

Multi-component microinjection moulding-trends and developments

V. Piotter · J. Prokop · H.-J. Ritzhaupt-Kleissl ·
A. Ruh · J. Hausselt

Received: 15 June 2008 / Accepted: 17 February 2009 / Published online: 5 March 2009
© Springer-Verlag London Limited 2009

Abstract With standard microinjection moulding becoming more and more established in practical manufacturing, special variants are attracting increasing attention. Especially, the approaches on multi-component microinjection moulding are worth mentioning: As handling and assembly are difficult procedures especially in microtechnology, methods to reduce mounting efforts are of high economic importance. By merging of shaping and mounting procedures in one step, economic progress as well as new material combinations can be obtained. An interesting approach for the fabrication of metal (or in principal, ceramic) microcomponents is the combination of insert injection moulding and metal deposition by electroforming. First, an electrically conductive base plate is produced by injection moulding of conductively filled polymers. In a second injection moulding step, microstructures consisting of insulating plastics are mounted on these plates. The quasi-infinite conductivity gradient allows controlled electroplating starting at the base plate only so that defect-free metal microcomponents can be achieved. As a further variant of microinjection moulding, the development of the so-called MicroPIM process facilitates a large-scale series fabrication technology for metal and ceramic microcomponents. Combined with multi-component technology, an interesting new approach for micromanufacturing is obtained, i.e. the realisation of magnetic/non-magnetic or conductive/non-conductive material combinations by two-component MicroPIM. But, also the combination of different mechanical properties like hard/tough pairings is possible.

Keywords Microinjection moulding · Two-component injection moulding · Powder injection moulding · Electroplating · Galvanoforming

1 Introduction

Up to now, the variety of materials for microtechnology applications has been dominated by silicon, polymers, and certain metals. On the other hand, there is still a lack of materials with high resistant and/or eminent surface properties and the related manufacturing processes for high aspect ratio (HARM) parts. Additionally, the reduction of assembly expenditures is also an important demand [1–5].

For the realisation of multi-material devices as well as the reduction of mounting costs, the development of micro two-component injection moulding represents not only an interesting but also very ambitious challenge. It enables the production of fixed connection between two components as well as movable junctions. The possibility to realise fixed connections by two-component metal injection moulding has been demonstrated on combinations like 316L/H13, 316L/17-4PH and 316L/Fe [6–8]. The bonding between both components is attributed to the diffusion of alloying elements across the interfaces.

Movable connections so far could be realised by multi-component powder injection moulding [8]. In this feasibility study, a feedstock filled with alumina was used as spacer. After sintering, this component could be separated to get a movable, metallic hinge. However, this demonstrator was realised in macroscopic scale. As a consequence, the challenge to realise movable junctions in microdimensions has gained momentum in microtechnology.

V. Piotter (✉) · J. Prokop · H.-J. Ritzhaupt-Kleissl · A. Ruh ·
J. Hausselt
Forschungszentrum Karlsruhe, Institute for Materials Research III,
P. O. Box 3640, 76021 Karlsruhe, Germany
e-mail: volker.piotter@imf.fzk.de

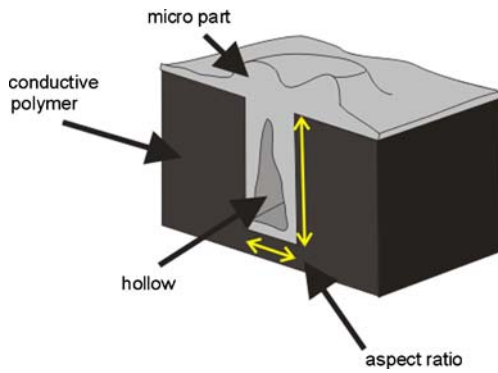


Fig. 1 Principle of one-component injection moulding with electroforming process

In this contribution, two possibilities will be presented: electroplating on lost plastic moulds and micropowder injection moulding.

2 Manufacturing of metal microparts by galvanofarming on partially conductive substrates

2.1 Basic evaluations

At present, micromanufacturing lacks processes suitable for both high-strength materials as well as large-scale series production. In order to transfer these microsystem research concepts of high-aspect ratio metallic microparts into new product developments, there is a need of new manufacturing methods for cost-conscious production [1]. One potential solution is a process sequence, combining injection moulding of polymer sacrificial substrates with a subsequent electroforming step for replication.

Development started with the injection moulding of microstructured fully conductive polymer parts, which had to serve as substrates for the electroplating step and as templates. Unfortunately, this kind of electroforming (undirected growth all over the conductive part) is limited to aspect ratios not larger than 2 because the galvanic

deposit will otherwise overgrow the apertures of the templates before they are completely filled. Thus, partially filled cavities would be left in the product (see Fig. 1).

The reason for this effect is the distribution of the electric field lines in the electrolyte. They are denser at edges and corners than at plain surfaces. Additionally, transport control limits the galvanic deposition rate in narrow cavities as the metal ion concentration there is determined only by diffusion, whereas the concentration near to the electrolyte bulk is usually stabilised by convective exchange. Consequently, the only starting point for galvanic deposition is the structure base. A quasi-infinite conductivity gradient between bottom and side walls of the microstructured substrates has to be guaranteed, i.e. two-component polymer parts are required [1].

2.2 The process line

First, electroconductive material is injection moulded to form a plain electrically conductive base plate. By a second injection moulding step, microstructures consisting of insulating plastic are mounted on the base plate. The mould inserts used are either LIGA-manufactured or micro-mechanically cut. The quasi-infinite conductivity gradient enables controlled electroplating starting at the base plate only.

The two-component mould acts as cathode in the galvanic step. For example, an aqueous 1.5 M solution of nickel sulphamate $\text{Ni}(\text{SO}_3\text{NH}_2)_2$ with standard current densities is used to achieve a deposition rate of $12\ \mu\text{m}/\text{h}$ nickel at a temperature of 52°C . Boric acid, H_3BO_3 , acts as buffer to prevent local formation of hydroxides, especially in the microcavities of the structure. The principle of the process chain is demonstrated in Fig. 2.

2.3 Practical investigations

Basic electroplating trials were carried out on one-component conductive substrates to determine the specific

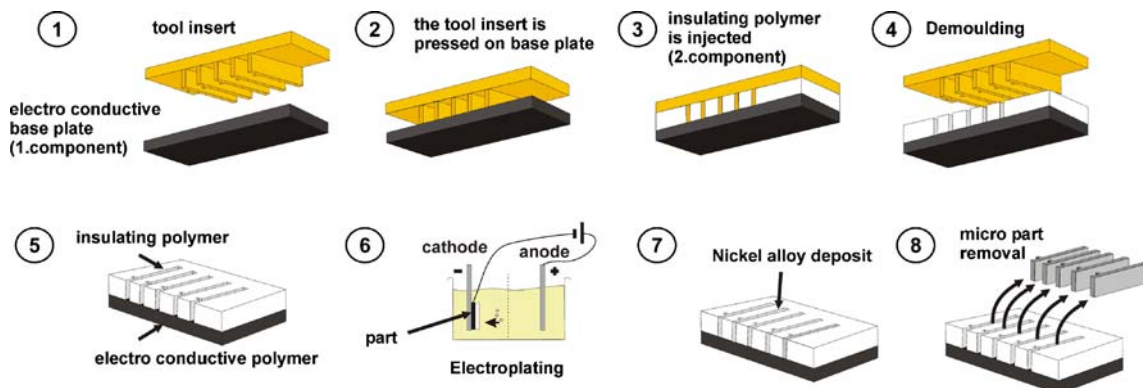


Fig. 2 Principle scheme of the process chain

characteristics of the material and on two-component substrates with microstructures of different types and shapes to study the characteristics and deposition behaviour of many different composites like PMMA, POM, PS, PA12, PA6.6 and their conductive versions.

Injection temperature and pressure, melt velocity, conductive filler type and polymer combination bear a specific influence on parameters like conductivity, roughness, surface structure and subsequently on the initial nucleation and the applicable maximum current densities.

It turned out that the conductivity of the filled polymers is principally lower than that of metallic and metal-coated substrates. The respective surface resistance of conductive polymer of a standard mould substrate (66 mm×26 mm) ranges from 10^1 to $10^3 \Omega$. Rising surface resistance results in consequences on the initial galvanic deposition conditions. The resistance and with it the deposition characteristics show a strong dependency on the kind of conductive filler. The homogeneity of the nucleation behaviour and the following metal growth increased in relation to the finer and more homogeneous distribution of the conductive filling in the polymer matrix, as did the formation speed of regular thick layers with electroformed structures.

Materials like PA6.6-CF and PEEK-CF filled with carbon fibres (length $\sim 10^2 \mu\text{m}$) show lower conductivity than the compounds filled with carbon black and often show an inhomogeneous distribution, causing conductivity lacks and hemispherical nucleation behaviour. As a consequence, many hemispherical, insulated nickel nuclei grow up before they unite to a closed layer or form a plate.

Carbon black of about 35 nm diameter as filler in polyamide (PA12-C) is more likely to show metal-like behaviour. These substrates are far more suitable for the replication of single microstructures from a closed structured resist layer (Fig. 3a–c).

2.4 Injection moulding of filled polymer

The properties of carbon black filled polymers showed a high dependency on the injection moulding parameters. In order to produce microparts on one base plate simultaneously, the conductivity of this base plate must be homogeneous. To understand the dependency of the homogeneity of the injection moulding parameters, a range of tests was carried out, and a new material mixture was produced.

The material mixture consisted of the polymer Grilamid L16A (EMS Chemie) and the carbon black Ketjenblack® EC-600 JD from AkzoNobel. Two different kinds of twin screw extruders were used in order to analyse the effects of the mixing process on the material properties. The machines used were Coperion Werner Pfleiderer lab size twin screw extruders, types ZSK25WLE and ZKS26MC.

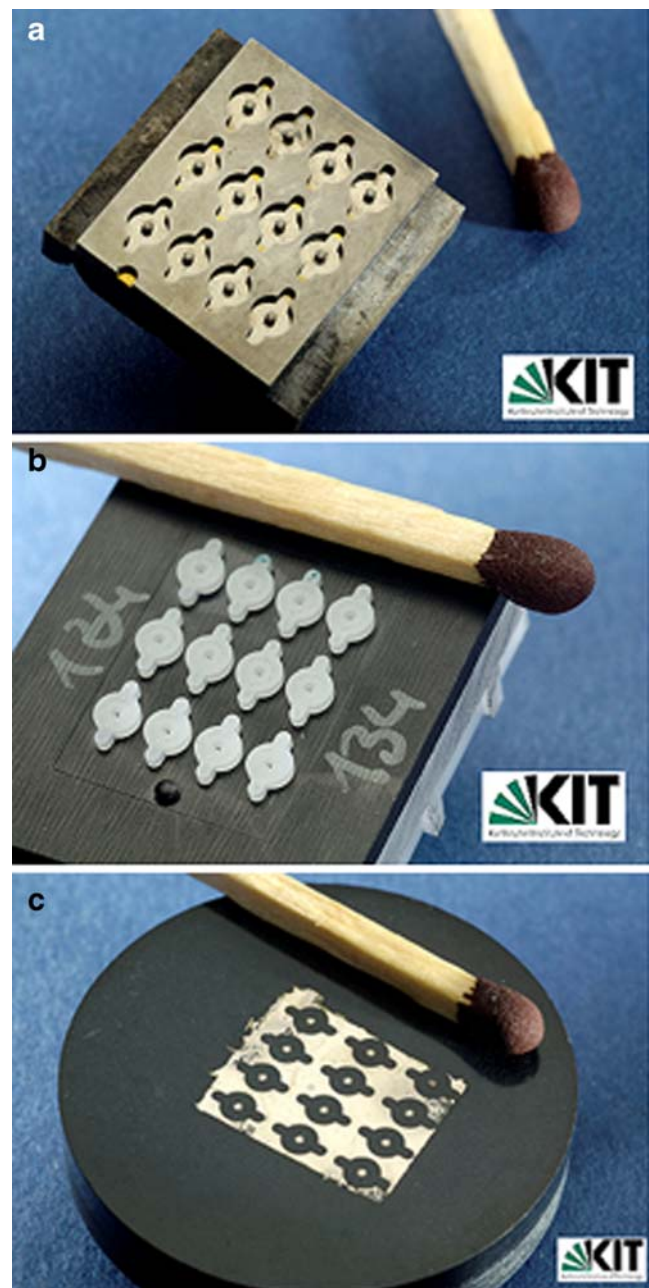


Fig. 3 a–c The process chain explained by pictures: LIGA-made mould insert with 12 microgear wheel cavities (*top*), injection moulded two-component polymer substrate (*middle*), and final electroplated metal parts (*bottom*)

The percentage of the carbon black was set to 3, 5, 10 and 12 wt.% for the processing on the ZSK25WLE in order to analyse the percolation threshold. On the ZKS26MC, material containing 12 and 15 wt.% carbon black was used to see differences in the behaviour with regard to the injection moulding parameters. First, a standard part with different thicknesses was analysed using the ring electrode test according to DIN VDE 0303 (Fig. 4).

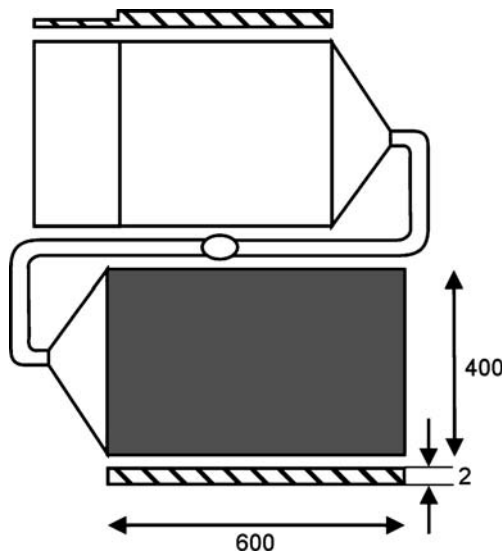


Fig. 4 Specimen for ring electrode test according to DIN VDE 0303

By analysing surface resistance for the different amounts of carbon black, the percolation threshold of the parts was determined. Figure 5 shows the measured values of the thicker plate injected with 40 mm/s. The percolation threshold is reached at between 5 and 10 wt.% of carbon black.

In order to understand the correlation between the surface resistance and the injection speed, resistance was measured against the motion speed of the screw (Fig. 6).

In amorphous thermoplastics, the injection speed has a high dependency on the accomplished conductivity of the produced part. To understand this behaviour for semicrystalline polymers like PA 12 in detail, this dependency was investigated by using material with 10, 12 and 15 wt.% of carbon black. By increasing the percentage of the conductive filler, the dependency on the injection moulding

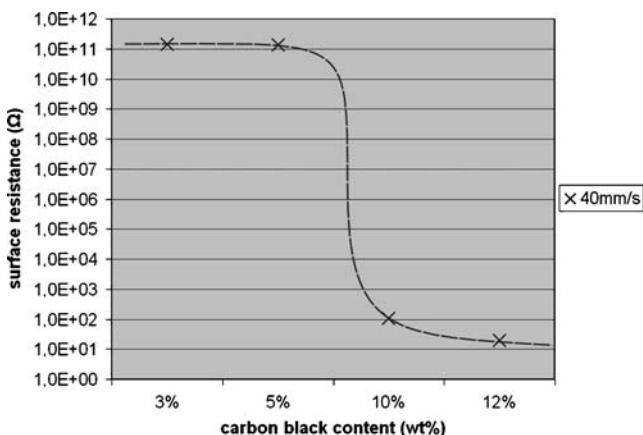


Fig. 5 Percolation threshold of Grilamid L16A mixed with Ketjenblack EC 600JD

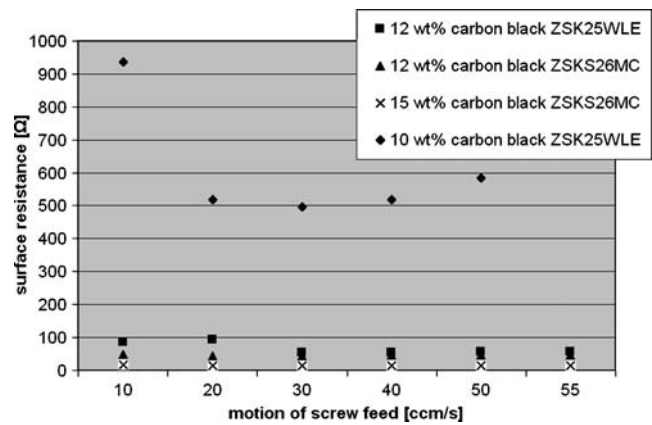


Fig. 6 Interdependency between injection speed as determined by screw forward rate and surface resistance

parameters could be decreased but not completely avoided (Fig. 6).

In order to see differences between the machining of the material, the same material was produced using the ZSK25WLE (20 kW drive rating) and the ZSKS26MC (31 kW drive rating). The difference between these machines is the applicable torque, which reaches higher values for the ZSKS26MC. Going into detail, it became obvious that the influence of the injection moulding speed can be reduced by using the machine with more torque. The effect on the material resistance is most probably caused by the higher degree of de-agglomeration obtained by operating an extruder with increased torques and thus higher shear rates.

TEM analyses will probably show this effect in more detail. To disclose this effect of the influence of the injection moulding speed and the conductivity, the process was analysed using Moldflow®. The simulation showed that an increase of the shear stress leads to a decrease of the conductivity (Fig. 7a, b).

An explanation can only be given by the shear-induced migration of the conductive filler during the process. The detailed analysis of this behaviour is quite difficult since the carbon black eliminates most of the existing analysing possibilities. Inquiries of the University of Akron and the IKV Aachen showed particle migration of carbon black in PP and PS during injection moulding caused by shear stress.

For the project presented, it was important to produce homogeneous conductive templates in the first step. This was accomplished by using the assumptions given and the produced material. By producing thicker base plates and applying shear reducing parameters—both measures to reduce the shear strain—base plates could be produced with 15–25 Ω, which was not possible before. Results of an electroplating test of such base plates are shown in Fig. 8.

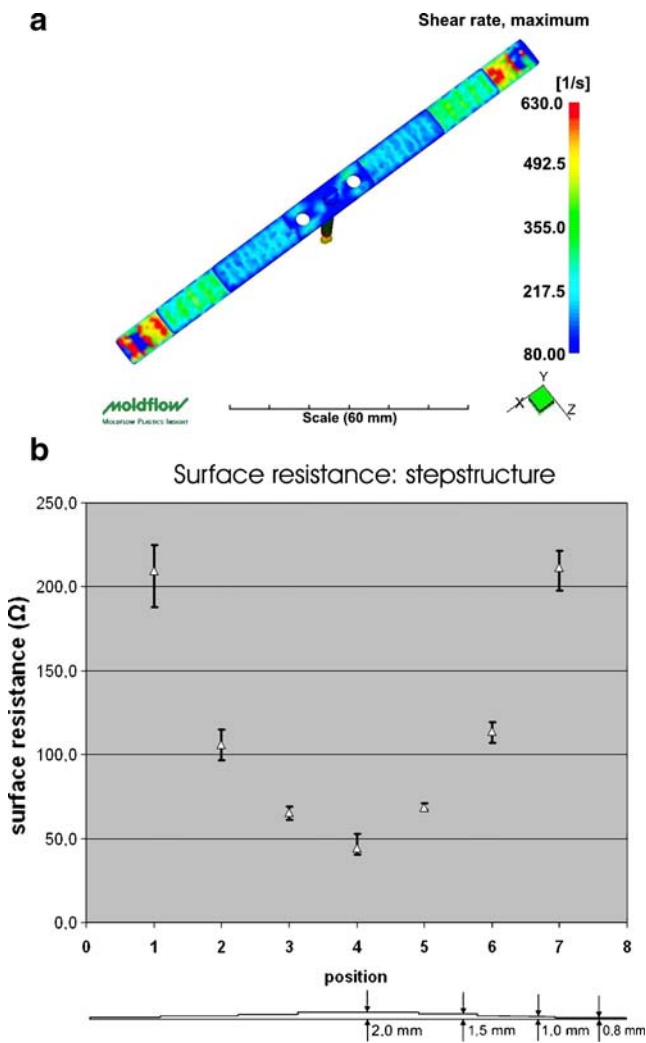


Fig. 7 a, b Simulation analysis for comparison of calculated shear rate (*top*) and surface resistivity measured (*bottom*)



Fig. 8 Electroplating test on base plate of almost homogeneous electrical conductivity distribution (homogeneity gradient of $\pm 8\Omega$)

By using the developed material sets for both injection moulding and electroplating, different microgear wheels could be fabricated as demonstrated by Fig. 9a, b.

3 Micro two-component powder injection moulding

Mounting and assembly are crucial issues in microfabrication but, fortunately, their criticality can be reduced by multi-component injection moulding [2]. Besides this, MicroPIM development aims at extending this shaping technology towards the integration of two metal or ceramic materials with different physical properties, here as different electrical resistivities, in a single piece [3, 4].

To facilitate the injection moulding of two-component parts, a moulding tool and a special process control had to be developed. The moulding tool allows the utilisation of various different microcavities. Some of these parts are

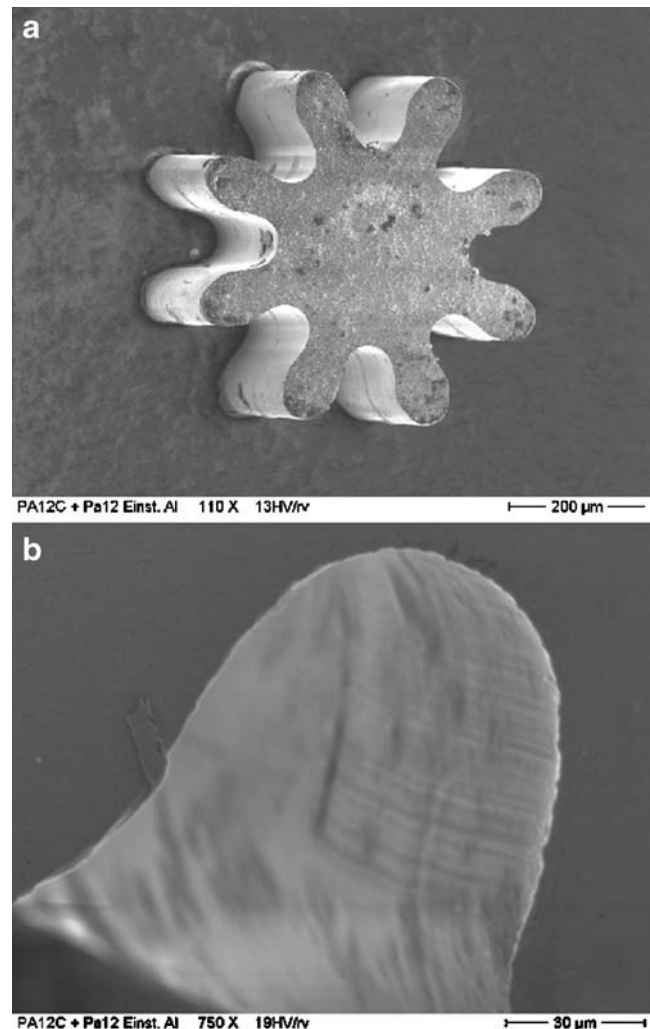


Fig. 9 a, b SEM pictures of microgear wheels produced by improved process chain of 2C injection moulding and electroplating

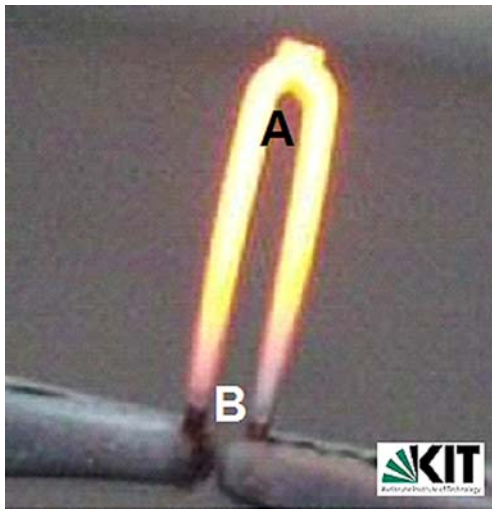


Fig. 10 Glooming test of a sintered two-component heating element (*A* section with low TiN content, *B* sections with high TiN content)

determined for tensile and bending tests so that the bonding strength of the junction between two materials can be investigated.

Being able to evacuate the moulding tool to avoid burners caused by compressed air is an important tool characteristic. Another important feature of the tool is the integrated partition dividing the cavity. This allows a sequential injection of both components: After the injection of the first material, the partition can be pulled, and the second component is injected onto the stationary front. It is also possible to work with an open partition. In this case, the position of the junction can be adjusted by varying injection speed.

The manufacturing of u-shaped heater elements as shown in Fig. 10 is one example for 2C micropowder injection moulding. It also presents a possible application. Here, a ceramic mixture consisting of Al_2O_3 and TiN was used. Due to the relatively high electrical conductivity of TiN, the specific resistivity of the whole system can be adjusted by varying the mixing ratio [5].

In case of the u-shaped heater elements, the curved section was moulded using a mixture with a low TiN content, whereas in the two straight sections, the Al_2O_3

content was reduced. After sintering, a two-material part with a significant gradient in electrical conductivity was obtained. If the whole sintered part is set under current, a glooming effect typical for resistivity heaters can be detected (Fig. 10).

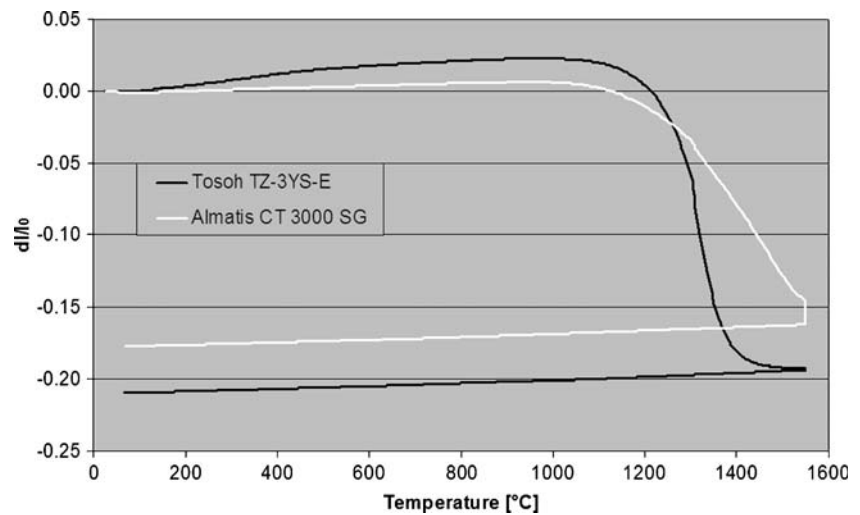
An advanced application of two-component micropowder injection moulding is the production of shaft-to-collar connections. Using these techniques, two different features can be realised: a fixed or a moveable connection, depending on the materials and the applied process. In principle, there are several processes, which influence the formation of fixed or loose connections. First of all, the behaviour of the feedstocks has to be regarded. Depending not only on the composition of the feedstocks but also on the techniques of the injection tool, intermixing of the components might be possible. This is advantageous for realising fixed connections. For the production of moveable connections, however, intermixing should be avoided, either by the injection parameters and/or by using incompatible feedstocks. Binder systems with different polarities show the tendency to de-wet mutually. Thus, intermixing of the two components might be avoided.

A more important role appertains to the thermal processes, especially sintering. By heating the samples up to sinter temperature, two superimposing effects occur: thermal expansion and shrinkage. As different ceramic or metal powders are used for two-component MicroPIM, the expansions of the components will diverge during heating. Thus, if the gear wheel of a shaft-to-collar connection shows a higher expansion than the axle, the gear wheel will uncouple from the axle, which is advantageous for realising movable connections. The converse case is beneficial for fixed connections, but very different thermal expansions may cause stresses inside the assembly and, as worst, even a breakdown. The total changes of the dimensions of axle and gear wheel have to be considered for the selection of suitable ceramic powders, for adjustment of the feedstock composition (especially powder load) and for the process control. Preliminary investigations on possible ceramic powders and feedstocks have shown that applicable materials and material combinations are principally available [9]. Based on these results, both fixed and moveable

Table 1 Powders used for the trials on 2C MicroPIM (powder loading)

| | Shaft (Al_2O_3) | Gear wheel (ZrO_2) |
|------------------|-----------------------------------|-------------------------------|
| Fixed junction | Approx. 50 vol.% each | |
| | Almatis CT 3000 SG | Tosoh TZ-3YS-E |
| | RC UFX-DBM | Unitec PYT05.0-005H |
| Movable junction | 45–50 vol.% | 50–55 vol.% |
| | RC UFX-DBM | Unitec PYT05.0-005H |
| | Almatis CT 3000 SG | Tosoh TZ-3YS-E |

Fig. 11 Sinter curves of alumina (Almatis CT 3000 SG) and zirconia (Tosoh TZ-3YS-E)



shaft-to-collar connections have been realised. To enable both modifications, material selection and process parameters have been adapted to the specific requirements. Especially, the different properties of the ceramic powders can be utilised. In this context, the particle size or rather the specific surfaces of the powders are particularly important since the specific surface affects the sinter activity. Table 1 provides an overview on the powders used.

For the fixed junction, for example, a ZrO_2 powder was chosen (namely TZ-3YS-E from Tosoh Corporation) that shows an early sintering start and accelerated shrinkage. As second component, the alumina powder CT 3000 SG from Almatis GmbH was used. During the sintering process, this powder shows a slow shrinkage rate in comparison to Tosoh TZ-3-YS-E; thus, both materials start to sinter at almost the same temperature (Fig. 11). In addition, CT 3000 SG has a lower total degree of shrinkage. Thus, the gear wheel could shrink on the axle.

With respect to the sinter parameters, the heating rate was decreased after reaching 1,300°C, and the maximum

temperature of about 1,550°C was sustained for 2 h. Thereby, the concentration changes by diffusion will be enhanced according to Fick's laws. Mutual diffusion involves enrichment of the components like ZrO_2 , Y_2O_3 and Al_2O_3 on the opposite side. By increasing time and/or temperature, diffusion is enhanced, and the components will diffuse deeper into the opposite material. Therefore, the bonding of the components at the interface can be improved due to the time extension of the sintering process. The degrees of shrinkage were adjusted by varying the powder load of the Al_2O_3 and ZrO_2 feedstocks. Thus, for forming the axle, the feedstock consisted of Al_2O_3 , polyethylene, paraffin and stearic acid. For the injection moulding of the gear wheel, a feedstock consisting of ZrO_2 , polyethylene, paraffin and stearic acid was used. An example of a sintered two-component assembly is shown in Fig. 12. SEM observations confirmed that the components are adhesively joined after sintering (Fig. 13).

For the successful realisation of movable connections, other powders and parameters have to be adopted as, for example, described in [10] for macroscopic applications. In contrast to fixed junctions, the designated zirconia powder



Fig. 12 Shaft-to-collar connection after sintering (fixed junction)

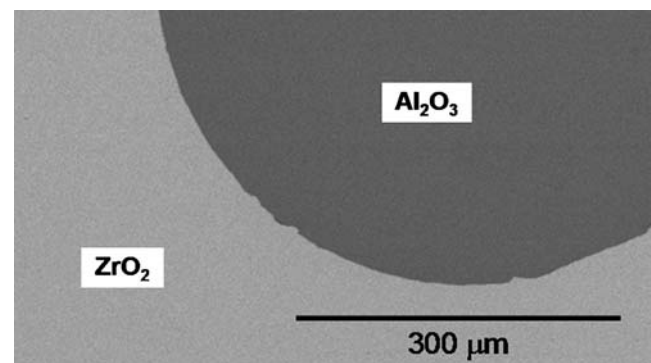


Fig. 13 Interface of a fixed shaft-to-collar connection after sintering

(PYT05.0-005H from Unitec Ceramics Limited) for movable junctions has decelerated shrinkage dynamics. In comparison to the selected alumina powder, this ZrO_2 powder shrinks at a higher temperature and with a reduced shrinkage rate. Hence, the axle will begin to shrink earlier and may detach from the gear wheel. In order to avoid any bounding at the interface, the above-mentioned diffusion processes should be reduced as far as possible. The zirconia powder chosen for realising movable connections (PYT05.0-005H from Unitec Ceramics) has a significant lower shrinkage rate than Tosoh TZ-3-YS-E and also a lower linear change resulting from sintering. In comparison to the alumina powder RC UFX-DBM from Baikowski Malakoff, which is designed as component for the axle of the movable junction, PYT05.0-005H shows a relatively late initiation of shrinkage. The dynamics of shrinkage of both powders are quite similar. Therefore, the change in diameter of the gear wheel at a given temperature is lower than that of the axle, possibly resulting in the formation of a gap between both components. Additionally, the degree of shrinkage of both components can be controlled by adjusting the powder load. The binder consists of the same organic components as used for producing fixed connections.

So far, movable connections can be produced by interrupting the sinter process and applying mechanical movements. In doing so, the rotatability of the gear wheel can be maintained when the sinter process will be continued. It was shown that the connection is movable at a temperature at which the organic binder components are already decomposed. Therefore, the characteristics of the powders and the sinter parameters predominantly influence the circumstances for forming a fixed or a loose connection.

4 Outlook

It was shown that multi-component microinjection moulding enables both the realisation of new material combinations as well as the economic enhancement by, e.g., reducing assembly efforts.

Concerning galvanofarming on partially conductive substrates, the next step is the set-up of an automatic 2C process for pre-series production. Additionally, variation of the second component and improvement of the injection moulding parameters of this process step will be carried out. In order to analyse the forces during de-moulding, it is planned to create a simulation of this process using Ansys. This will show the effects of the machining and will provide a tool to analyse structures giving an idea if these structures can be built or not. As base plates with nearly homogeneous conductivity distribution could be achieved further material optimisation is not predominant.

Regarding two-component micropowder injection moulding, a promising technological level has been reached but there is, of course, a lot of research work to be done, especially concerning the adjustment of feedstock formulations and the optimisation of sintering procedures. Further materials will be studied for their suitability for 2C MicroPIM, with a focus on producing movable assemblies. Even this study has shown that the result of forming movable connections depends primarily on the powder characteristics rather than on binder systems. Studies on feedstocks that are different from those used so far are scheduled. Objectives will be investigations on new incompatible binder systems in order to avoid intermixing as the combination of incompatible feedstocks show mutual de-wetting. Additionally, further ceramic powders which sinter at lower temperatures will be tested with the objective of clarifying if lower sinter temperatures can reduce the effects of diffusion and phase reactions at the interface. The aim of the oncoming studies is the development of movable connections in a closed manufacturing process without an intermediate step at which mechanical relative movements have to be applied.

Acknowledgements Parts of this study are carried out within the Collaborative Research Area (Sonderforschungsbereich) 499 and the Research Group (Forschergruppe) 702. The authors would like to thank the German Research Association (DFG) and the Federal Ministry of Education and Science (BMBF) for the financial support. We also wish to thank our industrial partners and all colleagues at Forschungszentrum Karlsruhe for their helpful support. Also, parts of this work were carried out within the framework of the EC Network of Excellence “Multi-Material Micro Manufacture: Technologies and Applications (4 M)”.

References

1. Piotter V, Holstein N, et al (2002) Methods for large-scale manufacturing of high performance micro parts. Proceedings of 3rd Euspen International Conference Eindhoven, pp. 337–340
2. Michaeli W, Opfermann D (2005) Micro assembly injection moulding—potential application in medical science. Proceedings of 4M 2005 Conference; ISBN 0-080-44879-8; Elsevier, pp. 79–82
3. Oerlygsson G, Piotter V, Finnah G, Ruprecht R, Hausselt J (2003) Two-component ceramic parts by micro powder injection moulding. Proceedings of the Euro PM 2003 Conference Valencia, Spain, pp. 149–154
4. Piotter V, Finnah G, Zeep B, Ruprecht R, Hausselt J (2007) Metal and ceramic micro components made by powder injection moulding. Mater Sci Forum 534–536:373–376 Trans Tech Publications
5. Piotter V, Oerlygsson G, Ruprecht R, Hausselt J, Nishiyabu K (2004) New developments in micro powder injection moulding. Proceedings of PM 2004 Powder Metallurgy World Congress 1:473–480 ISBN 1899072 15 2
6. Imgrund P, Rota A (2003) Multifunctional microparts by metal injection molding. Proc. MICRO SYSTEM Technologies Int. Conf.; Franzis Verlag GmbH, ISBN 3-7723-7020-9, pp. 218–225

7. Imgrund P, Rota A, Hartwig T, Petzoldt F, Simchi A (2005) Adjustment of materials and sintering processes for MIM of bi-material parts. Proc. Euro PM 2005 Congress & Exhibition, 2.-5. 10. 2005, Prague, Czech Republic—EPMA. Shrewsbury 2:307–312
8. Imgrund P, Rota A, Wiegmann M (2007) Getting better bonding at tiny interfaces. Met Powder Rep 62(3):31–34. doi:[10.1016/S0026-0657\(07\)70064-4](https://doi.org/10.1016/S0026-0657(07)70064-4)
9. Ruh A, Dieckmann A-M, Heldele R, Piötter V, Ruprecht R, Munzinger C, Fleischer J, Haußelt J (2008) Production of two-material micro assemblies by 2-component powder injection molding and sinter-joining. J Microsyst Technol 14:1805–1811. doi:[10.1007/s00542-008-0646-8](https://doi.org/10.1007/s00542-008-0646-8)
10. Maetzig M, Walcher H (2006) Assembly moulding of MIM materials. Proc. Euro PM 2006—Powder Metallurgy Congress & Exhibition 2:43–48