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"STRUCTURAL CHARACTERIZATION OF ADVANCED CERAMICS USING THE NEUTRON DIFFRACTOMETER DEVELOPED BY INSTITUTO DE PESQUISAS ENERGÉTICAS E NUCLEARES (IPEN)"

C. B. R. PARENTE

V. L. MAZZOCCHI

INSTITUTO DE PESQUISAS ENERGÉTICAS E NUCLEARES
COMISSÃO NACIONAL DE ENERGIA NUCLEAR
CAIXA POSTAL 11049, CEP 05499 - SÃO PAULO - SP - BRAZIL

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PROGRESS REPORT

'Structural Characterization of Advanced Ceramics Using Neutron Diffractometer Developed by Instituto de Pesquisas Energéticas e Nucleares (IPEN)'

C. B. R. Parente and V. L. Mazzocchi

IPEN-CNEN/SP

IN-PROGRESS RESEARCHES:

- Development of a methodology for analysis of the crystalline quality of single crystals.

Sabrina Metairon, Vera L. Mazzocchi , Carlos B. R. Parente and Sonia L. Baldochi.

Recently, a study of the crystalline quality of Czochralski grown $BaLiF_3$ single crystals has been published [1]. In the study, intensity curves (rocking curves) obtained by neutron diffraction are related to several parameters involved in the growth method. Although the methodology employed in the study led to useful results, characterization of domains obtained by its employment lacks completeness. This present study is an attempt to develop a better methodology for the analysis of the crystalline quality of single crystals by using rocking curves measured by neutron diffraction. Neutrons are better than x-rays for this purpose since they can give information about the bulk of the crystal. X-rays, on the other hand, give information about a very small portion of the crystal [1]. Tridimensional Intensity x ω x χ plots, obtained from a single crystal as several Intensity x ω rocking curves for χ stepping in a convenient interval, show, in general, more than a single mosaic domain. In principle, it is possible to determine number of domains, their relative intensities, mosaic spread 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 single crystal under study. Fig. 1. shows a tridimensional plot obtained from a $BaLiF_3$ single crystal and the corresponding contour map. A fit of Gaussians to

a few of the individual Intensity x ω rocking curves showed at least 5 mosaic domains. For this reason, one can say that the crystal has a bad crystalline quality. It should be noted that domains for the scans in the χ direction appear much more wider than in the ω direction. This is due to the worst resolution of the Soller collimator in the χ direction. Use of a special collimator for neutron multiple diffraction, which collimates in both vertical and horizontal directions, can partially eliminates this problem. Fig. 2. is similar to Fig. 1., except that it represents a $BaLiF_3$ single crystal with a much better crystalline quality. In fact, Gaussian fittings to a few individual Intensity x ω rocking curves showed no more than 2 mosaic domains, one being dominant over the other.

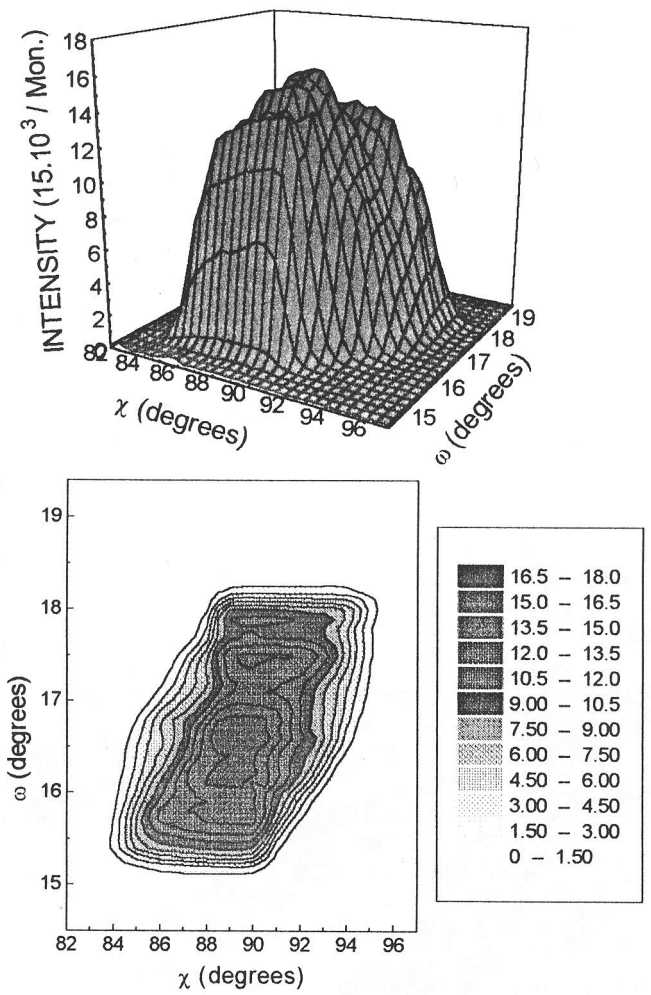

Figure 1. - Tridimensional plot (above) obtained with reflection 111 from a $BaLiF_3$ single crystal and corresponding contour map (below). Crystalline quality is very poor.

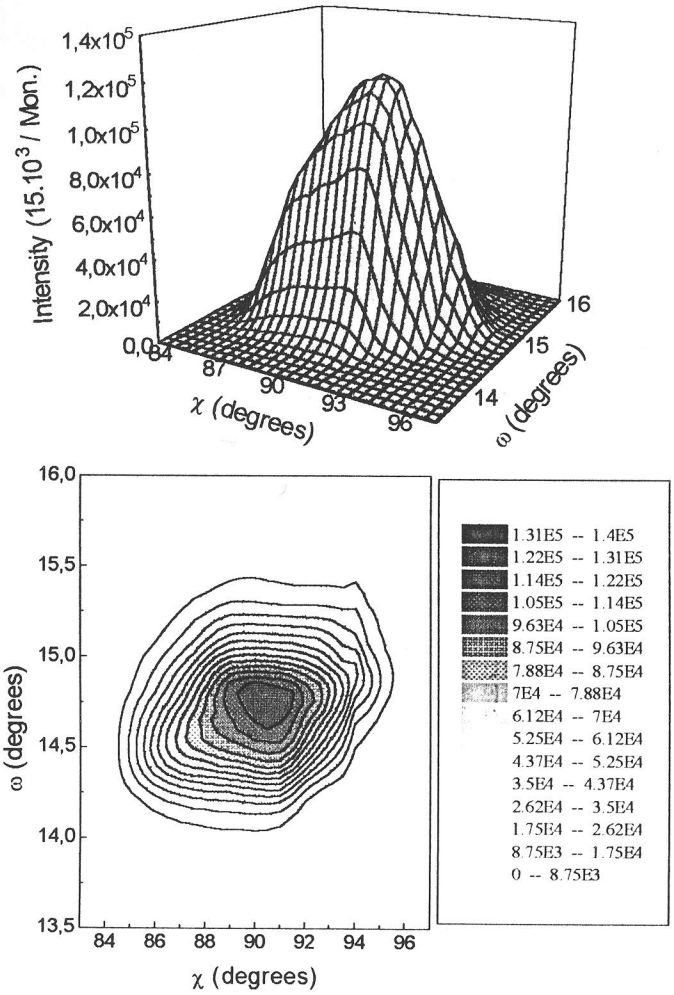

Figure 2. - Tridimensional plot (above) obtained with reflection 111 from a $BaLiF_3$ single crystal and corresponding contour map (below). Crystalline quality is good.

Fig. 3. shows 3 rocking curves obtained from a $Bi_{12}TiO_2$ (BTO) single crystal. Such curves were measured with reflections 200, 002 and 020. Scattering vector of reflection 200 was parallel to the growth direction [100]. Scattering vectors of reflections 002 and 020 were perpendicular to growth direction. Gaussian fittings revealed the existence of several domains in the crystal. Although obtained from a same crystal, rocking curves BTO (002) and BTO (020) revealed 5 mosaic domains whereas rocking curve BTO (200) revealed only 4 domains. It should be understood that it is impossible to determine unequivocally number and angular positions of domains from bidimensional rocking curves. Table I lists the parameters of the mosaic domains found for the rocking curves of Fig. 3.

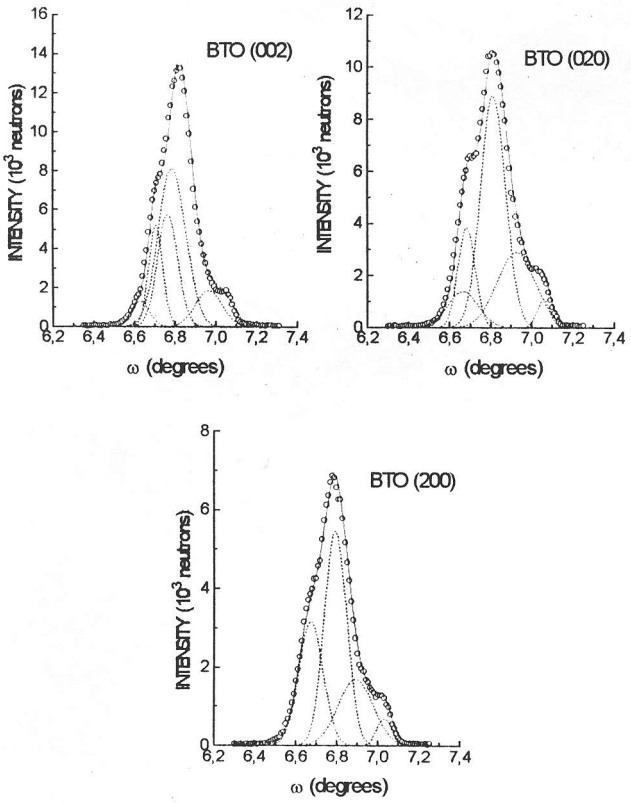

Figure 3. - Rocking curves for the growth direction, BTO (200), and for two directions perpendicular to growth direction, BTO (002) and (020) of a $Bi_{12}TiO_2$ single crystal. Gaussian fittings to curves reveal number, position and intensity of mosaic domains. Crystalline quality is poor.

Fig. 4. shows rocking curves obtained with reflections 111 and 110 from a Ni doped $BaLiF_3$ single crystal. Scattering vector of reflection 111 was parallel to the growth direction. Scattering vector of reflection 110 was perpendicular to growth direction. For the growth direction only one domain is observed. The crystal has undoubtedly a quite good quality. As expected for the perpendicular direction, the quality is not so good as for the growth direction [1]. A Gaussian fitting in this case shows 2 domains with different spreads but very close one to other. Table II lists parameters found in the Gaussian fittings for both curves.

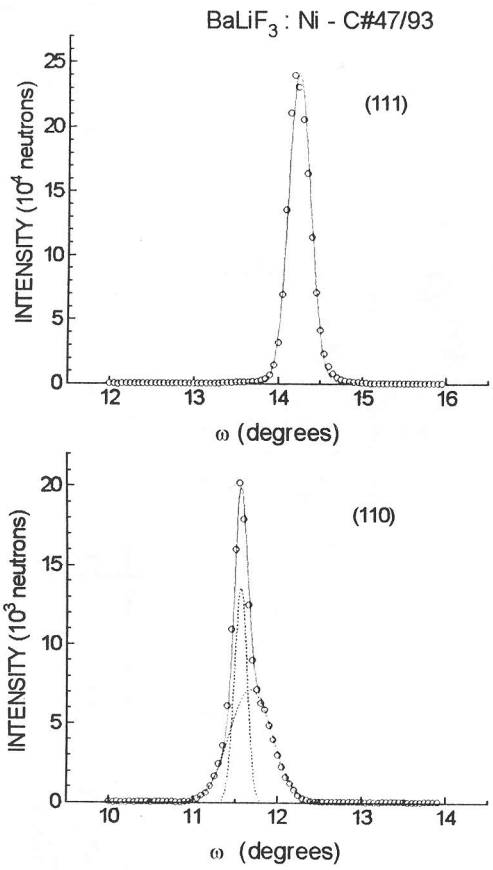

Figure 4. - Rocking curves for the growth direction [111] and a direction [110] perpendicular to growth direction of a Ni doped $BaLiF_3$ single crystal. Crystalline quality is good.

Table I - Parameters for the mosaic domains in the BTO single crystal, found in the Gaussian fittings of rocking curves.

	BTO (002)			BTO (020)			BTO (200)		
domain	**center pos. (degrees)**	**f.w.h.m. (degrees)**	**height (counts)**	**center pos. (degrees)**	**f.w.h.m. (degrees)**	**height (counts)**	**center pos. (degrees)**	**f.w.h.m. (degrees)**	**height (counts)**
1	6.6239	0.0876	1314.8	6.6600	0.1503	1387.3	6.6696	0.1182	3169,4
2	6.7007	0.0687	5217.9	6.6772	0.0831	3849.9	6.7899	0.1081	5463.2
3	6.8088	0.0997	5754.9	6.8067	0.1206	8903.5	6.8878	0.1690	1697.3
4	6.8310	0.1345	8112.0	6.9189	0.1855	2899.1	7.0270	0.0725	693.28
5	7.0101	0.1318	1870.8	7.527	0.0624	1081.2	-	-	-

Table II - Parameters for the mosaic domains in the Ni doped $BaLiF_3$ single crystal, found in the Gaussian fittings of rocking curves.

	$BaLiF_3$: Ni (111)			**$BaLiF_3$: Ni (110)**		
domain	**center pos. (degrees)**	**f.w.h.m. (degrees)**	**height (counts)**	**center pos. (degrees)**	**f.w.h.m. (degrees)**	**height (counts)**
1	14.24	0.1292	240600	11.55	0.0758	13530
2	-	-	-	11.67	0.2488	7173

Fig. 5. shows rocking curves for two Pb doped $BaLiF_3$ single crystals, both obtained in the growth direction. Crystals were doped with 5 wt. pct. and 2 wt. pct. of Pb and called C#41 and C#42, respectively. They were both grown by Czochralski method. Crystal C#42 has a very poor crystalline quality, probably due to extended defects produced by inclusion of Pb in the lattice. The same occurs with crystal C#41, although in a lesser degree owing to the smaller concentration of Pb in the lattice. It is expected that the crystalline quality be improved in next crystals in order to allow n.m.d. measurements. No Gaussian fittings were done for these samples.

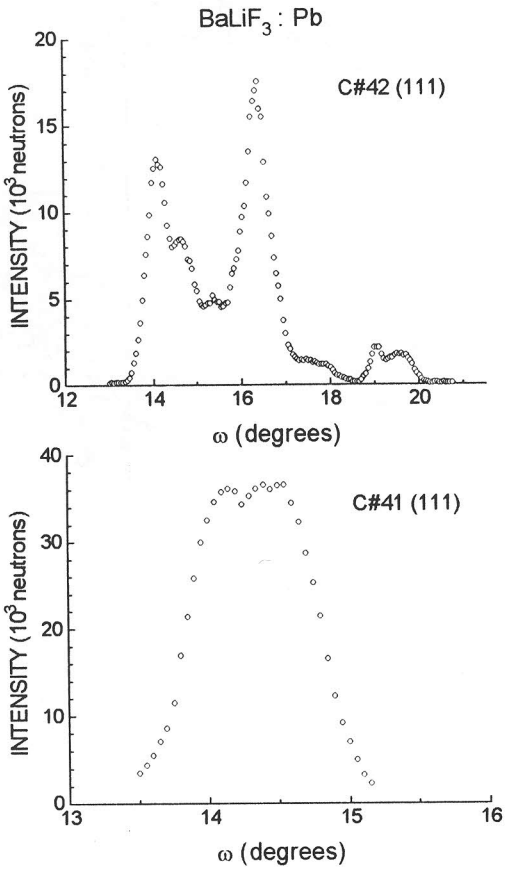

Figure 5. - Rocking curves for the growth direction of two Pb doped $BaLiF_3$ single crystals. Crystals C#41 and C#42 have concentrations 1 wt. pct. and 2 wt. pct. Pb, respectively. Although both have a bad crystalline quality, C#41 is much better than C#42.

- Phases present in high - Tc ceramic superconductors of the Bi-Sr-Ca-Cu-O system.

Reginaldo Muccillo, Eliana N.S. Muccillo, Vera L. Mazzocchi and Carlos B.R. Parente.

In a previous Report, Parente and Mazzocchi [2] observed that the signal-to-background ratios, calculated for the peaks in neutron powder patterns of superconductors of the Bi-Sr-Ca-Cu-O system, have very low figures. Peaks are very broad with low intensity. This was ascribed to the small size of the crystallites in the samples. We have also observed that a heat treatment of samples, consisting of annealing at approximately 1000K for several hours, produced no increase in the mean crystallite

size. As a matter of fact, such annealing produced even a decrease in the mean size. Results also included a neutron powder pattern for a sample obtained by addition of lead during sintering of the superconductor. The signal-to-background ratios in this case have even lower figures, indicating that the mean crystalitte size is smaller than in the case of annealed pure superconductors.

In continuation of the reasearch, we have used a different heat treatment which will be described later on. We decided to use samples prepared by addition of lead although, as mentioned above, they produce poor figures for the signal-to-background ratio. Addition of lead is reported to increase the fraction of the higher Tc phase 2223 increasing the uniformity in the composition of this phase as well [3]. Both are desirable characteristics of the Bi-Sr-Ca-Cu-O system. For this reason, we have opted for this kind of superconductors. It should be mentioned that addition of Pb provokes a partial substitution of Pb for Bi in the lattice. A general formula, for the superconductor containing Pb can be written as $Bi_{2-x}Pb_xSr_2Ca_2Cu_3O_y$.

Samples of high-Tc ceramic superconductors are being currently prepared in the Materials Science Division at IPEN. A sample containing Pb to be used in this work was prepared by the solution technique [4]. The initial stoichiometry had an excess of 10 at. pct. of Ca and 16.7 at. pct. of Cu resulting in the high-Tc superconductor $Bi_{1.7}Pb_{0.3}Sr_2Ca_{2.2}Cu_{3.5}O_{10.6}$. The sample was annealed at circa 1103K for 30 hours. With this sample, called SC-114A, we obtained the neutron powder pattern showed in Fig. 6.. After measurements, a new heat treatment was applied to the sample. It consisted of a quick annealing at 1113K for 30 minutes. This temperature is about 5K below the eutetic point of the compound. Fig. 7. shows the neutron powder pattern obtained with such sample, called SC-114B. It should be noted that the preset counting for the monitor in SC-114B pattern is twice that in SC-114A pattern. To allow an easy observation of differences between the two patterns, intensity scale in SC-114A has an extension twice that in SC-114B. A simple qualitative analysis of results shows that peaks for sample SC-114B are slightly greater than peaks for sample SC-114A. Another observation is that the background level in SC-114A is almost twice that in SC-114B. Both are evidences that crystallites in sample SC-114B have slightly greater sizes than in sample SC-114A.

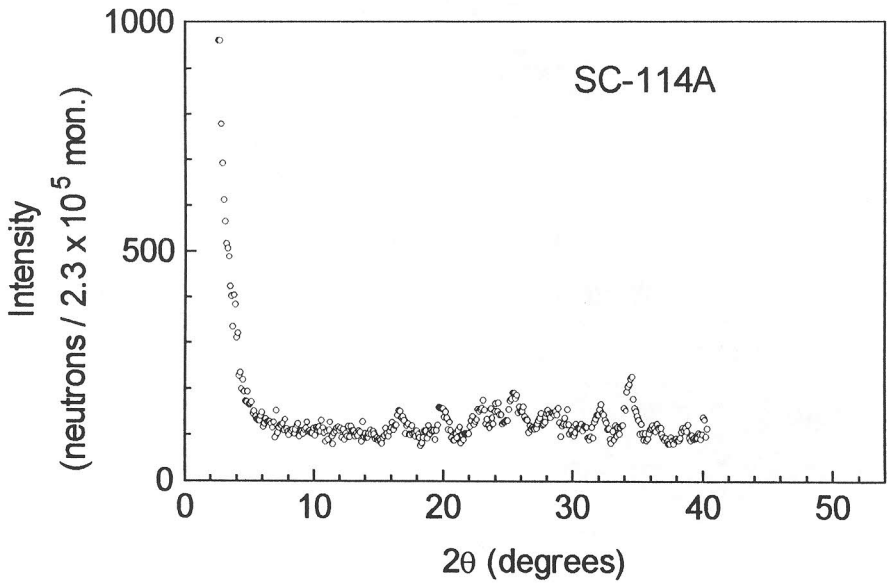

Figure 6. - Neutron powder pattern obtained with the high-Tc superconductor $Bi_{1.7}Pb_{0.3}Sr_2Ca_{2.2}Cu_{3.5}O_{10.6}$ after annealing at ca. 1103K for 30 hours.

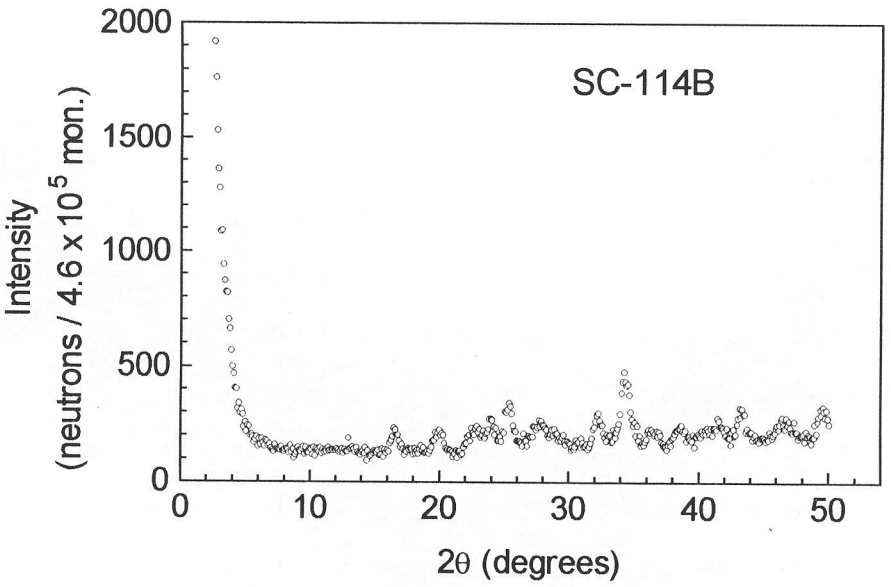

Figure 7. - Neutron powder pattern obtained with the same superconductor of Fig. 6. above after a quick second annealing at ca. 1113K for 30 minutes.

- Symmetry in multiple diffraction patterns.

Carlos B.R. Parente, Vera L. Mazzocchi, Sabrina Metairon, José M. Sasaki[1] and Lisandro P. Cardoso[1].

In 1972, Parente studied the multiple diffraction of neutrons in an aluminum single crystal [5]. He measured a neutron multiple diffraction (m.d.) pattern with the 111 primary reflection from the crystal. The experimental neutron m.d. pattern had an extension sufficient to show two of the symmetry mirrors normally existing in a m.d. pattern. Fig. 8. shows the pattern with indexing for the secondary reflections. It can be observed in Fig. 8. that, for one of the mirrors (at $\phi = 60°$ in the lower scale), in the unique pair of symmetrical peaks appearing in the pattern, peaks have different intensities. For the other mirror (at $\phi = 30°$) 3 pairs exhibit a perfect symmetry, i.e. peaks are in symmetrical positions around the mirror and have same intensity. The author explained the differences in intensity by showing that a few of the secondary reflections, as well as the coupling reflections, involved in the formation of the peaks had different reflectivities. Different reflectivities imply different intensities. Recently, Sasaki [6] observed the same phenomenon in a x-ray m.d. pattern obtained with $CuK\alpha_1$ radiation and primary reflection 222 from a galium arsenide (GaAs) single crystal. The crystal was epitaxially grown on top of an indium-galium arsenide (InGaAs) substrate. The pattern obtained with the crystal is shown in Fig. 9. Two symmetrical peaks having different intensities are indexed in the Figure.

This work is a systematic search for the phenomenon observed by authors above in x-ray and neutron m.d. patterns. Present authors intend to make a further investigation of the phenomenon by measuring and simulating m.d. patterns for different primary reflections from several different crystals. Simulated patterns are calculated by the computer programs MULTI [7] and MULTX [8], for neutrons and x-rays, respectively. Till the present, several simulated m.d. patterns have been calculated, for a full extension of the azimuthal angle ϕ ranging from 0 to 360°. Data were plotted in circular plots, where the intensity axis is radial and the ϕ axis is circular. A circular plot allows a better

[1] Instituto de Física Gleb Wataghin - Universidade Estadual de Campinas (UNICAMP) - Campinas, SP, Brazil.

visualization of the pattern symmetry. Using such a type of plot simulated patterns, we have identified two types of symmetry mirrors. One of them is a normal mirror we called 'isomorphic mirror'. It produces only pairs of fully symmetrical peaks like those in Fig. 8., around $\phi = 30°$ in the lower scale. Other is an abnormal mirror we called 'anamorphic mirror'. It produces pairs of symmetrical peaks having different intensities like those in Fig. 8., around $\phi = 60°$. It should be mentioned that such a type of mirror also produces pairs of fully symmetrical peaks. Another point to be noted is that, depending on some sample characteristics, partially symmetrical peaks can appear with so minute differences that they can be misinterpreted as being fully symmetrical peaks. Fig. 10. shows a pair of peaks, simulated by MULTI, corresponding to the experimental pair at $\phi = 60°$ in the lower scale of Fig. 8. For the simulations, a cylindrical Al single crystal was considered. By varying radius (r), height (h) of the cylinder and mosaic spread (η) of the crystal, peaks change their shapes and intensities in an astonishing way. It can be easily verified that η plays a much more important role in the shape modification than r or h does. Fig. 11. is the equivalent for Fig. 10, except that the simulated pair corresponds to the experimental pair at $\phi = 30°$ in Fig. 8. In this case, although a great variation in shape and intensity occurs, peaks are rigorously equal for a same pair.

Concerning the existence of isomorphic and anamorphic mirrors, we have simulated several different m.d. patterns. Examples of simulated patterns are shown in Figs. 12., 13. and 14. corresponding to the primary reflections 111, 220 and 042 from a cylindrical Al single crystal. In cubic symmetry, direction [111] has a 3-fold symmetry, [220] a 2-fold symmetry and [042] a 1-fold symmetry. As can be readily seen in Figs. 12., 13. and 14., each plot form a figure having the symmetry of the corresponding direction. Furthermore, circular plots for n odd have both anamorphic and isomorphic mirrors. Mirrors of one type intercalate into mirrors of other type, in a regular sequence. Circular plots for n even have only isomorphic mirrors. The number of isomorphic mirrors equals the order of the symmetry axis parallel to the scattering vector of primary reflection, i.e. $m_i = n$. For anamorphic mirrors $m_a = m_i = n$, if n is odd, and $m_i = 0$, if n is even. Of course, number total of mirrors m_t is given by $m_t = m_i + m_a$. In equalities above m_i is the number of isomorphic mirrors, m_a is the number of anamorphic mirrors and n is the order of

symmetry axis.. It should be noted that, in a normal plot, the number of mirrors is twice that in a circular plot. In a circular plot, a same mirror passing through a certain azimuthal position also passes through a position 180° from the first one. In a normal plot, these two positions are considered as being two individual mirrors.

We have also studied several experimental x-ray and neutron m.d. patterns in search for isomorphic and anamorphic mirrors. For instance, we have analysed the experimental 00.1 α- and β-quartz patterns measured by Mazzocchi [9]. We observed that they present no anamorphic mirrors. In this case, in a circular plot, m_a is equal to zero but m_i is still equal to n. It should be mentioned that α-quartz has a hexagonal structure. Consequently, the [00.1] direction has a 6-fold symmetry and $m_i = 6$. On the other hand, β-quartz has a trigonal structure and [00.1] direction has a 3-fold symmetry with $m_i = 3$. The patterns are shown in Fig. 15..

Although conclusions like those just above have been drawn from experimental patterns, existing patterns have limited extensions and are, in general, of poor quality. For this reason, we have decided to obtain full experimental patterns with good quality. It should be noted that obtainment of a full (360°) pattern is a very time-consuming procedure, particularly in a low flux reactor like ours. Nevertheless, we have already measured two full patterns. One of them is shown in Fig. 16.. It was obtained with the 111 primary reflection from a $BaLiF_3$ single crystal. Although the pattern is of a poor quality, positions of mirrors are easily found: they appear at every 30° from ca. 20° to 350°. On the other hand, it was not possible to determine unambiguously the beam type of the mirrors in the pattern. It should be noted that the number of mirrors, 12 in a normal pattern, is in accordance with the order of direction [111] in the cubic system of $BaLiF_3$. Other recently measured full pattern was obtained by measuring reflection 111 from a cylindrical copper single crystal. The scattering vector of this reflection was parallel to the cylinder axis. In this case, a quite good pattern was obtained. Fig. 17. shows such a pattern like in the pattern of Fig. 12., intercalated isomorphic and anamorphic mirrors can be observed. It should be noted that copper and aluminum are isostructural. The number of mirrors, 12 in the normal plot, is in accordance with reflection 111 from the copper cubic structure.

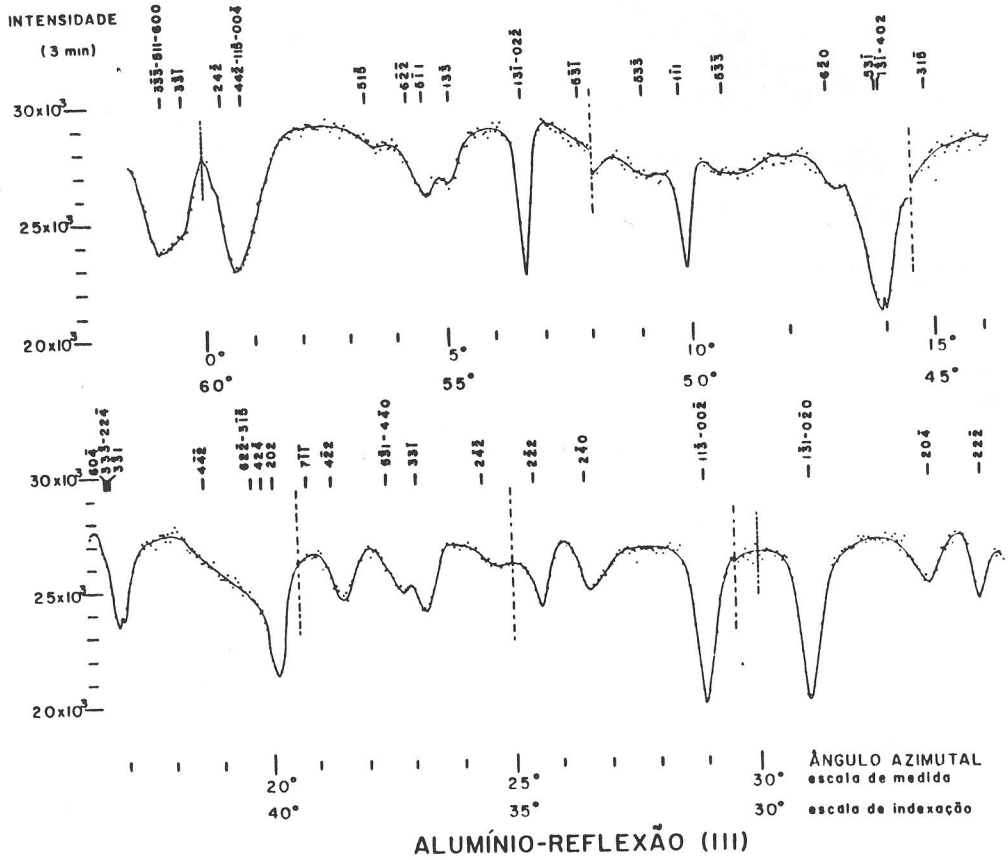

Figure 8 - Neutron m.d. pattern obtained with reflection 111 from an aluminum single crystal [5]. An anamorphic symmetry mirror is positioned at $\phi = 60°$ in lower scale; an isomorphic at 30°.

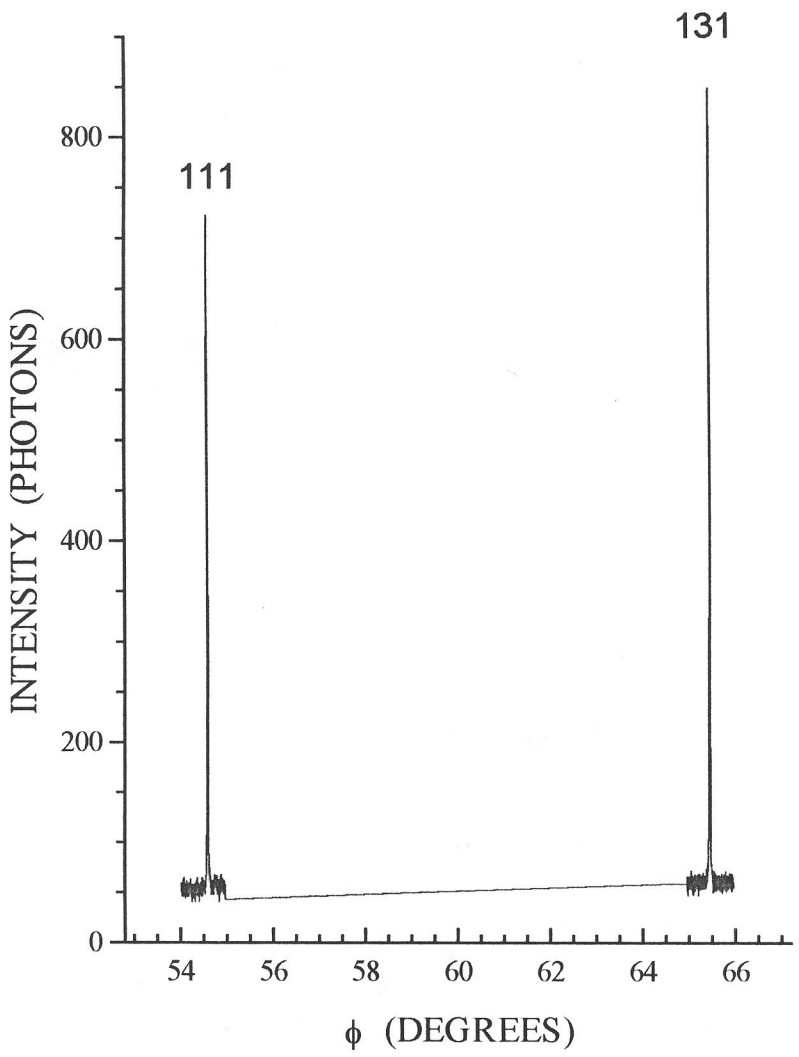

Figure 9 - X-ray m.d. pattern obtained with CuKα_1 radiation and reflection 222 from a GaAs / InGaAs single crystal [6]. Symmetrical secondary reflections have different intensities.

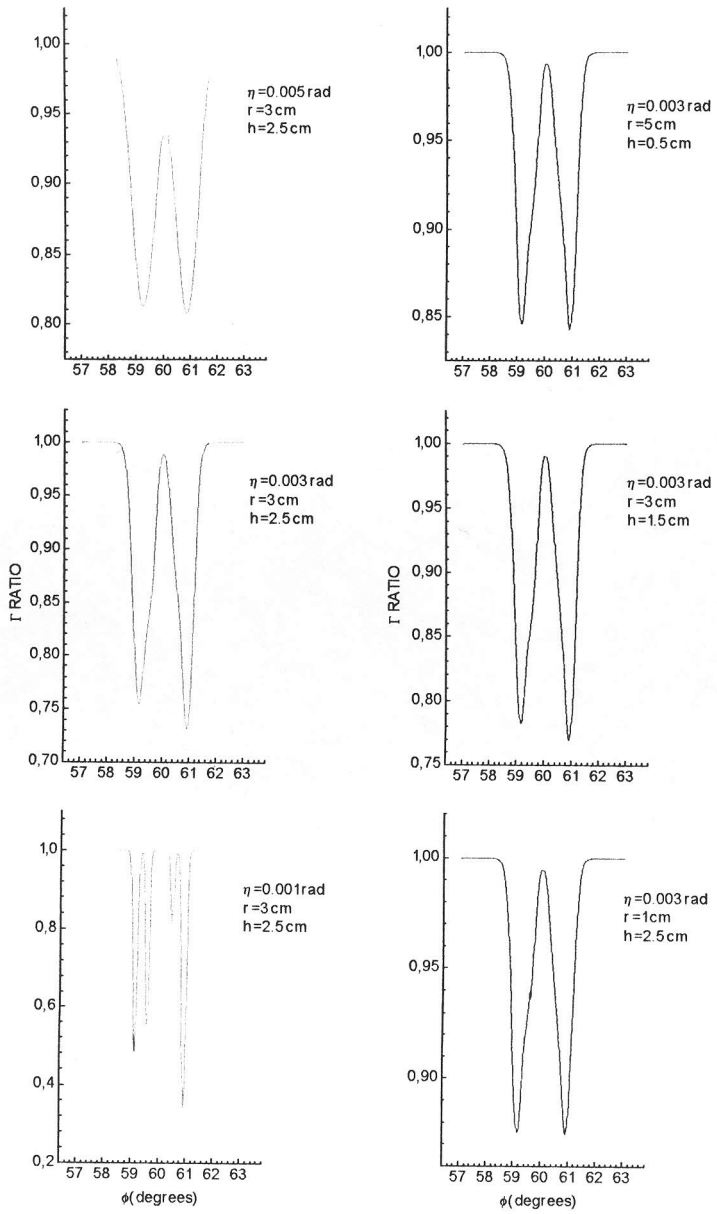

Figure 10 - Al (111) simulated peaks around an anamorphic mirror located at $\phi = 60°$. Different values for the radius (r) and height (h) of a cylindrical single crystal as well as for the mosaic spread (η) were assumed in the calculations.

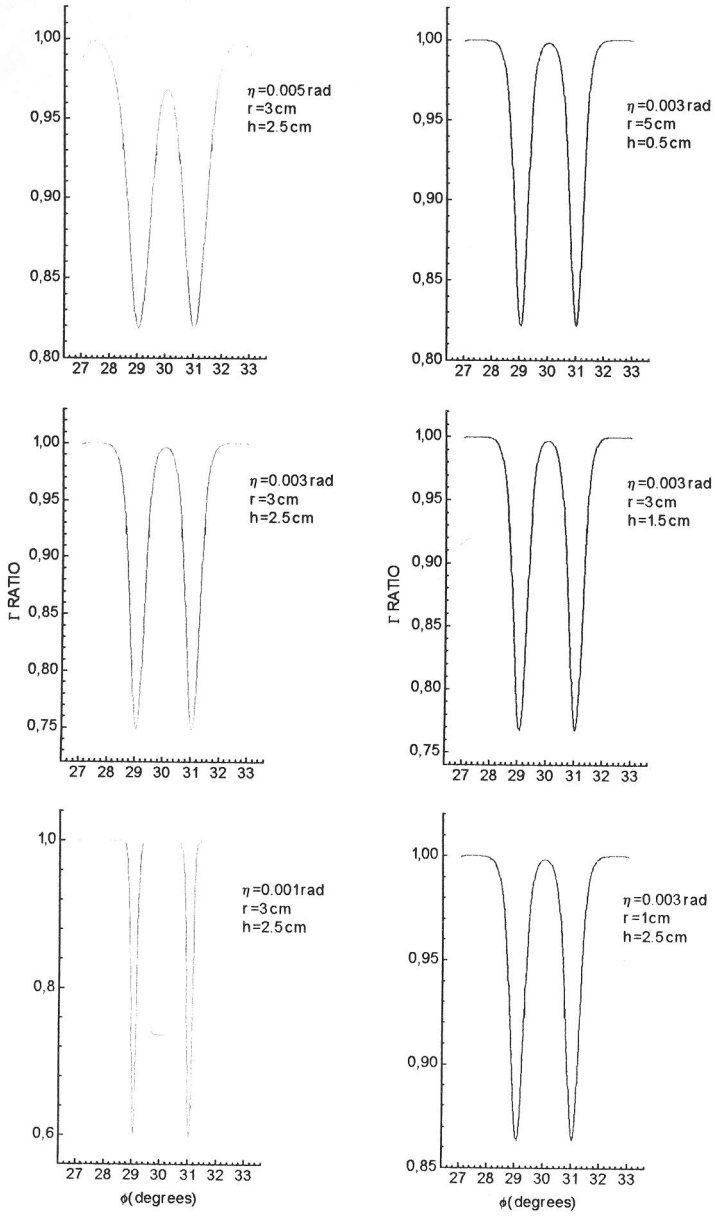

Figure 11 - Al (111) simulated peaks around an isomorphic mirror located at $\phi = 30°$. Different values for the radius (r) and height (h) of a cylindrical single crystal as well as for the mosaic spread (η) were assumed in the calculations.

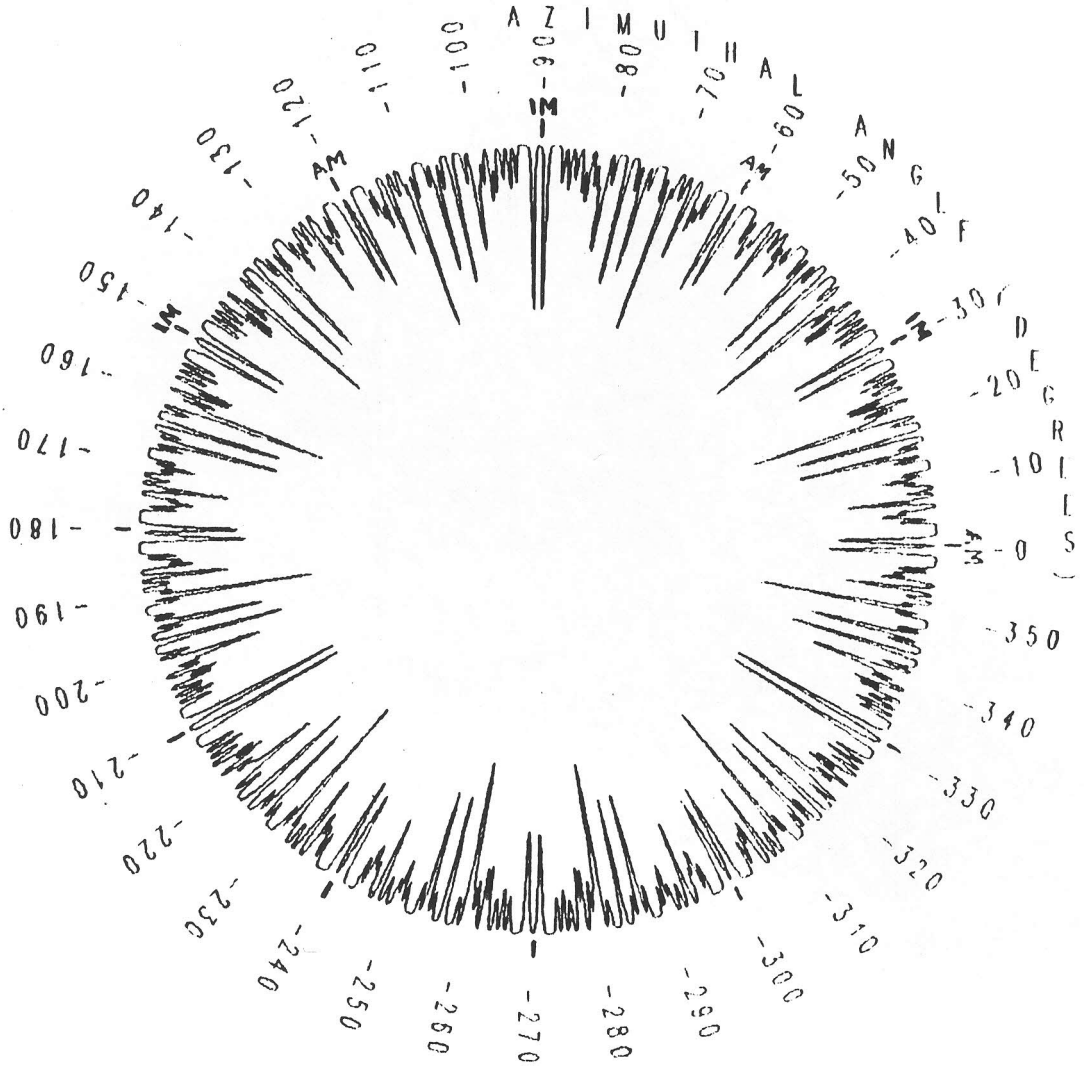

Figure 12 - Simulated Al (111) neutron m.d. pattern in a circular plot. The 3-fold symmetry of direction 111 is clearly visible in the plot. Three isomorphic mirrors (IM) intercalated into three anamorphic mirrors (AM) are shown in the plot.

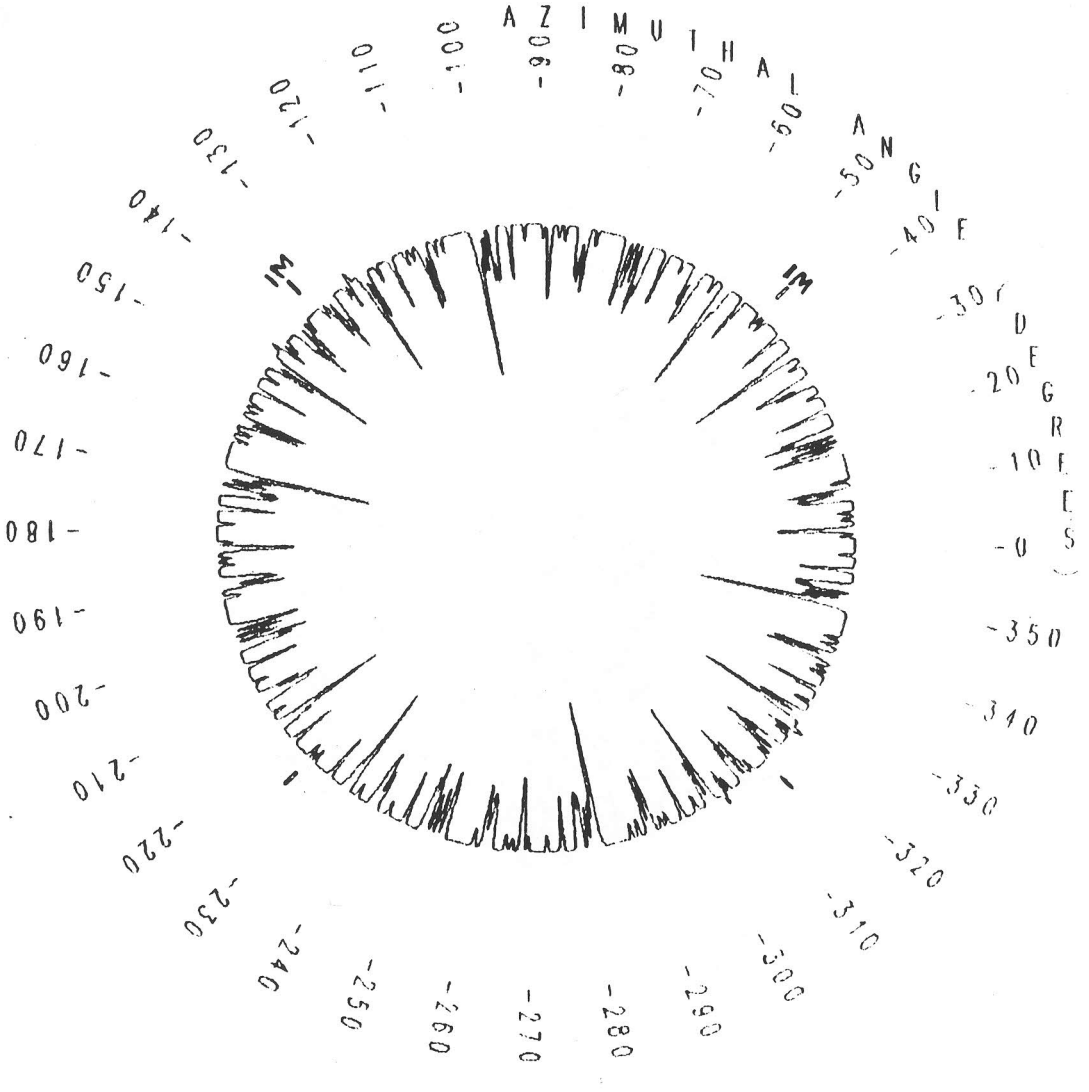

Figure 13 - Simulated Al (220) neutron m.d. pattern in a circular plot. Only two isomorphic mirrors (IM) are defined for the 2-fold symmetry of direction 110.

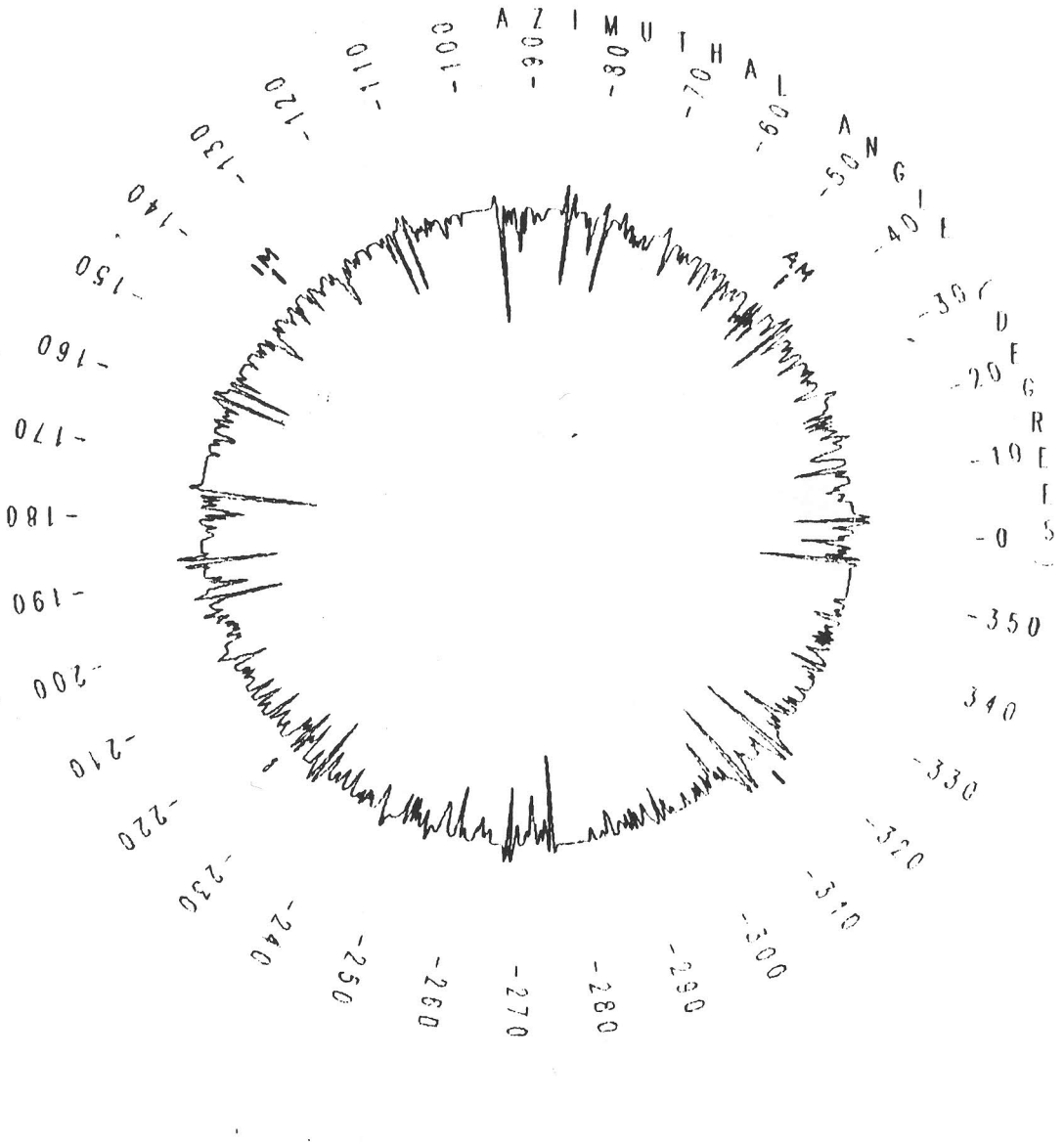

Figure 14 - Simulated Al (042) neutron m.d. pattern in a circular plot. According the 1-fold symmetry of direction 021 only one isomorphic mirror (IM) and one anamorphic mirror (AM) appear in the plot.

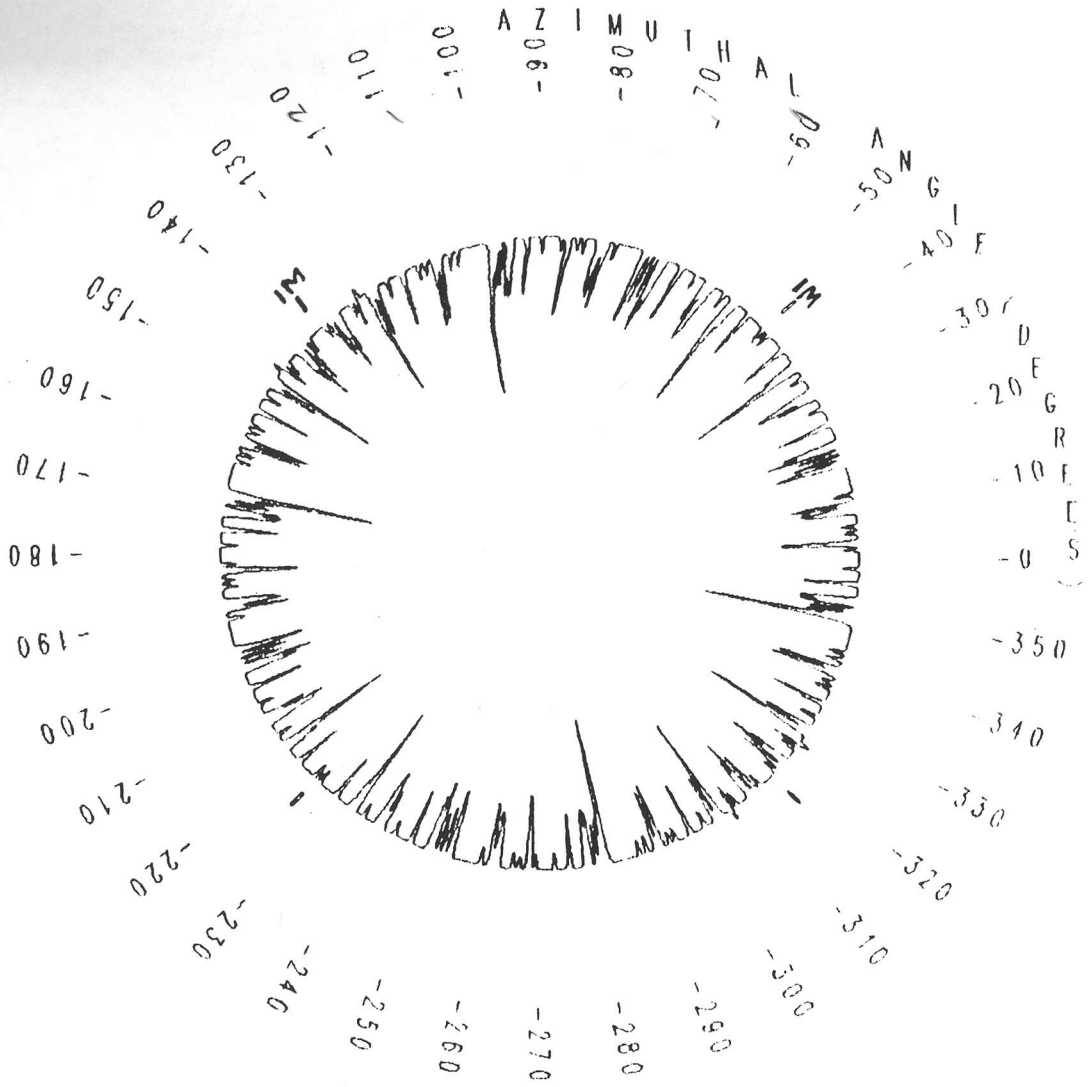

Figure 13 - Simulated Al (220) neutron m.d. pattern in a circular plot. Only two isomorphic mirrors (IM) are defined for the 2-fold symmetry of direction 110.

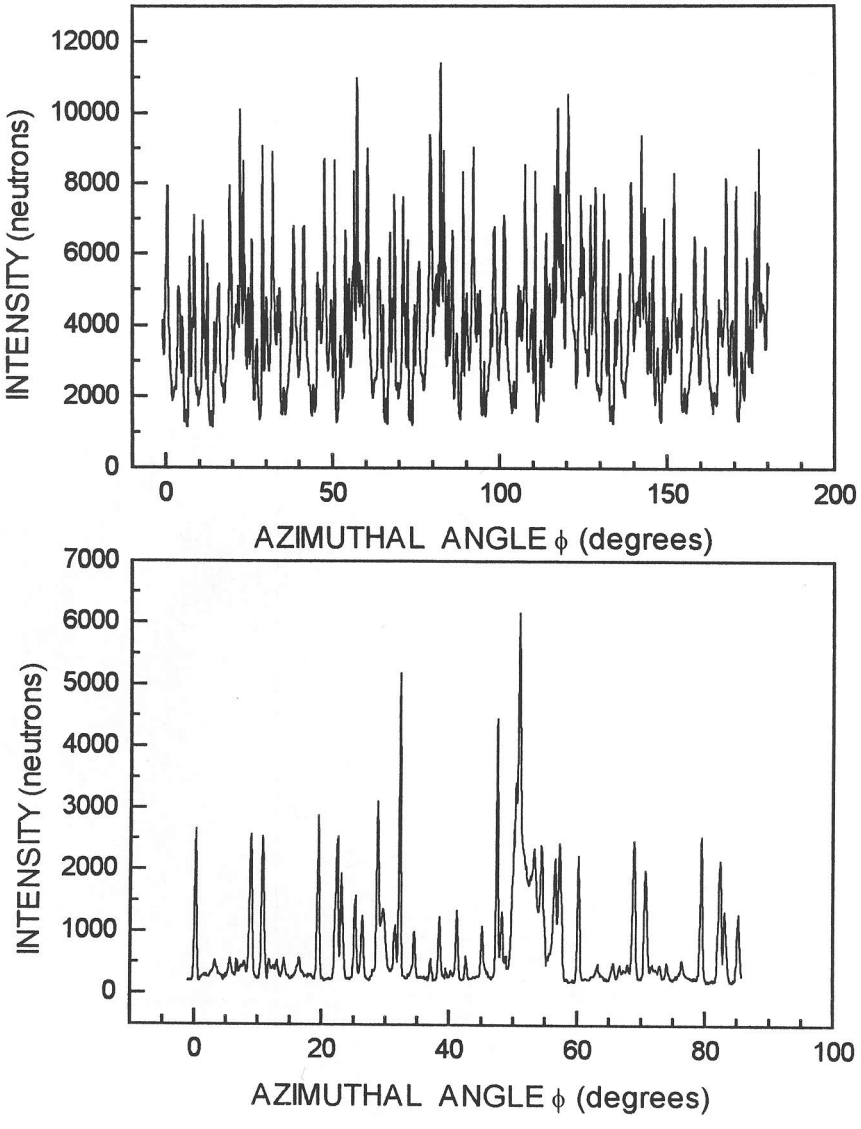

Figure 15 - Neutron m.d. patterns obtained with reflection 00.1 from a quartz (SiO_2) single crystal in phases α (above) and β (below) [9].

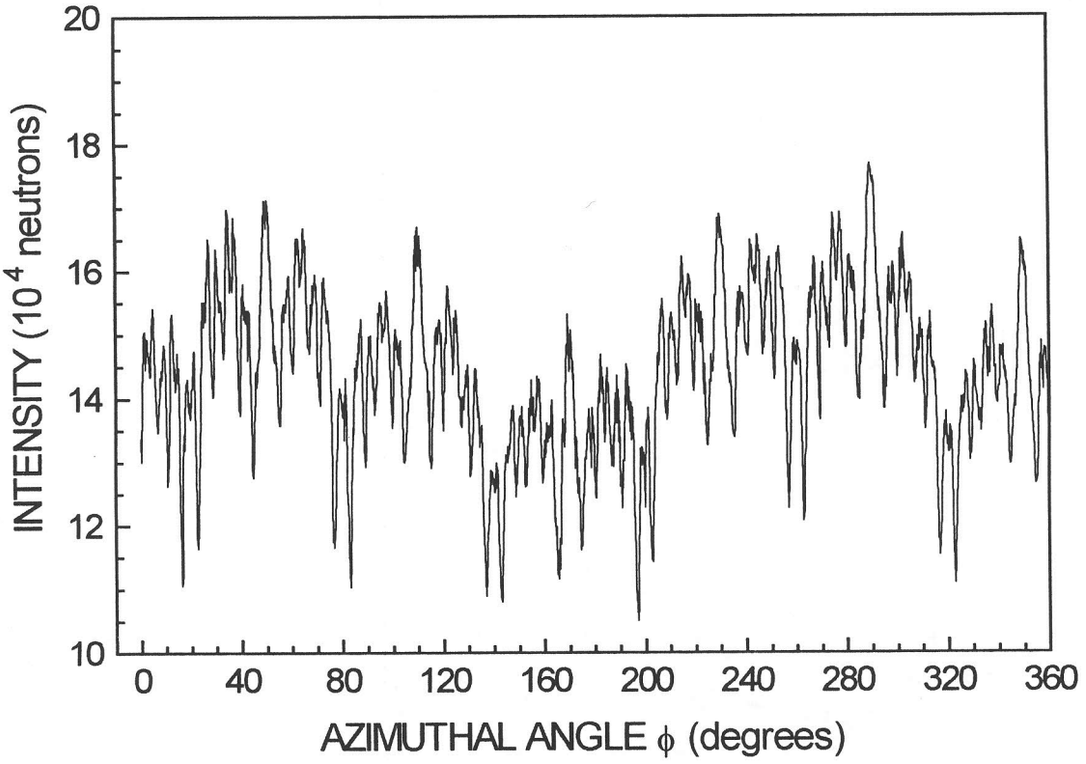

Figure 16 - Full neutron m.d. pattern measured with reflection 111 from a $BaLiF_3$ single crystal.

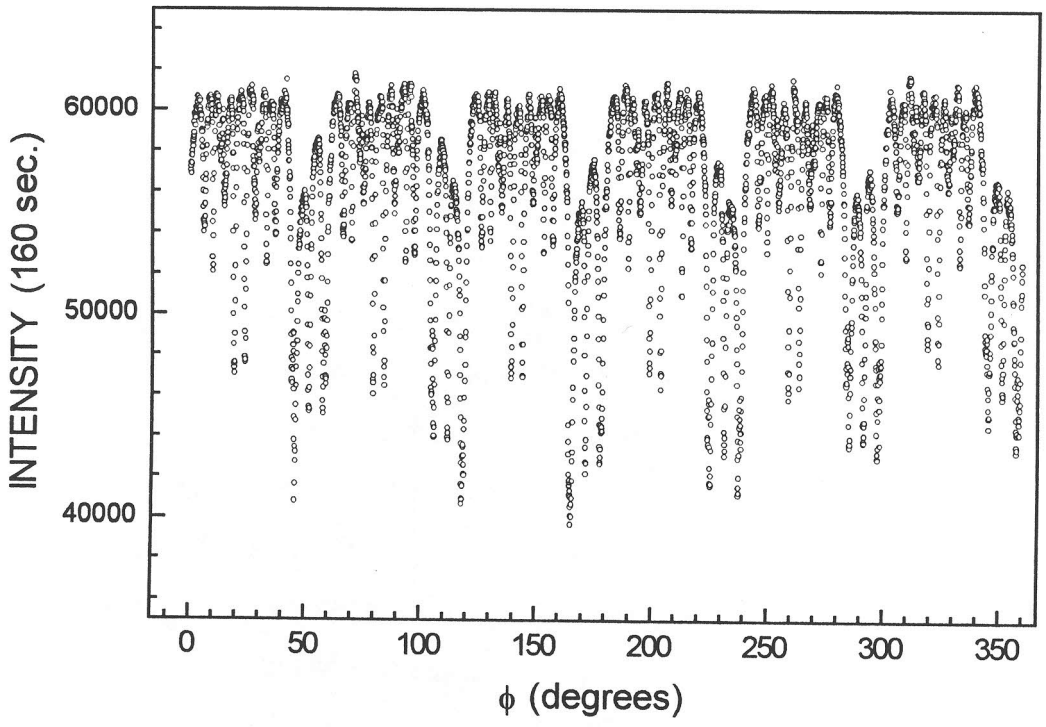

Figure 17 - Full neutron m.d. pattern measured with reflexion 111 from a Cu single crystal.

- Determination of the lattice parameter of aluminum at different temperatures by neutron multiple diffraction.

Carlos B.R. Parente and Vera L. Mazzocchi.

In a m.d. pattern the azimuthal position of a peak depends on the wavelength of the incident radiation as well as on the cell parameters of the crystal under investigation [10]. If the wavelength assumes a fixed value, azimuthal position becomes dependent only on the cell parameters.

In this work, the cell parameter a of an Al single crystal is determined for different temperatures. The azimuthal angular positions of several peaks in a neutron Al m.d. pattern, obtained at a certain temperature, are used to determine the a-value for that temperature. In a first method, the absolute position of a peak, i.e. its position referred to an arbitrary origin, is used. In this method, each peak produces an a-value. In a second method, the relative positions of two neighbor peaks are used. For a better result, two neighbor peaks with opposite behaviors must be chosen. In this method each pair of peaks produces an a-value. Values of a are found by comparing the absolute azimuthal position of a peak with azimuthal positions of the corresponding peak in simulated patterns, calculated by assuming different a-values. When the azimuthal position of corresponding peaks match for a particular simulated pattern, the a-value assumed in the simulation is assigned to the peak. In the second method, relative positions, i.e. azimuthal angular distances between two peaks, are used instead. For this method, angular distances are compared to find the a-value for a particular temperature. For both methods, the final a-value can be attained by linear regression of the several individual a-values plotted.

Fig. 18. shows partial neutron m.d. patterns obtained with an Al single crystal at 3 different temperatures. The azimuthal positions of the peaks are listed in the bottom of Fig. 18.. They resulted from Gaussian fittings of the patterns (full lines). Fig. 18. illustrates how the peaks change their positions with temperature. Peak 402, for example, goes to left as the temperature increases. Peak 042, on the other hand, goes to right. They form a pair with opposite behaviors and could be used in the second method. Nevertheless, each one separately could also be used in the first method.

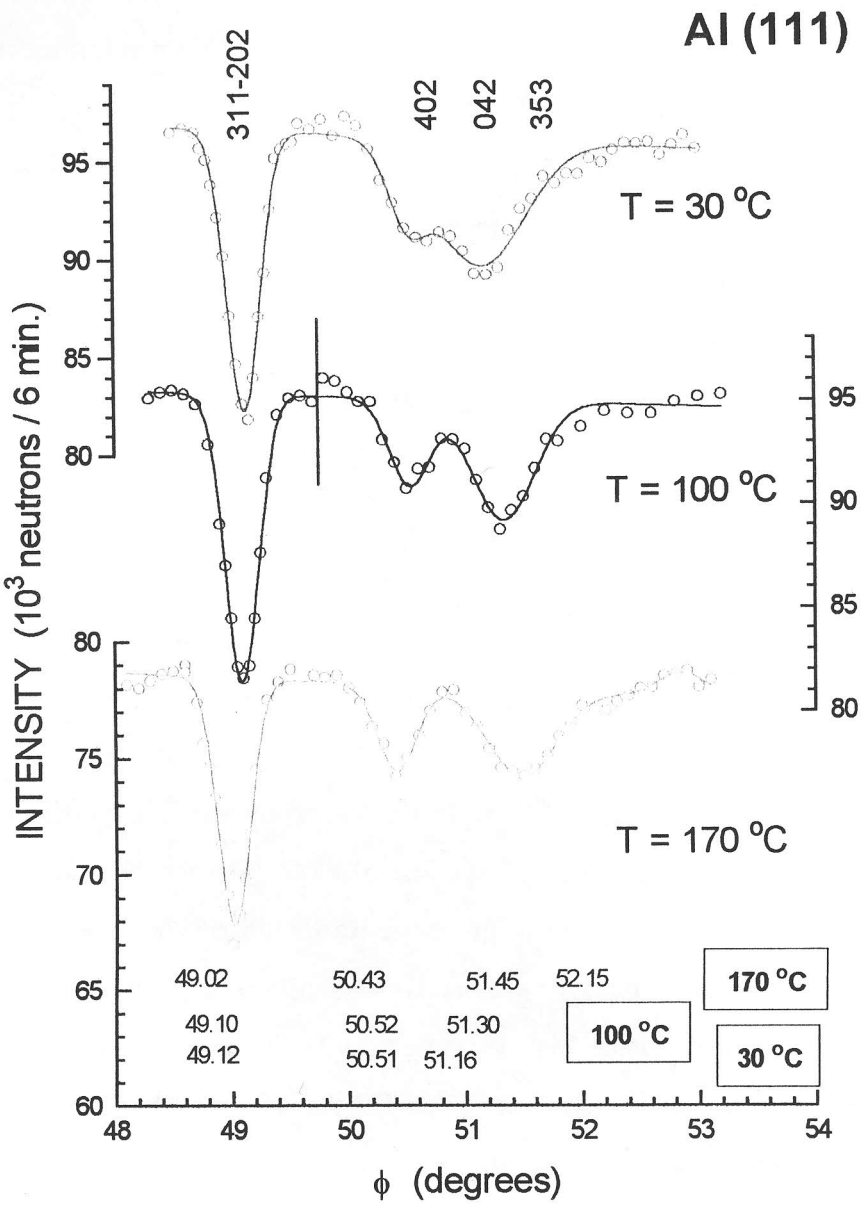

Figure 18 - Partial Al (111) neutron m.d. patterns measured at 30, 100 and 170 °C. They illustrate how peaks change positions with the temperature variation.

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