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Micro powder-injection moulding of metals and ceramics

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Abstract. Development of micro-MIM/-CIM was started at Forschungszentrum Karlsruhe with the aim of creating a process suitable for a wide range of materials as well as for medium-scale and large-scale production of micro components. Using enhanced machine technology and special tempering procedures, this process enables the manufacturing of metal and ceramic devices with smallest wall thicknesses of 50 µm and structural details of less than 3 μm. Using ultrafine ceramic powders (e.g. zirconia) and high-quality LIGA mould inserts, surface qualities of $R_a = 40$ nm or $R_{\max} \leq 3$ mm could be obtained. Possible practical applications are demonstrated by components of micro-annular gear pumps made of zirconia for future handling of very small volumes of dangerous fluids and micro samples (tensile and bending specimens) suitable for mechanical testing of metals (316L, 17-4PH) and ceramic materials (Al_2O_3, ZrO_2) in the micrometre range.

Keywords. Microsystems technology; MEMS/MOEMS; LIGA; powder-injection moulding; micro-MIM; micro-CIM.

1. Introduction

Manufacturing of microsystems products not only possesses a respectable market potential (Wechsung 2000) but is also regarded as one of the leading technologies of our time. An important condition for economic breakthrough is the availability of manufacturing processes suitable for medium-scale and large-scale series production. For micro parts made of silicon, some well-known processes from microelectronic production can be adapted, while for polymeric materials different types of micro injection moulding are suitable. The first plastic products manufactured by micro injection moulding have successfully entered the market.

The logical question now is how to proceed in case of applications that require better material properties than those offered by polymers. There are many such processes, each one being characterised by some advantages (Shimizu *et al* 1999), but in most cases their overall qualities are not sufficient. An exception is micro powder-injection moulding, because it combines the possibility of large-scale serial production with a wide range of materials, so that it has great economic potential (Rota *et al* 1998; Piotter *et al* 1999). Therefore, the special features and possibilities of micro injection moulding in general are described here, followed by micro-PIM experiments using powder-filled feedstocks.

2. Micro injection moulding

Micro injection moulding is an established and economic process for manufacturing micro-components of complex shapes in large numbers. As common injection moulding parameters like relatively low tool temperatures and injection pressures lead to incomplete filling of the microstructured mould insert, injection moulding of complex micro-components has to be carried out at elevated tool temperatures. For amorphous thermoplastics (e.g. PMMA, PC, PSU) these temperatures are above the glass transition temperatures. For semi-crystalline thermoplastics (e.g. POM, PA, PEEK) they often reach the crystallite melting points. Prior to demoulding, the moulded part, the injection moulding tool, and the mould inserts have to be cooled down to a certain demoulding temperature, which is determined by the material strength and the specific microstructure. This tempering cycle leads to relatively long cycle times in micro injection moulding and has to be compensated for, for example by increasing the number of microstructured mould cavities. As the microstructures in the mould inserts represent "blind holes" which are filled from the face of mould insert, venting of the cavity via gaps or the parting plane of the tools is not possible in micro injection moulding. Therefore, the machine periphery includes a special vacuum unit for the evacuation of the mould cavities.

3. PIM experiments

In an effort to adapt the micro injection moulding process to powder metallurgy, the so-called micro powder-injection moulding process is currently being developed at the Forschungszentrum Karlsruhe. This process operates at higher injection pressures (as typically known from macroscopic injection moulding) compared to the low-pressure injection moulding technique (Knitter & Bauer 2003). The main steps of the entire process line are shown in figure 1.

The experiments start with screening of established powders, binders, and feedstock systems as well as of debindering methods to test their applicability for micro injection moulding (Piotter *et al* 1998).

As an example of metals, carbonyl iron powder with a mean particle size of 4–5 μm was chosen and regarding the ceramic materials, most investigations were carried out with aluminum oxide powder (0·6 μm mean particle diameter) or ZrO_2-strengthened Al_2O_3. In

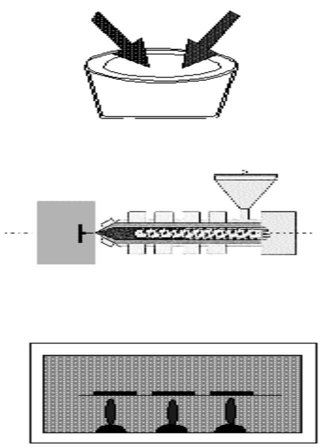

Figure 1. Schematic principle of powder injection moulding consisting of three steps: Mixing of feedstock, micro injection moulding, debindering/sintering.

certain cases, yttrium oxide-stabilized zirconium oxide with a mean particle size of 0·3–0·4 μm was used (Ruprecht & Piotter 1999).

For the investigations, mainly two commercially available binder systems were selected: Polyolefin/wax compounds and a polyacetal-based system. The experiments started with commonly available systems, followed by self-prepared feedstocks (Merz *et al* 2001).

In the first experiments, test structures were injection-moulded by MIM with 260 μm lateral width and an aspect ratio (comparable to the flow length to wall thickness ratio) of nearly 5, while for further investigations LIGA and UV-LIGA mould inserts with aspect ratios of more than 10 were applied. In more than five hundred injection moulding cycles so far, mostly with automated machine operation, no wear phenomena have been detected in the microstructured nickel mould inserts.

For debindering, three basically different methods have been investigated.

(1) Debindering by thermal elimination of the organic components. This method is often supported by a previous step of dissolving a certain amount of binder in organic solvents or simply in water;
(2) Debindering by supercritical carbon dioxide. This technology uses carbon dioxide under supercritical conditions in an autoclave which normally operates at temperatures of more than 330 K and pressures of $\approx$ 300 bar (Merz 1997);
(3) A catalytical debindering process as commonly used for polyacetal-based systems.

For debindering polyolefin/wax structures, both methods, i.e. thermal elimination and debindering by supercritical carbon dioxide, were examined. Although supercritical debindering allows an extraction of up to 80% of the binder within one hour and thus works sufficiently well, additional modifications of the process parameters are necessary. Hence a final assessment of supercritical debindering in micro powder injection moulding cannot yet be done, so that most micro components moulded with polyolefin feedstocks were debindered by thermal elimination. For debindering feedstocks based on polyacetal binders, a nitric acid atmosphere was appropriate.

The sintering procedure was carried out in a tube furnace with a diameter of 88 mm and a heating zone length of 600 mm. While the ceramic micro parts were sintered under air supply, a reducing H_2 atmosphere was necessary for the metal microstructures to prevent oxidation of metal.

4. Examples of micro components

As a typical example of micro components for micromechanical applications, samples of stepped gear wheel structures were manufactured using mould inserts made by the LIGA process. For demonstration purposes, carbonyl iron, stainless steel (316L), aluminum oxide, aluminum nitride (under development), and zirconium oxide powders have been chosen for these structures, but processing of other materials might also be possible. Up to now, minimal dimensions after sintering are 50 μm lateral width and 480 μm structural height, while the exact geometrical data vary according to the different shrinkage values of the feedstocks used (see figure 2).

Stepped grid test structures represent the actual limitations of micro-PIM. As demonstrated by figure 3, replication of structural details smaller than 1 μm is possible in principle. However, the performance of the whole part is determined by the surface quality rather than by the geometry.

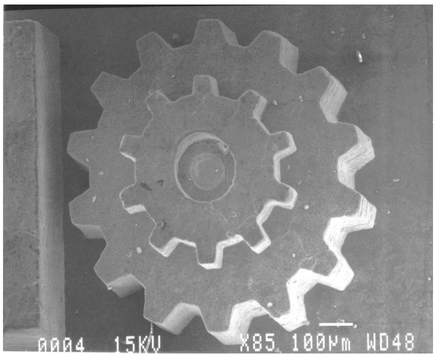

Figure 2. Stepped LIGA gear wheels made of 316L stainless steel. Structural height is about 480 μm and minimum tooth width about 50 μm; both values were measured after sintering.

A recent development with high market potential is the manufacturing of micro annular gear pumps. Use of metal or ceramic materials makes these pumps suitable for the processing of hazardous fluids which cannot be handled by polymer-made devices.

Figures 4 and 5 display some details of the micro annular gear pump.

Powder injection moulding was carried out with zirconia feedstock using a LIGA mould insert with a depth of 650 μm. Afterwards, the micro parts were isolated. Figures 6a-c show the surface of an isolated part as well as of the surface-finished micro parts (Gietzelt *et al* 2001).

Further examples are specimens for micromechanical tests, like bending bars and tensile test bars. The sizes are $250 \times 250 \times 2000$ μm for the green bending bars (see figure 7) and $260 \times 130 \times 4500$ μm for the green tensile test bars, respectively (see figure 8). These microparts could be manufactured without substrate plates and have aspect ratios between 8 and 12.

Another demanding microstructure is a micro gearwheel with an evolvent toothing and a total diameter of 1260 μm in the sintered state (see figure 9). It has to be mentioned, that the mould inserts for these microparts were manufactured by high precision micro milling of hardened steel with 56 HRC.

5. Results

One important aspect during the investigations on micro powder injection moulding was to establish the critical dimensions for successful replication and determine the various physical properties (densities, surface qualities etc.).

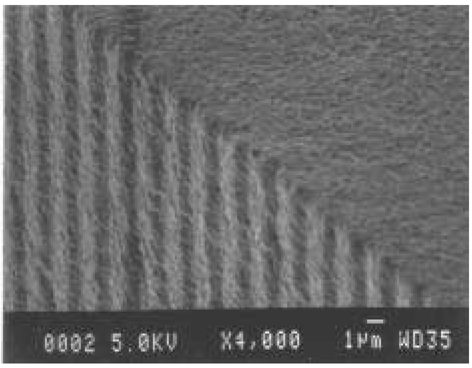

Figure 3. Detail of powder injection-moulded micro grid test structure with a zig-zag height of approx. 0·9 μm in zirconium oxide (bottom).

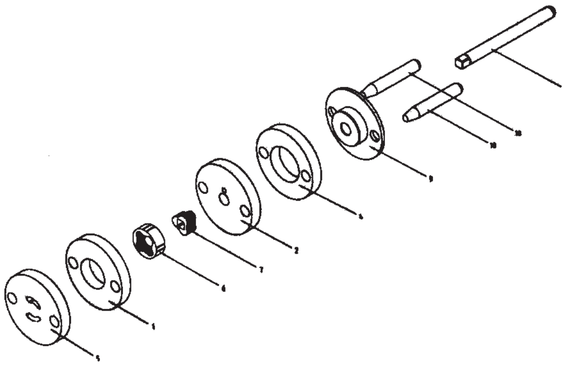

Figure 4. Principle drawing of the micro annular gear pump. Outer diameter: 3·2 mm.

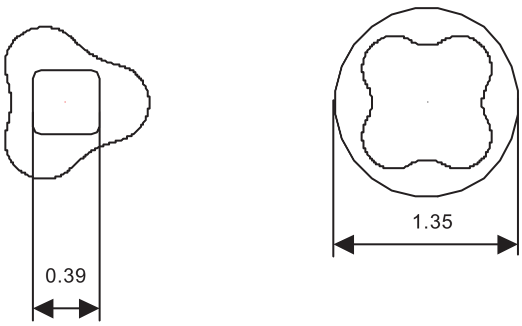

Figure 5. Details of the annular gear pump in millimetre.

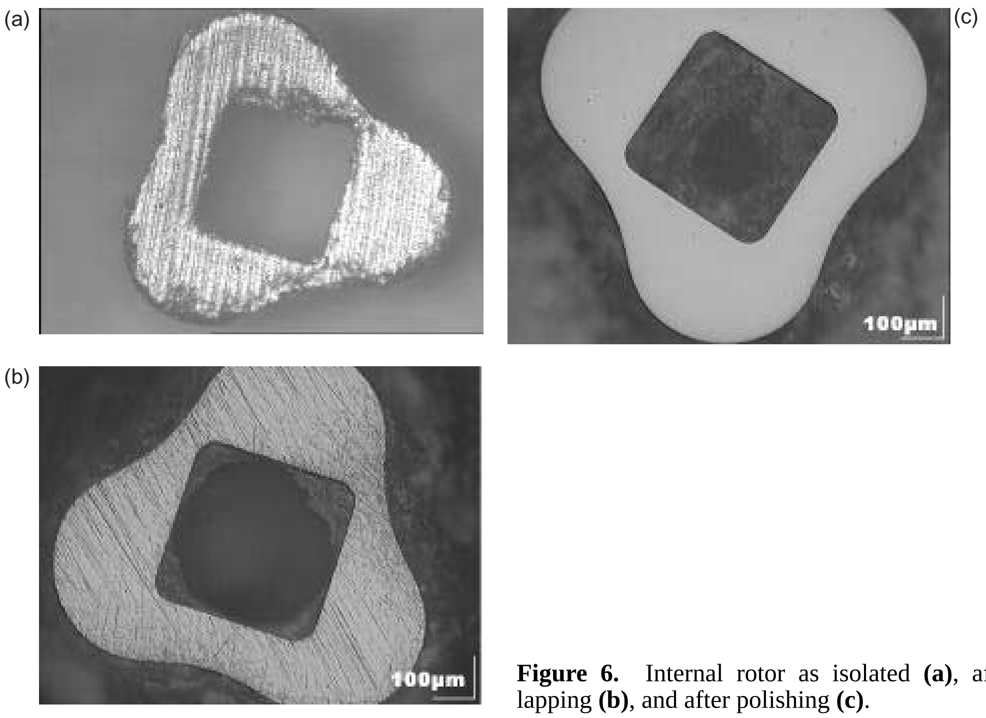

Figure 6. Internal rotor as isolated **(a)**, after lapping **(b)**, and after polishing **(c)**.

Figure 7. Bending bars (17-4PH steel, green parts) for micromechanical tests.

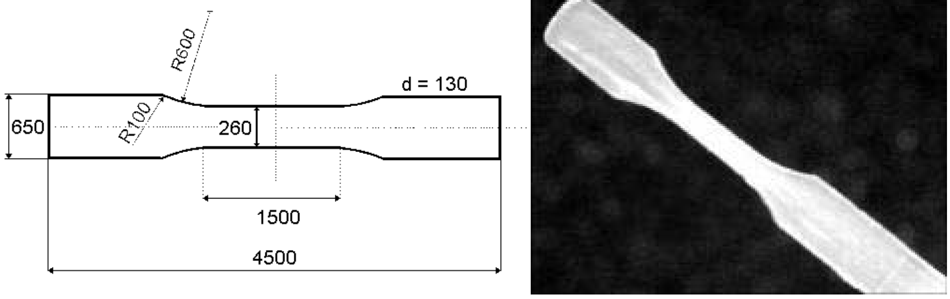

Figure 8. Tensile test bars (ZrO_2, green part) with details in μm.

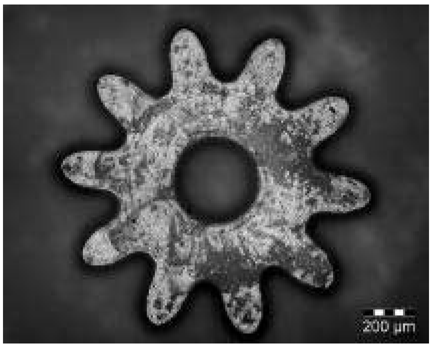

Figure 9. Sintered ZrO_2 micro gearwheel, outer diameter 1260 μm.

Table 1. Maximum densities and surface qualities achieved after sintering.

Material	d_{50} (μm)	Density (g/cm^3)	Density (% theor)	R_{max} (μm)
Carbonyl-Fe	4·5	7·53	95·6	> 8
Carbonyl-Fe	1·5	7·53	95·6	4
316L	4·5	7·78	96·7	8
Al_2O_3	0·4–0·6	3·85	96·7	3
ZrO_2	0·2–0·4	6·05	99·2	2

The lowest weights of the parts after separation and finishing were 0·25 mg only for LIGA gear wheels made of aluminum oxide. This is much lower than the values mentioned in literature (German 1998). On the other hand, the total shot weights currently exceed 20 g, because the typical injection-moulded arrays of micro parts are connected with runner systems as well as with auxiliary structures which are actually necessary to guarantee safe demoulding.

Another important step of manufacturing micro parts was the development of a technique for isolation and surface finishing that was suitable not only for laboratory, but also for large-scale production.

Densities of the final parts achieved after sintering were similar to typical macroscopic ones and are shown in table 1 together with the R_{max} roughness values for certain materials. In this case, LIGA structures with R_{max} lower than 1 μm were used.

Obviously, the mean particle diameter has a significant influence on the accuracy and surface quality of the replicated structures (Piotter *et al* 1999). Special metallic powders with mean particle dimensions in the range of 1–5 μm seem to be sufficient for many applications, but best results have been achieved by using ceramic powders with mean particle diameters of 0·6 mm or even smaller. Due to the smaller particle size of the ceramic powders, the micro structures were manufactured with a better surface quality of R_{max}2–3 μm compared to the metal microstructures that reached R_{max} values of 4–8 μm only.

The linear shrinkage ranged from 15–22% depending on the composition of the feedstocks. In all cases, nominal sizes in the micrometre range were obtained with a standard deviation of 0·5–0·7%.

As shown by the tensile test and bending bars, avoiding the substrate plate results in defect free surfaces if finishing steps can be omitted. This leads to mechanical properties, which are at least comparable to macrodimensional tests specimens or even better. The average bending strength for ZrO_2 specimens reached 2100 MPa.

6. Outlook

Presently, development of micro powder injection moulding is being continued in order to achieve an industrially feasible process. This includes new methods and moulding tools for economic isolating and finishing.

Further experiments deal with the utilization of very fine metal powders as well as nearly nanoscaled powders in case of ceramic materials. Two-component micro injection moulding using ceramic feedstocks represents an ambitious future task.

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