feedstock preparation

The pelletized mixture of powder and binder used in injection molding is termed the feedstock. The purpose of mixing is to coat the particles with the binder and break up the agglomerates to get a homogeneous feedstock. Five factors determine the attributes of the feedstock – powder characteristics, binder composition, the ratio of powder to binder, mixing method and pelletizing techniques [41].

2.3.1 Powder characteristics

Generally the selection of a powder is essential to achieve defect-free green part, and final parts as well as the successful control of process. Thus, an understanding of powder characteristics is important. Particle size is measured by determining the dimensions of a particle. The size depends on the measurement technique and particle shape. A suitable measurement method is coherent (laser) light-scattering technique [41]. Usually, size determinations are based on detecting discontinuities in the

property of a fluid stream (air or water) carrying dispersed particles. The particle size distribution can be shown as a histogram of the amount of powder in the measured size increments as shown in Figure 2.9(a). Most typical is a plot of the cumulative particle size distribution, which gives the amount of powder smaller than a given size. As shown in Figure 2.9(b) and (c), the percent of particles by mass smaller than a given size is plotted versus the particle size on a logarithmic scale [31]. A typical concern is with three points on the distribution, designated as D_{10} , D_{50} and D_{90} that correspond to the particle size at 10, 50 and 90% on the cumulative distribution.

Figure 2.9 Particle size distribution of water atomized stainless steel [31]

One important attribute is the selection of a powder that reduces defects during processing. Generally, this requires a spherical powder with a size below 20 μm and a packing density near 60 % of theoretical density that is free of agglomeration [31]. A wide particle size distribution gives a higher packing density that leads to less shrinkage, and easier processing as well as dimensional control. However, in the case of a wide particle distribution, special care must be taken during feedstock mixing. Due to the size difference between particles there will be segregation in the mixed powder. This can lead to uneven packing densities and distortion during sintering.

Particle shape has a large effect on the success of the process. Irregular particles exhibit better part shape retention during debinding, the packing coordination number and density are degraded by irregular particle shape [42]. Thus, more densification is needed in sintering to compensate for the poor initial packing. A compromise is to combine some spherical and irregular particles to attain the benefits of each shape [41]. Figure 2.10 gives a collection of particle shapes and shows the appropriate qualitative descriptors [31].

Figure 2.10 Qualitative particle shape descriptions [31]

2.3.2 Binder

The binder provides the powder with the fluidity necessary for molding. Many binders are being used industrially and can be categorized into: thermoplastic compounds, thermosetting compounds, water-based systems, gelation systems, and inorganics [31]. Although many binders are available on a fabrication basis, thermoplastics are so far the most widely used and understood.

Generally the binder consists of multiple components – a backbone polymer that provides strength, a filler phase that is easily extracted in the first phase of debinding, and a surfactant to bridge between the binder and the powder. Often, the most effective surfactants reduce the mixture viscosity and increase the solids content of the mixing by creating an interfacial bridge between the powder and the binder. During debinding, the binder components are removed progressively. The primary binder components such as filler phases and surfactants are removed first to partially open the pores. The fully debound but unsintered structure becomes so fragile that it is impossible to handle. The secondary polymer component, which usually remains in the debound part serving as backbone, retains the shape of the part, preventing slumping and distortion [31, 43]. Eventually the backbone polymers are removed during sintering, which occurs as part of the heating process before the sintering temperature is reached.

2.3.3 Ratio of powder to binder

The ratio of powder to binder largely determines the success or failure of subsequent steps. Generally, feedstock is prepared using minimum binder content, which gives rise to high green density and little shrinkage during sintering. The powder loading is the volumetric ratio of powder to the total volume of powder and binder. The optimal powder loading for molding has less powder than the critical powder loading (the critical powder loading is the composition where particles are packed as tightly as possible without external pressure and all space between the particles is filled with

binder), typically a value of 60_{vol.}%. At this point the feedstock has sufficiently low viscosity for molding but exhibits good particle-particle contact to ensure shape preservation during processing [31].

2.3.4 Mixing and pelletizing

In the mixing of powder and binder, failure to disperse or evenly distribute the powder in the binder may result in powder-binder separation, segregation and agglomeration in the feedstock. These may be translated into non-uniform shrinkage during sintering and cause inhomogeneities in the final properties. The best mixing occurs with a high shear, but not to the point where the work of mixing damages the particles or overheats the binder. A properly mixed feedstock consists of homogeneous powder dispersion in the binder with no internal porosity or agglomerates. Several high-shear mixers are used for feedstock preparation that include double planetary mixers, single screw extruders, plunger extruders, twin screw extruders, twin cam extruders, shear roll compounders, rotor-stators, sigma blade mixers and Z-blade mixers.

The homogeneously mixed feedstock is pelletized for injection molding and storage/transportation. The pellets should have a consistent shape and be free of air bubbles and defects. There are two goals in pelletizing feedstock. The first is to prepare easily transported pellets that can be loaded into molding machine. The second is to incorporate recycled feedstock back into the molding process. Two types of commonly used pelletizing equipment are shown in Figure 2.11 [41]. The first

pelletizing equipment comprises of rotary cutters encased within a cutting chamber. In this chamber, the rotary cutters shred the feedstock into pellets. A sizing screen ensures that only small enough pellets are collected. The second pelletizing equipment is actually an extruder with a chopper. The extruded feedstock is chopped into small pellets by the chopper.

Figure 2.11 Two types of commonly used pelletizing equipment for feedstock [41]

2.3.5 Special feedstock requirements in μ PIM

In μ PIM, the particle size of the metal powder is a very important parameter that limits the producible structure dimensions and aspect ratio. For this reason, feedstock of high strength with smaller metal/ceramic powders having a mean particle size far below $3\ \mu\text{m}$ is desired [8]. For all materials there exists a tendency to use smaller powder. The smaller powder helps to fabricate smaller structures and lead to sharper corners and higher sintered density in the structure. Metal powders in the sub-micron range are not as easily available as ceramic powders. The reason for this lies in the ductility and reactivity of the metals, which make it difficult and very expensive to

fabricate fine powders [44]. In Germany, metal powder with particle size down to $D_{50} = 2 \mu\text{m}$ was used, while at Karlsruhe University (Germany) and the Nanyang Technological University (Singapore), 316L stainless steel powder of $D_{50} = 4 \mu\text{m}$ and $D_{50} = 4.5 \mu\text{m}$ are used respectively to fabricate 3-D metal micro-structures [8, 28, 43, 45].

Most commercially available feedstocks are limited in their properties to fabricate micro-structures and microparts, especially in the case of high aspect ratio. To solve this problem, the development of suitable feedstocks with small particle size as well as appropriate binders is necessary [46]. To achieve sufficient green strength, the composition of the binder is very important. The binder must be appropriately selected in order to give higher green strength for demolding, lower viscosity for easy filling of cavity during molding, as well as good shape retention and lower shrinkage during debinding and sintering [43, 47-50].

2.4 Injection molding

The μPIM injection molding step is similar to that of PIM. The defects and problems associated with PIM also apply to μPIM . In its simplest form, the feedstock is heated to a sufficiently high temperature causing it to melt in the plasticating barrel of the molding machine. The feedstock melt is forced into the mold cavity by the reciprocating screw or plunger. There, the feedstock melt cools and solidifies assuming the shape of the cavity. Finally, the green part is ejected when the molding cycle ends.

2.4.1 Mold

The mold is used to produce the shape of the green part. It is typically (but not always) manufactured from tool steel material where machining is performed to obtain the mold cavity. The surface finish of the mold cavities will determine the finish of the part. Figure 2.12 shows a typical two-plate mold assembly [31]. A typical sprue, runner and gate system is shown in Figure 2.13 [51].

Figure 2.12 A typical two-plate mold assembly [31]

Figure 2.13 Sprue, runner and gate of green part [51]

The sprue is a channel through which feedstock melt is transferred from the injecting nozzle of injection molding machine into the mold. Sprues are normally tapered from 1.5° to 5° . In a two-plate mold the green part and sprue are ejected together. In a three-plate mold, the green part and sprue are separated during mold opening. The runner is a channel that guides the feedstock melt into the mold cavity. Typically, runners have diameter of 3 to 6 mm to improve the filling speed. Figure 2.14 shows some typical runner cross-sections [52]. The gate is an entrance through which the feedstock melt enters the cavity. The gate has various designs, some common gate designs are: standard edge gate, fan gate, pin point tab gate, direct sprue gate, disk gate, flash or film gate, bottom or jump gate, submarine gate, spoke or spider leg gate [52].

Figure 2.14 Types of runner cross-sections [52]

2.4.2 Main injection molding phases

The entire injection molding cycle can be divided into several phases, which partly overlap one another as shown in Figure 2.15 [53]. The main injection molding phases are filling phase, packing pressure phase and cooling phase.

Figure 2.15 Injection molding process cycle [53]

The term “filling phase” is used to describe the part of the injection molding between the beginning of mold filling and the point at which the machine is switched to packing pressure phase. Usually, the filling phase is under velocity-controlled conditions. This means that the screw forces the feedstock melt into the mold cavity with a velocity profile that changes in several steps. After leaving the runner channels, the feedstock melt flows into the mold cavity and is then subjected to simultaneous flow and cooling processes.

The packing pressure phase starts at the switchover point and ends at the end of packing pressure exerted by the machine. The packing pressure phase is performed under pressure control. Usually this means that the injection screw of the injection molding machine is loaded with a pressure that can be adjusted in several steps. The pressure profile has to be used to optimize shrinkage and warpage behavior of the part. In Figure 2.16, the injection hydraulic pressure and cavity pressure during a typical

injection molding cycle are presented.

$t_1 - t_4$ filling phase ($t_1 - t_2$ filling the mold, $t_2 - t_3$ cavity full-pressure rises
 $t_3 - t_4$ compression of the feedstock melt), $t_4 - t_5$ packing pressure phase

Figure 2.16 Injection hydraulic pressure and cavity pressure profile [52]

The cooling phase starts immediately after the injection of the material and includes both the injection and packing pressure phases. However, the cooling time must be extended beyond the packing pressure phase. This is because the green part would not have cooled down sufficiently and is not sufficiently stable for demolding.

2.4.3 Micromolding (for plastic)

As the injection molding step of μ PIM shares some common process features with micromolding for plastic and both face the same problem of incomplete filling, a review of micromolding follows. The novel mold and machine design, aimed at complete filling in micromolding for plastic, can be also used for μ PIM.

Micromolding is a key technology for the economic mass fabrication of plastic micro-structures or microparts for microsystems [54]. Once a mold insert has been made, several thousand parts can be molded with little effort. The cost of the raw material in most cases is negligibly low, because only small material quantities are required for micro-components. Therefore, micro-structures or microparts fabricated by micromolding, even from high-end materials, are suitable for applications requiring low-cost and disposable components [54].

Two typical micromolding technologies are micro plastic injection molding and hot-embossing.

a) Micro plastic injection molding

Micro plastic injection molding is adapted from conventional plastic injection molding technology. It requires a number of equipment modifications from conventional plastic injection molding technology, i.e. it usually requires more peripheral equipment than conventional injection molding [9, 54, 55]. This is mainly due to the small dimensions of the molded plastic micro-structures, the high aspect ratio and the demand for sub-micron precision [56, 57]. Therefore, the machines and tools used for micro plastic injection molding have been modified from those used for conventional plastic injection molding and are discussed subsequently.

For the injection of milligram-shot weight, the metering and dosing have to be very exact. In this aspect, separate plasticating (metering and dosing) units are used, and

smaller metering screw and smaller injection plungers are used to guarantee exact metering and fast injection [9, 58-60]. Figure 2.17 and Figure 2.18 show the material plasticating and injection units from Germany IKV and Battenfeld, respectively [58, 59].

In conventional molding process, air vent slots facilitate the escape of air in the mold cavity during mold filling. However, these slots are close to the size of the micro-structures. Therefore, the cavity has to be evacuated using an external evacuation system [8, 54, 61]. Further, the incorporation of vacuum system is necessary to avoid entrapped air and “diesel effect” in the blind micro-cavity during molding, and to enhance the mold-filling process. In some cases, a pressure of less than 1 mbar is used [8, 62].

Figure 2.17 IKV micromolding machine [58]

Figure 2.18 Battenfeld microsystem injection process [59]

In micro plastic injection molding, the extreme aspect ratios in combination with the small dimensions of the micro-structures, require a temperature variation technology termed as variotherm [9]. In the case of injection molding high aspect ratio micro-structures using conventional mold, the rapid heat loss often leads to incomplete filling of the mold cavity. Thus, it is necessary to use very high mold temperature for easy filling. However, such a high temperature leads to longer cycle time. As a consequence, the reduction of the cycle time is one of the key issues for economic success of micro-structures fabrication [16].

In a variotherm mold, a rapid mold heating/cooling system is fitted. In this case, the mold can be rapidly heated by electrical cartridges to approximately the melt temperature (before the actual injection process). After filling, the mold is then cooled to a demolding temperature which allows a safe and defect-free demolding of the

micro-structures [62, 63]. Although the variotherm heating/cooling system shortens the cycle time to a large extent as compared with conventional mold, the large temperature difference still leads to a longer cycle time. The typical cycle time of the variotherm mold is in the order of minutes.

The cooling medium used by variotherm mold can be low-temperature oil or water. In some cases, cooling is conducted with liquid carbon dioxide [64] or liquid nitrogen. One typical example is rapid thermal response heating and cooling systems (RTR) developed in universities in the USA [65-67], as shown in Figure 2.19 [66]. The RTR system can facilitate rapid change in the surface temperature of the mold as shown in Figure 2.20 and thereby improving the quality of the plastic molded parts without appreciable increase in the total cycle time. The mold surface temperature response of the RTR mold is: heating from 25°C to 250°C in 2 sec. and cooling to 50°C in 8 sec.

Figure 2.19 Schematic experimental setup of RTR molding [66]

Figure 2.20 Changes in temperature with time in variotherm mold [66]

Due to the small part weight, hot-runner or runnerless systems are adopted in order to increase material utilization. A thermally closable pin shut-off nozzle is commonly used. This guarantees that the melt enters the mold only in the injection phase and is not sucked into the mold by vacuum [62, 63].

In micro plastic injection molding, dimensional stability of the molded part has to be ensured. An effective and extremely precise dimension alignment device with an alignment quality of $\pm 10 \mu\text{m}$ has been used [62]. Further, high tolerance requirements on the closing joint, ejector pins and the mold inserts are also required. During fabrication, tolerance $< 1 \mu\text{m}$ must be observed [62]. Typical micro-structures exhibit dimensions in the range of some tens of microns and large aspect ratio. To facilitate separating these micro-structures from the mold insert, a separation or substrate unit is needed to demold the micro-structures [68].

b) Hot-embossing (for plastic)

Replication of micro-structures and nanostructures has been successfully achieved using hot embossing for plastic [5, 69-71]. As shown in Figure 2.21, hot embossing takes place in a machine frame similar to that of a press [72]. The force frame delivers the embossing force of the order of 5 to 20 tons after the mold is heated to a temperature higher than the softening temperature of polymer sheet. After embossing one can demold the micro-structures at room temperature or below the softening temperature (or glass transition temperature). A vacuum for hot embossing ensures a longer lifetime for the mold, absorbs any water released during the embossing process from the polymer materials, prevents bubbles from entrapped air and incomplete filling of micro-cavity [73].

Figure 2.21 Flowchart of hot embossing process for micro-structures [73]

Hot embossing offers excellent properties, particularly for microoptics, microfluidics and microcapillary systems. Replicating of large, plane plates with micro-structures of highest precision, smallest distortion, and minimum stress represents the prerequisite for micro-optical applications and ensures the reproducibility that is required for

fabrication in series. As a consequence of the short flow length in this process, and the use of very low filling speed, hot embossing allows very fine micro-structures to be fabricated. And most of thermoplastic materials can be used. However, hot embossing is suitable for small structures and low aspect ratios, and difficult for large structures or features. For higher micro-structures, a higher temperature and larger force must be used. This means a longer cycle time and a larger residual stress.

2.4.4 Mold inserts for μ PIM

The potential of molding minimum structural details is defined by the method selected for manufacturing the microstructured mold inserts, their quality as well as by the particle size of the powder [8]. For mass fabrication, a suitable mold insert with long lifetime plays an important role in the μ PIM process. Up to now, about 500 cycles have been performed using LIGA (LIGA is the Germany acronym for x-ray Lithography, Electroforming and Molding, i.e. Lithographie, Galvanoformung and Abformung) nickel mold insert, mostly in fully automatic fabrication, without wear being detected on the mold insert [8]. So far, the following processes are used for the fabrication of mold insert used in μ PIM as shown in Table 2.1.

Table 2.1 Methods used to fabricate mold insert used in μ PIM

No	Micro-machining Method	Insert material	Tolerance	Max.aspe-ct ratio
1	LIGA	Ni, Ni-Fe alloy and tungsten-cobalt alloy [74-77].	0.3 μ m	>100
2	Laser ablation	Hard metal[8], polyimide film mold pattern [78, 79]	1 μ m	50
3	μ PIM	Iron (Fe), 316L stainless steel and hard metal (WC-Co10) [40]	high surface roughness compared with advanced silicon etch and LIGA, but are comparable to micro laser ablation and micro cutting	Metal: 5 Ceramics : 10 [8, 40, 80]
4	Precision engineering techniques (micro-milling , μ EDM , wire-cut erosion etc) [81, 82]	Hard, brittle, conductive materials Nitinol, stainless steel and brass [16, 48, 74]	Lateral tolerance 5-20 μ m	100
5	Plasma etching [43, 45, 83, 84]	Silicon	1 μ m	100

In this research, silicon mold inserts made by plasma dry etching of silicon wafer (e.g. DRIE - deep reactive ion etching) were used. Plasma is a key ingredient in microfabrication, as it contains a large number of positive ions with extremely high kinetic energy that can be used to remove a portion of base material by knocking out the atoms from materials such as silicon [73, 85]. DRIE technology is useful in many different MEMS applications. DRIE technology allows the attainment of nearly vertical sidewall at high anisotropy and better sidewall surface roughness by selecting suitable wafer temperature and the addition of oxygen. Etching rates ranging from 50 to 2000 nm/min are available depending on plasma reactivity and plasma source [84].

The cross-sectional profiles of trenches etched by DRIE in silicon wafer are shown in Figure 2.22 [86].

Figure 2.22 Trenches etched by DRIE [86]

In order to obtain reasonable etch rates and higher anisotropic properties, the plasma densities have to be increased. In DRIE technology, inductively coupled plasma source was introduced to meet this requirement by applying RF power to a coil placed outside the reactor. With this technology, plasma densities of one to two orders of magnitude higher than those created by conventional RIE plasma have been achieved. In DRIE etching, increased etch rates, very high aspect ratio, good uniformity, profile control and reliable notching suppression are achieved [87, 88]. Figure 2.23 shows 1.5 μm trenches etched by DRIE, with some over etching at the bottom [89].

Figure 2.23 1.5 μm trenches etched by DRIE [89]

2.4.5 Demolding

In injection molding the release and ejection of the molded part after mold opening is termed demolding. In μ PIM, during demolding, where the green part is separated from the mold, the micro-structures broke easily, especially in the case of high aspect ratio micro-structures [46]. The fabrication of high aspect ratio micro-structures fails due mainly to the feedstock, which is too soft or brittle to withstand the demolding force [46]. Additionally, the vertical cavity wall and the lack of draft angle on the cavity lead to more difficult demolding. Only with small wall roughness, can demolding be carried out without any draft angle [16].

To facilitate demolding, different strategies were investigated to improve the yield of the μ PIM process. Reducing the surface roughness of the mold is one possibility [90], as this reduces the friction of the green micro-structures during demolding. For example, high surface qualities are obtained by LIGA, which is expensive and have long fabrication times, making LIGA method attractive for selected applications only. Another strategy is the enhancement of the mechanical strength of the green part. This goal can be achieved by the substitution of standard binder by high strength polymers [90]. However, these materials also increase the viscosity of the feedstock making the molding of micro-structures more difficult [90].

So far, there is no known analysis on the demolding of micro-structures fabricated by μ PIM. The demolding of metal micro-structures is more demanding than for plastic

micro-structures as the molded part has lower strength to resist the demolding force. But, they do share common features. Therefore, the demolding of conventional injection molded plastic part and hot embossed part is reviewed and presented here.

a) Demolding analysis of molded or hot-embossed plastic part

During demolding a number of movements have to be carried out by the mold in order to detach the molded part from the cavity. These movements depend on the shape of the molded part, e.g. release of the external surfaces of the part through opening of the mold and actuating slides, and the ejection, stripping or unscrewing of the part from cavities and cores (smooth or threaded). These operations and their control can be very complex in the case of complicated part shape. The ejector system for demolding can be operated mechanically, hydraulically, pneumatically or electrically, either singly or in combination. In this research work, hydraulic clamping and ejector systems were used.

The demolding force required to eject the molded part from the mold cavity is closely related to the thermodynamic conditions set by the processing parameters, the amount of friction between the part and mold surface and the rigidity of the mold itself [91, 92]. If the demolding force is inadequately estimated, or if the various relationships are unknown, this can cause mechanical defects in the mold as well as faulty molded part or even rejects. So, it is important to have a qualitative knowledge of how the demolding force is affected by the mold, molded part and molding

conditions. With this knowledge, a designer will be able to design the mold so that demolding force is reduced to a minimum. The demolding of molded parts usually consists of pushing the part off the core or of ejecting the part from deep recesses, e.g. such as ribs, struts, fastening lugs, partition walls etc. The demolding force can be differentiated into those produced through shrinkage (e.g. a molding onto a core), through jamming and finally those due to sticking. They can occur together in various combinations.

b) Methodology for the demolding of injection molded plastic part

Demolding is a complicated step that is influenced by a large number of factors, which are mainly derived from the complicated frictional relationship between the mold cavity and the molded part. The factors affecting the demolding force are presented in Figure 2.24 [62, 91]. Changes in the demolding force can be expected from four groups of factors: mold, molded part, molding material and processing.

T: demolding temperature of micro-structures
 p: contact pressure between micro-structure and micro-cavity
 μ : coefficient of friction between micro-structure and micro-cavity

Figure 2.24 Factors affecting magnitude of demolding force [62, 91]

The method for estimating the demolding force is simplified by the following equation [62, 91-93]:

$$F_E = \sum \mu \cdot F_N \quad (2.1)$$

Where,

$$F_E = p \cdot A_C$$

F_E : demolding force

F_N : normal force

μ : coefficient of friction

p : contact pressure between the molded part and mold cavity

A_C : contact area between the molded part and mold cavity

The coefficient of friction is dependent on the mold and molded part surface in contact. It is also greatly influenced by surface roughness and the contact temperature at the friction surface, as well as molding parameters. Some factors influencing the coefficient of friction in the case of flat, sheet-like part is presented in Table 2.2 [91].

Table 2.2 Factors influencing the coefficient of friction [91]

Factor		Magnitude of effect	Direction of change		Remarks
			F_E	μ	
Contact pressure	↑	>30 %	↑	↓	Damage to part surface by microcracks
Ejection rate	↓	<10 %	↓	↓	
Cooling time	↑	<30 %	↓	↓	Slight damage of part surface by microcracks
Average demolding temperature	↓	<30 %	↓	↓	Slight damage of part surface by microcracks
Cavity wall temperature	↓	10-40 %	↓	↓	Major shrinkage, slight damage, low coefficient of friction
Melt temperature	↑	<10 %	↓ ↑	↓ ↑	
Injection pressure	↓	<30 %	↓	↓	
Packing pressure	↓	>30 %	↓	↓	Major shrinkage, slight damage, low μ
Injection speed	↓	<30 %	↓	↓	
Release agent	↑	>30 %	↓	↓	Reduction of adhesion and smearing, low μ
Surface roughness	↓	>30 %	↓	↓	Less damage to part surface through surface peak

The normal force involved in the demolding process can be converted into contact pressure via the friction surfaces. The contact pressure can have two completely different causes and require different measures for reducing the force [91].

The first type of contact pressure results directly or indirectly from the pressure profile during the injection molding step. The molded part may be jammed in the mold cavity due to this residual pressure. Cylindrical columns or micro-structures on the base are typical examples in which the part is prone to jamming or sticking inside the mold cavity. Demolding force due to this cause should be kept as small as possible by suitable product and mold design.

The second type of contact pressure is governed by the cooling process during which the sleeve-like part shrinks onto the mold core. This method is based on the idea that the mold core prevents the contraction of the cooling sleeve-like part. This “prevented shrinkage” causes a specific amount of elastic extension at the moment of demolding and this extension imparts a stress to the part, which can then be converted into the corresponding contact pressure acting on the core. The remedy for this case is the estimate of the “prevented shrinkage” at the point of demolding. The method used to determine the immediate energy-elastic dimensional change (Δd_r) or 24 hours after demolding are presented in Figure 2.25 [62].

Figure 2.25 Flow chart for computing demolding force on sleeve-like part (adapted from [62])

In summary, the demolding force is dependent on the interaction between the molded part geometric shape, mold design, part material and molding parameters. Calculations of demolding force are still limited to simple problems involving sleeve-like part that shrink upon cores. It is thought that progress will be made in this direction through the development of suitable computer programs, although these will require a more accurate prediction of pressure and temperature profiles with respect to time and location, shrinkage as well as a knowledge of temperature-dependent stresses and adhesion friction [91].

c) Demolding analysis of injection molded plastic tubular part

Some researchers in the University of Minho, Portugal, studied the demolding force required for ejecting tubular injection molded parts [93-96]. Experiments and theoretical prediction of the demolding force were conducted. The dimensions of the molded part are diameter 60 mm $\times$ length 146 mm $\times$ thickness 2 mm, as shown in Figure 2.26 [95, 96].

Figure 2.26 Injection molded tubular part used in demolding analysis [95, 96]

The effects of processing conditions on the demolding force were studied. They included the surface temperature of the core at demolding, mold temperature, melt temperature and packing pressure. The temperature at the surface of the core substantially influenced the demolding force. The demolding force decreased when the surface temperature increased. That was caused by the reduction of the modulus and the shrinkage (at demolding) caused by the high surface temperature. An optimum melt temperature that minimized the demolding force was found. The behavior of the demolding force in relation to the melt temperature seemed to be the combined result of shrinkage and frictional resistance.

The shrinkage decreased because of the increasing pressure transmission during the packing phase. The frictional resistance associated with the coefficient of friction increased as a result of the better filling of the grooves and undulations on the surface of the core. The demolding force varied inversely with the mold temperature. The reason was that high mold temperature led to less shrinkage of the tubular part and accordingly smaller demolding force. The demolding force changed inversely with the packing pressure. This was attributed to the reduction of the diametrical shrinkage associated with a rise in the packing pressure, resulting in a smaller demolding force. However, at very low packing pressure a reduction of the demolding force was observed [96]. This resulted from the large through-thickness shrinkage that caused the detachment of the material from the mold cavity and consequently a molded part with higher temperature.

In addition to the experimental study of the demolding force, a model predicting the shrinkage and the demolding force of molded tubular part using some simplifying assumptions was proposed [93]. The schematic drawing of the demolding force analysis is shown in Figure 2.27 [93].

Figure 2.27 Schematic drawing of demolding force calculation [93]

The predicted demolding force is depicted in Equation 2.2.

$$F_E = \cos \beta \cdot (\mu - \tan \beta) \cdot \frac{2 \cdot \pi \cdot H_{part}}{1 - \nu} \cdot E(\overline{T_e}) \cdot \int_0^{L_t} \left(-\frac{1}{3} \cdot \ln \left(\frac{V(x, r, t_e)}{V_s(x, r)} \right) - \frac{H}{(1 + \nu) \cdot 2 \cdot R_m} \cdot Sh_r(t) \Big|_{t_r^*}^{t_e} \right) \cdot dx \quad (2.2)$$

Where

- $\beta =$ the draft angle of the core
- $\mu =$ the coefficient of friction
- $H_{part} =$ the thickness of the tubular molded part
- $H =$ the thickness of the mold cavity
- $\nu =$ Poisson's ratio of the material
- $E(\overline{T_e}) =$ Young's modulus at average demolding temperature
- $L_t =$ length of the tubular molded part
- $V_s(x, r) =$ the specific volume at the moment of solidification
- $V(x, r, t_e) =$ the specific volume at the moment of demolding

$R_m =$ the average radius of the wall of the tubular molded part
 $Sh_r(t) \Big|_{t_r}^{t_e} =$ the thickness shrinkage of the tubular molded part from the moment when the cavity pressure reached zero to the onset of demolding

Equation 2.2 indicated that the demolding force was directly dependent on the elastic modulus at the demolding temperature, the coefficient of friction (considered here as a constant), the thickness of the part, and the variation of the (average) volume shrinkage between the moment of solidification and at the start of demolding. The last term inside the integral was included if the tube shrank in the thickness direction. This might be the case for lower packing pressure or shorter packing time leading to the reduction of the demolding force.

To verify the prediction of the demolding force, a series of experiments were conducted under different packing pressures and demolding temperatures [96]. Three common thermoplastics polymers were used: a semicrystalline PP (polypropylene) and two amorphous polymers, i.e. PS (polystyrene) and PC (polycarbonate). The experimental demolding forces and the predicted values as a function of the packing pressure and demolding temperature respectively were compared. In general, for all materials, the model was in close agreement with the experimental data and a maximum deviation of 5% between the predicted and experimental data was observed. However, some divergence was observed in the case of the amorphous materials (PC and PS) at low packing pressures. This was probably because the detachment of the material from the mold cavity surface was not considered. This phenomenon probably

caused the temperature to remain higher due to the poorer heat conduction transfer.

d) Demolding analysis of stereolithography mold (for plastic injection mold and PIM)

Stereolithography (SL) molds have proven effective for short injection molding runs. However, they are susceptible to failure because of their poor mechanical properties and the stair-step profile on the surface of SL mold. The latter is due to the drawing characteristics of the laser beam of SL system, especially at elevated temperature. A majority of these failures occur during the demolding stage, due to excessive demolding forces. Therefore, some researchers studied the demolding associated with SL molds for plastic injection molding and PIM (powder injection molding), as shown in Figure 2.28 [97, 98].

Figure 2.28 Demolding of the part from SL mold [97, 98]

In the case of SL mold for plastic injection molding, a demolding force model was developed by combining the effects of thermal shrinkage and mechanical interlocking due to stair-steps on the surface of SL tools [98-100]. Utilizing a thick walled cylinder approximation, a closed-form demolding force equation was developed. This equation permits demolding forces in SL molds to be estimated based on mold dimensions and material properties, injection molding process parameters (temperature) and SL mold building parameters. Finite element analyses were performed to validate and complement the demolding force equation. Measured forces and temperatures from injection molding experiments indicated that the demolding force model was valid for an SL mold for both circular and non-circular shape. The average differences between measured and predicted demolding forces were approximately 10 %.

In the case of an SL mold for PIM, adhesion has been measured between a powder injection molded (PIM) part and the stereolithography epoxy mold surrounding it after cooling. Analysis of the release behavior suggested a link to the thermal properties of the mold material [97]. Subsequent measurements of cooling in the part and at the part/mold interface were consistent with a one-dimensional heat transfer model. Adhesion development at the part/mold interface showed a complex dependence on the thermal characteristics of both the mold and the PIM feedstock. The release behavior of green parts using SL-epoxy molds is an important consideration factor when considering rapid tooling for powder injection molding. The thermal exchange between the injected material and the mold during cooling would strongly influence subsequent demolding. In the case of SL-epoxy molds, relatively high injection

temperatures and subsequent large shrinkage during cooling led to the formation of a surface layer on the mold. Cohesive failure between this surface layer and the rest of the part allowed easy part removal but poor surface quality on the green part. This poor surface quality remained in the final sintered part if other measures were not taken to correct it (i.e. sanding or polishing of the final part). Lower injection temperatures, however, led to parts with good surface finish but which adhered to the mold surfaces during demolding, making it difficult to remove the cooled part from the mold.

e) Demolding analysis of hot-embossed plastic micro-structure

In the above sections, the demolding analysis of macro-scale plastic injection molded part was reviewed. In this section, some research work on the demolding analysis of micro-structures in hot embossing will be introduced.

Figure 2.29 shows the analysis of the demolding of plastic micro-structures in LIGA process using ABAQUS finite element analysis software [68]. A circular micro-structure is demolded from the nickel mold insert. The cooling mechanisms which can lead to problems during demolding were studied in terms of geometry and material properties of the micro-structures. It was shown that breakage was expected to occur at the transition from the micro-structure to the residual layer. The intensity of loading in this region depended on the thickness of the residual layer and the combination of the mold material with the substrate. Use of substrate had a negative influence on the

demolding process for thick residual layers. For thin residual layers, it turned out that the stress in the transition region is influenced by the choice of the substrate as well as by the thickness of the residual layer (h).

Figure 2.29 General sketch of geometry and boundary conditions in demolding of micro-structures [68]

A special demolding method – peel demolding and its finite element analysis were reported in the ultraviolet embossing for patterning high aspect ratio polymeric micro-structures, as shown in Figure 2.30 [101, 102].

Figure 2.30 Schematic drawing of the peel demolding in UV embossing [101, 102]

The parameters governing the demolding process were the peel angle, the pre-crack condition, shrinkage, interface fracture toughness, tensile strength and modulus of polymer. A pre-crack between the polymer and mold was introduced before peel demolding. Numerical analysis in terms of Cohesive Zone Modeling (CZM) was used to simulate the demolding process. Shrinkage caused by UV exposure was represented by thermal strain effects and the fully cured polymer was peeled off using displacement control. The ultimate tensile strength (UTS) of the cured polymer was used as a failure criterion. The stresses involved were crucial for determining clean demolding. As peeling progressed, stresses experienced in the polymer matrix increased rapidly in the region ahead of the crack with little or no stress at the cracked region. When stresses experienced by the polymer were below the ultimate tensile strength, demolding was regarded as successful.

2.5 ABAQUS finite element analysis software

In this research work, a general-purpose finite element method (FEM) software – ABAQUS was used to simulate the demolding of green micro-structures. Thus, the software is introduced in this section.

Since 1978, ABAQUS has been adopted by many major corporations across all engineering disciplines as an integral part of their design process. ABAQUS provides solutions for linear, non-linear, explicit and multi-body dynamics problems.

ABAQUS software used in this research includes:

- ABAQUS/CAE, an interactive environment used to create finite element models, submit ABAQUS analyses, monitor and diagnose jobs, and evaluate results;
- ABAQUS/Standard, a general-purpose finite element program;
- ABAQUS/Viewer, a subset of ABAQUS/CAE that contains only the post-processing capabilities of the visualization module.

Usually, the whole FEM analysis using ABAQUS includes three steps:

a) Pre-processing (ABAQUS/CAE)

In this stage the user must define the model of the physical problem and create an ABAQUS input file. The model is usually created graphically using ABAQUS/CAE or any other commercial preprocessor, although the ABAQUS input file for a simple analysis can be created directly using a text editor.

b) Simulation (ABAQUS/Standard)

The simulation, which normally is run as a background process, is the stage in which ABAQUS/Standard solves the numerical problem defined in the input file. Examples of output from a stress analysis include displacements and stresses that are stored in binary files ready for post-processing. Depending on the complexity of the problem being analyzed and the power of the computer being used, it may take anywhere from seconds to days to complete an analysis run.

c) Post-processing (ABAQUS/Viewer)

The user can evaluate the results once the simulation has been completed and the displacements, stresses, or other fundamental variables have been calculated. The evaluation is generally done interactively using ABAQUS/Viewer or any other commercial post-processor. ABAQUS/Viewer, which reads the neutral binary output database file, has a variety of options for displaying the results, including color contour plots, animations, deformed shape plots, and X–Y plots.

2.6 Debinding

After molding, the green part consists of powder particles held together by the binder phase. The powder particles are in point contact and the binder forms a continuous network throughout the part. At this juncture the concern is with removal of the binder, a step known as debinding. Some commonly used debinding methods are thermal debinding, supercritical debinding and catalytic debinding.

2.6.1 Thermal debinding

Thermal debinding appears to be the most viable method of binder removal for mass fabrication of parts because of the simplicity of method and equipment [41]. Thermal debinding involves the removal of the binder from the green part at slightly elevated temperatures. The binder may be thermally decomposed into low molecular weight species such as water, methane, carbon oxide etc and subsequently removed by diffusion in the case of low vapor pressure and small pore size or by permeation in the case of high vapor pressure and large pore sizes [103-106]. Figure 2.31 is a sketch of the pore model for debinding by permeation or diffusion [31].

Figure 2.31 Model showing binder permeation through open pore [31]

In thermal debinding there are certain defects that can be related to binder removal. The green parts may slump or deform as the binder reaches its softening point during thermal binder removal. The decomposition of the binder may cause cracks in the green parts or may lead to bloating [107]. These experiences have limited the use of conventional pyrolysis of wax or low molecular weight polymeric binder [108].

2.6.2 Supercritical debinding

Supercritical debinding using carbon dioxide as solvent was used in μ PIM [79]. This technology uses CO_2 under supercritical conditions in an autoclave, which is usually operated at temperature above 430 K and pressures above 300 bar [79]. Although supercritical CO_2 debinding fabricates satisfactory results for micro-structures with an aspect ratio of up to 1.5, further modifications will be required to obtain an economically viable process [8].

2.6.3 Catalytic debinding

The emergence of catalytic debinding with polyacetal as the main binder component makes it possible to remove the polymer vehicle from thicker section and with faster debinding rate. Catalytic debinding is a combination of solvent and thermal treatments. In catalytic debinding, the reaction depends on permeation of the catalyst vapor into the pores and permeation of the decomposition product out of the pores. In the presence of acid vapor the binder decomposes predominantly to formaldehyde well below its softening point, that is, in the solid state [109, 110]. These occur in nitrogen at atmospheric pressure and a temperature of 110 to 150 °C [111-113]. The low debinding temperature below the softening point of the binder gives the debound part good shape retention. Because poly-acetal based binder was used in this research, catalytic debinding must be used.

This polyacetal-based binder consists of a specially modified polyoxymethylene (POM) and some additives including copolymer and amorphous polymers, organic buffers, surfactants, and dispersant [114]. Polyacetal has the favorable property of depolymerizing catalytically under acidic conditions by an unzipping mechanism, yielding formaldehyde - a direct solid-gas transition, as shown in Figure 2.32 [115].

Figure 2.32 Catalytic debinding depolymerization mechanism [115]

The catalytic debinding process is performed well below the melting point of the polyacetal. This low debinding temperature prevents the formation of a liquid phase, and thus prevents deformation of the green part from gravitational distortion or stress relaxation. Since the polymer is not penetrated by the vaporized acid, the decomposition is catalyzed exclusively at the gas-binder interface. Gas exchange is limited to the already porous shell and no pressure is built up within the green body, as shown in Figure 2.33 [116, 117].

Figure 2.33 Schematic representation of the debinding mechanism [116, 117]

The binder decomposition front moves inward at a rate of 1-2 mm/h (depending on the debinding temperature, catalyst concentration, powder size and stage of debinding) making catalytic debinding about ten times faster than conventional techniques. [112-114]. Figure 2.34 illustrates the catalytic debinding process of a green part with a height of 7 mm [39]. The catalytic debinding is completed within just 150 min at a large debinding rate.

Figure 2.34 Catalytic debinding process [39]

2.7 Sintering

The final step of the μ PIM process is sintering. This is a necessary step once the binder is extracted. Pores are eliminated as part of particle bonding during high-temperature sintering. Consequently the debound part is densified to a smaller dimension with the desired composition [30, 31]. During sintering, the particulate system (debound part), which contains large amounts of free energy because of large surface area, is converted into a more stable state and a less porous body. The driving force in this “spontaneous” process is the difference in free energy between the initial and final conditions. A particle has a small stress associated with its curved surface. This surface curvature and the associated high surface area initially provide the motivation for sintering [30]. Figure 2.35 demonstrates the development of inter-particle bond during sintering [31].

Figure 2.35 Development of inter-particle bond during sintering [31]

As long as no special processes (e.g. sintering under externally applied pressure) are used, the general form of sintering consists of the following stages: non-isothermal heating to sintering temperature, isothermal sintering at sintering temperature and relatively slow cooling to room temperature. Special care must be taken to control the heating and cooling stages as they affect densification and chemical composition.

The proper selection of sintering temperature is of high importance. Higher sintering temperature gives more rapid sintering and higher sintered density [118]. In the sintering of 316L stainless steel, a low sintering temperature of 1200 °C gives low density and smaller grain. A high sintering temperature of 1300 °C coarsens the grains due to the separation of grain boundaries and pores. The medium sintering temperature of 1250 °C yields the highest density [119]. The sintering time plays an important role in the amount of shrinkage. Longer time and higher temperature will contribute to a higher sintered density. In the early stage of sintering the rate of

shrinkage is at a maximum. Any variation in the sintering time will give rise to larger shrinkage variation in the early stage. Thus the sintering time should be controlled carefully [120]. In the later stage of sintering, the shrinkage is lesser and any time variation is not critical [120]. However, long sintering time will cause grain growth and coarse pores, as well as greater energy consumption. Although porosity level decreases, the individual pore size and grain size increase.

The porous structure of debound parts reacts more readily with the surrounding atmosphere than fully dense materials. For this reason, the sintering atmosphere is very important. The protective atmospheres used are mainly gases, in special cases vacuum. Depending on the material being sintered, the sintering atmosphere must perform some or all of the following functions [30, 121]: (a) has no reaction with the material at the sintering temperature though sometimes the atmosphere is used to provide a small refinement to the chemical composition of the powder being processed, (b) excludes air from the furnace or compensate for the effect of its ingress, (c) reduces any surface oxides on the powder to improve metal to metal contact, (d) physically and/or chemically removes volatilized lubricants and binders from the debound parts and from the furnace and (e) provides a convective means of heat transfer in the furnace. For 316L stainless steel, basically six types of protective atmosphere are used, namely, hydrogen, dissociated ammonia, inert gas, nitrogen, natural gas and vacuum. There are also various combinations of the above-mentioned six atmospheres [31, 121].

Problems in sintering mainly reveal themselves either in dimensional control or adverse reaction between the part and the sintering atmosphere [122]. Table 2.3 demonstrates some typical sintering problems and possible solutions [31].

Table 2.3 Some typical sintering problems and possible solutions [31]

Problem	Source (s)	Solutions
Anisotropic shrinkage	<ul style="list-style-type: none"> • Molding • Powder packing 	<ul style="list-style-type: none"> • Eliminate flow separation • Eliminate binder or particle orientation in molding
Blisters	<ul style="list-style-type: none"> • Rapid heating 	<ul style="list-style-type: none"> • Slow heating to allow volatile species to escape prior to sintering
Cracking	<ul style="list-style-type: none"> • Heating rate • Molding • Weld line 	<ul style="list-style-type: none"> • Reduce heating rate at early portion of sintering cycle to increase part strength • Eliminate hairline cracks at same location in green part • Eliminate polymer segregation that forms hidden cracks
Distortion	<ul style="list-style-type: none"> • Residual stress 	<ul style="list-style-type: none"> • Remove long chain backbone polymer that has residual stress
Excess carbon	<ul style="list-style-type: none"> • Residual carbon 	<ul style="list-style-type: none"> • Slow heating rate • Increase atmosphere flow • Add oxidizing agent to atmosphere
Large pores	<ul style="list-style-type: none"> • Heating rate 	<ul style="list-style-type: none"> • Reduce heating rate to minimize grain growth and pore coalescence
Partial melting	<ul style="list-style-type: none"> • Debinding 	<ul style="list-style-type: none"> • Remove all carbon from part to avoid forming low melting phases
Sooting	<ul style="list-style-type: none"> • Binder decomposition 	<ul style="list-style-type: none"> • Slow heating rate to convert residual binder into vapor • Increase gas flow rate

2.8 Summary and area of research

In this chapter, the state-of-the-art of μ PIM and the problems faced by various researchers were reviewed. The successful fabrication of micro-structures by μ PIM depends on feedstock properties, injection molding, debinding and sintering as depicted in Sections 2.3 to 2.6. Figure 2.36 summaries the μ PIM process and key parameters for successful fabrication of μ PIM part.

Figure 2.36 μ PIM process and key parameters

In this research, a commercial feedstock (BASF Catamold 316L stainless steel feedstock) was used together with silicon mold insert fabricated by DRIE. As depicted in Figure 2.37, this research focuses on the design of a variotherm mold and demolding of 316L stainless steel free-standing cylindrical micro-structures array shown in Figure 2.38.

Figure 2.37 Area of research – injection molding

Figure 2.38 Array of micro-structures

As shown in Figure 2.39, silicon mold inserts fabricated by DRIE were used in this research work as high aspect ratio micro-cavities can be easily etched on silicon at a low fabrication cost due to the widespread use of silicon in various industries. Silicon has suitable material properties for mold inserts such as high strength, low thermal expansion coefficient and high thermal conductivity [123]. But, on the other hand silicon is brittle. DRIE technology allows the attainment of relatively vertical sidewall at high anisotropy and good sidewall surface roughness.

Figure 2.39 DRIE silicon mold insert showing the micro-cavities

The moldability of a part is related to how far the molten material can flow before it freezes. A high aspect ratio part can be molded easily if there is a supporting flow path.

Figure 2.40 Schematic drawing of parts (a) with supporting flow path and (b) without supporting flow path

For example, the micro-structures in part (a) and (b) have the same aspect ratio as shown in Figure 2.40, but part (a) is easier to mold due to its supporting flow path while part (b) is more difficult to mold as it is a free-standing part. The chosen pattern (b) used in this work is worse than pattern (a) in terms of geometrical/spatial configuration that inhibits complete filling. Therefore, the molding of an array of free-standing micro-structures in μ PIM is challenging because each micro-cavity has independent flow path. In this case one of the hurdles limiting the current technical capability is the freezing of the feedstock melt in the mold cavity when it contacts the cold cavity wall, resulting in incomplete filling. The problems increase when micro-structures with higher aspect ratio are molded. In μ PIM, the situation is more

pronounced because the feedstock melt has higher heat conductivity and viscosity than the polymer melt. The feedstock melt is more sensitive to heat loss. During injection molding of micro-structures, incomplete filling often occurs at the edges of the curvatures or at the front of the micro-structures [4]. In the injection molding of 316L stainless steel micro-structures array, there is incomplete filling at the flat end of the micro-cavities, which may be caused by trapped air in the micro-cavities [47].

To the author's knowledge, in-depth molding and demolding studies of high aspect ratio metal micro-structures for μ PIM have not been reported by other researchers.

This research aims at seeking answers to:

1. the highest possible aspect ratio moldable using a conventional mold (i.e. not variotherm mold),
2. the highest possible aspect ratio moldable using variotherm mold,
3. establish suitable processing conditions for items 1 and 2,
4. understand the demolding mechanism of micro-structures by theoretical analysis (using ABAQUS FEM software), e.g. where and when the breakage of micro-structures may take place and how packing pressure and demolding temperature affect the demolding force,
5. establish suitable demolding conditions based on the theoretical analysis and conduct experimental verifications.

CHAPTER 3

MATERIAL PROPERTIES AND EXPERIMENTAL EQUIPMENT

A commercially available 316L stainless steel feedstock was used in this research. The characterization of the feedstock included a series of density, thermal, rheological and mechanical tests. To determine suitable injection molding conditions for the feedstock in Chapter 4 and 5, differential scanning calorimetry (DSC) testing, thermogravimetric analysis (TGA) and capillary rheometer testing were conducted. To collect the necessary material properties for the demolding analysis in Chapter 6, thermomechanical analysis (TMA), pressure-volume-temperature (PVT) testing, tensile test, tensile adhesion strength testing between the feedstock and silicon wafer and coefficient of friction testing between the feedstock and silicon wafer were conducted. The main equipment used in this research are also described.

3.1 Experimental materials and characterization

3.1.1 Feedstock

BASF Catamold 316L austenitic stainless steel feedstock shown in Figure 3.1 is supplied by BASF AG of Germany. The 316L stainless steel powder has a particle size D_{50} of 4 μm . A polyacetal-based binder is used. The binder consists of the widely used thermoplastic polymer, polyoxymethylene (POM) as the main component, and some other additives in small quantities including copolymers and amorphous

polymers, organic buffers, surfactant and dispersant [114, 124].

Figure 3.1 BASF 316L stainless steel feedstock

3.1.2 Density of feedstock

A Quantachrome ultrapycnometer Model 1000 (Quantachrome Japan Science and Technology Corporation) shown in Figure 3.2, measures the volume and true density of solid objects. This is accomplished by employing Archimedes' principle of fluid displacement to determine the volume. The displaced fluid used in the ultrapycnometer was helium, which can penetrate the finest pores thereby assuring maximum accuracy. The measured density ρ of the feedstock is 5.547 g/cm^3 .

Figure 3.2 Quantachrome ultrapycnometer (Model 1000)

3.1.3 Differential scanning calorimetry (DSC) testing

Differential scanning calorimetry (DSC) is based on the fact that a transition such as crystallization is associated with a change in the thermal properties of the sample under study. This change can be identified when comparing the sample with a reference. By monitoring the temperature difference between the sample and the reference, or by measuring the energy input necessary to maintain both the sample and the reference at the same temperature, one can determine the location where the transition occurs and also the type and magnitude of this transition [125]. Thus, the melting and crystallization behavior of the feedstock can be investigated by means of DSC testing.

The DSC testing was conducted on a TA-2920 differential scanning calorimeter made by TA Instruments of USA. The temperature range was between 30 °C and 200 °C. Heating and cooling rate of 10 °C/min was used. The purging gas was a dynamic flow of purified nitrogen gas. Figure 3.3 shows the DSC plot of the feedstock. One

endothermic peak of 165.37 °C is seen during heating which is due to the melting of the binder. The melting temperature range is 161.26 °C to 169.43 °C. An exothermic peak at 144.36 °C is also seen during cooling, which is due to the crystallization of the polyacetal-based binder. The crystallization temperature range is 139.43 °C to 146.52 °C.

Figure 3.3 DSC curve of feedstock

3.1.4 Thermogravimetric analysis (TGA)

Thermogravimetric analyzer measures weight changes in materials upon heating as a function of temperature or time. This allows the accurate quantitative analysis of thermal reactions such as decomposition that are accompanied by mass changes. The change in weight of a sample is measured under isothermal conditions or under heating – usually a linear temperature program i.e. at constant heating rates.

TGA uses a balance where on one side of the balance, the sample is placed on a pan and the pan is suspended in the furnace. The other side of the balance holds additional weight to offset the mass of the apparatus. The deflection of the scale balance during the heating process is compensated automatically by a repelling force from an electromagnetic coil. The change in the current of the coil is directly proportional to the change in the weight of the sample. The balance and sample chamber can be filled with inert or reactive gas such as nitrogen.

A Perkin Elmer thermogravimetric analyzer (TGA) Model 7 was used to determine the decomposition temperature of the binder and powder loading of the feedstock. The test was conducted in the temperature range of 50 °C to 550 °C at a heating rate of 10 °C/min with argon purging. Figure 3.4 shows the TGA curve of the binder components in the feedstock.

Figure 3.4 TGA curve of feedstock

The polyacetal-based binder starts to degrade at about 215.3 °C and completes its degradation at about 487.5 °C. From 215.3 °C to 355.3 °C, there is only a weight loss of about 0.4 %. After 355.3 °C there is a sharp increase in the weight loss from about 0.4 % to 7.5 % at 424.8 °C, which results from the degradation of the main binder component (polyacetal). From 424.8 °C to 487.5 °C, there is a small weight loss, which is due to the degradation of other components in the binder.

To estimate the powder loading of the commercial feedstock, three samples were taken randomly from the feedstock for TGA testing. The weight left after burning at 550 °C is tabulated in Table 3.1. From the results, the estimated powder loading is 91.93 wt.%.

Table 3.1 Weight left after burnout test

Weight left (wt. %)			Average (wt.%)	Standard Deviation
1	2	3		
91.916	91.928	91.946	91.93	1.51×10^{-2}

The DSC and TGA analysis results of the feedstock are summarized in Table 3.2. According to the results, suitable molding temperature should be set within the range of 165.37 °C to 215.0 °C. And, the demolding temperature should be below the crystallization temperature of 144.36 °C.

Table 3.2 DSC and TGA results of the feedstock

Parameter	Value
Peak melting temperature (°C)	165.37 (range from 161.26 to 169.43)
Start degradation temperature (°C)	215.0
Peak crystallization temperature (°C)	144.36 (range from 139.43 to 146.52)

3.1.5 Rheological characteristics of feedstock

Having determined the molding temperature range of the feedstock, the next step was to establish the rheological behavior of the feedstock. Feedstock melts are usually of viscoelastic characteristics and exhibit pseudoplastic behavior [31, 41]. Accordingly, the viscosity must be measured over a range of conditions that are expected in practical molding. Capillary rheometry is very useful in characterizing PIM feedstock since it matches well the range of viscosities encountered in practice, although usually the peak molding shear rate exceeds the range obtainable with a rheometer [126-128].

The rheometer measures the pressure drop associated with a forced flow rate through a small capillary tube. One of the most widely used constitutive equations is the power law model, proposed by Ostwald and de Waele [129] given by $\tau = k_c \cdot (\dot{\gamma})^n$ where, τ (Pa) is the shear stress, $\dot{\gamma}$ (s^{-1}) is the shear rate, k_c ($Pa \cdot s^n$) is the consistency and n is flow index. For $n = 1$ the Ostwald de Waele model reduces to Newton's law

of viscosity with k_c being the viscosity. An apparent viscosity is defined as the local value corresponding to a newtonian behavior represented as $\tau = \eta_a \cdot \dot{\gamma}$. Then, the power law equation can be written as $\eta_a = k_c \cdot (\dot{\gamma})^{n-1}$.

For capillary rheometer using round capillary [130]:

$$\text{Apparent shear rate } \dot{\gamma}_a = \frac{32 \cdot q}{\pi D^3} \text{ and}$$

$$\text{Apparent viscosity } \eta_a = \frac{\pi \cdot D^4 \cdot p}{128 L_d \cdot q}$$

where,

q = flow rate

p = pressure

D = die diameter

L_d = die length

A double logarithmic plot of the apparent wall viscosity against apparent wall shear rate shows the rheological characteristics of the melt. Usually apparent values such as apparent wall viscosity and apparent wall shear rate are sufficient for such applications as quality control and test comparison. In cases where more accurate results are necessary, correction should be carried out. Two correction methods are widely used. One is the Bagley correction [131], another is Weissenberg-Rabinowitsch correction [130]. The Bagley correction takes into account the entrance/exit effect which negatively influences the viscosity-related pressure loss. The pressure measured in a capillary rheometer is the sum of the pressure drop through the capillary itself and the entrance pressure drop necessary to force the

material to flow from the large barrel into the capillary. The entrance pressure drop can be evaluated by making measurements using several capillaries of different length to diameter ratio according to Bagley [131]. The Weissenberg-Rabinowitsch correction takes into account the non-Newtonian behavior of the melt. This correction changes the apparent shear rate to true shear rate and results in a corrected viscosity curve. In this research, both the Bagley correction and the Weissenberg-Rabinowitsch correction were used.

The rheological behavior of the feedstock was investigated on a Göttfert Rheograph 6000 capillary rheometer (Göttfert GmbH, Buchen Germany) shown in Figure 3.5.

Figure 3.5 Capillary rheometer and capillary die

The capillary rheometer measures the pressure drop associated with a forced flow rate through a small capillary tube. It consists of a heating barrel whereby the loaded sample feedstock is melted. When the pre-set temperature of the feedstock inside the

barrel is reached, the feedstock melt is forced through the round die hole at different shear rates of $200 \text{ s}^{-1} \sim 4000 \text{ s}^{-1}$. After the whole process ends, the apparent viscosities of the feedstock at different shear rates are measured and calculated automatically. The diameter of the barrel and plunger is 12 mm. The diameter of the capillary die is 1 mm. To incorporate Bagley correction, three capillary dies are used whose dimensions are $\phi 1 \text{ mm} \times \text{length } 10 \text{ mm}$, $\phi 1 \text{ mm} \times \text{length } 20 \text{ mm}$ and $\phi 1 \text{ mm} \times \text{length } 30 \text{ mm}$ respectively. After Bagley correction, Weissenberg-Rabinowitsch correction was also conducted. Figure 3.6 shows the plot of viscosity after Bagley and Weissenberg-Rabinowitsch correction against shear rate at different temperatures.

Figure 3.6 Viscosity-shear rate relationship of feedstock

Four different temperatures of 170 °C, 180 °C, 190 °C and 200 °C were selected for the rheological test. The temperatures fall between the peak melting temperature of 165.37 °C and the start degradation temperature of 215 °C. Figure 3.6 shows that the

viscosity decreases with increasing shear rate for all temperatures, indicating pseudoplastic (shear thinning) behavior. Further, the viscosity increases as the temperature decreases. It has been reported that during injection molding the shear rate usually ranges between 10^2 and 10^5 s^{-1} [31]. In this range, the maximum useful viscosity for the feedstock melt at the molding temperature is $10^3 \text{ Pa}\cdot\text{s}$ [31, 132]. At the temperatures of 190°C and 200°C , the viscosity of the feedstock melt is less than $10^3 \text{ Pa}\cdot\text{s}$ in the shear rate range of 2×10^2 to $4 \times 10^3 \text{ s}^{-1}$. The melt temperature of 190°C was selected for injection molding as it could provide low viscosity (good moldability) and avoid binder degradation.

3.1.6 Thermomechanical analysis (TMA)

The mechanical behavior of the feedstock as a function of temperature can be examined with the aid of a thermomechanical analyzer. The main interest of TMA is the thermomechanical stability (softening point) [125]. Generally, TMA measures the dimensional changes of sample during heating or cooling.

Coefficient of thermal expansion (CTE) is the ratio of (a) the change in length of a line segment in a material body per unit of temperature change to (b) its length at a reference temperature. The CTE parameter was used to analyze the stress distribution of the micro-structures during the cooling and shrinking of the green part. To obtain the coefficient of thermal expansion of the feedstock, a TMA-2940 system made by TA Instruments of USA was used. Rectangular specimens were cut from the gage

section of a tensile bar. The cut surface was polished with fine sandpaper to achieve good flatness. The specimens were cut from three directions of the tensile bar: along the length of the tensile bar, the width of the tensile bar and the thickness of the tensile bar, as shown in Figure 3.7.

Figure 3.7 Tensile bar and the three testing directions

The tests were conducted in nitrogen gas, in the temperature range of 30 to 150 °C, with a heating rate of 5 °C/min. The dimensional change of the specimen was measured using a probe. The TMA curves are shown in Figure 3.8. The CTE is represented by the slope of TMA curve.

Figure 3.8 TMA curves for different testing directions

The coefficients of thermal expansion in the three different directions are shown in Table 3.3. It shows that there is a slight deviation in the CTE values tested in the width and the thickness directions. However, more than 10 % deviation between the CTE value tested in the length direction and the other two directions is noted. As the longitudinal direction is the direction where the feedstock flowed into the cavity during mold filling, it has more metal powder and less polymeric binder. The CTE of the metal powder is lower than that of the polymer; therefore, the powder-rich longitudinal direction has a lower CTE. The average CTE value is $48.7 \times 10^{-6}/^{\circ}\text{C}$.

Table 3.3 The coefficients of thermal expansion of different directions

Direction	Longitudinal	Width	Thickness	Average
Coefficient of thermal expansion ($10^{-6}/^{\circ}\text{C}$)	43.7	50.1	51.3	48.7

3.1.7 Pressure-volume-temperature (PVT) testing

The dependence of density of the material on pressure and temperature is called the thermodynamic material behavior or PVT behavior. Traditionally, instead of the density, the reciprocal value of the density, specific volume, is used. Knowledge of PVT diagram is important to understand the physical process in the mold cavity during injection molding, especially during the packing pressure phase. Also, it is important for understanding the characteristics of the final green part, particularly shrinkage and warpage [53].

The essential component of PVT testing is a measuring cell, into which a weighed quantity of the material is charged. The material can be heated or cooled via the surrounding cylinder wall. A pressure transducer mounted below the measuring cell measures the pressure. Above the cell, a hydraulic piston applies a range of pressure to the material. The piston displacement is measured at the same time, so that the enclosed volume can be determined as a function of pressure and temperature. The PVT interrelationship can be determined from the changes in piston pressure and temperature in the material.

Pressure-volume-temperature (PVT) test was conducted on a PVT100 tester manufactured by SWO Polymertechnik GmbH (Krefeld, Germany). A schematic drawing of the device is shown in Figure 3.9 [133].

Figure 3.9 Schematic drawing of the PVT cell [133]

The feedstock was put in the cylindrical cell, and the specific volume was deduced from the measured length of the feedstock. The weight of the feedstock is 3.98 g. The diameter of the cell is 7.4 mm. The lower piston is fixed, and the upper piston can move, ensuring the pressure. Between each piston and the feedstock is a PTFE sealing to avoid leakage. The measurement of the upper piston displacement provides the length of the feedstock after subtracting the sealing volume. The temperature control of the cell is ensured by an electric heater band, and is air cooled. The temperature is measured using a thermocouple placed very near to the feedstock. Moreover, the cell is mounted on springs allowing relative movements between the cell and the pistons. In this way, the pressure is always applied on both sides of the feedstock, even if it is stuck on to the cell (all the normal force is transmitted to the lower piston).

The specific volume - temperature curves expressed in isobaric condition at five different pressures are shown in Figure 3.10.

Figure 3.10 PVT curves of the feedstock

For the theoretical demolding analysis in Chapter 6, the PVT relationship for the feedstock is expressed as a two-dimensional curve fit relating pressure and temperature. In the curve fitting, the modified double-domain Tait PVT model was used. The model is used to account for material compressibility during injection molding simulation. The compressibility of a material affects the volume of feedstock required. The Modified Double-Domain Tait PVT model is given by Equation 3.1 [134]:

$$v(T, p) = v_0(T) \left\{ 1 - C \ln \left(1 + \frac{p}{B(T)} \right) \right\} + v_t(T, p) \quad (3.1)$$

Where,

$v(T, p)$ = the specific volume at temperature T and pressure P

$v_0(T)$ = the specific volume at zero gauge pressure

p = pressure

T = temperature of the feedstock

$C = 0.0894$ (universal constant)

$B(T)$ accounts for the pressure sensitivity of the feedstock

For $T > T_i$ ($T_i = b_5 + b_6 p$ = transition temperature of the feedstock at pressure p , b_5 and b_6 are data-fitted coefficients. As polyacetal binder is semi-crystalline, its transition temperature at atmospheric pressure is the upper limit of the crystallization temperature range as measured in the DSC test)

$$\begin{cases} v_0(T) = b_{1m} + b_{2m} \bar{T} & (\text{where } \bar{T} = T - b_5) \\ B(T) = b_{3m} e^{-b_{4m} \bar{T}} \\ v_i(T, p) = 0 \end{cases}$$

(b_{1m} , b_{2m} , b_{3m} and b_{4m} are data-fitted coefficients)

The upper region of the curve ($T > T_i$) can be described by Equation 3.2:

$$v(T, p) = (b_{1m} + b_{2m} \bar{T}) \left\{ 1 - C \ln \left(1 + \frac{p}{b_{3m} e^{-b_{4m} \bar{T}}} \right) \right\} \quad (3.2)$$

For $T < T_i$

$$\begin{cases} v_0(T) = b_{1s} + b_{2s} \bar{T} \\ B(T) = b_{3s} e^{-b_{4s} \bar{T}} \\ v_i(T, p) = b_7 e^{(b_8 \bar{T} - b_9 p)} \end{cases}$$

(b_{1s} , b_{2s} , b_{3s} , b_{4s} , b_7 , b_8 and b_9 are data-fitted coefficients)

The lower region of the curve ($T < T_i$) can be described by Equation 3.3:

$$v(T, p) = (b_{1s} + b_{2s} \bar{T}) \left\{ 1 - C \ln \left(1 + \frac{p}{b_{3s} e^{-b_{4s} \bar{T}}} \right) \right\} + b_7 e^{(b_8 \bar{T} - b_9 p)} \quad (3.3)$$

After mathematical curve fitting using Datafit software developed by Oakdale Engineering of USA, the PVT coefficients of the feedstock are summarized in Table 3.4. These results are used in the theoretical analysis in Chapter 6.

Table 3.4 PVT curve fitting coefficients for the feedstock

$b_{1m} = 1.87 \text{ e-}4 \quad \text{m}^3/\text{kg}$	$b_{1s} = 1.81744 \text{ e-}4 \quad \text{m}^3/\text{kg}$	$b_5 = 422 \text{ K} \quad (149 \text{ }^\circ\text{C})$
$b_{2m} = 4.1538 \text{ e-}8 \quad \text{m}^3/\text{kg}\cdot\text{K}$	$b_{2s} = 2.93 \text{ e-}8 \quad \text{m}^3/\text{kg}\cdot\text{K}$	$b_6 = 1.5 \text{ e-}7 \quad \text{K/Pa}$
$b_{3m} = 4.2543 \text{ e}8 \quad \text{Pa}$	$b_{3s} = 6.391 \text{ e}8 \quad \text{Pa}$	$b_7 = 4.8107 \text{ e-}6 \quad \text{m}^3/\text{kg}$
$b_{4m} = 0.003096 \quad 1/\text{K}$	$b_{4s} = 0.00255 \quad 1/\text{K}$	$b_8 = 0.1237 \quad 1/\text{K}$
		$b_9 = 2.0466 \text{ e-}8 \quad 1/\text{Pa}$

3.1.8 Mechanical properties

Tensile test was adopted for the measurement of the mechanical properties. Tensile tests were performed on an Instron 5500 tensile testing machine equipped with a thermal chamber as shown in Figure 3.11.

Figure 3.11 Instron tensile tester 5500 with thermal chamber

The tensile bars shown in Figure 3.7 were injection molded on a Battenfeld injection molding machine described in Section 3.2.1. As the mechanical properties of the feedstock are dependent on the temperature, tensile tests were conducted at the temperatures of 20 °C, 45 °C, 65 °C, 85 °C, 105 °C, 125 °C and 140 °C. Three tests were conducted at each temperature. The tensile rate was set at 2 mm/s, which is the demolding speed used during injection molding of the micro-structures. The true stress-strain curves are shown in Figure 3.12. The mechanical properties of the feedstock are summarized in Table 3.5.

Figure 3.12 True stress-strain curves

Table 3.5 Mechanical properties of the feedstock

Temperature (°C)	20°C	45°C	65°C	85°C	105°C	125°C	140°C
Young's Modulus (GPa)	1.47	1.31	1.26	0.98	0.60	0.39	0.27
Ultimate tensile strength (MPa)	6.66	5.71	5.03	4.28	3.48	2.74	1.91
Stress at fracture (MPa)	6.15	5.11	4.47	3.89	3.30	2.22	1.60
Fracture Strain	8.1%	12.8%	14.8%	21.6%	52.1%	58.1%	66.6%

Poisson's ratio ν is defined as the absolute value of the ratio of transverse strain to the corresponding longitudinal strain, resulting from uniformly distributed longitudinal stress below the proportional limit of the material. Four tensile tests were performed with strain gages specially mounted on the gage sections of each tensile bar prior to testing. Longitudinal strain is the linear strain in a plane parallel to the longitudinal axis of the tensile bar while transverse strain is the linear strain (in the width direction) in a plane perpendicular to the axis of the tensile bar. The longitudinal and transverse strains (in the elastic region) are sensed by the strain gages and recorded by a data logger. The calculated Poisson's ratio ν ranges from 0.252 to 0.323, giving an average of 0.274.

3.1.9 Tensile adhesion strength between the feedstock and silicon wafer

In the theoretical analysis of the demolding of green micro-structures in Chapter 6, the adhesive force and frictional force between the green micro-structures and the inner surfaces of the micro-cavities in the silicon mold insert were taken into account. Thus, tensile adhesion strength between the feedstock and silicon wafer, and coefficient of friction between the feedstock and the silicon wafer were measured and presented in Sections 3.1.09 and 3.1.10.

The set-up used to measure the tensile adhesion strength is shown in Figure 3.13. A $\phi 4$ inch silicon wafer was put on the plate of a hot press. The silicon wafer was etched in the DRIE (deep reactive ion etching) machine in order to achieve a surface roughness of $R_a = 32$ nm. This surface roughness was the same as the bottom surface of the $\phi 100$ $\mu\text{m} \times$ depth 200 μm micro-cavity in the silicon mold insert used in the injection molding experiments discussed in Chapters 4 to 6. A cylindrical chamber with inner diameter of $\phi 25$ mm was put on the silicon wafer. 24.5 g of feedstock was poured into the chamber. Then the set-up was heated to 170 °C to melt the feedstock. The feedstock was compacted and held for 1 minute in the heated chamber. The applied forces were 500 N, 1000 N and 2000 N. Force larger than 2000 N could break the brittle silicon wafer. After the compacted circular part was cooled to a specific temperature (20 °C, 40 °C, 60 °C, 80 °C, 100 °C, 120 °C and 140 °C), the force needed to detach the compacted circular part from the silicon wafer at the specific temperature was measured by an Instron tensile testing machine (Model 5569). The

results are shown in Figure 3.14. The adhesion tensile strength is lower than 0.02 MPa.

This peak value of 0.02 MPa is used in the FEM analysis in Chapter 6.

Figure 3.13 Set-up for tensile adhesion strength measurement

Figure 3.14 Tensile adhesion strength between the feedstock and the silicon

3.1.10 Coefficient of friction between the feedstock and silicon wafer

The set-up for measuring the coefficient of static and sliding friction between the feedstock and the silicon wafer is shown in Figure 3.15. A $\phi 32$ mm $\times$ thickness 3 mm disc was injection molded using the feedstock. This disc was put on a $\phi 4$ inch silicon wafer secured on the support plate for friction test. To investigate the effect of the surface roughness on the coefficient of friction, etched and polished $\phi 4$ inch silicon wafers with different surface roughness ($R_a = 4, 12, 30, 32$ and 500 nm) were used in the measurement. The result is shown in Figure 3.16. For the surface roughness range of $R_a = 4$ to 500 nm, the coefficient of friction between the feedstock and silicon wafer is not sensitive to the surface roughness. The coefficient of static friction μ_s is 0.66. The coefficient of sliding friction μ_d is 0.37. As the surface roughness of the inner frictional surface of the micro-cavity in the silicon mold insert is $R_a = 200$ nm, which is in the range of $R_a = 4$ to 500 nm, these values are used in the FEM analysis discussed in Chapter 6.

Figure 3.15 Set-up for coefficient of friction measurement

Figure 3.16 Coefficient of static and sliding friction

3.2 Main experimental equipment

3.2.1 Injection molding machine

When the development of micro metal injection molding started in the late eighties, no appropriate machine technology was available [135]. Therefore, only modified commercial injection molding machines, hydraulically driven and with a clamping force of usually 25-50 tons, were used for the fabrication of microparts or micro-structures using microstructured mold inserts with high aspect ratios. In this research, green parts with micro-structures were molded on a conventional injection molding machine, a Battenfeld BA 250CDC. The machine, shown in Figure 3.17, has a clamping force of 250 kN and an injection screw of $\phi 18$ mm.

Figure 3.17 Battenfeld BA 250 CDC injection molding machine

Table 3.6 summarizes some important features of the Battenfeld BA 250CDC injection molding machine [136]. There are four heating zones on the barrel: nozzle / zone 1 /zone 2 /zone 3, as shown in Figure 3.18.

Figure 3.18 Schematic drawing of injection molding machine

Table 3.6 Features of Battenfeld BA 250CDC injection molding machine

Description	Unit	Data	Description	Unit	Data
Screw diameter	mm	18	Screw torque	Nm	190
Specific injection pressure	bar	2760	Metering capacity	g/s	6.5
Theoretical stroke volume	cm ³	9.2	Injection rate into the open	cm ³ /s	47.1
Max. Injection weight (PE)	g	6.5	Number of heating zone		1+3
L/D ratio of screw		17.5	Ejector force	kN	17.6
Nozzle path	mm	150	Ejector stroke	mm	100
Max screw revolution	1/min	410			

The melt temperature setting consists of four temperatures corresponding to the four temperature zones. The injection molding machine uses progressive injection profile to achieve constant flow front velocities during the filling stage. As shown in Figure 3.19, the injection movement of the screw is divided into five sections (speed 1 / speed 2 / speed 3 / speed 4 / speed 5) [136]. The specific speed value and corresponding stroke value can be adjusted according to the volume and shape of the part. The injection speed setting is expressed as a percentage of the maximum allowable injection speed of 47.1 cm³/s. Even though the injection speeds at different sections are different, constant injection pressure is maintained as shown in Figure 3.19.

Figure 3.19 Flow chart of injection movement of injection screw

3.2.2 Mold insert

The DRIE silicon mold insert used in this research consists of 24×24 (576) micro-cavities. The micro-cavities were etched on an STS Deep Reactive Ion Etcher equipped with a high-density inductively coupled plasma source (ICP). With the ICP plasma source, very high aspect ratio micro-cavity with vertical profile can be achieved [137-139]. The DRIE process involves alternate etching and surface passivation. Etching gas SF_6 is introduced into the chamber (where plasma is generated) to etch the silicon. Passivation gas C_4F_8 is introduced to passivate the sidewall in order to protect the sidewall from being etched during the next etching.

Thus, a micro-cavity with a specific depth was fabricated by alternate etching and passivation.

After the micro-cavities were etched in standard $\phi 4$ inch wafer, the wafer was then cut by a dicing machine into smaller square dimensions of $5\text{ mm} \times 5\text{ mm}$. The cut square silicon was used as mold insert. Thus, the dimensions of the mold insert are $5\text{ mm} \times 5\text{ mm} \times 0.5\text{ mm}$ with 24×24 (576) micro-cavities. Figure 3.20 shows the 3-dimensional schematic drawing of the silicon mold insert with the micro-cavity dimensions of $\phi 100\text{ }\mu\text{m} \times \text{height } 200\text{ }\mu\text{m}$, giving an aspect ratio of 2.

Figure 3.20 Silicon mold insert with micro-cavities

A JSM-5600LV scanning electron microscope was used to observe the silicon mold insert and green micro-structures. Figure 3.21 and Figure 3.22 show the top view of the silicon mold insert and the cross-sections of the micro-cavities.

Figure 3.21 Top view and cross-section of micro-cavities fabricated by DRIE ($\phi 100 \mu\text{m}$)

Figure 3.22 Top view and cross-section of micro-cavities fabricated by DRIE ($\phi 60 \mu\text{m}$)

Due to the fragile property of silicon, it is not easy to fix the silicon mold insert onto the steel mold cavity. Hence, super glue was used initially to stick the silicon mold insert onto the surface of the mold cavity. But, the silicon mold insert was detached from the mold cavity as the glue is not able to withstand the high mold temperature of 125°C . A high mold temperature is required due to the use of polyacetal-based feedstock. Finally, a commercial adhesive (Wesbond silicone adhesive) that is able to withstand a high mold temperature of 150°C was used. The mold cavity surface and

the silicon insert were first cleaned using acetone to remove contamination that may weaken the bond between the mold cavity surface and the silicon mold insert. Then a small amount of adhesive was put on the back surface of the silicon mold insert. The silicon mold insert was lightly and evenly pressed against the mold cavity surface in order to form an even thin film. After that, the mold cavity and the silicon mold insert were kept at room temperature for 24 hours till good bonding strength was achieved. Experiments show that the number of injection molding cycles feasible before the silicon mold insert detaches itself from the mold cavity is highly dependent on the cleaning quality of the surfaces and how the silicon mold insert was used.

For example, if the silicon mold insert was used at 125 °C till the insert detached itself from the mold cavity, a maximum of 120 molding cycles per insert has been observed. On the other hand, if the silicon mold insert was used for some cycles and unused for several days, and then put into use again, the total number of feasible molding cycles is reduced. This is probably due to the expansion and contraction of the thin glue film.

3.2.3 Catalytic debinding oven

A gas-tight, acid-resistant and forced convection oven was used for debinding. Its photo and schematic working principle is shown in Figure 3.23. It is designed and fabricated by Heraeus Instruments GmbH (Germany). The model is VT6060 MU-2. In the debinding oven, the binder is depolymerized by means of gaseous catalyst (100% nitric acid). The depolymerization products are immediately incinerated in a natural gas flare.

The oven is first purged with an inert gas (nitrogen) and heated to operating temperature, and a continuous flow of inert gas is maintained. The catalyst is metered into a pan in the oven, where it evaporated. The gases are circulated to ensure uniform debinding of all the green parts in the oven.

Figure 3.23 Catalytic debinding oven and the schematic debinding principle [116]

The oven was heated and flushed with nitrogen at a pressure of 2 bar to avoid an explosive atmosphere during debinding and at the same time avoiding excessive

surface oxidation of sensitive powders. The flow rate of nitrogen was set at 500 l/h. After reaching the process temperature of 120 °C, the catalyst (100 % nitric acid) was metered into the hot purge gas. The concentration of the gaseous nitric acid ranges from 0.1 to 5vol.%, depending on the material (metal or ceramic) and the loading of the oven [116]. In this study, the acid rate was set 35 ml/h. The debinding rate was controlled by temperature and catalyst concentration. The process does not need complicated temperature profile and was not sensitive to small fluctuations of the temperature and gas flow. The exhausted gas, mainly formaldehyde in the nitrogen carrier gas was burnt with excess air.

3.2.4 Sintering furnace

A laboratory furnace (Model: 1730-12 HTF) manufactured by CM Furnaces Inc. was used to sinter the debound part. The furnace uses a horizontal ceramic tube as shown in Figure 3.24. It is able to run continuously at a maximum temperature of 1650 °C. An over-temperature safety device is used to protect the furnace against any damage due to over-heating. Its standard control instrumentation included a Eurotherm multiple segment programmable microprocessor used in conjunction with a B-Type thermocouple. A circulating water cooling system with a pump and hoses is used to cool the stainless steel covers of the ceramic tube during high-temperature sintering. The furnace is equipped for vacuum and inert atmosphere.

Figure 3.24 Tube sintering furnace

3.3 Summary

In this Chapter, a series of characterization experiments of the feedstock were conducted and the main experimental equipment was introduced. The experiments included density test, DSC test, TGA test, capillary rheometer test, TMA test, PVT test, tensile test, tensile adhesion strength test and the measurement of coefficient of friction between the feedstock and silicon wafer. The properties of the feedstock are summarized in Table 3.7.

Table 3.7 Properties of the feedstock

Parameter	Value						
Density (g/cm ³)	5.547						
Powder loading by weight (%)	91.93						
Peak melting temperature (°C)	measured				Supplied by supplier		
	165.37 (range from 161 to 168)				165 (from [140])		
Peak crystallization temperature (°C)	144.36 (range from 140 to 149)						
Start degradation temperature (°C)	215						
Melt temperature in injection molding (°C)	190 (from [140])						
Coefficient of linear expansion (10 ⁻⁶ /°C)	48.7						
PVT curve fitting coefficients of the feedstock in modified double-domain Tait model	b _{1m} =1.87 e-4 m ³ /kg		b _{1s} = 1.81744 e-4 m ³ /kg		b ₅ =422 K (149 °C)		
	b _{2m} =4.1538 e-8 m ³ /kg·K		b _{2s} =2.93 e-8 m ³ /kg·K		b ₆ =1.5 e-7 K/Pa		
	b _{3m} =4.2543 e8 Pa		b _{3s} =6.391 e8 Pa		b ₇ = 4.8107 e-6 m ³ /kg		
	b _{4m} = 0.003096 1/K		b _{4s} =0.00255 1/K		b ₈ = 0.1237 1/K		
					b ₉ = 2.0466 e-8 1/Pa		
Young's Modulus (GPa)	20°C	45°C	65°C	85°C	105°C	125°C	140°C
	1.47	1.31	1.26	0.98	0.60	0.39	0.27
UTS (MPa)	6.66	5.71	5.03	4.28	3.48	2.74	1.91
Stress at fracture (MPa)	6.15	5.11	4.47	3.89	3.30	2.22	1.60
Fracture strain	8.1%	12.8%	14.8%	21.6%	52.1%	58.1%	66.6%
Poisson's ratio	0.274						
Tensile adhesion strength between the feedstock and silicon wafer (MPa)	0.02						
Coefficient of friction between the feedstock and silicon wafer	Coefficient of static friction μ _s				Coefficient of sliding friction μ _d		
	0.66				0.37		

CHAPTER 4

μ PIM WITHOUT VARIOTHERM MOLD

In this chapter, μ PIM using a conventional mold (instead of a variotherm mold) was used to fabricate 316L stainless steel micro-structures for comparison with μ PIM using variotherm mold. As a commercial BASF Catamold 316L stainless steel feedstock was used in this research, only three processing steps (injection molding, debinding and sintering) were conducted.

4.1 Injection molding study

In order to obtain visually defect-free free-standing green micro-structures array, it is important to understand the injection molding process and to establish suitable injection molding parameters systematically. Examples of injection molding parameters are injection pressure, injection speed, packing pressure, packing time, melt temperature and mold temperature [141-143]. Potential defects such as incomplete filling, powder-binder separation, warpage and fracture can be due to inappropriate selection of injection molding parameters. Further, molding defects can propagate in subsequent processing steps. Hence, this study provides a systematic approach to the selection of suitable injection molding parameters.

4.1.1 Mold design

The green part is a round disc with a base of $\phi 16$ mm and thickness 1.5 mm, and an array of micro-structures in the center of the disc. Edge gate and runner with modified trapezoidal cross-section were used. A silicon mold insert of dimensions 5 mm $\times$ 5 mm $\times$ 0.5 mm was mounted on the movable half of the mold cavity. Figure 4.1 shows the dimensions of the runner, gate and cavity. Figure 4.2 shows the green part, with an array of 24 $\times$ 24 (576) micro-structures in the center area.

Figure 4.1 Runner, gate and cavity of mold design

Figure 4.2 Green part with an array of micro-structures

4.1.2 Effects of molding parameters

In the following sub-sections, the effects of injection molding parameters on the complete filling of the mold cavity will be discussed. Six sets of experiments were conducted by varying one parameter at a time: injection speed, injection pressure, packing pressure, packing time, melt temperature and mold temperature. When a parameter was changed from one value to another, the injection machine was stabilized for 10 minutes before injection molding was conducted. After changing the parameter, the first five parts were not used for evaluation so as to obtain more representative parts. And for each set of parameter, five green parts were collected for analysis.

a) Injection stroke and speed profile

In the first part of the study, the injection stroke profile was determined so as to ensure complete filling. The mold filling phase designates the period of time during which the feedstock melt moves from the machine nozzle to the extremities of the cavity, filling the cavity completely. Normally, the injection speed profile allows constant flow front velocities to be achieved [144]. During the injection molding of the microstructures, high injection speed (resulting in less temperature loss and reducing shrinkage potential) was used as the silicon mold insert needs to be filled rapidly to prevent early freezing [73]. Further, increasing the flow rate decreases the amount of residual stress, because the feedstock can fill the mold quickly and the binder can relax before solidification occurs [72, 145]. Consequently, the initial injection speed profile was set as 80 / 80 / 80 / 80 / 80 (%) of max. injection speed (186 mm/s) stated in the manual [136]. This corresponds to 149 / 149 / 149 / 149 / 149 (mm/s).

As five speeds are available in the BA 250 CDC molding machine, the material may flow with different speeds at different portions of the cavity volume. Flow in non-critical sections was set high while slow filling was preferred in sections that are prone to defects or where there is a sudden change in mold geometry. Hence, the flow was divided into five stages according to the flow path illustrated in Figure 4.3.

Figure 4.3 Injection speed profile based on different filling stage

Stage one involves flow in the sprue. Stage two involves flow till cold slug well. Stage three involves flow in the runner till the start of the gate. Stage four involves flow in the mold cavity till where the mold insert area is covered by the feedstock melt and stage five involves flow in the remaining volume of the cavity. The injection stroke profile was set at 21 / 17 / 13 / 11 mm after approximate calculation of the weight percentage of every flow stage.

With the initial injection speed profile of 80 / 80 / 80 / 80 / 80 (%) (149 / 149 / 149 / 149 / 149 (mm/s)), complete filling of the mold cavity was achieved but there was severe powder-binder separation at the gate area. To reduce powder-binder separation, the injection speed profile was reduced to 50 / 40 / 40 / 30 / 20 (%) (93 / 75 / 75 / 56 / 38 (mm/s)). This injection speed profile is high enough to achieve complete filling as shown in Figure 4.4.

Figure 4.4 Powder-binder separation at different injection speeds

a) 80 / 80 / 80 / 80 / 80 (%) (149 / 149 / 149 / 149 / 149 (mm/s))

b) 50 / 40 / 40 / 30 / 20 (%) (93 / 75 / 75 / 56 / 38 (mm/s))

b) Injection pressure and packing pressure

As soon as the filling phase starts, injection pressure (hydraulic pressure in hydraulic cylinder of injection unit during injection) is applied to the screw in order to inject the feedstock melt into the mold cavity through the runner system. Correspondingly, the cavity pressure is built up as the mold cavity is being filled by the feedstock melt. After volumetric filling of the mold cavity is completed, switchover from injection pressure to packing pressure occurs to ensure the addition of fresh melt into the mold cavity to compensate for the effects of thermal contraction during cooling [53]. At switchover point, the hydraulic pressure in the injection cylinder should be reduced to the desired packing pressure [52]. Since time-dependent switchover was used in this study the selection of switchover point is highly dependent on the injection speed. With the injection speed profile of 50 / 40 / 40 / 30 / 20 (%) (93 / 75 / 75 / 56 / 38 (mm/s)), the switchover point was set as 0.22 sec. according to *in-situ* monitoring of the hydraulic pressure curve.

After determining the switchover point, suitable injection pressure and packing pressure were selected. Three injection pressures of 90 bar, 110 bar and 130 bar were tested. For each injection pressure, different packing pressures were applied. The packing pressure profile shown in Figure 4.5 was used since the part had a simple geometry. Only one packing pressure P_H was selected and held for a period of t_H sec., after which it was decreased to 0 bar in 0.5 sec. With this profile, the packing pressure was arranged so that the packing pressure would not switch down to zero suddenly but followed a linear trend. A Mettler PB303 weighing equipment was used to measure the mass of the green part. The mass variation of green parts under different injection pressure and packing pressure is shown in Figure 4.6. Based on preliminary work, the packing time t_H was set as 3.5 sec.

Figure 4.5 Packing pressure profile

Figure 4.6 Variation of mass of green part with packing pressure and injection pressure

The molding results show that injection pressure plays an important role in the molding of the micro-structures. Compared with PIM, injection molding of micro-structures requires a higher injection pressure. For the injection molding of $\phi 16 \text{ mm} \times$ thickness 1.5 mm part (without micro-structures) an injection pressure lower than 90 bar gives complete filling. But for μ PIM, when the injection pressure is lower than 90 bar the micro-cavities were not completely filled even when using a high packing pressure of 80 bar. When the injection pressure increases to 110 bar and suitable packing pressure was used, the micro-cavities were completely filled. The mass increases as the injection pressure increases. The higher pressure overcomes the friction in the flow channel (including sprue, runner and cavity) and especially the micro-cavities, making the flow into the micro-cavities easier and more feedstock

melt is packed into the micro-cavities. The large number of micro-cavities required higher injection pressure for complete filling. Thus, in subsequent injection molding experiments an injection pressure of 130 bar is used.

The most important aspect of the packing pressure phase is the addition of fresh melt to compensate for the effects of thermal contraction of the feedstock melt during cooling. The result is that air bubbles and sink marks in the part are prevented, and shrinkage and warpage minimized [146]. As shown in Figure 4.6, the mass of the green part increases as the packing pressure increases. Packing pressure also has significant effect on the filling of the micro-cavities. Under low packing pressure, the micro-cavities are not filled completely. When the packing pressure reaches 80 bar (injection pressure of 130 bar) the micro-cavities are filled completely. And the mass of the green part starts to stabilize when the packing pressure reaches 100 bar. Thus, the packing pressure of 100 bar was selected for the next experimental study.

c) Packing time

In this set of experiments, packing time t_H was varied from 0 sec. to 3.5 sec. with a packing pressure of 100 bar. The mass variation of green parts fabricated at different packing time as shown in Figure 4.7 was used to find the gate solidification time.

Figure 4.7 Variation of mass of green part with packing time

At zero packing time, the material flows into the mold cavity by injection with incomplete micro-structure giving very low mass as shown in Figure 4.7. As the packing time increases more material is packed into the mold cavity before the gate solidifies. Starting from 2 sec. packing time, the mass of the green part is almost constant. The gate solidification time is about 2 sec. Consequently the packing time t_H of 2 sec. (1.5 sec. + 0.5 sec.) is used in subsequent experiments.

d) Melt temperature

The melt temperature is the temperature of the feedstock melt at the nozzle. On the molding machine, there are four heating elements: nozzle, zone 1, zone 2 and zone 3. As the commercial 316L feedstock has high viscosity, the temperature of the nozzle was set 5 °C higher than that of zone 1 and zone 2 in order to ensure low viscosity during injection. A constant packing pressure of 100 bar for 2 sec. and a mold

temperature of 125 °C were used for four different melt temperature settings. Since the suitable molding temperature should be set within the range of 165.37 °C to 215.0 °C, the melt temperature settings for the four heating elements were: 200 / 195 / 195 / 180 °C, 190 / 185 / 185 / 170 °C, 180 / 170 / 170 / 160 °C, 170 / 165 / 165 / 160 °C. Too high or too low temperatures have not been used so that binder degradation and low melt viscosity are avoided.

Figure 4.8 Variation of mass of green part with melt temperature

It was found that when the melt (nozzle) temperature increases, the mass of the green part also increases till at the melt temperature of 190 °C, as shown in Figure 4.8. The reason is because the higher melt temperature reduces the feedstock melt viscosity, thus improving the filling of the micro-cavities. At the melt temperature of 200 °C the mass of the green part remains constant. The reason is that the volume of the green

part could not be increased further due to complete filling. The melt temperature was set as 190 / 185 / 185 / 170 °C for the next experimental work. The melt temperature (at nozzle) of 200 °C has not been used in order to prevent possible degradation of the binder.

e) Mold temperature

Besides the thermal characteristics of the material, mold temperature plays an important role in determining the filling, cooling and demolding of the green part. From the DSC results in Chapter 3, the crystallization temperature range of the feedstock is from 139.43 °C to 146.52 °C. The mold temperature should not be higher than the crystallization temperature, otherwise demolding will be difficult due to the lower green strength of the micro-structures. The mold temperatures of 90 °C, 100 °C, 110 °C and 125 °C, constant packing pressure of 100 bar and packing time of 2 sec. were used in the experiments.

Figure 4.9 Variation of mass of green part with mold temperature

The part mass increases as the mold temperature increases, as shown in Figure 4.9. The reason is because the higher mold temperature prevents premature solidification of the feedstock melt. Thus, complete filling can be achieved at a higher mold temperature. Figure 4.10 and Figure 4.11 show the scanning electron micrographs of the green micro-structures ($\phi 100\ \mu\text{m} \times \text{height } 200\ \mu\text{m}$) molded at mold temperatures of $90\ ^\circ\text{C}$ and $125\ ^\circ\text{C}$ with DRIE silicon mold insert. At the mold temperature of $90\ ^\circ\text{C}$, incomplete filling of the micro-cavities occurred.

Figure 4.10 Micro-structures molded at mold temperature of $90\ ^\circ\text{C}$

Figure 4.11 Micro-structures molded at mold temperature of $125\ ^\circ\text{C}$

It can be seen from Figure 4.11 that the mold temperature of 125 °C is more suitable for the filling of $\phi 100\ \mu\text{m} \times \text{height } 200\ \mu\text{m}$ micro-structures. The micro-cavities are filled completely. The green strength of the micro-structures at the temperature of 125 °C is also sufficient for successful demolding. This is because the temperature of 125 °C is lower than the crystallization temperature of 144.36 °C and the polyacetal-based binder has relatively higher viscosity compared with common polymeric binders.

$\phi 80\ \mu\text{m} \times \text{height } 196\ \mu\text{m}$ micro-structures with aspect ratio of 2.5 and $\phi 60\ \mu\text{m} \times \text{height } 191\ \mu\text{m}$ micro-structures with aspect ratio of 3.2 were also molded using the same injection molding conditions. Complete filling was achieved for the molding of $\phi 80\ \mu\text{m} \times \text{height } 196\ \mu\text{m}$ micro-structures. However, incomplete filling occurred for the molding of $\phi 60\ \mu\text{m} \times \text{height } 191\ \mu\text{m}$ micro-structures, as shown in Figure 4.12. There are two reasons for the incomplete filling of the micro-cavities. As the diameter decreases and the aspect ratio increases, the surface area-to-volume ratio of the micro-structure increases. This makes the micro-structure more liable to heat loss. The rapid heat loss leads to the solidification of the feedstock before the micro-cavity is filled completely. Another reason is that as the diameter decreases entrapped air in the micro-cavities could not escape causing incomplete filling. Vacuuming of the mold cavity will help to prevent incomplete filling of the micro-cavities. Therefore, in order to fabricate high aspect ratio micro-structures, a new mold design called variotherm mold, was used.

The variotherm mold design should provide higher mold temperature than the conventional (existing) mold and with reasonable cycle time. High mold temperature can lower the viscosity of the feedstock melt and prevents pre-mature solidification. Thus, complete filling can be achieved. But, due to the high mold temperature, the cooling of the mold cavity to a suitable demolding temperature will be longer. Therefore, the variotherm mold should be properly designed to ensure a reasonably short cycle time. Further, a vacuum system is needed to evacuate the mold cavity before injection with the aim of avoiding air being trapped in the micro-cavities.

Figure 4.12 $\phi 60 \mu\text{m} \times \text{height } 191 \mu\text{m}$ green micro-structures

During injection molding using the conventional mold, in addition to incomplete filling, demolding problems also occurred as the diameter of the micro-structure decreased to $\phi 60 \mu\text{m}$ and the aspect ratio increased from 2 to 3.2. For example, some $\phi 60 \mu\text{m} \times \text{height } 191 \mu\text{m}$ micro-structures with aspect ratio of 3.2, located at the corner of the microstructured area consistently broke during demolding, as shown in

Figure 4.13. Thus, theoretical analysis of the demolding of the micro-structures will be helpful to reduce demolding problem.

Figure 4.13 Broken $\phi 60 \mu\text{m} \times \text{height } 191 \mu\text{m}$ micro-structures at the corner of microstructured area

4.1.3 Summary of injection molding study

After selecting suitable injection molding conditions (injection stroke and speed profile, injection pressure, packing pressure, packing time, melt temperature and mold temperature), $\phi 100 \mu\text{m} \times \text{height } 200 \mu\text{m}$ micro-structures with aspect ratio of 2 and $\phi 60 \mu\text{m} \times \text{height } 196 \mu\text{m}$ micro-structures with aspect ratio of 3.2, were molded successfully using silicon mold inserts.

- 1) Suitable metering stroke was set at 15 mm for the first stage (metering speed 50 %) and 27 mm for the second stage (metering speed 70 %) after “trial and error” until complete filling of the micro-cavities. Injection speed profile of 50 / 40 / 40 / 30 / 20 (%) (93 / 75 / 75 / 56 / 38 (mm/s)) and injection stroke profile of 21 / 17 / 13 / 11 mm were selected to keep constant flow velocity.

- 2) Compared with conventional PIM, the injection molding of micro-structures requires higher injection pressure in order to counteract the friction along the flow path, especially with a higher number of micro-cavities. In this study, the injection hydraulic pressure of 130 bar was used.
- 3) To ensure packing until gate solidification, a packing time of 2 sec. was used. A high packing pressure of 100 bar enabled more material to be packed into the micro-cavities. This avoided incomplete filling and gave increased green density, which will result in less shrinkage during sintering.
- 4) Higher melt temperature improved the filling of the micro-cavities. The melt temperature of 190 / 185 / 185 / 170 °C was used. A high mold temperature of 125 °C enabled more material to be packed into the micro-cavities. At this temperature, complete filling of micro-cavities was achieved.
- 5) As the diameter decreased and the aspect ratio increased, e.g. in the fabrication of $\phi 60 \mu\text{m} \times \text{height } 191 \mu\text{m}$ micro-structures with aspect ratio of 3.2, incomplete filling and breakage of the micro-structures took place. To resolve these two problems, a variotherm mold was designed and theoretical analysis of the demolding of the micro-structures (using the variotherm mold) was studied.

Based on the above analysis and experimental results, a set of suitable injection molding conditions for the conventional mold was established as shown in Table 4.1 for the successful molding of $\phi 100 \mu\text{m} \times \text{height } 200 \mu\text{m}$ micro-structures.

Table 4.1 Suitable injection molding conditions

Parameter	Value
Metering stroke (mm)	15 (50 %) / 23 (70 %)
Injection stroke (mm)	21 / 17 / 13 / 11
Injection speed	50 / 40 / 40 / 30 / 20 (%) (93 / 75 / 75 / 56 / 38 (mm/s))
Injection pressure (bar)	130
Packing pressure (bar)	100
Packing time (sec.)	1.5 / 0.5
Melt temperature ($^{\circ}\text{C}$)	190 / 185 / 185 / 170
Mold temperature ($^{\circ}\text{C}$)	125

4.2 Debinding study

After injection molding, the next step in the μ PIM process is debinding, where the binder is removed from the green part. In the debinding step, it is important that most of the binder is removed. This is because sudden internal vapor pressure build-up at high sintering temperature would cause severe part deformation in the case of failure to remove the binder. After debinding, the part should be free of defects such as cracks,

distortion, slumping and contamination. Occurrence of any defect will affect the final mechanical properties. In this research, catalytic debinding was used to remove the polyacetal-based binder.

Owing to the use of catalytic debinding, the green part was well debound. Distortion and warpage were avoided with good shape retention. Scanning electron micrographs of the debound micro-structures are shown in Figure 4.14 and Figure 4.15.

Figure 4.14 $\phi 100 \mu\text{m} \times 200 \mu\text{m}$ debound micro-structures

Figure 4.15 $\phi 60 \mu\text{m} \times 191 \mu\text{m}$ debound micro-structures

4.3 Sintering study

The sintering of the debound parts was conducted on a CM tube furnace under vacuum atmosphere.

4.3.1 Sintering temperature profile

After debinding, there may be some remaining organic matter, dispersants and some polymer fragments, which can be removed quickly by pyrolysis at 600 °C at the first stage of sintering. A higher amount of carbon will be kept in the sintered parts if a faster heating rate is used, since sintering occurs before all of the organic components have been removed from the part [41]. The sintering profile shown in Figure 4.16 was used.

Figure 4.16 Sintering temperature profile in vacuum atmosphere

Sintering was conducted under a vacuum level of 8 millitorr. First, the debound part was heated from room temperature 25 °C to 600 °C at a heating rate of 7 °C/h, then hold for one hour at 600 °C. The part was then heated from 600 °C to the sintering temperature and held for one hour. After that, the sintered part was naturally cooled in the furnace till room temperature. Sintering at 1200 °C, 1250 °C, 1300 °C and 1350 °C were conducted. Figure 4.17 shows the green, debound and sintered part. Scanning electron micrographs of the sintered micro-structures are shown in Figure 4.18.

Figure 4.17 Green, debound and sintered parts ($\phi 100 \mu\text{m} \times 200 \mu\text{m}$ micro-structures)

Figure 4.18 Sintered micro-structures at different sintering temperature

4.3.2 Density of sintered part

From the scanning electron micrographs of the micro-structures, the micro-structures become smaller as the temperature increases because of higher shrinkage giving higher density. The density of the sintered part was measured by water immersion technique using Mettler electronic balance. At each temperature two sintered parts were measured. The fractional densities at different sintering temperatures are tabulated in Table 4.2. The fractional density is the percentage ratio of the density of the sintered part to its theoretical density 7.99 g/cm^3 .

Table 4.2 Fractional density of sintered part

Temperature (°C)	Fractional density (%)		
	No.1	No.2	Average
1200	95.08	95.12	95.10
1250	95.19	95.21	95.20
1300	95.60	95.56	95.58

Table 4.2 shows that the fractional density increases as the sintering temperature increases. At higher sintering temperature, the interparticle bonds grew and the pore volume shrank, thus higher densification is obtained. The highest average fractional density is 95.58 % for the sintering temperature of 1300 °C. At 1350 °C, melting occurred.

4.3.3 Polished and etched micro-structure

The sintered parts and the micro-structures were sectioned, and then polished and etched with suitable etchant. The etchant is 70 °C nitric-hydrofluoric acid (10_{wt.}% nitric acid + 3_{wt.}% Hydrofluoric acid + 87_{wt.}% de-ionized water). The results are shown in Figure 4.19 to Figure 4.21. Figure 4.19 shows that the number of pores and the pore size were reduced as the sintering temperature increases. Figure 4.20 and Figure 4.21 show that the grain size increases as the sintering temperature increases. At the low temperature of 1200 °C, grain growth had taken place. A higher sintering

temperature of 1300 °C results in higher densification and smaller pore size, and some isolated pores in the grain hinder further densification.

Sintering temperature of 1200 °C

Sintering temperature of 1250 °C

Sintering temperature of 1300 °C

Figure 4.19 Scanning electron micrographs of polished micro-structures

Sintering temperature of 1200 °C

Sintering temperature of 1250 °C

Sintering temperature of 1300 °C

Figure 4.20 Scanning electron micrographs of etched sintered micro-structures

Sintering temperature of 1200 °C

Sintering temperature of 1250 °C

Sintering temperature of 1300 °C

Figure 4.21 Scanning electron micrographs of etched base area

4.4 Summary

In this chapter, $\phi 100\ \mu\text{m} \times \text{height } 200\ \mu\text{m}$ 316L stainless steel micro-structures with aspect ratio of 2 were successfully fabricated by μ PIM using a conventional mold instead of a variotherm mold. In the injection molding step, with suitable injection molding parameters such as high injection pressure and high mold temperature, micro-structures with dimensions of $\phi 100\ \mu\text{m} \times \text{height } 200\ \mu\text{m}$ were successfully injection molded. This is because the $\phi 100\ \mu\text{m} \times \text{height } 200\ \mu\text{m}$ micro-structures have relatively large dimensions and a small aspect ratio of 2. As the dimensions of the micro-structures decreases to $\phi 60\ \mu\text{m}$ (height of $191\ \mu\text{m}$) and the aspect ratio of the micro-structures increases to 3.2, incomplete filling occurred. This could be because air is trapped in the micro-cavity and cannot escape when the feedstock melt flows in to fill the micro-cavity and/or the feedstock melt flowing into each micro-cavity freeze instantaneously upon contacting the cold micro-cavity wall of the conventional mold. Furthermore, demolding problem such as the breakage of the micro-structure occurred during molding of the $\phi 60\ \mu\text{m} \times \text{height } 191\ \mu\text{m}$ micro-structures. Owing to the shortcomings of the conventional mold (i.e. not suitable for the fabrication of high aspect ratio small micro-structures), a variotherm mold was used and discussed in Chapter 5.

CHAPTER 5

μ PIM WITH VARIOTHERM MOLD

In order to mold smaller micro-structures with higher aspect ratios and to reduce demolding problem encountered with a conventional mold, a variotherm mold was designed and fabricated. Micro-structures with dimensions of $\phi 40\ \mu\text{m} \times \text{height } 174\ \mu\text{m}$ and $\phi 20\ \mu\text{m} \times \text{height } 160\ \mu\text{m}$ with aspect ratio of 4.4 and 8, respectively, were successfully injection molded using the variotherm mold.

5.1 Variotherm mold design

The new variotherm mold comprises a mold body, heating system, cooling system, vacuum system, hot sprue system and measuring system (cavity pressure and cavity temperature), as shown in Figure 5.1. The injection molding machine with a variotherm mold is shown in Figure 5.2.

Figure 5.1 Variotherm mold design layout

Figure 5.2 Injection molding machine with variotherm mold

5.1.1 Variotherm mold heating/cooling system

In the case of using conventional mold, one of the important reasons for the incomplete filling is the rapid solidification of the feedstock melt when in contact with the cool wall of the micro-cavity. The situation is worse when molding high aspect ratio green micro-structures for two reasons. The first is that the large surface-to-volume ratio of the high aspect ratio micro-structure leads to fast cooling or even solidification of the feedstock melt in the micro-cavities. The second is that the feedstock melt has higher thermal conductivity than polymers that made the cooling and solidification of the green micro-structures even faster. Therefore, the temperature of the injected feedstock melt will decrease to that of the micro-cavity within a very short duration, and the injected feedstock melt will solidify before the micro-cavities are filled completely.

To achieve complete filling of the micro-cavities, a high mold temperature is required during injection molding. However, after mold filling, the mold has to be cooled to a temperature to facilitate successful demolding of the micro-structures. However, the mold heating/cooling system in conventional mold does not facilitate rapid and frequent mold temperature adjustment. Usually, the mold temperature in a conventional mold is kept constant during injection molding by an oil/water mold temperature controller. The cycle time is in the region of 1 min. depending on the size of the green part. In the case where frequent mold temperature adjustment is needed for high aspect ratio micro-structure molding, the cycle time using conventional mold

is more than 10 min. depending on the heating/cooling capacity of the oil mold temperature controller. This is because it takes a long time to heat/cool the oil in the mold temperature controller. Therefore, a variotherm mold was designed to mold high aspect ratio micro-structures with a reasonably short cycle time. The variotherm mold has a powerful fast heating system to provide rapid heating and direct circulation of cooling water for fast cooling.

The heating/cooling system of the variotherm mold is shown in Figure 5.3. Heat transfer oil with a temperature of 150 °C is pumped from a Tool-Temp mold heater into the heating channel 5 to 7 of the variotherm mold. The temperature of the mold base is kept stable by oil heating. Concurrently, electrical heating cartridges in heating channels 3 and 4 are used to heat the mold cavity. Thus, the temperature of the mold cavity can be increased rapidly. Before demolding, the cooling water from a Tool-temp chiller is pumped into the cooling channels 1 and 2. The temperature of the cooling water is 20 °C. As the cooling channels are very close to the mold cavity, the cooling rate is quite high. Hence, rapid cooling of the green micro-structures can be achieved. Accordingly, the demolding temperature can be adjusted rapidly.

In general, the heating time from the demolding temperature of 100 °C to the mold temperature of 150 °C is around 4.5 minutes. The cooling time from the mold temperature of 150 °C to the demolding temperature of 100 °C is around 12 sec., giving a cooling rate of 4.2 °C/sec. The total cycle time is around 5.5 min in the case where the demolding temperature is 100 °C. The cooling rate of the variotherm mold

is sufficient but the heating rate can be improved further. Because of the space constraint it is very difficult to arrange heating cartridges and cooling channels perfectly in order to achieve high heating rate and cooling rate at the same time. The heating rate is restricted due to the relatively large gap between the heating cartridges and the mold cavity.

Figure 5.3 Variotherm mold heating/cooling system

5.1.2 Vacuum system

Apart from rapid solidification of the feedstock melt, trapped air in the small micro-cavity is another reason for the incomplete filling, particularly when the micro-cavities are so small that they cannot be vented in the conventional way, i.e., through the parting plane or special bore holes. In this case, evacuation of the micro-cavities prior to injection is necessary.

To prevent air being trapped in the micro-cavity, a vacuum system is incorporated into the variotherm mold, including the venting channel. An O-ring seal and an external vacuum pump are connected to the mold by a hose as shown in Figure 5.4. To ensure good sealing of mold cavity, temperature-resistant O-ring seals are also placed around every ejection pin. Before injection started, the cavity is evacuated by the vacuum pump. The vacuum level reached is 0.27 Pa.

Figure 5.4 Vacuum system for variotherm mold

5.1.3 Hot-sprue system

The variotherm mold is equipped with a hot-sprue system, as shown in Figure 5.5. Compare with the hot-runner system, the hot-sprue system is more suitable for μ PIM. Usually the hot-runner system is based on a hot nozzle, which ends at the mold cavity. The heated nozzle keeps the feedstock melt at a temperature higher than the melting

point. The outlet of the hot runner is in direct contact with the mold cavity. If the hot-runner nozzle is insufficiently insulated, the runner would heat up the cavity and the local temperature of the mold cavity would increase. This can lead to non-homogeneous shrinkage and even demolding problems [147, 148]. Owing to challenges concerning thermal insulation between hot-runner system and the mold cavity, hot-runner system seldom finds application in the μ PIM process [148]. If necessary, a hot-sprue is an alternative [147].

As shown in Figure 5.5, the portion of the hot-sprue set in contact with the nozzle of the injection molding machine is heated to the temperature of 190 °C. The temperature is the same as that of the nozzle of the injection machine. The length of the portion is around three fourths of the total length of the hot-sprue set. Because of the high temperature, the feedstock melt in this portion is kept at melted state. Owing to this portion of feedstock melt, the sealing of the mold cavity is available before injection. Another portion is a conventional sprue whose length is around one fourth of the total length of the hot-runner set. Its volume is much smaller than that of the sprue in the existing conventional mold design. Thus, the material cost using a variotherm mold with hot sprue system is reduced as compared to existing conventional mold.

Variotherm mold

Conventional mold

Figure 5.5 Cross sectional view of variotherm mold and conventional mold

5.1.4 Measuring system

A Kistler 6190A combined pressure and temperature sensor (Kistler Instrument Corp., USA) was installed, so that cavity pressure and cavity temperature during injection molding can be monitored precisely. The electronic signals were converted to pressure and temperature values and recorded by a computer in real time through two electronic module cards. Furthermore, it is possible to control the demolding temperature more precisely with the sensor.

Figure 5.6 Combined pressure and temperature sensor

5.1.5 Mounting of silicon mold insert in mold cavity

The adhesive bond between the silicon mold insert and the mold cavity had a short lifetime when using the conventional mold in Chapter 4. This is because the feedstock melt entered the mold cavity at a high speed giving the silicon mold insert a large lateral impact, which caused the detachment of the silicon mold insert from the mold cavity. To prolong the life of the silicon mold insert, the mounting method of silicon mold insert was modified, i.e. the top surface of the silicon mold insert was designed to be flush with the mold cavity surface. The new mounting method was used on both modified conventional mold and variotherm mold in the subsequent experiments.

As shown in Figure 5.7, the modified mounting method is different from that in the conventional mold mentioned in Chapter 4. In the case of the modified mounting method, the lateral impact of the feedstock melt on the silicon mold insert is eliminated. The filling is more uniform since the mold insert does not impose any interference to the feedstock melt during the filling of the micro-cavities. This ensures complete filling of the micro-cavities. Further, demolding is easier since the silicon mold insert is not laterally displaced during demolding.

Figure 5.7 Mounting of silicon mold insert in the mold cavity before and after modification

5.2 Evaluation of the variotherm mold

The features of the variotherm mold are introduced and evaluated with respect to process cycle, injection molding conditions, cavity pressure and temperature curves, and molding of high aspect ratio microstructures.

5.2.1 Process cycle for the variotherm mold and processing parameters

The schematic injection molding cycle of the variotherm mold is shown in Figure 5.8.

The typical cavity pressure and temperature curves of the variotherm mold during injection molding $\phi 100 \mu\text{m} \times \text{height } 200 \mu\text{m}$ micro-structures are shown in Figure 5.9.

The key injection molding parameters include a packing pressure of 100 bar and a demolding temperature of 100 °C.

Figure 5.8 Injection molding cycle for variotherm mold

Figure 5.9 Typical cavity pressure and temperature curve of variotherm mold (for $\phi 100 \mu\text{m} \times \text{height } 200 \mu\text{m}$ micro-structures)

As shown in Figure 5.8, after the closing of the mold, the mold cavity is evacuated to a pressure of 2.7 Pa via the vacuum unit. The mold base is heated through oil heating via channels 5, 6 and 7 of Figure 5.3. Concurrently, four electrical cartridges with the power of 400 watt in heating channels 3 and 4 heat the mold rapidly to the desired temperature of 150 °C. This is between the melting temperature of 163.5 °C and the crystallization temperature of 144.36 °C of the feedstock. When the Kistler sensor detects and displays 150 °C, the injection unit is moved forward against the mold and the injection screw injects the feedstock melt into the mold cavity and micro-cavities.

Because of the smaller temperature difference (as compared to PIM) between the temperature of the feedstock melt and the mold temperature, the feedstock melt was injected with little heat loss during the filling phase. The higher mold temperature facilitated complete filling of the micro-cavities with lower melt temperature and slower filling rate than that used normally. This is because the mold temperature was higher than the crystallization temperature of the feedstock. Thus, the feedstock melt would not solidify at the mold temperature.

As shown in Figure 5.9, water cooling is started after 5 seconds of packing pressure. The temperature of the green part is reduced via cooling channels 1 and 2. Since the cooling channels are very close to the mold cavity, rapid cooling of the green micro-structures can be achieved. The cooling rate is around 4.2 °C/sec., e.g. the cavity temperature is reduced from 150 °C to 100 °C in just 12 sec. Accordingly, the demolding temperature can be adjusted easily and rapidly. The combined pressure and temperature sensor monitors the temperature of the green part and the cavity pressure. After the cavity temperature reaches the desired demolding temperature, the demolding temperature is held for 30 sec. to stabilize the temperature. Then the mold opens and the green part is ejected.

For the variotherm injection molding process, the suitable injection molding parameters for the $\phi 100\ \mu\text{m} \times \text{height } 200\ \mu\text{m}$ micro-structures are listed in Table 5.1.

Table 5.1 Suitable injection molding conditions for $\phi 100 \mu\text{m} \times \text{height } 200 \mu\text{m}$ micro-structures using variotherm mold

Parameter	Value
Injection stroke (mm)	19 / 17 / 15 / 15
Injection speed	50 / 40 / 40 / 30 / 20 (%) (93 / 75 / 75 / 56 / 38 (mm/s))
Packing pressure (bar)	100
Packing time (s)	8 (7.5 / 0.5)
Melt temperature ($^{\circ}\text{C}$)	190 / 185 / 185 / 170
Mold temperature ($^{\circ}\text{C}$)	150

5.2.2 Cavity pressure and temperature curves

To show the difference between the variotherm mold and the conventional mold, cavity pressure and temperature curves of both molds (using the modified mounting method of silicon mold insert) are presented in Figure 5.10. As the cavity pressure and temperature curves for different dimensions of silicon mold inserts have the same trend, silicon mold inserts with $\phi 100 \mu\text{m} \times \text{depth } 200 \mu\text{m}$ micro-cavities were used due to easy demolding and suitability for conventional mold.

The mold temperature of 150°C used in the variotherm mold is higher than that for the conventional mold. Accordingly, the cavity temperature is also higher. However, due to the effective cooling system, the cavity temperature reduces rapidly to the demolding temperature at a cooling rate of $4.2^{\circ}\text{C}/\text{sec}$. In the case of the conventional mold, the cavity temperature decreases slowly to the constant mold temperature.

Higher cavity temperature gives higher cavity pressure according to the PVT properties of the feedstock discussed in Section 3.1.8. Thus, during isochoric packing, the cavity pressure of the variotherm mold is higher than that in the case of conventional mold where the same packing pressure is used. This will improve the complete filling of the mold cavity and micro-cavities.

Figure 5.10 Cavity pressure and temperature curves of variotherm mold and conventional mold for $\phi 100 \mu\text{m} \times \text{height } 200 \mu\text{m}$ micro-structures

In the injection molding using variotherm mold, the effects of packing pressure were investigated. Based on the demolding temperature of 100°C , different packing pressures were used to mold $\phi 100 \mu\text{m} \times \text{height } 200 \mu\text{m}$ micro-structures. The packing pressures were 20 bar, 40 bar, 60 bar, 80 bar, 100 bar and 120 bar. Cavity pressure and temperature curves at different packing pressures are shown in Figure 5.11.

Figure 5.11 (a)

Figure 5.11 (b)

Figure 5.11 (c)

Figure 5.11 (d)

Figure 5.11 (e)

Figure 5.11 (f)

Figure 5.11 Cavity pressure and temperature curves at different packing pressures for $\phi 100 \mu\text{m} \times \text{height } 200 \mu\text{m}$ micro-structures

The curves show that the packing pressure has a large influence on the pressure curve during the packing pressure phase. A rise in the packing pressure causes a rise in the cavity pressure and an increase in the duration of the cavity pressure. At low packing pressures such as 20 bar and 40 bar, the cavity pressure decreases quickly to the minimum value within 5 sec. and 7 sec. respectively. However, low packing pressure such as 40 bar results in incomplete filling of the micro-cavities, as shown in Figure 5.12. As the packing pressure increases to 60 bar, 80 bar and 100 bar, the cavity pressures are much larger and last over longer time than in the case of low packing pressures. This has positive influence on the filling of the micro-cavities. For example, $\phi 20\ \mu\text{m} \times \text{height } 160\ \mu\text{m}$ micro-structures were molded with complete filling using 100 bar packing pressure as shown in Figure 5.13. However, the extreme high packing pressure will make the feedstock melt undergo high pressure for a longer time that may result in high residual stress in the green micro-structures. The selection of suitable packing pressure is a compromise between complete filling and low residual stress. The lowest packing pressure that ensures the complete filling of the micro-cavities is preferred for minimizing the residual stress.

Figure 5.12 $\phi 20\ \mu\text{m} \times \text{height } 160\ \mu\text{m}$ green micro-structures molded at packing pressure of 40 bar

Figure 5.13 $\phi 20\ \mu\text{m} \times \text{height } 160\ \mu\text{m}$ green micro-structures molded at packing pressure of 100 bar

Packing pressure has significant effect on the filling of the micro-cavities. As shown in Figure 5.14, the average mass of three green parts increases as the packing pressure increases, thus ensuring the complete filling of the micro-cavities.

Figure 5.14 Variation of mass of green part with packing pressure

5.2.3 Molding high aspect ratio micro-structures using variotherm mold

Using the injection molding conditions mentioned in Section 5.2.1, smaller micro-structures with higher aspect ratio were produced. As shown in Figure 5.15, the maximum attainable aspect ratio using variotherm mold nearly increases two-folds as compared with that using modified conventional mold with the silicon mold insert being flush with the inner surface of the mold cavity. (Note: The micro-cavities are etched on 4-inch silicon wafer with 450 μm thickness. The maximum etching depth to ensure vertical sidewall is around 200 μm , thus the maximum available aspect ratio in the large diameter is limited).

For example, $\phi 40 \mu\text{m} \times \text{height } 174 \mu\text{m}$ micro-structures with aspect ratio of 4.4 and $\phi 20 \mu\text{m} \times \text{height } 160 \mu\text{m}$ micro-structures with aspect ratio of 8 were successfully injection molded, as shown in Figure 5.16 and Figure 5.17, respectively.

Figure 5.15 Maximum attainable aspect ratios with variotherm mold

Figure 5.16 $\phi 40\text{ }\mu\text{m} \times \text{height } 174\text{ }\mu\text{m}$ green micro-structures Figure 5.17 $\phi 20\text{ }\mu\text{m} \times \text{height } 160\text{ }\mu\text{m}$ green micro-structures

After injection molding, the green part were catalytically debound and sintered. The $\phi 40 \mu\text{m} \times \text{height } 174 \mu\text{m}$ micro-structures sintered at 1300°C in hydrogen are shown in Figure 5.18.

Figure 5.18 $\phi 40 \mu\text{m} \times \text{height } 174 \mu\text{m}$ micro-structures sintered at 1300°C

5.3 Summary

A variotherm mold was designed to fabricate 316L stainless steel high aspect ratio micro-structures and facilitate the demolding of the micro-structure by rapid adjustment of the demolding temperature. A hot sprue system, combined pressure and temperature sensor, vacuum unit and rapid heating/cooling system are incorporated into the mold. The variotherm mold has the following advantages compared with conventional mold: the melt fills the micro-cavities with lower heat loss during injection, thus facilitating the filling of micro-cavities, and the fabrication of micro-structures with smaller dimensions and higher aspect ratio (such as $\phi 20 \mu\text{m} \times \text{height } 160 \mu\text{m}$ micro-structures with aspect ratio of 8) becomes possible. With the rapid heating/cooling system (on the variotherm mold), the duration of cooling from 150°C

to the demolding temperature of 100 °C is just 12 sec, giving a cooling rate of 4.2 °C/sec. Finally, the demolding temperature can be adjusted to a specific value for successful demolding. $\phi 40 \mu\text{m} \times \text{height } 174 \mu\text{m}$ micro-structures with aspect ratio of 4.4 and $\phi 20 \mu\text{m} \times \text{height } 160 \mu\text{m}$ micro-structures with aspect ratio of 8 which could not be fabricated using conventional mold were successfully fabricated using the variotherm mold.

CHAPTER 6

ANALYSIS OF THE DEMOLDING OF MICRO-STRUCTURES

In the previous chapter, a new variotherm mold was designed and fabricated with the aim of improving the filling of the micro-cavities and facilitating demolding. This was carried out by adjusting the mold temperature rapidly using a fast heating/cooling system in the variotherm mold. Thus, $\phi 20\ \mu\text{m} \times \text{height } 160\ \mu\text{m}$ micro-structures with high aspect ratio of 8 were fabricated successfully using the variotherm mold. A theoretical analysis of micro-structure demolding is needed so that suitable demolding conditions can be established for successful demolding. In this chapter, PVT analysis and FEM analysis using ABAQUS software were conducted to study the demolding behavior of the array of 24×24 (total of 576) micro-structures mentioned in Chapter 5. The stress distributions of the micro-structures were studied to establish possible fracture and the moment when the fracture takes place. The demolding forces required to eject the micro-structures from the micro-cavities were calculated based on the results of the FEM analysis. To achieve the lowest demolding force for successful demolding, the effects of packing pressure and demolding temperature on the demolding force were analyzed and then experimentally verified.

6.1 Analysis of demolding of the micro-structures

During demolding, the green part (consisting of the base and the micro-structures) is ejected out from the mold cavity after the feedstock melt cools and solidifies, and the mold opens. A schematic drawing of the demolding of the green part is shown in Figure 6.1.

Figure 6.1 Schematic drawing of the demolding of green part

Although demolding takes place after packing pressure and cooling phases, the demolding behavior is still affected by the packing and cooling of the feedstock melt during injection molding. This is because the pressure-volume-temperature (PVT) status of the feedstock in the mold cavity is governed by its PVT properties as

mentioned in Section 3.1.8. Theoretically, the dimensions of the green part after demolding are determined by the packing pressure and the demolding temperature.

There are two main tasks in the demolding analysis:

- a) To calculate the dimensions of the green part (base and micro-structures) at a specific demolding temperature based on the PVT properties of the feedstock and processing conditions. This task is discussed in Section 6.2.
- b) Based on the calculated dimensions, analyze the stress distribution of the green part during demolding using FEM and calculate the demolding force for the micro-structures. This task is discussed in Section 6.3.

During demolding, the stresses on the micro-structures can be derived from the following: (a) thermal stress caused by the cooling of the green part and (b) shear stress from friction between the micro-structure and the micro-cavity.

During molding, the feedstock melt is forced into the mold cavity. After the packing pressure phase and gate solidification, the feedstock melt cannot be forced into the mold cavity. The amount of the feedstock melt packed into the mold cavity is finalized. In the mold cavity, this amount of feedstock melt within the mold cavity

cools, and then solidifies after the gate solidifies. The variation of its pressure, specific volume and temperature follows the PVT equation of the feedstock. After the feedstock fully solidifies to form the green part, it will continue to shrink as the mold temperature keeps decreasing. Thus, each micro-structure will shrink toward its own center of mass, as does the base of the green part, as shown in Figure 6.2.

Figure 6.2 Shrinking direction of green part (base and the micro-structures)

Because of the shrinking of the base towards the center of mass of the green part (which is located on the centerline of the green part due to geometric symmetry) any micro-structure in the array will be given a radial displacement towards the centerline. The displacement of the individual micro-structure will increase as its distance from the centerline of the green part increases. However, the displacement of the micro-structure is hindered by the micro-cavity of the silicon mold insert. Thus, thermal stress is induced.

As each of the solidified green micro-structure is in contact with the inner surface of the micro-cavity, there is contact pressure between the micro-structure and the inner surface of the micro-cavity. The micro-structure is thus subject to: (a) frictional force (between the circumferential surface of the micro-structure and the inner circumferential surface of the micro-cavity) and, (b) the adhesion force (between the top end surface of the micro-structure and the bottom inner surface of the micro-cavity), during ejection out of the micro-cavity. Therefore, shear stress will be induced. The stress endures by the micro-structures during the demolding is the combination of the thermal stress and the shear stress.

6.2 PVT analysis

In order to calculate the amount of feedstock melt packed into the mold cavity after gate solidification, it is necessary to find the gate solidification time during injection molding.

6.2.1 Determination of gate solidification time

Each green part has a total of 576 micro-structures. The total volume of all the 576 micro-structures accounts for a very small percentage of the volume of the green part. For example, total volume of all the $\phi 100 \mu\text{m} \times \text{height } 200 \mu\text{m}$ micro-structures with

aspect ratio of 2 accounts for only 0.3 % of the volume of the green part. For smaller micro-structures such as $\phi 80\ \mu\text{m}$, $\phi 60\ \mu\text{m}$, $\phi 40\ \mu\text{m}$ and $\phi 20\ \mu\text{m}$, the percentages are even smaller. Therefore, for $\phi 16\ \text{mm} \times \text{thickness } 1.5\ \text{mm}$ green parts with different micro-structures, their gate solidification time are approximately equal to each other. The dimensions of the micro-cavities do not matter in the experiment measuring gate solidification time. In this demolding analysis, silicon mold inserts with $\phi 100\ \mu\text{m} \times \text{depth } 200\ \mu\text{m}$ micro-cavities were used due to easy molding/demolding and also because larger size micro-structures give larger demolding force that can be more precisely measured by the load cell.

Experiments were conducted to measure the gate solidification time during injection molding. The masses of green parts fabricated using different packing time were measured using a Mettler PB303 weighing equipment and the results shown in Figure 6.3. The average mass was based on three green parts.

In this experiment, the molding conditions in Table 5.1 were used. Packing time was varied from 0 sec. to 8 sec. At zero packing time, the material flowed into the cavity by injection with incomplete micro-structure giving very low mass as shown in Figure 6.3. As the packing time increased more material was packed into the mold cavity before the gate solidified. Starting from 6 sec. packing time, the mass of the green part

Figure 6.3 Variation of mass of green part with packing time

was almost constant. Thus, the solidification time was 6 sec. At this time, the cavity temperature (taken as the gate solidification temperature) was around 145 °C according to Figure 5.11. To prevent any possible backflow of the feedstock melt into the mold cavity a packing time of 8 sec. was used in subsequent experiments.

6.2.2 PVT analysis

The gate solidification temperature of 145 °C is lower than the transition temperature of the feedstock (i.e. the upper limit of the crystallization temperature range). The PVT behavior of the feedstock follows the lower portion of its PVT curves shown in Figure 3.10, which is represented by Equation 3.3. Thus, the mass of the feedstock melt packed into the micro-cavity when the gate solidifies can be calculated according to Equation 3.3.

The specific volume of the feedstock packed into the mold cavity at the moment of gate solidification $v(T_s, p_s)$ is:

$$v(T_s, p_s) = v_0(T_s) \left\{ 1 - C \ln \left(1 + \frac{p_s}{B(T_s)} \right) \right\} + v_t(T_s, p_s) \quad (6.1)$$

where,

$$v_0(T_s) = b_{1s} + b_{2s} \bar{T}_s = b_{1s} + b_{2s} (T_s - b_5)$$

$$B(T_s) = b_{3s} e^{-b_{4s} \bar{T}_s} = b_{3s} e^{-b_{4s} (T_s - b_5)}$$

$$v_t(T_s, p_s) = b_7 e^{(b_8 \bar{T}_s - b_9 p_s)} = b_7 e^{[b_8 (T_s - b_5) - b_9 p_s]}$$

T_s = temperature of micro-structure at the moment of gate solidification

p_s = cavity pressure at the moment of gate solidification

The corresponding mass of the feedstock packed into mold cavity is:

$$M = \frac{V_c}{v(T_s, p_s)} = \frac{N_m \pi d^2 h + \pi D_c^2 H}{4v(T_s, p_s)}$$

where,

V_c = the volume of the mold cavity

N_m = the number of micro-cavities (micro-structures)

D_c = the diameter of the mold cavity

H = the thickness of the mold cavity

d = the diameter of the micro-cavity

h = the depth of the micro-cavity

The green part continues to cool and is demolded when it reaches the demolding temperature T_d . The specific volume of green part at atmospheric pressure after demolding is:

$$v(T_d, p_a) = v_0(T_d) \left\{ 1 - C \ln \left(1 + \frac{p_a}{B(T_d)} \right) \right\} + v_t(T_d, p_a) \quad (6.2)$$

where,

p_a = atmospheric pressure, which is assumed to be zero as it is very small compared with the peak cavity pressure of 1160 bar.

Therefore, Equation 6.2 is equal to:

$$v(T_d, p_a) = v(T_d, 0) = v_0(T_d) + v_t(T_d, 0) = [b_{1s} + b_{2s}(T_d - b_5)] + b_7 e^{b_8(T_d - b_5)} \quad (6.3)$$

As the mass of the feedstock packed into the mold cavity (i.e. the mass of green part) does not change after demolding, the corresponding volume of green part after demolding V_d can be calculated as below.

$$M = \frac{V_c}{v(T_s, p_s)} = \frac{V_d}{v(T_d, p_a)} \quad (6.4)$$

$$V_d = \frac{v(T_d, p_a) \cdot V_c}{v(T_s, p_s)} = \frac{\pi}{4} D_d^2 H_d + N_m \cdot \frac{\pi}{4} d_d^2 h_d$$

where,

D_d = the diameter of the base of green part at demolding temperature T_d

H_d = the thickness of the base of green part at demolding temperature T_d

d_d = the diameter of the micro-structure at demolding temperature T_d

h_d = the height of the micro-structure at demolding temperature T_d

Here an assumption is made that the shrinkage of the green part is isotropic. Thus,

Equation 6.4 can be rewritten as below:

$$\frac{V_c}{v(T_s, p_s)} = \frac{V_d}{v(T_d, p_a)} = \frac{(1-\gamma)^3 \cdot V_c}{v(T_d, p_a)} \Rightarrow \gamma = 1 - \left[\frac{v(T_d, p_a)}{v(T_s, p_s)} \right]^{1/3}$$

where γ = linear shrinkage ratio

Therefore, the diameter of the micro-structure d_d at the demolding temperature is depicted by:

$$d_d = (1-\gamma)d = d \cdot \left[\frac{v(T_d, p_a)}{v(T_s, p_s)} \right]^{1/3}$$

Substituting Equation 3.3 into the above equation gives:

$$d_d = d \cdot \left[\frac{[b_{1s} + b_{2s}(T_d - b_5)] + b_7 e^{b_8(T_d - b_5)}}{[b_{1s} + b_{2s}(T_s - b_5)] \left\{ 1 - C \ln \left(1 + \frac{p_s}{b_{3s} e^{-b_{4s}(T_s - b_5)}} \right) \right\} + b_7 e^{[b_8(T_s - b_5) - b_9 p_s]}} \right]^{1/3} \quad (6.5)$$

The three-dimensional plot for $\phi 100 \mu\text{m} \times \text{height } 200 \mu\text{m}$ micro-structures is shown in Figure 6.4.

Figure 6.4 Three-dimensional plot of the diameter of the micro-structure

The diametric variation Δd of the micro-structure at the demolding temperature is:

$$\Delta d = d_d - d = d \cdot \left[\frac{v(T_d, p_a)}{v(T_s, p_s)} \right]^{1/3} - d$$

$$= d \cdot \left(\left[\frac{[b_{1s} + b_{2s}(T_d - b_5)] + b_7 e^{b_8(T_d - b_5)}}{[b_{1s} + b_{2s}(T_s - b_5)] \left\{ 1 - C \ln \left(1 + \frac{p_s}{b_{3s} e^{-b_{4s}(T_s - b_5)}} \right) \right\}} + b_7 e^{[b_8(T_s - b_5) - b_9 p_s]} \right]^{1/3} - 1 \right)$$

(6.6)

When $\Delta d = 0$, the demolding temperature $T_d = T_{cr}$, where T_{cr} is called the critical temperature. According to Equation 6.6, T_{cr} is determined by the cavity pressure when the gate solidifies and the feedstock properties, i.e. PVT coefficients of the feedstock. For example, when the packing pressure is 120 bar, T_{cr} is 108 °C for all the

different micro-structure diameters shown in Figure 6.5.

ϕ100 μm micro-structure

ϕ80 μm micro-structure

ϕ60 μm micro-structure

ϕ40 μm micro-structure

Figure 6.5 Diameter of micro-structure d_d at demolding temperature T_d using packing pressure of 120 bar

$T_d < T_{cr}$, $\Delta d < 0$. In this case, theoretically the diameter of the green micro-structure is smaller than that of the micro-cavity at the demolding temperature. Owing to isotropic shrinkage assumption, the total volume of the green part at the demolding temperature is smaller than that of the mold cavity.

$T_d > T_{cr}$, $\Delta d > 0$. In this case, theoretically the diameter of the micro-structure is larger than that of the micro-cavity at the demolding temperature on condition that no micro-cavity constrains the micro-structure. Likewise, the total volume of the green part at the demolding temperature is larger than that of the mold cavity on condition that no mold cavity constrains the green part.

During the cooling phase, two scenarios can occur. The first is the volumetric expansion due to cavity pressure decrement. The PVT properties of the feedstock in the mold cavity follow its PVT curves. As the cavity pressure decreases, its specific volume increases, i.e. its volume increases. The second concerns the volumetric shrinkage from cooling. As the temperature decreases, its volume decreases. At the critical temperature T_{cr} , a balance is struck between these two scenarios. The volume of the green part is equal to that of the mold cavity. In the case of $T_d < T_{cr}$, the volumetric shrinkage due to cooling prevails over the volumetric expansion due to cavity pressure decrement. Thus, the volume of the green part is smaller than that of

the mold cavity and the diameter of the micro-structure is smaller than that of the micro-cavity. In the case of $T_d > T_{cr}$, the reverse situation occurs.

6.3 FEM stress analysis of micro-structures demolding

From Section 6.2, the dimensions of the micro-structures at the demolding temperature T_d can be calculated in the PVT analysis. With these data further stress analysis during the course of demolding becomes possible.

6.3.1 Model and boundary conditions

The model for the demolding of the micro-structures is shown in Figure 6.6. The total demolding force needed to eject the green part from the mold cavity can be depicted as follows:

Figure 6.6 Model of demolding micro-structures

$$F_D = F_{Fb} + F_{Sb} + N(F_{Fm} + F_{Sm} + F_v)$$

where

F_D = demolding force for the whole green part

F_{Fb} = frictional force between cavity wall and the circumferential surface of the base of green part

F_{Sb} = sticking force between top surface of the base of green part and cavity inner surface

N = the number of micro-structures

F_{Fm} = frictional force between micro-structure and the inner surface of the micro-cavity

F_{Sm} = sticking force between top surface of the micro-structure and the bottom surface of the micro-cavity

F_v = additional release force due to vacuum effect for the case where contact between the micro-structure and the micro-cavity exists

The three-dimensional geometric model of the green part with 24×24 micro-structures was established in order to simulate the demolding of the micro-structures. The dimensions of the micro-structure are $\phi 100 \mu\text{m} \times 200 \mu\text{m}$. The pitch of the micro-structures array is $200 \mu\text{m}$. The reason for using relatively big size micro-structures in the analysis was that larger size micro-structures gave larger demolding force that can be more precisely measured by the load cell. The geometric model and meshing are shown in Figure 6.7. Because of symmetry, it is sufficient to calculate only one quarter of the model.

Figure 6.7 Geometric model and its meshing in the FEM simulation

a) In the case of $T_d < T_{cr}$

In this case, green part shrinks at the demolding temperature T_d as compared with the dimensions of the mold cavity and the micro-cavities. The boundary conditions of the model are shown in Figure 6.8. The left center of the mold insert is fixed. The right center of the mold insert is constrained only in vertical direction and the left edge is constrained in horizontal direction so that the mold insert can undergo free expansion or contraction. The centerline of the green part is fixed only in vertical direction so that there is no constraint in radial displacement except in the direction of the axis of symmetry. Hard contact and classical isotropic coulomb friction model (i.e. $F = \mu N$, where F is frictional force, μ is the coefficient of friction and N is normal force between the frictional surfaces) between the micro-structure and the micro-cavity are used. When surfaces are in hard contact, any contact pressure can be transmitted between them. The surfaces are not in contact if the contact pressure reduces to zero. The surfaces come into contact when the clearance between them reduces to zero.

Figure 6.8 Boundary conditions of the model in the FEM simulation for the case where $T_d < T_{cr}$

The simulation is divided into three steps.

- i) The first step is surface contact. The green part is held inside the mold cavity and the micro-cavities. According to the PVT analysis, the dimensions of the green part at the demolding temperature are slightly smaller than those of the mold cavity and the micro-cavities. In this step, the shrunk green part (the base and the micro-structures) is in contact with the mold cavity and the micro-cavities. The thermal stress will be calculated in this step. The coefficient of expansion of the silicon mold insert ($2.5 \times 10^{-6} / ^\circ\text{C}$) is much smaller than that of the feedstock ($48.7 \times 10^{-6} / ^\circ\text{C}$). In order to decrease the calculation time the dimensional variation of the silicon mold insert is neglected.

- ii) The second step is the static friction stage. In this step, the green part starts to move. As measured in Section 3.1.11, the coefficient of static friction between the feedstock and the silicon of 0.66 will be used in the theoretical analysis.
- iii) The third step is the sliding friction stage, when the green part is ejected from the mold cavity. In this step, the friction between the micro-structures and the micro-cavities is sliding friction. The coefficient of sliding friction of 0.37 measured in Section 3.1.11 will be used in the theoretical analysis.

b) In the case of $T_d > T_{cr}$

According to the PVT analysis, in the case where $T_d > T_{cr}$ the green part expands after demolding provided the mold cavity does not constrain the green part. Thus, contact pressure is established between the micro-structures and the micro-cavities. Likewise, there is also contact pressure between the circumferential surface of the base of the green part and the mold cavity wall. Therefore, the circumferential surface of the base of the green part is in contact with the mold cavity wall and constrained along the radial direction. The surface can only move along the demolding direction. Other boundary conditions are the same as those in the case where $T_d < T_{cr}$. The boundary conditions of the model in the FEM simulation are shown in Figure 6.9.

Figure 6.9 Boundary conditions of the model in the FEM simulation for the case where $T_d > T_{cr}$

6.3.2 Simulation results in the case of $T_d < T_{cr}$

In the simulation, the typical injection molding conditions used in the experiments as mentioned in Table 5.1 were used. For example, a packing pressure of 100 bar was used and a demolding temperature of 105 °C that is lower than the critical temperature of 122 °C was used.

a) Stress and strain distributions during demolding

As there are a total of 576 micro-structures in the array, it is difficult to show the stress distribution of all the micro-structures clearly. Some representative micro-structures are selected to reflect the stress distribution of the micro-structures array.

The stress endured by the micro-structures is mainly caused by the shrinkage of the green part, and the shrinkage is always towards the center of mass of the green part which is located on the centerline of the green part, shown in Figure 6.2. Therefore, a cross-sectional plane is created as the representative plane as shown in Figure 6.10. The plane is the cross-sectional plane of twelve micro-structures along the diagonal direction of the micro-structures array. The stress distribution of the micro-structures in the cross-sectional plane will be displayed and analyzed.

Figure 6.10 The diagonal cross-sectional plane of the micro-structures

The stress and strain distributions of the micro-structures in the cross-sectional plane at different displacements during the course of the demolding are shown in Figure 6.11 and Figure 6.12 respectively. Due to complex three-dimensional stress conditions and the ductile property of the feedstock, equivalent Mises stress was used. The maximum stress endured by the individual micro-structure increases with increasing distance from the centerline of the green part during the whole course of the demolding. This is because the micro-structure farther away from the centerline has a larger strain due to the shrinkage of the base of the green part.

Displacement: 80 μm
Figure 6.11 (c)

Completely ejected out
Figure 6.11 (d)

Figure 6.11 Equivalent Mises stress distribution of the cross-section of the micro-structures in the case of $T_d < T_{er}$
(point X is maximum stress point of the micro-structure)

Just before demolding
Figure 6.12 (a)

Onset of demolding
Figure 6.12 (b)

Figure 6.12 Equivalent plastic strain distribution of the cross-section of the micro-structures in the case of $T_d < T_{cr}$

Before the demolding is initiated, thermal stress exists due to cooling and shrinkage. The maximum equivalent Mises stress of the two farthest micro-structures reaches the ultimate tensile strength (3.48 MPa) of the feedstock at 105 °C. The maximum equivalent plastic strain (termed PEEQ in the ABAQUS software) at the base of the micro-structure is 1.49 % and 1.75 %, as shown in Figure 6.12(a). As the maximum PEEQ is smaller than the fracture strain of 52.1 % mentioned in Section 3.1.9, the micro-structures did not break.

At the onset of demolding, the micro-structures undergo shear stress caused by the friction between the micro-structure and the inner surfaces of the micro-cavity. This is in addition to the thermal stress experienced by the micro-structures. Hence, the equivalent Mises stress is larger than that before demolding. As shown in Figure 6.11(b), the maximum equivalent Mises stresses of the four farthest micro-structures reach the ultimate tensile strength (3.48 MPa) of the feedstock compared with the two farthest micro-structures before demolding. The equivalent plastic strain also increases to the maximum value of 3.13 %. The onset of demolding is the most critical moment during the whole course of demolding.

As a micro-structure is ejected out of the micro-cavity, the maximum stress point of the micro-structure is the point closest to the centerline of the green part, indicated by point X in Figure 6.11 (a) to (c). The maximum equivalent Mises stress of the individual micro-structure becomes smaller and smaller as the micro-structure is ejected out. After the micro-structure is completely ejected out, there exists residual

stress in the green part. After the maximum PEEQ reaches its maximum value of 3.13 % at the onset of demolding, the value does not increase any more during the course of demolding.

Due to the shrinking effect, the maximum PEEQ of the micro-structure will increase as the demolding temperature decreases. Therefore, too low a demolding temperature is not recommended. A suitable demolding temperature must ensure the stress/strain and demolding force experienced by the micro-structures be as low as possible. Once the maximum PEEQ exceeds the fracture strain of the feedstock, the micro-structure will fracture and this is at the base of the micro-structure (at the point X in Figure 6.11(b)). For micro-structure with smaller diameter, the possibility of fracture due to shrinking effect will become more apparent.

b) Demolding force for the micro-structures

The demolding force for the individual micro-structure in the cross-sectional plane is calculated and shown in Figure 6.13. The data were taken from the FEM result by summing the reaction forces along the demolding direction of all the nodes (of the FEM model) of the individual micro-structure. For each of the twelve micro-structures, the demolding force at the onset of the demolding is the largest, while the demolding force decreases to a large extent as the micro-structure is ejected out of the micro-cavity. The reasons are that the coefficient of static friction of 0.66 is much higher than the coefficient of sliding friction of 0.37 and the area of the contact

surface between the micro-structure and the micro-cavity becomes smaller and smaller as the micro-structure is ejected out of the micro-cavity. The smaller contact area gives a smaller frictional force during the course of demolding.

Figure 6.13 Demolding force for the individual micro-structures in the case of $T_d < T_{cr}$

The results also show that the farther the micro-structure is away from the centerline of the green part, the larger the demolding force needed to eject the micro-structure out of the micro-cavity. For example, the demolding force needed to eject the farthest micro-structure (No.12) out of the micro-cavity is the largest as compared with the other eleven micro-structures that are closer to the centerline of the green part. A micro-structure that is farther from the centerline will be pushed more severely against the circumferential surface of the micro-cavity due to the shrinkage of the base (of the green part) towards the centerline. This gives a larger contact pressure, consequently the static frictional force needed to move the micro-structure increases.

The maximum demolding force for the array of 144 micro-structures (based on 1/4 simulation model due to symmetry) at the onset of ejection are shown in Figure 6.14. It further illustrates the trend that the demolding force increases as the distance of the micro-structure from the centerline of the green part increases. Moreover, for the micro-structures with the same distance from the centerline of the green part the demolding forces are the same.

Figure 6.14 Demolding forces of the micro-structures in the case of $T_d < T_{cr}$

c) Effects of packing pressure and demolding temperature on demolding force

In order to investigate the effects of packing pressure and demolding temperature on the demolding force, a series of simulations were conducted. The packing pressures of

80 bar, 100 bar and 120 bar were used. The demolding temperatures studied range from 60 °C to 140 °C. Other molding conditions listed in Table 5.1 were used. The demolding force curves at different packing pressures and different demolding temperatures for the array of 576 micro-structures are shown in Figure 6.15.

Figure 6.15 Demolding force curves at different demolding temperatures for the array of 576 micro-structures in the case of $T_d < T_{cr}$

The demolding force for the array of 576 micro-structures increases as the packing pressure decreases. At lower packing pressure, the volumetric expansion due to cavity pressure decrement is smaller. Thus, larger volumetric shrinkage takes place after cooling as compared to the case where higher packing pressure is used. Likewise, larger shrinkage gives larger contact pressure between the micro-structure and the

inner surface of the micro-cavity. Therefore, larger demolding force is needed to eject the micro-structure out of the micro-cavity.

6.3.3 Simulation results in the case of $T_d > T_{cr}$

In the simulations, a packing pressure of 100 bar and a demolding temperature of 125 °C which is higher than the critical temperature of 122 °C were used.

a) Stress distribution during demolding

The stress distribution of the twelve micro-structures in the cross-sectional plane at different demolding displacements is shown in Figure 6.16. All the micro-structures in the array have the same stress distribution. The maximum equivalent Mises stress in the micro-structure always appears at the area which is in contact with the edge of the micro-cavity entrance as shown in Figure 6.16. The maximum equivalent Mises stress of the individual micro-structure decreases as the micro-structure is ejected out of the micro-cavity.

Figure 6.16(a) Just start demolding

Figure 6.16(b) Demolding displacement 40 μm

Figure 6.16(c) Demolding displacement 120 μm

Figure 6.16 Equivalent Mises stress distribution of the cross-section of the micro-structures in the case of $T_d > T_{cr}$

In the case of $T_d > T_{cr}$, the green part and the micro-structures will expand (as compared with the dimensions of the mold cavity and the micro-cavity) at the demolding temperature as they are being ejected out. Before the mold opens, the slightly larger green part (the base and the micro-structures) is constrained in the smaller mold cavity and the micro-cavities, accordingly stress is built up. Even after the mold opens the micro-structures are still constrained inside the micro-cavities and the circumferential surface of the base (of the green part) is constrained.

As the circumferential surface of the base of the green part is constrained by the circumferential surface of the mold cavity (see Figure 6.9), the radial displacing tendency of the micro-structure due to the expansion of the base is hindered. The micro-structure has no radial displacement. Thus, the expansion of the base of the green part does not induce stress to the micro-structures. Compressive stress induced in the micro-structure is due only to the interference fit between the expanded micro-structure and the micro-cavity. All the micro-structures have the same expansion due to the assumption of isotropic expansion. Therefore, they are under the same stress condition and have the same stress distribution during demolding.

Once the demolding is initiated, the micro-structure undergoes shear stress due to the friction between the micro-structure and the circumferential surface of the micro-cavity. The area that is in contact with the edge of the micro-cavity entrance is subjected to a combination of maximum shear stress (due to maximum contact area between the micro-structure and the circumferential surface of the micro-cavity) and

compressive stress due to the expanding tendency of the micro-structure. As the demolding temperature of 125 °C is just slightly higher than T_{cr} , the PEEQ is equal to zero.

b) Demolding force for the micro-structures

The demolding forces for the individual micro-structures in the cross-sectional plane were calculated and shown in Figure 6.17. The required demolding force for any individual micro-structure in the array is the same. The reason is that they have same stress distribution during demolding as shown in Figure 6.16. Just like the case of $T_d < T_{cr}$ (demolding temperature of 105 °C), for all the twelve micro-structures, their demolding forces at the onset of demolding are the largest. The demolding force decreases as the micro-structure is ejected out of the micro-cavity, that is, as the demolding displacement increases. This is because the contact surface area between the micro-structure and the micro-cavity decreases as the demolding displacement increases, giving a smaller frictional force.

Figure 6.17 Demolding force for individual micro-structures for the case of $T_d > T_{cr}$

c) Effects of packing pressure and demolding temperature on demolding force

To investigate the effects of the packing pressure and demolding temperature on the demolding force, a second series of simulations were conducted. Packing pressures of 80 bar, 100 bar and 120 bar were used. The demolding temperatures studied range from 110 °C to 140 °C. The demolding force curves at different demolding temperatures for the array of 576 micro-structures using different packing pressures are shown in Figure 6.18.

Figure 6.18 Simulated demolding force curves at different demolding temperatures for the array of 576 micro-structures in the case of $T_d > T_{cr}$

In the case for $T_d > T_{cr}$ and T_{cr} lower than 135 °C, the demolding force for the array of 576 micro-structures increases as the demolding temperature increases. The micro-structures are under compressive stresses from the interference fit between the

micro-structure and the inner surface of the micro-cavity. The higher the demolding temperature, the lesser the cooling and shrinking effects and therefore the more severe the interference fit. This gives a larger contact pressure and the static frictional force needed to move the micro-structure increases, i.e. the demolding force increases.

However, the gradient of the demolding force curve decreases as the demolding temperature increases, since the feedstock strength decreases as the temperature increases. Accordingly, the demolding force decreases as the feedstock strength decreases. At 135 °C, a balance is struck between an increase in the demolding force due to greater interference fit and a decrease in the demolding force due to a decrease in the feedstock strength. At the demolding temperature of 140 °C, the decrease of the demolding force due to the decrease of the feedstock strength prevails over the increase of the demolding force caused by greater interference fit. Therefore, the demolding force is lower than that at the demolding temperature of 135 °C.

The demolding force for the array of 576 micro-structures increases as the packing pressure increases. At higher packing pressure, the volumetric expansion due to cavity pressure decrement is larger. Thus, the interference fit after cooling is larger as compared to the case where a lower packing pressure is used. Likewise, the greater interference fit gives a larger contact pressure between the micro-structure and the inner surfaces of the micro-cavity. Therefore, a larger demolding force is needed to eject the micro-structures out of the micro-cavities.

6.3.4 Demolding force curves for the case of $T_d < T_{cr}$ and $T_d > T_{cr}$

To show the whole scenario of the effects of packing pressure and demolding temperature on the demolding force, the demolding force curves for the case of $T_d < T_{cr}$ and $T_d > T_{cr}$ are combined as shown in Figure 6.19. It is clear that the temperature giving the lowest demolding force is T_{cr} . Thus, to reduce the possibility of micro-structure fracture, demolding should be conducted at or close to T_{cr} . The trends are the same for the three packing pressures, except that as the packing pressure increases, T_{cr} decreases.

Figure 6.19 Demolding force curves at different demolding temperatures for the array of 576 micro-structures

6.4 Experimental verification of the demolding force curves

In the earlier sections, the demolding of an array of 24×24 (total of 576) $\phi 100 \mu\text{m} \times$ height $200 \mu\text{m}$ micro-structure was analyzed. To verify the results of the analysis, the following experiments were conducted to:

- 1) measure the demolding force at different demolding temperature and packing pressure and
- 2) verify the effects of the distance of the micro-structure from the green part centerline on the demolding force

6.4.1 Demolding force at different demolding temperature and packing pressure

The demolding force was measured on an Instron tensile testing machine (Model 5569). The demolding force needed to eject all the 24×24 (total of 576) micro-structures could not be measured directly because of the frictional force and adhesion force between the surfaces of the base of the green part and the mold cavity wall. An indirect method was used to measure the demolding force as shown in Figure 6.20.

Figure 6.20 Schematic drawing of demolding force measurement

Firstly, the green part was injection molded using a specific packing pressure. The demolding force needed to eject the green part was measured at a specific demolding temperature as shown in Figure 6.20(a). A 500 N load cell was used in the measuring experiments. The peak value F_1 in the upper curve is the maximum demolding force at the onset of movement of the micro-structures. Secondly, the silicon mold insert with micro-cavities was replaced by a silicon mold insert without micro-cavities. Accordingly, a disc with the same dimensions as the base of the green part was injection molded using the same molding parameters. Then the demolding force required to eject the disc was measured as shown in Figure 6.20(b). The peak value F_2 in the lower curve is the maximum demolding force at the onset of the movement of the disc. Finally, the difference between F_1 and F_2 is the demolding force F_D for 24×24 (total of 576) micro-structures for a specific packing pressure.

During the measurement of the demolding force, the mold was maintained at the demolding temperature by heating cartridges. The mold half with the ejection system and green part was detached so that the ejection pins were exposed. During the course of detaching the mold, any contact with the ejection pins was strictly avoided. Then the mold half with the green part was placed onto the Instron tensile testing machine, making sure that the mold half was secured and the top surface was horizontal (using spirit level). The green part was ejected at a speed of 2 mm/s, which is the same as that used on the injection molding machine.

The demolding forces in the case of different packing pressures (80 bar, 100 bar and 120 bar) and demolding temperatures (from 60 to 140 °C) were measured. Three measurements were conducted at each data point. The results were compared with those calculated in the FEM analysis and shown in Figure 6.21.

Figure 6.21 shows the theoretical and measured demolding forces at different demolding temperatures for packing pressures of 80 bar, 100 bar and 120 bar. Both the theoretical and measured demolding force curves show similar trend. The discrepancy between the calculated demolding force and the measured demolding force is below 25 %. The experiments show that the demolding force at the critical temperature T_{cr} is the lowest and this is in accordance with the theoretical results discussed earlier. Therefore, demolding temperature at the critical temperature T_{cr} is the most suitable for successful demolding.

Figure 6.21 Theoretical and measured demolding force at different packing pressure and demolding temperature

6.4.2 Verify the effect of distance from the centerline on the demolding force

As shown in Figure 6.14 in Section 6.3.2 (b), the demolding force of the individual micro-structure was calculated in the case where $T_d < T_{cr}$. The demolding force of the individual micro-structure increases as the distance from the centerline of the green part increases. In the case where $T_d > T_{cr}$, the results in Figure 6.17 in Section 6.3.3 (b) shows that the demolding force of any individual micro-structure in the array is the same. To compare whether the trend is in accordance with the results of the FEM analysis, experiments were designed and conducted for the above two cases.

a) In the case of $T_d < T_{cr}$

To verify the above calculated results, measuring the demolding force of the individual micro-structure is required. However, the demolding force of the individual micro-structure is too small to be precisely measured. Considering that the demolding force for a number of micro-structures in the array is large enough to be detected and measured precisely, all the 24×24 micro-structures were divided into two halves: inner micro-cavities and outer micro-cavities. The demolding forces for these two halves were measured respectively and compared. Three measurements were conducted at each demolding temperature. The average demolding force and its range is shown in Figure 6.22

As shown in Figure 6.22, the inner 288 micro-cavities (half of the total number of the micro-cavities) on the silicon mold insert were blocked using silicone adhesive before

injection molding. After the silicone has solidified, the silicon mold insert was used for injection molding. Hence, only 288 outer micro-structures were molded when using the partially blocked silicon mold insert. In this case, the demolding force for all these outer 288 micro-structures was measured.

Figure 6.22 Different half number of micro-cavities on the silicon mold insert

Likewise, in another case, the outer 288 micro-cavities on the silicon mold insert were blocked using silicone adhesive (without the other inner 288 micro-cavities). Thus, only 288 inner micro-structures were injection molded using the partially blocked silicon mold insert. In this case, the demolding force for all these inner 288 micro-structures was measured.

The packing pressure of 80 bar was used in the verification experiment as the demolding force in this case is larger compared to packing pressures of 100 bar and 120 bar. This helps to reduce the effect of measuring errors. The demolding temperatures of 60 °C, 80 °C and 100 °C were used. The comparison results are shown in Figure 6.23. The experimental results show that demolding force for the outer 288 micro-structures is larger than that for the inner 288 micro-structures. This is in accordance with the theoretical result that the demolding force increases as the distance of the micro-structure from the centerline of the green part increases.

Figure 6.23 Demolding force for inner and outer half micro-structures in the case of $T_d < T_{cr}$

b) In the case of $T_d > T_{cr}$

In this case, the verification experiments were similar to those in the case of $T_d < T_{cr}$. The packing pressure of 120 bar was used in the verification experiment as the demolding force is relatively larger compared with that of 80 bar and 100 bar. Likewise, this helps to reduce the impact of measuring errors. The experimental results are shown in Figure 6.24.

Figure 6.24 Demolding force for inner and outer half micro-structures in the case of $T_d > T_{cr}$

From Figure 6.24, the demolding force for the outer 288 micro-structures is almost equal to that for the inner 288 micro-structures. This is in accordance with the theoretical result that the demolding force of any individual micro-structure in the array is the same.

6.5 Summary

This chapter studies the demolding of micro-structures based on variotherm mold using finite element method. The simulation results show that there exists a “critical temperature” T_{cr} at which the demolding force for the micro-structures array is a minimum. In the case where the demolding temperature $T_d < T_{cr}$, demolding force for the individual micro-structure at the onset of demolding is the maximum and the demolding force for the individual micro-structure decreases as the micro-structure is ejected out of the micro-cavity. The demolding force for the array of 576 micro-structures increases as the demolding temperature decreases and as the packing pressure decreases. In the case where the demolding temperature $T_d > T_{cr}$, the demolding force for the array of 576 micro-structures at the onset of the demolding is the largest too. The demolding force decreases as the micro-structures are ejected out of the micro-cavities. The demolding force increases as the packing pressure increases. In order to reduce the demolding force to the minimum so as to prevent demolding problem, it is recommended to use the critical temperature for demolding. Demolding force measuring experiment was designed to verify the theoretical demolding force (for the array of micro-structures) established in the FEM analysis. The theoretical prediction is in good agreement with the experimental results. Although the experimental results are always higher than the theoretical results, the general trends are similar.

CHAPTER 7

CONCLUSIONS AND FUTURE WORK

7.1 Conclusions

The development of the μ PIM process for the fabrication of an array of 24 x 24 (total of 576) high aspect ratio micro-structures using BASF Catamold 316L stainless steel feedstock was conducted successfully. Silicon mold inserts with micro-cavities etched by DRIE were used.

Initially, injection molding of the micro-structures was conducted using a conventional mold. A series of experiments were conducted to determine suitable injection molding parameters. $\phi 100\ \mu\text{m} \times \text{height } 200\ \mu\text{m}$ micro-structures with aspect ratio of 2 were successfully molded, debound and sintered. However, incomplete filling of the micro-cavities and demolding problem (breakage of micro-structure) occurred when molding $\phi 60\ \mu\text{m} \times \text{height } 191\ \mu\text{m}$ micro-structures with aspect ratio of 3.2.

In order to overcome the problems associated with using the conventional mold and to investigate the feasibility of the variotherm process for μ PIM, a variotherm mold was designed and fabricated. The variotherm mold was equipped with variotherm mold heating/cooling system, vacuum system, hot-sprue system and combined pressure and temperature sensor. The variotherm mold heating/cooling system facilitates the fast

adjustment of the demolding temperature. High mold cavity temperature of 150 °C reduces the heat loss of the feedstock melt during injection molding, thus ensuring the complete filling of the high aspect ratio micro-cavities. The rapid cooling of the mold temperature to a suitable demolding temperature helps to provide strength to the green microstructures, thus eliminating the micro-structure breakage problem. The typical injection molding conditions for the variotherm mold are listed in Table 7.1. Compared with using conventional mold, $\phi 40\text{ }\mu\text{m} \times \text{height } 174\text{ }\mu\text{m}$ micro-structures with aspect ratio of 4.4 and $\phi 20\text{ }\mu\text{m} \times \text{height } 160\text{ }\mu\text{m}$ micro-structures with aspect ratio of 8 were successfully injection molded using the variotherm mold.

Table 7.1 Typical injection molding conditions for the variotherm mold

Parameter	Value
Injection stroke (mm)	19 / 17 / 15 / 15
Injection speed	50 / 40 / 40 / 30 / 20 (%) (93 / 75 / 75 / 56 / 38 (mm/s))
Packing pressure (bar)	100
Packing time (s)	8 (7.5 / 0.5)
Melt temperature (°C)	190 / 185 / 185 / 170
Mold temperature (°C)	150

An FEM analysis (using ABAQUS software) of the demolding of the micro-structures using variotherm mold was conducted with the aim of reducing demolding problem. The analysis shows that there exists a critical temperature T_{cr} for the demolding of the micro-structures. At T_{cr} , the demolding force for the array of microstructures is minimum. T_{cr} is determined by the cavity pressure at the moment where gate solidification occurred and the cavity pressure is affected by the packing pressure. The higher the packing pressure, the lower the critical temperature T_{cr} .

In the case where the demolding temperature $T_d < T_{cr}$, the micro-structures experience both shear stress (resulting from the friction between the micro-structure and the inner surfaces of the micro-cavity) and thermal stress (due to the shrinkage of micro-structure). During the whole course of demolding, the maximum equivalent Mises stress endured by the individual micro-structure increases as its distance from the centerline of the green part increases. As the micro-structure is ejected out of the micro-cavity, the maximum equivalent Mises stress of the micro-structure occurs at the area where it is in contact with the edge of the micro-cavity entrance. However, the magnitude of the maximum equivalent Mises stress for individual micro-structure becomes smaller and smaller as the micro-structure is ejected out.

The farther away the micro-structure is from the centerline of the green part, the larger the demolding force required to eject the individual micro-structure out of the micro-cavity. The demolding force for the individual micro-structure at the onset of demolding is the maximum and the demolding force for the individual micro-structure

decreases as the micro-structure is ejected out of the micro-cavity. The demolding force for the array of 576 micro-structures increases as the demolding temperature decreases and as the packing pressure decreases.

In the case where the demolding temperature $T_d > T_{cr}$, all the micro-structures are under the same stress condition and have the same stress distribution during the course of demolding. The shear stress resulting from friction between the micro-structure and the micro-cavity is the main cause for demolding problems. The maximum equivalent Mises stress in the micro-structure always occurs at the area where the microstructure is in contact with the edge of the micro-cavity entrance. The magnitude of the maximum Mises stress of the individual micro-structure decreases as the micro-structure is ejected out of the micro-cavity. Likewise, the demolding force for the array of 576 micro-structures at the onset of the demolding is the largest. The demolding force decreases as the micro-structures are ejected out of the micro-cavities. The demolding force increases as the packing pressure increases.

Therefore, in both cases of $T_d < T_{cr}$ and $T_d > T_{cr}$, the most critical moment during the demolding is at the onset of the ejection. At this moment, the micro-structures are subjected to maximum equivalent Mises stress and the maximum demolding force. The demolding force at T_{cr} is the minimum.

Demolding force measuring experiments were conducted to verify the theoretical demolding force (for the array of microstructures) established in the FEM analysis.

The experimental results are higher than the theoretical results but the general trends are similar, that is: (a) for $T_d < T_{cr}$, as the demolding temperature increases, the demolding force decreases and (b) for $T_d > T_c$, as the demolding temperature increases, the demolding force increases and (c) a minimum demolding force exists. In order to reduce the demolding force to the minimum so as to prevent demolding problem, it is recommended to use the critical temperature for demolding.

7.2 Future work

Future work are discussed below:

1. Better thermal cycle time

The total cycle time of the existing variotherm mold is around 5.5 min when demolding temperature of 100 °C is used. The cooling rate of 4.2 °C/sec. is sufficiently high but heating rate can be further improved. Because of the space constraints, it is difficult to arrange heating cartridges and cooling channels perfectly to achieve higher heating rate and cooling rate at the same time. The heating rate is restricted due to the large distance between the heating cartridges and the mold cavity. The mold design can be further modified to achieve higher heating rate with little cooling rate loss by adopting new heating methods and adjusting the layout of the heating cartridges locations. For example, more powerful electric heating cartridges or quartz glass induction heating elements can be used, or a small plate of heating elements can be placed directly underneath the mold cavity. Liquid nitrogen could be

used as the cooling medium to increase the cooling rate. The targeted cycle time is less than 3 minutes. However, this may not be practical in production because of high cost.

2. Better way to sense the pressure at individual micro-cavity

In the design, the combined pressure and temperature sensor is positioned at the bottom of the base (of the green part). The measured pressure is not exactly inside the micro-cavity. This is because it is impractical, if not impossible, to machine a $\phi 100\ \mu\text{m}$ hole at the base of the silicon mold insert to install the sensor. Sealing could be a problem after installation. A possible solution is to fabricate built-in pressure sensor inside the micro-cavity using MEMS technology.

3. Non-isotropic behavior of the feedstock

In Chapter 6, it was assumed that the shrinkage/expansion of the feedstock is isotropic. This is because the feedstock was cooled rapidly from $150\ ^\circ\text{C}$ to $100\ ^\circ\text{C}$ in just 12 sec. The high cooling rate avoids causing a large temperature gradient in the green part (the base and the micro-structures). However, a more accurate analysis that considers non-isotropic behavior of feedstock could be studied.

4. Effects of different spatial configuration

Silicon mold inserts with a fixed pitch of $200\ \mu\text{m}$ were used in this research work. Smaller or larger pitches can be used in the simulation and experimental work. In the

case of small pitch for large-area micro-structures molding the interference between the micro-structures can be investigated.

5. Effects of different geometrical configuration

Cylindrical micro-structures were injection molded in this research work. The effects of different geometrical configuration on micro-structure demolding can be studied. For example, the micro-structures with square or triangle cross-section or asymmetrically-distributed micro-structures can be used for theoretical and experimental analysis.

6. Design of thermal stress absorber

A thermal stress absorber can be designed on the silicon mold insert in order to reduce the thermal stress due to thermal shrinkage/expansion of the feedstock. For example, a strong circular rib with large width (in the radial direction of the green part) can be designed surrounding the microstructured area on the base of the green part. During demolding, this circular rib can act as stress absorber so that the thermal stress in the micro-structures will be reduced.

7. Effects of different particle size

Commercial feedstock with $D_{50} = 4 \mu\text{m}$ was used in this research work. In future work, in-house feedstock can be mixed using powders with different particle sizes. The effects of particle size on the micro-structure demolding can be studied.

8. Effects of types of powder

In this research work, the commercial BASF Catamold 316L stainless steel feedstock was used. However, the effects of different types of powders with different particle shapes on demolding can also be studied.

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APPENDIX

A sample of Abaqus program in this research is shown as below:

<pre> *Heading ** Job name: 100bar-105C Model name: Model-1 *Preprint, echo=NO, model=NO, history=NO, contact=NO ** ** PARTS ** *Part, name=mold *End Part ** *Part, name=molding *End Part ** ** ** ASSEMBLY ** *Assembly, name=Assembly ** *Instance, name=molding-1, part=molding *Node *Element, type=C3D8R ** Region: (feedstock_sec:Picked) ** Section: feedstock_sec *Solid Section, elset=_PickedSet28, material=feedstock 1., *End Instance ** *Instance, name=mold-1, part=mold *Element, type=C3D8R ** Region: (silicon_sec:Picked) ** Section: silicon_sec *Solid Section, elset=_PickedSet27, material=silicon 1., *End Instance ** *Nset, nset=cross_section, instance=molding-1 *Elset, elset=cross_section, instance=molding-1 *Nset, nset=s-1, instance=molding-1 *Elset, elset=s-1, instance=molding-1 *Nset, nset=s-1, instance=molding-1 *Elset, Elset=s-1, instance=molding-1 *Nset, nset=s-2, instance=molding-1 *Elset, Elset=s-2, instance=molding-1 *Nset, nset=s-3, instance=molding-1 *Elset, Elset=s-3, instance=molding-1 *Nset, nset=s-4, instance=molding-1 </pre>	<pre> *Elset, Elset=s-4, instance=molding-1 *Nset, nset=s-5, instance=molding-1 *Elset, Elset=s-5, instance=molding-1 *Nset, nset=s-6, instance=molding-1 *Elset, Elset=s-6, instance=molding-1 *Nset, nset=s-7, instance=molding-1 *Elset, Elset=s-7, instance=molding-1 *Nset, nset=s-8, instance=molding-1 *Elset, Elset=s-8, instance=molding-1 *Nset, nset=s-9, instance=molding-1 *Elset, Elset=s-9, instance=molding-1 *Nset, nset=s-10, instance=molding-1 *Elset, Elset=s-10, instance=molding-1 *Nset, nset=s-11, instance=molding-1 *Elset, Elset=s-11, instance=molding-1 *Nset, nset=s-12, instance=molding-1 *Elset, Elset=s-12, instance=molding-1 *Nset, nset=s-whole, instance=molding-1 *Elset, Elset=s-whole, instance=molding-1 *Surface, type=ELEMENT, name=_PickedSurf7, internal *Surface, type=ELEMENT, name=_PickedSurf8, internal *Surface, type=ELEMENT, name=_PickedSurf9, internal *Surface, type=ELEMENT, name=_PickedSurf10, internal type=ELEMENT, *Surface, type=ELEMENT, name=_PickedSurf11, internal type=ELEMENT, *Surface, type=ELEMENT, name=_PickedSurf12, internal type=ELEMENT, *Surface, type=ELEMENT, name=_PickedSurf13, internal type=ELEMENT, *Surface, type=ELEMENT, name=_PickedSurf14, internal type=ELEMENT, *Surface, type=ELEMENT, name=_PickedSurf15, internal type=ELEMENT, *Surface, type=ELEMENT, name=_PickedSurf16, internal type=ELEMENT, *Surface, type=ELEMENT, name=_PickedSurf17, internal type=ELEMENT, *Surface, type=ELEMENT, name=_PickedSurf18, internal type=ELEMENT, *Surface, type=ELEMENT, name=_PickedSurf19, internal type=ELEMENT, *Surface, type=ELEMENT, name=_PickedSurf20, internal </pre>
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*Surface, name=_PickedSurf21, internal	type=ELEMENT,	1.70358e+06, 0.00123329
*Surface, name=_PickedSurf22, internal	type=ELEMENT,	1.74768e+06, 0.00137545
*Surface, name=_PickedSurf23, internal	type=ELEMENT,	1.78572e+06, 0.00152508
*Surface, name=_PickedSurf24, internal	type=ELEMENT,	1.82836e+06, 0.00166756
*Surface, name=_PickedSurf25, internal	type=ELEMENT,	1.87377e+06, 0.00180355
*Surface, name=_PickedSurf26, internal	type=ELEMENT,	1.90947e+06, 0.00195856
*Surface, name=_PickedSurf27, internal	type=ELEMENT,	1.96285e+06, 0.00208581
*Surface, name=_PickedSurf28, internal	type=ELEMENT,	1.99589e+06, 0.00224124
*Surface, name=_PickedSurf29, internal	type=ELEMENT,	2.03927e+06, 0.002383
*Surface, name=_PickedSurf30, internal	type=ELEMENT,	2.08137e+06, 0.00252748
*Surface, name=_PickedSurf31, internal	type=ELEMENT,	2.12406e+06, 0.00266888
*Surface, name=_PickedSurf32, internal	type=ELEMENT,	2.16531e+06, 0.00281628
*Surface, name=_PickedSurf33, internal	type=ELEMENT,	2.21028e+06, 0.00295039
*Surface, name=_PickedSurf34, internal	type=ELEMENT,	2.24942e+06, 0.00310169
*Surface, name=_PickedSurf35, internal	type=ELEMENT,	2.29262e+06, 0.00324285
*Surface, name=_PickedSurf36, internal	type=ELEMENT,	2.34229e+06, 0.00337816
*Surface, name=_PickedSurf37, internal	type=ELEMENT,	2.37669e+06, 0.00352983
*Surface, name=_PickedSurf38, internal	type=ELEMENT,	2.42704e+06, 0.00366011
*End Assembly	type=ELEMENT,	2.46316e+06, 0.00381142
**		2.5023e+06, 0.00395922
** MATERIALS		2.55654e+06, 0.00408754
**		2.58956e+06, 0.0042435
*Material, name=feedstock		2.63578e+06, 0.00438065
*Elastic		2.67365e+06, 0.00453105
6.02e+08, 0.274		2.71764e+06, 0.0046739
*Expansion, zero=129.11		2.75854e+06, 0.00481977
4.87e-05,		2.7948e+06, 0.00497085
*Plastic		2.8398e+06, 0.00510802
1.20428e+06, 0.		2.87607e+06, 0.00526309
1.25133e+06, 6.0262e-05		2.91437e+06, 0.00541138
1.31369e+06, 0.000172098		2.95381e+06, 0.00555968
1.36877e+06, 0.000291924		2.99296e+06, 0.00570796
1.4336e+06, 0.000401658		3.03145e+06, 0.00585744
1.47763e+06, 0.000539836		3.06828e+06, 0.00601157
1.53682e+06, 0.000655936		3.10708e+06, 0.00616054
1.5698e+06, 0.000814465		3.13923e+06, 0.00631944
1.62683e+06, 0.000934053		3.17455e+06, 0.00647409
1.66479e+06, 0.00108391		3.2059e+06, 0.00663542
		3.24014e+06, 0.00679536
		3.26832e+06, 0.00695996
		3.29114e+06, 0.00713286
		3.31572e+06, 0.00730844
		3.33374e+06, 0.00749181
		3.35394e+06, 0.00767307
		3.36197e+06, 0.00787213
		3.36654e+06, 0.00807484
		3.37753e+06, 0.00827349
		3.3753e+06, 0.00848849
		3.38899e+06, 0.00868056
		3.38558e+06, 0.00890012
		3.39133e+06, 0.00910287
		3.40239e+06, 0.0093009
		3.39812e+06, 0.0095193
		3.41069e+06, 0.00970872
		3.41485e+06, 0.00991921
		3.41819e+06, 0.010127

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3.42789e+06, 0.0103227	_PickedSet36, 3, 3
3.42747e+06, 0.0105373	** Name: vertical_symmetry Type:
3.43494e+06, 0.0107361	Symmetry/Antisymmetry/Encastre
3.44471e+06, 0.0109378	*Boundary
3.45167e+06, 0.0111376	_PickedSet34, YSYMM
3.4488e+06, 0.0113541	**
3.45535e+06, 0.0115592	** FIELDS
3.46106e+06, 0.0117605	**
3.45823e+06, 0.0119801	** Name: temperature Type: Temperature
3.46614e+06, 0.0121782	*Initial Conditions, type=TEMPERATURE
3.46878e+06, 0.0123886	_PickedSet410, 129.11
3.47608e+06, 0.0125934	**
3.46964e+06, 0.0128149	** INTERACTIONS
3.47561e+06, 0.0130204	**
3.47645e+06, 0.0132293	** Interaction: contact-1
3.47671e+06, 0.0134432	*Contact Pair, interaction=_contact-1-Property
3.48147e+06, 0.0136502	_PickedSurf8, _PickedSurf7
*Material, name=silicon	** Interaction: contact-10
*Elastic	*Contact Pair, interaction=_contact-10-Property
1.6e+11, 0.22	_PickedSurf26, _PickedSurf25
*Expansion	** Interaction: contact-11
2.5e-06,	*Contact Pair, interaction=_contact-11-Property
**	_PickedSurf28, _PickedSurf27
** INTERACTION PROPERTIES	** Interaction: contact-12
**	*Contact Pair, interaction=_contact-12-Property
*Surface Interaction, name=_contact-1-Property	_PickedSurf30, _PickedSurf29
1.,	** Interaction: contact-2
*Friction, slip tolerance=0.005	*Contact Pair, interaction=_contact-2-Property
0.66,	_PickedSurf10, _PickedSurf9
*Surface Behavior, pressure-overclosure=HARD	** Interaction: contact-3
*Surface Interaction, name=_contact-10-Property	*Contact Pair, interaction=_contact-3-Property
1.,	_PickedSurf12, _PickedSurf11
*Friction, slip tolerance=0.005	** Interaction: contact-4
0.66,	*Contact Pair, interaction=_contact-4-Property
*Surface Behavior, pressure-overclosure=HARD	_PickedSurf14, _PickedSurf13
*Surface Interaction, name=_contact-11-Property	** Interaction: contact-5
1.,	*Contact Pair, interaction=_contact-5-Property
*Friction, slip tolerance=0.005	_PickedSurf16, _PickedSurf15
0.66,	** Interaction: contact-6
*Surface Behavior, pressure-overclosure=HARD	*Contact Pair, interaction=_contact-6-Property
*Surface Interaction, name=_contact-12-Property	_PickedSurf18, _PickedSurf17
1.,	** Interaction: contact-7
*Friction, slip tolerance=0.005	*Contact Pair, interaction=_contact-7-Property
0.66,	_PickedSurf20, _PickedSurf19
*Surface Behavior, pressure-overclosure=HARD	** Interaction: contact-8
*Surface Interaction, name=_contact-2-Property	*Contact Pair, interaction=_contact-8-Property
1.,	_PickedSurf22, _PickedSurf21
*Friction, slip tolerance=0.005	** Interaction: contact-9
0.66,	*Contact Pair, interaction=_contact-9-Property
*Surface Behavior, pressure-overclosure=HARD	_PickedSurf24, _PickedSurf23
*Surface Interaction, name=_contact-3-Property	** -----
1.,	-----
*Friction, slip tolerance=0.005	**
0.66,	** STEP: fit
*Surface Behavior, pressure-overclosure=HARD	**
*Surface Interaction, name=_contact-4-Property	*Step, name=fit

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<pre> *Static 1., 1., 1e-05, 1. ** ** FIELDS ** ** Name: temperature Type: Temperature *Temperature _PickedSet410, 105. ** ** OUTPUT REQUESTS ** *Restart, write, frequency=0 ** ** FIELD OUTPUT: F-Output-1 ** *Output, field *Node Output CF, RF, U *Element Output, directions=YES LE, PE, PEEQ, PEMAG, PS, S *Contact Output CDISP, CSTRESS ** ** HISTORY OUTPUT: H-Output-1 ** *Output, history, variable=PRESELECT *End Step ** ----- ** ** STEP: static ** *Step, name=static *Static 0.25, 1., 1e-05, 1. ** ** BOUNDARY CONDITIONS ** ** Name: ejection Type: Displacement/Rotation *Boundary _PickedSet37, 3, 3, -6e-06 ** ** LOADS ** ** Name: sticking Type: Pressure *Dload _PickedSurf38, P, -20000. ** ** FIELDS ** ** Name: temperature Type: Temperature *Temperature _PickedSet410, 105. ** ** OUTPUT REQUESTS ** </pre>	<pre> *Restart, write, frequency=0 ** ** FIELD OUTPUT: F-Output-1 ** *Output, field *Node Output CF, RF, U *Element Output, directions=YES LE, PE, PEEQ, PEMAG, PS, S *Contact Output CDISP, CSTRESS ** ** HISTORY OUTPUT: H-Output-1 ** *Output, history, variable=PRESELECT *End Step ** ----- ** ** STEP: ejection-20 ** *Step, name=ejection-20 *Static 0.2, 0.25, 2.5e-06, 0.25 ** ** BOUNDARY CONDITIONS ** ** Name: ejection Type: Displacement/Rotation *Boundary _PickedSet37, 3, 3, -1.4e-05 ** ** LOADS ** ** Name: sticking Type: Pressure *Dload _PickedSurf38, P, 0. ** ** FIELDS ** ** Name: temperature Type: Temperature *Temperature _PickedSet410, 105. ** ** INTERACTIONS ** ** Interaction: contact-1 *Change Friction, interaction=_contact-1- Property *Friction, slip tolerance=0.005 0.37, ** Interaction: contact-10 *Change Friction, interaction=_contact-10- Property *Friction, slip tolerance=0.005 0.37, ** Interaction: contact-11 </pre>
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<pre> *Change Friction, interaction=_contact-11- Property *Friction, slip tolerance=0.005 0.37, ** Interaction: contact-12 *Change Friction, interaction=_contact-12- Property *Friction, slip tolerance=0.005 0.37, ** Interaction: contact-2 *Change Friction, interaction=_contact-2- Property *Friction, slip tolerance=0.005 0.37, ** Interaction: contact-3 *Change Friction, interaction=_contact-3- Property *Friction, slip tolerance=0.005 0.37, ** Interaction: contact-4 *Change Friction, interaction=_contact-4- Property *Friction, slip tolerance=0.005 0.37, ** Interaction: contact-5 *Change Friction, interaction=_contact-5- Property *Friction, slip tolerance=0.005 0.37, ** Interaction: contact-6 *Change Friction, interaction=_contact-6- Property *Friction, slip tolerance=0.005 0.37, ** Interaction: contact-7 *Change Friction, interaction=_contact-7- Property *Friction, slip tolerance=0.005 0.37, ** Interaction: contact-8 *Change Friction, interaction=_contact-8- Property *Friction, slip tolerance=0.005 0.37, ** Interaction: contact-9 *Change Friction, interaction=_contact-9- Property *Friction, slip tolerance=0.005 0.37, ** ** OUTPUT REQUESTS ** *Restart, write, frequency=0 ** ** FIELD OUTPUT: F-Output-1 ** </pre>	<pre> *Output, field *Node Output CF, RF, U *Element Output, directions=YES LE, PE, PEEQ, PEMAG, PS, S *Contact Output CDISP, CSTRESS ** ** HISTORY OUTPUT: H-Output-1 ** *Output, history, variable=PRESELECT *End Step ** ----- ** ** STEP: ejection-40 ** *Step, name=ejection-40 *Static 0.25, 0.25, 2.5e-06, 0.25 ** ** BOUNDARY CONDITIONS ** ** Name: ejection Type: Displacement/Rotation *Boundary _PickedSet37, 3, 3, -2e-05 ** ** FIELDS ** ** Name: temperature Type: Temperature *Temperature _PickedSet410, 105. ** ** OUTPUT REQUESTS ** *Restart, write, frequency=0 ** ** FIELD OUTPUT: F-Output-1 ** *Output, field *Node Output CF, RF, U *Element Output, directions=YES LE, PE, PEEQ, PEMAG, PS, S *Contact Output CDISP, CSTRESS ** ** HISTORY OUTPUT: H-Output-1 ** *Output, history, variable=PRESELECT *End Step ** ----- ** ** STEP: ejection-60 ** </pre>
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<pre> *Step, name=ejection-60 *Static 0.25, 0.25, 2.5e-06, 0.25 ** ** FIELDS ** ** Name: temperature Type: Temperature *Temperature PickedSet410, 105. ** ** OUTPUT REQUESTS ** *Restart, write, frequency=0 ** ** FIELD OUTPUT: F-Output-1 ** *Output, field *Node Output CF, RF, U *Element Output, directions=YES LE, PE, PEEQ, PEMAG, PS, S *Contact Output CDISP, CSTRESS ** ** HISTORY OUTPUT: H-Output-1 ** *Output, history, variable=PRESELECT *End Step ** ----- ----- ** ** STEP: ejection-80 ** *Step, name=ejection-80 *Static 0.25, 0.25, 2.5e-06, 0.25 ** ** FIELDS ** ** Name: temperature Type: Temperature *Temperature PickedSet410, 105. ** ** OUTPUT REQUESTS ** *Restart, write, frequency=0 ** ** FIELD OUTPUT: F-Output-1 ** *Output, field *Node Output CF, RF, U *Element Output, directions=YES LE, PE, PEEQ, PEMAG, PS, S *Contact Output CDISP, CSTRESS </pre>	<pre> ** ** FIELD OUTPUT: F-Output-1 ** *Output, field *Node Output CF, RF, U *Element Output, directions=YES LE, PE, PEEQ, PEMAG, PS, S *Contact Output CDISP, CSTRESS ** ** HISTORY OUTPUT: H-Output-1 ** *Output, history, variable=PRESELECT *End Step ** ----- ----- ** ** STEP: ejection-140 ** *Step, name=ejection-140 *Static 0.25, 0.25, 2.5e-06, 0.25 ** ** FIELDS ** ** Name: temperature Type: Temperature *Temperature PickedSet410, 105. ** ** OUTPUT REQUESTS ** *Restart, write, frequency=0 ** ** FIELD OUTPUT: F-Output-1 ** *Output, field *Node Output CF, RF, U *Element Output, directions=YES LE, PE, PEEQ, PEMAG, PS, S *Contact Output CDISP, CSTRESS ** ** HISTORY OUTPUT: H-Output-1 ** *Output, history, variable=PRESELECT *End Step ** ----- ----- ** ** STEP: ejection-160 ** *Step, name=ejection-160 *Static 0.25, 0.25, 2.5e-06, 0.25 </pre>
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<pre> ** ** FIELDS ** ** Name: temperature Type: Temperature *Temperature _PickedSet410, 105. ** ** OUTPUT REQUESTS ** *Restart, write, frequency=0 ** ** FIELD OUTPUT: F-Output-1 ** *Output, field *Node Output CF, RF, U *Element Output, directions=YES LE, PE, PEEQ, PEMAG, PS, S *Contact Output CDISP, CSTRESS ** ** HISTORY OUTPUT: H-Output-1 ** *Output, history, variable=PRESELECT *End Step ** ----- ----- ** ** STEP: ejection-180 ** *Step, name=ejection-180 *Static 0.25, 0.25, 2.5e-06, 0.25 ** ** FIELDS ** ** Name: temperature Type: Temperature *Temperature _PickedSet410, 105. ** ** OUTPUT REQUESTS ** *Restart, write, frequency=0 ** ** FIELD OUTPUT: F-Output-1 ** *Output, field *Node Output CF, RF, U *Element Output, directions=YES LE, PE, PEEQ, PEMAG, PS, S *Contact Output CDISP, CSTRESS ** ** HISTORY OUTPUT: H-Output-1 ** </pre>	<pre> *Output, history, variable=PRESELECT *End Step ** ----- ----- ** ** STEP: ejection-198 ** *Step, name=ejection-198 *Static 0.25, 0.25, 2.5e-06, 0.25 ** ** BOUNDARY CONDITIONS ** ** Name: ejection Type: Displacement/Rotation *Boundary _PickedSet37, 3, 3, -1.8e-05 ** ** FIELDS ** ** Name: temperature Type: Temperature *Temperature _PickedSet410, 105. ** ** OUTPUT REQUESTS ** *Restart, write, frequency=0 ** ** FIELD OUTPUT: F-Output-1 ** *Output, field *Node Output CF, RF, U *Element Output, directions=YES LE, PE, PEEQ, PEMAG, PS, S *Contact Output CDISP, CSTRESS ** ** HISTORY OUTPUT: H-Output-1 ** *Output, history, variable=PRESELECT *End Step ** ----- ----- ** ** STEP: ejection-203 ** *Step, name=ejection-203 *Static 0.25, 0.25, 2.5e-06, 0.25 ** ** BOUNDARY CONDITIONS ** ** Name: ejection Type: Displacement/Rotation *Boundary _PickedSet37, 3, 3, -5e-06 ** </pre>
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<pre> ** FIELDS ** ** Name: temperature Type: Temperature *Temperature _PickedSet410, 105. ** ** OUTPUT REQUESTS ** *Restart, write, frequency=0 ** ** FIELD OUTPUT: F-Output-1 ** *Output, field *Node Output CF, RF, U *Element Output, directions=YES LE, PE, PEEQ, PEMAG, PS, S *Contact Output CDISP, CSTRESS ** ** HISTORY OUTPUT: H-Output-1 ** *Output, history, variable=PRESELECT *End Step </pre>	
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