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MODIFICATION OF THE X-RAY DIFFRACTION EFFICIENCY OF LITHIUM FLUORIDE CRYSTALS BY SURFACE TREATMENT

Barton O. Sellick

July 22, 1976

MASTER

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Abstract

Convex-curved crystals of lithium fluoride demonstrate good dispersion and efficiency when used in reflection for x-ray spectral analysis. The crystals are stable and reasonably unaffected by harsh environments. In addition, they are mechanically strong, easily cleavable or machinable, and plastically deformable with heat.

In the present study, flat

crystal wafers were left either clear as cleaved or were subjected to surface treatment by sandblasting or lapping. Some wafers were then bent in a press mold to obtain convex-curved crystals of differing radii. The diffraction efficiency data presented show how surface treatment affects the efficiency of these various crystals when used as x-ray diffracting agents.

Introduction

Since crystals have come into use for x-ray analysis, many people have contributed to increasing the efficiency and resolution of such crystals. Our increased use of lithium fluoride crystals over the past eight years has paralleled the continuous upgrading of our crystal technology. Our techniques for cleaving, surface treatment, bending,

etching and evaluation of the finished crystal are detailed in a recent report.¹

This paper compares the diffraction efficiencies of crystals either clear or surface-treated and either flat or curved. The relative diffraction efficiencies of convex-curved crystals of differing radii are also shown.

Experimental Methods

Wafers cleaved from a lithium fluoride boule, $38 \times 38 \times 0.7$ mm in thickness, were evaluated on a spectrogoniometer for "d" spacing and diffraction efficiency of the copper K α energy. The results of surface treatment were assessed using a 10-channel, x-ray fluorescence spectrometer using wavelength dispersive analysis (6 to 60 keV, i.e., 1.9 to 0.22 Å). All the techniques and procedures employed were detailed and illustrated in a previous report.¹

Diffraction efficiency (ϵ) is calculated according to the following simplified formula¹

$$\epsilon = \frac{I_d - (\text{background} \cdot \text{pinhole area})}{I_o - \text{background}} \cdot \frac{d}{D}$$

$$I_d = I_{\text{diffracted}}$$

$$I_o = I_{\text{main beam}}$$

d/D = Geometry correction factor

Pinhole area = zero-order pinhole correction

Experimental Results

Comparisons were made of the diffraction efficiencies of individual flat wafers, as cleaved, without surface treatment. As can be seen in Fig. 1, differences are evident even between like-oriented and adjacent wafers from the same boule.

As shown in Fig. 2, we compared the diffraction efficiencies of flat vs convex-curved crystals and untreated vs surface-treated crystals. With respect to flat crystals, the diffraction efficiency increases six to eight times after surface treatment (compare A and B). Generally, the sandblasted crystal (#1 grit, 30 s/side) is superior to the lapped

crystal (600 grit aluminum oxide, 2 min/side) both in efficiency and in the ease and speed of the method.

On the other hand, prior surface treatment of the convex-curved crystal does little to enhance its efficiency as a x-ray dispersing monochromator. That is, if a flat, surface-treated crystal (A) of Fig. 2 is heated to 475-575°C and rolled³ or pressed in a mold¹ to a convex-curved crystal of a desired radius (C), its efficiency as an x-ray diffracting agent generally is found to be less than for a flat, untreated crystal (B). Enhancement of the diffraction efficiency that occurred after

Fig. 2. Diffraction efficiencies of flat vs convex-curved crystals ($r = 25$ mm). (A) Flat crystals, surface treated, (B) Flat crystals, clear and untreated, (C) curved crystals, treated and untreated. Flat crystals are designated by open symbols, curved crystals by solid symbols.

Fig. 3. Diffraction efficiencies of convex-curved lithium fluoride crystals (200) having different radii (3, 6 and 25 mm). The separate $K\alpha_1$ (solid line) and $K\beta_1$ (dashed line) curves are shown for each radius.

the Bragg angle. Therefore, the overall diffraction efficiency is reduced.

Plastic deformation of lithium fluoride wafers (0.2 to 2.0 mm thick) to curved crystals with radii of 2 to 10 cm can be readily accomplished by either rolling³ or pressing.¹ Pressing gives a more uniform radius and generally better crystals are obtained. The pressing procedure was used in all of the present work.

Rolling of the hot crystal results in a variable radius because clamping leaves a flat end and a tendency to warp and to spring back. Diffraction intensity variations, causing a "woodgrain" effect in filmed spectra, seem more prevalent in rolled than in pressed crystals. Thermal shock may

cause crystal breakage unless great pains are taken to preheat the crystal and to provide even cooling upon its removal from the rolling tool.

When successive crystals are being bent, the rolling method is much slower than the press mold procedure because it is more difficult and time-consuming to clamp the pre-heated crystal in the hot rolling tool.

When the crystal is slipped into the press mold, self-centering is automatic. When the press mold comes to temperature in the oven, the mold is closed momentarily. Upon reopening, the hot crystal is removed with preheated tongs and let cool at room temperature on a hot carbon block.

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References

1. B. O. Sellick, Lawrence Livermore Laboratory Rept. UCRL-52086 (1976).
2. L. S. Birks and R. T. Seal, J. Appl. Phys. 28, 541 (1957).
3. L. S. Birks, Rev. Sci. Instrum. 41, 1129 (1970).