MECHANICAL CHARACTERIZATION OF SiC FILM AT HIGH TEMPERATURES BY TENSILE TEST

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ABSTRACT

This paper reports the mechanical properties of silicon carbide (SiC) films at elevated temperatures up to 500ºC. Poly-crystalline SiC film (poly-SiC) was deposited by LPCVD on a silicon wafer and patterned into a free-standing specimen on an "on-chip" tensile test device. The fracture strength of poly-SiC films showed little temperature dependence over the test temperature range. The tensile strength was 2.89 GPa at room temperature (RT), and decreased slightly to 2.66 GPa at 500ºC. The fracture surface at 500ºC showed almost the same morphology as that at RT, without any slippage. The potential of SiC films as a material for micromechanical devices working at high temperatures has been heretofore confirmed.

1. INTRODUCTION

The demands for sensors that can be operated at high temperature are increasing in fields such as the automobile, aerospace, energy, and chemical industries. Si-based MEMS (microelectromechanical systems) physical sensors have already been commercialized for detecting physical quantities such as pressure, acceleration, angular, and velocity. Single-crystal-silicon (SCS) is an excellent mechanical material in the room temperature range. However, the strength of SCS films with a thickness less than 5 μm significantly decreases above 300ºC [1], and micromechanical structures show plastic deformation above 500ºC.

Silicon carbide (SiC) is a promising material for application in harsh environments such as being exposed to high temperature, high radiation, corrosive chemical, etc. SiC has a high elastic modulus, high mechanical strength, high thermal resistance, and excellent resistance to corrosion. Various approaches to fabricating SiC thin films on silicon wafers have been developed in the last twenty years. Of particular interest here is that researchers have demonstrated SiC MEMS sensors for harsh environment applications [2-4]. To understand the reliability of these devices, it is necessary to study the mechanical properties of SiC films in the same dimensions and fabrication processes of MEMS devices in order to identify potential size effects and process dependencies. While several researchers have reported the mechanical properties of micron-sized SiC films [5-9] for MEMS devices, very little has been done at high temperatures [10]. The mechanical properties at high temperatures are necessary for poly-SiC based MEMS sensors for harsh environments.

We have previously developed a tensile testing system for mechanical characterization of thin films at high temperatures [1]. In the present study, we fabricated SiC tensile specimens based on our tensile testing systems. We report the tensile testing results, up to 500ºC, of polycrystalline SiC (poly-SiC) films deposited by LPCVD, and show the films' mechanical properties at high temperatures for MEMS structures.

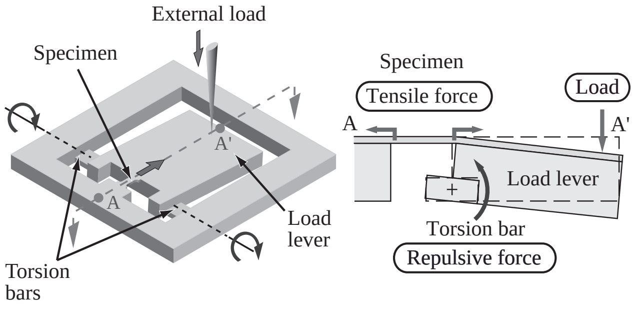
2. DEVICE AND FABRICATION

"On-chip" tensile test

We applied the "on-chip" tensile test system developed by Sato [11] to evaluate mechanical properties of poly-SiC thin films at high temperatures. In this method, we use a unique test device that consists of a thin film tensile specimen and a loading system, as shown in Fig. 1. The loading system is composed of torsion bars, a load lever, and a support frame. When a normal load is applied to the load lever, the lever rotates around the axis of the torsion bars, and the specimen is uniaxially stretched (Fig. 1 (b)). The applied load balances the tensile force to the specimen and the repulsive force of the torsion bars before the specimen fractures. The tensile force is calculated from the applied load by subtracting the repulsive force of the torsion bars, which can be measured after the specimen fractures.

This testing method has two advantages for testing brittle materials like SiC. First, this device enables easy tensile testing of thin film materials only by pushing-down action of the load lever end without having to deal with bothersome specimen gripping problems. Second, the system is suitable to detect small strains of SiC which is a very stiff material. The elongation of the specimen is proportionally enlarged and detected at the loading point of the lever end according to the lever ratio of the system.

The test device is clamped on the heating stage containing cartridge heaters and a thermocouple, and the



(a) Schematic of the test device *(b) Cross-sectional view*

Figure 1. "On-chip" tensile testing method.

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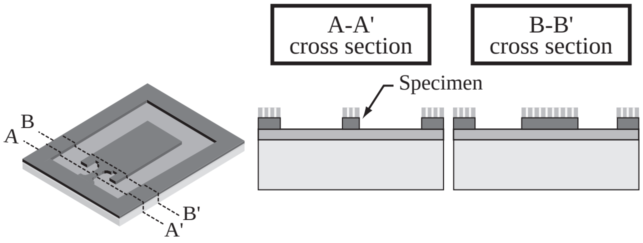
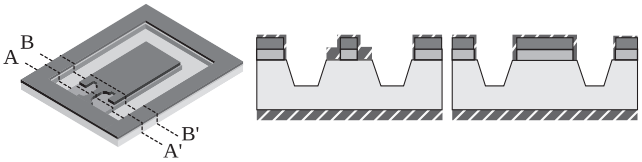
temperature of the stage can be controlled during the test. The details of the measurement system are described in [1].

Fabrication process of test chip

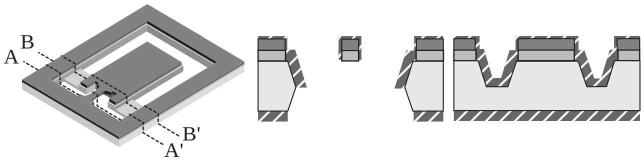
Fig. 2 shows the fabrication process of the SiC film specimen on the tensile test device. We prepared (100) single crystal silicon with a thickness of 450 μm. A 2-μm-thick LTO layer was deposited on the front side to protect the silicon surface. Poly-SiC was then deposited on the LTO layer by LPCVD deposition; the total thickness was achieved by accumulative deposition in three runs. A Ni mask was deposited and patterned, and used as the etch mask to define the poly-SiC specimen profile by inductively-coupled-plasma etching using SF_6 (Fig. 2 (a)). After removing the Ni RIE mask, a thermal silicon dioxide layer was grown and patterned on the top surface of the Si substrate to serve as an etching mask for the next step, which was anisotropic wet etching using a TMAH solution. This bulk etch of the silicon substrate was continued until the desired thickness of the torsion bars was attained (Fig. 2 (b)). Silicon dioxide was thermally grown and patterned again on the back side of the Si substrate, and wet bulk etching was again used to define the profiles of the torsion bars and load lever (Fig. 2 (c)). Finally, the oxide mask was removed using buffered HF solution.



(a) Formation of SiC specimen profile



(b) Defining the load lever profile and thickness of torsion bars



(c) Formation of the torsion bars and load lever profile

▥ Ni ☐ LTO ▨ Thermal oxide ■ SiC □ Si

Figure 2. Fabrication process of SiC film test device.

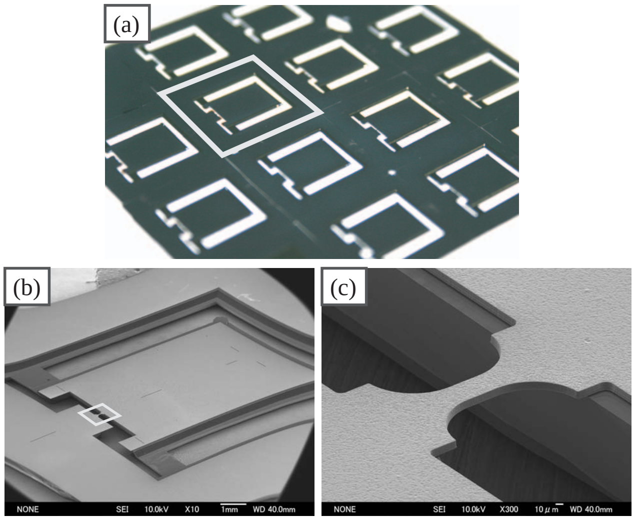
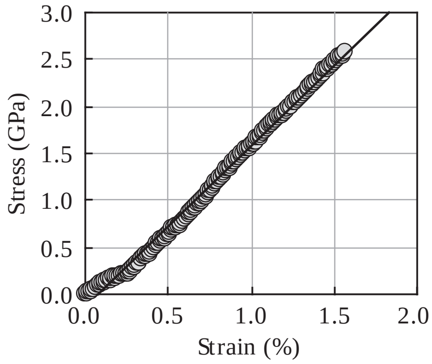


Figure 3. Fabricated testing device. (a) Arrayed tensile devices on wafer, (b) SEM image of the device, (c) SiC film specimen.

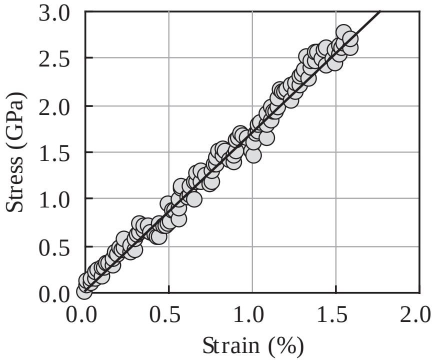
The wafer was cut into 15-mm squares. A typical fabricated poly-SiC test specimen is shown in Fig. 3. The specimen size is 50 or 100 μm long, 45 μm wide, and has an average thickness of 11.7 μm.

3. RESULTS AND DISCUSSION

Tensile testing of the poly-SiC specimens was carried out at temperatures ranging from room temperature (RT) to 500ºC. Four tests were conducted at RT, three at 300ºC, and four at 500ºC. Figure 4 shows the stress-strain relationships at RT and 500ºC. The measured data represented by the open circles show that the poly-SiC specimens deformed



(a) RT

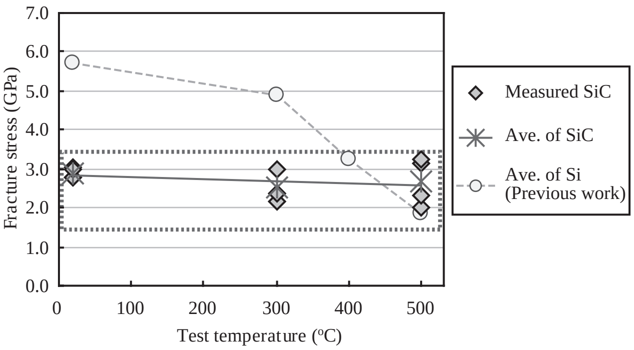


(b) 500 °C

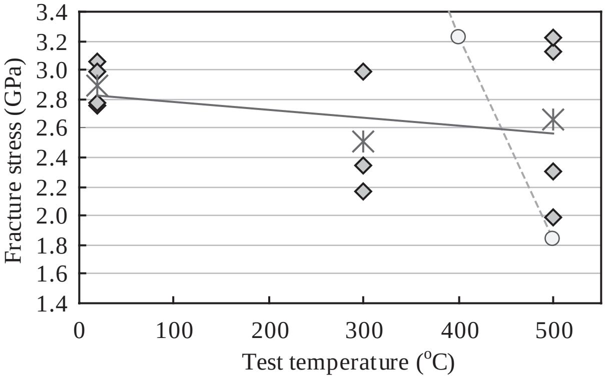
Figure 4. Stress-strain relationship.

elastically until they fractured, although the graph at 500ºC includes some thermally induced noise. All specimens showed the expected brittle fracture behavior like above.

Figure 5 shows the relationship between fracture stress of poly-SiC films and test temperature, as well as a comparison with previous study of SCS films [1]. The average values at each temperature are summarized in Table 1. The fracture stress of poly-SiC film at RT was 2.89 GPa, which is relatively large compared to other reports [5, 6]. This difference can be a result of differences in the fabrication process conditions from film deposition to specimen patterning. In particular, etching conditions that dominate the morphology of the specimen sidewall, where the weak point of thin film material is prone to occur. As the test temperature increased, fracture stress slightly decreased, reaching 2.66 GPa at 500ºC with a slight increase of variation.



(a) Full scale graph from RT to 500ºC



(b) Magnified graph of the dashed line

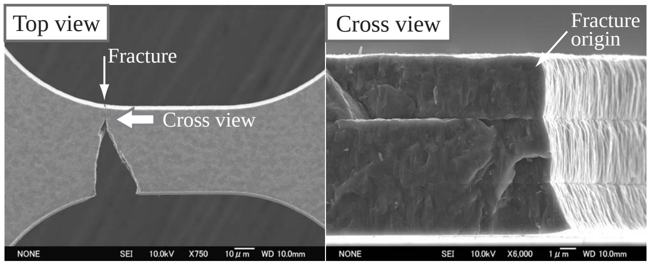
Figure 5. Measured fracture stress of SiC film at each temperature.

Table 1. Average value of fracture stress of SiC and SCS.

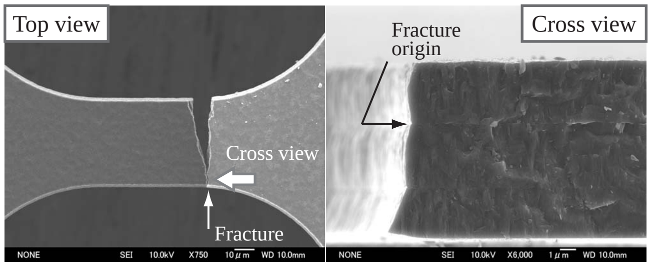
Test Temp. (ºC)		RT	300	400	500
SiC	Average Thickness (μm)	11.73	11.75	–	11.69
	Number of Tests	4	3	–	4
	Fracture Stress (GPa)	2.89	2.50	–	2.66
SCS <110> loading [1]	Average Thickness (μm)	3.97	4.33	4.35	4.26
	Number of Tests	5	5	5	5
	Fracture Stress (GPa)	5.73	4.91	3.22	1.84

In our previous work on SCS film [1], the fracture stress was 5.73 GPa at RT, more than twice that of the SiC film. The possible reasons of such a low strength of SiC relative to SCS can be that: (i) the poly-SiC film here is fabricated by from three separate depositions, providing an opportunity to introduce flaws in the specimen; (ii) the RIE etched sidewall morphology of the poly-SiC specimen is different than the previous SCS specimen; and (iii) the SiC specimen thickness is much larger than that of SCS. Actually, a size effect on the strength with Si has been reported elsewhere [12]. With SCS films, the fracture stress decreased significantly above 300ºC, and further decreased to 1.84 GPa at 500ºC with cross-slippage in the crystal. Here, we find that the fracture strength of poly-SiC films is stable in a range from RT to 500ºC.

Figure 6 shows SEM images of poly-SiC specimens fractured at RT and 500ºC. All specimens fractured at the end of the straight portion, where the stress concentration occurs. The cracks originated at the corner of the rectangular cross section or at a flaw between the layers of three consecutive LPCVD depositions. The multi-layer film has higher probability of fracture because of flaws at the layer interfaces. We speculate that a film with the same total thickness from one continuous deposition will have improved tensile strength. The fracture surfaces at 500ºC showed almost the same morphology as those at RT,



(a) RT



(b) 500ºC

Figure 6.SEM images of fractured SiC specimens at RT and 500ºC.

without any slippage in the crystals or grain boundary. This indicates that the fracture properties do not change at 500ºC. These results demonstrate the high mechanical robustness of SiC films at high temperatures and guarantee reliable MEMS devices operating under such harsh environments.

4. CONCLUSION

"On-chip" tensile testing was used for mechanical characterization of poly-SiC thin films at high temperatures. Poly-SiC films deposited by LPCVD at a thickness of 11.7 μm were patterned into tensile specimens based on an "on-chip" tensile test device. Tensile testing of poly-SiC films was carried out in a range from RT to 500ºC. The fracture stress of poly-SiC film at RT was found to be 2.89 GPa. The value then slightly decreased as the test temperature increased, reaching to 2.66 GPa at 500ºC. Compared to previous results from SCS films that showed a significant decrease in strength at temperatures above 300ºC, SiC films exhibited highly stable mechanical strength. Fracture surfaces of poly-SiC films at 500 ºC showed almost the same morphology as those at RT, without any slippage. We found that SiC films are excellent for high-temperature MEMS devices.

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