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X-ray Diffraction Profiles from Neutron-Irradiated LiF Single Crystals*

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The detailed measurement of the Bragg line profiles of several X-ray reflexions from six single-crystal samples of LiF irradiated under different and well-defined conditions in the core of a swimming-pool type nuclear reactor are reported. The crystals were irradiated with three different spectra: (*a*) total neutron spectrum plus the γ background radiation, (*b*) same as (*a*) without thermal neutrons, the crystal being wrapped in a cadmium foil, and (*c*) the background γ radiation alone. The experimental results presented display the changes in the profiles due to the different radiation spectra used and to the change in irradiation temperature within the interval from 113 to about 173 °C. That peak intensities decrease while integrated intensities increase with irradiation is a well-known result amply confirmed here. Integrated intensities in these experiments increase from 4 to 14 times with respect to the unirradiated crystal. The strongest reflexions (111, 200 and 220) experienced the highest increases. The influence of the temperature of irradiation has been verified through measurements of reflexions 111, 200 and 220 from crystals irradiated at temperatures of 113, 148 and 173 °C. As the irradiation temperature rises the peak intensities clearly tend to those of the unirradiated crystal while integrated intensities attain a maximum at about 150 °C. The results also indicate that at least under the conditions prevailing in the present experiments the highest amount of damage is due to thermal neutrons ($E \leq 0{\cdot}45$ eV).

1. Introduction

Neutron-irradiated LiF has been the subject of numerous X-ray diffraction studies (for instance, Binder & Sturm, 1954; Keating, 1955; Senio & Tucker, 1957; Smallman & Willis, 1957; Lambert & Guinier, 1957, 1958). Most of the papers referred to are concerned with powder samples of LiF; we were unable to find any previous publication on line-profile analysis of neutron-irradiated single crystals of LiF, where the conditions of irradiation were clearly established. The importance of accurate knowledge of the neutron spectrum and of the temperature of the sample in radiation damage analysis has, however, often been pointed out, by Billington & Crawford (1961), Kohler (1971), and others. It is then surprising that a systematic study of the influence on the radiation damage of such parameters as the neutron spectrum, temperature of the crystal during irradiation and of the accompanying γ radiation, present as a background in the reactor core, has not yet been published.

In this paper, the experimental Bragg line profiles of several reflexions of LiF single crystals neutron and γ irradiated under well-defined conditions are presented as recorded. The deconvolution process and the interpretation of the deconvoluted profiles are currently under elaboration.

2. Experimental

In the experimental setup use was made of a single-crystal diffractometer with an incident-beam plane monochromator (Ge 111), scintillation counter and pulse-height analyser; Cu $K\alpha$ radiation of a highly stabilized ($\pm 0{\cdot}005\%$ in intensity) X-ray generator was used throughout.

The monochromator and the crystal were mounted in an antiparallel arrangement with an interposed collimator of 6′ of angular divergence. When a monochromator was used, the intensity ratio $I(K\alpha_1)/I(K\alpha_2)$ in the monochromatized beam depended strongly on small adjustments of the setting of the monochromator–collimator system, a fact which invalidates the use of Rachinger's method of correction for the residual $K\alpha_2$ radiation. The corrections were avoided by using a pure $K\alpha_1$ line, obtained by careful adjustment of the system.† The background around the $K\alpha_1$ lines was minimized by means of the pulse-height analyser, a condition maintained for all the profiles analysed in order to minimize errors due to profile truncation.

The measured value of the horizontal divergence of the monochromatized beam was of 20″ of arc. This is satisfactory for the present purpose since the widths of the profiles of the irradiated crystals analysed are of the order of 20′. Since the shape of the incident beam is

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† The residual $K\alpha_2$ intensity was less than $0{\cdot}1\%$ of $K\alpha_1$

convoluted in the profile, no physical significance is to be attached to details of about 20″ or less.

The LiF single crystals were mounted with the cleavage face perpendicular to the rotation axis of the goniometer head. Accurate setting of the crystals was carried out by monitoring the intensity of the 200 Bragg reflexion while the crystal turned around the reciprocal-lattice vector 200, coincident with the φ axis in this experiment and perpendicular to the surface of the crystal plate. By this alignment procedure the nearly perfect crystal can be oriented within a few seconds of arc.

The line profile was measured by point-to-point manual operation of a Rigaku Denki SG8 diffractometer, so that advantage could be taken of the fact that in this mode the apparatus was able to measure angular variations of 0·001° with good reproducibility, whereas when the automatic step-scanner was used the minimum angular variation was of 0·01°.

Following Wilson (1967), an odd number of observations, numbered from $-r$ to $+r$, were made. The central observation, $r=0$, corresponded to the maximum counting rate in the profile. The angular separation between consecutive measurements varied from 0·01 to 0·001° for some reflexions, and r was sufficiently high so that the first or the last few measurements were practically indistinguishable from the background, which was measured on both sides at intervals of 0·10°; in this way no serious error due to profile truncation is to be feared (Ladell, Parrish & Taylor, 1959; Young, Gerdes & Wilson, 1967). Fixed-time rather than fixed-count measurements were used systematically, following Wilson (1967), but in any case a sufficient number of counts was accumulated at each point, so that the statistical error was less than about 1%.

The size of the receiving slit was checked for every reflexion so that it would not affect the counting rate at that particular position.

For a given *hkl* reflexion, the measurements for irradiated and unirradiated crystals were performed under the same instrumental configuration to make sure that the instrumental function was the same for different crystals; absorption and other corrections are then practically identical. The small changes in θ due to changes of the crystal parameters during irradiation were negligible from this point of view since for all samples the dispersion in the values of a was about 0·005 Å.

Care was taken not to interchange reflexions equivalent for symmetry reasons; since the neutron flux in the reactor core is not isotropic it is likely to affect, say, reflexions *hk*0 and *h*0*k* in different ways. This view was substantiated in a parallel project on the diffuse scattering from the same samples, to be published elsewhere.

3. Irradiated LiF single crystals

Single crystals of LiF grown by Harshaw Chemical Co. cleaved most easily after a preliminary irradiation with X-rays in agreement with Ives & Ramachandran (1967). Crystal plates of about 12 × 8 × 1 mm were then annealed at 520 °C for 30 min in order to eliminate the colour centres introduced by the X-irradiation process (Mayer, Perio, Gigon & Tournarie, 1955). The samples were cut from the crystal block; those not optically perfect were discarded at the outset and the others checked for crystal perfection. Only samples whose 200 linewidth, measured with the previously described experimental setup, was about 20″ were used in the following. The selected samples were irradiated in the same position in the core of the swimming-pool type reactor of the Instituto de Energia Atômica, São Paulo, under well-defined conditions of neutron flux and spectrum, temperature and orientation with respect to the core. One of the unirradiated samples was kept protected from radiation at room temperature and served as a standard of reference.

The neutron spectrum for the particular position of irradiation used in the experiment, as well as the neutron and γ fluxes, were determined by Pimentel and co-workers (Pimentel, 1973; Pimentel, Amaral, Moura & Frugoli, 1975). The temperature of the sample during irradiation was kept constant within 5 °C. Table 1 summarizes the conditions of irradiation: fluence of thermal, intermediate and fast neutrons, background γ radiation, exposure time and temperature for the different samples.

Table 1. *Conditions of irradiation*

In this table thermal neutrons have energies $E_n \leq 0{\cdot}45$ eV, intermediate neutrons, $0{\cdot}45$ eV $< E_n < 1$ MeV and fast neutrons, $E_n > 1$ MeV. Errors for fast and intermediate neutron fluxes are about 5% and for thermal neutrons about 3%.

		Neutrons								γ-radiation		
		Flux (n cm^{-2} s^{-1})				Fluence (n cm^{-2})						
Sample	Total time of irradiation (h)	Thermal ($\times 10^{13}$)	Intermediate ($\times 10^{13}$)	Fast ($\times 10^{12}$)	Total ($\times 10^{13}$)	Thermal ($\times 10^{17}$)	Intermediate ($\times 10^{17}$)	Fast ($\times 10^{17}$)	Total ($\times 10^{18}$)	Flux (Photons cm^{-2} s^{-1}) ($\times 10^{13}$)	Fluence (Photons cm^{-2}) ($\times 10^{18}$)	Temperature of the sample (°C)
1		Unirradiated										
2	8·22	1·58	1·9	6·3	4·3	4·68	5·6	1·9	1·2	3	~1	113±3
3	8·55	1·42	1·9	6·0	4·0	4·37	5·8	2·0	1·0	3	~1	148±4
4	8·35	1·57	1·9	7·2	4·2	4·71	5·7	2·2	1·2	3	~1	173±6
5	8·50	—	1·9	6·5	2·5	—	5·8	2·0	0·78	3	~1	121±3
6	13·95	—	–	–	–	—	–	–	–	0·2	~0·1	ambient

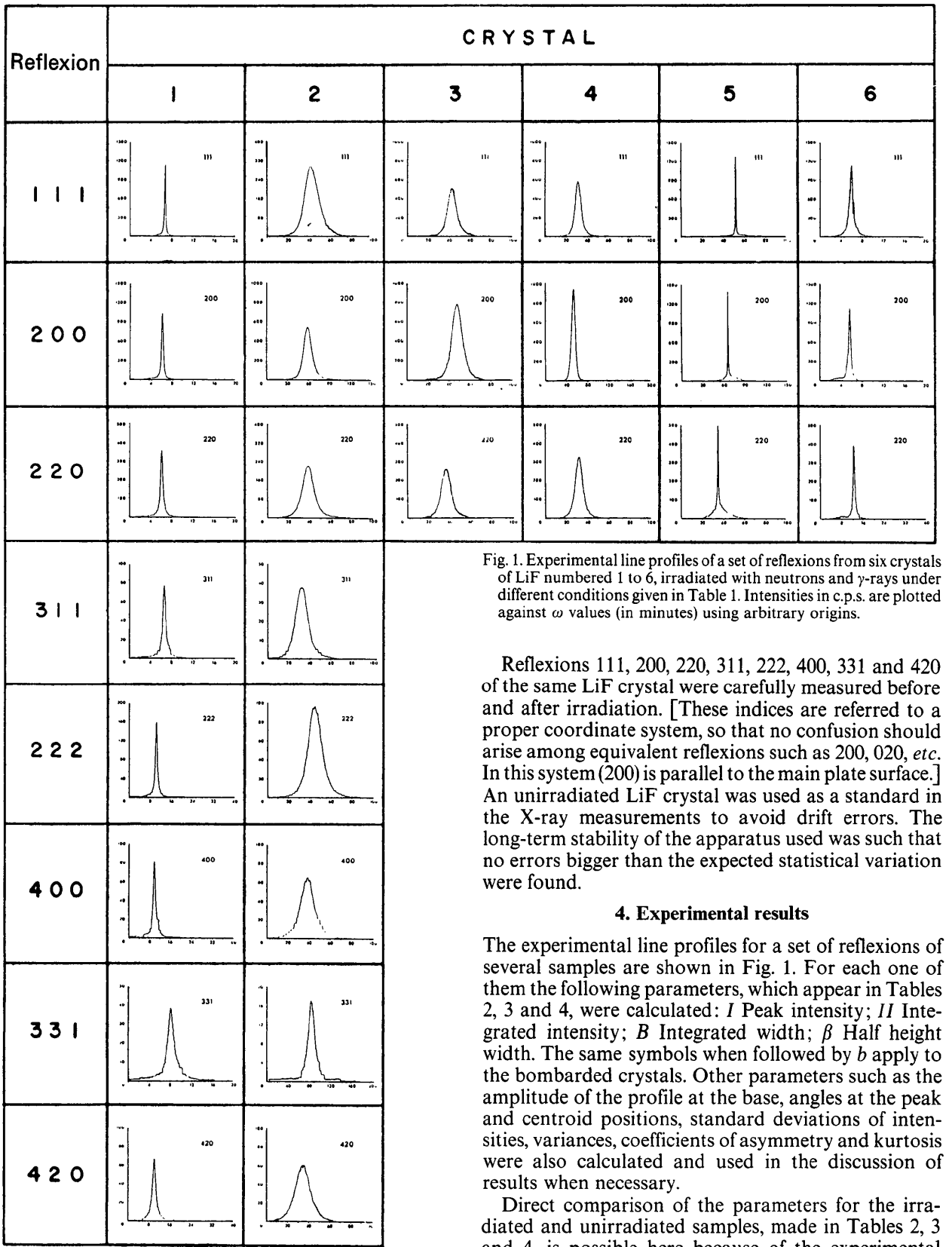

Fig. 1. Experimental line profiles of a set of reflexions from six crystals of LiF numbered 1 to 6, irradiated with neutrons and γ-rays under different conditions given in Table 1. Intensities in c.p.s. are plotted against ω values (in minutes) using arbitrary origins.

Reflexions 111, 200, 220, 311, 222, 400, 331 and 420 of the same LiF crystal were carefully measured before and after irradiation. [These indices are referred to a proper coordinate system, so that no confusion should arise among equivalent reflexions such as 200, 020, *etc.* In this system (200) is parallel to the main plate surface.] An unirradiated LiF crystal was used as a standard in the X-ray measurements to avoid drift errors. The long-term stability of the apparatus used was such that no errors bigger than the expected statistical variation were found.

4. Experimental results

The experimental line profiles for a set of reflexions of several samples are shown in Fig. 1. For each one of them the following parameters, which appear in Tables 2, 3 and 4, were calculated: *I* Peak intensity; *II* Integrated intensity; *B* Integrated width; β Half height width. The same symbols when followed by *b* apply to the bombarded crystals. Other parameters such as the amplitude of the profile at the base, angles at the peak and centroid positions, standard deviations of intensities, variances, coefficients of asymmetry and kurtosis were also calculated and used in the discussion of results when necessary.

Direct comparison of the parameters for the irradiated and unirradiated samples, made in Tables 2, 3 and 4, is possible here because of the experimental precautions taken to ensure that the instrumental

Table 2. *Peak intensities Ib and their ratios with respect to those of the unirradiated crystal I1*

Crystals 2, 3 and 4 are related to crystal 1′ and crystals 5 and 6 to crystal 1″.

Re-	*I*1		*Ib* (c.p.s.)					*Ib*/*I*1				
flexion	1′	1″	2	3	4	5	6	2	3	4	5	6
111	1151±8	1300±8	299±4	512±5	587±5	1282±8	1147±8	0·26	0·44	0·51	0·98	0·88
200	1057±7	1301±8	544±5	792±6	958±7	1371±8	1197±8	0·51	0·75	0·91	1·05	0·92
220	362±4	398±4	222±3	259±4	327±4	504±5	393±4	0·61	0·71	0·90	1·26	0·98
311	78±2		38±1					0·49				
222	160±2		97±2					0·61				
400	82±2		66±2					0·80				
331	39±1		17±1					0·43				
420	67±1		61±2					0·91				

Table 3. *Integrated intensities IIb and their ratios with respect to those of the unirradiated crystal II*

Crystals 2, 3 and 4 are related to crystal 1′ and crystals 5 and 6 to crystal 1″.

	II (c.p.s. deg.)							*IIb*/*II*				
Reflexion	1′	1″	2	3	4	5	6	2	3	4	5	6
111	8±1	9±1	88±4	107±3	80±2	34±4	20±4	11·0	13·4	10·0	3·7	2·2
200	11±1	13±2	149±3	186±6	155±4	63±4	15±4	13·5	16·9	14·1	4·8	1·1
220	5·3±0·6	6·0±0·7	56±2	57±2	54±1	30±4	8±1	10·5	10·5	10·2	5·0	1·3
311	1·5±0·2		10·3±0·4					6·8				
222	3·9±0·4		29±1					7·4				
400	2·5±0·4		20·2±0·8					8·1				
331	1·5±0·1		6·3±0·3					4·2				
420	2·7±0·2		19·6±0·7					7·2				

function was small and practically the same for a given reflexion measured from different samples.

The ratios of the values obtained for some parameters of the irradiated and unirradiated samples are plotted in Fig. 2 as a function of $N=h^2+k^2+l^2$ and as a function of temperature in Fig. 3.

5. Discussion of results

The experimental results presented in Tables 2, 3 and 4 show the changes on the profiles due to (*a*) temperature of the sample during irradiation, (*b*) neutron spectra and (*c*) background γ irradiation.

Crystal 2, which was irradiated at the lowest temperature exhibits the most pronounced changes in the profile parameters. When this sample is compared with sample 1 (unirradiated), as shown in Table 2, it is observed that the peak intensities decrease considerably in the process of irradiation, coming down to about 25% of their initial values for reflexion 111, while the other reflexions come down to values between 50 and 90%. These ratios have practically no correlation with $N=h^2+k^2+l^2$.

Integrated intensities were increased by the irradiation process by factors ranging from 4 to 14 for these two particular samples. The strongest reflexions (111, 200 and 220) experienced the highest increase in these ratios (Table 3). Although Fig. 2(*b*) shows a tendency for the ratio *IIb*/*II* for different reflexions to decrease with N, no functional relation between these two magnitudes was found. Also, there is a definite tendency for this ratio to increase with the integrated intensity *IIb* or with *II* (Table 3).

The integral width B of sample 1 increases with N as expected, while the corresponding value Bb for crystal 2 follows a seemingly chaotic pattern. However, the ratio Bb/B when plotted against N follows a regularly decreasing curve, which will be further analysed elsewhere.

The ratio of the standard deviation of the profiles of crystals 2 and 1 follows a pattern similar to that of the ratio of the integral widths, with the exception of 331. The integrated intensity of this reflexion was increased by a factor of only 4·2, the smallest for any of the measured reflexions and also one of the smallest increases in integrated width, while the standard deviation increased by a factor of 11, one of the biggest for any of the measured reflexions. Reflexion 331 is unusually broad at the base and it is also quite broad for the unirradiated crystal. Since this result seemed rather curious the measurements were repeated for both cases; no changes bigger than the statistical fluctuations were found.

5.1 *Influence of temperature during irradiation*

Samples 2, 3 and 4 were irradiated with neutrons under the same geometrical conditions, with practically identical fluences and total spectra (Table 1) but at

Table 4. *Measured line widths Bb and βb and their ratios with respect to those of the unirradiated crystal B and β*

Crystals 2, 3 and 4 are related to crystal 1′ and crystals 5 and 6 to crystal 1″.

Reflexion	Integrated widths B (′)						Bb/B					Half height widths β (′)					$\beta b/\beta$	
	1′	1″	2	3	4	5	6	2	3	4	5	6	1′	5	6	5	6
111	0·42±0·05	0·42±0·05	17·7±0·8	12·5±0·3	8·2±0·2	1·6±0·2	1·0±0·2	42·1	29·8	19·5	3·8	2·4	0·42±0·03	0·82±0·03	0·42±0·03	1·95	1·00
200	0·62±0·05	0·60±0·05	16·4±0·3	14·1±0·4	9·7±0·2	2·7±0·2	0·8±0·2	26·5	22·7	15·6	4·5	1·3	0·60±0·03	0·64±0·03	0·60±0·03	1·07	1·00
220	0·88±0·09	0·90±0·09	15·1±0·5	13·2±0·2	10·0±0·2	3·6±0·5	1·22±0·15	17·1	15·0	11·4	4·0	1·4	0·75±0·03	1·01±0·03	0·80±0·03	1·31	1·06
311	1·15±0·15		16·3±0·7					14·2									
222	1·46±0·15		17·9±0·9					12·3									
400	1·9±0·3		18·4±0·8					9·7									
331	2·3±0·2		22·2±1·5					9·6									
420	2·4±0·2		19·3±0·8					8·0									

different temperatures, 113, 148 and 173 °C respectively. Fig. 3(*a*), (*b*) and (*c*) shows the ratios of peak intensities, integrated intensities and integral widths of these crystals, with respect to sample 1, as a function of temperature. Fig. 3(*a*) shows that as the temperature of irradiation increases, the peak intensity tends to that of the unirradiated crystal or, in other words, the defects tend to disappear as they are formed with increasing facility at the higher temperatures. Fig. 3(*b*) displays a rather unexpected feature in that the ratio of integrated intensities has a maximum around 150 °C. At higher temperatures the integrated intensities probably tend to those of the unirradiated crystal. In Fig. 3(*c*) the integral width is seen to decrease regularly with the temperature, the high value, 42, attained by this ratio for the 111 reflexion at 113 °C being worthy of note. On the other hand, the standard deviations* follow a pattern similar to that of the integral widths; so there is no qualitative change in the shape of the profile.

Consideration of Fig. 3 indicates that the temperature dependence of the radiation damage in this case is quite complicated. In fact, one expects, as indicated above, the parameters of the profiles to tend to those of the unirradiated crystal as the temperature increases. While this is probably so, the different parameters evolve in different ways. The behaviour of the integrated intensities is very interesting; the ratio IIb/II displays a maximum at about 150 °C, which is, by chance, close to the middle of the temperature range of our measurements. $IIb/II \simeq 10$ at 173 °C for reflexions 111 and 220 and $\simeq 14$ for 200; at the same temperature the peak intensities of reflexions 220 and 200 are 90% while 111 has attained only 50% of the corresponding peak intensity of the unirradiated crystal. In contrast with the integrated intensities the integral-width ratios Bb/B decrease monotonically with the temperature.

5.2 *Influence of the neutron spectrum*

In this section we compare the effects produced by irradiation with the whole neutron spectrum (samples 2, 3 and 4) with those where thermal neutrons ($E < 0{\cdot}45$ eV) were filtered out by means of a cadmium foil (sample 5).

The results obtained show important changes in peak intensity, integrated intensity and integral width which are mostly due to the thermal neutrons. In fact, sample 5 experienced comparatively small changes in these magnitudes. For instance, reflexion 111 of a crystal irradiated with the total spectrum at the same temperature, 121 °C, as sample 5, would be expected, according to Fig. 3(*c*), to show an increase in the integral width of a factor of 38, while the actual increase was only 3 times, or 8% of that due to the total spec-

* It is probably preferable to use the term 'second moment', but since we are actually referring to the square root of the second moment, we have preferred to use the simpler expression 'standard deviation' in this paper.

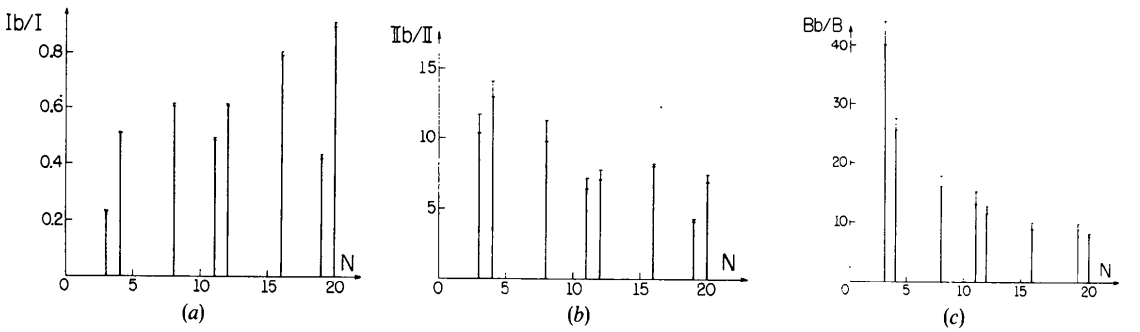

Fig. 2. (*a*) Ratio of peak intensities of the irradiated crystal 2 to those of the unirradiated crystal 1 as a function of $N = h^2 + k^2 + l^2$. (*b*) Ratio of integral intensities for crystal 2 to those of crystal 1 as a function of N. (*c*) Ratio of integral widths for crystal 2 to those of crystal 1 as a function of N.

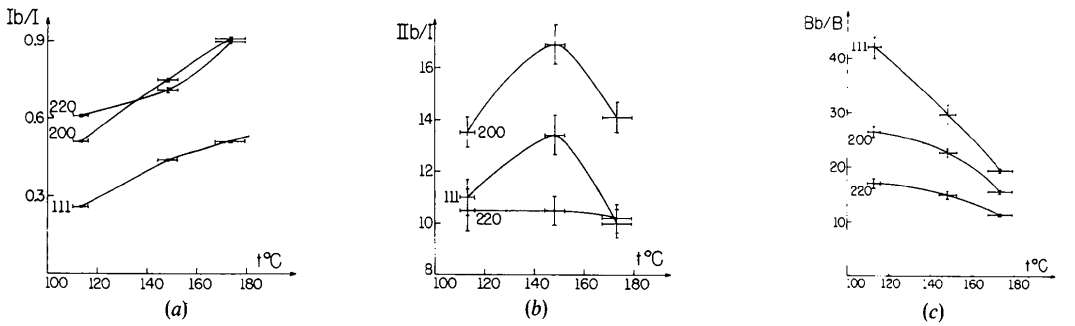

Fig. 3. (*a*) Ratio of peak intensities of reflexions 111, 200 and 220 of irradiated crystals 2, 3 and 4 to those of the unirradiated crystal 1 as a function of the temperature during irradiation. (*b*) Ratio of integrated intensities of crystals 2, 3 and 4 to those of crystal 1 as a function of temperature. (*c*) Ratio of integral widths for the same crystals.

trum. Theoretical calculations made by Pimentel *et al.* (1975), confirm that only about 6 % of the total damage is due to neutrons with $E > 0{\cdot}45$ eV, by assuming that the (n, α) reaction is the predominant one in the neutron irradiation of LiF.

Profiles 200, 111 and 220 from sample 5 ($E > 0{\cdot}45$ eV) were entirely different from those of either the unirradiated crystal or crystals 2, 3 and 4, irradiated with the total spectrum, the main difference being that the profiles of sample 5 had long tails, the amplitude at the base for reflexion 200 being about 200 times the half-height width while for the same reflexion in sample 2 this ratio was only about 7. This indicates that sample 5 probably contains predominantly point defects and clusters; however, no attempt was made at this stage to separate them.

Since sample 2 received practically the same fluence of intermediate and fast neutrons as sample 5 plus an additional dose of thermal neutrons, one would expect that the number of point defects would be much higher and consequently the profile would have even longer tails. However, the experimental profiles indicate that exactly the opposite occurs. This apparent contradiction can probably be interpreted by observing that the integral width of sample 2 is much higher, indicating a far more distorted lattice which certainly contains extended defects of different types.* As is known, point defects migrate, and may become trapped by extended defects. This mechanism and also a coalescence process may explain this apparent decrease in the concentration of point defects.

5.3 *Influence of* γ *radiation*

Sample 6 was γ-irradiated in the reactor core, during a shut-down period and at room temperature, with a fluence of 10^{17} photons/cm^2 or about ten times less than the estimated γ fluence for the other samples. The Bragg line profiles for this sample (Fig. 1) display a large base in comparison with the half-height width or with the integrated width. They are qualitatively similar to those of crystal 5, which have even larger bases. The general shape then indicates the existence of point defects and a small number of extended defects, as implied by the small increase in the peak width. This is in agreement with previous results given by Ives & Ramachandran (1967), Spalt & Peisl (1971) and Wohofsky & Waidelich (1971) among others.

* Evidence for this has recently been found in X-ray topographs of the same specimens (Suzuki & Caticha-Ellis, to be published), which confirm that sample 2 contains a higher density of extended defects than sample 5.

Conclusions

Bragg profiles of irradiated LiF single crystals obtained with careful experimental techniques have been presented.

As is known, radiation damage under the same conditions of neutron and γ fluence is higher the lower the temperature of the crystal during irradiation. This was indeed the case for samples 2, 3 and 4 irradiated with total neutron and γ spectra at temperatures of 113, 148 and 173 °C, respectively. This is a conclusion that would probably be confirmed for a more extended interval of temperature. This is attributed to a process of diffusion of point defects, which would be favoured at higher temperatures. However, the results displayed in Fig. 3 do indicate that the phenomenon is quite complicated and particularly so because of the appearance of a maximum in the ratio of integrated intensities at about 150° C, which was in the middle of the temperature range of our measurements. Other profile parameters, such as the peak intensities and the integral widths, follow monotonic patterns with a tendency towards the corresponding values of the unirradiated crystal as the temperature of irradiation increases.

As is seen from the comparison between sample 2, irradiated with the total spectrum and sample 5, where thermal neutrons were filtered out by means of a cadmium foil, the highest amount of damage produced in LiF in the reactor core, at least under the conditions prevailing in our experiment, is due to thermal neutrons. This is in agreement with calculations of Pimentel *et al.* (1975), according to which thermal neutrons would produce 93% of the total damage.

The influence of the background γ radiation in the production of defects is by no means negligible, as is seen from the study of sample 6, which, however, received a γ fluence about ten times less than the other samples.

As is known, it is theoretically and experimentally possible to detect the existence of a planar distribution of defects from Bragg profile analysis. The experimental conditions used in these experiments provided enough resolution for that purpose. None of the crystals examined exhibited a profile compatible with a planar defect parallel to the {001} planes as found by Lambert & Guinier (1957, 1958) for LiF crystals irradiated under presumably different conditions of irradiation.

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