# **Evaluation of Oxygen Precipitates in Si Wafer by Resonance Ultrasound Microscopy**

共振ヤング率顕微鏡を用いた Si ウェハー内の酸素析出物の評価

Hiroki Yoshida<sup>1†</sup>, Hirotsugu Ogi<sup>1</sup>, Masahiro Hirao<sup>1</sup>, Kazuhiro Matsukawa<sup>2</sup>, Hirofumi Harada<sup>3</sup> (<sup>1</sup>Graduate school of Engineering science, Osaka Univ., <sup>2</sup>Renesas Technology inc., <sup>3</sup>Siltronic Japan inc.)

吉田裕希  $^{12}$ , 荻博次  $^{1}$ , 平尾雅彦  $^{1}$ , 松川和人  $^{2}$ , 原田博文  $^{3}$  ( $^{1}$ 大阪大 基礎工;  $^{2}$ (株) ルネサス テクノロジ、 $^{3}$ シルトロニック・ジャパン(株))

#### 1. Introduction

Si wafers are extensively used as substrates or structure components in many devices, and their functions are highly related with defects near the surface of Si. Because this influence becomes marked as the miniaturization proceeds, more accurate control on their density and size is needed. Oxigen presipitates, called the bulk microdefects (BMD), especially affect mechanical and electrical properties of micro electromechanical systems (MEMS) and their configuration and concentration should be controlled precisely. BMDs appear in Si wafers manufactured by the Czochralski method. They behave as gettering sources to capture heavy metal impurities. However, their mechanical properties are expected to be significantly different from those of the matrix Si, deteriorating the functions of MEMS devices. Thus, the nondestructive evaluation method of the mechanical properties of BMD has been desired. Existing methods such as the infrared light scattering method<sup>1)</sup> and the transmission electron microscope can visualize and evaluate BMDs, but they cannot provide us with any mechanical information.

To evaluate the elastic constant of BMD, we use resonance ultrasound microscopy (RUM). This method quantitatively measures Young's modulus at the local regions of materials by the change in the resonant frequency of the probing oscillator. The amount of the deformation around the defects increases by the stress concentration, and Young's modulus there will decrease. The base structure of BMD is known to be equivalent to the amorphous SiO<sub>2</sub>, and its Young's modulus is expected to be lower than Si by 45%. Thus, RUM is effective for the BMD evaluation, because it is high sensitivity for material's Young's modulus<sup>2)</sup>.

## 2. Measurement Procedure

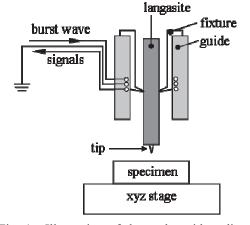


Fig. 1 Illustration of the probe with a diamond tip. Three antennas are embedded in the cylindrical guide.

RUM is the method to measure Young's modulus at local regions of materials. When the resonance oscillator touches the specimen, the resonance frequencies of the oscillator change. This change reflects the elastic constants of the specimen. Figure 1 shows an illustration of the RUM probe. A monocrystal rectangular parallelepiped langasite is used as the oscillator. For the quantitative measurement, langasite is supported by a nylon fixture at the nodal point, which was identified by Rayleigh-Ritz method with Lagrangian minimization<sup>3)</sup>. The longitudinal direction of the crystal is selected to be along the X direction of the trigonal system for three reasons. First, the X direction Young's modulus  $E_{[100]}$  takes a minimum value, leading to a high sensitivity as shown later. Second, the temperature derivative of Young's modulus is smaller in the X direction, yielding the stable resonance frequency for the temperature change. Third, because the piezoelectric coefficient  $e_{11}$  takes a large absolute value, the longitudinal vibration along the X direction is easily excited.

Excitation and reception of the langasite oscillator are performed by three line wires. They consist of the generation wire, the detection wire, and a grounding wire. We applied tone-burst voltages to the generation wire to induce the

ogi@me.es.osaka-u.ac.jp

quasi-static electric field in the X direction at the nodal line of the side face of the crystal. The electric field along the longitudinal direction predominantly causes longitudinal vibrations (Ag vibration groups<sup>4</sup>). After the excitation, the detection wire picks up the resonance vibration through the piezoelectric effect<sup>5, 6)</sup>. A frequency scan yields the resonance spectrum contactlessly, and the Gaussian-function fitting provides the resonance frequency. For a quicker measurement, we monitored the phase of the received signal at a fixed frequency to determine the resonance frequency change from the linear relationship between the phase and frequency near the resonance frequency<sup>6, 7)</sup>.

To convert the difference of the resonance frequency to Young's modulus, a Hertzian contact model is adopted<sup>8)</sup>. Assuming a longitudinal vibration, we derived a frequency equation of the oscillator in contact with the specimen

$$kL\tan(kL) = \frac{K}{K_{osc}}.$$
 (1)

Here, k is the wavenumber, K is the contact stiffness between the tip and the specimen.  $K_{OSC}=E_{[100]}A/L$ , where  $E_{[100]}$ , A, and L are Young's modulus along the X axis, the cross-section area, and the length of oscillator, respectively. K is represented by below.

$$K = \sqrt[3]{6E^{*2}RF_0} \ . \tag{2}$$

Here,  $E^*$ , R,  $F_0$  are the effective Young's modulus of with the specimen, the tip radius, and the biasing force. Hertzian contact model assumes that the material is elastically isotropic. However, applying the stress analysis procedure proposed by Willis<sup>9</sup>, the error related with the elastic anisotropy is estimated to be less than  $10\%^{2}$ .

## 3. Results and discussion

Young's modulus images annealed Si wafer at 1000 °C for 30 min was measured by RUM with a 1 µm step. Figure 2 shows a result. Note that there are many small spots where Young's modulus (~80-90 GPa) is significantly lower than the matrix region. Because the BMD structure is similar to that of the amorphous SiO<sub>2</sub>, whose Young's modulus equals 72 GPa, these softened regions are caused by the concentration of BMD. Thus, the present RUM can be used to detect the soften regions where BMD is concentrated. However, because the macroscopic volume fraction of BMD is significantly smaller, it is difficult to detect BMD in uniformly distributed BMD areas. Therefore, the present RUM needs to be

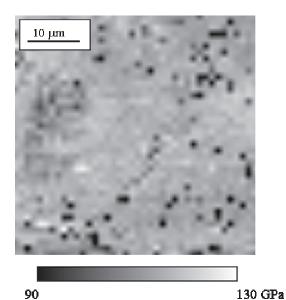


Fig. 2 Young's modulus image measured by RUM on an annealed Si wafer. The modulus mapping has been done every 1  $\mu$ m.

improved for detecting a single BMD.

#### 4. Conclusion

We measured Young's modulus of Si wafer at local regions by RUM. Mapping image of Young's modulus for Si wafer shows many regions where Young's modulus is lower than the matrix region. Their stiffness correspond to the amorphous SiO<sub>2</sub>, indicating that they are BMD.

## References

- K. Moriya, K. Hirai, K. Kashima, and S. Takasu, J. Appl. Phys. 66, 5267 (1989).
- 2. H. Ogi, T. Inoue, H. Nagai, and M. Hirao, Rev. Sci. Instrum. **79**, 053701 (2008).
- 3. H.Ogi, N. Nakamura, K. Sato, M. Hirao, and S.Uda, IEEE Trans. Ultrason. Ferroelectr. Freq. Control **50**, 533 (2003).
- 4. I. Ohno, Phys. Chem. Miner. 17, 371 (1990).
- 5. H. Ogi, K. Motohisa, T. Matsumoto, K. Hatanaka, and M. Hirao, Anal. Chem. **78**, 6903 (2006).
- H. Ogi, K. Motohisa, T. Ohmori, M. Hirao, and M. Nishiyama, Biosens. Bioelectron. 22, 3238 (2007).
- 7. H. Ogi, K. Matsumoto, K. Hatanaka, M. Hirao, and M. Nishiyama, Jpn. J. Appl. Phys. **46**, 4693 (2007).
- 8. K. Johnson, Contact Mechanics (Cambridge Univ. Press, Cambridge, 1985).
- 9. J. R. Willis, J. Mech. Phys. Solids **14**, 163 (1966).