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Micro Metal Powder Injection Molding

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1. Introduction

Powder injection molding (PIM), which encompasses metal powder injection molding (MIM) and ceramic powder injection molding (CIM) is a net-shape process for the manufacturing of high volume and high precision components for use in a variety of industries. The micro-miniaturization of dimension and structures in MIM is facing with various technical problems, such as incomplete filling to narrow cavity, failure in demolding of fragile green compacts, and deformation in debinding and sintering process. Therefore micro MIM (μ -MIM) process is a more sophisticated process for tiny metal components and micro structured parts. This chapter introduces a general flow of MIM process, the material properties of the feedstock and focuses on the unique phenomena in the micro injection molding and the filling behaviour. A flow simulation of micro gear and micro dumbbell tensile specimen will be carried out and the flow pattern by short shot test and internal pressure measured will be compared to the simulation results. The production method of micro sacrificial plastic mold insert MIM (μ -SPiMIM) process has been proposed to solve drastically the specific problems involving the miniaturization of MIM parts. The sacrificial plastic mold (SP-mold) is prepared by injection-molding polymethylmethacrylate (PMMA) polymer into Ni-electroform. Micro-sized stainless steel 316L powder feedstock is injection-molded into the SP-mold which consists of micro multi-pillar structures. The effects of metal particle size and processing conditions on the quality of molded and sintered parts are evaluated. For the higher quality of μ -SPiMIM process, the feedstock composed of nano-sized Cu powder and oxymethylene-based binder is adequately prepared and molded into PMMA films with fine line-scan structures which are prepared by nano-imprint lithography (NIL) technique. From the evaluation results on the effects of particle size of metal powder and processing conditions toward the high precision of sintered parts, it is concluded that the μ -SPiMIM process has a great potential to produce precisely the complex metallic parts with fine micro-structures.

1.1 What is metal powder injection molding (MIM)?

Metal powder injection molding (MIM) is a manufacturing method that combines traditional powder metallurgy (P/M) with plastic injection molding as shown in Fig.1. Over the past decade it has established itself as a competitive manufacturing process for small precision components that would be costly to produce by alternative methods. It can be used to produce comparatively small parts with complex shapes from almost all types of materials such as metals, ceramics, inter-metallic compounds, and composites (German, 1984). Recently MIM

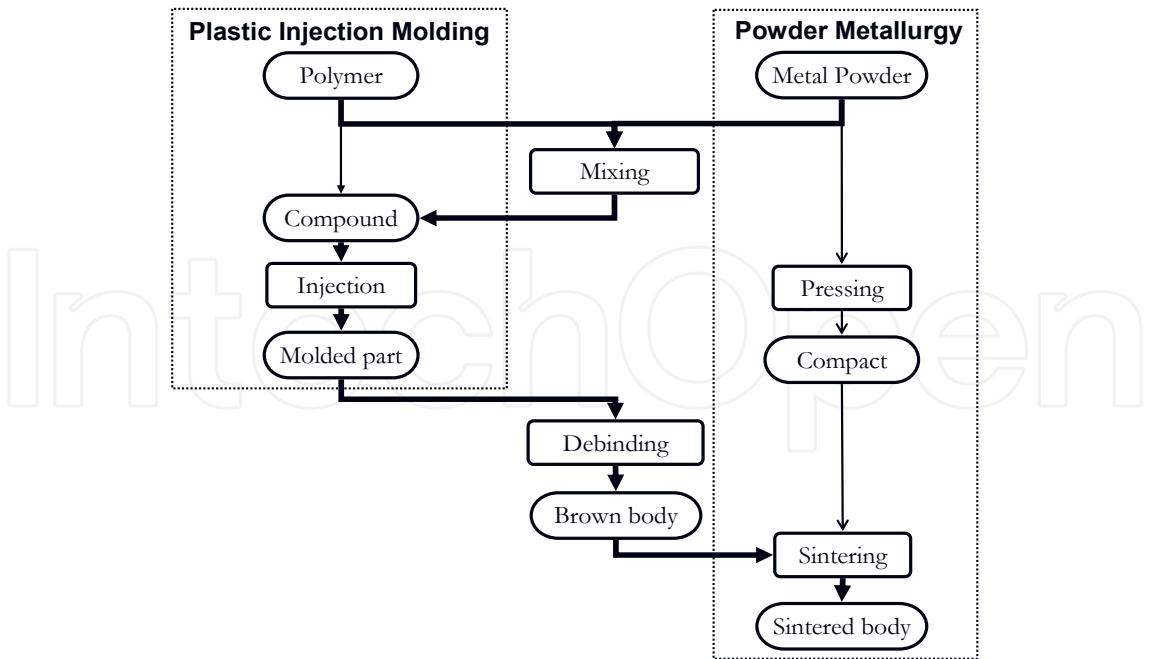


Fig. 1. Flow of metal powder injection molding (MIM) process indicated by bold line.

has been studied not only for hard metals, but also for materials such as titanium, copper and aluminium (German and Bose, 1997). Unlike in the case of P/M, MIM requires mixing metal powders with a large amount of polymeric binder. Afterwards the organic constituents are removed in a debinding step using solvent extraction or pyrolysis. The brown body is held in the molded form by only metal powder after debinding. The mixing and debinding of polymer with metal powder is a very unique and an original process of MIM. Thus it is very effective for manufacturing higher functional metal parts to apply this unique process.

1.2 Micro-miniaturization techniques by MIM

Fig.2 shows some typical examples of commercial MIM products, such as connector, impeller, cam, planetary-gear set and micro-reactor. In these conventional MIM products, as the size become smaller, the dimensional accuracy becomes higher as it falls within a few tenth micrometers. As the size of MIM products decreases much further, the dimensional accuracy is hoping to ensure within a few micrometers. In practical productions, however, it cannot be achieved easily. The production method of these tiny metallic parts which have micro-size and micro-structure are called μ -MIM, which is capable of manufacturing the micro-structured parts such as micro-pillars (Löhe and Haußelt, 2005).

1.3 Technical problems and solutions to miniaturize MIM parts

The μ -MIM process is very useful for the manufacturing of micro-sized and micro-structured metallic parts, but it is facing with various technical problems in each process. For example, it is difficult to fill feedstock completely into a narrow cavity and to demold fragile green compacts from a metallic mold in injection molding process. A careful handling is also required in the debinding and sintering processes. There are many other technical problems such as measuring of the density, the shape and mechanical properties of

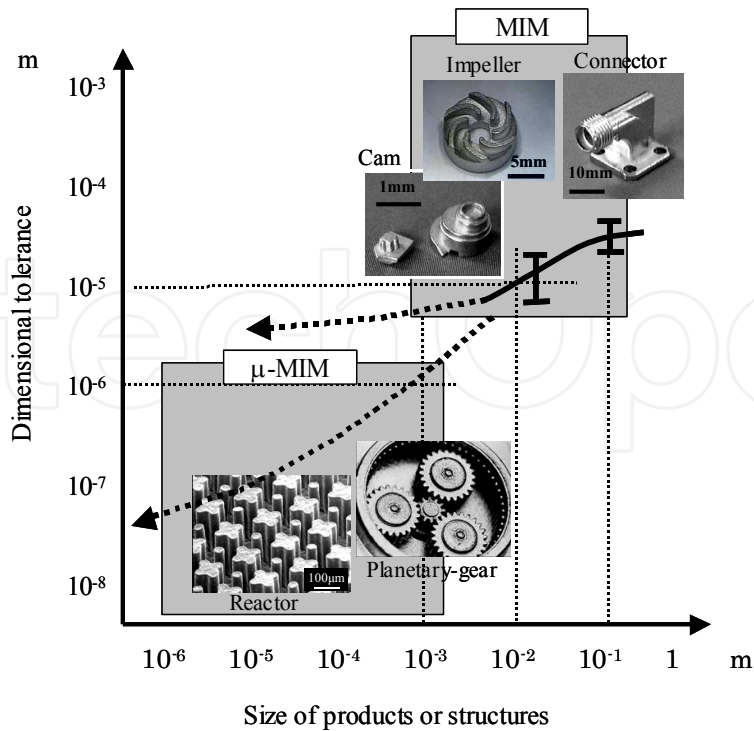


Fig. 2. Dimensional tolerance versus size of MIM Products.

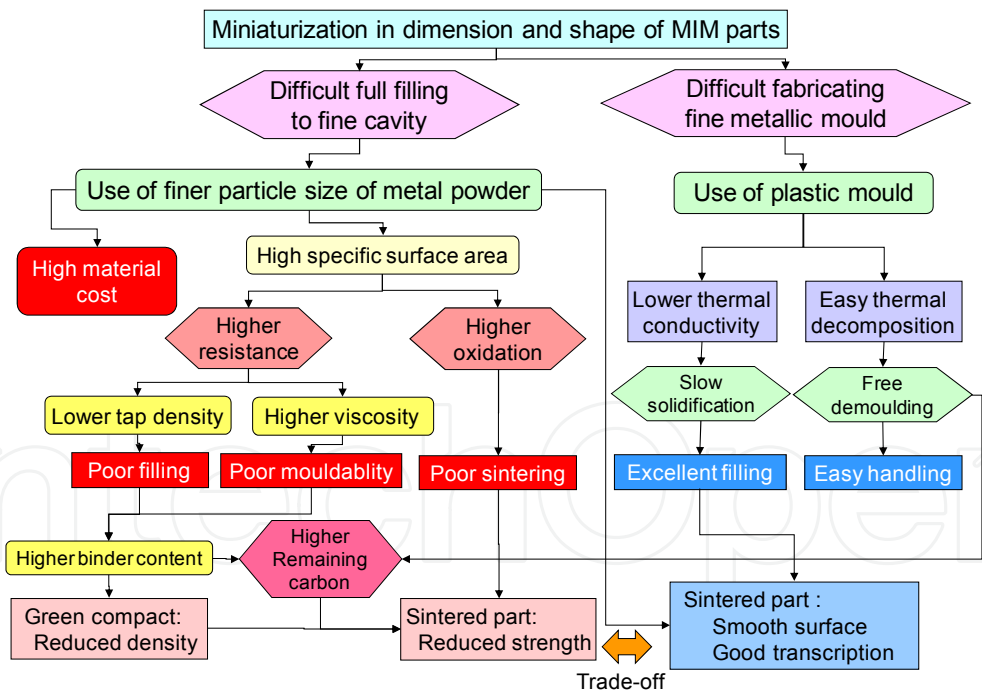


Fig. 3. Technical problems and its solutions to miniaturize MIM parts.

sintered parts. In Fig.3, the solutions for filling of viscous feedstock into the fine mold and fabricating of fine mold are shown in addition to the effectiveness and the disadvantages. The use of finer powder is essential to fill the feedstock into several tenth and a few micron-sized cavity. However, nano-sized powder has extremely high specific surface area, thus the tap density is very low and the viscosity of the feedstock increases remarkably. It is also

susceptible to oxidation and relatively high production cost. Therefore these properties result in adverse affect for the production and the utilization. The fluidity of the feedstock is improved generally by increasing the binder content, but it makes lower the quality and the mechanical property of the sintered parts because the density of green compact become lower and the remaining carbon content increases. On the other hand, it is not easy to fabricate precisely a metallic mold, but a plastic is more superior for processability of the fine mold. In addition, a plastic mold is much superior for filling of the feedstock because its low thermal conductivity which delays solidification of melted materials. Moreover it is not necessary to demold a green compact from a plastic mold which can be decomposed thermally in debinding process. As a result, if there is a solution to the problem in sintering and injection of finer metal powder into plastic mold, a sintered part with smooth surface and good transcription can expect to be manufactured by MIM process. Therefore the trade-off problem needs to be solved by any innovative technologies.

2. Metal powder injection molding for small components

2.1 Material properties of MIM feedstock

The metallic powders used for MIM process are plain and low alloy steels, high speed steels, stainless steels, superalloys, intermetallics, magnetic alloys, hardmetals, and titanium and so on (Osada et al, 2007). Among them, stainless steels are commonly used. Fig.4 shows distributions of particle diameter of 316L stainless steel powder (10 μ m and 2 μ m in mean diameter, Epson Atmix Co., Ltd., PF-20J, PF-2F) produced by water-atomization method. Fine powders sintered more readily than coarser ones, but there is a number of limiting factors. The metallic powders are compounded with wax and polymeric binder by high-pressure kneader and are granulated by plunger-type extruder. The least possible amount of binder should be used, but an appropriate volume fraction of binder to powder exists. In industrial practice, the ratio varies from about 0.5 to 0.7.

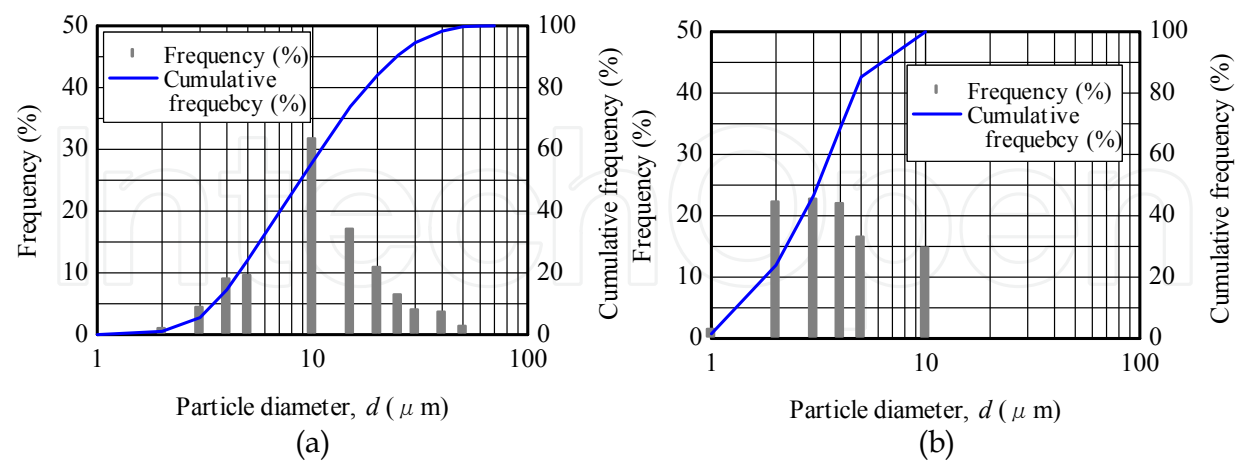


Fig. 4. Distributions of particle size of stainless steel powders used for MIM feedstock; (a) 316L powder (D_{50} =10 μ m); (b) 316L powder (D_{50} =2 μ m).

Fig.5 shows the melt viscosity of the feedstock with various fractions of 316L stainless steel powders with different mean particle sizes, which was measured by a capillary graph. The binder used for the feedstock was polyacetal polymer and paraffin wax. The volume

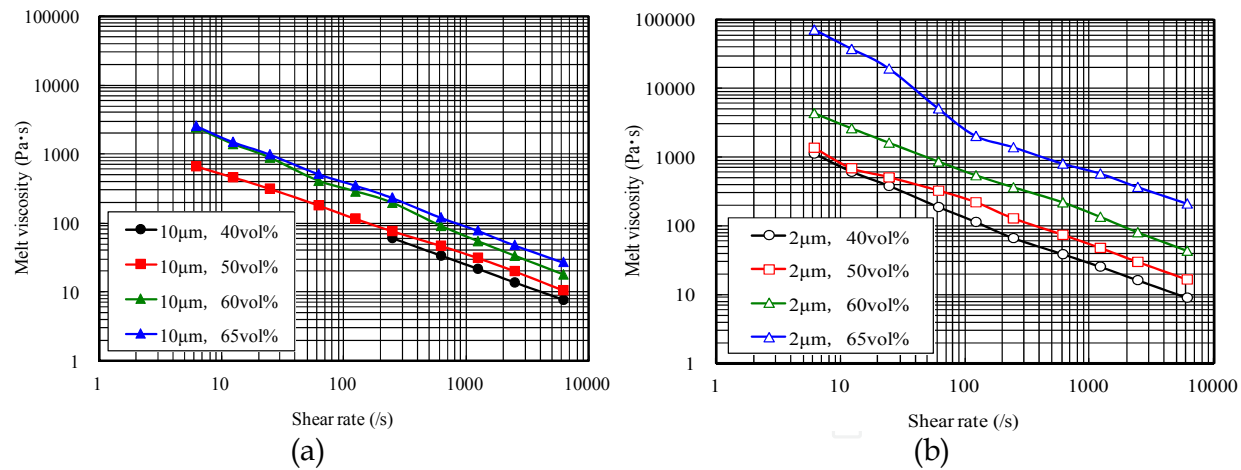


Fig. 5. Melt viscosities of MIM feedstock with various fractional metal powders; (a) 316L powder (D_{50} =10 μ m) feedstock; (b) 316L powder (D_{50} =2 μ m) feedstock.

fraction of the metal powder was varied from 40 to 65%. It is obvious that the melt viscosity of the feedstock increases remarkably as volume fraction of metal powder increases, and fine powder makes the melt viscosity of feedstock much higher than the coarse one.

2.2 Effects of capacity of mixing machine and injection molding machine

The feedstock used for MIM is generally prepared by mixing metal powder and binders with a twin screw extruder and kneader. A highly-homogenized feedstock is significant to manufacture the high quality of sintered parts, but it is not easy due to a big difference in specific gravity between metal powder and binders. The homogeneity of the feedstock was evaluated with the coefficient of variation (CV). Fig.6 shows the CV values of binder weight contained in a pellet of feedstock which was prepared with various mixing volumes such as 100cc, 1000cc and 40,000cc in a lot. The experimental result shows that the variation of binder content can reduce significantly with decreasing of the mixing volume.

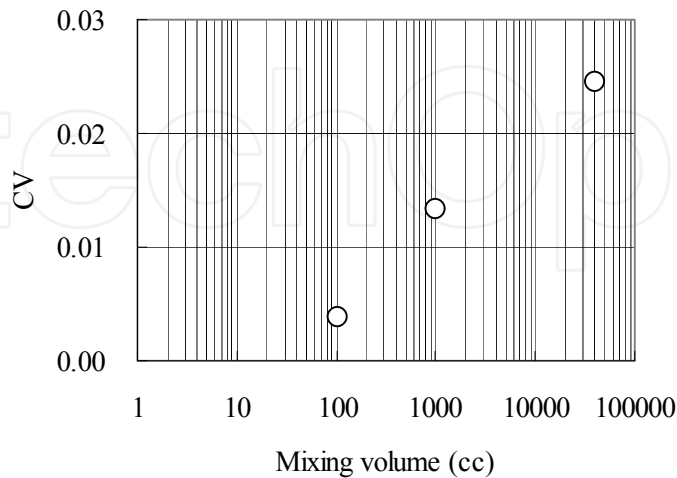


Fig. 6. Variation of binder weight in a pellet prepared with various mixing volumes.

Fig.7. shows the distributions of weight of green compacts prepared by two types of injection molding machines with varied capacities. Micro injection molding machine

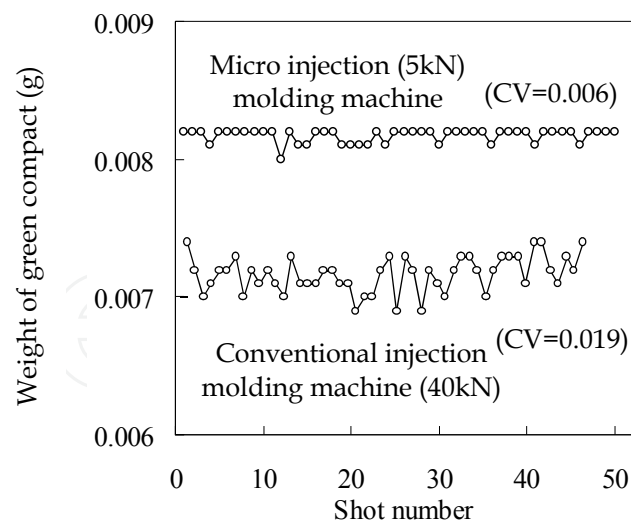


Fig. 7. Distributions of weight of green compacts prepared by injection molding machines with varied capacities.

(Battenfeld GmbH, Microsystem50) has 50kN clamping force and is smaller than conventional one with 400kN (Nissei Plastic Industrial Co., Ltd., PN40-2H). The diameter of injection plunger in micro injection molding machines is 3mm which is much smaller than that of 19mm in conventional one. Minimum volume for fabricating of micro injection molding machine is 120mm³ which is much smaller than the conventional one. It is found that small capacity injection molding machine can be reduced the variation in weight of green compacts as compared to conventional one.

2.3 Filling behavior and flow simulation

2.3.1 Micro gear

In recent years, some advanced micro-manufacturing processes and the micro-sized gears made of metals and some advanced ceramics were demonstrated (Löhe and Haußelt, 2005). The micro-planetary gear motors made of Ni-Fe and Ni-based bulk metallic glasses were developed by X-ray lithography, electro-deposition and injection molding method (Ishida et al., 1995). However, micro-sized gears made of general-purpose durable materials are demanded for miniaturization and reliability improvement of products, and also the manufacturing is aiming to achieve a high economical efficiency for industrial needs. Authors have studied the tribological properties of micro-gear manufactured by MIM process and were evaluated quantitatively, thus the wear mechanisms were clarified (Kameo et al., 2006) and the accuracy of the ultra-compact planet gear was also evaluated by measuring the variation in dimensions of the gear teeth with digital image analysis (Nishiyabu et al., 2008). Fig.8 shows the figures of the micro-planetary gear composed of three types of gearwheels manufactured by μ -MIM process and the dimension of the planet gear (module: $m=0.07\text{mm}$, number of teeth: $z=24$). The materials used for producing the ultra-compact gears are stainless steel 17-4PH water-atomized powder ($D_{50}=2\mu\text{m}$) and oxymethylene-based binders. The volume fraction of powder in the feedstock is 60%. The feedstock was injection-molded using high-speed injection molding machine (FANUC Ltd., S-2000i 50A). The green compacts were debound at 600°C for 2hrs in N₂ gas, and sintered at 1150°C for 2hrs in Ar gas. Also the sintered parts were age-hardened at 480°C for 1hrs. From

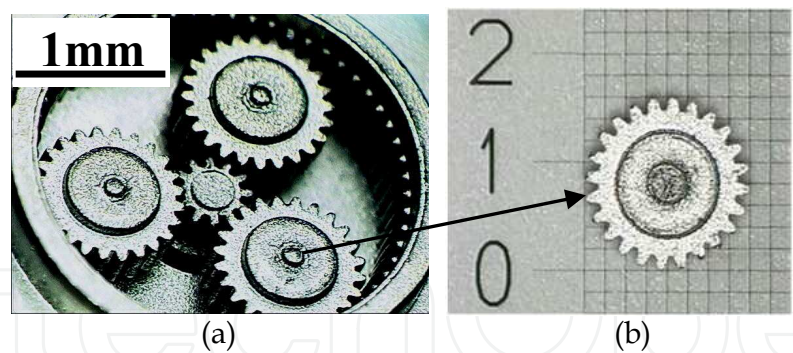


Fig. 8. Micro-planetary-gear manufactured by MIM process; (a) Planetary-gear set; (b) Planet gear.

the evaluation results, it was clarified that the accuracy class of the ultra-compact planet gear have not come up to that of ground precise gear which is equivalent to around five. However it is reviewed that the accuracy class of ultra-compact gear is around seven which is an acceptable level for general-purpose applications from a practical point of view.

Computational fluid dynamics (CFD) analysis can provide valuable information to mold designers and manufacturers (Nishiyabu et al., 2008). Flow simulation of micro-planetary-gear manufactured by MIM process was done using Moldex™ software. The finite element model shown in Fig.9 is used. In total, the cavity meshes and mold base meshes included approximately 2.3million elements. Whilst the material for the analysis has the material properties of MIM feedstock, it is not currently possible to accurately model all of the complex flow characteristics of MIM feedstock, such as layer slip, compressibility and jetting. The material in the analysis has similar flow characteristics to a heavy plastic. Most commercial injection-molding CFD programs cannot accurately create micro-parts model using standard machine settings as they have unsuitable values for shot weights and flow rates etc. From the flow simulation results shown in Fig.10, it is clarified that the filling time is approximately 1.8ms for spur-runner part but only 0.2ms for cavity one. Also the filling of gear teeth is at the end of flow, and is hard to apply high pressure into fine wall of teeth. Extra care must be taken with runner shapes and gate locations in micro-analysis due to the exceptionally small and thin cavity shapes.

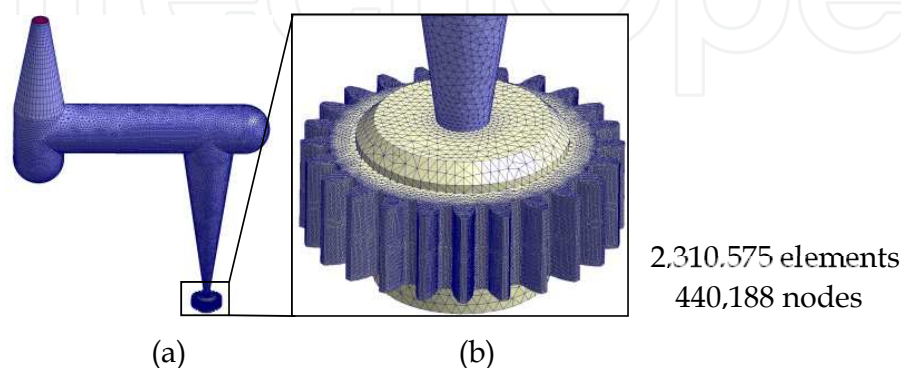


Fig. 9. Micro-planetary-gear manufactured by MIM process; (a) Runner and spur part; (b) Micro-gear part.

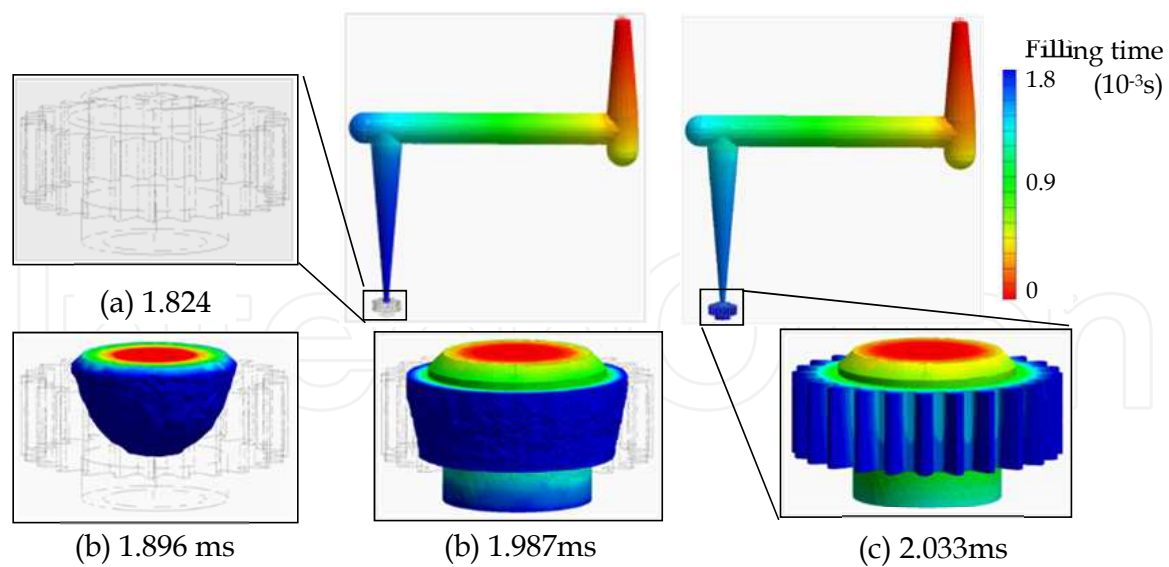


Fig. 10. Filling state of micro-planetary-gear with spur-runner part.

2.3.2 Micro-dumbbell tensile specimen

As the size of MIM parts decrease, the test specimen is also necessary to downsize appropriately for actual evaluation. Authors (Nishiyabu et al., 2005) used micro-dumbbell tensile specimen with 0.1mm wide in narrowest portion as shown in Fig.11. The reason why the dumbbell shape with large volumes of clamps at the ends was attached is to investigate the performance of filling and the change of internal pressure when it was molded with an in-line screw type injection molding machine. The jigs for clamping are also manufactured by MIM, and the specimen can be observed by scanning electron microscope (SEM). The motion of deformation and damages occurred on the surface is recorded.

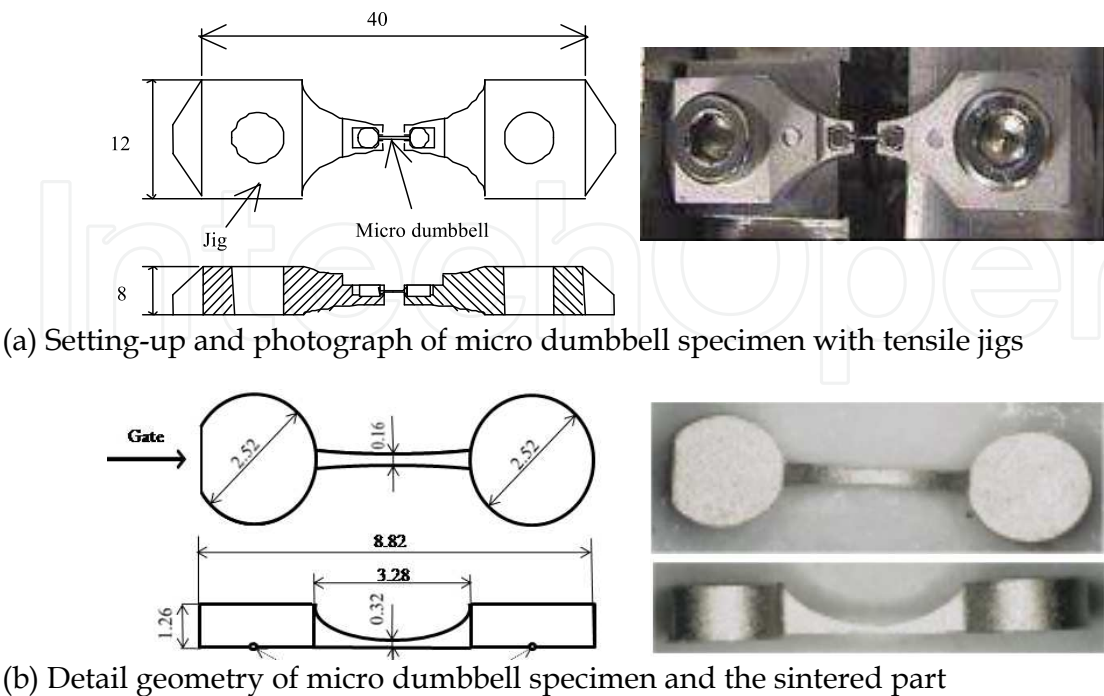


Fig. 11. Geometry of micro dumbbell specimens.

The conditions of injection molding for micro dumbbell specimens were examined with various injection pressures and speeds, but the other conditions such as holding and back pressure, injection and holding time, metered value, molten material temperature and mold temperature were constantly-applied. The results of the experiments using $D_{50}=10\mu\text{m}$ and $D_{50}=2\mu\text{m}$ 316L stainless powder feedstock in constancy of metal powder fraction of 50vol.% are shown in Fig.12(a) and (b), respectively. These diagrams show the filling behaviour. In case of $10\mu\text{m}$ powder feedstock, the maximum injection pressure was limited at 150MPa because of the high melt viscosity, while no limitation existed for $2\mu\text{m}$ powder feedstock. In case of $2\mu\text{m}$ powder feedstock, the short shot phenomenon was observed in the wide range of injection pressure and speed. In either case, the short shot was likely to occur at lower injection speed, while flash is significant at higher injection speed, because the melt viscosity of feedstock depends on shearing rate as shown in Fig.5. With this preliminary examination, it is concluded that the suitable injection pressure and speed are 20-70MPa and 300-400mm/s for $10\mu\text{m}$ powder feedstock while 70MPa and 400mm/s for $2\mu\text{m}$ one.

The finite element model of micro-dumbbell with spur-runner parts as shown in Fig.13 was used for flow simulation. The cavity pressure profiles obtained by numerical analysis and

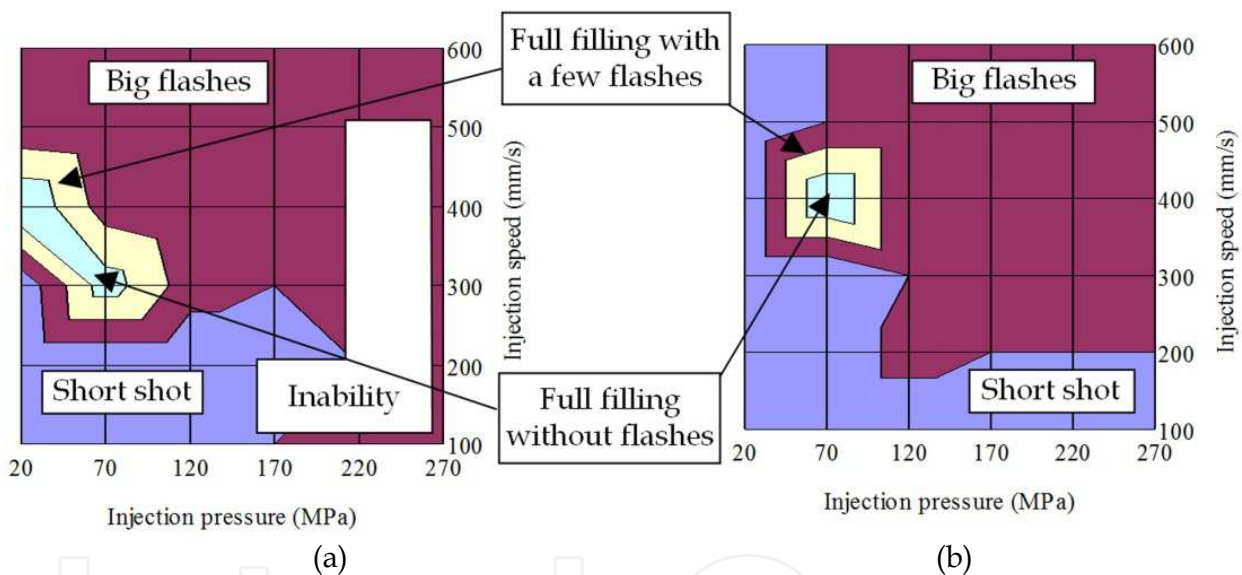


Fig. 12. Dependence of injection molding conditions on filling behaviour in micro dumbbell specimens molded using varied particle size feedstock; (a) $D_{50}=10\mu\text{m}$ powder feedstock; (b) $D_{50}=2\mu\text{m}$ powder feedstock.

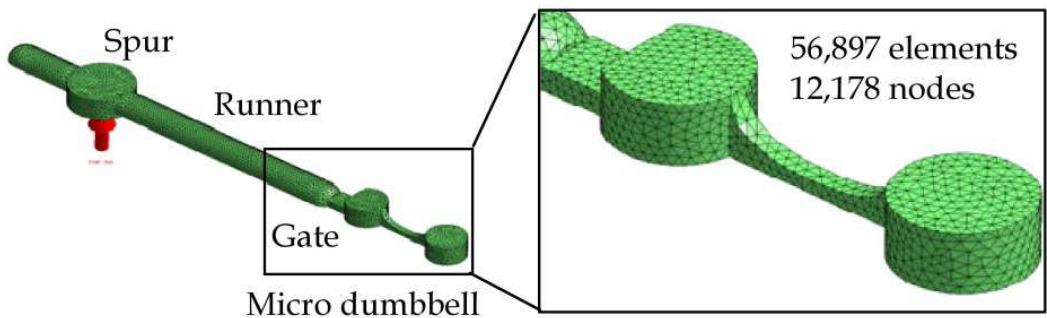


Fig. 13. FE model for micro-dumbbell with spur-runner parts.

experiment are shown in Fig.14. The far gate side is applied much lower pressure than the near gate. The results agree qualitatively, however it has large differences quantitatively between the simulation and the experiment.

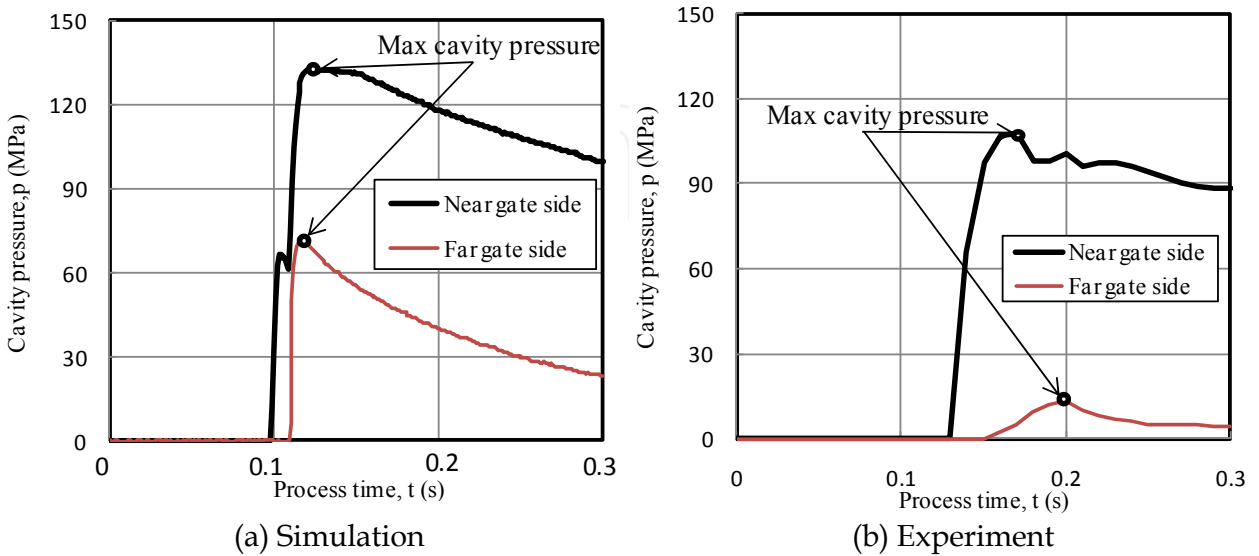


Fig. 14. Cavity pressure versus process time.

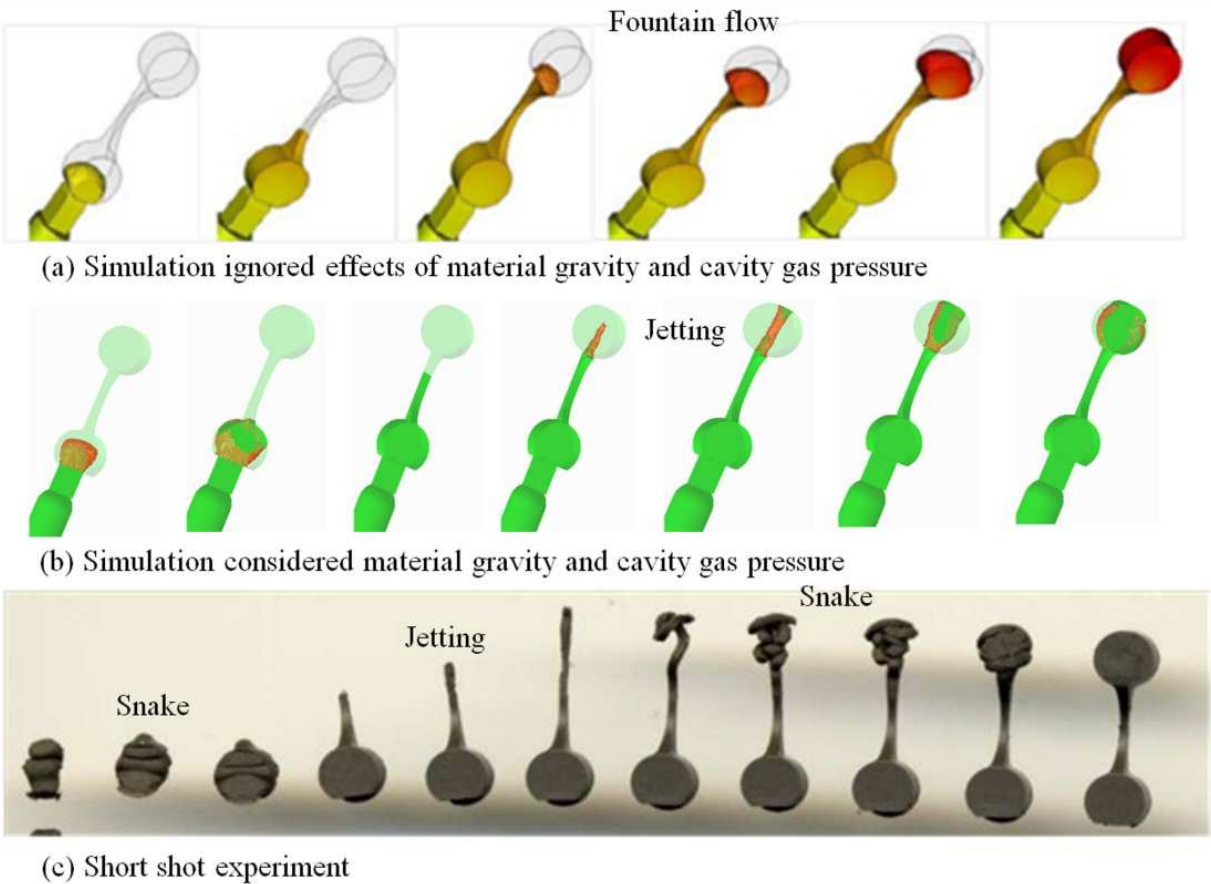


Fig. 15. Comparison of filling between simulation and short shot results.

The result in Fig.15 shows that one of the reasons to be considered is difference in filling between simulation and short shot. When the flow simulation considered the effects of material gravity and cavity gas pressure (Fig.5(b)), the results could be simulated as a jetting phenomenon and it could not be identified the unstable filling state such as snake phenomenon (Fig.5(c)). Thus it is considered that the pressure is resulted to reduce significantly in the experiment.

3. Metal powder injection molding for micro pillar-structured parts

3.1 Concept of sacrificial plastic mold insert MIM (SPiMIM) process

Fig.16 shows the flow of sacrificial plastic mold insert MIM (SPiMIM) process which is basically divided into three steps; 1) manufacturing of SP-mold, 2) injection molding of MIM feedstock into SP-mold insert, and de-molding the green compact and SP-mold insert part as one component, handling to the debinding-sintering process, and finally 3) debinding to eliminate the SP-mold and polymeric binder followed by sintering process. Therefore, the μ -SPiMIM process has great potentials to improve the filling, de-molding and handling, and to produce the tiny parts with 3 dimensional complex shapes and fine structures. The SP-molds used in this process can be manufactured by several types of methods such as injection molding, machining, rapid-prototyping, hot-embossing and lithography and so on (Nishiyabu et al., 2007).

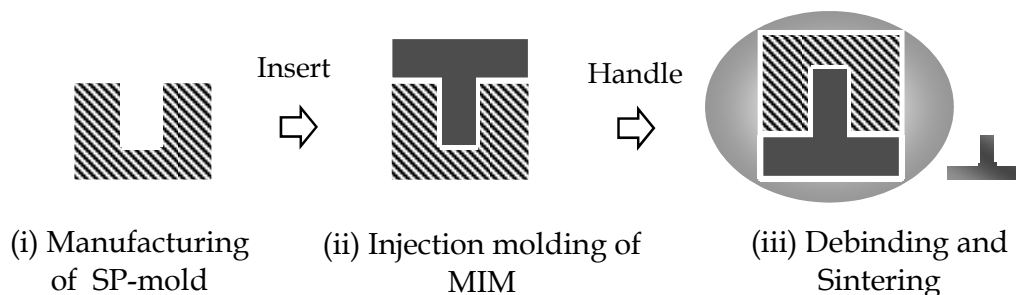


Fig. 16. Flow of micro sacrificial plastic mold insert MIM (SPiMIM) process.

Fig.17 shows some examples of SPiMIM products such as an optical fibre connector, a zigzag spring and micro impeller. These shapes cannot be manufactured easily because they have hollow, under-cuts and external screw and very narrow portions. The core and mold insert are made of polymethylmethacrylate (PMMA) polymer by conventional plastic injection molding for optical fibre connector and a zigzag spring, while micro rapid prototyping is used to make the closed mold to manufacture a micro-impeller.

3.2 Differences in filling behavior of metallic mold vs. plastic mold

The differences of mold materials on the filling behaviour were investigated by flow analysis using commercial plastic injection molding simulation software (Moldflow™, MPI ver.4.0). The main difference in property of mold materials is thermal conductivity, which of steels (46.2W/mK) is 230 times higher than plastics (0.2W/mK). From the analytical results in zigzag spring specimen as shown in Fig.18, it was confirmed that filling of feedstock could be accomplished with lower injection pressure by using plastic mold. This is

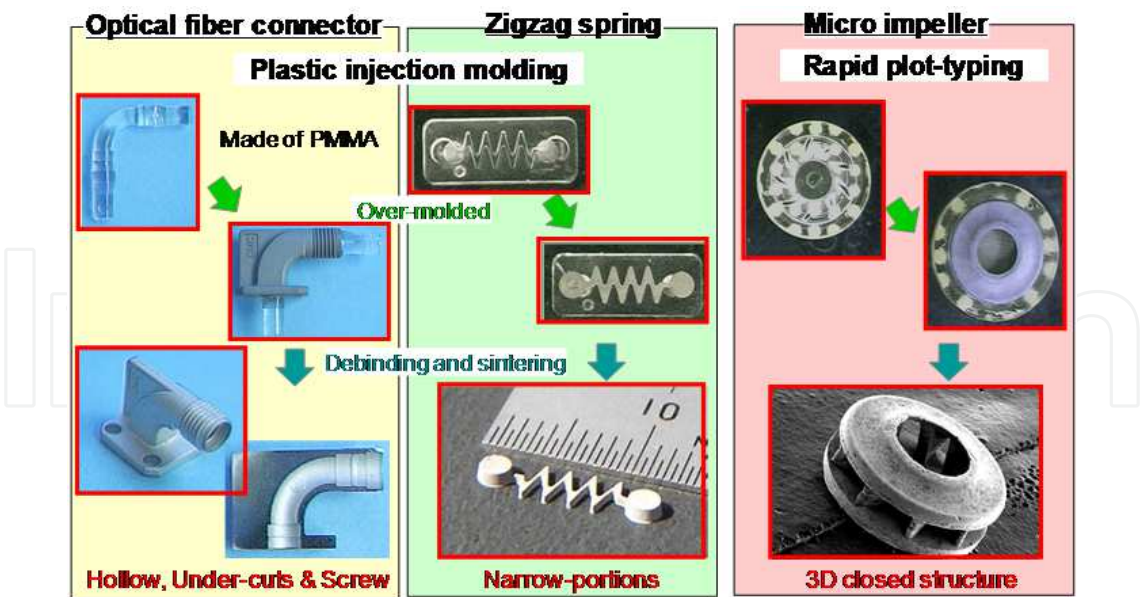


Fig. 17. Examples of metal parts manufactured by SPiMIM process.

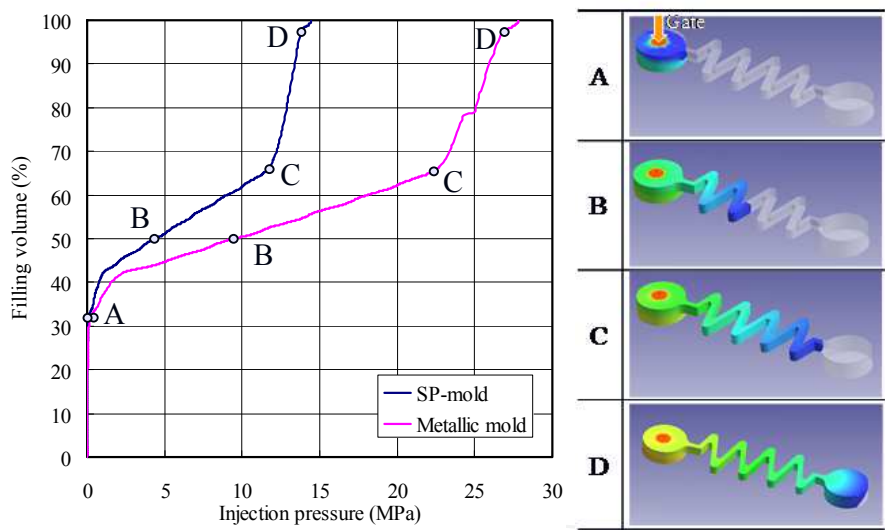


Fig. 18. Effects of mold material on filling behaviour in zigzag spring specimen.

considered because the plastic mold cools the feedstock much slower than metallic one. This is due to the low thermal conductivity of plastic and heat generated by friction between viscous flowing feedstock and plastic mold. In consequence of high viscosity of the feedstock, there is no rapid increase in plastic mold. Therefore, it can be expected to easily fill the viscous feedstock in narrow cavity of micro parts. In particular, with injection molding of micro pillar-structured parts, it is effective to use plastic mold.

3.3 Effectiveness of plastic in mold fabrication for μ-MIM

The products shown in section 3.1 cannot easily be manufactured by conventional MIM process, but they are not so tiny parts. Therefore, the SPiMIM method was applied into μ-MIM process by using finer tooling production method as below. Fig.19 shows the situation of MIM process overwritten in a road map on micro-nano processing developed by Japan

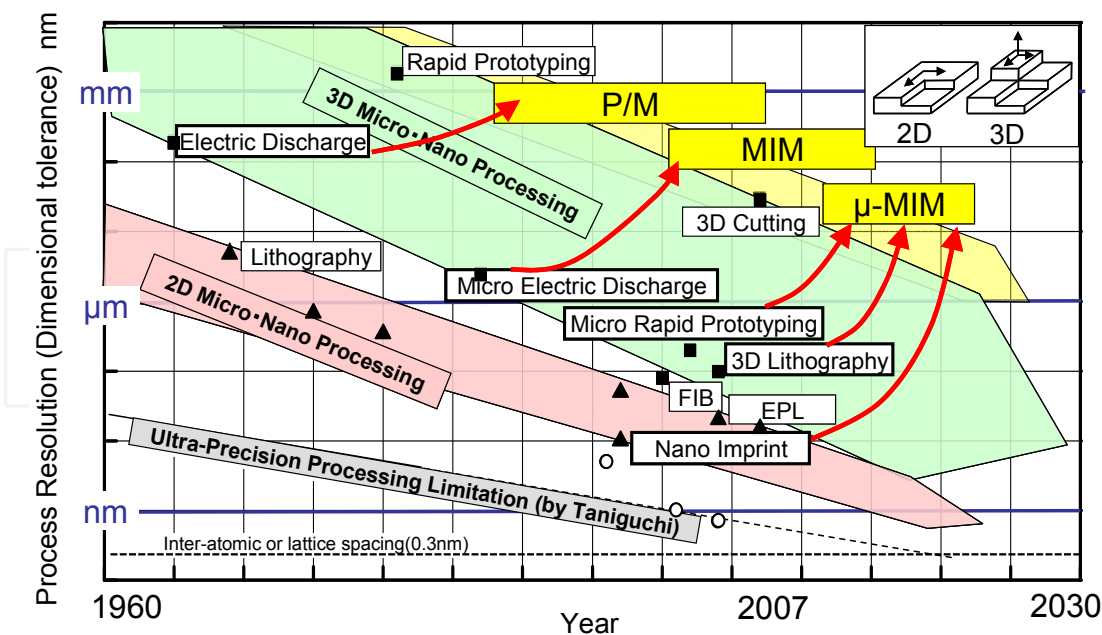


Fig. 19. Situation of MIM process in road map on micro-nano processing.

Society of Mechanical Engineers (JSME), which was originally surveyed by Prof. Hata. It is obvious from this road map that the process resolution is becoming progressively more accurate from micro-scale to nano-scale with the passing of the year by two or three dimensional micro-nano processing such as several lithography techniques, nano-imprint, micro electric discharge and micro-rapid prototyping and so on. By using these three dimensional micro-nano processing's to make the molds, the dimensional tolerance of P/M and MIM is becoming higher and it is even more so high for μ -MIM. However, most of these ultra-precision processing's is capable of applying for polymers, not for metals. Therefore, it is suggested that the use of plastic mold in MIM process is effective for the manufacturing of fine mold in addition to providing a high performance in filling and easy handling.

3.4 Combination of LIGA and MIM process

In producing of the higher precision parts, it is necessary to use the molds that are more precise than conventional molds manufactured by cutting and electric discharging. LIGA ((Lithographie , Galvanoformung , Abformung) is the abbreviation of German words for lithography, electroforming and molding, which is one of the micro-processing techniques applied for semiconductor production such as Micro-Electro-Mechanical-System (MEMS) or Micro System Technology (MST). LIGA is mainly consisted of 3 processes; the first is irradiation process to transcript the mask shape to resin, namely resist, the second is developing process to remove unnecessary part of the resist, and to make negative structure of the desired body, the third is molding process by electro-forming metal structure in the resist. The main features of LIGA process are to create the fine profiles with dimensional accuracy in nanometre order, and micro-structures with high aspect ratio. However, the materials applicable to LIGA are limited, and the process is not suitable for mass-production because of its high cost. The manufacturing method of micro sacrificial plastic mold insert metal injection molding (μ -SPiMIM) combined LIGA process, namely LIGA/ μ -SPiMIM process has been proposed to solve specific problems involving the miniaturization of MIM. As shown in Fig.20, the μ -SPiMIM method inserted ultra-fine molds which were fabricated

by LIGA process, namely LIGA/ μ -SPiMIM process, was proposed (Nishiyabu et al, 2007). Two types of SP-molds with fine structures such as 1) PMMA resist and 2) PMMA mold injected into Ni-electroform which is a typical LIGA process, are named as “One-step transcription method” and “Two-steps transcription method”, respectively. In one-step transcription method, PMMA resist is used for SP-mold. Therefore, this is very good for transcription, because original shaped resist is used as SP-mold, and out-gassing is very easy in injection molding. However the cost for producing PMMA resist is extremely high. In two-steps transcription method, Ni-electroform is used only for injection molding of SP-mold, it is preferable for mass-production with cost efficient, but the quality reduction of transcription due to the twice injection molding operations is a main issue.

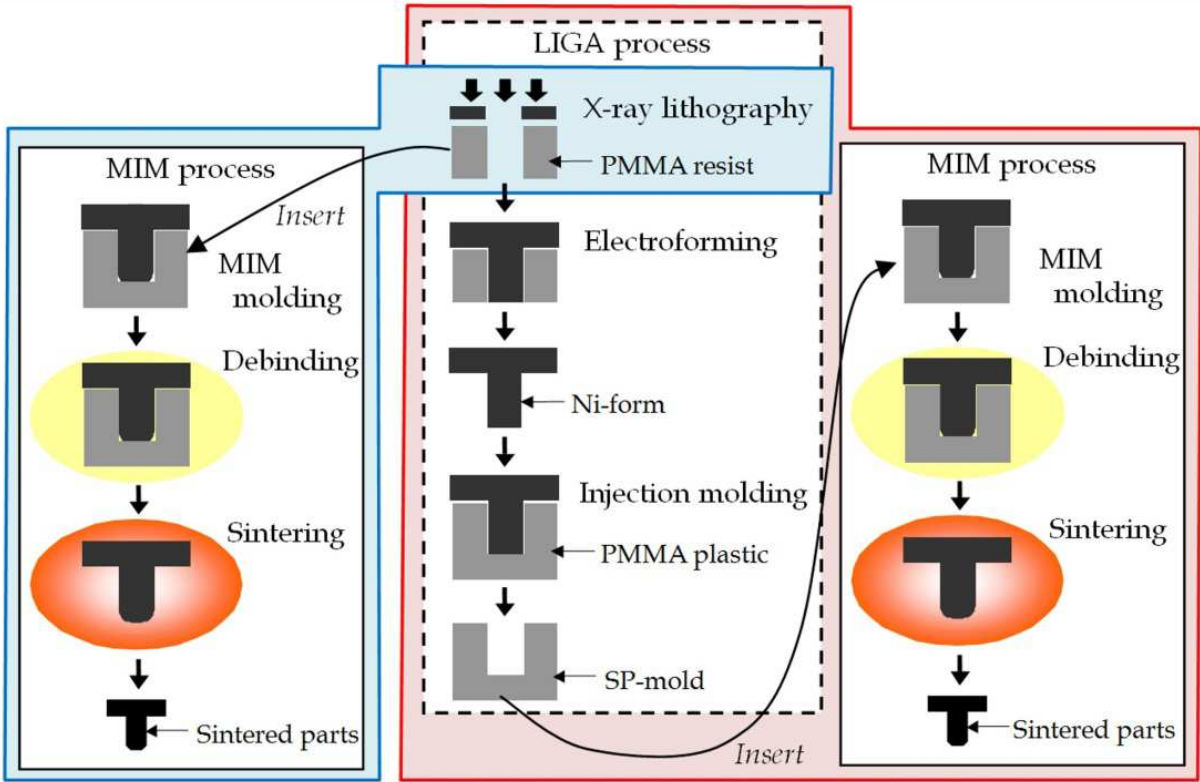


Fig. 20. Flow of two types of transcription methods in LIGA based μ -SPiMIM Processes.

3.5 Manufacturing by resist/ μ -SPiMIM process

To demonstrate the possibility of Resist/ μ -SPiMIM process, authors tried to manufacture multi-pillar structures assuming a micro-fluidic device, such as micro-reactor and micro-mixer as shown in Fig.21 (Nishiyabu et al, 2007). The resist film shown in Fig.21(a) was used as SP-mold. The dimension of a cylinder is 200 μ m in height and 50 μ m in diameter. The sintered part with 30-40% shrunk dimension after sintering was obtained as shown in Fig.21(b). It is also shown in Fig.21(c) that micro fluidic chip made of stainless steel 316L can be fabricated with relative ease by Resist/ μ -SPiMIM process. Though the aspect ratios of these structures are not so high, it is very difficult to mold such a fine structure with conventional injection molding using metallic mold. Due to the fragility of green compact and fine structures with high aspect ratio, the de-molding is extremely difficult, because the surface area of multi-pillar structures increase exponentially with increasing in number of

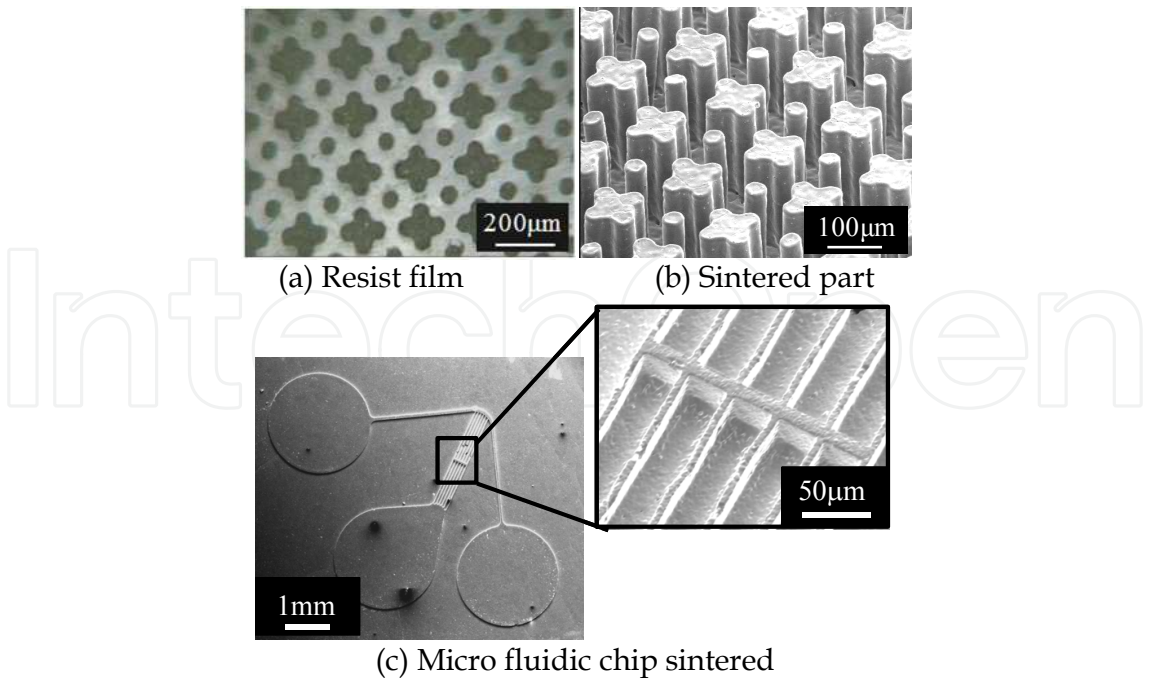


Fig. 21. Resist film and sintered parts manufactured by Resist/ μ -SPiMIM process.

	(a) Ni-form	(b) Sintered parts: $D_{50}=3\mu\text{m}$ powder	(c) Sintered parts: $D_{50}=9\mu\text{m}$ powder
(i) Square column			
(ii) Cylindrical column			
	Arithmetic average surface roughness, R_a (μm)		
	0.17	0.43	1.40

Fig. 22. SEM images, profiles and roughness of Ni-forms and sintered parts manufactured by Resist/ μ -SPiMIM process.

pillars. However, the de-molding is not required in μ -SPiMIM process, therefore this process possesses great advantages in producing the tiny parts with micro-structures. Fig.22 shows SEM images, profiles and roughness of Ni-forms and sintered parts manufactured by Resist/ μ -SPiMIM process using varied particle diameter of stainless steel 316 powder. The shape accuracy and surface roughness of sintered parts manufactured using finer powder is considerably improved compared to coarse ones and it is close to Ni-forms. It is also obvious to be able to manufacture metallic parts smaller than SP-mold.

3.6 Defects in molding in LIGA/ μ -SPiMIM process

LIGA/ μ -SPiMIM process requires two steps transcription, which is 1) plastic injection molding for SP-mold, and 2) metal powder injection molding for green compact. Thus, it is very important for obtaining accurate green compact to manufacture precisely SP-mold. However, the defects such as the development of weld line and short filling often occurred around pillars of molded parts in plastic injection molding. In order to reduce those defects to the scale of having no influences after sintering, the molding conditions for manufacturing the SP-mold should be optimized and also the effects of metal particle size and processing conditions on the shrinkage, transcription and surface roughness of sintered parts should be investigated. The specimen for SP-mold with numerous micro-pillars is shown in Fig.23. The size of square column is 150 μ m on a side and 200 μ m in height. In case of improper molding conditions, defects such as weld line and short filling caused by injection molding of SP-mold as shown in Fig.24(a). As the result, the sintered parts with unwanted shape such as convex line and rounded edge as shown in Fig.24(b) were obtained.

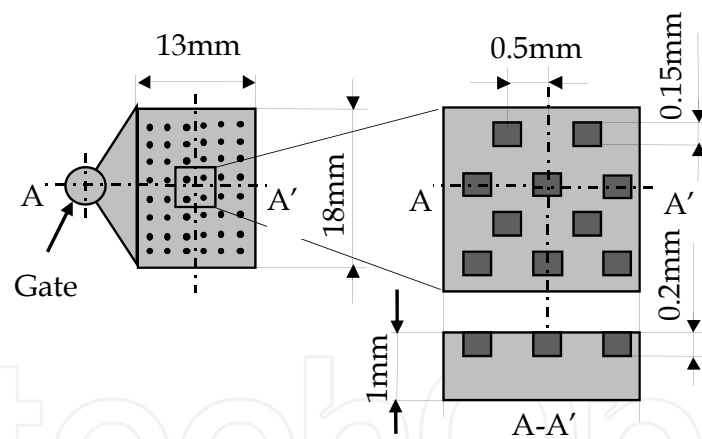


Fig. 23. Geometry of SP-mold with numerous micro-pillars.

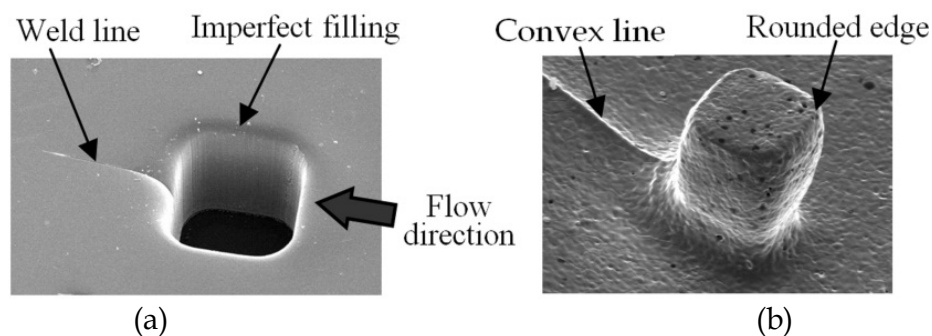


Fig. 24. Defects caused by injection molding of SP-mold; (a) SP-mold; (b) Sintered part.

The effects of injection molding condition on defects were investigated. Fig.25 shows the depth of weld line and width of short filling developed in SP-molds fabricated under various injection speeds and mold temperatures, but the material temperature and holding pressure were held constant. It is obvious from these graphs that the both sizes of weld line and short filling decreased as injection speed and mold temperature increased. As the results of the manufacturing of SP-molds under the maximum injection speed and highest mold temperature, the size of the defects was reduced as shown in Fig.26. The size of weld

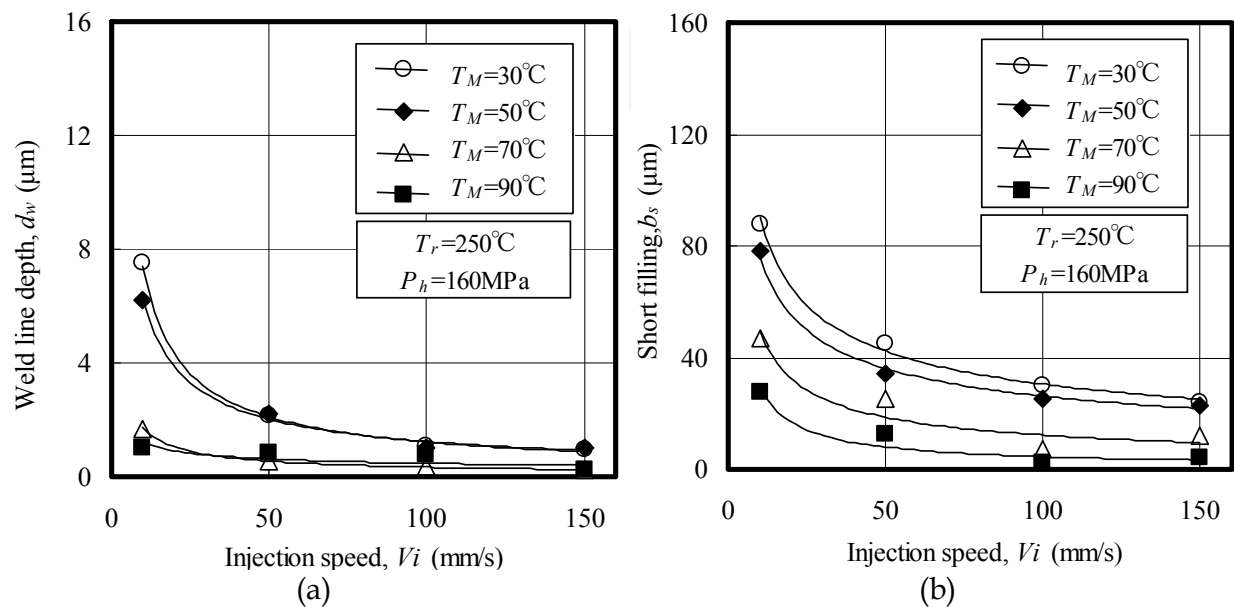


Fig. 25. Effects of injection speed and mold temperature on molding quality of SP-mold; (a) Depth of weld line; (b) Width of short filling.

	(a) Origin condition	(b) Optimized condition
Molding condition	$v_i=10\text{mm/s}$, $T_M=60^\circ\text{C}$ $T_r=195^\circ\text{C}$, $P_h=30\text{MPa}$	$v_i=150\text{mm/s}$, $T_M=90^\circ\text{C}$ $T_r=250^\circ\text{C}$, $P_h=160\text{MPa}$
SEM image		
A-A' Profile		
Size of weld line	4 μm deep, 90 μm wide	0.4 μm deep, 9 μm wide

Fig. 26. SEM images and profiles of SP-molds prepared before and after optimization of molding condition.

line is 0.4 μ m deep and 9 μ m wide, which the size is equivalent to disappear after sintering. Micro-pillar structured parts were manufactured by LIGA/ μ -SPiMIM process as shown in Fig.27. The SP-mold (Fig.27(b)) was prepared by injection molding PMMA polymer into Ni-form (Fig.27(a)). Although the defects that occurred during the injection molding such as weld line and rounded edge, SP-mold can be manufactured with low cost and high cycle time. MIM feedstock prepared varied particle diameter of stainless steel 316L powder were injection-molded in the SP-mold, the sintered parts (Fig.27(c)(d)) were obtained after debinding and sintering. As similar to sintered parts manufactured by Resist/ μ -SPiMIM process, micro-parts with much higher quality in shape and surface could be obtained by using fine powder better than coarse one.

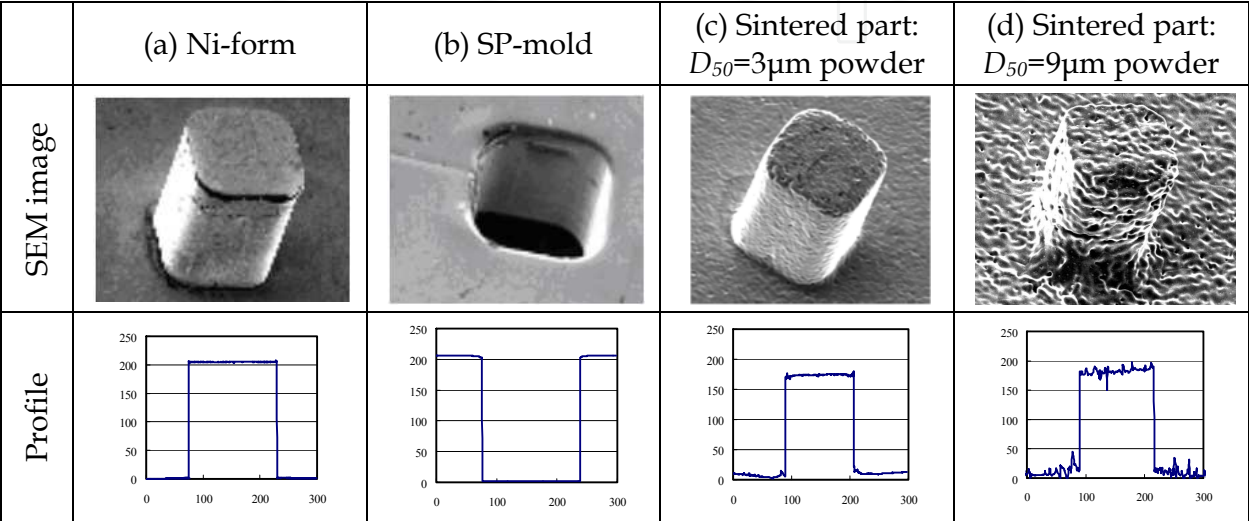


Fig. 27. SEM images and profiles of Ni-form, SP-mold and sintered parts manufactured by LIGA/ μ -SPiMIM process.

4. Use of nano-scale powder in micro sacrificial plastic mold insert MIM

4.1 Material properties of MIM feedstock using nano-scale powder

Further improvements on the quality of μ -MIM products are required in practical productions. In general the use of finer metal powders is one of solutions to improve the dimensional accuracy and surface roughness of μ -MIM products. It can be also expected to inhibit the grain growth by reducing the sintering temperature. On the other hand, sintering inhabitation by higher oxidation, lower packed density, higher viscosity and higher cost are some of drawbacks in MIM production. MIM feedstock is prepared using various particle sized metal powders from micro-scale to nano-scale and the effects of particle size on material properties of MIM feedstock were investigated.

The metal powders used for the experiments are five types of pure Cu powders as shown in Fig.28. The reasons for pure Cu powder is used as the research material are that Cu has a high thermal conductivity, thus it is expected to be used for microscopic structures with high-specific surface area but it is not easy to mass-produce the metallic parts with such a structure, besides Cu can be reduced easily by H₂ gas because of the high Gibb's energy. The micro-sized Cu powder is a conventional material manufactured commercially by a water-atomization method (d_{50} =8.2 μ m, d_{50} =20 μ m) or wet-electrolytic method (d_{50} =1.5 μ m,

$d_{50}=0.30\mu\text{m}$). On the other hand, the nano-sized Cu powder is an ultra-fine material produced by a radio-frequency thermal plasma method ($d_{BET}=0.13\mu\text{m}$). The primary particles are completely different in size and shape from micro-sized Cu powder. As nano-sized powder has a large specific surface, melt viscosity of the feedstock increases more significantly. Therefore it is a key technology for deriving the effectiveness of nano-scale powders to select the component of binder and its fraction of MIM feedstock. Multi-component binder composed of polyacetal polymer and paraffin wax is used. The binder content is predicted by referring 35vol.% of the practical optimum binder content for Cu powder ($d_{50}=8.2\mu\text{m}$) which are using in conventional MIM production. Tap densities of various sized Cu powders are shown in Fig.29(a). Tap density of Cu powder increases exponentially as the particle size of Cu powder increases. As for the binder content, it decreases when the particle size increase as shown in Fig.29(a). As the results, a finer

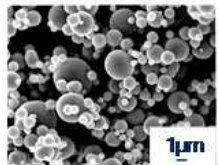
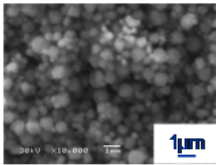
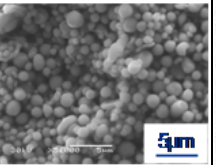
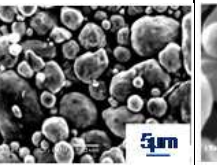
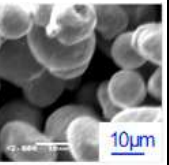
	(a) $d_{BET}=0.13\mu\text{m}$	(b) $d_{50}=0.3\mu\text{m}$	(c) $d_{50}=1.5\mu\text{m}$	(d) $d_{50}=8.2\mu\text{m}$	(e) $d_{50}=20\mu\text{m}$
Production method	Radio frequency plasma method	Wet reduction method	Water atomization method		
SEM image					
Tap density	0.66g/cm ³	2.10 g/cm ³	3.50 g/cm ³	4.80 g/cm ³	4.81 g/cm ³
Specific surface	5.1 cm ² /g	1.6 cm ² /g	0.65 cm ² /g	0.101 cm ² /g	0.125 cm ² /g

Fig. 28. Properties of Cu powders with various particle sizes.

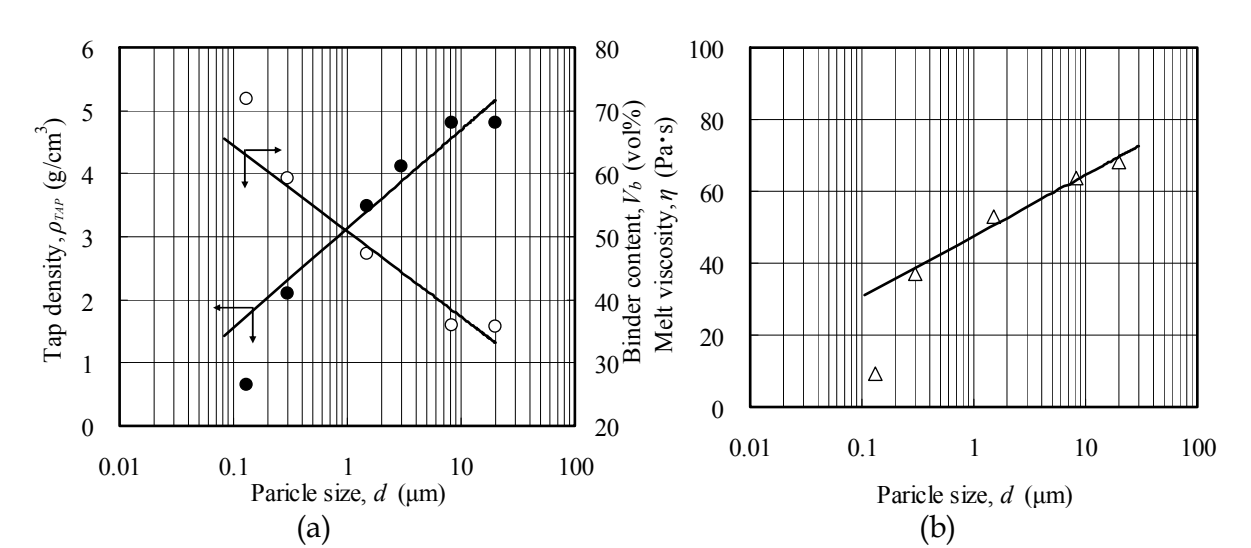


Fig. 29. Properties of MIM feedstock with various particle sizes of metal powder; (a)Tap density and binder content; (b) Melt viscosity.

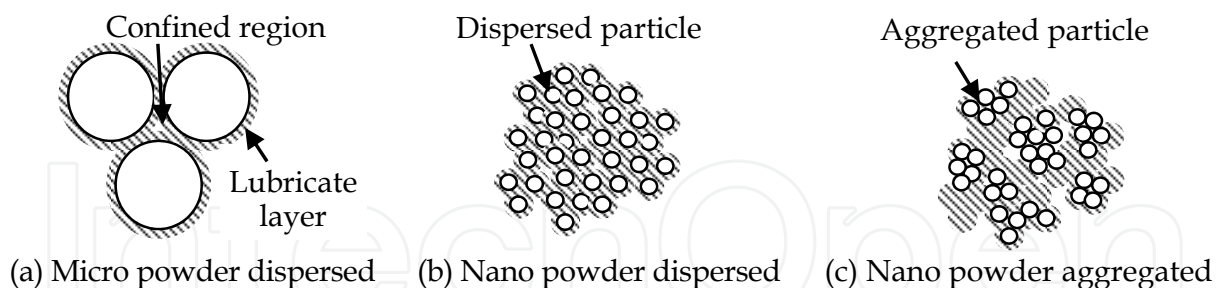


Fig. 30. Configuration interaction of binder in MIM feedstock with varied particle.

powder has a larger specific surface, the melt viscosity of the feedstock increases more significantly as shown in Fig.29(b). When the binder content is predicted from the equation based on the space rate estimated from tap density, and the calculation of the binder content which the spaces are filled up, were made sometimes it shows that the binder content is too high and the melt viscosity becomes much lower. This reason is assumed to be due to remarkable agglomeration of nano-sized particles as shown in Fig.30. Thus the melt viscosity of feedstock was tried to keep constant by changing binder content using the equation based on the space rate estimated from tap density.

4.2 Molding machine for micro sacrificial plastic mold insert MIM

It is often desirable to conduct a material parametric study using a very small amount of nano-sized powder. The use of relatively large amount of feedstock is required for a conventional injection molding machine. Therefore, a direct mixing-injection molding machine as illustrated in Fig.31 has been developed. This machine is small enough to be placed on top of a table, and it enables the mixing of metal powder and binders followed by injection molding, therefore it can achieve molding without pelletizing. The capacity of the furnace is 0.05cm³ in volume which is equivalent to the general size of a single feedstock pellet. A mixing is completed by a rotation of the plunger with 3mm in diameter. The mixing condition is basically controlled by furnace temperature, rotation speed and mixing time. The procedure of injection molding on this machine which is metal powder and binders are previously metered with balance and homogeneously mixed into the furnace. The feedstock is fully injected into the cavity at high speed when the plunger is pressed by compressed air with 0.1-0.85MPa. Therefore, this operation can achieve an accurate control on the volume of feedstock for each shot. In case of μ -SPiMIM process, resist film and NIL film is inserted into the mold cavity as shown in Fig.31(d) and (e), respectively. The green compact and SP-mold are ejected as one component.

4.3 Effects of particle size in resist sacrificial plastic mold insert MIM

In Resist/ μ -SPiMIM process, the resist film made of PMMA polymer with numerous micro-holes was used as SP-mold. The feedstock was prepared using Cu powders with various particle sizes and the green compacts could be prepared with a high efficiency in experiment using a small amount of feedstock by a small molding machine introduced in previous section. The sintered parts with micro-pillar structure were obtained after

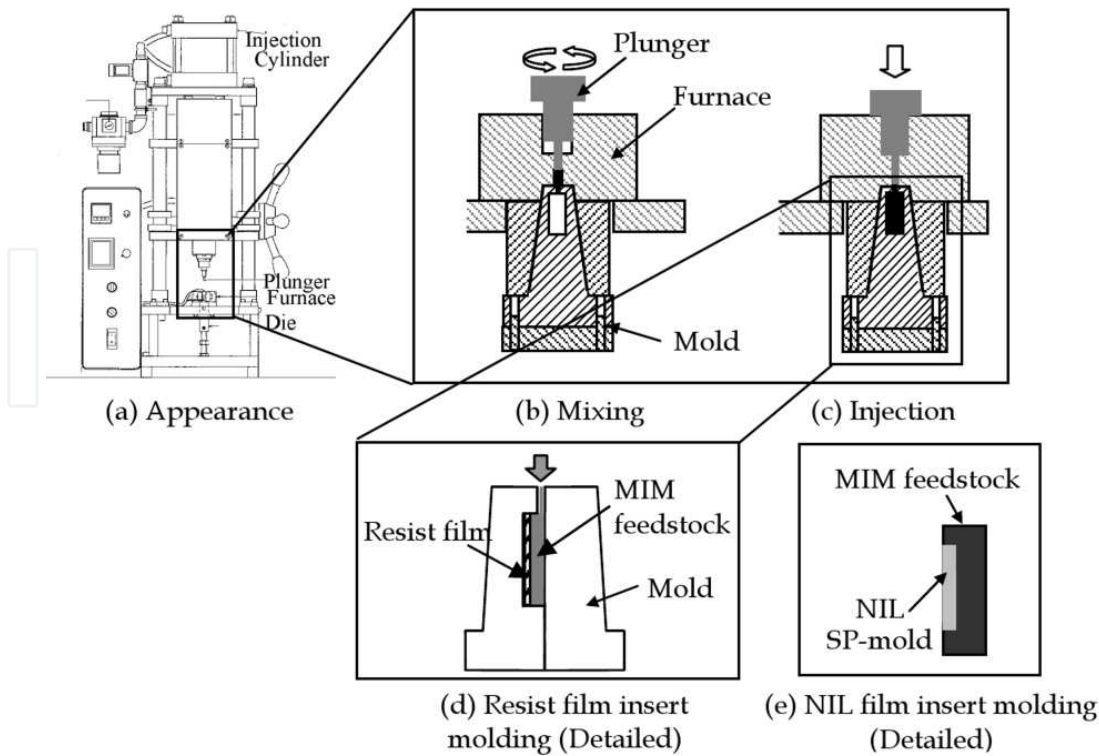


Fig. 31. Scheme of small direct mixing-injection molding machine.

debinding and sintering process. Fig.32 shows the SEM images of green compacts and sintered parts produced by various sized Cu powders. As the particle size of Cu powder used decreases, the filling of the green compacts in molding was improved except for $d_{50}=0.13\mu\text{m}$ powder specimen. The decreasing of particle size results in a marked improvements of surface roughness, transcription and dimensional variation of sintered parts. Fig.33 shows SEM images of green compact and sintered part manufactured by using $d_{50}=0.3\mu\text{m}$ Cu powder. For the entire body of green compact, the feedstock was filled fully in fine pillar structure, but a slight deformation is visible in sintered part. This is considered to be due to large amount of binder, and further study on the decision of binder content in particle agglomeration is needed for the quality improvement.

	(a) $d_{BET}=0.13\mu\text{m}$	(b) $d_{50}=0.3\mu\text{m}$	(c) $d_{50}=1.5\mu\text{m}$	(d) $d_{50}=8.2\mu\text{m}$	(e) $d_{50}=20\mu\text{m}$
Green compact					
Sintered part					

Fig. 32. SEM images of green compact and sintered part manufactured by using Cu powders with various particle sizes.

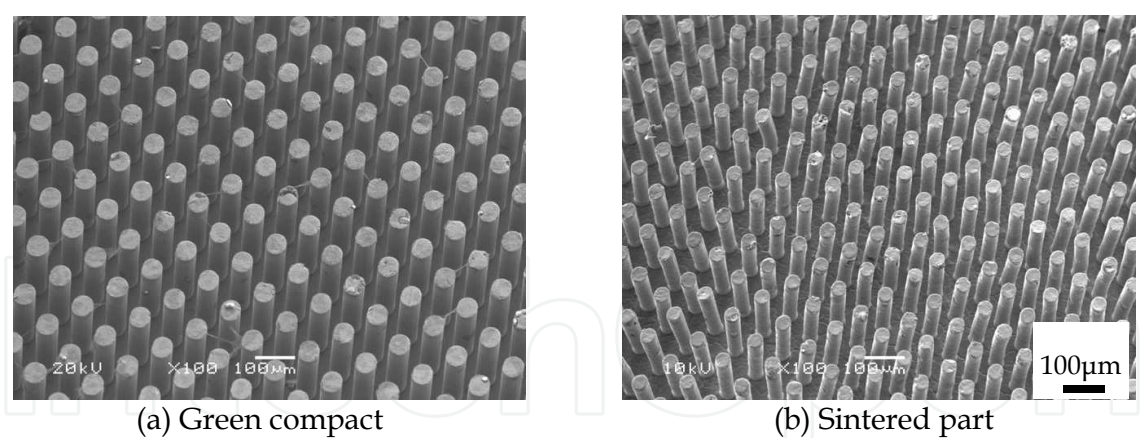


Fig. 33. SEM images of green compact and sintered part manufactured by using $d_{50}=0.3\mu\text{m}$ Cu powder.

4.4 Nano-imprint lithography (NIL) sacrificial plastic mold insert MIM

Fig.34 shows the flow of NIL/ μ -SPiMIM process. Thermal NIL technique is an application for the hot embossing technique but it can achieve higher resolution than conventional ones. The main features of NIL process are to create fine profiles with dimensional accuracy in nanometre order, and microstructures with several micrometers. However, the materials applicable to NIL process are limited only to polymers and glasses. Thus the combination process named as NIL/ μ -SPiMIM which was used NIL process for manufacturing SP-mold in μ -SPiMIM process was proposed. As shown in Fig.34(i), thermal NIL process mainly consists of three steps; 1) Heating to soften PMMA film more than the glass-transition

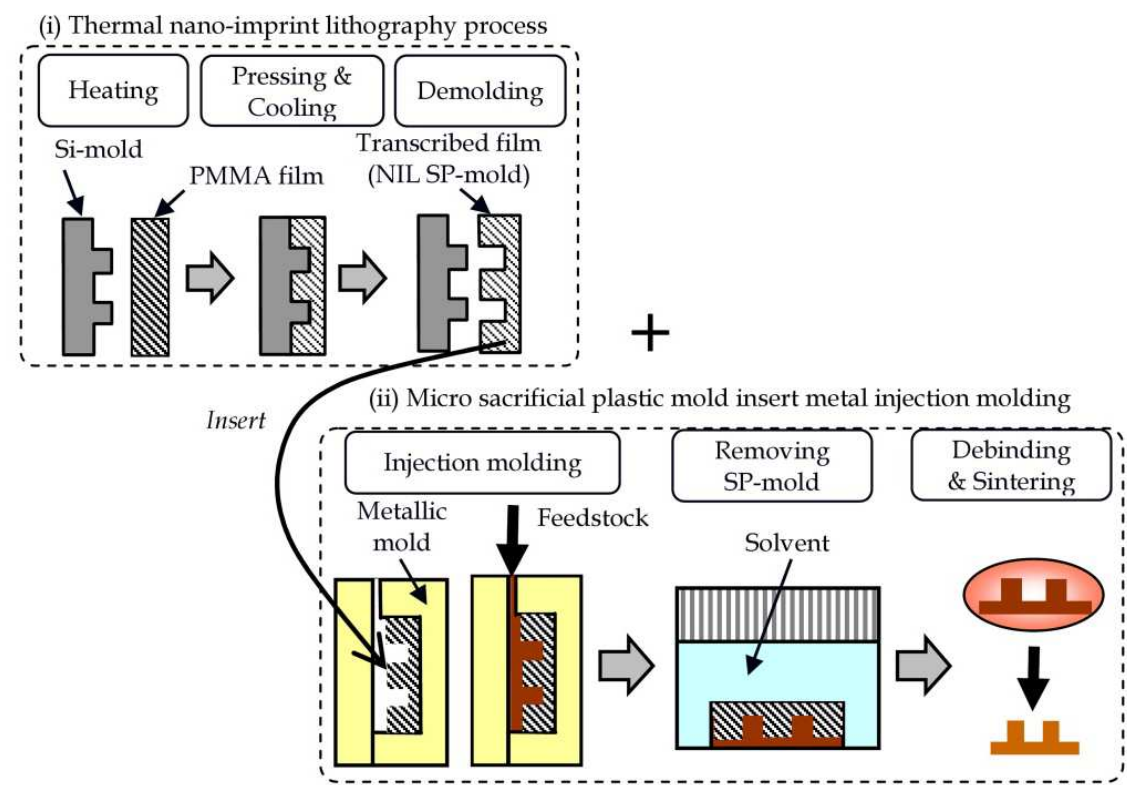
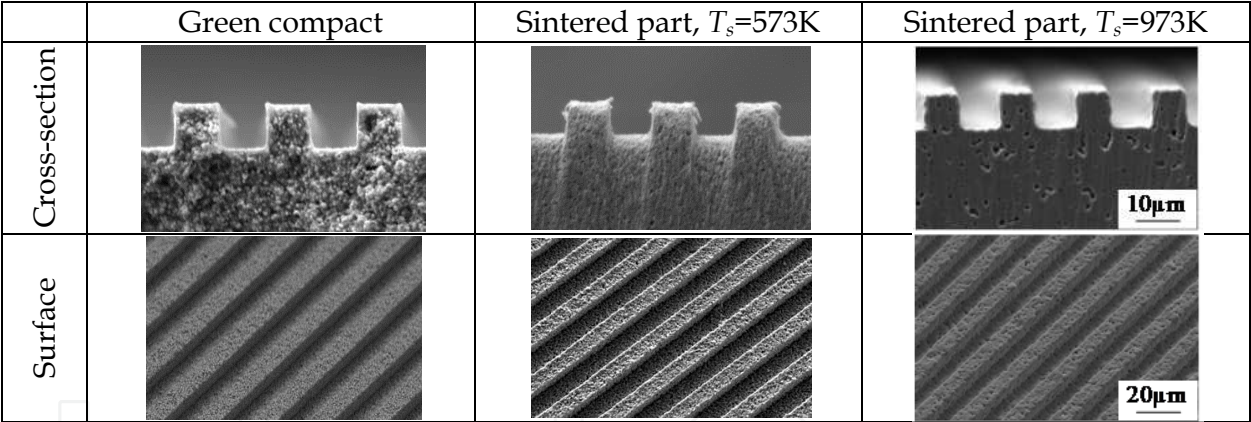


Fig. 34. Flow of NIL/ μ -SPiMIM process.

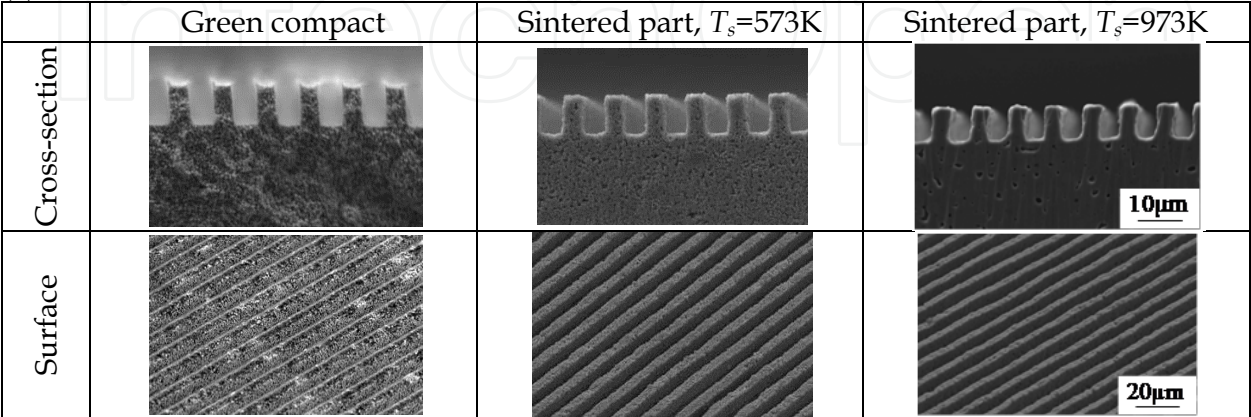
temperature, 2) Pressing and cooling to transcript Si-mold shape to PMMA film and 3) De-molding the film from Si-mold. Subsequently the μ -SPiMIM process is proceeded by three steps as shown in Fig.34(ii); 1) Injection molding of MIM feedstock into the SP-mold, and ejecting the green compacts and SP-mold as one component, 2) Removing the SP-mold and 3) Debinding polymeric binders followed by sintering.

The metal powder used for the experiments is nano-sized Cu powder which is manufactured commercially by a wet-reduction method (d_{50} =0.7 μ m, tap density: 3.17g/cm³, specific surface area: 1.69m²/g). The solid loading is 50vol.% and melt viscosity of the feedstock is attained to 43.7 Pa-s. The feedstock composed of nano-sized Cu powder and oxymethylene-based binder was adequately prepared and molded into NIL film made of PMMA polymer with fine line-scan structures (5 μ m or 10 μ m in width and 10 μ m in height), and the molded parts (with a single length of 4mm) were sintered in a reductive gas atmosphere followed by solvent debinding of the films. The debinding and sintering condition was optimized by investigating the effects of sintering temperature and atmosphere gas on density, shrinkage, composition and profile accuracy with thermo-gravimetric analysis, carbon and oxygen analysis and SEM observation.

Fig.35 shows the SEM images of green compacts, and sintered parts processed at 573K and 973K. In the green compacts with both L/S=5 μ m and 10 μ m, the feedstock has be seemingly filled into the micro channels completely, but the polymer binder builds up at the upper corners of microscopic structures. The sintered parts processed at 573K have many



(a) In L/S=10 μ m structure



(b) In L/S=5 μ m structure

Fig. 35. SEM images of green compacts and sintered parts.

sub-micron pores, because both debinding and oxidizing have not been completed at the low temperature. Then the microscopic structures are kept in accurate shape. In general the sintered parts processed at higher temperature shrink more, and the corner of microscopic structures becomes dull correspondingly. However the sintered parts processed at 973K keep the edge sharpness under optimized debinding-sintering conditions. The green compacts have slight concave portions on the top face of microscopic structure, but it was attained to fill the feedstock into SP-mold with sufficiently high transcription. On the other hand, the sintered parts processed at 973K shrank 20% in both height and width, and became round at the top and bottom of corner portions. As the sintering temperature is raised, the shrinkage ratio increased remarkably up to 873K and further increased gradually. It was also seen that the shrinkage ratios of $L/S=5\mu\text{m}$ structures are larger than that of $L/S=10\mu\text{m}$ ones and the whole bodies.

The processability of a variety of μ -SPiMIM processes as above-described is summarized in Fig.36 in compared with the other precision processing and machining methods. A conventional rapid plot typing is difficult to manufacture SP-mold with micro-scale structure and high dimensional accuracy. Then RP/ μ -SPiMIM method is not superior compared to micro machining such as micro-cutting, micro-EDM and micro-casting on the size of products, but it has a great potential to manufacture complex shaped parts such as micro-impeller shown in Fig.17. Further micro-miniaturization and surface quality improvement of rapid plot typing are required for μ -SPiMIM process, micro rapid plot-typing using stereo-lithography and 3D-printing technology is a prospective method for manufacturing of fine structured SP-mold. On the other hand, LIGA/ μ -SPiMIM and Resist/ μ -SPiMIM are very hopeful combined methods for manufacturing metallic micro-structured parts with high aspect ratios. The size possible for manufacturing is ranged from hundreds micrometers to tens micrometers. The problems are included high manufacturing cost and shape limitation of SP-mold. Furthermore, NIL techniques has a possibility for

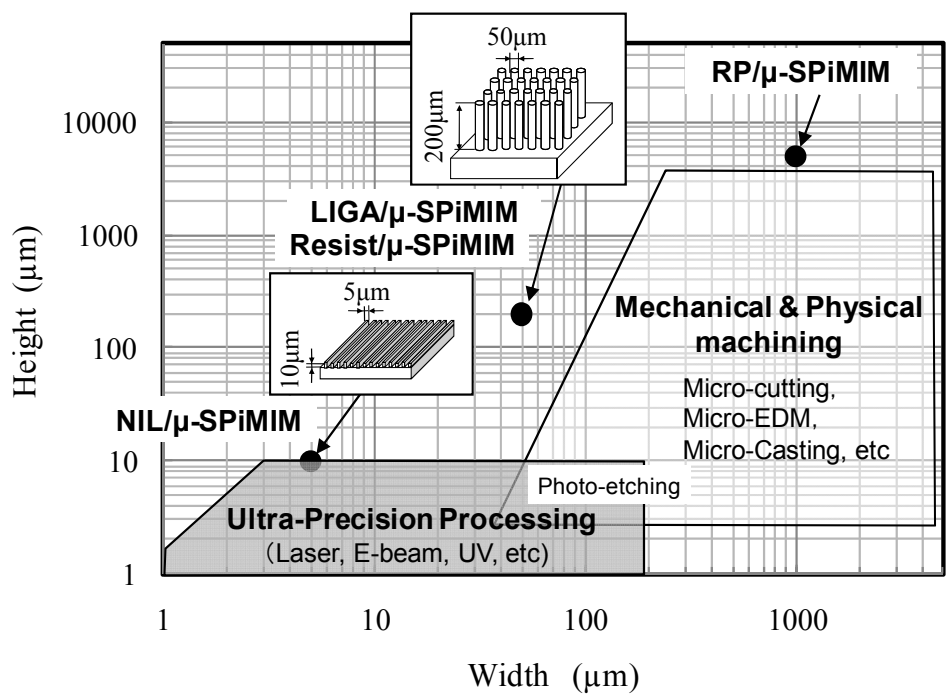


Fig. 36. Processability of a variety of μ -SPiMIM processes.

manufacturing a single digit micrometer-sized structured part, but technical problems on quality such as high-densification and sintering by using nano-scale metal powders should be cleared in NIL/ μ -SPiMIM process at this moment. In addition, evaluation methods and designing of micro devices are hoped with effectiveness of these micro-structure and a variety of metallic own properties. These μ -SPiMIM processes have a unique advantage on fabricating as a one component with macro-scale and micro-scale structures made from a variety of materials. Also the micro-scale open and closed porous structures can be formed in sintering parts. This is not accomplished by semiconductor processes and depositions methods. Therefore it is hoping to use advanced applications such as micro reactor and micro-patterned electrodes with catalyst activity for fuel cell and battery manufacturing or micro-sensors for medical devices.

5. Conclusion

In this chapter, a general characteristic of MIM process on materials and conditions for manufacturing of small metallic parts with high quality was described utilizing actual data, and the complex flow characteristics of MIM were introduced on two kinds of small components, such as micro gear and micro dumbbell specimen. Then the technical problems to be solved for micro-miniaturizing of MIM parts were addressed, and the effectiveness of sacrificial plastic mold in micro-MIM process was shown by citing some example productions of micro-structured parts. A variety of methods for fabricating of the sacrificial plastic mold such as rapid proto-typing, plastic injection-molding, LIGA process and nano-imprint lithography process, were introduced by showing the investigation results on the effects of metal particle size and processing conditions. The use of nano-sized metal powder was applied in micro MIM process inserted sacrificial plastic molds made of resist or nano-imprint lithography, the results that the decreasing of particle size improved the surface roughness and shape-transcription of sintered parts were shown obviously. In collusion, micro sacrificial plastic mold insert metal injection molding, named as μ -SPiMIM method has a great potential to solve technical problems occurring in the μ -MIM process, it can be produced precisely the 3 dimensional complex metallic metal components with single-digit micrometer structures.

6. Acknowledgment

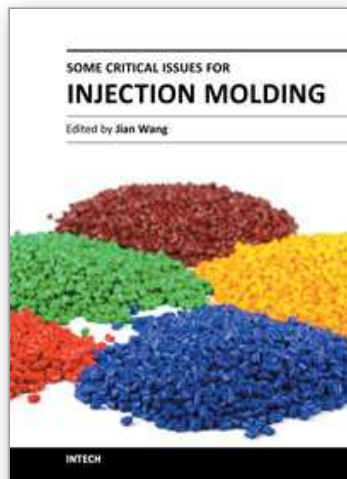
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Some Critical Issues for Injection Molding

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This book is composed of different chapters which are related to the subject of injection molding and written by leading international academic experts in the field. It contains introduction on polymer PVT measurements and two main application areas of polymer PVT data in injection molding, optimization for injection molding process, Powder Injection Molding which comprises Ceramic Injection Molding and Metal Injection Molding, and some special techniques or applications in injection molding. It provides some clear presentation of injection molding process and equipment to direct people in plastics manufacturing to solve problems and avoid costly errors. With useful, fundamental information for knowing and optimizing the injection molding operation, the readers could gain some working knowledge of the injection molding.

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