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Fracture behavior of single crystal silicon with thermal oxide layer

Toshiyuki Tsuchiya *, Kenji Miyamoto, Koji Sugano, Osamu Tabata

Department of Micro Engineering, Kyoto University, Kyotodaigaku-Katsura C3, Nishikyo-ku, Kyoto 615-8540, Japan

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This paper reports on the effect of oxidation on fracture behavior of single crystal silicon (SCS). SCS specimens were fabricated from (100) silicon-on-insulator wafer with 5-μm-thick device layer and oxide layer were thermally grown. Quasi-static tensile testing of as-fabricated, oxidized and oxidized layer removed specimens was performed. The fracture origin location transited from the surface to silicon/oxide interface and inside of silicon. The transition may be caused by surface smoothing, thickening oxide layer and formation of oxide precipitation defects in silicon during oxidation. The radius of the oxide precipitation defects was estimated, which is well agreed with the fracture-initiating crack sizes.

1. Introduction

Fracture of both single crystal and polycrystalline silicon has been widely investigated for applications in microelectromechanical systems (MEMS). Failure of silicon is dominated by a fracture initiating flaw which mostly exists on the surface [1,2], the morphology and roughness of which are created during microfabrication processes, such as photolithography, wet/dry etching, thermal treatment, and wafer bonding. The relationship between the roughness, often regarded as a fracture initiating flow, and strength has been discussed extensively [3]. Control of the surfaces roughness of silicon is important for developing highly reliable micro mechanical structures.

There are various methods to improve the surface roughness and strength of silicon microstructures, such as anisotropic wet etching of silicon [4], hydrogen annealing [5], oxidation [6], and laser ablation [7]. Among them, smoothing using oxidation is simple and easy to implement to standard fabrication process. Ericson et al. reported improvement of the bending strength by oxidation and subsequent removal of oxide [6]. The improvement was caused by healing of removal of defect on the surface. Since the cantilever beams of heavily doped silicon layer was fabricated by means of dopant-selective etching and the damages were introduced by polishing process, it is required to investigate combined effect of oxidation process to the scalloping introduced by Bosch process, which is a standard process of silicon MEMS fabrication.

We have conducted tensile testing on single crystal silicon specimen oxidized after specimen fabrication to investigate the effect of an oxide surface layer on fracture of 3-μm-thick silicon structures using the Bosch process [8]. We reported the fracture origin transition from surface to inside of silicon possibly due to oxide precipitation. However, the etched surface was too rough to investigate the details of fracture process. In this paper, the specimen is fabricated using thicker 5-μm-thick silicon film with the Bosch process of finer scallops to reveal fracture behavior of oxidized silicon structures.

* Corresponding author.
E-mail address: tutti@me.kyoto-u.ac.jp (T. Tsuchiya).

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Nomenclature

SOI	silicon on insulator
SCS	single crystal silicon
MEMS	microelectromechanical system
RTA	rapid thermal anneal
FEM	finite element method
FE-SEM	field emission scanning electron microscope
LVDT	linear variable differential transformer
L, L_1, L_2	length of specimen gauge part
S_1, S_2	slope of stress-stage displacement curve
$r(t)$	radius of oxygen precipitation defects
t	oxidation time
D	diffusion constant of oxygen in silicon
C_0	interstitial oxygen density in silicon
C_p	oxygen density of silicon dioxide
C'	oxygen concentration in the silicon matrix
E	Young's modulus
E_n	nominal Young's modulus
E_{Si}	Young's modulus of silicon
E_{Ox}	Young's modulus of silicon dioxide
A_{Si}	cross section area of silicon
A_{Ox}	cross section area of silicon dioxide
K_{IC}	fracture toughness (of silicon)
a	half of crack length
σ_f	fracture strength

2. Experimental

Uniaxial tensile specimen of single crystal silicon was fabricated from silicon-on-insulator (SOI) wafer of 5-μm-thick device layer, using a standard SOI-MEMS fabrication process with underlayer substrate removal. The specimen is shown in Fig. 1. The gauge part is 4 μm wide, 5 μm thick and 120 or 600 μm long. The specimen surface is (1 0 0) plane and the tensile axis is ⟨1 1 0⟩ direction, which is the standard orientation in MEMS device structures. One end of the part is fixed to the substrate and the other is connected to the large paddle for gripping. Oxidation was done after specimen release, that is, the specimen (called as as-fabricated) shown in Fig. 1 was put into a RTA furnace (ULVAC, MILA-5000) and oxidized at 1100 °C. The oxygen flow rate was 0.7 l/min. The oxidation time was 5.5, 22.5 and 90 min, which correspond to the expected oxide thicknesses of 50, 100 and 200 nm, respectively. Since it is difficult to measure the oxide thickness on the micro-scale specimen, we used these expected thicknesses. These samples denoted as 50-nm-oxide, 100-nm-oxide and 200-nm-oxide. The 200-nm-thick samples were also tested after oxide removal using hydrofluoric acid etching (called as oxide-removed). The as-fabricated, oxidized and oxide-removed specimens were tested using a custom made tensile testing system under an optical microscope [9], shown in Fig. 2. The specimen-carrying substrate was aligned to the sample stage driven by piezoelectric stage (PI-polytec, P-780.20). The stage displacement was measured using a built-in LVDT sensor. The free paddle of the specimen is clamped using electrostatic gripping [2] and connected to a loadcell (Kyowa, LTS-50GA). The sample stage was covered with a small chamber connected to a temperature and humidity controlled chamber (Espec, SH-221) to control the test environment. The humidity and temperature were monitored by a sensor placed at the vicinity of the specimen. Quasi-static tensile testing was conducted at 26 °C and 50%RH. The loading rate was controlled at the stage speed of 0.5 μm/s. The nominal tensile stress was calculated by dividing the tensile force by the nominal cross sectional area. The fractured surfaces were observed using field emission scanning electron microscope (Hitachi, S-4500).

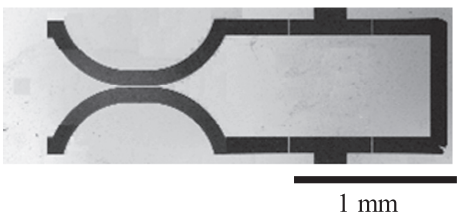

Fig. 1. Single crystal silicon specimen.