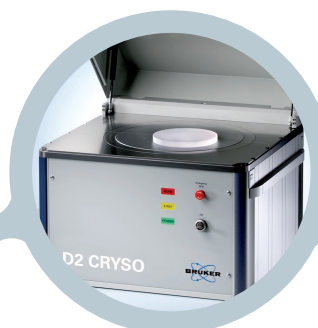


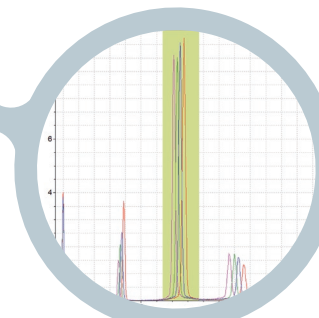
Single crystals



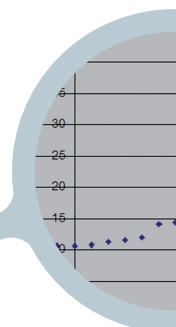
XY-stage with sample holder



D2 CRYSO



Energy spectra



Radius of curvature

Lab Report XRD 73

D2 CRYSO

Determination of the Radius of Curvature on Bent Crystals

The D2 CRYSO is a desktop diffractometer utilizing energy dispersive X-ray diffraction (EDXRD). Due to its flexible sample handling the D2 CRYSO is the right tool for both research&development and quality control, respectively. Furthermore, the easy-to-use software of the D2 CRYSO enables routine operation and data evaluation with minimum to no user intervention.

Introduction

A newly designed XY-stage with motorized 150 mm travel extends the analytical capabilities of the D2 CRYSO. Fully software supported, the crystallographic orientation of single crystals up to 150 mm diameter can be investigated with spatial resolution. Fully automated, the D2 CRYSO determines the orientation map of the sample based on customer defined measurement grids.

The orientation maps are graphically displayed or, alternatively, the results can be listed in a table. The entire set of measurement data and the results of the evaluation process are automatically stored in a project file for archiving and potential later data verification purposes.

This report describes the measurements on several bent single crystal materials such as a Si-substrate of a multilayer, a graphite crystal, and SiGe-gradient-crystals.

Method

For determining the radius of curvature the following method was applied. For a circularly bent single crystal with the bending radius r the angle β between surface normal on location x and the z -axis (figure 1) can be described by equation 1:

$$\beta(x) = \frac{x}{r} \cdot \frac{180^\circ}{\pi} \quad (1)$$

Considering a lattice plane which is aligned approximately parallel to the crystal's surface and if the orientation angle $\beta(0)$ in the x - z -plane (figure 1) is determinable, an analogue equation applies:

$$\beta(x) = \frac{1}{r} \cdot \frac{180^\circ}{\pi} \cdot x + \beta(0) \quad (2)$$

The D2 CRYSO with its XY-stage allows measuring the orientation angles δ and γ of the lattice plane with reference to the measurement coordinate system for any desired location on the crystal.

The angles δ and γ are orientation angles in spherical coordinates. The angles $\beta(x)$ can be calculated according to equation 3:

$$\beta(x) = 2 \cdot \arctan\left(\sin \gamma \cdot \tan \frac{\delta}{2}\right) \quad (3)$$

Finally, the radius of curvature can be calculated from the slope of the linear regression curve of β versus x -position.

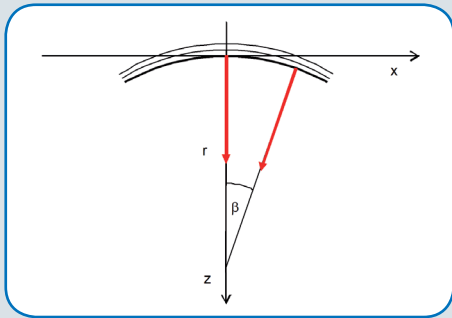


Fig. 1: Schematic illustration of determination of the orientation angle



Fig. 2: Photo of the XY-stage and sample holder

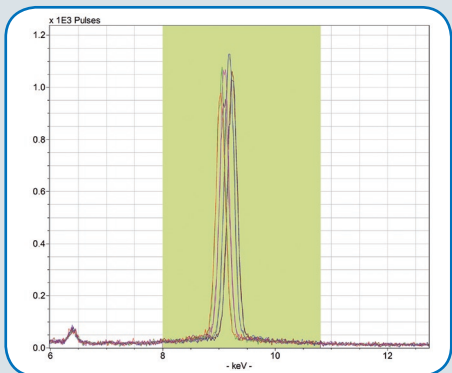


Fig. 3: EDRXRD spectra collected at the Si (400) reflection of the multi-layer sample

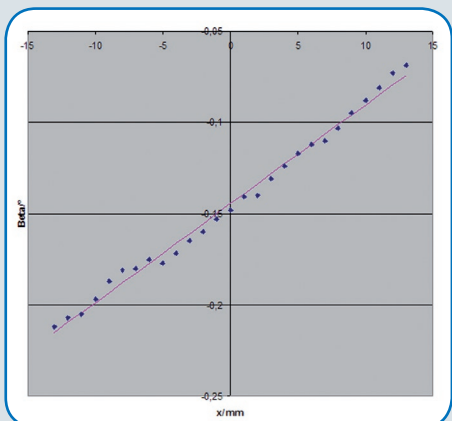


Fig. 4: Orientation angle $\beta(x)$ and linear regression for the multi-layer sample

Experiment

The crystals to be examined were positioned concentrically in the sample holder of the XY-stage (figure 2). They were aligned parallel to the x-direction, the sample surface upside down.

The measurements were performed using CRYSO MEASURE. Line scans were performed in x- and in y-direction, respectively. Each orientation angle β was calculated automatically and was added to the results list.

Results

Bent multilayer

The first sample investigated was a bent multi-layer mirror of $29 \times 9 \text{ mm}^2$ size. A bent multi-layer mirror acts as a monochromator and beam conditioner for the primary beam of a total external X-ray fluorescence (TXRF) spectrometer for example. As such the multi-layer uniformity as well as the figure error essentially influences the shape of the beam of the mirror, thus the performance of the TXRF instrument.

For determining the orientations with spatial resolution the (400) reflection of the silicon substrate of the mirror was investigated. The change of the angle β is smaller at larger radii of curvature. Therefore, measurements were performed for eight rotation angles at each position (figure 3). For the center line ($y=0$) a radius of $10,560 \pm 190 \text{ mm}$ was determined (figure 4).

An overall topography of the bending was generated by determining the orientation angles across the sample surface of $26 \text{ mm} \times 7 \text{ mm}$ and with a grid of 1 mm . Figure 5 shows a colour coded map of the orientation angle $\beta(x,y)$. The referring bending radii $r(x, y=\text{constant})$ and $r(x=\text{constant}, y)$ are shown on figure 6 and 7 respectively. Obviously the bending radii are increasing towards the edges of the mirror. So, it can be concluded that the investigated mirror will show some aberration error caused by the deviation of the perfect curvature figure.

Bent graphite monochromator

Another commonly used bent crystal is a graphite monochromator. It is for example used to suppress $K\beta$ -lines or fluorescence radiation when investigating powder samples with an X-ray diffractometer. Besides other parameters, the bending radius of the graphite crystal influences the reflection characteristic. The graphite crystal examined had dimensions of $25 \times 15 \text{ mm}^2$ with a nominal bending radius of 400 mm .

The (004) reflection was used for measurement of the orientation angle. Figure 8 shows the EDXRD data taken for different rotation angles. Figure 9 shows the orientation angle $\beta(x)$ and the linear regression resulting in a radius of curvature of 377 ± 10 mm, significantly below specification.

Radius of curvature of SiGe-gradient-crystals

The change of the Ge concentration within the bulk of SiGe-gradient-crystals causes a strain in the crystal lattice. The caused macroscopic curvature was determined with the D2 CRYSO. Additionally, it was possible to determine the intensities of the Ge-K α fluorescence emission from the EDXRD energy spectra (figure 10). Using these intensities it was possible to determine a relative Ge content with spatial resolution.

Two different single SiGe crystals with a (111) surface orientation were investigated: crystal #1 had a diameter of 30 mm, crystal #2 a diameter of 50 mm. Measured cross the crystal surface along two orthogonal directions radii of curvature of 18.4 ± 0.80 m and 21.2 ± 0.85 m were determined for crystal #1, respectively.

Crystal #2 had a radius of curvature of 33.3 ± 0.66 m in one direction. The inner range between $y = -8$ mm and $y = 8$ mm showed a distinctly smaller radius of 26.3 ± 0.7 m (figure 11). This result represents the so-called crystal core, which formation happens to occur in the [111] direction during the crystal growth. This radius change correlates with the detected significant change of the Ge-concentration (figure 11, bottom part).

The d-spacing of the reflection used for the measurements of the orientation is calculated automatically. With the knowledge of the crystal structure obviously the lattice constant can be derived. Assuming the validity of Vegard's law the linear dependence of lattice spacing versus concentration in a solid solution crystal, the Ge content can be calculated. The results are given in table 1.

Taking the values from table 1 a ratio of 0.76 of the Ge content of crystal #1 versus crystal #2 was calculated. This result corresponds very well with the value of 0.75, which was calculated from the Ge-K α emission line intensities, thus the results are self-consistent.

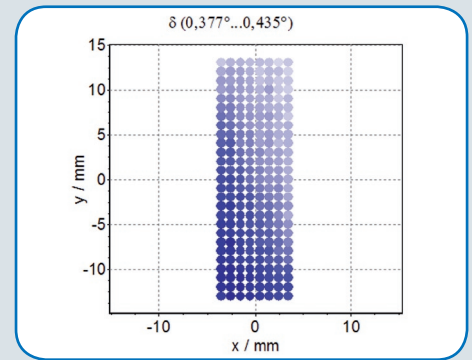


Fig. 5: Orientation map of the multi-layer sample

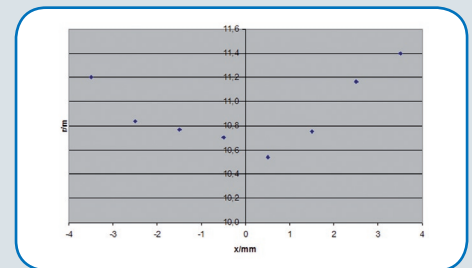


Fig. 6: Radius of curvature $r(x)$

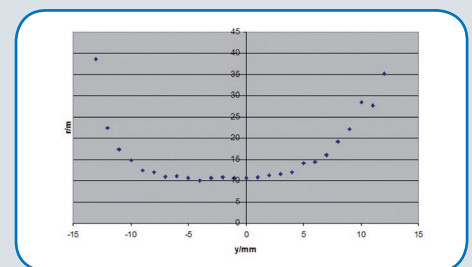


Fig. 7: Radius of curvature $r(y)$

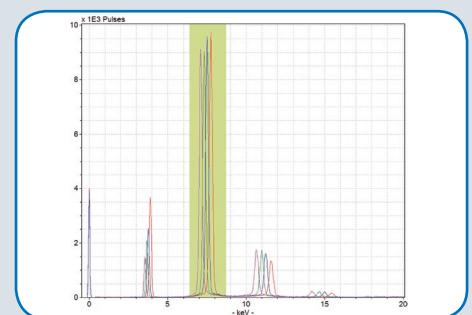


Fig. 8: Energy spectra collected on the graphite (004) reflection

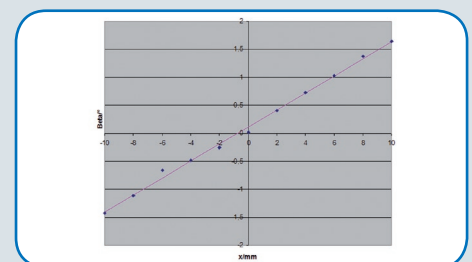


Fig. 9: Orientation angle $\beta(x)$ and linear regression

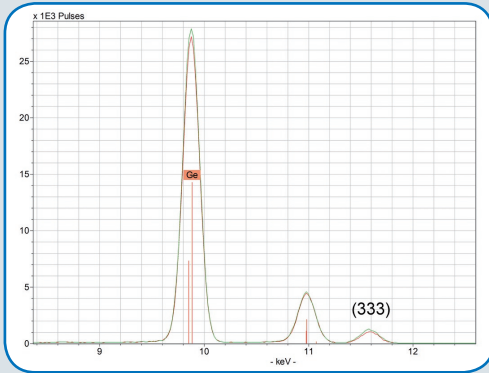


Fig. 10: Two energy spectra obtained at crystal #2, showing the Ge K-lines and the (333) reflection; red: crystal center, green: crystal border

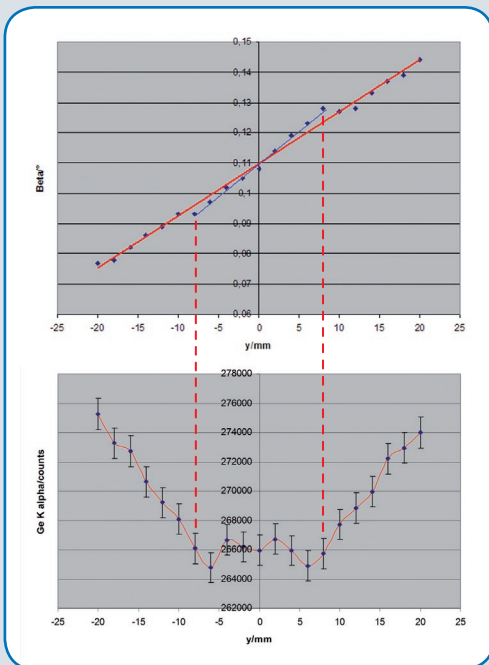


Fig. 11: Experimentally determined angle β dependent on crystal location and regression line (above) and intensities of Ge $K\alpha$ line dependent on y (below)

	d obs. (333) / Å	a / Å	Ge concentration
Si		5.4309	0.00 %
Ge		5.6570	100.00 %
Crystal #1	1.04685	5.4396	3.85 %
Crystal #2	1.04737	5.4423	5.04 %

Tab. 1: Results of the investigation of the two SiGe samples

Conclusion

The new version of D2 CRYSO with its XY-stage was used to determine the radius of curvature of three different types of bent single crystals. The applied method is based on the measurement of orientation angles of lattice planes in dependence of the location on the sample surface. Performing fully automatic measurements of line and area maps, the D2 CRYSO is particularly suited for determining the radius of curvature in R&D as well as in quality control.

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