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SURFACE MORPHOLOGY OF MECHANICALLY AND CHEMICALLY POLISHED SEMICONDUCTOR WAFERS

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Abstract In the X-ray measurement of crystal truncation rod (CTR) scattering from a flat crystal surface, the conventional symmetric reflection geometry with the use of a 4-circle diffractometer is shown to be still effective. Some advantages of using an imaging plate for the observation of X-ray CTR scattering are also presented. Several fundamental aspects of X-ray CTR scattering are demonstrated using several Si wafer surfaces with different degree of flatness. It is possible to characterize, by non-destructive methods, semiconductor surfaces which are treated for industrial use.

1- INTRODUCTION

In the X-ray scattering from the surface of semiconductor single crystal wafers, a rod-shaped scattering can be observed emanating along the surface normal direction from each Bragg point, if the surface is treated by etching after lapping so as to be flat and smooth like a mirror plane /1/. The simple Fourier transform operation for such an observation (on the basis of kinematical diffraction theory) leads directly to the existence of plate-like scatterers which should be parallel to the crystal surface. Yasuami and Harada /2/ interpreted the rod shaped scattering (RSS) as arising from extrinsic plate-like defects under the wafer surface. They surmised that such defects were induced during the mechanical treatment of the wafer surface, because the...
Intensity of the RSS was reduced or disappeared, if a portion of the surface layer was removed by chemical etching.

Extending the study for many other crystals such as lapped and mechano-chemically polished Si and GaAs wafers, liquid phase epitaxially grown GaAs and GaP and cleaved KCl with different surface states, Kashiwagura and Harada, however, found that some contradiction still remained in their interpretation of the observed data. For instance the wafer surfaces that show pronounced RSS were observed to be very flat and smooth by a scanning electron microscope, while the surfaces that did not show such strong RSS were rather rough and irregular. Furthermore, when the depth of the damaged layer under the surface was estimated from the intensity change of the RSS due to the change of the glancing incidence angle of x-rays to the crystal surface, it was approximately 0±1μm. These facts suggest that the RSS may arise from the surface itself.

The characteristics of the above observation are consistent with the surface scattering demonstrated by Andrews and Cowley and also by Robinson: that is the scattering due to the abrupt change of charge density at a crystal surface which is now usually referred to as crystal truncation rod (CTR) scattering. The CTR scattering is essentially the same sort of scattering as that known as specular reflection in RHEED patterns. From the analysis of this specular reflection, Ichimiya has shown that it is possible to obtain fairly accurately the atomic arrangement on a crystal surface. The analysis is based on the dynamical diffraction theory, but it is essentially the same kind of analysis as that in x-ray scattering.

The confirmation of the fact that RSS is not due to the existence of plate-like defects and is in fact the CTR scattering was demonstrated by the direct observation of lattice images from high resolution transmission electron microscopy. Once it has been confirmed that the RSS is scattering due to an abrupt truncation of charge density at a crystal surface, all the existing data could be interpreted on the basis of the CTR scattering. As the intensity distribution of the CTR scattering is very sensitive to the treatment of the crystal surface, the analysis of such CTR scattering provides diverse information about the lattice modulation at a crystal surface and interface, such as the surface morphology and surface lattice relaxation on an atomic scale.

In this paper we present, firstly, our method of x-ray measurement of CTR scattering, in which the conventional symmetric reflection geometry, with the use of a 4-circle diffractometer, is shown to be still effective. The use of imaging plates for the measurement of CTR scattering is also illustrated and its advantages are compared with the conventionally used method. After presenting some fundamental aspects of CTR scattering by using Si (111) wafer surfaces with different degrees of flatness, we show that it is possible to characterize semiconductor surfaces which are treated in different ways.

It should be mentioned that all the investigated surfaces of crystals are those which have been mechanically and also chemically treated without any special care for protecting against oxidation. The surfaces are, therefore, naturally oxidized. In the
x-ray measurement of CTR scattering from these samples we have not used any vacuum chamber. Thus, the surfaces of concern in this paper are the interfaces between the crystal surface and the naturally oxidized layers.

2- MEASUREMENT OF X-RAY CTR SCATTERING

We employed three different sorts of instruments for the measurement of x-ray CTR scattering, namely an ordinary 4-circle diffractometer installed on 6kW and 12kW Rigaku rotating-anode x-ray generators, a triple axis diffractometer system in conjunction with a synchrotron radiation source, beam line (BL) 4-C at Photon Factory (PF), KEK, and a Weissenberg type camera on BL-6-A also at PF which was designed for macromolecular crystallography.

2.1- CONVENTIONAL 4-CIRCLE DIFFRACTOMETER

In the measurement of X-ray diffuse scattering, the symmetric reflection geometry is useful in conjunction with a conventional 4-circle diffractometer; in which a sample with extended surface is utilized and the incident beam and the scattered beam make equal angles with respect to the sample face. Because the absorption correction in this geometry is simply given by $1/2\mu$, $\mu$ being the linear absorption coefficient, the observed intensity into an absolute unit is easily converted to $I_0$, if necessary. For the measurement of x-ray CTR scattering this conventional method is still effective, although the background scattering from bulk TDS may be enhanced a little bit, as compared with that obtained from the glancing incidence method.

Aspects of the CTR scattering can be visualized on an intensity contour map of a two dimensional section of reciprocal space. As an illustration of the sort of experiments to be carried out, we show the intensity contour map around the 111 reciprocal lattice point on the (hkl) reciprocal lattice plane obtained from the mechano-chemically (MC) polished surface of a Si (001) wafer in Fig.2.1. In addition to the elliptically-shaped thermal diffuse scattering (TDS), a strong rod-shaped scattering is observed emanating along the [001] direction from the 111 reciprocal lattice point. Such a RSS is the CTR scattering from the (001) surface. It should be noted that the vertical resolution is usually poor compared with the horizontal resolution so that the intensity contour map so obtained is sometimes deformed by such an anisotropic instrumental function.

Intensity profiles across the CTR scattering can be obtained using the \( \omega \)-scan mode. This is very similar to the measurement of a rocking curve of a Bragg reflection where the crystal can be set by \( \chi \) and \( \phi \) circle movements in such a way that the scattering vector \( \mathbf{K} \) is in the scattering plane. In this measurement the observed width of the CTR is given by the convolution of the instrumental resolution function and intrinsic width of the CTR. The resolutions \( \Delta K_x \) and \( \Delta K_y \) in the scattering plane are given in terms of horizontal divergences of the incident and the scattered beam and the scattering angle \( 2\theta \). The approximate magnitudes are of order $2 \times 10^{-3}$ A$^{-1}$. If we set an analyzer crystal in front of the detector we attain high resolution in the horizontal directions. This is the triple crystal diffractometry. Some advantages of
using the triple crystal diffractometry have been demonstrated by Eisenberger /11/ and Iida and Kohra /12/.

**Fig. 2.1** Isointensity contour map in the vicinity of the 111 reciprocal lattice point in the ⟨hhl⟩ reciprocal lattice plane for the mechano-chemically polished Si ⟨001⟩ surface.

**Fig. 2.2** Schematic diagram of the experimental setup for the measurement of x-ray scattering. ωs, 2θs and ωa represent the ω-rotation and the 2θ-rotation of the sample and the ω-rotation of the analyzer crystal, respectively.

### 2.2- TRIPLE CRYSTAL DIFFRACTOMETRY

A synchrotron radiation source monochromated by a double crystal monochromator,
consisting of two Si single crystals, provides a very powerful, parallel and especially narrow beam so that it is very effective for high resolution measurements. We employed the beam line 4-C (designed by Matsushita) of Photon Factory, KEK to which a vertical type Huber 4-circle diffractometer with a Si (111) crystal analyzer was installed /13,14/. In Fig. 2.2, a schematic arrangement of the system is shown; it corresponds to the four crystal arrangement of (+n,−n,+n,−n) setting with a χ−φ cradle at the sample table. The instrumental resolution, investigated by 3-dimensional step scanning of the profiles of the 220 Si Bragg reflection, showed that the FWHM values along 2θ, ω and χ angular directions are 0.0029′, 0.0015′ and 0.5′, respectively. The resolution in the diffracting plane, i.e. the ω−2θ plane, is, therefore, quite good, while the resolution perpendicular to the plane, i.e. along χ−rotation, is almost the same order of magnitude as that of a usual 4-circle diffractometer system (see 2.1). The resolution function in K-space is of a needle-shape with the cross section of $2.6 \times 10^{-4}$ $\AA^{-1}$ $\times 5.7 \times 10^{-4}$ $\AA^{-1}$ and the length of $8.6 \times 10^{-4}$ $\AA^{-1}$. This is a typical characteristic of the ribbon-shaped incident beam attained by the combination of a double crystal monochromator and a synchrotron radiation source.

2.3- OBSERVATION BY IMAGING PLATE

The recent development of the imaging plate (IP) detector /15,16/, having high-quantum efficiency, unlimited count-rate capacity, high resolution (presently around 150 $\mu$m, but this is soon to be improved to around 25 $\mu$m), a dynamic range of from 1 to $10^5$, and high sensitivity (approximately 30 times that of normal photographic x-ray film), offers a solution to nearly all the limitations of current position sensitive detectors except energy discrimination. For these reasons, any diffraction measurement system based on the IP is useful for the measurement of CTR scattering. We present the result of CTR scattering obtained by using a Weissenberg camera on BL-6-A at PF, KEK.

3- FUNDAMENTAL ASPECTS OF CTR
3.1- ORIENTATION OF CTR

Theoretically the orientation of the CTR scattering is expected to be parallel to the normal of the truncated surface, and not to the crystallographic net plane. This is one of the important features of the CTR scattering. Such an effect should be confirmed experimentally.

Harada et al measured the orientation of the CTR scattering from two Si wafer samples the surfaces of which were polished mechano-chemically in the same way but they were of slightly different orientation /8/. The surface of the sample A is almost parallel to the (001) crystallographic net plane, while that of the sample B is tilted by 3 degree from the (001) net plane in the (110) plane. Their results are reproduced in Fig. 3.1 (a) and (b). The intensity profiles across the CTR scattering at q=0.14 a* [see Fig. 2.1, dashed line] from the 111 reciprocal lattice point are illustrated for the two samples A and B, where q is the distance from the reciprocal lattice point along the CTR in the units of the reciprocal lattice constant a*. 

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In the figures we see that the intensity distribution across the CTR scattering along the [110] direction is broader than that along the [110] direction. This is due to the anisotropy of the instrumental resolution, not to an inherent anisotropy of the scattering. However, the peak positions are clearly observed at (1.000, 1.000, 1.140) for $q>0$ and (1.000, 1.000, 0.860) for $q<0$ for the sample A, while they are at (0.994, 1.006, 1.140) and (1.006, 0.994, 0.860) for $q>0$ and $q<0$, respectively, for the sample B. From these results we see that the CTR observed around the 111 reciprocal lattice point is parallel to the [001] direction for the sample A, but it is inclined from the [001] direction for the sample B. The angle of the CTR inclination from the [001] crystallographic direction is estimated to be 3.5 degree in the (110) plane in agreement with the nominal angle of 3 degrees between the crystal surface and (001) plane. These observations are consistent with the theoretical prediction.

**Fig. 3.1** Intensity profiles across the CTR scattering at $q=\pm 0.14[a^*]$ for two samples. (a) The surface of sample A is just parallel to the crystallographic (001) net plane and (b) the surface of sample B tilted nominally by 3° in the (110) plane.

3.2 - WIDTH OF CTR

The width of the CTR scattering is expected to be sensitive to the surface morphology. We prepared two Si (111) wafer samples with a different degree of flatness. One of them, sample C, has a surface which has been MC polished, as a
representative for the flattest surface of all available. The other, sample D, is prepared as a representative for a rough surface, which has been chemically etched by an amount of 30 μm after lapping. This etching is performed for removing a heavily damaged mosaic layer on the surface. Hereafter, this sample is referred to as a lapped surface.

As stated in §2, triple crystal diffractometry presents a high resolution technique for investigating the detailed distribution of the intensity of CTR scattering /13/. The CTR scattering profiles were measured using an ω-scan mode with a step size of 0.001°~0.005° in such a way that the needle shaped resolution function goes across perpendicularly to the rod-shaped scattering. Fig.3·2. shows the results. In the case of MC polished Si(111), Fig.3·2. (a), the full width at half maximum (FWHM) values are very small and independent of q. They are all 12 seconds, including the instrumental broadening of 9 seconds. Thus the intrinsic width is estimated to be 3 seconds. This is of the order of the width of the Bragg reflection peaks expected from the dynamical diffraction theory. In the case of the lapped sample, Fig.3·2. (b), the FWHM values are rather large and depend on q value. The CTR from this sample is, therefore, not of a rod-shape but a cone-shape. The angular divergence of the cone is estimated to be about 2.3 degrees. From this fact it is conceivable that the surface is smoothly modulated something like a sand dune, since the orientation of the CTR should always be normal to the truncated surface. At the present stage, it is difficult to formulate an exact relationship between the observed shape of the CTR

![Graph](image-url)  

Fig.3·2  The ω-scan profiles of the CTR scattering. (a) for sample C; ○, □ and △ represent the ω-scan profiles at q=3.5x10^{-3}, 1.7x10^{-2} and 3.1x10^{-2}[a *], respectively. (b) for the sample D; ○, □ and △ represent the ω-scan profiles at q=6.9x10^{-3}, 2.1x10^{-2} and 3.5x10^{-2} [a *], respectively.
scattering and the surface morphology. We leave this problem open for future discussion.

3.3- INTENSITY DISTRIBUTION ALONG CTR

The intensities of CTR scattering integrated with respect to the $\omega$-scan (Fig. 3.2 (a) and (b)) are plotted against $q^2$ in Fig. 3.3. The solid circles and the open circles represent the intensities for $q<0$ and $q>0$, respectively, for the MC polished Si(111) sample. The solid triangles and the open triangles represent those for the lapped samples in the same way. Roughly speaking, the observed intensity distribution along the rod has a $q^2$ dependence when $|q|$ is small in agreement with the prediction from kinematically diffraction theory for a sharp boundary without modulation at the surface /4,13/. Some deviation from the $q^2$ dependence is noticed for the large $|q|$ region. Besides, an asymmetry between the intensity distribution along the low angle side ($q<0$) and that along the high angle side ($q>0$) from the 111 Bragg point is noticed, especially for the MC polished sample.

![Integrated intensity distributions of the CTR scattering along the central axis are plotted against $q^2$. A considerable asymmetry is noticed between the intensity distribution for $q<0$ and that $q>0$, especially for the sample C.](fig3_3.png)

This asymmetry can be more clearly visualized in the oscillation photograph of the IP. One such observation is reproduced in Fig. 3.4 (a), which was taken from a MC polished Si (111) surface under the following conditions: the incident beam was
monochromated to be 1.02 Å by a Si (111) crystal, the ω-angle around the axis perpendicular to the <111> axis was ±10° around the 111 reciprocal lattice vector and the exposure time was about 2 hours. A strong needle-shaped scattering is observed emanating along the [111] direction from the 111 Bragg point around which elliptical TDS exists. This is the CTR scattering. The asymmetry between the intensity distribution along the + side and that along the − side of q is clearly seen.

![CTR scattering image](image)

Fig. 3.4 Intensity distribution observed by the imaging plate in the vicinity of the 111 reciprocal lattice point from a mechano-chemically polished Si (111) surface. (a) is the contour map in which the horizontal direction coincides with the surface normal direction. (b) is the integrated intensity distribution along the central axis of the CTR scattering against q. The solid line indicates the best fit in a model calculation with least squares fitting.

The intensity distribution perpendicular to the CTR are integrated with an interval of 5 pixels along the CTR in order to evaluate quantitatively the profile of the CTR. The results are plotted against q in Fig. 3.4 (b). The asymmetry as well as the dumping of the intensity with |q| are also seen. These observations show that a deviation from an ideal boundary exists at the surface. It is in fact possible to
interpret the results by introducing a lattice relaxation as well as an atomic density modulation at the interface boundary between the crystal surface and the amorphous oxide layer. The solid curve in the figure shows the best fit to the observed data on the basis of the formula given by Kashihara et al./13/; the parameters which were refined are the atomic population and the lattice distortion on the crystal surface. The result is that the lattice plane at the interface expands by about 3~4% of the bulk Si lattice spacing.

As demonstrated, the intensity asymmetry of the CTR scattering with respect to the reciprocal lattice points provides us with information about the lattice relaxation at the crystal surface or the interface boundary. By using the optical diffraction method it is easy to show that the CTR intensity on the low angle side of a Bragg spot is higher than that on the high-angle side if the surface or the interface lattice spacing has expanded, and vice versa if it has shrunk. We have found by using this kind of CTR analysis that the lattice spacing of a GaAs (001) wafer shrinks at the interface boundary between the (001) crystal surface and the amorphous layer.

4- OBSERVATION OF CTR SCATTERING FROM VARIOUS CRYSTAL SURFACE

4.1 LAPPED AND ETCHED SURFACE

The effect of chemical etching of semiconductor surfaces on the CTR scattering was investigated for the (111), (110) and (001) surfaces of Si and GaAs wafers. Fig.4.1 shows the results for the Si crystals: (a) and (d) for the (111) Si surface and (b) and (c) for the (110) and (001) Si surfaces, respectively. In each figure, the isocontour maps in the vicinity of the 111 reciprocal lattice point on the (hhl) reciprocal lattice plane are reproduced. As for the (111) Si surface, we see from Fig.4.1 (a) and (d) that the CTR remains even after chemical etching by an

![Fig.4.1 Isocontour maps for lapped and chemically etched Si surface; (a) for the (111) surface etched by an amount of 45μm after lapping, (b) for the (110) surface etched by 19μm, (c) for the (001) surface etched by 14μm and (d) for the additionally etched (111) surface of (a) by amount of 117μm. P-P and Q-Q indicate the surface normal direction in each map.](image-url)
amount of 45 μm, and it vanishes almost completely when the surface is etched more than 100 μm. On the other hand, for the (110) and (001) Si surfaces, the CTR disappears almost completely after etching only 15–20 μm as seen from Fig. 4.1 (b) and (c).

These observations indicate that in general the (111) surface becomes flat and smooth, while the (110) and (001) surfaces do not if the surfaces are chemically etched. This seems to strongly relate to the fact that the (111) net planes are the first cleavage planes and are composed of a double layer structure. It is, therefore, considered that the (111) net planes may be removed easily layer by layer during the etching, while the (110) and also (001) planes may not be. In view of the fact that the chemically etched (110) or (001) surfaces are very irregular, whereas the etched (111) surface has a tendency to become easily flat, those (110) and (001) surfaces are considered to consist of micro facets of the (111) planes. The morphology of these surfaces should be confirmed by direct observation by electron microscopy.

Fig. 4.2 shows the results for GaAs crystals. The CTR persists even after heavy chemical etching of 50–200 μm for all the (111), (110) and (001) surfaces. This is the great difference between the GaAs surface and the Si surface. We postulate that this tendency is attributed to the fact that appreciable ionic character still remains in the interatomic bonding of GaAs, although it is essentially of a covalent type. Such a small ionicity may make the surface flat and smooth during the chemical etching by keeping microscopic charge neutrality.

Fig. 4.2 Isointensity contour maps for lapped and chemically etched GaAs surfaces; (a) for the (111) surface etched by an amount of 45 μm after lapping, (b) for the (110) surface etched by 19 μm, (c) for the (001) surface etched by 14 μm and (d) for the additionally etched (110) surface of (a) by 117 μm. P–P and Q–Q indicate the surface normal direction in each map.

4.2- MECHANO-CHEMICALLY POLISHED SURFACE

A similar study to that in 4.1 was carried out for the MC polished (111), (110)
and (001) Si surfaces. In contrast to the case of the as-lapped surface, from which the CTR is not observed because of the heavily damaged mosaic layer, the CTR from as-MC polished surface can be clearly observed. Fig. 4.3 shows the results for the surfaces of the [111], [110] and [001] orientation. The CTRs are observed elongated remarkably and much longer than those from an etched surface [see Fig. 4.1]. This shows the effectiveness of the MC polishing for making surfaces flat and smooth for any surface orientation with low indices.

This is also true for the GaAs surface. The CTR from the MC polished (001) GaAs surface was also elongated, although it still remained after heavy chemical etching in agreement with the result in 4.1. We may say from this CTR observation that the effect of chemical etching on GaAs crystal is quite different from that on the Si crystal.

Fig. 4.3 Isointensity contour maps for a MC polished Si surface; (a) for the (111) surface, (b) for the (110) surface and (c) for the (001) surface.

4.3- EFFECT OF CHEMICAL ETCHING ON MECHANO-CHEMICALLY POLISHED SURFACE

In order to see the effect of chemical etching on the MC polished surface we compared the CTR scatterings from the surfaces before and after the chemical etching. Fig. 4.4 (a) shows the result for an as MC polished Si (111) surface, which shows almost the same pattern as that of Fig. 4.3 (a). Fig. 4.4 (b) and (c) show the CTR scatterings from the etched surfaces of 15 μm and 21 μm, respectively. We see that the CTR scattering which was seen in Fig. 4.4 (a) vanishes almost completely in Fig. 4.4 (b), but remains unchanged in Fig. 4.4 (c), although the amounts of etching are almost of the same order for both the cases.

In spite of many trial observations of CTR scattering, each with different etching conditions, no systematic tendency was found for the effect of chemical etching on the MC polished surface. An unknown factor still seems to exist that plays
an important role in the chemical etching after the MC polishing.

**Fig. 4.4** Isointensity contour maps from the mechano-chemically polished Si (111) wafer, (a) for the as polished surface (almost the same as that of Fig. 4.3 (a)), (b) for the chemically etched surface by an amount of 15μm after the polishing, (c) for the same kind of surface by an amount of 21μm.

4.4 - EFFECT OF ALKALI ETCHING ON Si SURFACE

It is known that alkali etching has a chemical part in mechano-chemical polishing of semiconductor surface. In order to see its role on the surface treatment we examined to see how the surface state changes with the alkali etching by detecting the

**Fig. 4.5** Isointensity contour maps for Si (111) surface; (a) for the chemically etched surface by an amount of 125 μm after lapping in order to reduce the CTR scattering intensity, (c) also for the chemically etched surface by amount of 50μm for the same purpose, (b) and (d) for the alkali etched surfaces of (a) and (c), respectively, which were simply dipped in NaOH (pH~11) for 2.5 hours.
change of the CTR scattering. We prepared two Si (111) wafer samples by treating the surfaces by the usual chemical etching in order to be as rough as possible. One sample started from a lapped surface and the other from a MC polished surface. Fig.4.5 (a) and (c) show the CTRs from such initial surface states and Fig.4.5 (b) and (d) show those from the surfaces after dipping these wafer samples into NaOH solution for 2.5 hours. We see that the CTR scattering which is scarcely visible in Fig.4.5 (a) and (c) is enhanced in Fig.4.5 (b) and (d). Since the intensity of the CTR scattering is considered to be a good measure for the flatness of a surface, these facts indicate that the original rough surfaces changed into flat and smooth surfaces, on an atomic scale, by the simple alkali etching.

4.5- SURFACE OF AN EPITAXIALLY GROWN LAYER

It is possible to apply the x-ray CTR scattering technique to the characterization of the semiconductor surfaces which are epitaxially grown by using the recent advanced technique such as MBE, CVD and LPE methods.

The CTR scattering from epitaxially grown surfaces was compared to that from the substrate wafer. Fig.4.6 shows such comparisons: (a) the results for an epitaxially grown Si/Si (001) surface by chemical vapor deposition (CVD) in which the thickness deposited on the surface is 90μm, and (b) for a MC polished Si (001) surface which is used as the substrate for CVD. We see that the CTR scattering from the CVD surface is much more elongated and sharper than that from the substrate surface. We may, therefore, say that the flatness of the Si (001) surface is remarkably improved on an atomic scale by the deposition in spite of the large thickness of CVD layer.

![Isointensity contour maps](image)

**Fig.4.6** Isointensity contour maps; (a) from the mechano-chemically polished Si (001) wafer, (b) from the epitaxially grown Si/Si (001) surface by CVD on the substrate of the sample in (a).

**Fig.4.7** shows a similar comparison between the liquid phase epitaxially (LPE) grown GaAs/GaAs (001) surface and the MC polished surface as the substrate for LPE. We
see that the length of the CTR scattering is reduced appreciably when a GaAs layer is epitaxially deposited on a GaAs surface by the LPE method. This forms a striking contrast to the case of the CVD grown surface of Si/Si (001). It is clear from this observation that in the present stage the LPE surface of GaAs/GaAs (001) is much less flat than the substrate surface.

Fig. 4.7 Isointensity contour maps; (a) from the mechano-chemically polished GaAs (001) wafer, (b) from the epitaxially grown Si/Si (001) surface by LPE on the substrate of the sample in (a).

CONCLUSIONS

The results presented in this paper clearly demonstrate power of the x-ray CTR scattering technique for non-destructive characterization, on an atomic scale, of semiconductor surfaces which are treated or grown by various methods. It should be remarked that a conventional symmetric reflection geometry with the use of a 4-circle diffractometer in conjunction with a rotating anode x-ray generator is still effective even for the characterization of a crystal surface without using the glancing incidence technique.

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