

## DEVELOPMENT OF A METHODOLOGY FOR ANALYSIS OF THE CRYSTALLINE QUALITY OF SINGLE CRYSTALS

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Recently, a study of the crystalline quality of Czochralski grown  $\text{BaLiF}_3$  single crystals has been published [1]. In the study, certain characteristics of intensity curves (rocking curves), obtained by neutron diffraction, are correlated to several parameters involved in the growth method. As pointed out by authors, neutrons are better than x-rays for this purpose since they give information about the bulk of the crystal. A neutron beam has, in general, a large cross-sectional area and a sample can be completely immersed in it. Since neutrons are high-penetrating particles, for most materials, it is possible to observe all crystalline domains of a sample simultaneously. An x-ray beam, on the other hand, is infinitely less penetrating than a neutron beam and has, in general, a small cross-sectional area. In this case, if the crystal is large, the x-ray diffraction gives information about a very small portion of the crystal [1]. Unless the beam impinges upon a domain boundary, a single domain is observed in a rocking curve.

Although the methodology employed in the above study led to useful results, it is not suitable for a complete characterization of the crystalline domains of a sample. This present work establishes a better methodology for the analysis of the crystalline quality of single crystals, by using tridimensional rocking curves measured by neutron diffraction. Tridimensional Intensity ( $I$ ) $\omega\chi$  plots, measured as  $I\omega$  rocking curves for  $\chi$  stepping in a convenient interval, show, in general, several domains located at different positions in the  $\omega\chi$  grid. In principle, it is possible to determine the number of such domains, their relative intensities, mean breadth of each one and angular dispersion between them. From a macroscopic point of view, determination of these characteristics corresponds to an evaluation of the crystalline quality of the crystal under study.

For the development of the method, a tridimensional rocking curve  $I\omega\chi$  was constructed with data obtained with an aluminum (single) crystal, shaped as a square plate  $8 \times 8 \times 1.5 \text{ cm}^3$  and oriented with (111) planes parallel to the  $8 \times 8 \text{ cm}^2$  face. Data were collected on the IPEN neutron diffractometer, installed at the 2-MW IEA-R1m research reactor. In the beginning of the data analysis, the individual  $I\omega$  and  $I\chi$  rocking curves, which constitute the tridimensional rocking curve, were fitted by Gaussians. In continuation of the analysis, the instrumental broadenings in both directions  $\omega$  and  $\chi$  were deconvolved from the individual Gaussians found in the fitting procedure applied for each rocking curve  $I\omega$  and  $I\chi$ , respectively. The instrumental broadenings were determined with a (perfect) lithium fluoride (LiF) single crystal as the full widths at half maximum (FWHM) of the rocking curves measured around the  $\omega$  and  $\chi$  axes. Owing to an enhanced Lorentz factor in direction  $\chi$ , the scale in this direction had to be "shrunk" by a correction factor. This was done in order to make the FWHM of domains in direction  $\chi$  equivalent to those found in direction  $\omega$ . The final step of the analysis was carried out by using contour maps. The construction of contour maps, on the bases of the tridimensional rocking curves, turns easier the characterization of

domains. A contour map for the aluminum crystal was constructed. In this map, the five domains found in the analysis appear individualized. Individualization of domains facilitates the determination of the breadth of each one, as well as the angular distances between them. Table 1 lists the results found for the breadth of the domains, represented by the FWHM in both directions, and their relative intensities ( $I_r$ ) Table 2 lists the angular distances  $\Delta$  between each domain and all others.

*Table 1:FWHM, for both directions  $\omega$  and  $\chi$ , and relative intensities of the domains in the aluminum crystal.*

Domain	FWHM <sub><math>\omega</math></sub> (°)	FWHM <sub><math>\chi</math></sub> (°)	I <sub>r</sub> (%)
1	0.1228	0.1248	16.70
2	0.1173	0.1678	38.26
3	0.2236	0.2692	100.00
4	0.2061	0.2497	77.16
5	0.0953	0.1756	11.71

*Table 2:Angular distances between domains in the aluminum crystal.*

Combination	Angular distance $\Delta$ (°)
1-2	0.1510
1-3	0.2655
1-4	0.4083
1-5	0.5628
2-3	0.1379
2-4	0.2864
2-5	0.4373
3-4	0.1487
3-5	0.3011
4-5	0.1544

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#### REFERENCE

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