

## Development and Prototyping of a Ceramic Micro Turbine

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### Abstract

Within the framework of the Collaborative Research Center 499 (SFB 499) of the German Research Foundation (DFG), a zirconia micro turbine was chosen as a demonstrating tool to enhance the interaction of the participating workgroups. This case study describes the evolution of the demonstrator and experiences gained with the design and the manufacturing of a micro device. Although it was not the aim of this basic research project to develop a commercial product, the experiences can be useful for improving the performance of industrial product development processes for ceramic micro devices.

The various parts of the zirconia micro turbine were prepared by a Rapid Prototyping Process Chain (RPPC) that allows for a fast and inexpensive manufacturing of ceramic parts with details down to the micron range. A first design concept was made to demonstrate mainly the shaping feasibility of the process in the micro range. However, some features affected the performance due to their low strength. Thus, a modified design was optimized for power output and durability. Models of the design parts were produced either in polymer by a Rapid Prototyping method or as metallic models by micro machining. After some process optimizations, dense and homogenous ceramic micro parts could be replicated from the models. Currently, an assembling process takes place, preparing the micro turbine for performance tests.

Keywords: micro component, prototyping, micro machining, LPIM, zirconia

### Introduction

In 2000, the Collaborative Research Center 499 (Sonderforschungsbereich 499, SFB 499) "Development, production and quality control for molded micro components made of metallic and ceramic materials" was established by the German Research Foundation (Deutsche Forschungsgemeinschaft, DFG). Participating research institutes are from the University Karlsruhe (TH), the University Freiburg and from the Research Center Karlsruhe (Forschungszentrum Karlsruhe) [1]. It is the aim of the SFB 499 to work out the scientific basics for the development of a continuous and stable process chain for the mass production of molded micro components made of metallic and ceramic materials. This includes the construction of micro components, the manufacturing and an attendant quality management. The core of the SFB 499 consists in the production techniques of powder injection molding (PIM) that can be used for the manufacturing of complex three-dimensional metallic or ceramic micro components, and the micro casting, exclusively used for metallic micro components.

A demonstrator system was defined to act as a link between the individual projects of the SFB. Although it was aspired to build a functioning system, one always has to bear in mind that it is not the mission of the project to develop a commercial product with competitive costs and optimized functionality. The purpose of the demonstrator was rather to represent a physical interface which allows to optimize the cross-linking between workgroups, to integrate a variety of manufacturing challenges into a single system, and to demonstrate the problems which arise from assembling a complex system instead of manufacturing only individual components. With the help of the demonstrator it was possible to detect restrictions and to cross frontiers in the micro fabrication processes for ceramics and for metals.

Various demonstrator systems were used during the runtime of the SFB. One of them is a micro turbine that drives a planetary gear set. This paper is focused only on the micro turbine section. It describes experiences which were gained with the design and the manufacturing of the turbine

components by a prototyping method and the assembly of the components to the device. Restrictions and problems which were identified in the manufacturing and handling of a first design concept lead to a design evolution and to progresses in the manufacturing processes.

### Design of the micro turbine demonstrator

Designing micro parts is very much different from macroscopic design tasks. From the macroscopic point of view, the detailed design is usually one of the last steps. The design of micro parts deals from the beginning with the question what is possible with available manufacturing methods. Even more than in other design tasks the limitation and restrictions for the design possibilities come from downstream process steps [2,3]. Therefore, it is important to stay in close contact to the other process disciplines and to start testing and simulating very early. First concepts are needed to validate the details in the design of the parts.

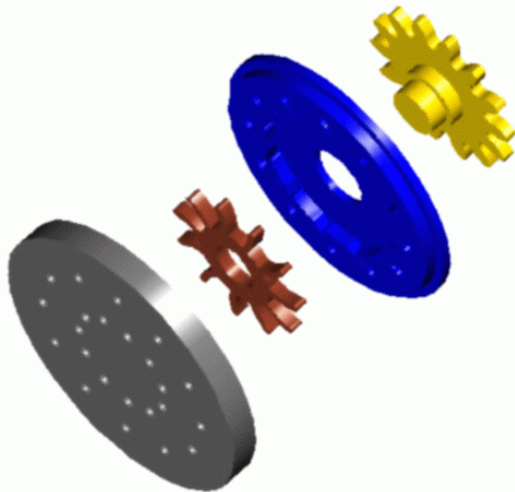


Fig. 1: Exploded assembly drawing of the micro turbine demonstrator device consisting of perforated plate, rotor, nozzle plate and a gear wheel with integrated axis.

Design guidelines that might support the development process of the fluidic components of a turbine are generally known for large systems, but it is not certain that they will be valid for the aspired size range. For that reason the design is mainly based on experience gained in the macro range. Another aspect that influences the design is the specific purpose of the demonstrator. The integration of demanding features was necessary to reveal manufacturing limits and to demonstrate deficits in the practicability of assembling strategies.

Fig. 1 shows a design scheme with the demonstrator components. The system consists of a perforated plate with media inlets and outlets, the

rotor, nozzle plate and a gear wheel for power output. All rotating components should be fixed on a central axis. An outer diameter of 4 mm was intended for the final, air-driven system.

Two concepts of the demonstrator device are presented. In concept 1 especially the micro dimensions of the rotor wings and nozzle channels were challenging for the molding process. Rotor, gear wheel and axis, which have been manufactured separately, were designed for assembly by press fit. In concept 2 the assembling effort was reduced by the combination of gear wheel and axis and by a form fit connection of the rotor. Additionally, the rotor wings were enlarged in width and height for improved strength and for higher torque output. In this case the high aspect ratios (ratio of height/depth to width) of the rotor wings and nozzle channels were found to be a challenge.

### Ceramic injection molding

Ceramic injection molding (CIM) has established itself as a standard process for the manufacturing of complex micro patterned ceramic parts. On the equipment side, the process is based on machines similar to those used in conventional injection molding which, however, had to be extensively modified and upgraded in terms of tool design and process control, to accommodate the specific requirements of micro devices and feedstock properties [4]. There are two variants of ceramics injection molding, the widely used high-pressure injection molding (HPIM) process [5] and the less common low-pressure injection molding (LPIM) process. While the potential of high-pressure injection molding lies in cost-effective mass production, low-pressure injection molding is predestined for the fast manufacture of small production lots [6,7]. One reason for the positioning of HPIM for mass production are the high costs associated with the manufacturing of the molds. This makes the process only profitable when a large number of parts is molded. On the other side, LPIM can work with simple and inexpensive molds and is even economical for small series. Both variants complement one another in an ideal manner and cover the entire range from one-off prototype to mass production.

A central objective of SFB 499 is the development of a processing technique adapted to the manufacturing of middle and large series. Therefore, HPIM takes up a central position in the project. On the other side, the demonstrator system consists of a large number of individual parts. Manufacturing the complete demonstrator by HPIM would produce large costs for the required molds. Thus, it was decided to limit the application of HPIM to the molding of specific parts, mainly for the planetary gear set, and to establish a prototyping project based on LPIM for the other parts, including the manufacturing of the complete micro turbine.

### Prototyping of ceramic micro parts

During the last 20 years a variety of different Rapid Prototyping (RP) or Solid Freeform Fabrication (SFF) methods for the fast production of models and prototypes were developed in the macro world. They are mainly generative methods, like stereolithography or laser sintering, enabling the production of three-dimensional parts directly from the 3-D CAD data. Originally developed for polymer materials, meanwhile, numerous methods also exist for the production of large ceramic models [8,9].

Yet the Rapid Prototyping of micro parts is still limited with regard to accuracy and materials variety. Extremely fine details have only been realized with stereolithographic methods which allow the manufacturing of polymer parts with a resolution of a few micrometers [10]. The principle feasibility of expanding RP methods to the manufacturing of small ceramic parts was already demonstrated on a lab scale for stereolithography or ink jet printing [11,12], but difficulties in the preparation of suited dispersions limited the applicability to a few materials only, and the missing availability of commercial equipment prevents a wider proliferation of the technique.

The current restrictions of RP methods in the micro range can be overcome by a Rapid Prototyping Process Chain (RPPC), where a primary model is first manufactured from a polymer or metal material and then replicated into the requested ceramic material by low-pressure injection molding (Fig. 2) [13,14]. Micro stereolithography is particularly suited for the fabrication of a micro patterned polymer model. Furthermore, primary models can also be produced by micromechanical machining of polymers or metals which is described later in this paper. The second step of the process chain is the fabrication of a mold suited for LPIM.

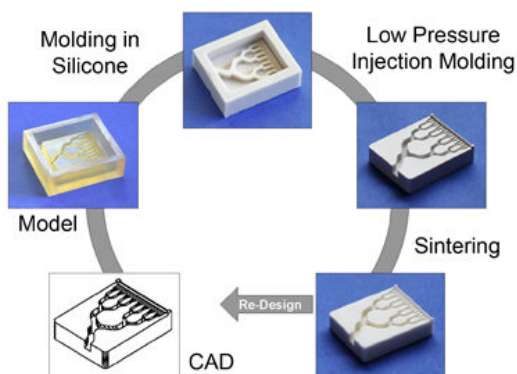


Fig. 2: Schematic representation of a Rapid Prototyping Process Chain (RPPC) for the production of ceramic parts.

Using molds from silicone rubber has proven to be advantageous for several reasons. Such molds can simply be produced by casting the primary model into a two-component silicone rubber. Cross linking of the material only lasts a few hours till the model can be extracted and the mold can be used. Silicone rubber is characterized by an excellent reproduction ability of the finest structures; even details in the sub-micrometer range are replicated. The elasticity of the silicone rubber is not only favorable for the extraction of the primary model but also for the subsequent removing of the soft green body. This makes it possible to demold also fragile details and vertical walls with high surface roughness, as they are often characteristic for RP methods. Even undercuts can be removed without a complex tool design due to the compliancy of the material. However, this compliancy makes some demands on the control of the LPIM process. To avoid discrepancies in the dimensions of the part, it must be guaranteed that the molding process ends in a stress-free state. Deformations may also occur due to the weight of the green body, but this problem is usually negligibly for micro parts. Another challenge is the high thermal expansion of the silicone rubber. For the production of precise parts there are high demands with respect to temperature control during hardening the silicone rubber as well as during the shaping process. Silicone rubber molds can withstand a large number of moldings, as long as the mechanical loads remain in a moderate frame. According to experience, a minimum batch of some 100 parts can be manufactured by a single mold.

### Prototyping of the micro turbine (concept 1)

Zirconia ( $ZrO_2$ ) is proposed as the most promising ceramic material for micro components. It offers a fine microstructure and excellent mechanical properties. Additionally, for micro parts the high costs of the material only play a minor role. For the micro turbine the widespread zirconia TZ-3YS-E powder from TOSOH (Japan) was used. The starting powder has a mean particle size of less than 400 nm and a BET surface of 6-7  $m^2/g$ . The powder was dispersed at a solid content of 52 vol.% in the commercial binder system Siliplast LP65 by Zschimmer & Schwarz (Lahnstein, Germany). The binder already contains dispersants and other additives, thus, no additional organics are required. Melting temperature of the binder is located at app. 65°C. For the preparation of the LPIM feedstock a heatable dissolver mixer (VMA Geetzmann, Reichshof, Germany) was used, resulting in a viscosity of 17 Pa·s (at 85°C and a shear rate of 100  $s^{-1}$ ).

Polymer models for the concept 1 of the micro turbine were mainly manufactured by the so-called RMPD<sup>®</sup> method, developed by microTEC (Duisburg, Germany) [16]. This company has a

large experience in the prototyping of polymer micro parts down to the micro range. In the RMPD technique thin layers of photocurable acrylic or epoxy resins are exposed via a mask. By stacking the layers, a three-dimensional micro part is formed. The layer thickness can be reduced down to a few micrometers resulting in very smooth vertical surfaces (Fig. 3), but for reasons of time and effort usually a layer thickness of 25 or 50  $\mu\text{m}$  is preferred. Lateral resolution is in the range of some micrometers.

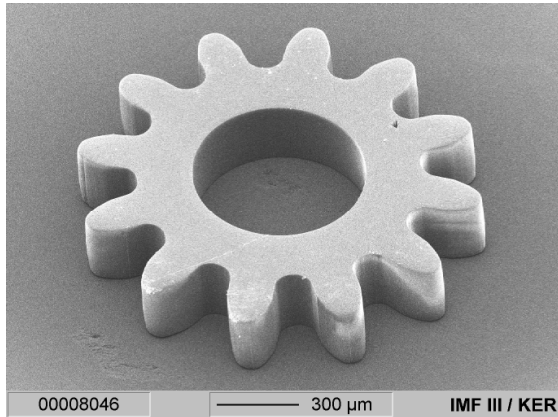


Fig. 3: SEM micrograph of a polymer gear wheel with smooth surfaces, made by the RMPD process.

For the manufacturing of the molds a two-component silicone rubber NEUKASIL<sup>®</sup> RTV 20 with a shore A-hardness 50 from Altropol Kunststoff GmbH (Stockelsdorf, Germany) was used. At a temperature of 40°C cross-linking took place within six hours, enabling the manufacturing of additional molds within short time.

The molding of the micro parts was performed in a piston-driven injection molding machine GC-MPIM-2-MA-X from GOCERAM Ltd. (Sweden). With this machine it was possible to work at injection pressures as low as 0.1 to 0.3 MPa to prevent distortion of the compliant silicone mold. A feedstock temperature of 95°C and mold temperature of app. 40°C was used. Prior to injection, the air was removed from the mold to prevent incomplete filling by entrapped gas. Demolding was performed manually. The process is promoted when the micro parts are molded on a base plate. This supports the mold-filling as the plate creates an additional heat storage and thus reduces the risk of premature freezing of the feedstock. Additionally it works as a support to facilitate the demolding of the fragile micro parts. The plate was removed by grinding before sintering took place. For that purpose the micro parts were recast with wax or paraffin to protect them during machining. The wax is simply removed by melting at the beginning of the debinding process.

Standard debinding procedures were performed up to 500°C at a rate of less than 1°C/min and dwell times at 150°C and 240°C. For sintering a 3°C/min ramp up to 1450°C was used. For the thermal treatment, the micro parts were placed on an alumina substrate which could be heated up to 1450°C, thus it was not necessary to transfer the sensible parts to another support after debinding.

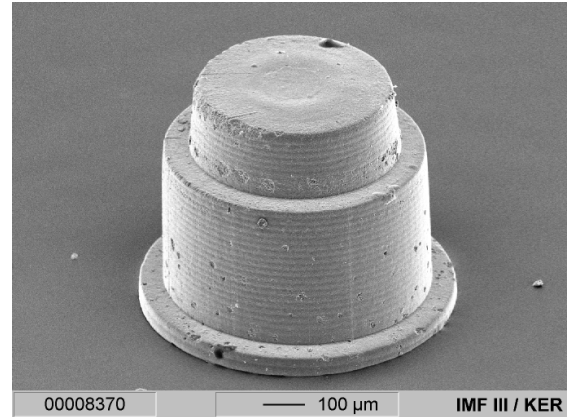


Fig. 4: SEM micrograph of the turbine axis. The layer structure of the polymer model is replicated in the sintered zirconia part.

Examples of sintered micro parts are shown in Fig. 4-6. All structural details of the turbine design could be manufactured with the described method. The high precision of the replication process is evidenced by a complete replication of the layer characteristic of the polymer models into the ceramic micro parts (Fig. 4). However, at a closer look surface defects are revealed arising from insufficient feedstock homogeneity. Powder agglomerates were not destroyed during feedstock preparation, leading to a rough and porous surface. Direct measuring of the sintering density was not feasible with sufficient accuracy due to the low weight of the micro parts but could be performed with parts where the base plate was not removed. The result was a mean sintering density of 97% th. d. (at a theoretical density of 6.10 g/cm<sup>3</sup>) confirming the remaining porosity.

The precision of the micro parts is exemplarily demonstrated by the sintered gear wheel part. The axis diameter ( $d_i$ ), the addendum circle diameter ( $d_a$ ) and the height ( $h$ ) of the parts are given in table 1. All values were measured on a dozen samples with an optical microscope.

Table 1: Measurements of sintered ZrO<sub>2</sub> micro gear wheels

	Mean / $\mu\text{m}$	Standard deviation / $\mu\text{m}$
$d_i$	491	5
$d_a$	1158	6
$h$	155	17

With the exception of the height a precision of better than 10  $\mu\text{m}$  can be obtained by LPIM with silicone molds. Comparable values were achieved for the precision of the other components. For all samples, the relative precision seems to become lower for the small features. This is mainly due to the error of measurement, that was identified to be in the range of 3-4  $\mu\text{m}$  for the used measuring equipment, but for still smaller features it will be caused also by the grain size of the sintered ceramic (400-500  $\mu\text{m}$ ). The lower precision of the height of the gear wheel has another reason. It results from the grinding of the base plate that was performed manually without precise process control.

All components were produced with a high yield of more than 90 %. The only exception is the rotor part (Fig. 5). Here problems arose from the susceptibility of the wings with a cross-section of only 150 x 80  $\mu\text{m}$ . During demolding, grinding of the base plate and other handling steps wings broke away easily. This reduced the yield of this part to less than 20 %.

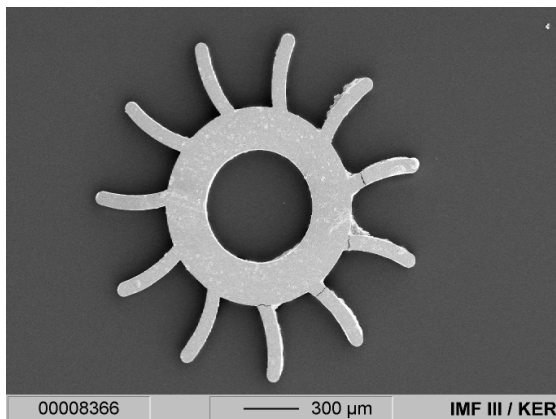


Fig. 5: SEM micrograph of the zirconia rotor. The cracks in the wings were introduced during SEM preparation.

The individual components were tested for assembling to build the complete turbine. It was intended to fix the gear wheel and the rotor on the axis by a press fit as it is common for macroscopic parts. However, the scattering of the diameters led to an insufficient fit of the components. Rotor and gear wheel were usually either loose or broke during the assembly. For thin parts like the rotor even the overcoming of the wall roughness produced tensile stresses which were high enough to break the central ring. Due to this, the press fit could not be used for the assembly of the micro turbine. In addition, suited rotors were fixed on the axis with glue and turned from the outside via the gear wheel. The vulnerability of the design was so high that even a slight contact with the surrounding parts caused failure of the wings.

The described problems evidenced the need to improve various processing steps. Besides the design also the feedstock properties and the machining was updated. These steps optimized the properties and facilitated the handling of the micro parts.

### Improvement by process optimization

The properties of the zirconia feedstock could be improved by using a different binder system. The use of a paraffin binder Type TerHell 6403 (Sasol Wax, Hamburg, Germany) and of Hypermer<sup>®</sup> LP1 (Uniqema, Emmerich, Germany) as a dispersant were found to be effective. The dispersant was added to the paraffin with an amount of 2 mg per square meter of the particle surface. Although the viscosity of this binder system is higher (23 Pa·s at 85°C, shear rate of 100 s<sup>-1</sup> and a solid content of 50 vol.%) the homogeneity of the feedstock is obviously improved as an enhanced sintering density of more than 99.6 % of theoretical density and a reduced surface roughness were obtained (Fig. 6). The linear sintering shrinkage was 20.6%.

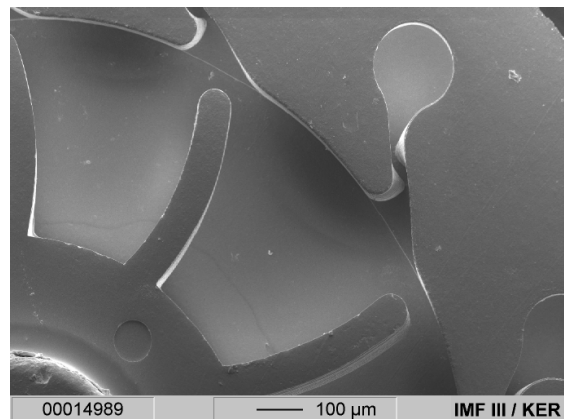


Fig. 6: SEM micrograph of the zirconia rotor within the nozzle plate. The twisted shape of the nozzle is caused by a distortion of the silicone mold during the filling with the high viscous feedstock.

The viscosity of this feedstock is near the upper limit compatible with silicone molds. Although all structural details are replicated in the sintered micro part, deformations can already be seen at the channels in Fig. 6. During the filling of the mold the silicone walls for the 40  $\mu\text{m}$  wide channel structure are already distorted by the increased shear stresses of the feedstock.

Another optimization of the process was achieved by an adapted machining process. As parts were often destroyed during the grinding of the base plate in the green state, the strength of the parts was increased by a pre-sintering step. After debinding, the micro parts were annealed for 1 h

at a temperature of 1080°C to induce the formation of sintering necks at the particles. Thereby the strength of the parts was sufficiently increased to improve the handling without significantly aggravating the machining. In particular, the yield of the rotor parts was considerably increased by this procedure.

### Turbine design optimization (concept 2)

In concept 2 the design was optimized for better permanence and for higher torque output by a new rotor profile and by increasing the height of the rotor from 150 to 800  $\mu\text{m}$ . Gear wheel and axis are now a single unit. Additionally, the connection between rotor and axis was now designed as a triangular form fit junction.

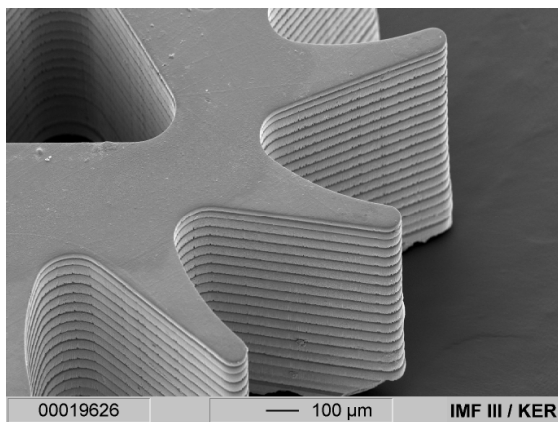


Fig. 7: SEM micrograph of the zirconia rotor (concept 2). The polymer model was made by the RMPD process.

Models for the concept 2 were manufactured either as polymer model by the RMPD<sup>®</sup> process or by micromachining of brass. A challenge of the new design was the increased height of the internal features. With the layer based RMPD<sup>®</sup> technique thick layers and an undulated vertical surface were produced for this sample height (Fig. 7). This aspect aggravated the demolding of the nozzle plate and the axis/gear wheel component drastically. For that reason, a micromachining method was chosen to produce these two components with smoother surfaces (Fig. 8).

Micro milling enables the manufacturing of metal models with accurate geometrical tolerances in the range of a few microns and smooth surfaces with an attainable roughness as low as a  $R_z$  of 0.3  $\mu\text{m}$  [17-19]. Brass was selected as work piece material, featuring a very good machinability while still being quite wear resistant.

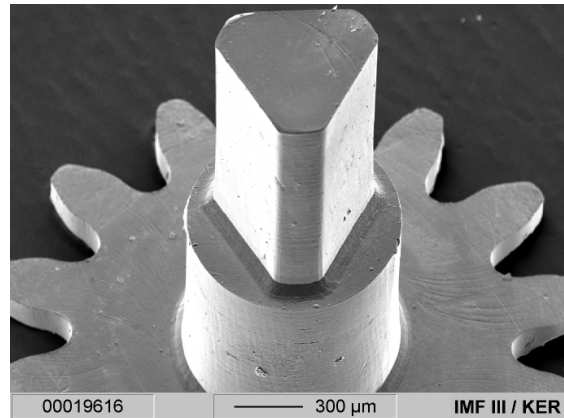


Fig. 8: SEM micrograph of the zirconia axis/gear wheel combination (concept 2). The model was fabricated by micro milling of brass.

The most complex part of the turbine was the nozzle plate with thin slots and a high aspect ratio in order to guarantee the proper functioning of this fluidic part. The width should be as small as possible while maintaining a total depth of 1000  $\mu\text{m}$  (corresponding to 800  $\mu\text{m}$  after sintering).

Smallest available tool diameters in micro milling with a sufficient reproducibility rate are 30  $\mu\text{m}$  wide. However, these tools currently only allow machining of aspect ratios of up to three. To reach the desired depth a 100  $\mu\text{m}$  tool had to be used with a flute length of 1 mm and therefore an aspect ratio of 10. The process parameters employed with this tool were 24.000 rpm, a feed per tooth of 2  $\mu\text{m}$  and an axial feed of 2  $\mu\text{m}$ .

For smaller nozzles, EDM (Electro discharge machining) had to be used as an additional machining step. Therefore, the complete geometry has been machined by milling except for the narrow slots. Subsequently this work piece was clamped to a Micro-EDM machine tool by Sarix (SX 100). Round electrodes with a diameter smaller than 10  $\mu\text{m}$  could be produced by WEDG (Wire electro discharge grinding), but the electrode wear is very high resulting in uneven surfaces (slopes) at the bottom of the work piece. In a second approach small feeler gauges, commercially available as electrodes with a thickness of 30  $\mu\text{m}$ , were successfully employed. The used process parameters were: pulse frequency 180 kHz, pulse width 2  $\mu\text{m}$ , gap 70 V, pulse peak 100 A, idle voltage 90 V.

In first tests, the total slot width includes the width of the electrode and the process inherent gap on all sides and accumulates to 63  $\mu\text{m}$  and a depth of 800  $\mu\text{m}$  (Fig. 9). Further test showed that the desired depth of 1 mm could easily be reached demonstrating the potential of EDM to produce high aspect ratios.

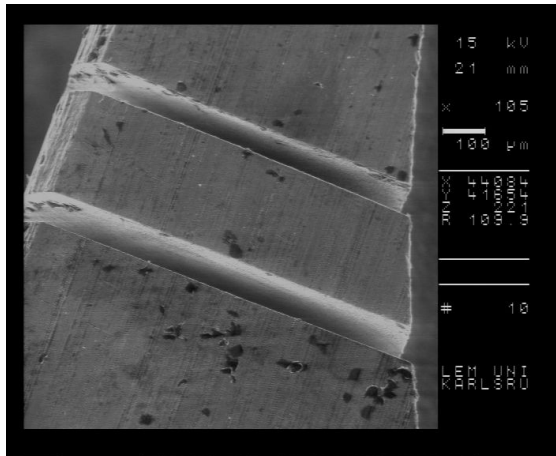


Fig. 9: SEM micrograph of 63  $\mu\text{m}$  test slots made by EDM in brass.

Due to the smooth vertical surfaces of the micromachined model the zirconia nozzle plates with 100  $\mu\text{m}$  slots could be demolded from the silicone molds without problems. After sintering a slot width of approximately 80  $\mu\text{m}$  is achieved (Fig. 10). The preparations for the manufacturing of the brass model with 63  $\mu\text{m}$  slots are still running. If the demolding step works for this sample, a slot width of 50  $\mu\text{m}$  at a depth of 800  $\mu\text{m}$  is expected for the sintered nozzle plate.

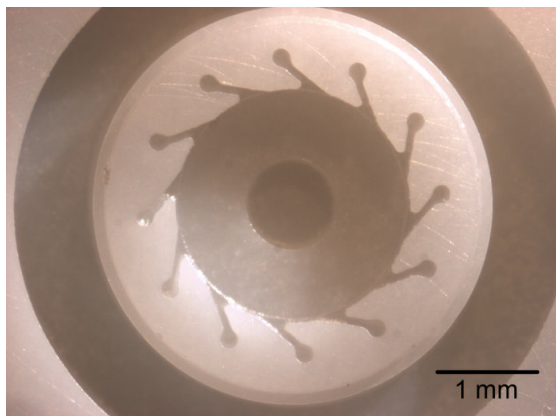


Fig. 10: Optical micrograph of the sintered zirconia nozzle plate (concept 2). Width of nozzle channels is app. 80  $\mu\text{m}$  at a depth of 800  $\mu\text{m}$ .

### Outlook

It is obvious that an operative and reliable system will only emerge if it is possible to manufacture all components with tight tolerances. This is not only a prerequisite for the proper assembling of the parts on the axis but also obligatory with respect to the long-term behavior of the device. The outstanding assembling and operating tests will indicate whether the obtained status is already adequate for turbine applications.

Also intended are performance tests which measure the power output of the device as the turbine must provide sufficient torque to move the subsequent planetary gear drive. Results will be published in a following paper.

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