TRANSDUCERS & EUROSENSORS '07
The 14th International Conference on Solid-State Sensors, Actuators and Microsystems, Lyon, France, June 10-14, 2007

EFFECTS OF ENVIRONMENTAL CONDITION ON THE STRENGTH OF SUBMICRON-THICK SINGLE CRYSTAL SILICON FILM

S. Nakao[1], T. Ando[1], M. Shikida[2] and K. Sato[1]
[1]Department of Micro-Nano Systems Engineering, Nagoya University, Nagoya, JAPAN
[2]EcoTopia Science Institute, Nagoya University, Nagoya, JAPAN

Abstract: We developed a quasi-static tensile test system that controls environmental conditions, such as pressure, temperature, and surrounding gasses. Using this system, we evaluated the fracture properties of micron- and submicron-thick single-crystal-silicon film under several conditions. The strength of silicon measured in vacuum or helium was slightly higher than that in laboratory air. We measured the fracture toughness at different temperatures ranging from room temperature (RT) to 500ºC and found a brittle-to-ductile transition at 70ºC for micron-sized silicon film. The fracture toughness drastically increased at the transition temperature and saturated at a level of 2.5 MPa$\sqrt{m}$, which is twice the value at RT. On the other hand, submicron-thick silicon was less brittle: its fracture toughness was already 2.7 MPa$\sqrt{m}$ at RT.

Keywords: single crystal silicon, fracture strength, size effect, environmental effect

1. INTRODUCTION

In order to make reliable MEMS devices, we need sufficient knowledge about the mechanical properties of functional thin films. In previous studies by several researchers, the properties of thin films showed some differences from those of the bulk materials because of size effects [1, 2]. Other studies confirmed that the fabrication processes also affect the strength of thin materials due to differences in the surface conditions [3, 4]. Thus, there are many factors that can affect the mechanical properties of thin film materials. This is essential information for MEMS producers trying to commercialize these devices.

In addition, the applications of MEMS technology are spreading in many fields, and the devices are being used under various environmental conditions. Many researchers are therefore concerned about the effects of environmental factors on the mechanical properties of thin films. Some groups have evaluated the effects of temperature on the mechanical properties of thin films [5, 6]. Other groups have reported that the fracture strength and fatigue life are affected by humidity [7, 8]. However, the fracture mechanism has not been clarified yet.

In this paper, we report developing a tensile testing system that can control the pressure (from vacuum to atmosphere), temperature (from RT to 500ºC), and ambient gas (N_2, Ar, He, etc.) in the chamber. We evaluated the static fracture strength of submicron-thick single-crystal-silicon films in laboratory air, vacuum, and helium and evaluated fracture toughness at different temperatures in laboratory air. The effects of film thickness and environmental conditions on the strength and toughness of the film specimens are discussed.

2. MATERIALS AND TESTING METHOD

2.1 "On-chip" tensile testing method

We used the "on-chip" tensile testing method to evaluate the fracture properties of submicron-thick films (Fig. 1) [5]. The test device was fabricated on two types of SOI wafers, which had top layers 2 and 5 μm thick, respectively. The specimen was fabricated in the top layer by RIE, and two steps of wet etching formed a load lever and torsion bars that worked as a loading system. The test device was cut in a 15 mm square.

When an external load was applied to the load lever in the vertical direction, the lever rotated around the axis of the torsion bars and the

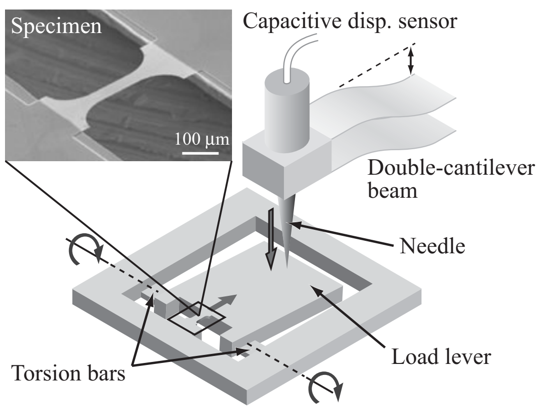


Fig. 1 "On-chip" tensile testing method.

1-4244-0842-3/07/$20.00©2007 IEEE

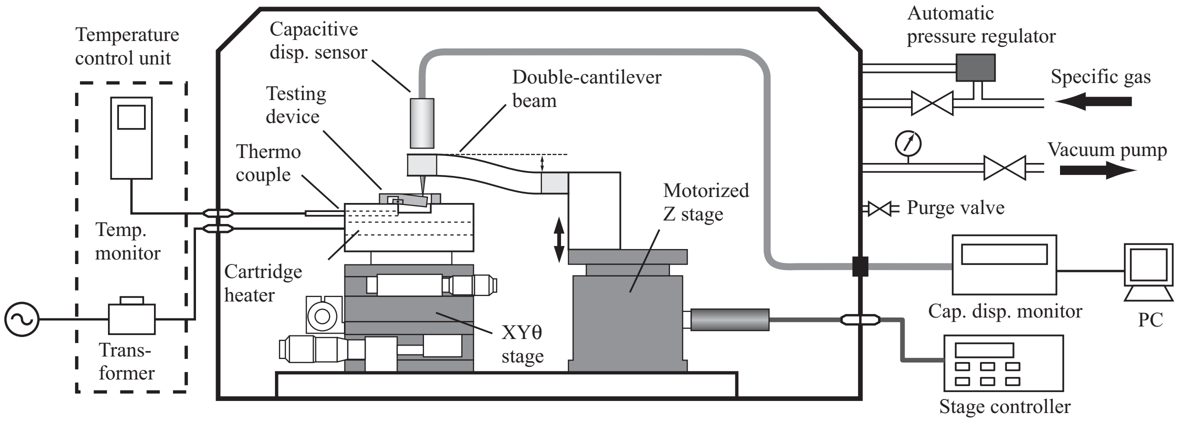


Fig. 2 Environmental control system.

specimen was stretched. The tensile force could be calculated from the applied load by subtracting the repulsive force of the torsion bars, which was measured independently after the specimen was broken.

2.2 Tensile test specimens

We used single-crystal-silicon film specimens having a (100) surface orientation and a tensile direction of <110>. The specimen length was 50 or 100 μm and the width was about 45 μm. The thickness was thinned from 2 and 5 μm to 0.17–1.03 and 3.7–4.6 μm, for submicron- and micron-thick specimens, respectively, by two steps of thermal oxidization and etching of the thermal oxide layer. When we evaluated the fracture toughness, a notch was introduced on one side of the straight portion using a FIB. The notch length was about 1–2 μm.

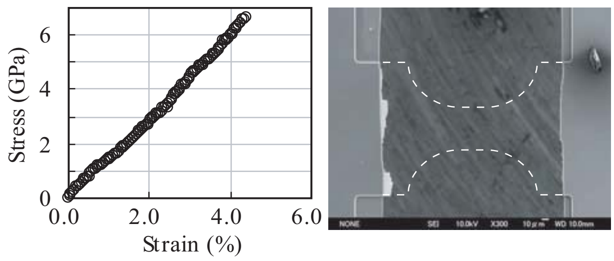
2.3 Environmental control system

The developed tensile testing equipment in a controlled environment is schematically shown in Fig. 2. The test device is clamped on the heating stage, and the temperature can be controlled by means of the heater current. We can change the environment in the chamber from laboratory air to a vacuum of lower than 13.3 Pa. Various kinds of gasses can be introduced in the chamber. The double-cantilever beam for applying the external load is controlled by remotely operating the z-stage. The force can be calculated from the deflection of the double-cantilever beam and the beam spring constant, which is hardly influenced by the environmental condition.

3. FRACTURE STRESS

3.1 Test procedure

We evaluated the fracture stress of submicron-



(a) Stress-strain relationship *(b) fractured specimen*

Fig. 3 Results of tensile test in vacuum.

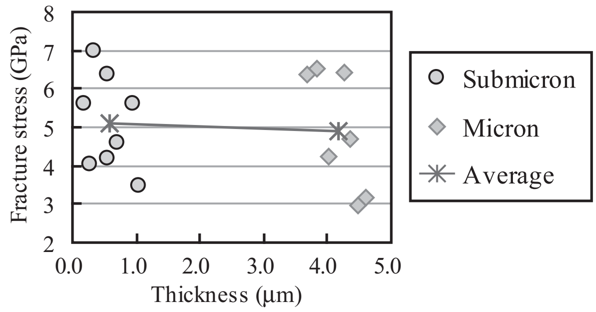


Fig. 4 Relationship between fracture stress and thickness.

thick single-crystal-silicon films in laboratory air, vacuum, and helium. For the tests in vacuum and helium, the test chamber was evacuated, while the test device was baked at 150ºC to evaporate the adsorbed water. After cooling the stage down to 15ºC in vacuum, or blowing helium gas into the chamber, we evaluated the fracture stress in each environment. The tests were carried out successfully in each condition. The specimen was brittle fractured, and a major portion was lost as a result of the fracture (Fig. 3).

3.2 Relationship between fracture strength and specimen thickness

We compared the fracture strength of submicron-thick (average thickness: 0.574 μm) specimens and that of micron-thick (average thickness: 4.17 μm) specimens in laboratory air, as

TRANSDUCERS & EUROSENSORS '07
The 14th International Conference on Solid-State Sensors, Actuators and Microsystems, Lyon, France, June 10-14, 2007

1-4244-0842-3/07/$20.00©2007 IEEE

TRANSDUCERS & EUROSENSORS '07
The 14th International Conference on Solid-State Sensors, Actuators and Microsystems, Lyon, France, June 10-14, 2007

shown in Fig. 4. The results were widely scattered because of their brittleness. The average values for submicron- and micron-thick specimens were 5.12 and 4.92 GPa, respectively. We found that the submicron-thick specimens showed slightly higher strength than the micron-thick specimens.

3.3 Environmental effect on the film strength

Table 1 shows the average fracture stress of submicron-thick single-crystal-silicon films in laboratory air, vacuum, and helium. The average values in vacuum and helium were higher than that in laboratory air. The similarity between the results for vacuum and helium was attributed to the low humidity in both these environments. It has been reported that a high-humidity environment leads to lower fatigue strength and shorter life [7, 8]. Our results show that the static strength of silicon film is also decreased by H_2O in the air.

4. FRACTURE TOUGHNESS

4.1 Thickness dependence of fracture toughness

We evaluated the fracture toughness of submicron- and micron-thick single-crystal-silicon films in laboratory air and compared the results. The average values for the submicron- and micron-thick specimens were 2.71 and 1.28 MPa $\sqrt{m}$, respectively. The submicron-thick specimens were twice as tough as the micron-thick ones. One reason for this may be that the stress state transited from plane strain to plane stress at a notch tip. Another possibility cause is the behavior of dislocations, as described in the following section.

4.2 Micron-thick specimens at high temperatures

We also measured the fracture toughness of micron-thick specimens at high temperatures. Fig. 5 shows the fracture toughness of micron-thick specimens from RT to 500ºC and of a submicron-thick specimen at RT. The fracture toughness of the micron-thick specimens drastically increased at 70ºC to a value about double that at RT. This increase in toughness shows that the fracture mode clearly changed at 70ºC. We observed TEM images of specimens fractured at RT and 300ºC and compared the dislocation patterns. The fractured specimens were thinned to 200–300 nm using FIB apparatus to prepare samples for TEM observation. Fig. 6 shows TEM images (bright

Table 1. Average values of fracture stress in each environment.

Environment	Lab. Air	Vaccum	Helium
Test number	8	7	6
Average strength (GPa)	5.12	5.65	5.77

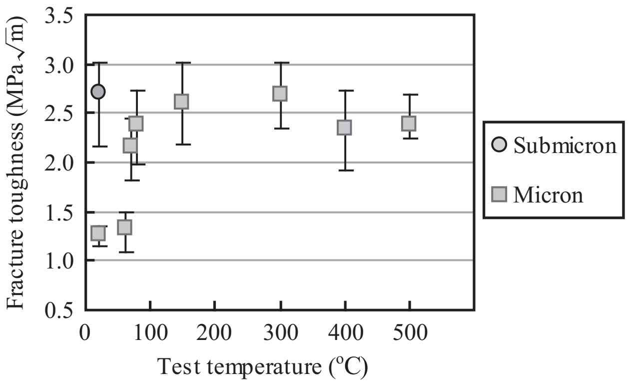


Fig. 5 Relationship between fracture toughness and temperature.

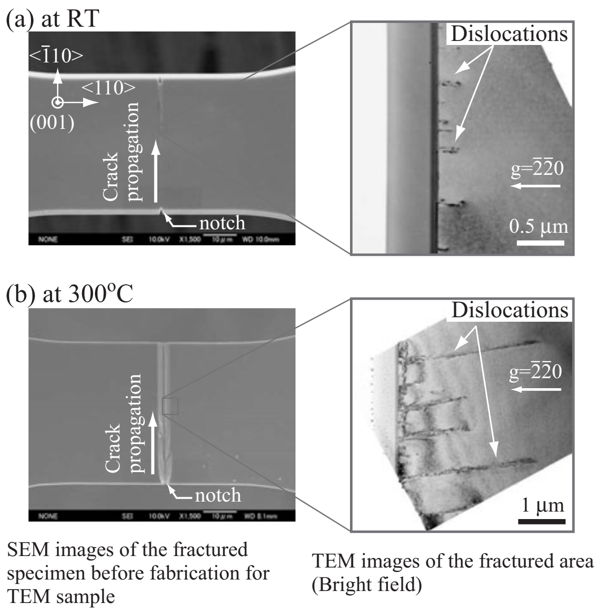


Fig. 6 TEM images of micron-thick specimens at RT and 300ºC.

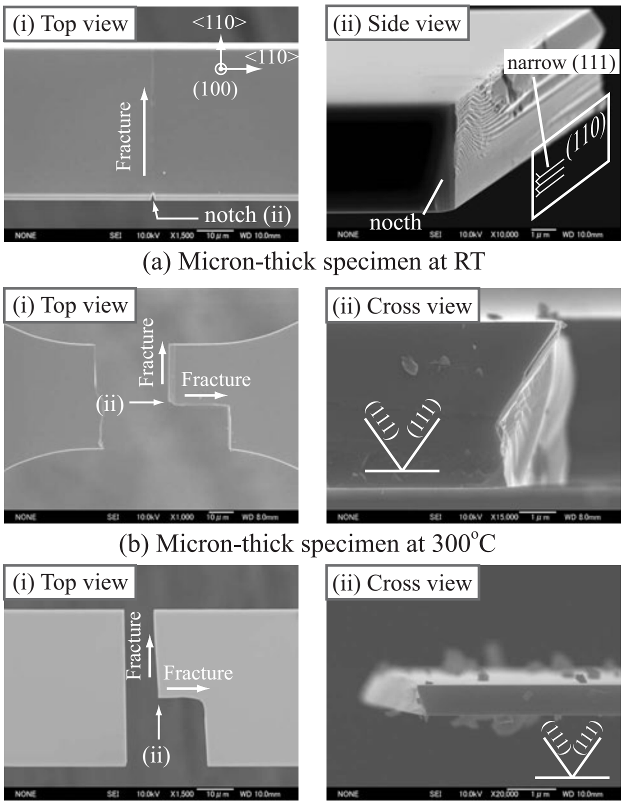
field) of specimens fractured at RT and 300ºC when the incident beam direction was [001] and the diffraction vector was set to $[\bar{2}20]$. Some small dislocations with a length of 200–300 nm were induced from the fracture surface in the tensile direction at RT. On the other hand, they spread to 2–3 µm in length at 300ºC. The dislocations observed in a specimen fractured at 300ºC grew ten times longer than at RT. The more active movement of dislocations at elevated

1-4244-0842-3/07/$20.00©2007 IEEE

temperature seems to have contributed to the relaxation of the stress concentration in the microstructure at the crack tip, so the micron-thick silicon film changed fracture mode at 70ºC.

4.3 Submicron at RT and micron at high temperatures

On the other hand, the submicron-thick specimen showed high toughness even at RT. The value was almost the same as for micron-thick specimens at high temperatures. Moreover, we could see by SEM observation that the specimen fractured with similar morphology to micron-thick specimens at high temperatures. Fig. 7 shows SEM images of fractures of micron-thick specimens at RT and 300ºC and of the submicron-thick specimens at RT. The 4-µm-thick specimen at RT was fractured from notch tip straight along the (110) plane composed of narrow inclined (111) planes. However, it fractured along with large (111) planes, and cracks propagated in the tensile direction on many fractures at high temperatures. The submicron-thick specimen at RT fractured in a very similar shape to those of micron-thick specimens at high temperatures. Although there is



(a) Micron-thick specimen at RT

(b) Micron-thick specimen at 300°C

(c) Submicron thick specimen at RT

Fig. 7 Fractured specimens of micron-thick specimen at RT and 300ºC and submicron-thick specimen at RT.

clearly some relationship between temperature and thickness effect on the fracture toughness, a more detailed analysis of these results is needed.

5. CONCLUSION

An environmental control system that can control the pressure, temperature, and ambient gas has been developed for "on-chip" tensile test. Using this system, we evaluated the fracture strength of submicron-thick single-crystal-silicon films in laboratory air, vacuum, and helium. In laboratory air, the strength of submicron-thick film was slightly higher than that of micron-thick film. In addition, we could see the effect of the environment on the static strength: the average strength in vacuum and helium were higher than that in laboratory air.

We also evaluated the fracture toughness of a submicron-thick specimen at RT and micron-thick specimens at high temperatures. We found a transition in fracture toughness value at 70ºC with micron-thick specimens. The submicron-thick specimen showed fracture toughness values equivalent to those at high temperatures with micron-thick specimens, even though they were tested at RT.

ACKNOWLEDGEMENT

This work was partially supported by the 21st COE program (Micro- and Nano-Mechatronics for Information-based Society) from the Ministry of Education, Culture, Sports, Science, and Technology, Japan.

REFERENCES

[1] T. Tsuchiya et al., J. Microelectromech. Syst., Vol. 7, No. 1, pp. 106-113 (1998).
[2] W. N. Sharpe, Jr. et al., J. Microelectromech. Syst., Vol. 10, No. 3, pp. 317-326 (2001).
[3] T. Yi et al., Sens. Actuators A, vol. 83, pp.172-178 (2000).
[4] K. -S. Chen et al., J. Microelectromech. Syst., Vol. 11, No. 3, pp. 264-275 (2002).
[5] S. Nakao et al., J. Micromech. Microeng., Vol. 16 no. 4, pp.715-720 (2006).
[6] T. Namazu et al, J. Microelectromech. Syst., Vol.11, no.2, pp.125-134 (2002).
[7] C. L. Muhlstein et al., Sens. Actuators A, Vol. 94, pp. 177-188 (2001).
[8] Y. Yamaji et al., Proc. MEMS 2007, Kobe, Japan, Jan. 21-25 2007, pp. 267-270.

1-4244-0842-3/07/$20.00©2007 IEEE