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Embossing of ceramic micro-pillar arrays

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Abstract

A microfabrication method for producing ceramic micro-pillars is reported. The method is based on embossing of a gelforming ceramic substrate using a sacrificial polymer mould to produce ceramic micro-pillars with aspect ratios of up to 10. Such high aspect ratio ceramic micro-pillars have been demonstrated for the fabrication of 1–3 PZT piezocomposites. It is also suitable for producing any ceramic MEMS or micropatterned ceramics or microcomponents. The results indicate that the crosslinkable polymer binder system used in the gelforming processing is crucial to retain the structural integrity during mould removal and sintering stages, especially for the high aspect ratio ceramic micro-pillar structures. © 2012 Elsevier Ltd. All rights reserved.

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1. Introduction

Ceramic pillar arrays with high aspect ratios have many potential applications. Examples include 1-3 composites for ultrasound and thermal imaging,¹⁻⁶ electromagnetic bandgap structures,^{7,8} catalyst support and bioreactors,⁹ heat exchangers¹⁰ and thermoelectric devices.¹¹ Among them, 1-3 piezoelectric ceramic/epoxy composites are the most explored.¹⁻³ 1–3 piezocomposite has a structure in which piezoelectric ceramic (e.g. lead zirconate titanate, PZT) pillars array embeds in an epoxy matrix, while ceramic pillars are one-dimensionally connected and the polymer has threedimensional connectivity.⁴ 1–3 piezoelectric composites benefit from high coupling factors, low acoustic impedance, mechanical flexibility, and broad bandwidth in combination with a low mechanical quality factor. The good acoustic match to tissue or water enables 1-3 piezoelectric composites especially useful for underwater sonar and medical diagnostic ultrasonic transducer applications.5,6

Three categories of fabrication methods for such ceramic pillar structures have been reported. These include direct machining; mould-free solid freeform fabrication and

0955-2219/\$ - see front matter © 2012 Elsevier Ltd. All rights reserved. http://dx.doi.org/10.1016/j.jeurceramsoc.2012.04.009 mould-based replication. The conventional way to fabricate ceramic pillar structures is dicing sintered ceramic blocks using a precision saw.¹² This is simple and low-cost method. But there are several drawbacks associated with this method. For example, the pillar and gap size are limited by the saw thickness which is normally $>50 \,\mu$ m. The pillar's cross section is restricted to square shape. Other advanced direct machining methods such as laser micromachining have been used.¹³ It is a flexible technique with good resolution. Arbitrary shape and feature size ($<10 \,\mu$ m) are feasible. It is ideal for rapid prototyping. But it is expansive, the pillars exhibit tapered angle when producing high aspect ratios. Many mould-free rapid prototyping or solid freeform fabrication (SFF) methods such as ink jet printing have been developed for ceramic micro-structures based on direct writing technology.^{10,14,15} However, they suffer from problems of either low lateral resolution or low production rate, especially for repeated periodic structures. Direct ink jet writing has relatively poor surface finishing because of its stepwise bottom up approach. The ink fluid dynamics within a nozzle and layer-by-layer deposition process play a limiting role in defining minimum feature size and surface roughness. The resolution limitations for these techniques are normally ca. 30 µm or above. Therefore, for ceramics with feature sizes in the range of a few micrometers, techniques which can combine both the resolution of lithographically generated patterns and colloidal processing are highly desirable. Namely,

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Fig. 1. Schematic representation of the embossing of gelformed ceramics to fabricate ceramic micro-pillar arrays.

microfabricated moulds are needed to define the minimum feature size and surface finish of the ceramic pillars. In these mould-based techniques, issues such as mould microfabrication and demoulding of unsintered or 'green' ceramics are the key for the success in the fabrication of ceramic micro-pillar arrays. Injection moulding was reported as a possible mass-production method.¹⁶ But due to strong demoulding forces required, the pillar arrays produced are normally with feature sizes $>100 \,\mu m$ and aspect ratios <5. Replication via lost mould is more cost effective and widely reported. Microfabricated silicon mould has been reported.¹⁷ Slurry casting and hot isostatic press (HIP) were employed to fabricate piezoceramic pillar arrays with high aspect ratios. Extremely high aspect ratio (>12) and small pillar size $(7 \,\mu\text{m})$ have been achieved. The main problem, however, is the interaction between Si mould and ceramics during high temperature sintering process. Polymer moulds have therefore been reported as alternatives for silicon. The main challenge associated with these methods for the fabrication of ceramic pillars with fine structures is effective demoulding or removal of polymer moulds.

In this paper, a cost-effective method to fabricate ceramic pillar arrays using an embossing technique is described. PZT micro-pillar arrays have been used as a demonstrator. Based on the previous developed viscous polymer processing (VPP) technique,¹⁸ a modified ceramic gelforming processing is developed which could produce crosslinked green ceramics with high green strength and excellent chemical resistance to solvents, whereby could be used for the fabrication of ceramic micro-pillar arrays with different aspect ratios.

2. Experimental

Poly(methyl methacrylate) (PMMA) moulds used to fabricate ceramic micro-pillar arrays were made by X-ray lithography. They were negative micro-channels with diameters from 15 to 100 μ m and thicknesses from 0.1 to 1 mm. The ceramic powder used in gelforming processing was PZT-5A (Morgan, UK). The powder was pre-treated with a polyacrylic acid (PAA) dispersant (Duramax D-3021, Rohm and Haas) of different concentrations (0–4 mg relative to PZT powder, g) in an aqueous solution. After mixed with the PAA solution for several hours using ball milling, the surface treated PZT powders were freeze dried for the subsequent gelforming processing. The astreated PZT powder with a solid loading of 52 vol.% was mixed with a polyvinyl alcohol (PVA) polymer binder (Gohsenol KH-17, Nippon. Gohsei, Inc., Osaka, Japan) and distilled water in a weight ratio of 100:5:7, the mixture was subject to a high shear field on a twin-roll mill as in the VPP process. The ceramic dough was highly plastic and deformable after milling for a few minutes. It was then calendered into ceramic tapes of a thickness of 1 mm. The ceramic tape was laminated with the PMMA mould. Embossing was carried out in an INSTRON mechanical testing machine at a loading rate of 0.1 mm/min. The load was <80 MPa, below the compressive yield point of PMMA. The embossed samples were afterwards put in an oven at the temperature of 80 °C for 2 h and then 140 °C for crosslinking. The gelformed samples were immersed in chloroform overnight to dissolve away the PMMA mould. Finally, the green ceramic micro-pillar arrays were sintered in a Pb-rich atmosphere at 1200 °C for 1 h. The microstructure of the sintered ceramic pillar arrays were characterised using a scanning electron microscope (Philips XL30).

3. Results and discussion

The fabrication of high-resolution polymer micropatterns has been relatively well established. The (PMMA) resist moulds were produced using X-ray lithography.^{19,20} They have the advantages of high resolution and easy removal by solvents such as acetone and chloroform. UV lithography using SU-8 resist has



Fig. 2. Ceramic micro-pillar arrays using the conventional VPP process after the polymer mould removal.

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