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Tensile Testing of Single-Crystal Silicon Thin Films at 600°C Using Infrared Radiation Heating

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In this paper, we report the development of a new tensile tester operable at 800°C using an infrared radiation heating method to evaluate the mechanical properties of thin films at high temperature. The tester uses an electrostatic grip system for specimen chucking. We evaluated the grip force at high temperature and concluded that the electrostatic grip is effective up to 800°C. Single-crystal silicon specimens of 3 μm thickness and 4 μm width were tested at room temperature (RT) and 600°C. At RT, the fracture strength and Young's modulus were 3.33 and 163.2 GPa, respectively. At 600°C, they were 2.71 and 151.8 GPa, respectively. The stress and stage displacement curves at 600°C with lower strain rate showed yield points and the fractured specimens exhibited slip lines.

1. Introduction

Expanding application of micro-electromechanical systems (MEMS) demands rapid development of MEMS devices for various systems, such as automobile, aerospace, mobile, and security systems. For such development, a database of the properties of MEMS materials will help with their appropriate design. The materials used in MEMS are the same as those used in semiconductor devices, since the fabrication processes of MEMS were established from those of semiconductor devices. The lack of knowledge on the mechanical properties of these materials and their small size hinder the establishment of a material database. The development of evaluation techniques for these small materials is also important and widely undertaken.

The smallness and compactness of MEMS have led to their application in harsh environments, which demands the evaluation of material properties under specific conditions. Operations at high temperature are necessary, such as for sensing in engine rooms and the exhaust systems of cars, and geophysical instrumentation. Recently, research on the high-temperature mechanical properties of silicon, a typical material

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in MEMS devices, has been reported,(1–3) in which plastic behaviors at the relatively low temperature range of 100 to 200°C were exhibited. This plastic deformation at low temperature may arise from the small dimensions of the specimens. To understand the transition temperature from brittle to ductile fracture in silicon, a wide range of measurement temperature is required. However, it is difficult to heat specimens over 500°C and up to 800°C, because the heat capacity of the specimen holder used in the previously reported test apparatuses is high.

To overcome the difficulty of heating specimens over 500°C and to evaluate the properties of various materials, we have developed a new high-temperature tensile tester for thin films equipped with an electrostatic grip system using an infrared radiation (IR) heating method. In this paper, the feasibility of the electrostatic grip system at high temperature is evaluated first. Secondly, the details of the developed tester and the experimental conditions for single-crystal silicon thin films are described. Then, the results of both the high-temperature tester and the mechanical properties of silicon are described and discussed.

2. Electrostatic Grip at High Temperature

We considered that two important components of a tensile tester were heating and chucking of a thin-film specimen at 800°C. For specimen heating, noncontact heating by IR was chosen because of its rapid temperature ramping and low heat loss. For specimen chucking, we applied an electrostatic force grip (Fig. 1),(4) because it has a low heat capacity, which is suitable for IR heating, as well as easy specimen handling.

However, the feasibility of the electrostatic grip at high temperature has not been investigated. The dielectric strength voltage of films tends to decrease with increasing temperature. Low strength voltage may cause faulty chucking in tensile testing. We measured the dielectric strength using a method simulating the test environment. The measured strengths of 0.2-μm-thick silicon nitride films used as the insulating layer of the electrostatic grip were 230 and 86 V at room temperature (RT) and 800°C, respectively.

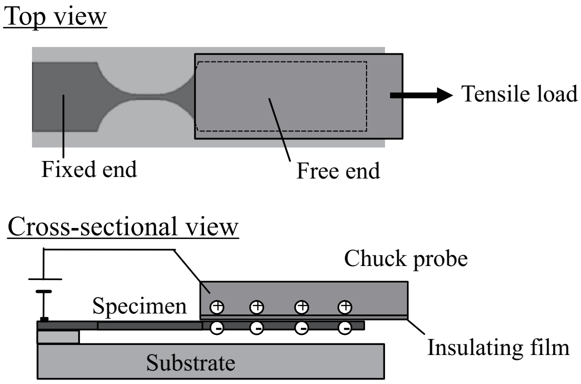


Fig. 1. Electrostatic grip.

We concluded that the electrostatic grip can be used by reducing the specimen cross-sectional area to one-tenth of that of the specimen for RT testing, since the applicable voltage to the electrostatic grip at 800°C is one-third of that at RT.

3. Experimental Procedure

3.1 *High-temperature thin-film tensile tester*

Figure 2 shows a schematic drawing of a high-temperature thin-film tensile tester, whose major specifications are listed in Table 1. Specimens were heated through a molybdenum plate by IR. The specimen temperature was calibrated as a function of that of the Mo plate, monitored using a thermocouple. Tensile force was applied by moving a motorized *x*-axis stage where the specimen holder was placed. The force was measured using a load cell connected to the electrostatic grip. The grip was a silicon strip covered with a 0.2-μm-thick silicon nitride film and held with a molybdenum block

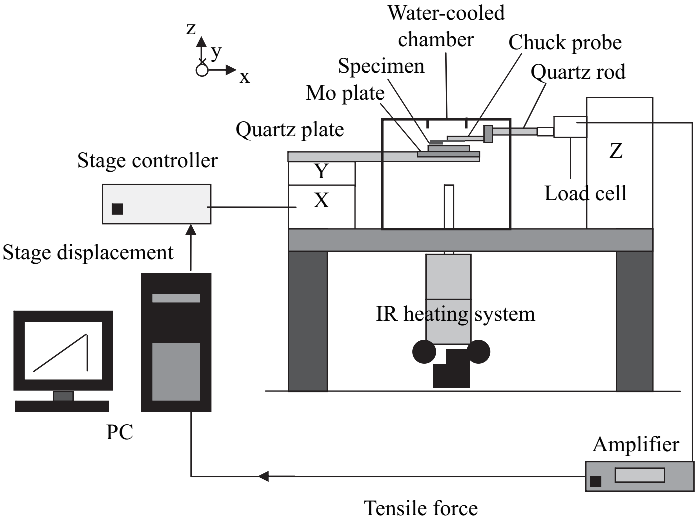


Fig. 2. Schematic diagram of high-temperature thin-film tensile tester.

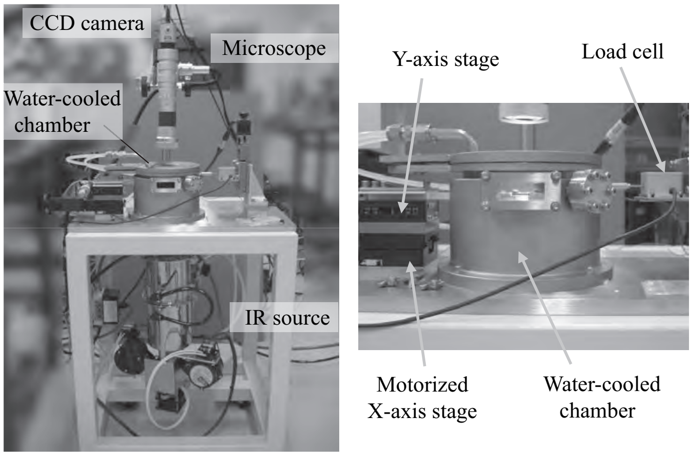
Table 1
Major specifications of the high-temperature thin-film tensile tester.

Load cell	Range	500 mN
	Resolution	1.5 mN
X-axis stage	Stroke	20 mm
	Resolution	0.02 μm
	Min. speed	0.02 μm/s
Max. temp.		800°C

to connect the strip and the load cell. We used a quartz plate and rod, which suppressed heat conduction from the Mo plate and block to the stage and the load cell, respectively. In addition, the stages for alignment and the load cell were placed at the outside of a water-cooled stainless steel chamber, which covered the testing space. This is important in order to achieve heating of specimens to a high temperature while maintaining the low temperatures of the other components. Figure 3 shows photographs of the constructed tensile tester.

3.2 *Tensile testing of single-crystal silicon*

The tested material was single-crystal silicon (SCS). Specimens were fabricated from (100) silicon-on-insulator (SOI) wafers by a standard SOI device fabrication technique using deep reactive ion etching (RIE). The specimen tensile axis was in the <110> direction. The width and thickness of the test part of the specimen were 4 and 3 μm, respectively. The length of the test part of the specimen was 120 or 600 μm, as shown in Fig. 4. Tests were carried out at RT and 600°C. The voltage applied to the electrostatic grip was 100 V. The displacement rate of the motorized *x*-axis stage was 0.04, 0.08, or 0.2 μm/s.



(a) (b)

Fig. 3. High-temperature thin-film tensile tester: (a) outlook and (b) close-up view of chamber during heating.

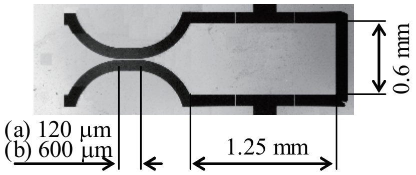


Fig. 4. Single-crystal silicon specimen.

4. Results

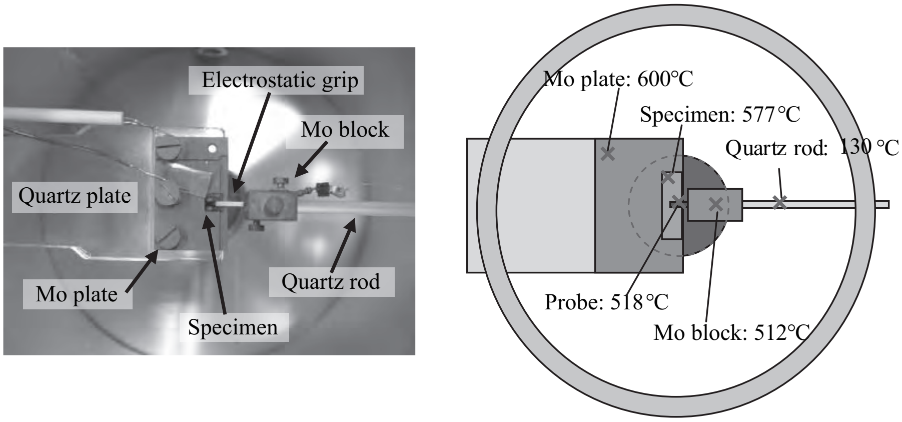
4.1 *Specimen temperature*

When the center of the IR heating area was aligned to the center of the Mo plate, we found that the specimen was heated to 800°C before chucking with the electrostatic grip. However, the temperature at the electrostatic grip was about 200°C after chucking. Therefore, the heating position was moved along to the end of the Mo plate to heat the grip (Mo block) at the same time, as shown in Fig. 5(a). As a result, the temperature difference between the specimen and the probe decreased from 200 to 30–50°C when the test part length was 120–600 µm, whereas the maximum temperature at the Mo plate decreased to 600°C. In this paper, we report the test results at 600°C under the modified heating condition. Under this condition, the measured temperature at the specimen chip and the grip were 577 and 518°C, respectively (Fig. 5(b)).

4.2 *Stress-stage displacement curves*

Figure 6 shows the stress-stage displacement curves of the specimen tested at 600°C when the stage displacement rate was 0.2 µm/s. The calculated strain rates of specimens 120 and 600 µm long were 3.65 and 1.94×10^{-4}/s, respectively. The tensile stress increased linearly with the stage displacement. The specimens deformed elastically and fractured as brittle materials. Tensile testing was carried out successfully at 600°C.

As shown in Fig. 6, small signs of yield were observed immediately prior to fracturing when the length of the test part was 600 µm. Therefore, the stage displacement rate was changed from 0.2 to 0.04 or 0.08 µm/s because we considered that the strain rate had been too high for the specimens to show ductile behavior. Figure 7 shows the stress-stage displacement curves of the specimens whose test part lengths were 120 and 600



(a) (b)

Fig. 5. (a) Top view in chamber. (b) Measured temperature while the Mo plate temperature was maintained at 600°C.

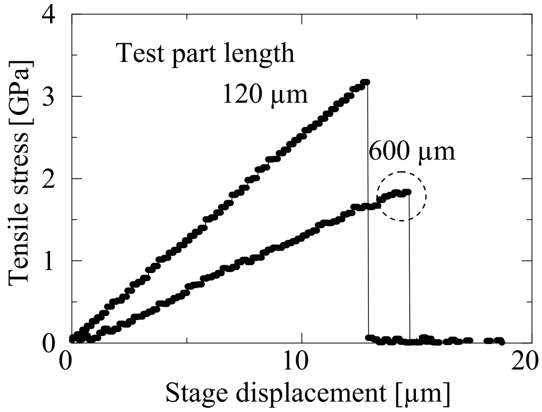


Fig. 6. Stress-stage displacement relationships of SCS films tested at 600°C. The stage displacement rate was 0.2 µm/s.

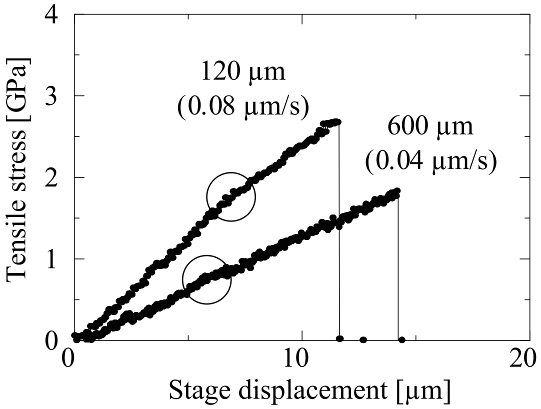


Fig. 7. Stress-stage displacement relationships of SCS films tested at 600°C. The stage displacement rates were 0.04 and 0.08 µm/s.

µm and stage displacement rates were 0.04 and 0.08 µm/s, respectively. The calculated strain rates of the 120- and 600-µm-long specimens were 1.63×10^{-4} and 4.11×10^{-5}/s, respectively. Each plot, where the stage displacement rates were 0.04 and 0.08 µm/s, showed a yield point in the middle. This shows that the specimens deformed plastically. From Figs. 6 and 7, the yield point did not exist when the strain rate was 3.65×10^{-4}/s but the points did exist at 1.9, 1.8, and 0.8 GPa in tensile stress when the strain rates were 1.94×10^{-4}, 1.63×10^{-4}, and 4.11×10^{-5}/s, respectively. The threshold stress at the yield point depends on the strain rate.

4.3 *Mechanical properties*

Figure 8 shows the measured fracture strength of SCS films as a function of temperature. The average fracture strength was 3.33 GPa at RT and 2.71 GPa at 600°C, which indicates a 19% decrease in strength at 600°C. The Weibull plots of the fracture strength are shown in Fig. 9. The fitted Weibull moduli were 5.53 and 5.37 at RT and 600°C, respectively.

The Young's modulus was calculated using the differential method, in which the elongation of the test part was measured from the difference in the slopes of the stress-stage displacement plots of 120- and 600-μm-long specimens adjacent on the same test chip. Figure 8 shows the Young's modulus as a function of the testing temperature. The averaged Young's modulus was 163.2 GPa at RT and 151.8 GPa at 600°C. The Young's modulus at 600°C decreased by 7.0% compared with that at RT. In the case of bulk silicon, the theoretical values are 168.9 and 160.5 GPa at RT and 600°C, respectively, which is a 5.0% decrease.(5)

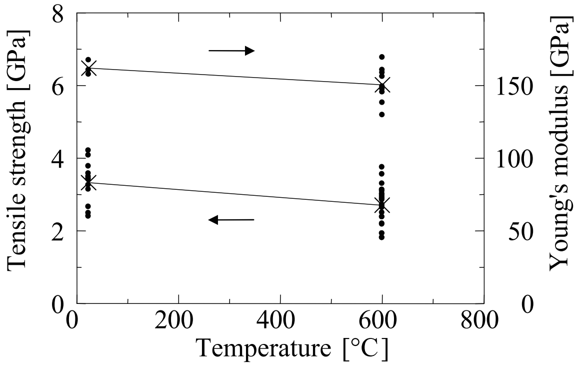


Fig. 8. Fracture strength and Young's modulus of SCS films as a function of temperature.

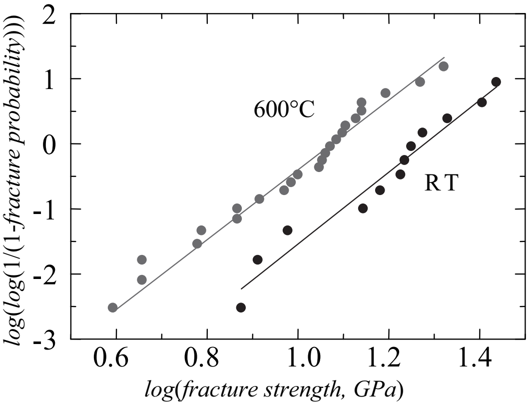
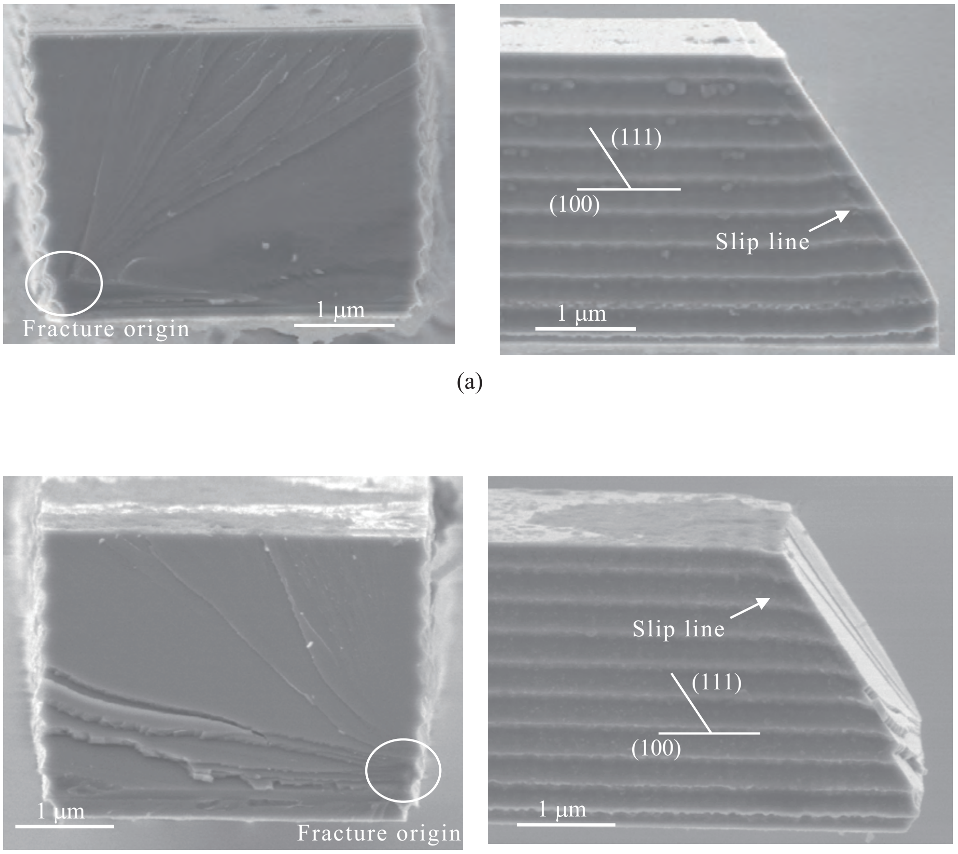


Fig. 9. Weibull plots of tensile strength of SCS specimens tested at RT and 600°C.

4.4 *Fractography*

The fracture surfaces were observed by scanning electron microscopy (SEM). The fractured specimens had no slip line at RT or 600°C when the stage displacement rate was 0.2 µm/s. On the other hand, the fractured specimens had step and slip lines when the testing temperature was 600°C and the stage displacement rates were 0.04 and 0.08 µm/s, as shown in Fig. 10. The slip plane seemed to be (111) oriented. The fracture origin was at the bottom corner and the fracture surface was in the (111) plane. One of the edge scallops produced during reactive ion etching acted as a notch and the specimen was cleaved from the notch to the (111) plane, which has the lowest surface energy among silicon crystal faces.(6)



(a)

(b)

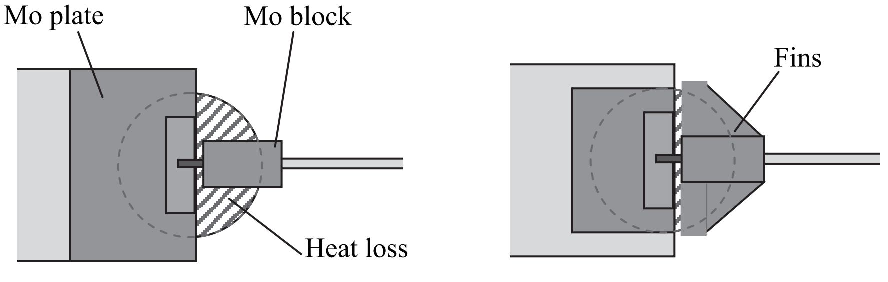
Fig. 10. Fractured surfaces of SCS specimens tested at 600°C. The stage displacement rate was (a) 0.04 and (b) 0.08 µm/s.

5. Discussion

5.1 *Specimen heating*

The developed specimen heating system can heat the specimen to 800°C in air before chucking the specimen's free end to the electrostatic grip. In the initial setup of the tester, since the Mo block was not heated well, the temperature at the grip was only 200°C, which caused cooling of the specimen parallel part. After reconfiguration of the tester to equalize the temperature of the plate and grip, the temperature difference between them was substantially reduced. All the tests were carried out under this condition. However, the difference between both ends ranged from 30°C for the shorter specimen to 50°C for the longer specimen. The tester should be improved for better uniformity in temperature as well as highest reachable temperature.

A schematic drawing of the configuration around the specimen is shown in Fig. 11. To heat the grip, the IR ray irradiates both the plate and grip, which causes heat loss from the IR source, as indicated by the hatched area in Fig. 11(a). In addition, the plate was larger than the specimen and had a high heat capacity. The loss from the surface of the plate might be sufficiently high to limit the maximum temperature. Therefore, the lateral dimensions of the Mo plate were decreased to reduce the heat capacity and surface area. Fins were attached to both sides of the Mo block to reduce heat loss. The positions of the plate and grip were optimized to equalize the temperature of both ends, as shown in Fig. 11(b). As a result, the temperature difference was reduced to 5–25°C. However, the temperature of the specimen was about 550°C, which was not improved. The reason for this might be the surface oxidization of the molybdenum parts, which deteriorates the heat absorption. Therefore, parts made of ceramic materials should be used.



(a) (b)

Fig. 11. Modification of the plate and block for improving the temperature uniformity. (a) Original parts. The temperature difference was 30–50°C. (b) Modified parts. The temperature difference was 5–25°C.

5.2 *Properties of SCS at 600°C*

We observed the slope change in the stress-stage displacement curve, which indicates the ductile deformation of the SCS specimens. This was confirmed by SEM observation of the fractured specimens that showed slip lines near the fracture surfaces, as shown in Fig. 10. This slope change that is similar to the results in previous works,(1,3) shows the work-hardening in silicon. The mechanism of the work-hardening has not been clarified, but one possible explanation is that the oxide impurities segregate in silicon crystals and the oxide segregations are presumed to act as obstacles to dislocation motion. The yield stress of the SCS at high temperatures depended on the strain rate. Table 2 shows a summary of the results shown in Figs. 6 and 7. Such strain rate dependence on yield stress was observed in ductile metal films.(7) Further investigation on the dependence of temperature and strain rate is required to determine the mechanisms of silicon ductile behaviors.

In the RT environment, the measured Young's modulus agreed well with that of bulk SCS and the observed fracture strength of the SCS specimen was similar to that in previous works, which indicate that the tester was properly operated. The measured decrease in the modulus at 600°C was slightly larger than that of bulk silicon. It is possible that the ductile deformation caused this difference, although the modulus was calculated at a stress lower than the yield stress. The measured decrease in strength may be caused by the ductile deformation.

We could not find any influence of oxidation through this experiment, although silicon is said to be oxidized above 600°C. The reason for this is the short testing time of about ten minutes including the time to heat a specimen. The surface of silicon is usually covered with a native oxide film whose thickness is one to several nanometers. The thickness of oxide grown at 600°C at atmospheric oxygen pressure is only ~1 nm.(8) It is difficult to discuss the influence of the oxidization, since this thickness is comparable to that of the native oxide layer.

Table 2
Measured yield stress for different specimen lengths and strain rates.

Length (μm)	Stage disp. (μm/s)	Strain rate (/s)	Yield stress (GPa)
120	0.2	3.65×10^{-4}	>3.2
600	0.2	1.94×10^{-4}	1.9
120	0.08	1.63×10^{-4}	1.8
600	0.04	4.11×10^{-4}	0.8

6. Conclusion

We have developed a high-temperature thin-film tensile tester and demonstrated the tensile testing of SCS films at 600°C. The specimens were heated to 800°C using IR heating. The mechanical properties of SCS films were evaluated at 600°C. The fracture strengths at RT and 600°C were 3.33 and 2.71 GPa, respectively, and the Young's moduli were 163.2 and 151.8 GPa, respectively. The fracture strength and Young's modulus decreased at 600°C compared with those at RT and the temperature coefficient of Young's modulus agreed with that of bulk silicon. When the strain rate was low, the stress-stage displacement curves had yield points and the fractured specimens at 600°C exhibited slip lines.

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