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Direct Observation of Tensile Stress in Silicon Oxide Films Using Cathodoluminescence Spectroscopy

Shigeru Kakinuma[1], Kentaro Nishikata[1], Naoaki Yamashita[2], Nobuyuki Naka[3], Shinsuke Kashiwagi[3], Koichi Matsumoto[1], Takahiro Namazu[2], and Shozo Inoue[2]

[1]R&D Center, HORIBA, Ltd., 2 Miyanohigashi, Kisshoin, Minami-ku, Kyoto 601-8510, Japan
Phone: +81-75-313-8121, E-mail: shigeru.kakinuma@horiba.com
[2]Department of Mechanical and System Engineering, University of Hyogo, 2167 Shosha, Himeji, Hyogo 671-2201, Japan
[3]Semiconductor Systems R&D Dept., HORIBA, Ltd., 2 Miyanohigashi, Kisshoin, Minami-ku, Kyoto 601-8510, Japan

1. Introduction

In-situ stress measurement in SiO_x films using cathodoluminescence (CL) spectroscopy is described. Stress evaluation is required for nano-scale devices related to LSI and MEMS to improve their characteristics and reliability [1]. CL that indicates the light stimulated by electron beam is known to yields information about the composition [2], impurity [3], dislocation [4] and stress [5] of a specimen. To date, CL stress distribution measurements with nano-meter scale spatial resolution have been reported [6,7], but the reliability of the calibration methods is not enough in the light of reproducibility and complexity of inhomogeneous stress fields.

In this paper we propose a new CL stress calibration method based on the combination of CL observation and tensile testing. The tensile tester, which can be installed in a vacuum chamber, is able to generate homogeneous stress fields in a specimen. CL spectral peak shifts, when tensile stress was applied to SiO_x specimen, were successfully observed.

2. Experimental Procedure

Tensile tester and Specimen

The uniaxial tensile tester we designed is shown in Fig. 1. Tensile stress is applied to a specimen by rotating the micrometer. Difference in heights between moving and holding parts is reduced to be less than 10 µm to avoid bending deformation of a specimen. Fig. 2 shows a photograph and optical microscope images of a SiO_x film. Si-based specimen was fabricated using MEMS techniques, followed by wet thermal oxidation to grow the film on the specimen.

CL system (SEM and Spectrometer)

A CL system (HORIBA, Ltd. MP-32M) was equipped to a SEM (HITACHI S-4300SE). CL was collected with an ellipsoidal mirror, which was led by an optical fiber to a spectrometer with a CCD detector. The excitation energy, probe current and exposure time were set to be 3 kV, 140 pA and 5 s, respectively. The obtained CL spectra were analyzed by curve fitting with Gauss/Lorentz functions. The curve fitting procedure enhances the spectral resolution as compared to the spectrometer resolution.

3. Results and Discussions

A typical CL spectrum of SiO_x and the fitting result are

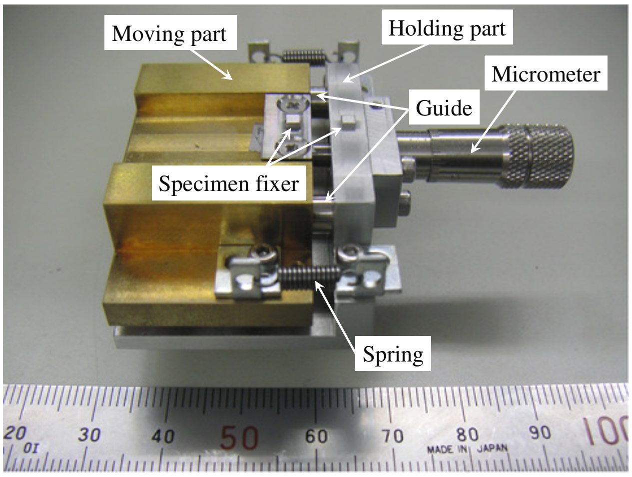

Fig. 1 A photograph of an uniaxial tensile tester

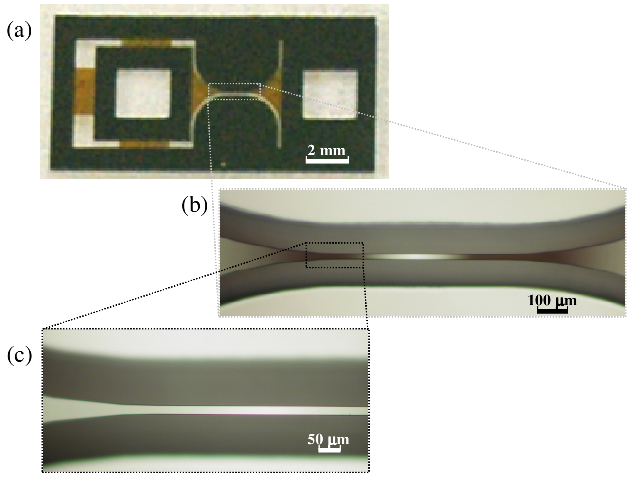

Fig. 2 Specimen images; (a) Photograph, (b) Optical microscope image, (c) Magnified view of (b).

shown in Fig. 3. Table I shows the band assignment of SiO_x originating from three defect peaks [8-10]. The 1.85 eV band was monitored for stress analysis. Fig. 4 shows the relation between displacement values of a micrometer and the peak position. Higher energy shift was observed as the displacement increased. At the same time, lower energy shift was observed when the displacement decreased. Fig. 5 shows the SE image for the fracture surface of a SiO_x film specimen. The mirror, mist and hackled surfaces were observed. These results confirmed that the specimen was tensioned precisely without bending deformation.

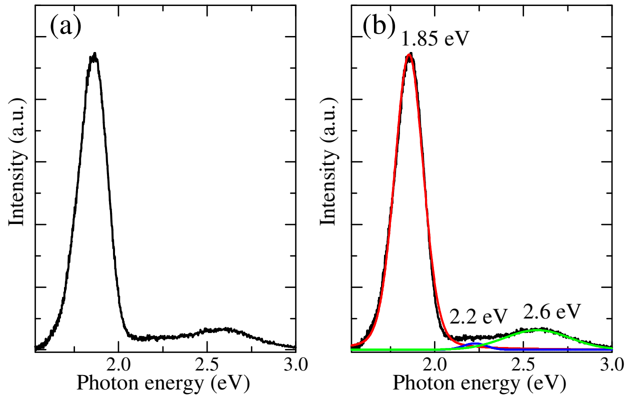

Fig. 3. (a) A typical CL spectrum of SiO_x and (b) the fitting result

Table I Band assignments of SiO_x

Emission	Assignment	Models
1.85 eV	NBOHC (Non-bridging oxgen hole center)	≡SiO·
2.2 eV	Trivalent silicon	≡Si·
2.6 eV	Neutral oxgen vacancy defect	≡Si−Si≡

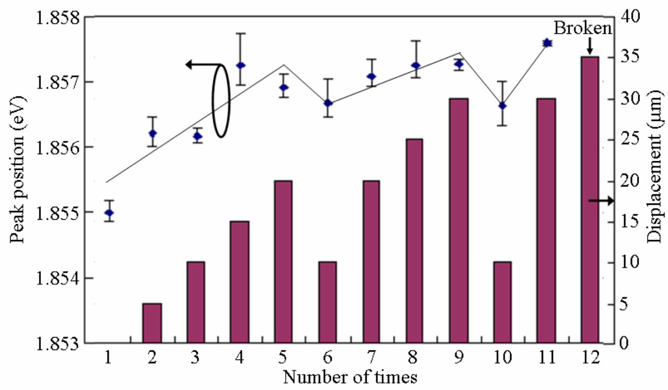

Fig. 4. The relation between peak position (line plot) and displacement value of the micrometer (bar chart).

4. Conclusions

We have performed tensile tests of SiO_x film with in-situ CL observations, and have demonstrated that the peak position of CL spectra shifts toward the higher energy side when tensile stress is applied to the specimen. This result suggests that CL is applicable to stress analysis in nano-meter scale devices related to LSI and MEMS. In future we will design and develop high-precision tensile tester using a piezo-actuator to accurately obtain the calibration curve between stresses and CL peak positions.

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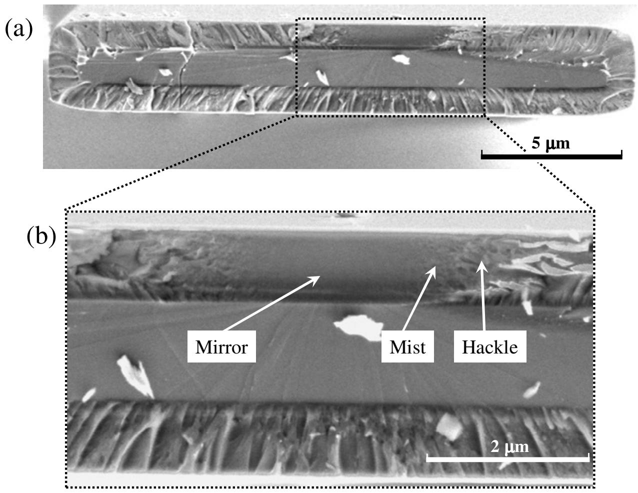

Fig. 5. The SE image for (a) the fracture surface and (b) the magnified view.

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