Advanced Micro & Nanosystems
Volume 4

Microengineering of Metals and Ceramics

Part II
Special Replication Techniques, Automation and Properties

Volume Editors
Detlef Löhe and Jürgen Haußelt
Advanced Micro & Nanosystems
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Microengineering of Metals and Ceramics

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Microengineering of Metals and Ceramics

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Special Replication Techniques, Automation and Properties

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Preface

Machines and their design and production have fascinated mankind from the very beginnings of culture. The last decades have shown mechanical contraptions decreasing in size to almost invisible dimensions. The implementation of micromechanics has become not only a technological challenge, but also a necessity for a successful future development of whole industrial branches. Adequate design and replication techniques of micromechanical components as well as a deep knowledge of their properties are indispensable for further progress in this field. At the same time the variety of materials used in micro system technology has increased significantly. Today not only silicon and polymers, but also metals and ceramics are of increasing interest for a large number of applications. In contrast to silicon and polymers, however, which can be structured by technologies well known from microelectronics, metals and ceramics require new forming and structuring techniques for dimensions in the sub millimeter range. In addition, mechanical properties of metallic and ceramic microparts are of special interest because they differ significantly from those measured in macroscopic dimensions.

It is because of these considerations that the German Research Council (Deutsche Forschungsgemeinschaft, DFG) has decided to fund a collaborative research center (SFB 499) with approximately 2 million euros p.a. which tackles the problems arising when trying to design, produce and characterize advanced microstructures made of metals and ceramics.

The two-volume book in hand presents the results of five years of research on micro engineering utilizing metallic and ceramic materials. It comprises the whole process chain from design and modeling of microcomponents along production preparation and two central replication techniques (micro powder injection molding and micro casting) to characterization and quality insurance, the scope encompassing both theoretical and experimental topics. The book is structured roughly according to the project groups which form SFB 499:

The first volume contains sections on design, tooling and replication techniques based on injection molding. The first section focuses on micro-component design including design environment, design flow, modeling and validation as well as on the modeling of micro powder injection molding (cf. chapters 1 to 3).
The second section on *tooling* describes preparatory steps for the production process. The production of mold inserts by micro milling, laser ablation, micro electro discharge machining and techniques based on lithography and electro-forming is investigated, surface treatment methods using shot peening and ultrasonic energy are presented, and optimized mold materials for micro casting are identified (cf. chapters 4 to 9).

The third section on *micro injection molding* (chapters 10 to 12) is concerned with the production itself, focusing on the actual molding processes. Following an introductory chapter on general aspects of micro injection molding, micro injection molding of metals and ceramics including the challenging process steps of debinding and sintering (cf. chapters 11 and 12) are described.

The second volume comprises three sections on replication techniques other than injection molding, on automation and on properties of the components produced.

The section on special replication techniques focuses on microcasting (chapter 13) and electroforming (chapter 14) of metals and on selected techniques for the manufacturing of ceramic microcomponents (chapter 15).

The second section of volume 2 deals with *automation and quality insurance* and includes chapters on the automation of μPIM, on assembly and on quality insurance and dimensional measuring techniques (chapters 16 to 18).

The last section addresses the *properties* of metals and ceramics and of the components produced. Microstructure and mechanical properties including micromechanical testing under quasi-static and cyclic loading as well as tribology are investigated and numerical wear simulation is performed (cf. chapters 19 to 22).

Working groups concerned with aspects touching all five project sections (e.g. on relationships between manufacturing processes) act as links between the projects. Cooperation within SFB 499 is further enforced by concerted work on a demonstration device consisting of a micro-turbine and a sun-and-planet gearing.

For a comprehensive treatment the chapters which directly deal with the research projects of SFB 499 are supplemented by several important research topics concerning micromechanical components (e.g. laser structuring, lithographic processes, electroforming, assembly) which are not part of the collaborative research center. In these cases guest authors have been asked to contribute. They come from Bremen University (Institut für angewandte Strahltechnik, BIAS), from Braunschweig University (Institut für Werkzeugmaschinen und Fertigungstechnik, IWF) and several scientific institutes of Forschungszentrum Karlsruhe.

By covering most aspects of the design, production, and properties of micromechanical components outside the silicon world, the authors hope to present a useful guide to students and readers looking for a comprehensive overview as a starting point of in-depth research in this field. However, the detailed presentation of latest SFB 499 research results as well as contributions from literature should also be a source of new insights and inspiration for micro-engineering experts from research institutions and industry.

_May 2005, Karlsruhe_  
_Detlef Löhe and Jürgen Haßelt, Volume Editors_
Foreword

We are proud to present the third and fourth volumes of Advanced Micro & Nanosystems (AMN), entitled Microengineering of Metals and Ceramics.

Although microtechnology is often associated with semiconductor cleanroom processes, this is by no means the only means of production available. The processes we associate with traditional mechanical engineering mass production have also been the focus of microtechnologists, with tremendous successes already in place and a huge potential for further progress. Of course, every new technology pairs the development of suitable materials with that of production technology, and in the sub-millimeter range the challenges become immense. Not only must raw materials be produced in particulate form fine enough to reproduce the molds they are formed into, and molds need to be prepared at the correct dimensions and surface quality, but new ideas are needed to make use of machine parts produced in this manner, and new methods to assemble parts into complete systems. In these two volumes you will find a comprehensive treatment of a variety of challenges that arise in the process of producing microparts from metals and ceramics, from materials, testing, production, computer-aided engineering all the way to assembly. We hope that these volumes will inspire the transfer of these fascinating techniques not only to other research groups, but also to industry and so broaden the range of items that can be successfully miniaturized.

Covering recent advances from the world of micro and nanosystems, future AMN issues will either focus on a particular subject, such as CMOS-MEMS and the present twin topical volumes Microengineering of Metals and Ceramics, or be a carefully chosen set of cutting-edge overview and review articles like the first AMN volume on Enabling Techniques for MEMS and Nanodevices.

Looking ahead, we hope to welcome you back, dear reader, to the upcoming fifth member of the AMN series, in which we take a close look at the fascinating field of Micro Process Engineering. The articles will range from the fundamentals and engineering over device conception and simulation to fabrication strategies and techniques, and finally cover application and operational issues. To cover such a wide spectrum, we are very glad to have the support of Dr. Nor-
H. Kockmann from the University of Freiburg, Germany, who will edit this volume.

Henry Baltes, Oliver Brand, Gary K. Fedder, Christofer Hierold, Jan G. Korvink, and Osamu Tabata

Series Editors

May 2005

Zurich, Atlanta, Pittsburgh, Freiburg and Kyoto
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Replication Techniques – Micro Casting, Micro Electro Forming and Further Techniques
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Abstract
Microcasting is a metal forming process based on the well-known lost-wax – lost-mold technology of investment casting. The further development of this technique for casting structures in the range of some tens of micrometers requires special patterns, investments and casting parameters. First, this chapter describes the general casting process, highlighting differences from conventional dental and jewelry casting. Additionally, the parameters of a typical microcasting process are given. Next, some alloys used for microcasting and their chemical compositions, melting and casting temperatures and phase transitions during the solidification process are described in detail. Thereafter, the two basic investments used for microcasting and the influence of the investment on the surface roughness of the cast parts are discussed. Finally, cast microparts are shown and their properties such as microstructure, dimensional accuracy, surface roughness, mechanical properties, smallest achievable structure size and highest obtainable flow length and aspect ratio are presented.

Keywords
investment casting; dental casting; gold base alloy; bronze; CoCrMo alloy
13.1 Introduction

Microcasting is the manufacturing process of small structures in the micrometer range or of larger parts carrying microstructures by using a metal melt which is cast into a microstructured mold. Fields of application are, e.g., instruments for minimal invasive surgery, dental devices and instruments for biotechnology. Additionally, the manufacturing of miniaturized devices for mechanical engineering is a desired outcome.

At present, two different techniques for casting structures in the micrometer range are known: capillary action microcasting and microcasting based on investment casting. The first manufacturing method was developed by Bach et al. [1] and Moehwald et al. [2]. They applied capillary action microcasting for form filling of structures in the range of some micrometers. Similar to die casting, this technique uses a permanent mold which can be opened in order to remove the cast structure. The cavities in the mold are shaped by high-precision grinding [2]. For casting, two different principles to fill these cavities exist: the suction principle and the displacement principle [1]. In the first case the melt is sucked into a specially coated mold by the capillary pressure. In the second case the casting alloy is melted inside the divisible mold and fills the microstruc-
tured cavities owing to the capillary force. Subsequently pressure is applied to the mold to displace the excess melt through the slit. Owing to absorption of the coating during solidification, the casting detaches from the mold’s surface, but at the same time the alloy composition changes slightly compared with the original material. In capillary action microcasting the castable geometries are limited to structures which can be filled by application of capillary forces. Microcasting based on the investment casting technique, which will be discussed in the following, does not suffer from these limitations.

13.2 Investment Casting

Microcasting, also named microprecision casting [3, 4], is generally identified with the investment casting process, a casting technology also known as the lost-wax, lost-mold technique [5, 6]. This forming process excels in near net shape manufacturing and is an established technology with great freedom in design [7]. It offers the chance to produce very complicated formed parts in metal even with undercuts. Another advantage of the investment casting process over other shaping processes is the rapidity of the casting procedure itself and the low loss of material due to the possibility of recycling the runners and sprues. However, the process cannot be fully automated, so it is best suited for small and medium series and for parts with highly complex shape. This is the reason why investment casting has, in addition to technical application, a high relevance for jewelry and dental casting. For both applications, precise manufacturing is achieved [8–13], especially by using precious alloys. For jewelry and dental casting, the sizes of the produced parts are in the millimeter up to the centimeter range with structural details in the millimeter and submillimeter ranges [1]. Further development and improvement of these techniques allowed the casting of microparts with structural details even in the micrometer range, which was confirmed by the replication of small-scale LIGA structures (see Section 13.2.1.1) with high accuracy [15]. The new microtechnology, derived from the conventional production process, requires different pattern materials, other investments, special alloys and other casting parameters compared with the standard investment casting process. Additionally, microcast parts cannot be machined mechanically after manufacturing. Sand blasting, as applied in dental and jewelry casting, cannot be used to remove residue of the investment, nor can surfaces be polished to increase their quality. Sand blasting would reduce the sharpness of the edges and therefore influence the accuracy of the part, and polishing is not possible because of their small size. Therefore, precious alloys are particularly suitable for microcasting, because here the investment can be removed chemically from the cast metal part using hydrofluoric acid without influencing the cast part. Recent progress in the development of investments, however, opened the possibility of casting microstructures with bronze as a non-precious alloy [17]. In the following, the typical microcasting process will
be illustrated first. Later, details on the alloys and investments and variables specifically influencing this process are given.

13.2.1 General Process

The microcasting process, which is described in Section 13.2.1.1, has enormous potential in manufacturing microparts of high quality without the need for further processing, as opposed to the dental and jewelry casting technique. The patterns used in the microcasting process (see Section 13.2.1.2) guarantee a higher strength and are thus of advantage when assembling microstructures. Sections 13.2.1.3–13.2.1.5 give the basics on melting, casting and solidification.

13.2.1.1 Process Description

The microcasting process itself is based on the lost-wax, lost-mold technique. It is widely comparable to casting of dental protheses or jewelry [18]. In contrast to the wax patterns used there, microtechnology mostly works with injection-molded plastic patterns which have much higher mechanical strength. The improved mechanical properties permit easier handling and assembling of the pattern during the manufacturing process.

The shaping of the microcavities in the mold insert, used for injection molding, can be achieved by several methods. We applied mainly the technique of micromilling [19–21], which is a further development and improvement of the standard milling process towards miniaturized manufacturing (see Chapter 4), but in some cases also microelectro discharge machining [22, 23]. More details on the latter process can be found in Chapter 7. Other ways for the production of microstructured mold inserts are the laser technique [24–26] and the LIGA process [28–30]. The LIGA process, which is described in detail in Chapter 8, includes a lithographic and a galvanic process and is beneficial for microreplication owing to the very good surface quality of the mold inserts and the high potential of generating minimum structures. However, in contrast to the milling process, which allows the production of free form faces and real 3D structures, the LIGA technique is limited to 2.5-dimensional structures because of their necessarily vertical walls.

The microcasting process requires a lost plastic pattern to be mounted on a gate and feeding system made of wax (Fig. 13-1). The assembly is then completely embedded in a ceramic slurry. This process differs from the technical investment casting process where normally a ceramic shell is built-up by repeatedly dipping the pattern in a ceramic slurry followed by stuccoing. After drying, the ceramic is sintered, resulting in a ceramic mold with high mechanical strength. Simultaneously, the plastic melts during the burning process and is pyrolyzed.

In order to fill the mold with the metallic melt, either the vacuum pressure casting or the centrifugal casting technique can be used. In the first case, the
ceramic investment mold is evacuated, then the melt is poured into the mold, filling the cavity only due to gravitational forces. After that, pressure is applied to the melt. In the second case, the centrifugal force is used for form filling. Both techniques will be explained later in detail. After solidification, the investment is mechanically removed without destroying or influencing the cast surface. Depending on the casting alloy and the investment material, additional chemical cleaning processes may be sometimes necessary. Finally, the single parts are separated from the runner system. Unlike dental or jewelry casting, there is no further treatment such as grinding or polishing of the cast surface. This is due to the much smaller geometry and inapproachability of details on cast microparts and also to the necessity for high contour precision without any rounding of the edges.

Fig. 13-2 shows the most important replication steps for the example of a microturbine plate. On the left, the mold insert for injection molding of the pattern, the negative form, is shown. It is made by micromilling in brass. In the
center is the injection-molded plastic turbine plate made of PMMA [poly(methyl methacrylate)]. This is the positive pattern required for microcasting. The plastic pattern is replicated by the investment and forms the negative mold. The third replication process – the real casting – yields the desired positive form, in this case made of a gold base alloy (right). It is worth mentioning that the replication is so precise that even scratches with depths of a few micrometers in the mold insert for injection molding are perfectly replicated on the cast part.

The investment casting procedure for manufacturing microparts is influenced by many different parameters. The most important ones are the casting alloy, the ceramic investment, the preheating temperature of the mold and the casting pressure. The molten casting alloys must exhibit a low viscosity in order to fill the small microstructures completely. Additionally, a minor tendency for oxidation is of high interest. For the ceramic investment, the most important factors are the ability for high-precision replication, an expansion behavior adjusted to the alloy used and a low surface roughness. The preheating temperature and the pressure influence the entire form filling process [31] and, as a consequence, the achievable grain size and the resulting mechanical properties.

13.2.1.2 Pattern Design

For cost-effective casting, the assembly of single patterns in form of a so-called tree is necessary, whereas the design rules of good castability should be considered to allow homogeneous form filling of all mounted structures. In microcasting, single polymer patterns are normally fixed with wax. As an example, Fig. 13-3a shows a pattern with 15 injection-molded polymer tensile test specimens fixed on a sprue system made of wax. In Fig. 13-3b, the resulting cast part (gold base alloy) can be seen. Single microstructured patterns should be made at least with a small runner owing to the difficult handling of the small parts. Forming of complete plastic or wax assemblies is even better. Especially patterns which are injection-molded on a substrate plate proved to be advantageous because the substrate plate can be used as feeder. However, the melt flow
in the plate is not easy to control. In industry, similar problems are solved by simulating the casting process. For microdimensions such specialized tools are not yet available.

Like patterns for macrocasting, patterns for microcasting should be constructed according to the well-known design rules for casting [32–35]. In order to produce faultless patterns, different wall thicknesses and sharp edges should be avoided [36]. Furthermore, the form filling process is of great importance. The cross-sectional thickness of the sprue system should increase in the direction of the sprue bottom, because solidification must begin in the microparts and end in the bottom of the tree. On the one hand, this design is beneficial for good form filling; on the other, it helps to avoid shrinkage holes in the casting. This design rule, however, does not normally cause any problems in microcasting, because the parts are generally distinctly smaller than the feeder and runner system. Nonetheless, the heat capacity of the mold should also be taken into account because the compact molds used for microcasting show a comparatively high heat capacity. This results in the inner part of the massive form being still hot while the surface cools rapidly after the mold has been taken out of the furnace. Therefore, thin-walled parts should be positioned in the outer and thick-walled parts in the inner area of the mold. The melt will then remain liquid in the thick-walled parts for the longest time so that they can work as feeder for the thinner parts. As mentioned before, an adequate sprue system is necessary in order to avoid shrinkage holes in the thick-walled parts. More detailed information on the sprue design is given in the literature [37].

A special aspect in microcasting is the flow behavior in very fine channels. Owing to the much higher surface to volume ratio in microchannels compared with macrostructures and the distinct influence of surface roughness, the occurrence of turbulent flow needs to be taken into account. Another aspect is the extremely high cooling rate and therefore extremely fast solidification in the small structures, which hinders form filling much more than in macrostructures. This aspect will be discussed in more detail in Section 13.5.6.

13.2.1.3 Melting

For casting in different atmospheres, various set-ups are available. Some casting machines work with vacuum, some with air and others with an inert gas atmosphere. Also the furnaces can vary. There is electrical resistant heating, heating by an open flame, induction heating and melting by an arc furnace.

For resistance heating, the heat is produced by a heat winding which encloses the crucible. The heat winding can be made, for example, of platinum–rhodium. The method is used for alloys with casting temperatures up to about 1300 °C. Such set-ups with resistance heating are predominantly used for casting precious metals.

For the open flame technique, the metal is melted by a propane–oxygen flame in a ceramic crucible. The method is limited to relatively small amounts of metal and is especially used in dental casting workshops for heating high-melting
alloys to temperatures between 1300 and 1500 °C. Here, the use of a reducing flame is of great importance in order to eliminate oxides in the melt. The open flame technique requires good craftsmanship, but given this, good casting results can be achieved.

Induction heated casting machines allow for a higher automation level. They are now commonly used [38] in dental and jewelry casting because they exploit the widest range of casting atmospheres and temperatures. In induction heated equipment the metal is melted in a crucible surrounded by a water-cooled copper coil. An alternating current excites a magnetic field inducing eddy currents in the metal charge. This results in strong Joule heating due to the resistance of the charge carriers in the metal. The amount of energy injected depends on the alloy and the frequency (of the furnace). Modern equipment works with high frequencies in the region of 100 kHz. A benefit of this method is the very high melting rate. Owing to the direct injection of energy, higher melting alloys requiring casting temperatures above 1300 °C can be cast, compared with electrical resistance furnaces which are limited to 1300 °C in general. The induced eddy currents result in a strong convection in the melt. Hence good mixing and homogenization are achieved.

For melting with an arc flame, a pure argon atmosphere is necessary because the gas atoms work as charge carriers for the current flow. For the same reason, the metal to be molten must have electric contact with the crucible, which is normally connected as anode. The arc is then ignited between a tungsten cathode above the crucible and the crucible itself. Arc furnaces are very powerful and are also able to melt higher melting metals such as CoCrMo alloys or titanium.

The melting crucibles are made of ceramic or graphite. At high temperatures the graphite crucibles produce a reducing CO atmosphere as a result of the reaction of the carbon with the oxygen in the air. This is especially beneficial for precious metals because the melt is protected against oxidation. On the other hand, graphite crucibles tend to react with the melt so that for carbon-sensitive alloys ceramic crucibles are used normally. For titanium alloys, however, graphite crucibles are used although titanium is known to be a strong carbide former. In this case, a thin titanium carbide layer is formed in the crucible during the first melting process, which protects the melt during the following uses against reaction with the graphite crucible.

13.2.1.4 Casting
Metals to be processed by microcasting must have sufficient castability. The term embraces properties such as flowability and form filling ability, little contraction and shrinkage, reduced segregation, low porosity and shrinkage cavitation, little hot crack susceptibility, high surface quality and good mechanical properties. A metal is considered castable if the mentioned properties can be sufficiently achieved by using a given casting method. Based on the criteria form filling, surface quality, microstructure and dimensional accuracy, the cast-
13.2 Investment Casting

13.2.1.5 Solidification

The molten metal is poured into a preheated form distinctly cooler than the melt. The solidification starts with nucleation and crystal growth at the cooler mold wall [43]. At the same time, the volume of the melt decreases owing to the normal shrinkage process, which may then cause casting defects [39]. Therefore, it is important that the cast part solidifies first while the metal in the sprue still remains liquid. Another important aspect is the changing of the chemical composition during the solidification due to segregation. This segregation can occur in the center of cast blocks because companion elements and inclusions are pushed aside by the solidification front and accumulate in the rest of the melt. Graduated microsegregation inside dendrites or in general inside one phase, formed during solidification, also known as coring [43], is found in alloys which show a solidification interval. In this case, a difference in alloy composition between the center and the extremities of dendrite arms occurs owing to an enrichment of one element in the forming crystals at the expense of an impoverishment of the same element in the liquid. Alloys are prone to coring if the solidification is too fast to reach an equilibrium state according to the phase diagram [44]. The chemical composition can be homogenized by a subsequent long heat treatment at relatively high temperature.

13.2.2 Vacuum Pressure Casting

For vacuum pressure casting of microparts, dental casting machines can be used. Fig. 13-4 shows a scheme of the process. The metal is melted in the crucible located in the center of a heating winding. On top of the crucible the open mold is fixed upside down. After evacuation, the machine turns itself upside down (Fig. 13-4, right). As a result, the melt flows into the mold by gravity.
Complete form filling even of small cavities is achieved by subsequent application of pressure to the melt. Fig. 13-5 shows a view in the opened vacuum pressure casting machine Prestomat® from Degussa Dental GmbH. Vacuum pressure casting machines typically work at pressures of 3.5–4 bar, which is sufficient for the form filling of parts in the millimeter range. Depending on the special geometry of the microparts and the feeding system, this pressure may even be adequate for the casting of structures in the micrometer range. However, if extremely high aspect ratios are to be cast, a higher pressure is necessary. Calculations of the form filling behavior [45] show that the pressure which is necessary for the melt to enter an extremely small pinhole increases hyperbolically with decreasing radius. Neglecting several actual influences on the casting procedure, it was found that for fibers with a diameter of 1 μm a pressure of 20 bar is necessary to overcome the negative capillary forces which hinder the melt entering a small hole owing to the bad wetting behavior of the melt on