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Micromachining of Silicon Carbide by Silicon Lost Molding, Chemical Vapor Deposition And Reaction-Sintering

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1. Introduction

Silicon carbide (SiC) has heat resistance, chemical inertness and high hardness, and its micromachining technology has various potential applications in harsh environments. One of the promising applications is pressing of various kinds of glass at high temperature using a microstructured SiC mold. This technology will replace the pressing of plastics and low melting point glass with poisonous lead, which is often used for mass-producing micro-optics.

Previously, we developed novel SiC micromachining process, "micro-reaction-sintering process", which combines silicon micromachining, reaction-sintering by hot isostatic pressing (HIP) and lost molding [1]. This process allows us to produce millimeter-sized SiC parts with microstructures, however, the surface is too rough (2-4 $\mu\text{m Ra}$) to be used for applications such as a glass press mold. In this paper, we report the SiC micro-reaction sintering process combined with SiC chemical vapor deposition (CVD) to improve the surface roughness.

Figure 1 illustrates the process. First, a silicon female mold is fabricated by photolithography and etching, and a SiC film is deposited on the silicon mold by CVD. Next, the SiC film is backed thick with material powder which contains SiC, graphite and binder resin, and then the material powder is reaction-sintered by HIP. Finally, the silicon mold is etched away to release a SiC product like in Fig. 2. The surface of the SiC product derived from the CVD SiC film is expected to show a smooth feature.

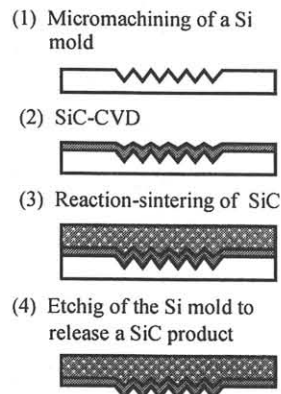


Fig. 1 SiC micromachining process

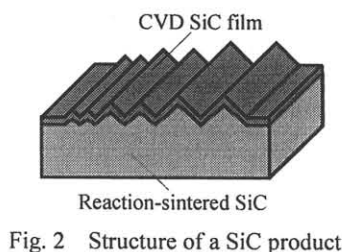


Fig. 2 Structure of a SiC product

2. Experimental Process and Result

A silicon mold which has a triangular-section grating with pitch of 5-20 μm was fabricated by a standard silicon wet etching process using tetramethyl ammonium hydroxide (TMAH). Figure 3 shows the process. A SiC film is deposited using a home-made atmospheric pressure CVD (APCVD) system shown in Fig. 4. In this experiment, a 30- μm -thick amorphous SiC film was deposited from tetramethyl silane (TMS) at 1100 $^{\circ}\text{C}$ in 300 min. Figure 5 shows the cross section of the silicon mold covered with a 3- μm -thick SiC film.

The SiC reaction-sintering for backing the CVD SiC film was performed as shown in Fig. 6. First, the silicon mold was set in a silicon frame, and the frame was filled with the material powder (α -SiC: 60 wt%, graphite: 30 wt%, phenol resin: 10 wt%) by cold isostatic pressing (CIP). Next, the frame was wrapped in silicon powder and subsequently in boron nitride (BN) powder by CIP. This sample was

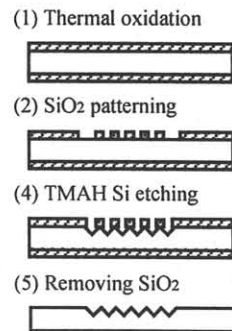


Fig. 3 Fabrication process of the Si mold

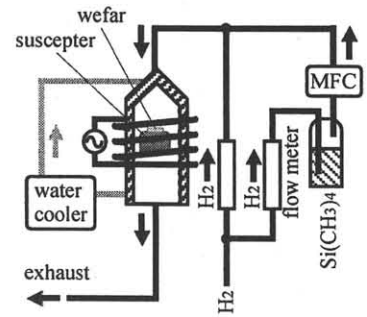


Fig. 4 Diagram of the SiC APCVD system

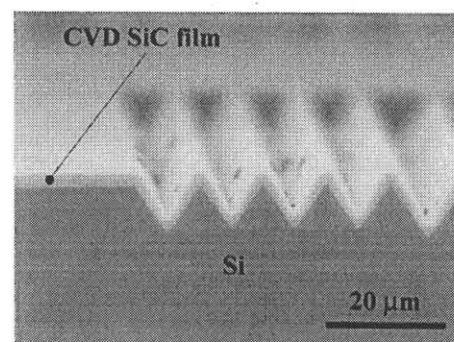


Fig. 5 Cross section of the Si mold with a CVD SiC film

encapsulated in vacuum using a Pyrex glass tube. The BN powder prevents the sample from reacting with the glass tube. Reaction-sintering was performed by HIP at 100 MPa and 1700 °C for 2 hours. In this step, the silicon powder melts, infiltrates into the materials powder and reacts with the graphite powder to generate SiC. The original SiC powder is bonded with this newly-generated SiC, and dense SiC backing is realized. Finally, the sample was dipped in etchant composed of hydrofluoric acid and nitric acid to etch away residual silicon after taken out from a glass capsule.

Figure 7 shows the cross section of the reaction-sintered sample fractured before silicon etching. The CVD SiC film is successfully backed with reaction-sintered SiC, and the shape of the triangular-section grating is maintained. Figure 8 shows the surface of the CVD SiC film backed with the reaction-sintered SiC, where the residual silicon was etched away. The shape of the silicon mold is transferred to the SiC product, however, the surface is not as smoothly as expected. Close-up views in Fig. 8 (b) suggest that the CVD SiC film was damaged during reaction-sintering and/or that poly-crystal SiC grew on the CVD SiC film. The surface roughness of the SiC product is 50-80 nm Ra, which is approximately one fiftyth of that without a CVD SiC film.

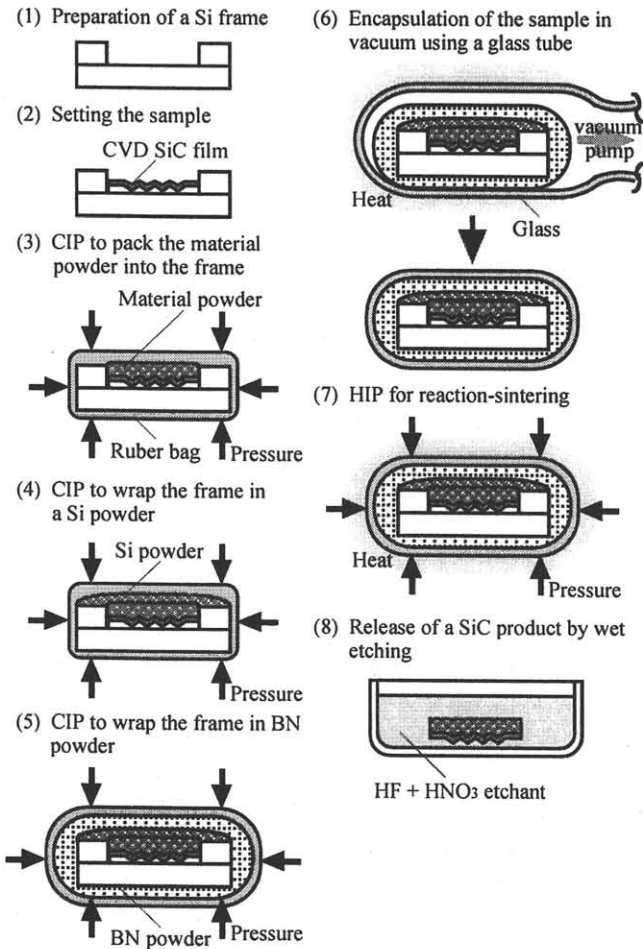


Fig. 5 Silicon carbide reaction-sintering process

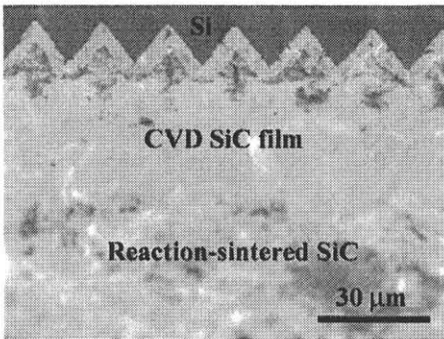
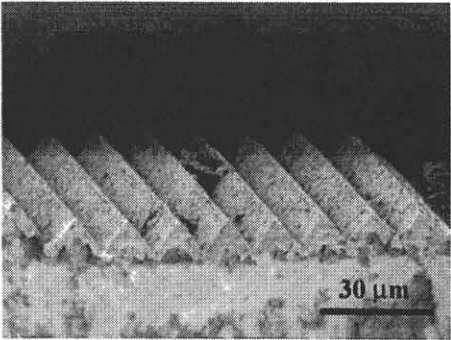
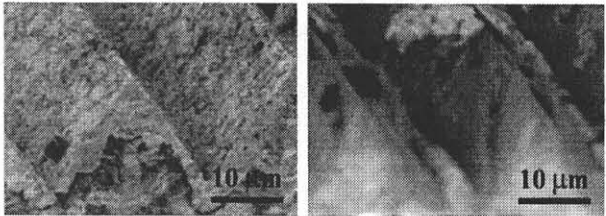


Fig. 7 Cross section of the reaction-sintered sample fractured before Si etching



(a)



(b)

Fig. 8 Surface of the CVD SiC film backed with reaction-sintered SiC

3. Conclusion

To obtain a SiC microstructure with a smooth surface, a SiC film was deposited on a micromachined silicon mold by APCVD, and the SiC film was backed by reaction-sintering of SiC. The SiC film was successfully backed with reaction-sintered SiC, however, the surface roughness is 50-80 nm Ra, worse than expected. Such rough surface could be caused by the damage of the CVD SiC film and/or the growth of poly crystal SiC during reaction-sintering. In future work, we will decrease the temperature and duration of HIP, and/or deposit a single- or poly-crystal SiC film on a silicon mold to improve the surface roughness.

Acknowledgment

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Reference

- [1] S. Tanaka *et al.*, J. Microelectromechanical Systems, **10**, 55 (2001).