X-Ray White Beam Interferences on Thin Crystals

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Results of white beam X-ray interference measurements on almost perfect semiconductor wafers are presented. A specific measurement geometry allows for the investigation of diffraction effects on thin wafers down to at least 375 μm with a simple experimental setup (standard lab CT with microfocus X-ray tube). Furthermore, the dynamic diffraction effect of double refraction has been studied in detail for thicker samples. This might lead to a new wafer testing method as the observed dynamic effects are very sensible on crystal quality.

1. Introduction

Transmission X-ray white beam radiography and interferences (TXWRI) is a measurement technique used to obtain radiographic and crystallographic information of a sample with a single X-ray transmission experiment. It can be performed on any X-ray computed tomography or radiography device with X-ray energies of about 30–300 keV. These systems are a widespread tool in material science and engineering. In the past 30 years especially, digital detectors have been improved significantly. Flat panels now offer high speed, a good dynamic, and acceptable spatial resolution compared to X-ray film. Due to this development, we had the opportunity to perform comprehensive experiments of white beam diffraction on semiconductor wafers. The goal was to improve defect localization and quantification as well as to develop a model to calculate the minimum sample thickness for TXWRI measurements.

First experiments with white radiation diffraction leading to high intensity lines (dark on film) have been reported by Determann.[2] Many experiments have been conducted by Brümmer on this topic[3] with a simultaneous imaging of radiography and diffraction lines presented in the latter article and more recently in [1].

TXWRI allows for the investigation of the bulk of thick crystals such as turbine blades made of Ni-based superalloys due to the high X-ray energy. However, the contrast of the diffraction lines is relatively low as they are always superimposed with the radiographic image. This leads to a minimal sample thickness of a few millimeters for metal samples. Additionally, most of the diffracted beam is not registered on the detector in a normal TXWRI geometry due to the overlay with the undiffracted (direct) beam (see Figure 1). The part of the direct beam which fulfills the diffraction condition has a reduced intensity compared to the background because it lacks the flux of the diffracted beam.

The diffraction may be explained by Bragg’s law nλ = 2d sin(Θ) where Θ is the diffraction angle, λ the wavelength, d the lattice plane distance, and n the order of diffraction. For a single wavelength and a thin sample, the resulting beampath is outlined in Figure 2. In this setup, usually not all X-rays are diffracted so that a part of the primary beam remains with a lowered intensity. These remaining rays are called deficiency rays here as their intensity is lower than the average background intensity without diffraction. Furthermore, all rays passing through the sample are partially attenuated. For perfect crystals, anomalous absorption may significantly lower the absorption for rays in diffraction condition.

When using polychromatic radiation (bremsstrahlung), the diffracted beams for each set of lattice planes are spread on a relatively large detector area. The spatial intensity distribution of the bremsstrahlung after diffraction can be calculated from Bragg’s equation and a spectral model like Kramers law. [5] The same applies to the deficiency (undiffracted) rays in diffraction condition. As the diffracted rays in Figure 1 originate from the other side of the sample as the undiffracted ones, a geometric offset between the two spectra arises. The visible intensity on the detector is then the sum of the two spectra—a thin bright line that originates from the high energy side of the spectrum and a broader area of slightly reduced intensity.

In this paper, we present experimental results of a measurement geometry where the detector is placed at (or close to) the focal point of the diffracted beams of different wavelength visible in Figure 1. The sample is then located exactly at the middle between X-ray source and detector; therefore, we call this the symmetric position. For each lattice plane set, a thin line is then visible on the detector where all beams of constructive interference aggregate. The position of this line is independent of the X-ray wavelength and the information about the lattice constant of the sample is lost, but the line contrast increases significantly and the information about crystal orientation and the angles between lattice planes is conserved.

A similar setup has previously been used by Stockmeier for high-energy investigations of thick crystals where the fine

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structure of the reflection lines was of no concern (some of the results presented there were obtained with a non symmetrical setup). The earliest measurements in this geometry known to the authors of this paper were performed by Guinier and Tennevin.[7]

2. Experimental Setup

All experiments were performed on a diondo d2 CT/CL-System with a maximum focus-detector-distance (FDD) of 1167 mm. The X-ray tube (X-RAY-WorX XWT-190-CT with Cu-target) can be operated from 20–190 kV with a current of 0.05–3 mA and has a JIMA-resolution of 2 µm. At normal operation and especially at high currents, the source size is larger (10–50 µm diameter for the experiments described here). The tube window is made of beryllium and no additional filters have been used. The detector is a flat panel (Varex PaxScan 4343DX-I) with 3072 x 3072 px² and 139 µm pixel pitch. The cover plate of the active area is made of carbon fiber composite; the minimum X-ray energy for detection is 30 keV according to the manufacturer. In practice, X-rays of lower energies can be detected but their contribution is negligible due to the low detection efficiency.

In the experiments described here, the focus-object distance (FOD) was varied while the focus-detector-distance (FDD) was kept constant to avoid brightness changes. FDD is 800 mm unless otherwise noted, FOD is always given relative to the center of the sample. When an appropriate FOD value for a given sample is found, the sample can then be moved vertical through the beam to scan the whole area for defects (see Figure 12). The image scale is always specified in relation to distances on the detector and not on the sample.

3. Results

In Figure 3, a series of TXWRI images can be found with a FOD varied around the symmetric position (400 mm). At this position, one sharp line is visible for each set of lattice planes. At sample positions near to that value, this line splits into two relatively sharp lines. At an FOD of 350 mm, far from the symmetric case, the reflections are hardly visible at all as the intensity of the different wavelengths is then spread over a larger area.

Due to the high amplification of the line contrast in the symmetric geometry, interference lines could be observed on silicon wafers down to a thickness of 375 µm (see the image series in Figure 4).

For a more detailed investigation of the line structure near the symmetric sample position, an 8 mm thick silicon wafer has been used. A image series with a fine stepping of the sample position is reproduced in Figure 5. It can be observed that two sample positions (FOD = 396 mm and 404 mm) exist where the reflections converge into one line. For sample positions between these values, the lines split again and appear more diffuse.

Line profiles of the 220 reflections from the measurements in Figure 5 have been extracted and are plotted in Figure 6 versus the sample position. They have been integrated over a 300–400 pixels wide strip of the reflection to reduce noise. The intensity
Figure 4. TXWRI images of a Si-Wafer (111) with a thickness of 375 $\mu$m, 50 kV, FDD: 600 mm, FOD from left to right: 295.5, 298.5, 301.5, 304.5 mm. The width of each image equals 69.5 mm.

Figure 5. TXWRI images of a 8 mm thick Si wafer (100 orientation), 50 kV, FOD from left to right: 380, 390, 396, 400, and 404 mm. The width of each image is 55.6 mm.

Figure 6. Line profiles across the 220 reflection of the 8 mm thick Si Wafer (50 kV) versus sample position. Profiles for each sample position are normalized to reduce the influence of fluctuations of the X-ray source intensity.

of the individual profiles (one for each FOD value) was divided by the average intensity of all profiles. This normalization was necessary to reduce the influence of small fluctuations of the X-ray source intensity over time.

In the plot in Figure 6, two pairs of parallel lines become visible forming a central rhombus with four intersection points. Two bright spots at FOD = 396 mm and 404 mm, where a single bright line is visible in Figure 5 and two spots at the exact symmetric position of FOD = 400 mm. The latter spots belong to two thin lines of equal intensity that are visible on the detector image for this FOD value.

The appearance of this two focus positions might be explained by double refraction in the sample. This effect is a consequence of the dynamic theory of X-ray diffraction. An X-ray beam that hits a perfect single crystal under a diffracting angle spreads on incidence with the sample like a fan that is often called Borrman triangle $^8$ (see inset in Figure 7). This triangle restricts the energy flow through the crystal to an angle of plus/minus the diffraction angle $\Theta$ around the lattice planes.

Kato $^9$ calculated the angular energy distribution dependent on the transmission angle with respect to the product of absorption coefficient $\mu$ and thickness $t$ of the sample. For the 8 mm Si wafer and 50 keV X-ray energy ($\mu \cdot t = 0.8$), the intensity should be centered around the border of the triangle. This leads to two X-ray beams traversing the crystal. When exiting the sample, these beams are diffracted again so that two parallel beams form for each wavelength $\lambda$ (see Figure 7). This effect was first observed by Authier. $^{10}$ For experiments with white X-rays, this applies to every energy in the spectrum leading to two parallel beams for every X-ray energy. In Figure 7, this is illustrated for two exemplaric X-ray energies. It can be seen that the rays that have been diffracted by $2\Theta$ when entering the sample (blue lines) all focus on a point mirroring the source behind the sample. The rays that traverse the sample almost undeflected and are diffracted by $\Theta$ when exiting the sample (red lines) focus on a point that is double the sample thickness behind the focus point of the blue lines.
Figure 7. Beam paths for X-rays of two exemplaric wavelengths leading to diffraction angles $\Theta_{1/2}$ of 4° and 10° with a sample thickness of 100 mm. These unrealistic values were chosen for better visibility of the geometric relations. The insets show the Borrmann triangle inside the sample for one wavelength and the resulting images on the detector for a sample movement (from Figure 6).

Figure 8. Line profiles across the 220 reflection of a 8 mm thick Si Wafer, 35 kV (left) and 100 kV (right) versus sample position.

The fact that the two focus positions for both lines are visible at two FOD values with a displacement of exactly the sample thickness leads to the conclusion that the energy in the crystal is only transported at the borders of the Borrmann triangle. This fits well with the calculations of Kato for higher X-ray energies where the factor $\mu \cdot t$ is close to 0 ($\mu \cdot t = 0.34$ for 100 keV and 8 mm of Si). However for $\mu \cdot t = 2$, Kato predicted an almost homogeneous intensity distribution with only a slight rise at the edges. In Figure 8, it can be observed for a tube voltage of 35 kV ($\mu \cdot t = 1.8$) that the two separate beam paths belonging to the edges of the Borrmann triangle are still clearly visible. One has to keep in mind that the detector used only registers X-rays of more than 30 keV according to the manufacturer, so the visible energy range for the 35 kV measurement is far smaller than for the high energy measurements. This explains why even the 440 lines are visible in Figure 8 (left) while for higher energies, the diffraction angles for 440 and 220 overlap due to the broader energy range of the available X-rays.
Figure 9. Line profiles versus sample position of two Si wafers measured at 50 kV. Left image is the 111 reflection of a 2 mm thick wafer, the right image shows the 220 reflection for a thickness of 0.75 mm.

Figure 10. GaAs wafer (thickness 0.5 mm, (100) surface orientation, FOD 400 mm, 100 keV) that was bent by the sample holder (left image). The right image is from a flat sample. The width of each image is 41.7 mm.

For the 2 mm Si sample in Figure 9, only two (broader) lines are visible that intersect at the symmetric sample position. The four intersection points visible in Figure 6 all unite to a bright intersection area around FOD = 400 mm due to the limited resolution of the detector. In the case of a 0.75 mm thick wafer, the intersection area and the widths of the lines in Figure 8 is reduced further.

Similar results could be obtained for a GaAs wafer of 0.5 mm thickness (see Figure 10). In the left image of Figure 10, the GaAs wafer was bent by the sample holder leading to non-parallel diffraction lines while in the image on the right, it was undisturbed.

With a very simple experimental setup, diffraction effects that can only be explained by the dynamic theory of diffraction could be observed on relatively big samples of nearly perfect semiconductor materials. This might enable new testing methods for this type of samples as the dynamic effects are very sensible for imperfections of the crystals. In the next section, further results and possible testing methods are presented.

3.1. Localization of Defects

In order to localize defects, it has to be considered that, as illustrated by Figure 1, a defect can be visible on different sides of the two diffraction lines according to the measurement geometry. If the detector is on the sample side of the symmetric position (FDD < 2FOD), the defect will be visible as a distortion of the diffraction line nearer to the defect. Whereas if FDD > 2FOD, the defect will be visible on the opposite diffraction line. In the exact symmetric geometry, it is impossible to determine on which side the defect is located. A series of TXWRI images with varying FOD values of a single crystal sample with a slightly misoriented region is presented in Figure 11. The diffraction line originating from the misoriented part of the crystal (short vertical streak)

Figure 11. CaF₂ sample with 6 mm thickness rotated by 90°, measured at 100 kV. The dark area on the left is the sample support (floral foam); the defective area is on the top left side, FOD from left to right: 350, 390, 420, 450 mm. The width of each image equals 97.9 mm.
Figure 12. Procedure to create defect localization graphs. The sample is moved through the X-ray beam at a FOD close to the symmetric position. Then the intensities for one of the two diffraction lines are extracted for every sample position and composed to a new image [reslice command in the software ImageJ]. The diameter of the Si wafer is 149 mm.

is easily visible in various TXWRI-positions and moves with the corresponding diffraction line.

3.2. Defect Screening of Wafers

For defect screening of whole wafers, the samples were moved through the X-ray beam in a (almost) symmetric position. It is advantageous to align one diffraction line horizontal on the detector. This reflection will keep its position while moving the sample when the wafers are exactly parallel to the detector. Defects are then shown as intensity and/or position irregularities of this reflection when moving the sample. An convenient way to visualize this is to reslice the image stack of the sample movement as explained in Figure 12.

In Figure 13, four defect screening graphs are shown for decorated silicon wafers and a unetched GaAs wafer with and without visible defects. These samples were measured with a small offset (1–2 mm) with respect to the symmetric position. This allows for a localization of the defects as they are visible on one of the two diffraction lines dependent on their position (see also Figure 14). Furthermore, in this geometry, the contrast of the defects is higher as the signal from the defect is not superimposed with signal from the other side of the sample.

For the defect-free sample, only vertical streaks are visible originating from the varying sensitivity of each detector pixel. In Figure 13b, the two relatively small defective areas near the rim of the wafer show up as two bright points (see also Figure 14). Defects spread over a larger area lead to the more diffuse pattern in Figure 13c. There the diffraction line used for reslicing the image stack was not exactly horizontal; therefore, the streaks originating from the defects are tilted which allows to tell the defect signal apart from the detector noise and irregularities. No defects are visible in Figure 13d for an unetched GaAs wafer, even though it contained a higher dislocation density near the rim.

3.3. Simultaneous Imaging of Radiographic and Crystallographic Information

In Figure 14, a section of the Si wafer with dislocations concentrated near the rim is shown. The defects are visible as dark vertically elongated areas due to the heavy metal incorporation of the
Figure 14. Simultaneous imaging of radiographic and crystallographic defects on an etched silicon wafer (100 orientation, thickness 1.5 mm, 50 kV). Areas with higher dislocation density are dark in the radiography due to heavy metal incorporation from the etchant.

etchant used to make the dislocations visible (chromium was detected with a handheld XRF in these areas). The lower one of the two horizontal diffraction lines, which originates from the defective areas, shows a higher intensity. This is possibly due to the higher scattering power of the metal atoms.

4. Conclusion

TXWRJ measurements in standard geometry (Figure 1) allow only for the investigation of thick and/or strongly scattering samples. With the symmetric geometry, it is possible to investigate silicon wafers with a thickness of only 375 μm which enables the testing of standard wafers for semiconductor production. With thicker wafers, double refraction effects could be investigated, an energy transport through the crystal only near the edges of the Borrmann triangle. This leads to two diffracted rays exiting the sample toward the center of the detector for each incoming ray. This effect was studied at various X-ray energies. For higher energies, our observations are in good agreement with the simulations of Kato, but this model cannot explain why the splitting in two rays is strong even for X-ray energies of only 35 keV.

Additionally, a possible measurement technique for wafer screening was developed. Currently, it only allows for the detection of dislocations on etched silicon wafers due to the heavy metal incorporations from the etchant. However, the experiments on wafers with dislocations have been conducted on relatively thin crystals where the splitting of the beam paths inside the sample could not be observed due to the limited detector resolution. As this splitting can only be explained by dynamic diffraction effects, it should not be present on thick, non-perfect crystals. This applies, for example, to the measurements of CaF₂ in Figure 11; the two main lines visible there can be explained by the kinematic theory of diffraction. When analyzing semiconductor wafers with a thickness of several millimeters and higher dislocation densities, it might therefore be possible to infer from the absence of the double focus position that dislocations are present. Alternatively, a detector with higher resolution could be implemented. This might enable the implementation of this method as a quick and flexible nondestructive wafer testing technique.

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Conflict of Interest

The authors declare no conflict of interest.

Data Availability Statement

The data that support the findings of this study are openly available in “figshare” at https://doi.org/10.6084/m9.figshare.14554836

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[1] J. Bauch, M. Böhling, H.-J. Ullrich, D. Wünsche, Cryst. Res. Technol. 2010, 45, 805.
[2] H. Determann, Schriften der Naturforschenden Gesellschaft in Danzig 1937, 10, 5.
[3] O. Brümmern, Z. Naturforsch. 1958, 13a, 571.
[4] O. Brümmern, Z. Naturforsch. 1960, 15a, 875.
[5] H. A. Krámers, London, Edinburgh, Dublin Philos. Mag. J. Sci. 1923, 46, 275.
[6] M. Stockmeier, A. Magerl, J. Appl. Crystallogr. 2008, 41, 754.
[7] A. Guinier, J. Tennevin, Acta Crystallogr. 1949, 2, 133.
[8] G. Borrmann, G. Hildebrandt, H. Wagner, Z. Angew. Phys. 1955, 142, 406.
[9] N. Kato, Acta Crystallogr. 1960, 13, 349.
[10] A. M. Authier, C.R. Hebdo. Séances Acad. Sci. 1960, 251, 2003.
[11] C. A. Schneider, W. S. Rasband, K. W. Eliceiri, Nature Methods 2012, 9, 6715.