SPECIAL LECTURE

ENVIRONMENTALLY ASSISTED FRACTURE BEHAVIOR OF SILICON MICROELEMENTS

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ABSTRACT

The influence of notch and water environment on the quasi-static and fatigue fracture behavior was investigated in single crystal silicon microelements. The tests were conducted in smooth and notched microcantilever beam samples. Single-crystal Si microelements deformed elastically until final failure, giving a brittle nature. The maximum fracture strength of a smooth microcantilever specimen reached about 7.7 GPa. However, the fracture strength decreased with an increase in notch depth, even though the notch depth was on the order of nanometer. In laboratory air, no fatigue damage was observed even though the surface was nanoscopically examined by an AFM. However, when the fatigue tests were conducted in pure water, the fatigue lives in water were decreased. Atomic force microscopy was capable of imaging nanoscopic cracks, which caused the failure in water.

KEYWORDS

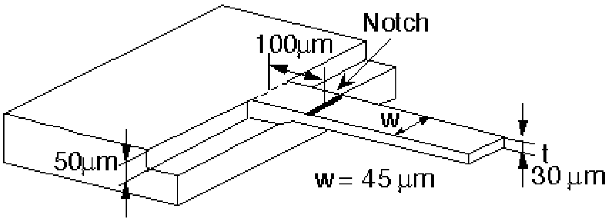
Single-crystal silicon, Fracture, Fatigue, Notch, Focused ion beam, Water, Fractography, Atomic force microscopy

INTRODUCTION

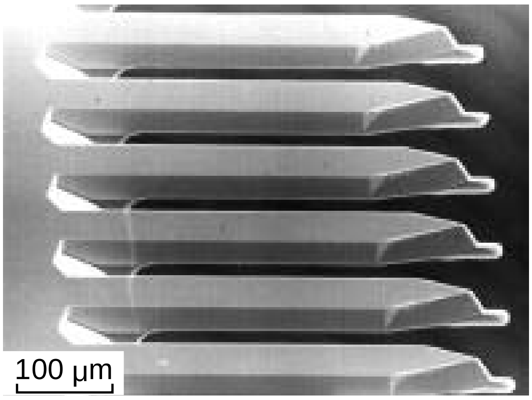
In order to develop a reliable micromachine in a service operation, much care must be taken not only to processing methods, but also to micromechanical evaluation, i.e., mechanical properties of μm-sized microelements including fatigue and wear. For mechanical evaluation, Johansson and Schweitz [1] showed that single-crystal silicon microelements on the order of µm have an extremely high bending strength over 10 GPa. The authors have also developed a specially designed testing machine for microelements [2], and have shown that bending strength increases with a decrease in specimen size, and the maximum bending strength obtained reaches about 8 GPa [3].

Beside these, fatigue behavior of microelements is also very important, when they are used in micromachines and micro electro-mechanical systems (MEMS) that are subjected to varying loads. The authors [3] have conducted fatigue tests in single-crystal Si microelement under three-point loading, and have shown that contact forces that work between a loading stylus and a sample promote crack initiation, and cause so-called fretting-fatigue. However, when a microelement is subjected to a simple bending in laboratory air, no fatigue damage is observed even though a sample surface is closely examined by an atomic force microscope. Note that fatigue behavior is observed when tests are conducted in water. Similar results such that water environments induce damage in single crystal silicon microelements were also demonstrated by Conally et al. [4, 5]. Our nanoscopic observation[3] showed that a crack of about 20 nm deep existed, which might be promoted by a synergistic effect of water and dynamic loading. Beside these, mechanical properties of single-crystal Si are very much sensitive to processing methods, and this is attributed to surface roughness. Therefore, it is very important to analyze the effects of nanometer-sized notch on the fracture behavior of a microelement.

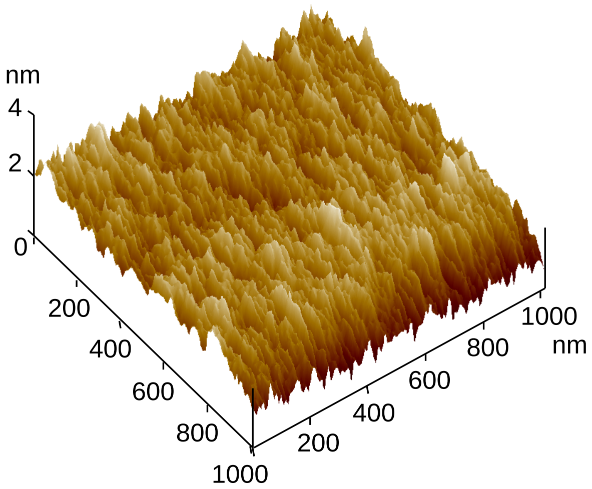
In this investigation, mechanical tests including fatigue were conducted in single-crystal Si microelements, and special attention was paid to the effects of nanometer-sized notch and of water on the quasi-static and fatigue strength.



(a) Schematic view



(b) SEM image



(c) AFM image of a microcantilever beam specimen (polished surface)

Fig.1 Microcantilever beam specimens.

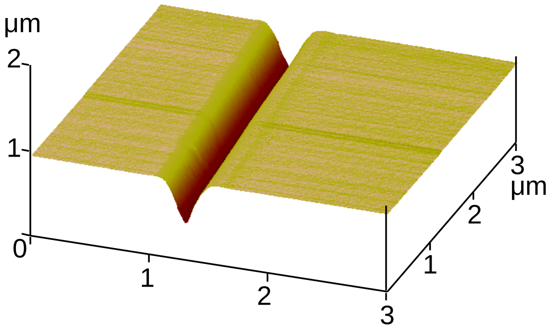


Fig.2 AFM image of V-shaped notch.

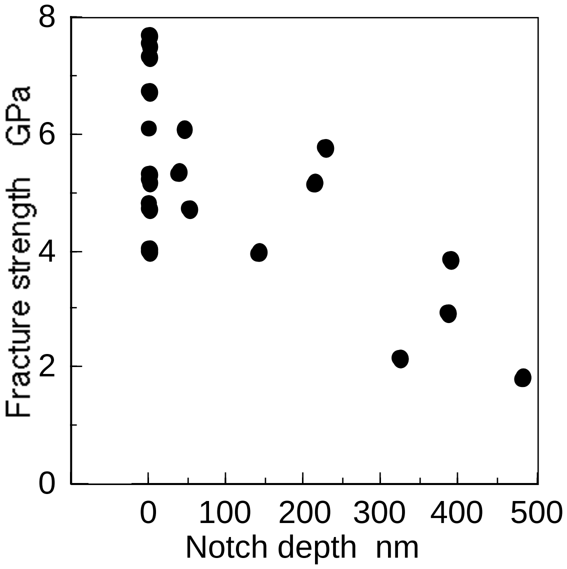


Fig.3 Fracture strength of single crystal as a function of notch depth.

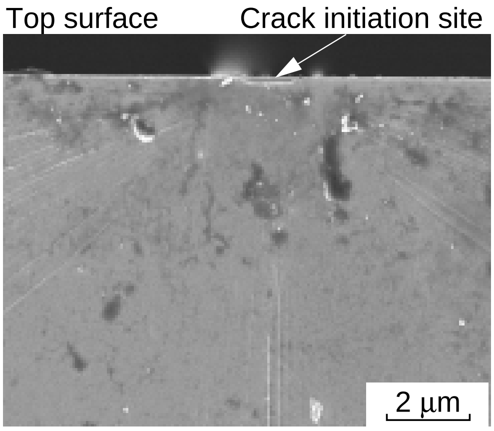


Fig.4 Quasi-static fracture surface of a notched specimen, imaged with SEM.

EXPERIMENTAL PROCEDURES

Single-crystal Si microcantilever beam specimens (Fig. 1) were prepared by micromachining (photo-etching) of (110) silicon wafers [3]. They were oriented along a <112> direction with a rectangular cross section. The sample used in this experiment was 30 µm thick, 45 µm wide, and 500 µm long. For some specimens, nanometer deep notch was milled 100 µm away from a sample root (Fig. 1(a)) by using a focused ion beam (FIB, ion source:Ga^+). Figure 2 illustrates AFM images of the cross-section of a V-shaped notch thus introduced by FIB milling. Bending force is applied to a microelement by a stylus positioned at the end of the actuator, which is a spherical diamond tip of 20 µm radius and 60° cone angle.

EXPERIMENTAL RESULTS AND DISCUSSIONS

Quasi-Static Fracture Strength and Influence of Notch

The load-displacement curve of a microcantilever with and without notch was linear until final catastrophic failure, indicating that single crystal Si microelement behaves elastically until final catastrophic failure. In the case of smooth microcantilever specimen [3], the fracture strength had large scatter band. The minimum fracture strength remained about 2 GPa, irrespective of specimen width ranging from 45 µm to 195 µm with thickness of 30 µm. However, the maximum or average fracture strength of each set of data increased with a decrease in specimen width. The maximum strength in all test results was 7.7 GPa, which was higher than that of mm order single crystal Si specimens machined by dicing which were subjected to four-point bending. The maximum fracture strength obtained in mm-sized sample was only 600 – 800 MPa [2].

Figure 3 shows the fracture strength as a function of notch depth. The fracture strength decreased with an increase in notch depth, even though the notch depth was on the order of nanometer.

Figures 4 shows the micrographs of the failed notched microcantilever sample. From the radial markings we could thus trace back to the initiation site, where a notch existed. Beside these, the fracture surface was normal to the top surface of a sample, and the intersection of the fracture surface and the top surface was straight and was about 71 to 72 degrees against the longitudinal direction, or <112> direction. A crack that nucleated at a notch propagated on a {111} plane, resulting in normal crack to the top surface.

Fatigue Fracture Behavior in Air

Figure 5 shows fatigue lives of notched microcantilevers in laboratory air as a function of notch depth. The applied load was 100 mN or 50 mN, which was loaded 350 μm away from the specimen root. These gave the nominal maximum stress of 3.7 GPa and 1.9 GPa at a notch position, respectively. From this figure, it is shown that the fatigue lives were more than about 5 x10^4 cycles below a certain notch depth. However, it became extremely small when the notch depth exceeded a threshold value. The threshold value was smaller when an applied load was larger.

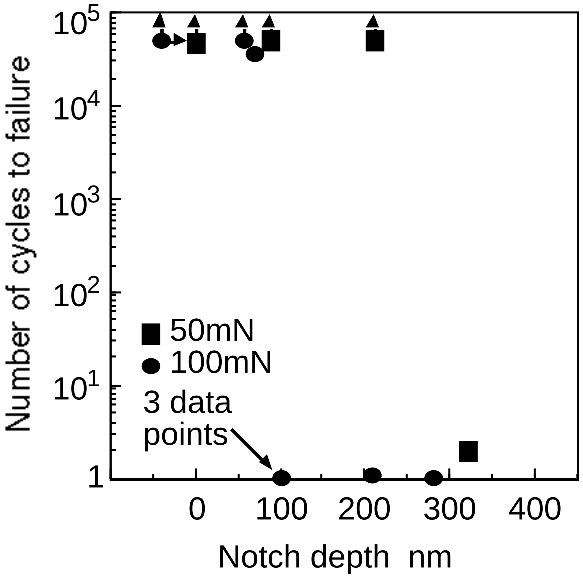
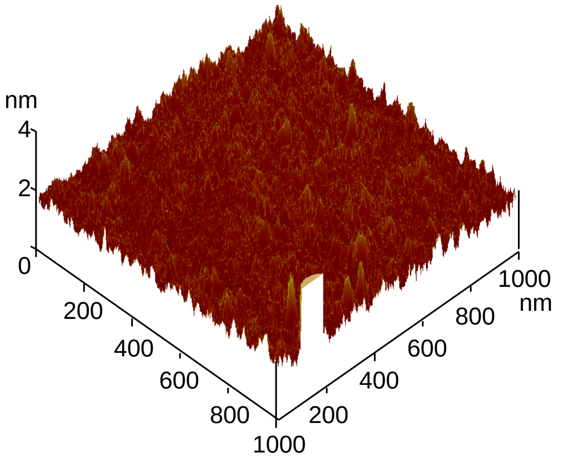
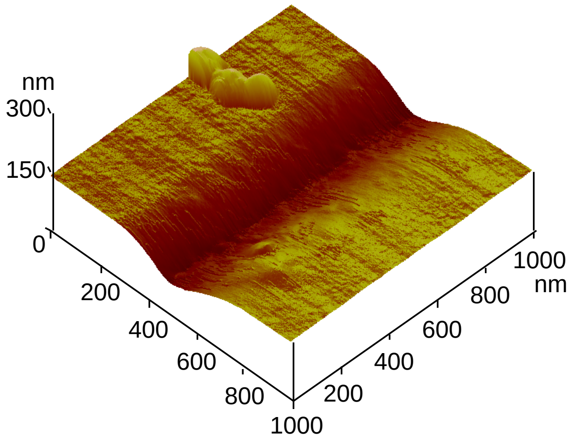


Fig.5 Fatigue strength in laboratory air as a function of notch depth.

Figure 6 shows an AFM image of top surface near and at a notch of a fatigued sample. The roughness of the fatigued sample surface was R_a=0.19 nm that was the same as that of the polished surface before fatigue tests. Nanoscopic fatigue damage could be seen neither near nor at a notch, indicating that single crystal Si might be immune to fatigue fracture in laboratory air.



(a) top surface near a notch
(notch depth: 68 nm, N= 3.6 × 10^4)



(b) notch (notch depth: 55 nm, N > 5 × 10^4)

Fig.6 AFM image of fatigued samples in laboratory air.

Influence of Water on Fatigue Fracture Behavior

Figure 7 shows fatigue lives in pure water of smooth and notched cantilever specimens as a function of pre-immersion time in water. The applied load was 100 mN, which gave the maximum stress of 3.7 GPa at a notch position. In this investigation, the water environment was achieved by placing wet absorbent cloth at a specimen root, and therefore, the adjacent microcantilevers to the testing one were also exposed to water. This means that the microcantilever fatigued later was immersed longer in water than the sample fatigued earlier. Therefore, the

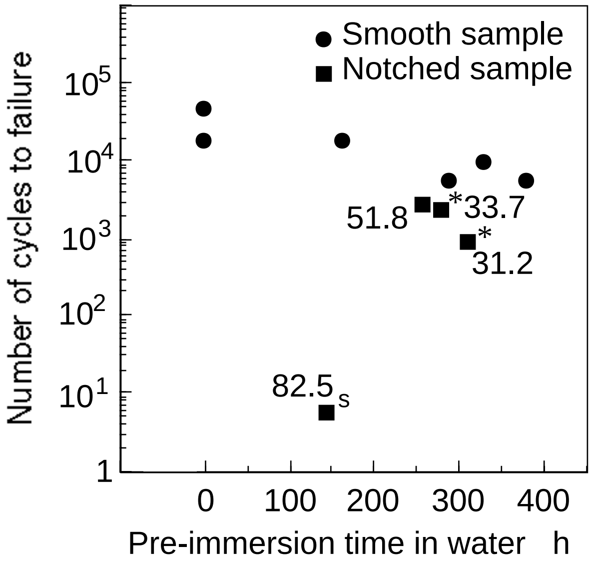


Fig.7 Fatigue strength in pure water as a function of pre-immersion time in water. (R = 0.1, f = 0.1Hz).

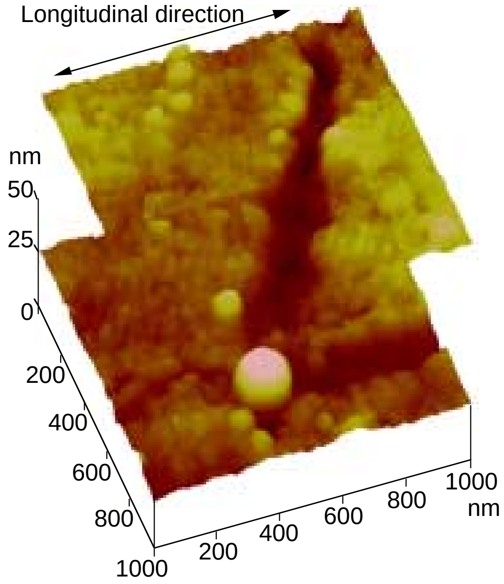


Fig.8 Fatigued sample imaged with AFM. The observed area is the smooth surface near a notch.

fatigue lives are plotted against pre-immersion time in water. The numbers in the figure indicate the notch depth in nm. Except the case with a superscript “s”, the notch depth machined was smaller than the threshold notch depth for fatigue in air.

For smooth cantilevers, the fatigue lives in water were shorter than those fatigued in air and became slightly shorter when the immersion time became longer. When a notch depth was large as is shown by a superscript “s”, the fatigue life was extremely short. In this case, the notch depth was almost equal to the threshold notch depth, above which static failure occurred, indicating that the failure was caused by the quasi-static one induced by stress concentration. Except this, similarly to the smooth microcantilevers, the fatigue lives of notched cantilevers were smaller than 3000 cycles, indicating shorter fatigue life than that conducted in air.

In the case of notched samples under quasi-static loading, a crack always initiated at a notch because of stress concentration. However, when a notch depth was smaller than a threshold depth below which no fatigue failure in air occurred, some sample failure in water was not associated with a notch. In Fig. 7, a superscript “*” is added when failure was not associated with a notch.

Figure 8 shows the notched sample surfaces fatigued in water, imaged by the AFM. The failure of the sample was not associated with a notch. The AFM tip used was a super sharp type with tip radius of about 5 nm. The groove type crack of about 16 nm in depth could be seen, and the direction of this crack was about 70 degrees against the longitudinal or <112> direction. This nanoscopic crack orientation agreed with that of the static failure, or {111} plane.

These observations indicate that a synergistic effect of fatigue loading and water environment caused a nanoscopic crack, that oriented along {111} plane. The crack gradually propagated, and when its depth exceeded the critical depth, unstable or catastrophic failure occurred. Single-crystal Si was reported insensitive to water environment, and stress corrosion cracking does not occur [6]. In a microelement, however, we must note that crack initiation and propagation are promoted by a synergistic effect of fatigue loading and water environment [3, 4, 5].

CONCLUSIONS

Mechanical tests including fatigue were conducted in single-crystal Si microelements fabricated by photoetching using a specially designed testing machine that enables mechanical testing including fatigue. Special attention was paid to the effects of nanometre-deep notch machined by FIB and of water environment on fracture. The investigation yielded the following conclusion.

1. Focused ion beam is suitable processing method for machining a nanometre-deep notch. The cross-section is V-shaped, and the radius of the curvature decreases with an increase in notch depth. It ranges from about 20 nm to 100 nm, when notch depth exceeds about 100 nm.
2. Single-crystal Si microelements deform elastically until final failure, giving a brittle nature. However, they are sensitive to notch, and manometer-deep notch causes a decrease in the fracture strength.
3. For notched samples, a quasi-static fracture initiates at a notch, and a crack propagates in the direction normal to the sample surface on a {111} plane.
4. When fatigue load is applied in air, no fatigue damage is observed even though the surface is nanoscopically examined by an atomic force microscope. However, the fatigue lives in water are decreased compared with those conducted in air. An atomic force microscope is capable of imaging nanoscopic cracks on a {111} plane. Depth of a crack observed on a fracture surface was long enough to cause unstable fracture. These mean that a synergistic effect of fatigue loading and water causes a nanoscopic crack, and when its length, or depth, exceeds a critical length, catastrophic failure occurs, leading to lower fatigue lives in water.

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