Measurement of local minute strain by using synchrotron X-ray microbeam

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Abstract: We have demonstrated to form an x-ray microbeam with a narrow angular divergence in both vertical and horizontal polarization directions of synchrotron radiation x-rays by the use of successive asymmetric Bragg reflections. Using the highly parallel x-ray microbeam thus obtained, we are able to analyze the local minute strain of less than 10^{-5} in semiconductor materials and devices. A series of x-ray rocking curves have been obtained by scanning the sample against the x-ray microbeam. Variations of the reflection peak intensity, angular shift value and/or half widths inform us how strain is distributed in the materials with high spatial resolution. In addition, reciprocal space maps have been drawn with an analyzer crystal put behind the sample on the x-ray path. From those data, strain distribution can be analyzed in terms of both lattice tilt variation and lattice parameter variation independently. Strain near Si-oxide film edges on Si substrate or that in silicon-on-insulator (SOI) crystals or other semiconducting materials has been measured. The results of reciprocal space maps have shown that the strain in the bonded SOI layers is mainly due to the lattice tilt variation rather than the lattice parameter variation.

Key words: Minute strain, Rocking curve, X-ray microbeam, SOI, Synchrotron radiation

1. INTRODUCTION

It has been well known that semiconductor device characteristics are influenced by crystalline defects such as dislocations, especially in optoelectronic devices such as light emitting diodes (LEDs) and laser diodes (LDs). Under a given environmental condition like a high temperature or steep temperature gradient in the crystals, those defects are sometimes generated at areas where local strain is exerted exceeding a critical value. Study of the local strain distribution in the crystals after growth or device processes is of much importance in terms of improvement of the device characteristics or the product yield.

For detecting the minute strain in high-quality single-crystals like silicon (Si) or gallium arsenide (GaAs), x-ray diffraction is useful because of non-destructivity of the sample and high strain sensitivity of the measurement. It is usually difficult, however, to measure directly and accurately the local strain because a proper x-ray probe of μm -order magnitude in size has not been obtained from a conventional x-ray.

In order to perform the measurement of local and minute strain in the crystals, X-ray microbeam combined with synchrotron radiation is one of the excellent candidates. We have established highly parallel x-ray microbeam by employing successive asymmetric Bragg reflections for both horizontal and vertical polarization directions of incoming synchrotron x-rays. By scanning the microbeam on the sample surface, a series of x-ray reflection intensity curves, i.e., x-ray rocking curves (hereafter, this will be referred as rocking curve maps, RCMs) have been obtained.

For more precise analysis of the lattice deformation at various fixed points of the sample, we put an analyzer crystal behind the sample and rotate both the sample and the analyzer crystal. Thus we can draw reflection intensity distribution maps around a reflection point in the reciprocal lattice space, which are so-called reciprocal space maps (hereafter, this will be referred simply as RSMs). From inspection of those RSMs it is possible to clarify the lattice strain in the samples in terms of both lattice tilt variation and lattice parameter variation.

2. EXPERIMENTAL

2.1. Formation of X-ray microbeam

So far there have been several technologies to prepare an x-ray microbeam, e.g., by the use of a pinhole [1], a glass capillary or an optical guide [2], mirrors of Kirkpatrick-Baez type [3], a Fresnel zone plate [4], a Wolter mirror [5], etc. But those x-ray optics cannot supply any x-ray microbeams with high parallelism being capable to detect the local strain of 10^{-4} or less at a small area of a few μm in size. It may be difficult to detect accurately the local minute strain by using the microbeams with a wide angular divergence, even if the probe size is small.

In order to measure the local strain with a high strain sensitivity of less than 10^{-5} , we need to design new x-ray optics being possible to supply the x-ray microbeam with a narrow angular divergence and a small beam size. Strain sensitivity and spatial resolution depend basically upon the angular divergence and the probe size, respectively, of the x-ray microbeam impinging on the sample.

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As an x-ray source, synchrotron radiation has become an excellent source because of its high brilliance, high parallelism, coherency, polarization, pulsation and so on. Using the synchrotron radiation, especially from an undulator, we can put various types of the x-ray optics in front of the sample without significant loss of the x-ray photons.

The present experiments have been carried out at the Hyogo Beamline (BL24XU) in SPring-8 [6]. The beamline has employed an in-vacuum type 'figure-8' undulator [7] for the hard x-ray region, which can provide both vertically and horizontally polarized x-rays as integer and half-integer harmonics, respectively. The undulator has been tuned to a horizontally polarized x-rays of 15 keV photon energy. A silicon double-crystal monochromator of horizontal dispersion with 111 symmetric reflection has been placed at a distance of 61 m away from the light source. The x-ray beam size at the undulator light source was 203 μm (horizontal) $\times$ 29 μm (vertical) and the angular divergence was 185 μrad (horizontal) $\times$ 11 μrad (vertical). In the experimental hutch 'C' of BL24XU, a four-quadrant slit of 100 μm (W) $\times$ 40 μm (H) was placed at 66 m from the light source to suppress the angular divergence of the x-rays to be less than 4.6 μrad (horizontal) $\times$ 1.0 μrad (vertical). Then, two double-crystal beam collimators were installed consecutively, as schematically shown in Fig. 1.

Fig. 1. Setup of x-ray rocking curves and reciprocal space maps.

The crucial concept of the present x-ray optics is a combination of 115 (+, -) successive asymmetric Bragg reflections [8] from (001) Si surface of perfect silicon crystals mounted on separate goniometer systems to realize the x-ray microbeam possessing a small angular divergence and a narrow energy bandwidth not only in the vertical polarization direction but also in the horizontal polarization direction [9]. The x-ray beam size becomes

small with an asymmetric factor of b , which is written as

$$b = \sin(\theta_B - \alpha) / \sin(\theta_B + \alpha), \quad (1)$$

where θ_B is the Bragg angle and α is the angle between the beam compressor crystal surface and the diffraction plane. The total demagnification determined by a factor of b^2 was almost 0.04. The beam sizes measured by a NaI scintillation counter with the use of a knife-edge scanned in the both directions were 7.6 μm (horizontal) and 5.7 μm (vertical) at the sample position. The microbeam actually obtained had an angular divergence of less than 2 arcsec for the both polarization directions.

2.2. Strain measurement

Samples of Si crystals or compound semiconductors like GaAs or indium phosphide (InP) crystals were mounted on a three-axes (x-y-z) translation stage fixed on a high-precision ω - 2θ goniometer being driven by pulse motors of high rotation accuracy with an angular step of 0.18 arcsec for the sample rotation (ω rotation). By scanning the sample after each ω rotation as schematically shown in Fig. 2, an RCM can be obtained part by a scintillation counter placed on a 2θ arm of the goniometer.

Fig. 2. Schematic illustration of the RCM measurement.

In addition, by putting a channel-cut Si analyzer crystal behind the sample [10, 11] on the x-ray path as seen in Fig. 1, the intensity distribution contour maps in a reciprocal lattice space (q_x , q_y), i.e., RSMs, are obtained from the data of reflection intensity. This is performed by rotating both the sample (ω rotation) and the 333-reflection analyzer crystal (2θ rotation) independently. Thus one can clarify whether the intensity fluctuation appearing in the RCM is due to the lattice tilt variation or due to the lattice parameter variation. A horizontal axis (q_x) of the RSM corresponds to lattice tilt variation (ω) and a vertical axis (q_y) corresponds to lattice parameter variation ($\Delta d/d$) of the sample.

3. RESULTS AND DISCUSSIONS

3.1. Strain at SiO₂/Si film edges

For a sample with a thermally oxidized SiO₂ film of 0.48 μm thickness, being formed partially by surface oxidation using a SiN mask, the RCMs of Si 004 symmetric reflection were obtained. The sample was translated stepwise against the x-ray microbeam both in the [110] and [1 -1 0] directions parallel to the (001) wafer surface after each ω rotation. The extinction distance of

x-rays in the present case was estimated to be $1.7 \mu\text{m}$. Figure 3 shows one of the RCMs for the local area of $300 \times 300 \mu\text{m}^2$ obtained by scanning the x-ray microbeam crossing the film edge in the $[110]$ direction with a scan step of $1 \mu\text{m}$ across the SiO_2 film edge. The vertical axis in Fig. 3 indicates the relative sample positions against the microbeam. An upper half of Fig. 3 corresponds to the region covered by SiO_2 film and a lower half corresponds to the bare Si surface region.

It is seen that the RCM at the area close to the film edge extends intensity tails of the curves to both the lower and the higher angles, the fact being probably caused by inferior crystallinity due to existence of the large strain around the film edge. Furthermore, the maximum peak shift to a higher angle for the bare Si region occurs at around $5 \mu\text{m}$ apart from the film edge and that to a lower angle for the SiO_2 -covered region also occurs at around $5 \mu\text{m}$ apart in another way from the film edge. The lattice parameter changes $|\Delta d/d|$ at those positions are nearly 5×10^{-6} . Those values seem to be depended, of course, upon the SiO_2 film thicknesses.

Fig. 3. RCM near a SiO_2 film edge obtained by scanning the x-ray microbeam. It is seen that rocking curve peaks shift around the film edge.

Fig. 4. Schematic illustration of strain feature around the SiO_2 film edge.

Another RCM of the reflection intensity distribution was obtained by scanning the sample in the $[1 -1 0]$ directions in such a way that a plane of x-ray incidence was perpendicular to the film edge direction. By observing carefully the result of those data, a possible feature of the lattice strain around the SiO_2 film edge can be drawn as schematically shown in Fig. 4. Lattice parameters in the direction normal to the surface are increased in the SiO_2 -covered region while those in the bare Si region are decreased.

Above-mentioned situation of the lattice strain near the SiO_2 film edge was previously predicted by Isomae [12] from the calculation of material dynamics. However, no real measurement of the local strain near the SiO_2 film edge has been carried out, since a brilliant x-ray microbeam with the high parallelism has not been made until the third-generation synchrotron radiation becomes available. It was also observed that the strain near the SiO_2 film edge, especially when the line-and-space patterns of the SiO_2 film exist, gives rise to a complicated pattern of the intensity distribution in the RSMs [13].

3.2. Strain in SOI layers

Silicon-on-insulator (SOI) crystals consisting of thin-top-Si-layer/buried-oxide- SiO_2 -layer ("BOX")/Si-substrate has been expected as one of the promising silicon substrates for extending three-dimensional metal-oxide-silicon (MOS) devices. There have been two principal methods for preparing the SOI materials; bonded silicon with a polished-back surface substrate [14] and oxygen-implanted silicon as simply called "SI-MOX" (separation by implanted oxygen) [15].

High-quality SOI layers seem to lead desired performance of three-dimensional Si devices like high-speed signal processors. Therefore, a direct characterization of the SOI quality is important. By using high-resolution x-ray microbeam diffraction technique, one can analyze the SOI layer crystallinity, especially the strain fluctuation in the SOI layers [16], regardless of the existence of the underlying BOX layer.

Firstly, bonded SOI layers have been investigated. Thickness of the BOX layer used in the present experiment was almost $1 \mu\text{m}$ and that of the SOI layers varied ranging from $5 \mu\text{m}$ to $60 \mu\text{m}$. By scanning the SOI sample against the x-ray microbeam, RCMs were obtained in the same way as in the SiO_2 film edge case.

Since lattice plane of the SOI layer and that of the substrate are not completely parallel, the x-ray rocking curves usually give two peaks of the Bragg reflection, one coming from the SOI layer and the other from the substrate. Figure 5 shows one of the 004-reflection RCMs measured along a line of about $200\text{-}\mu\text{m}$ length on a $5\text{-}\mu\text{m}$ -thick bonded SOI sample. It is interest to point out that the peak profiles corresponding to the SOI layer vary in shapes and that half widths of the curves fluctuate depending upon the irradiated positions. A vertically straight line of high intensity on the right hand side of Fig. 5 corresponds to the diffraction peaks from the substrate. A fluctuating intensity distribution on the left hand side of Fig. 5, however, indicates that the lattices of the SOI layer are strained from point to point.

In a second stage, RSMs were taken at some fixed peak positions of the SOI layer in the RCM shown in Fig. 5.

Figure 6 shows an example of RSM measured for the SOI layer at a dotted white line in Fig. 5. It is clear from this figure that the lattice distortion in the SOI layer is mainly due to the lattice tilt variation because several high-intensity spots appearing at different ω and standing only in a line of $\Delta d/d = 0$ can be seen. The fact may suggest the existence of several crystal grains of different lattice plane tilt angles. In addition, the lattice parameters for those grains scarcely change and thickness fringes belonging to each grain are recognized in the vertical direction in Fig. 5. From those results, it is suggested that the each grain in the SOI layer has almost perfect crystallinity.

Fig. 5. RCM of 004-reflection taken from a 5- μm -thick SOI layer. The vertical axis indicates the positions of the sample irradiated by the scanning x-ray microbeam.

Fig. 6. RSM of a bonded SOI layer measured at a dotted line position of the RCM shown in Fig. 5. Several high-intensity spots standing in a line at $\Delta d/d = 0$ correspond to the grains of different lattice plane tilts in the SOI layer. Additional spots arrays lying along the vertical direction are due to thickness fringes belonging to each grain in the SOI layer.

It is interesting to see how the lattice planes extend with the same lattice tilt in the SOI layer. Then, the sample was scanned against the microbeam and the reflection intensity was recorded at a fixed tilt angle of the crystal rotation (ω). Figures 7 shows the intensity distribution maps, i.e., equi-tilt maps, for an area of about $300 \times 300 \mu\text{m}^2$ of the SOI layer, being measured at two different rotation angles, (a) $\omega = -7.9$ arcsec and (b) $\omega = +7.9$ arcsec. From these images, it can be safely said that the magnitude of lattice tilt varies totally more than 16 arcsec within the measured area. Typical spatial sizes of the equi-tilt grains range from 10 to 100 μm in this sample.

Fig. 7. Equi-tilt maps taken at two different angles at (a) -7.9 arcsec and (b) $+7.9$ arcsec around the Bragg peak ($\omega = 0$ arcsec). Contrasts seem to be complementary between (a) and (b).

A similar feature of the lattice plane tilt, but the tilt of much larger angles, was also observed in SIMOX wafers. Then, it is clear that the SOI layers formed on the BOX layer are more or less strained regardless of SOI wafer processes and magnitude of the lattice tilt depends on the BOX thickness and the SOI process conditions.

At present, major reason for such a large lattice tilt in the SOI layer has not been completely understood yet. However, it is clear that, before the initial BOX formation or the wafer bonding process or the thinning process, lattice planes in the SOI layer may be as flat as that of the substrate and no lattice tilt exists in the SOI layer. Question is when and how those lattice tilts in the SOI layer are introduced. It is much likely that the polishing damages can be excluded because any peak shift or any angular spread of the reflection curves has not been observed in the case of thicker (more than 100 μm) SOI

layers. A similar experiment has been proved to be effective for the study of grain orientation distribution or crystal structure variation in some metals, e.g., electrochemically plated chromium layers, which results being presented at the meeting.

4. CONCLUSIONS

An x-ray microbeam formed by successive asymmetric Bragg-reflections from double-crystal collimators combined with synchrotron radiation x-rays was used to measure the local minute strain in Si crystals.

Strain distribution around SiO₂/Si film edges and that in SOI layers were measured by means of RCMs and also RSMs using the analyzer crystal. From those results, the local strain can be analyzed in terms of both the lattice tilt variation and the lattice parameter variation. In the case of SiO₂/Si film edge, the lattices shrink vertically at bare Si regions and expand at SiO₂ film regions near the SiO₂ film edge.

Strain measurements of the SOI layers reveal that the top SOI layer consists of many small grains inclined from each other without any lattice parameter change. The present strain analysis using the parallel x-ray microbeam is a powerful technique to get information of the strain features and distribution in single crystals used for other electronic devices such as light emitting diodes [17] or laser diodes [18].

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