Uniaxial Tensile Testing System for Quantitative Stress Analysis in Silicon Oxide Thin Films by Cathodoluminescence Spectroscopy

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1. Introduction

Non-contact stress evaluation is required for nano-scale devices related to LSI and MEMS to improve their characteristics and reliability [1]. Cathodoluminescence (CL) indicating the light stimulated by electron beam yields information about the composition [2], impurity [3], dislocation [4], and stress [5] of a specimen. To date, CL has become the candidate of non-contact stress measurement techniques with nano-scale spatial resolution [6,7]. We have proposed the stress calibration method, and have conducted tensile tests of silicon oxide (SiO_x) film with in-situ CL observations [8]. However, the tensile tester employed showed low repeatability and operationality.

The purpose of this study is to develop a uniaxial tensile testing system for quantitatively investigating the influence of tensile stress on CL spectra in SiO_x thin films. The uniaxial tensile tester installed in a scanning electron microscope (SEM) chamber has been developed to precisely generate homogeneous stress fields in a SiO_x film specimen.

2. Experimental Procedure

Tensile testing system and uniaxial tensile tester

Fig. 1 (a) shows a block diagram of the uniaxial tensile testing system operating in a SEM. The tensile stress can be controlled from the outside of the vacuum chamber. Fig. 1 (b) shows the developed tensile tester. The tester is fixed on a SEM specimen holder. This tester that has designed to be compact includes a piezoelectric actuator in a case, a load cell, specimen holders, and micro connector. The case has a lever structure that can amplify the displacement of the PZT actuator in the tensile direction. The amplification factor and the maximum displacement in the tensile direction are 2.27 and 67 μ m, respectively. The force measurement resolution of the load cell is 2 mN. The electrical micro connector enables us to easily desorb this tester from the SEM stage. The specimen tested can be replaced to a new one by using a load lock system only.

Specimen for tensile test

Figs. 2 show photographs of the produced SiO_x film specimen for tensile test. The specimen was fabricated using MEMS fabrication techniques. The figures indicate that the specimen consists of the parallel gauge section with 4-µm-thick single crystal silicon (SCS) covered with 820-nm-thick thermal SiO_x film, SCS springs, chucking



Fig. 1 (a) Block diagram of the uniaxial tensile testing system (b) photograph of the uniaxial tensile tester.

holes, and frame.

CL measurement system (SEM and Spectrometer)

A CL analysis system (HORIBA, Ltd. MP-32M) was equipped to a SEM (HITACHI S-4300SE) as illustrated in Fig. 1(a). CL was collected with an ellipsoidal mirror, and 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 sec, respectively. The obtained CL spectra were analyzed by Gauss/Lorentz function curve fitting.



Fig. 2 Photographs of the prepared tensile test specimen.



Fig. 3 Typical stress-strain relation of SiO_x -coated SCS specimen by uniaxial tensile test.

3. Results and Discussions

To check the performance of the produced tensile tester, we have firstly evaluated stress-strain relations of two types of specimens; a SCS specimen and a SiO_x-coated SCS specimen, before SiO_x stress evaluation with CL. The measured Young's modulus of 4- μ m-thick SCS specimen was 167 GPa, almost the same as the analytical value of SCS, though the data are skipped here.

Fig. 3 shows the stress-strain relation of the specimen. When the Poisson ratios of SCS and SiO_x films are the same, the effective elastic modulus, E_{1+2} , that is the combined Young's modulus of SiO_x and SCS can be calculated by the following equation:

$$E_{1+2} = \frac{E_1 t_1 + E_2 t_2}{t_1 + t_2}$$

where E_1 and E_2 are the Young's modulus of SCS and SiO_x respectively, and t_1 and t_2 are the those thicknesses. In Fig. 3, E_{1+2} was calculated to be 124 GPa. By substituting E_{1+2} , E_1 (= the measured value: 167 GPa), t_1 and t_2 into the equation above, E_2 was determined to be 50 GPa. This is comparable to the literature values on SiO_x film.

Fig. 4 depicts typical CL spectra of SiO_x under various tensile stresses. SiO_x is known to show three defect peaks in CL measurement. In this study, we monitored only 1.85 eV band for stress analysis. As tensile stress increased, the peak position gradually shifted to high energy side though the increments were very small.

Fig. 5 shows the relationship between applied tensile stress and the peak position. The obtained linear relation



Fig. 4 CL spectra around 1.85eV under various tensile stresses.



Fig. 5 Relationship between applied tensile stress and photon energy of CL spectra around 1.85 eV.

indicates that the peak position is a spectrum parameter for detecting stress on SiO_x film by CL analysis.

4. Conclusions

The developed uniaxial tensile testing system enabled us to perform tensile tests of SiO_x film with in-situ CL observation. In CL point measurements at non-damaged area in specimen, the peak position of CL spectra depended on the applied stress. In future we will investigate the relationship between stress and other CL spectral parameters using this system.

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