Nanoimprint technology for patterning functional ZrO₂ ceramic materials

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Functional materials such as metals, ceramics and nanomaterials are widely used in modern devices. However, it remains a challenge to pattern these materials especially the ceramics because most of the conventional microfabrication methods are not applicable to them ^[1]. Nanoimprint lithography (NIL), which creates patterns by mechanical deformation of imprint resist, is rather promising for the functional material patterning due to its simplicity, high throughput and low cost. Different NIL-based methods have been developed such as direct NIL, reversal NIL, solid-state electrochemical stamping, and sol-gel NIL to pattern functional materials. All the derivatives in the existing publications still require much help from the conventional methods thus reducing the fabrication efficiency. In this study, we develop another direct NIL-based method to pattern the functional ceramic materials with the goal to improve the fabrication efficiency by taking full advantage of nanoimprint.

The imprint resist is prepared as the following procedure. Zirconium oxide (ZrO₂) nanoparticle (with an average diameter of 50 nm) powder together with a dispersant of 3-glycidyloxypropyltrimethoxysilane (GPTMS)^[2] are added into acetone. The suspension is heated at 100°C while being stirred in a closed bottle for one hour to ensure enough surfactant attachment to the nanoparticles. The mixture is transferred to a beaker followed by the addition of poly(methyl methacrylate) (PMMA, Mw≈990k) solution in acetone. The weight ratio of the components in the mixture is 5:1:1 (ZrO₂: GPTMS: PMMA). The heating (200°C) and stirring continue until all the solvent is visually gone. The final ceramic-imprint resist is in solid form at room temperature, but its viscosity decreases drastically at a temperature beyond the glass transition point of PMMA.

The resist is first flattened on a piece of silicon wafer by pressing against another anti-adhesion layer treated smooth silicon wafer at 20MPa pressure and 175°C. After separating the two substrates, a standard nanoimprint procedure with 40MPa pressure at 175°C is conducted on the substrate with resist. The substrate and the mold are separated from each other after cooling to room temperature. Then the substrate with the imprinted ceramic-imprint resist is sintered by a programmed temperature profile based on the TGA analysis of the ceramic-imprint resist (Fig.1) in argon atmosphere.

XRD spectrum (Fig.2) shows three main monoclinic diffraction lines which correspond to the (111), (211) and (200) crystal planes of zirconia with small but non-negligible shifts. These shifts may be attributed to displacement of zirconium atoms with carbon atoms thus causing the crystal distortions. SEM images of the imprinted structures are shown in Fig.3. Successful fabrication of grating patterns combined with later work to remove the residual layer will demonstrate the applicability of nanoimprint for high-throughput patterning of ceramics. *References*

[1] Yu, C. C. and Chen, H. L., Microelectron Eng, 2015, 132, 98.

[2] Luo, K., Zhou, S., and Wu, L., Thin Solid Films, 2009, 517, 5974.



Fig. 1. TGA curve of ceramic-imprint resist







Fig. 3. SEM images of the imprinted structures afer sintering: top view (left) and cross section view (right)