TECHNICAL PAPER

Production of two-material micro-assemblies by two-component powder injection molding and sinter-joining

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Received: 26 October 2007/Accepted: 21 May 2008/Published online: 4 June 2008 © Springer-Verlag 2008

Abstract In the field of micro-technology the production of metallic and ceramic micro-components by powder injection molding (PIM) has become a more and more established fabrication method. But in order to fulfill the demand for more complex-shaped high-precision microcomponents further development work has to be performed. This is especially true if more efficient production routes for multi-component-micro-assemblies consisting of different materials or sub-components are envisaged. To meet these challenges, investigations are performed to realize and to establish two primary shape micro-processes. These are two-component micro-injection molding (2C-MicroPIM) and sinter-joining. The realization of these technologies will lead to a markedly reduction of the efforts for handling, adjustment, and assembling of metallic and ceramic microassemblies. Furthermore, an increased integration level and functionality can be yielded. For an effective transfer of scientific results to industrial applications the whole process chain must be considered, from development and construction of the tooling as well as of the components to the quality assurance and determination of the properties of the assemblies after sintering. These primary shape processes shall enable the mutual processing of different materials within the fabrication process, so avoiding separate mounting or assembling steps. Additionally fixed and loose junctions between at least two components shall be realized.

A.-M. Dieckmann · C. Munzinger · J. Fleischer Universität Karlsruhe (TH), Institut für Produktionstechnik (wbk), Kaiserstraße 12, 76128 Karlsruhe, Germany The progress in research and development will be demonstrated especially by the implementation of shaft-to-collar connections between micro-gearwheels and corresponding shafts. Regarding two-component micro-injection molding, the tool construction for shaft-to-collar connections will be presented as well as first experimental results on the properties of selected ceramic powders and feedstocks for the special requirements of the 2C-MicroPIM process. With the assembly step being performed outside the injection molding tool before sinter-joining different parts and geometries can be combined quite easily. The presented article gives an overview on the concept and on preliminary testing results for the fabrication of a shaft-to-collar-connection. Additionally, a solution for an automated assembly of a shaft and a toothed wheel outside the injection molding tool is presented.

1 Introduction

The industrial application of micro-components led to a variety of new products in recent years. In the future the production of micro-assemblies is expected to extend towards higher toughness, more complex shapes, and further miniaturization. For this purpose two-component micro-injection molding (2C-MicroPIM) and sinter-joining as two different micro-primary shape processes for manufacturing complex, three-dimensional, metallic or ceramic micro-assemblies with free-form surfaces are under investigation. Sinter-joining is preferentially suited for the production of small- and medium-scale series since a flexible alignment for changing assembly combinations is possible. In contrast, two-component micro-injection molding is a process most advantageous for large-scale production.

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The process enables two components of different materials to be joined in one injection molding cycle so resulting in a more or less complex micro-assembly. Using appropriate injection molding and sinter parameters, the formation of a flexible or of a fixed junction can be accomplished by both techniques. Multi-component injection molding based on plastics could be successfully applied several times in micro-technology (Seifert and Münch 1999). The usage of metallic powder within the 2C-MicroPIM process for establishing a fixed junction of the stainless steels 316L with 17-4PH could be demonstrated (Imgrund and Rota 2003; Rota et al. 2004; Imgrund et al. 2005).

Powder injection molding of assemblies made up of two different ceramics is also feasible. Here a u-shaped heating element consisting of an Al_2O_3/TiN mixed ceramic with different mixing ratios has been developed as demonstrator (Finnah et al. 2005; Piotter et al. 2007).

Sinter-joining is an innovative fabrication method with green PIM parts being assembled and joined during sintering resulting in a defined connection. This opens up the opportunity to produce multi-component assemblies from single injection molded parts during the sintering process.

The development of this processing route has two challenges. These are the reduction of the complexity of (automated) assembly on the one hand and also the adjusting the sintering behavior of the joint parts from different materials on the other hand (Avisihai et al. 2005; Fleischer and Dieckmann 2006a). Subject of the current investigations is to sinter-join two micro-components for the fabrication of different matchings. This shall be performed in a fully automated process in order to realize an as small as possible scatter of process parameters.

2 Two-component powder injection molding

2.1 Work plan and methods

The development of a fixed compound and of a movable junction between a gearwheel and a shaft, both of different materials, is the objective of the current investigations. Therefore, it is necessary to have a precise dimensioning concept and also to generate sufficient information about the applied materials, which are not only the different ceramics or metals themselves but also their feedstocks compounded for injection molding, which contain additional binders and plasticizers. Key activities of the current work are the construction of a tool allowing the realization of fixed as well as of loose junctions but also experimental investigations on powders and feedstocks. This paper presents the properties and characteristics of the materials and material combinations that will be considered for the 2C-MicroPIM process. The tool design allows the formation of junction areas by simultaneous or sequential injection of the feedstocks. Using a gate/core puller should be warranted if required.

Focus of the experimental investigations is the materials characteristics of adequate Al_2O_3 and ZrO_2 powders. As the shrinkage characteristics during sintering are relevant for the formation of fixed and moveable junctions, shrinkage and sinter parameters of isostatically pressed, cylindrical samples (length 7–10 mm; diameter about 5 mm) have been measured using a Netzsch dilatometer of the type DIL 402C. The dimensional changes have been determined at a heating rate of 5 K/min up to a temperature of 1300°C and subsequently 2.5 K/min up to 1550°C, 2 h dwell at 1550°C and a cooling rate of 5 K/min down to room temperature.

For producing the feedstocks, different volume contents of the ceramic powders have been mixed with polyethylene, paraffin, and stearic acid as dispergator and compounded using a Brabender measuring mixer of the type W 50 EHT. The behavior of the feedstocks during dispersing has been determined by measuring the torque as a function of time.

2.2 State of work, results and discussion

For 2C-injection molding an index-plate tool with two hot runners and an electric ejector unit was developed and put into operation. This enables cycles with a low ejector velocity. Special tool engineering characteristics like variotherm process control, evacuation of the mold cavity, and easy exchangeability of the mold insert, which are necessary for micro-powder injection molding, were incorporated into the tool design. A gating concept consisting of two C-shaped arcs was chosen for the injection of the gearwheels. In contrast to a point-shaped gate system, the concept with two C-shaped arcs offers a larger gate cross-section in order to avoid the risk of demixing in the feedstocks. Further advantages are the simple production of the gate system and the short demolding distance. Possible welding lines may be reduced by using a variotherm process. Thus, mold filling takes place at higher tool temperature. Changeable mold inserts enable a variable design of the tool cavity.

The demonstrator gearwheel–shaft will combine a tough zirconium oxide ceramic as micro-gearwheel and wearresistant aluminum oxide as aligned shaft. The assembly composed of a micro-gearwheel and a shaft with collar is shown in Fig. 1.

2.2.1 Influence of the different coefficients of thermal expansion

During heating to sinter temperature the brown body consisting of zirconia ($\alpha_{th} \sim 10\text{--}11 \times 10^{-6} \text{ K}^{-1}$) shows a



Fig. 1 Geometry of a 2C-Micro-PIM assembly, consisting of gearwheel and shaft (FZK-IMF-III)

higher thermal expansion than the alumina shaft ($\alpha_{th} \sim 7-8 \times 10^{-6} \text{ K}^{-1}$). In the zone of the driving collar the expansion of the zirconia gearwheel will exceed that of the shaft by about 2 µm. This behavior is generally beneficial for the realization of the moveable junction, viz. a movable shaft in the collar. During cooling from sintering temperature to room temperature, the gearwheel will shrink 2 µm more than the shaft. This is beneficial for the realization of a fixed junction. For realizing a moveable junction this effect must be considered. A higher shrinkage and/or a higher sinter density may eliminate the disadvantage of that effect.

2.2.2 Influence of shrinkage

For realizing a moveable junction, an earlier start of the sinter shrinkage of the shaft in comparison to the shrinkage of the gearwheel is advantageous. A higher shrinkage and/ or a higher sinter density for the shaft is necessary. Furthermore, the shrinkage rate of the shaft must be faster or at least as fast as that of the gearwheel. However, for realizing a fixed shaft-to-collar connections there are reverse conditions.

All those conditions can be met by the right selection and by adjustment of the following parameters:

- Thermal characteristics of the chosen materials.
- Choice of suitable powders and their sintering characteristics.
- Solid content in the feedstock.

These investigations were performed, showing the following results.

For determining the temperature of the sinter process and dynamic of shrinkage, dilatometric measurements have been carried out on several Al_2O_3 and ZrO_2 powders. The specifications of the investigated powders are given in Table 1.

The sintering of both ZrO_2 powders, ZrO_2 -type 1, used so far within SFB 499, and ZrO_2 -type 2, starts at a temperature of about 1100°C, but ZrO_2 -type 1 shows an accelerated shrinkage dynamic (Fig. 2). In contrast to alumina powder Al_2O_3 -type 1 (start of sintering at about 1020°C), ZrO_2 -type 2 shows a relatively late initiation of shrinkage. Furthermore, the sinter rate, viz. the dynamic of

Table 1 Specifications of ceramic powders used in this work

Denotation	Composition	Average particle size (µm)	Specific surface (m ² /g)
ZrO ₂ -type 1	ZrO ₂ -3 mole% Y ₂ O ₃	0.63	6.6
ZrO ₂ -type 2	ZrO ₂ -3 mole% Y ₂ O ₃	1.02	5.86
Al ₂ O ₃ -type 1	Al_2O_3	0.26	12.2
Al ₂ O ₃ -type 2	Al_2O_3	0.7	7.28

shrinkage of both the last-mentioned powders, is similar making them a promising choice for the realization of a moveable junction (see Fig. 2). The densities of the pressed body before and after sintering are also relatively similar (green density of Al_2O_3 -type 1 54.2%, sinter density 97.3 %; green density ZrO_2 -type 2 55.9%, sinter density 98.7%). In contrast, the green density of zirconia powder ZrO_2 -type 1 is considerably lower (49.2%) with the sinter density being higher (99.8%) than ZrO_2 -type 2 and Al_2O_3 -type 1. Using an identical filling rate of the powders ZrO_2 -type 2 and Al_2O_3 -type 1.

The applicability of feedstocks for the micro-powder injection molding process depends on the filling rate of the powder as well as the type and concentration of the applied plastics, waxes, and dispergators. Therefore torques have been measured on the feedstocks with the alumina powders Al_2O_3 -type 1 and Al_2O_3 -type 2 as well as on the zirconia powders ZrO_2 -type 2 and ZrO_2 -type 1 during mixing experiments by variation of the powder filling rate and of the concentration of stearic acid (C₁₇H₃₅COOH), used as dispergator.

The results after 1 h mixing of Al_2O_3 powder Al_2O_3 type 1 (specific surface 12.2 m²/g) with polyethylene, paraffin, and stearic acid are shown in Fig. 3.



Fig. 2 Relative length changes of selected ceramic powders during sintering (FZK-IMF-III and IMTEK Freiburg). Parameters see Chap. 2.1



Fig. 3 Final torques measured after 1 h dispersing during the production of feedstocks based on Al_2O_3 powder (Al_2O_3 -type 1), polyethylene, paraffin, and stearic acid, in dependence on the amount of stearic acid and powder load (FZK-IMF-III)

The addition of $1.1-4.4 \text{ mg/m}^2$ (relative to the powder surface) stearic acid is advantageous with respect to the viscosity of the feedstocks. The optimum amount of stearic acid was found to be 2.2 mg/m^2 . Investigations performed in order to investigate the influence of the solid content in the feedstock on the processability demonstrate a good processability of the feedstocks up to a powder load of 60 vol.%. These results show that a sufficiently wide parameter range is available, which enables the production of appropriate feedstocks with different solid contents for 2C-MicroPIM. These feedstocks allow the controlled variation of the shrinkage characteristics during sintering via the powder load. So it will be possible to realize moveable or fixed junctions between shaft and gearwheel by using 2C-MicroPIM.

3 Sinter-joining

For sinter-joining two green PIM parts are assembled and sintered afterwards (Fig. 4). Due to the different rates of

Fig. 4 Process chain for sinterjoining (wbk) shrinkage during sintering the parts will form a defined connection. The connection can be loose or fixed. In this way, already the sintering process will produce a component fit to be used as a finished sub-assembly later.

3.1 Shaft-to-collar connection

One of the possible applications of sinter-joining is the fabrication of a shaft-to-collar connection (SCC) enabling different materials to be joined in one assembly (Fig. 5).

The SCC requirements on precision and the quality of the connection itself are considerable (Avisihai et al. 2005). Different material combinations such as metal/metal and ceramics/metal have to be developed. In addition, work is focused on the development of various joints, fixed and loose.

3.2 Preliminary tests

In the beginning, the connection of tungsten steel rods and powder injection-molded green carbon steel toothed wheels formed the center of analysis. For that purpose the shrinkage rate of the carbon steel toothed wheels was determined experimentally. The diameter of the tungsten steel rods was adjusted to the resulting diameter of the toothed wheels. Substantial preliminary testing has proven that the sintering conditions have to be considered from a holistic point of view (Fleischer and Dieckmann 2006a). Hitherto existing experiments of realizing a fixed connection have proven to be incomplete under SEM inspection (Fig. 6).

Analysis of polished cut images showed that the process of connection had started already. But obviously it had not been completed yet (Fig. 7). According to grain growth in





Fig. 5 Configuration of SCC (wbk)

the connected area we deduce that the sintered connection had been nascent already but not completed yet.

The investigation showed that the mounting of the assemblies during sintering is of considerable influence on the connection. In the first trials the steel rods had been mounted horizontally. The SEM images prove that the force of gravity has a measurable influence on the sintering activity.

3.3 Measures of optimization

As a reaction to the experimental outcome sinter trays positioning the rods in a vertical manner during sintering have been developed to improve the quality of the joint. The rods are positioned upright into $400 \ \mu m$ boreholes



Fig. 7 Polished cut image of SCC (iwk 1, wbk)

which is the optimum position for joining them to the toothed wheels (Fig. 8). The defined position on the sinter trays helps keeping up the order induced by mechanical assembly. Additionally, the vertical positioning during sintering diminishes the negative influence of the force of gravity upon the growth of the joining seam in the shaft-to-collar connection.

By means of an automated solution the assembly can be carried out in the delicate green parts (Walcher 2001). This way other disruptive factors and sources of variation during



Fig. 6 SEM image of the SCC (iwk 1, wbk)



Fig. 8 Positioning during sintering (wbk)

the generation of the parts' connection can be excluded. Based upon the experiences in handling green parts (Fleischer and Dieckmann 2006b) a vacuum-based solution has been selected for the automation problem. Additionally, mechanical catches provide the necessary precision in the transfer points.

The critical part of the automation solution is the assembly station for the rod and the wheel (Fig. 9). Within the assembly station the toothed wheel is being fixed by a primary vacuum and consequently assembled with the rod. Mechanical fits of parts are in use during the assembly operation. They support the positioning of the wheel and the rod in relation to each other.

The assembly tolerances and the shrinkage of the toothed wheel's diameter relative to its diameter in a green state are interdependent. Therefore the tolerances result directly from the toothed wheels' diameter. Accordingly,



Fig. 9 Function of assembly station (wbk)

the tolerances of concentricity for the toothed wheels being assembled upon the non-shrinking metal rods are tight.

For a non-destructive assembly of parts tolerances of 10 μ m must be maintained for a total part's height of 600 μ m as this is the length of the rod dipping into the toothed wheel.

4 Outlook

The oncoming studies on the realization of moveable and fixed ceramic junctions by two-component powder injection molding focus on process parameters of injection molding and sintering as well as on junction zone and geometries of 2C-MicroPIM assemblies. Special attention will be given to diffusion processes within the system Al_2O_3 -ZrO₂-Y₂O₃, possibly resulting in the formation of a mixed oxide or an intermediate phase. This would be advantageous for the formation of a fixed junction, but would possibly lead to problems for a moveable junction. The current investigations will be extended with respect to the development of moveable junctions consisting of metal/metal pairings. Research work also will take into account the question if two feedstocks of the steel powders 17-4PH with different powder loads can stay separate during sintering by choosing suitable process parameters which utilize the differences of shrinkage in a way that the gearwheel may rotate around the shaft.

In the following experiments on sinter-joining metal toothed wheels shall be joined with sintered ceramic rods. For the enhancement of the SCC the rods and toothed wheels are both to be assembled in a green state, followed by shrinkage of the rods during sintering. Therefore the mechanical fits must be enhanced down to $1-2 \ \mu m$. In addition to that, the automation of the assembly and the entire process chain shall be realized.

Toothed wheels and rods are to be unloaded automatically from the injection mold and transferred automatically to the assembly process. After the assembly operation the assemblies are to be transferred automatically into the continuous debinding and sintering furnace. By these means it can be ensured that neither parts nor assemblies are damaged during the process.

Acknowledgments This work was performed within the Sonderforschungsbereich 499 (Collaborative Research Center 499). The authors are grateful for financial support of the subprojects C3 und C5 provided by the Deutsche Forschungsgemeinschaft. Further thanks go to Dipl.-Ing (FH) Klaus Plewa for his efforts on the design and construction of the injection molding tool. We thank Dr. rer. nat. Brando Okolo for the analysis documented within Figs. 6 and 7 and Mr. Roman Keding (IMTEK, Universität Freiburg) for the dilatometric measurements on Al_2O_3 -type 1. Additionally, the authors would like to thank the all other colleagues from IMF III (Forschungszentrum Karlsruhe) and from wbk (University of Karlsruhe) for the good collaboration.

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