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S-E-4

X-Ray Topographic Observations of Defects in As-Grown Silicon Single Crystals

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The advent of the synchrotron radiation opened up several new possibilities for X-ray topography. One of the most interesting one is the capability of producing highly collimated incident beam at desired wavelength. In this paper are shown some X-ray optics developed for the topographic observations of defects in as-grown silicon single crystals together with some recent results.

1. INTRODUCTION

The extremely high photon flux produced by synchrotron radiation makes it possible to develop many kinds of pre-processing technique of the incident x-rays used for synchrotron x-ray topography. Among them are (i) enlargement of the beam size (ii) highly monochromatization and (iii) highly collimation. These pre-processing techniques are especially useful for the x-ray topographic studies on as-grown silicon single crystals because the crystal quality of them nowadays are too excellent to observe inhomogenity or defects by usual laboratory equipments.

On the other hand, a number of perfect crystal optical elements are necessarily used as x-ray monochromators, collimators etc. in synchrotron radiation facilities. For those purposes which need ultra-high resolution, a slightest imperfection in optical elements should be avoided. Accordingly, sensitive characterizing methods for crystal imperfections were needed to be developed.

At the Photon Factory, commissioning of a high-precision instrument for topography started in 1983, as a collaborated work between NEC and the Photon Factory[1]. Since then, continual improvement has been made both for the diffractometer itself and peripheral utilities[2]. Recently, the instrument was opened for general users and research groups from NEC, SONY, NKK, Kyushu Institute of Technology and Photon Factory/Tokyo University share the same x-ray optics for their own samples.

2. X-RAY OPTICS

Two different x-ray optics are developed for the topographic study of the defects in as-grown silicon crystals. Perfect crystal optical elements based on the dynamical x-ray diffraction are extensively used for these optics.

One is essentially same as the double-crystal plane wave topography with asymmetric collimator, but a pre-monochromator crystal is added upstream of the asymmetric collimator so as to make (+,+) setting between them[3),4)] as shown in Fig.1. Since the original beam from the bending magnets of the storage ring is narrow vertical and aperture-limited horizontal sizes, we enlarged the vertical size of the beam by an asymmetric collimator to suit large sample wafers. Maximum size of the wafer ever taken by this scheme was as large as 6 inches[2)]. This optics have been mainly used for the studies of the growth-striation behavior under different growth conditions in Bragg geometry. Hereafter, this scheme is referred to as PWT.

Another is for "Ultra-Plane-Wave X-Ray Topography (UPWT)", which was originally constructed for the measurement of the coherence length of the highly collimated x-rays[5)]. Four crystal arrangement shown in Fig. 2. was used for UPWT. By the two succesive asymmetric

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diffractions of the second and third crystals, the angular divergence of the incident x-rays on the sample crystal (the 4th crystal) is reduced to be less than 0.01 arcsec, approximatedly 1/200 of the angular width of the intrinsic diffraction curve.

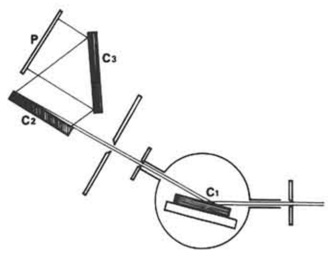

Fig. 1. X-ray optics for synchrotron PWT. White synchrotron x-rays (SR) are monochromatized by a pre-monochromator crystal (C_1), where 111 symmetric reflection in Bragg geometry is used. This crystal is mounted on a water-cooled crystal holder in order to remove heat due to irradiation of white SR. The second crystal (C_2) is a collimator, which is prepared from (111)Si slab. For characterization of (001)Si wafers, oblique 008 reflection is used so as to make asymmetric factor to be 1/40. This determines the available wavelength of x-rays around 0.113 nm. The third crystal is a sample (C_3) where 008 reflection in Bragg geometry is used. Topographs are recorded on a nuclear emulsion plates (P) placed perpendicular to the diffracted beam.

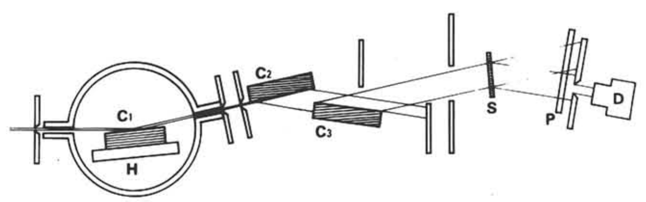

Fig. 2. X-ray optics for synchrotron UPWT. C_1; pre-monochromator, (111)Si, symmetric 111 reflection, H; water-cooled crystal holder. C_2 and C_3; asymmetric 220 collimator with asymmetric factor of 1/36 at the wavelength of 0.072 nm. S; sample crystal, symmetric 220 reflection in Laue geometry. P; nuclear emulsion plate for recording. D; monitoring detector.

3. OBSERVATION OF AS-GROWN SILICON CRYSTALS BY PWT

Figures 3 and 4 are plane-wave x-ray topographs of silicon wafers grown by magnetic Czochralski (MCZ) method and floating zone method, respectively. Strong striated patterns due to fluctuating oxygen concentration are clearly seen in topographs of MCZ crystal, whereas those for FZ crystal are quite faint.

A systematic study of the striation patterns for different growth conditions is now underway by SONY group. Results will be reported elsewhere[6]. Up to now, we cannot find those ingots for which striation patterns are not observed at all.

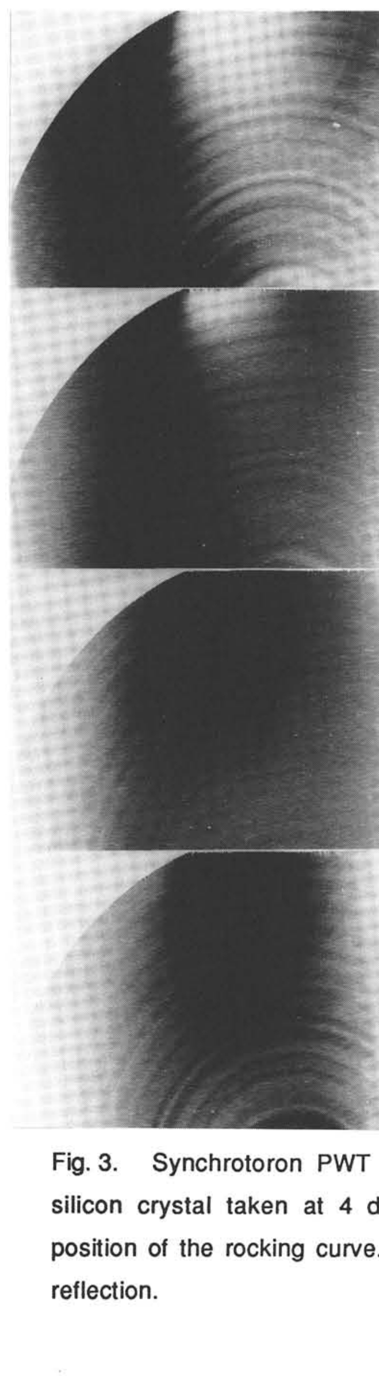

Fig. 3. Synchrotoron PWT of 4 inch MCZ silicon crystal taken at 4 different angular position of the rocking curve. 008 symmetric reflection.

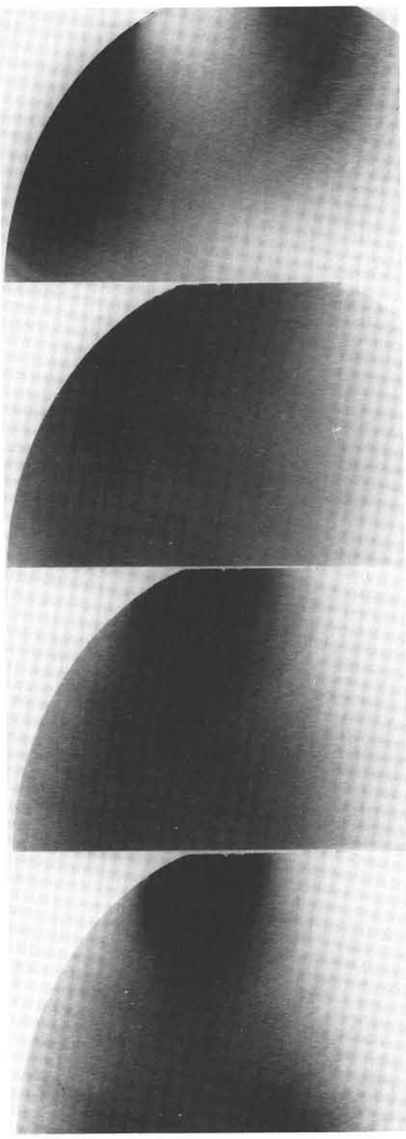

Fig. 4. Synchrotoron PWT of 4 inch FZ silicon crystal taken at 4 different angular positions of the rocking curve. 008 symmetric reflection.

4. OBSERVATION OF AS-GROWN SILICON CRYSTALS BY UPWT

In the ultra-plane-wave x-ray topographs, fine interference patterns usually smeared out in the double crystal system can be clearly observed because the angular divergence of the incident beam is much less than the intrinsic angular width of diffraction. Oscillatory profiles, with a period of 10^{-6} - 10^{-7} radian, of the diffraction curves in the transmission geometry are experimentally observed even for a rather thick crystal. In this case, minute strain fields around defects are imaged on a topograph as an equal-inclination fringes, which reflect the local effective deviation from the Bragg condition. Because the period of oscillation of the equal-inclination fringes is as small as 10^{-7} radian, minute strain field corresponding to the deviation of Bragg angle between 10^{-8} and 10^{-9} radian range can be imaged as a difference in the contrast. In addition, combining the equal-inclination fringes with equal-thickness fringes by using a wedge-shaped sample crystal, both sense and magnitude of atomic displacement around a defect can be analyzed. Figure 5 shows an example of the incident angle dependence of defect images for a wedge-shaped sample.

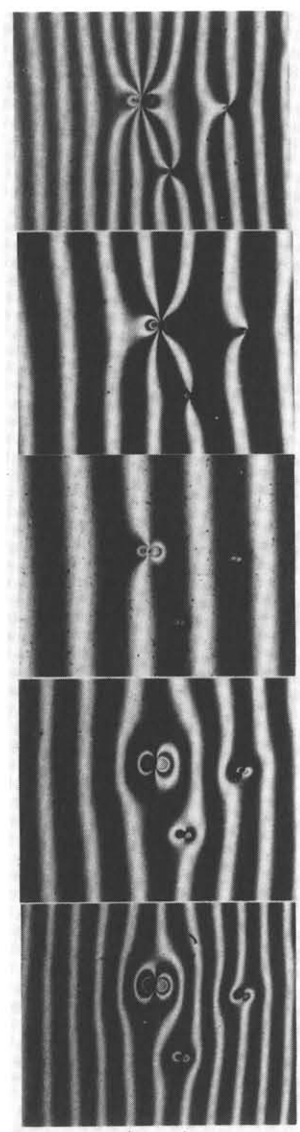

Fig. 5. UPWT of a wedge-shaped FZ silicon wafer taken at 5 different angular positions of the rocking curve. 220 symmetric reflection in Laue geometry. Scale make shown is 0.5 mm.

Even for a parallel-sided wafer, macroscopic equal-inclination fringes are observed due to resiual strain field when unsuitable sample preparation is made. Figure 6 is an example of such equal-inclination fringes. Besides of the equal-inclination fringes, two growth striation systems are observed in Fig. 6. A vertical one is from the collimator crystal while a horizontal one is from the sample crystal.

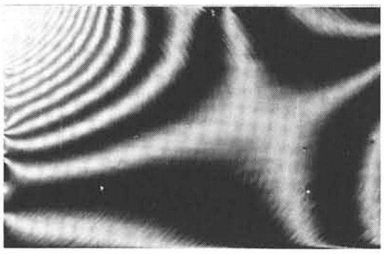

Fig. 6. Equal-inclination fringes observed for a poorly prepared parallel-sided sample.

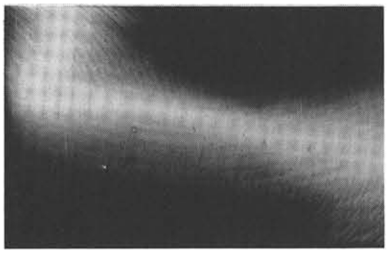

Fig. 7. UPWT of the same sample wafer shown in Fig. 6 after chemical etching.

In order to remove the strain, the sample crystal was chemically etched by HF(1) and HNO_3(10) mixture. UPWT of the sample after etching is shown in Fig. 7. According to Cu-decoration technique, this sample wafer contains three different regions; swirl, intermediate and D-defects[7]. Up to now, we cannot find distinctions of these three regions from the topographic contrasts nor the profiles of the rocking curves, but can observe similar striation patterns for every region and some individual contrast due to minute strain field.

Since the exposure time for recording UPWT is relatively short, 3-5 min for Ilford L4 nuclear emulsion plate, with SR from a bending magnet of the Photon Factory, utilization of much more stress-sensitive x-ray optics will be possible by using SR from insertion devices such as a multi-pole wiggler. In this case, a precision diffractometer with nano-radian anglar resolution is necessary. We have designed and constructed such a diffractometer and are now testing at the Photon Factory.

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