Fabrication and Mechanical Properties of Silicon Carbide Micropillars

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1. Introduction

Silicon carbide (SiC) has outstanding thermal and mechanical properties under high temperature and high neutron irradiation. SiC and SiC/SiC composites have been proposed as a promising candidate material for structural components in fusion reactors [1]. Characterization of the mechadnical properties such as fracture strength is important in ensuring the reliability of these ceramic structures [2].

This study demonstrates a micro-compression test of SiC micropillars which are fabricated by mask and dry-etching technique. Our fabrication method involves lithographic pattering of spun and baked photoresist on chemically vapor-deposited (CVD) polycrystalline beta-SiC substrates, followed by lift-off process of electroplated metal into the prescribed photoresist template. This metal works as an etch cap for inductively coupled plasma (ICP) etching. Our fabrication method enables the production of more than a few hundred micropillars under an identical fabrication condition, which is a great benefit for the statistical analysis of the fracture properties of brittle ceramic materials. The diameters of fabricated SiC micropillars range from 6 down to 0.5 μm. The ratio of micropillar diameter to height is set to 1:3 ~ 1:4.

Uniaxial compression tests have been conducted using flat punch nanoindentation at room temperature. We observed the specimen size effect on the measured fracture stress of SiC micropillars. In this paper we present the results of the micro-compression tests of SiC micropillars with the diameters of 0.8 and 2.6 μm.

2. Methods and Results

In this section we detail the fabrication method of SiC micropillars. The fabricated SiC micropillars have been observed by scanning electron microscopy (SEM). The results of micro-compression tests are presented along with the SEM micrographs of SiC micropillars after deformation.

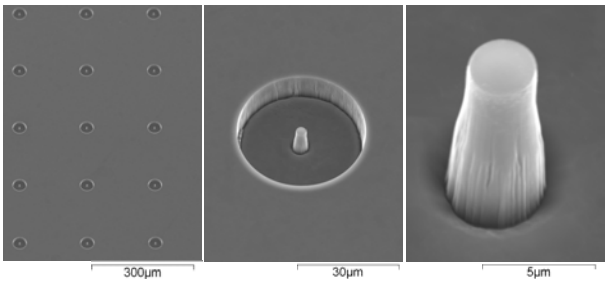
2.1 Fabrication of SiC micropillars

Commercially available CVD 3C-SiC (Morgan Technical Ceramics) specimens were used in this study. Electron backscatter diffraction measurement shows that the grains are columnar in shape along the growth direction and the grain size is 2 ~ 10 μm.

Photoresist was spun and baked on 1cm×1cm SiC substrates to form the resist layer. This photoresist was then patterned by using a Cr-mask on a quartz plate to produce micropillars with the diameter larger than 2 μm or by the electron beam lithography techniques to produce micropillars with the diameter less than 1 μm.

Ti(30 nm)/Ni(300 nm) films were then electroplated on the patterned photoresist template. These metal thin films were used as an etch cap. The final metal patterns were formed by using the conventional lift-off technique of photoresist layer. The patterned SiC substrates with the thin metal film etching mask were etched by using inductively coupled plasma etcher [3].

Fig. 1 shows the SEM micrographs of SiC micropillars with the prescribed diameter of 2.6 μm. The observed diameter of the micropillars is ~2.9 μm with the height of ~11.5 μm. This is similar with the results of the measurement by using optical surface profiler. The SiC micropillar is a little bit tapered, and the bottom diameter is ~4.2 μm. The outer circle around the pillar is designed for finding the pillar in the proceeding micro-compression tests which operates under optical microscopy.



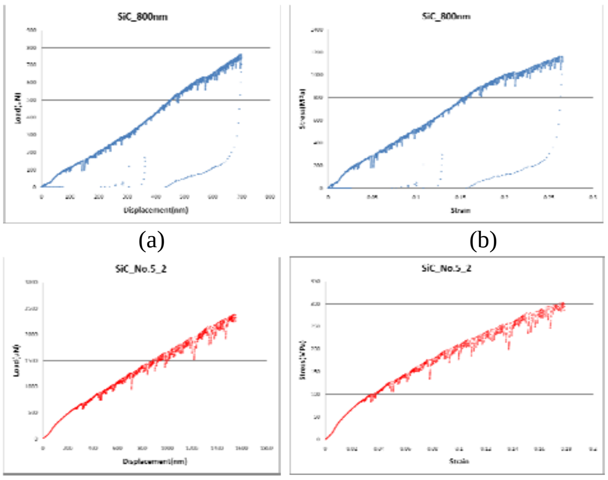
(a) (b) (c)

Fig. 1. SEM image of SiC micropillar array, (a) part of 20×20 array, (b) one crater with a pillar at the center, (c) magnified image of a pillar

2.2 Uniaxial micro-compression tests of SiC micropillars

Uniaxial micro-compression experiments of the SiC pillars were conducted using Hysitron Ti-750 nanoindenter with flat ended punch. The test has been performed with the indent rate of 16 nm/sec to maintain a constant strain rate.

Fig. 2(a) and 2(c) shows the measured load and displacement curves for SiC pillars with the diameter of 0.8 μm and 2.6 μm respectively. Load increases almost linearly. The abrupt load drop is due to the fracture of SiC pillars. The stress and strain curve calculated from the measured pillar diameter and height are shown in Fig. 2(b) and 2(d). Engineering stress was calculated by dividing the load P by the initial cross-section A, determined from the measured diameter. Strain was calculated from the displacement measurement of the instrumented stack piezo and corrected for pillar sink-in. This can be done using Sneddon's equation for the indentation of a flat punch into an infinite half space [4]. The calculated Young's modulus is much less than the expected value from the literatures. It is expected that the strain is overestimated and other effects, such as the deformation of flat punch is required to be considered for correct calculation. Indeed recent observation in in-situ SEM deformation of Si pillars demonstrates that strain measurement from the piezo displacement is not suitable for the measurement of elastic properties [5].



(a) (b) (c) (d)

Fig. 2. Measured Load-displacement curve for (a) 0.8 μm diameter, (c) 2.9 μm diameter pillar, and calculated stress-strain curve for (b) 0.8 μm diameter, (d) 2.9 μm diameter pillar

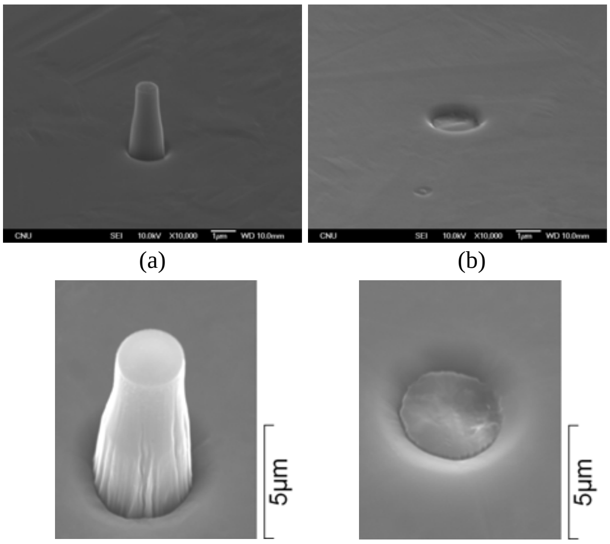
Compressive strength increases with decreasing pillar diameter, e.g. ~300 MPa for 2.5 μm diameter and ~1.5 GPa for 0.8 μm diameter.

Fig. 3 shows the SEM image of SiC micropillars before and after micro-compression test. All the pillars were completely fractured at the bottom of the pillar, probably due to the stress concentration around the trench at the bottom of the pillar.

3. Conclusions

We successfully fabricated and tested SiC micropillars. The micro-compression tests showed that the specimen size effect exists on the fracture strength of SiC. Statistical analysis of this size effect on the fracture strength will be discussed by comparing with earlier experiments on SiC fracture strength as soon as more data is available.

Our method for mechanical characterization of SiC has an advantage in evaluating the effects of irradiation. For example, the height of micropillars with 0.5 μm diameter is close to 2 μm, which is comparable with the penetration depth of 5 MeV Si ions. The effect of ion irradiation on the fracture strength of CVD SiC micropillars can be evaluated.



(a) (b) (c) (d)

Fig. 3. Fraction of counts lost with voltage and charge sensitive preamplifiers as a function of the true count rate.

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