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ELECTRON DIFFRACTION ANALYSIS OF MIXTURES OF HEXAGONAL AND CUBIC CdS

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Estimation of hexagonal cadmium sulfide in mixtures with the cubic form can be obtained by reflection electron diffraction of the polycrystalline material. Photographic plates are microphotometered and the integrated intensities are measured for several lines. Intensity ratios for the sample are compared to similar data for calibration standards and the hexagonal content is obtained graphically.

EXPERIMENTAL

Apparatus

A Trueb, Taueber KD3 electron diffractograph was used with Kodak medium-contrast projector slide plates. Plates were scanned with a recording microphotometer, and integrated intensities were then obtained by planimetry.

Reagents

General Electric luminescent-grade CdS was used for the hexagonal standard. Cubic CdS was obtained by passing H_2S[1, 2] into a hot solution 0.3F in HNO_3 and 0.1F in $Cd(NO_3)_2$. After thorough washing, the precipitate was dried in vacuum at 180° C. Only cubic lines appeared when

[redacted]

its purity was tested by electron and X-ray diffraction. Calibration standards containing from 50-100% hexagonal CdS were thoroughly mixed with a spatula on glazed paper.

Procedure

Samples were prepared using a 1/4-in.-wide metal strip moistened with Ambroid cement (excess cement wiped off). The CdS was firmly pressed onto the strip and smoothed with a spatula. No cement appeared on the surface that was examined by electron diffraction. The making of several photographic plates of differing transmission for each standard permitted the ultimate selection of plates having approximately the same background regardless of composition. The plates were microphotometered along four to six different radii to minimize spottiness. The lines scanned are shown in Table I, and a typical pattern is shown in Fig. 1.

Table I

INTERPLANAR SPACINGS

Hexagonal	Cubic
2.068 A	2.058 A
1.898	---
1.791	1.753

The line at 1.898 A was selected for determining percent hexagonal CdS. Overlapping occurs, but this line is moderately intense and sufficiently removed from the shadow edge to prevent marked absorption. The dashed

lines in Fig. 1 indicate the backgrounds selected for these peaks. The areas A and B were obtained with a planimeter and the ratio A/B was computed. The calibration curve appears in Fig. 2. The hexagonal crystallites were large and produced spotty lines. Grinding increased line width and the difficulty in assigning backgrounds. Furthermore, grinding of thin oriented films did not[3, 4] produce normal powder-pattern intensities, and annealing ultimately converted[5, 6] the cubic material to the hexagonal form. Hence, grinding was restricted to that occurring during the mixing process. Large standard deviations resulted from microphotometry of spotted lines. The ratio of peak height to background at 1.89 A shows similar deviations while the ratios at 1.79 A, 2.06 A, and 2.45 A were even less satisfactory. Consequently, even with ideal calibration standards a standard error of about 10% of the hexagonal form is expected for oriented samples yielding spotty patterns. Significantly better results should be obtained for randomly oriented films of small uniform crystal size.

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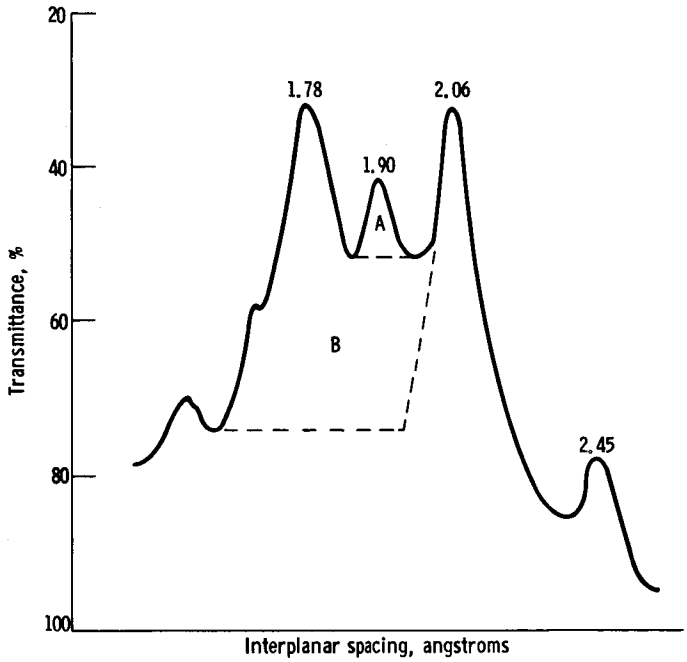

Fig. 1. - Diffraction pattern for 87.20% hexagonal CdS.

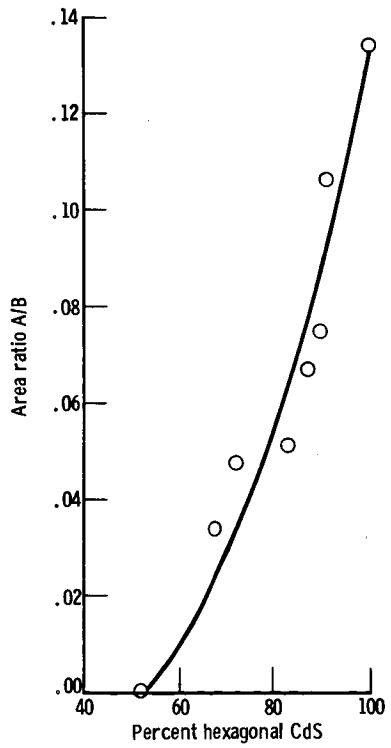

Fig. 2. - Calibration curve based on A/B.