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On the micrometre precise mould filling of liquid polymer derived ceramic precursor for 300-μm-thick high aspect ratio ceramic MEMS

Jonas Grossenbacher[a,*], Maurizio R. Gullo[a], Vadym Bakumov[b], Gurdial Blugan[b], Jakob Kuebler[b], Juergen Brugger[a,**]

[a]Microsystems Laboratory, Ecole Polytechnique Fédérale de Lausanne (EPFL), Station 17, Lausanne CH-1015, Switzerland
[b]EMPA, Swiss Federal Laboratories for Materials Science and Technology, Laboratory for High Performance Ceramics, Ueberlandstrasse 1239, CH-8600 Duebendorf, Switzerland

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Abstract

This paper describes a novel and scalable method for the fabrication of polymer derived ceramics (PDC) structures with high aspect ratio and micrometre scale features. Elastomeric micro-moulds composed of a filling pot are used to deliver via sacrificial micro-channels a precise amount of the liquid ceramic precursor to the target location with the micro-scale functional structures. To improve the filling properties of the mould and to ease the de-moulding of the fragile green body parts, we investigated various channel and mould coating materials, such as carbon and Teflon®-like C_4F_8. The coating properties were characterised by measuring the contact angle and the advancing speed of the PDC inside micro-channels. We found that, the C_4F_8 Teflon®-like coating yields the best de-moulding results for high aspect ratio moulds, whereas the carbon coating yields a two-fold increase in filling speed compared to bare PDMS. The fabricated samples and their side-wall properties were characterised in detail by means of optical and scanning electron microscopy. We present process parameters for well-defined ceramic samples containing micrometric features fabricated with this new approach opening the use of this outstanding material for new MEMS applications where resistance to harsh environment such as mechanical wear, high temperatures or corrosion is required. The presented fabrication method has the potential to be scalable up to cost-efficient mass production.

Keywords: Polymer derived ceramic; Micro-moulding; Capillary filling; Wetting angle

1. Introduction

There is an increasing interest in fabricating MEMS dedicated to extreme temperature and harsh environment applications. Ceramics, which possess outstanding temperature and mechanical properties, are investigated to be used in such environments. The standard polymer derived ceramic (PDC) processing starts with a thermal or UV crosslinking to form the green body (GB) which is subsequently sintered between 800–1600 °C for the densification into a ceramic [1]. The use of PDC for the fabrication of MEMS was proposed by Liew et al. [2]. Different routes to shape PDC materials have been investigated and the main fabrication approaches reviewed by M. Schulz et al. [3] are: moulding [2,4–6], photolithography [7–9] and nanostereolithography [10]. During the thermal processes, the increase of bonds and the gas release from the PDC induces an overall linear shrinkage of about 25%. Therefore, the components must be designed to compensate this volume loss. Furthermore, the green body has to be completely released from its substrate before the sintering at high temperature, to avoid thermal expansion mismatch and crack formation. We previously showed the ability to fabricate high resolution sub micrometre structured surfaces by using a moudling approach [6]. The work presented here focuses on

*Correspondence to: BM 3.202, Station 17, 1015 Lausanne, Switzerland. Tel.:+41 21 693 64 94.
**Correspondence to: BM 3.107, Station 17, 1015 Lausanne, Switzerland. Tel.: +41 21 693 65 73.
E-mail addresses: jonas.grossenbacher@epfl.ch (J. Grossenbacher), juergen.brugger@epfl.ch (J. Brugger).

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the objective to fabricate shape controlled bulk samples and improve filling and release characteristics in view of automated large volume fabrication of ceramic micro-components.

There are following challenges related to the use of moulding for the fabrication of bulk high aspect ratio PDC samples:

Challenge 1: Typically used hard materials moulds limit the degree of freedom of possible shapes due to the shrinkage occurring during the polymer crosslinking.
Challenge 2: By down-sizing the mould, it becomes difficult to manually fill the micrometric sized structures, due to limited pipette positioning and volume control.
Challenge 3: De-moulding of green body becomes difficult as the higher surface/volume ratio increases the sample adhesion to the mould
Challenge 4: The small samples are challenging to handle as they can easily be damaged by tweezers.

In this paper, we address these different issues and propose remedies for each limitation:

Remedy 1: We propose soft polymer moulds since it allows absorbing mechanical deformation related to the PDC shrinkage, and thus facilitates the release of the GB before sintering as explained in section 3.1.
Remedy 2: For miniaturised ceramic moulding, we propose to use a dedicated design for the filling of small moulds. The design is composed of 4 segments: (1) filling pot; (2) filling channel; (3) breaking notch; (4) target sample (Fig. 1). The filling pot is designed with a diameter of several millimetres, in order to easily align the pipette and deliver the appropriate amount of PDC as explained in Section 2.2.
Remedy 3: In order to facilitate the filling and releasing process, we used different combination of coatings on the PDMS mould surface. The resulting wetting angles are measured and reported in Section 3.2.
Remedy 4: Safe handling of the sample is guaranteed by using the cross-linked micro-channels as handling structure. Before the sintering, the samples are separated from the filling structures, by breaking them off at the predefined notch (Section 3.1).

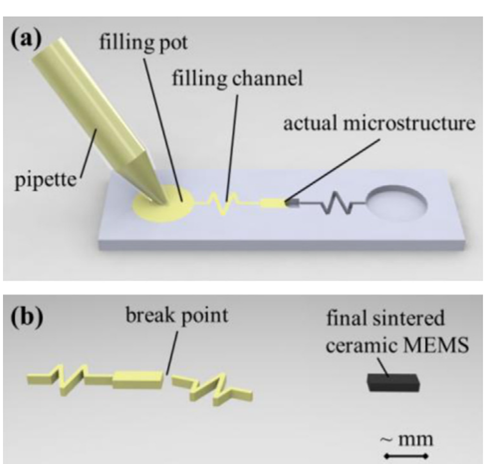

Fig. 1. Illustration of the shaping process of PDC. (a) Mould filling process during liquid state of the PDC and (b) cross-linked and sintered state.

2. Materials and methods

2.1. Materials

The liquid ceramic precursor used for the production of the MEMS component is Ceraset® polysilazane (from Clariant Charlotte, NC, USA). 1,1-Azobis(cyclohexanecarbonitrile) (Sigma-Aldrich, Buchs, Switzerland) was used as radical initiator for thermal cross-linking of the polymer precursors. Polymer mixing ratios, subsequent processing and properties of the resulting ceramics are described in more detail in a previous publication (Bakumov et. al [11]).

2.2. Design

The design of the filling structures (Fig. 1) consists mainly of four parts: the filling pot, the filling channel, the breaking notch and the target functional sample. In this proposed design, the presence of large filling pots (Fig. 1a) enables manual pipetting and increases the volume to be injected, which is calculated in function of the total mould volume. The precise volume of liquid needed to fill the mould is then dispensed in the pot resulting in a flat top surface of the liquid in the mould. The filling channels main function is to transport the PDC to the target sample micro-mould. The spring-like design has the purpose of mechanically damping the shrinkage occurring during cross-linking. A straight filling channel would lead to a pulling force acting on the sample, potentially inducing deformation and cracking of the sample as will be explained in Section 3.2. After the cross-linking, the filling channels will also serve as a bulk structure to enable safe handling with tweezers during the manual unmoulding. Before the sintering, the samples will be detached from the filling channel (Fig. 1b). Therefore, we designed a notch (30° in tip angle, protruding 50 µm into the 150 µm wide channel) at the inlet to concentrate stresses at this point and therefore inducing a crack that will separate the sample from the filling structure without inducing damage to the sample. Moreover, this notch also acts as a security, in case that shrinking material inside the channel pulls excessively on the sample, all stress will concentrate in the sharp angle of the notch, and will thus break at this precise point. This novel design enables the fabrication of structures at the millimetre scale with micrometre to sub-micrometre structures [6] by moulding processes.

The fabrication process is composed of two parts: Firstly, the fabrication of the mould with coatings; secondly the moulding of the PDC material, crosslinking, releasing and sintering.

2.3. Mould fabrication

The fabrication process is based on a counter mould approach. The counter mould is made of SU-8 (GM1075 SU-8, from Gersteltec, Pully, Switzerland), an epoxy based negative photo-resist, commonly used for the pattering of high aspect ratio