

# Comparison of micropatterning methods for ceramic surfaces

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## Abstract

The fabrication of defined ceramic micropatterns smaller than 100  $\mu\text{m}$  is due to the hardness and brittleness of ceramic materials still very challenging. However, in recent years, micropatterned ceramic surfaces have become highly interesting for biomedical applications or the fabrication of energy converting devices, such as solid oxide fuel or solar cells. In this study we evaluate six modern techniques for ceramic pattern fabrication with feature sizes ranging from 5 to 100  $\mu\text{m}$ . Ceramic materials such as alumina, zirconia, silica and hydroxyapatite are discussed. Advantages and disadvantages for each process are highlighted and compared to the other techniques. Three of these techniques, namely microtransfer molding, modified micromolding and Aerosol-Jet® printing generate patterns starting with aqueous ceramic suspensions. The other three techniques, micromachining and two different types of laser treatment produce micropatterns by material removal from solid ceramic substrates. The detailed analysis yields that properties such as the desired micropatterning size, shape or the production time are strongly dependant on the chosen technique. © 2011 Elsevier Ltd. All rights reserved.

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## 1. Introduction

Micropatterned surfaces are gaining ongoing interest in the field of materials research and industrial manufacture. They have become state of the art and opened new fields of potential applications for the fabrication of e.g. micromolds with modulated microtopographies<sup>1</sup> or for the modification of micro-bio-interfaces to guide interactions between cell tissue and medical implant surfaces.<sup>2,3</sup> The development and improvement of surface micro- and nanopatterning was in the focus of materials research for the last 20 years. Thereby, various techniques for the fabrication of patterned surfaces have been developed. These techniques were reviewed by a large number of research groups.<sup>4–8</sup>

Ceramic materials are of great interest for micropatterning processes, because they exhibit advantageous properties compared to polymers and metals such as high thermal resistance, chemical inertness, high hardness and biocompatibility. However, obtaining ceramic micropatterns with high accuracy, e.g. precise edge contours, is very challenging due to the particular

hardness and typically brittle behaviour of ceramic materials. For the surface patterning of ceramic materials mainly two different strategies can be applied. First of all there is the possibility of machine-aided ceramic micropatterning with a high effort of technical equipment. Examples are laser surface treatment, injection molding of microparts, slip pressing or casting of microdevices and micromachining of surfaces by e.g. CNC-machining (Computer Numerical Control). Beyond doubt, the results of the machine-aided methods are convincing. Bauer et al. reported the reliable fabrication of microparts with high accurate edge contours and surface details by the use of ceramic slip pressing. Thereby, alumina arrays of more than a thousand columns have been fabricated with smooth sidewalls and sharp edges. The fabricated patterns were 455  $\mu\text{m}$  high and 115  $\mu\text{m}$  wide.<sup>9</sup> Other groups reported reproducible results from the fabrication of microdevices via ceramic injection molding (CIM), in particular with low pressure injection molding (LPIM) of ceramic feedstocks. Various ceramic materials such as alumina, hydroxyapatite and zirconia have been used to reliably produce micropatterned components of a few  $\text{cm}^2$  in size.<sup>10–12</sup> Some of these machine-aided processes yielded remarkable results in terms of micropattern edge contours and process reliability. One main advantage is the potential fabrication of a high number of items per time. The limiting factor however is the difficulty in obtaining micropatterns or components smaller

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than 100  $\mu\text{m}$ . A different approach to fabricate highly defined ceramic patterns is the possibility of using low cost methods such as soft-lithography techniques.<sup>8</sup> Soft-lithography is capable to fabricate micropatterns with a low technical effort and very high accuracy – even smaller than 100  $\mu\text{m}$  – at the same time.

This study evaluates the results of six different micropatterning techniques for ceramic materials for obtaining structural features beneath 100  $\mu\text{m}$ . Non-oxide ceramic hydroxyapatite and at least one oxide material such as alumina or zirconia were used as material to be patterned. The following techniques are evaluated: Microtransfer molding ( $\mu\text{TM}$ ), modified micromolding (m- $\mu\text{M}$ ), Aerosol-Jet<sup>®</sup> printing, CNC-micromachining and two types of laser treatment. Each technique was tested and evaluated for ceramic micropatterning size features ranging from 5 to 100  $\mu\text{m}$ .

## 2. Experimental procedures

Six different processing techniques were applied to fabricate micropatterned ceramic surfaces. For CNC-micromachining and two types of laser treatment solid ceramic substrates were used. From these samples material was removed to form micropatterned surfaces. For the other three techniques, namely microtransfer molding, modified micromolding and Aerosol-Jet<sup>®</sup> printing, the micropatterns were obtained by starting with aqueous ceramic suspensions.

### 2.1. Fabrication of non-patterned substrates

Plane cylindrical, non-patterned platelets were fabricated by uniaxially die-pressing with 15 kN of 1.7 g commercially available calcium phosphate hydroxyapatite powder (HA,  $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$ , Prod.-No. 04238, Lot: 8345A, Sigma–Aldrich Chemie GmbH, Munich, Germany). The HA powder had a theoretical density of  $3.03 \pm 0.04 \text{ g/cm}^3$  and particle sizes of  $151 \pm 0.24 \text{ nm}$ . After die-pressing all samples were sintered for 2 h at 1200 °C in a furnace at ambient conditions (LHT08/17, Nabertherm GmbH, Lilienthal, Germany). The heating rate was 50 °C/h, the cooling rate at 100 °C/h. Except for modified micromolding, the non-patterned samples were used for all patterning processes.

### 2.2. Fabrication of aqueous ceramic suspensions for molding and printing

Three different ceramic suspensions were used for the patterning processes. One suspension contained 11.15 g of HA particles. The HA powder was stirred (RW20, IKA Werke GmbH, Staufen, Germany) into 20 g of double deionised water (Synergy<sup>®</sup>, Millipore, resistivity 18 M $\Omega$  cm, Schwalbach, Germany) to obtain a suspension of 15 vol.% solid loading. The ceramic suspension was adjusted to pH 9–10 by the addition of ammonia solution (25%) to achieve electrostatic stability and to prevent agglomeration of the particles. This ceramic suspension was used as a stamping liquid for the microtransfer molding ( $\mu\text{TM}$ ) process. Another ceramic suspension was used as a ceramic “ink” for Aerosol-Jet<sup>®</sup> printing processes.

It was similar to the first suspension, but it was diluted to a solid loading of 6.6 vol.%. The third suspension was used for modified micromolding. Its composition was similar to the one used for liquid stamping, but additionally a polyacrylic acid-based dispersant/binder was added (12 mg/g ceramic powder). Prior to micropatterning all ceramic suspensions were homogenised using ultrasonic treatment (Sonifier 450, Branson Ultraschall, Dietzenbach, Germany) for 3 min to disperse potential agglomerates.

### 2.3. Micropatterning techniques

#### 2.3.1. Microtransfer molding

For the microtransfer molding ( $\mu\text{TM}$ ) process a ceramic suspension was pipetted onto the micropatterned area of a soft mold (PDMS, Sylgard<sup>®</sup> 184 silicone elastomer, Dow Corning, Wiesbaden, Germany). Excessive suspension was carefully removed with a doctor's blade. Subsequently, the filled mold was deposited on a plane ceramic substrate. After drying the mold was lifted with care. The patterned thin film remained on the substrate during the removal of the mold (Fig. 1a). Hereafter the ceramic substrate and ceramic patterns were sintered. A more detailed description of this method can be found in Holthaus and Rezwan.<sup>13</sup> Generally, adjustable parameters for microtransfer molding are e.g. drying conditions, dispersants and binders, particle sizes and solid loadings of used ceramic suspensions.

#### 2.3.2. Modified micromolding

Similar to the microtransfer molding process, micropatterned soft PDMS master molds were used for modified micromolding (m- $\mu\text{M}$ ). The cylindrical molds were sealed with polyethylene tubes and aqueous ceramic suspension was pipetted into the molding chamber. During the drying process the ceramic particles sink to the bottom of the mold, while water is continuously evaporating. After drying the mold could be lifted with care from the micropatterned solid ceramic sample. Afterwards sintering was performed as mentioned above. Additional details about this method are described by Holthaus et al.<sup>14</sup> (Fig. 1b). Adjustable parameters for modified micromolding are e.g. drying conditions, binders and dispersants, solid loading of the ceramic suspensions and particle size.

#### 2.3.3. Aerosol-Jet<sup>®</sup> printing

Aerosol-Jet<sup>®</sup> printing (Optomec Inc., Albuquerque, USA), also known as maskless mesoscale materials deposition (M<sup>3</sup>D), was used for the direct printing of ceramic aerosols on non-patterned ceramic substrates. The aerosol of a ceramic suspension was generated with an ultrasonic transducer. The printing device is computer-aided and was used to deposit the ceramic particles on specific positions onto the plane substrate. The particles were carried by a gas and deposited by a nozzle onto the substrate. Subsequent sintering of the printed microstructures by a NdYAG-laser (Newport Spectra-Physics, Darmstadt, Germany) was proceeded. Further information about this experiment is provided in Holthaus and Rezwan<sup>13</sup> (Fig. 1c). Adjustable parameters for Aerosol-Jet<sup>®</sup> printing are related to the used ceramic suspensions e.g. dispersants

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