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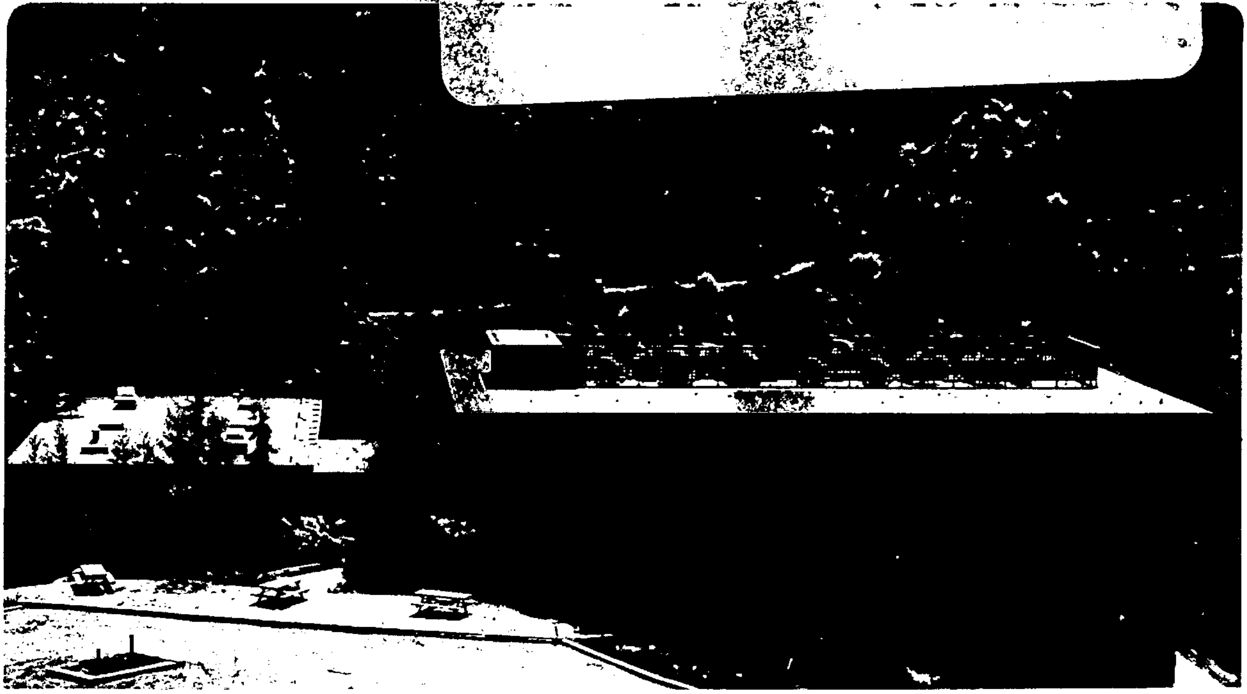

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COMPARISON OF CONVERGENT BEAM ELECTRON DIFFRACTION METHODS FOR DETERMINATION OF FOIL THICKNESS

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ABSTRACT

The methods of determining foil thickness from convergent beam diffraction patterns, the Kelly method and the Ackermann method, were compared in experiments using silicon and iron foils. It was necessary to use the Kelly method to determine the effective extinction distances experimentally. However, tests showed that the thickness determined by the Ackermann method was less sensitive to both systematic and random variations in the data, particularly to variations in the value of the first intensity maxima, for which the percentage errors are largest. The precision in thickness measurement achieved here was on the order of five percent. The deviation in thickness determinations by both methods was less than two percent. The two methods are roughly equivalent unless errors can be reduced below this level.

INTRODUCTION

Foil thickness may be determined using convergent beam electron diffraction (CBED) by either of two methods, the Kelly method (Kelly, Jostons, Blake and Napier, 1975) or the Ackermann method (Ackermann, 1948). Both methods are based on the relationship between the variation of the extinction distance with the deviation from exact Bragg conditions and the foil thickness as it is described by the two-beam approximation to the dynamical theory. The accuracy of the Kelly method was analyzed by Allen and Hall (1982), who used a Co-based alloy with a high density of stacking faults to provide an experimental check on the results. Castro-Fernandez, Sellars, and Whiteman (1985) have recently evaluated the Kelly method. They have concluded that the original recommended procedure can be ambiguous in thickness determination because of systematic reflections. They show that comparison of observed intensity profiles with those from two-beam dynamical calculations can resolve this problem.

No detailed theoretical or experimental analysis of the errors in the Ackermann method has been published. The present work was undertaken to provide an analysis of the Ackerman method and to evaluate the relative accuracy of the Ackermann and Kelly methods.

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EXPERIMENTAL PROCEDURES:

All CBED patterns were taken using a Philips EM400 in TEM mode at an operating voltage of 100kV and a nominal probe size of 400A. All patterns were imaged at a camera length of 750mm to reduce measurement errors. The specimens were tilted to obtain two-beam conditions for various low-order reflections. The convergence angle was approximately one degree or 18 mrad in all cases. High purity silicon and iron were used in this study. The locations of the maxima and minima in the diffraction pattern were measured optically as suggested by Blake, Jostons, Kelly and Napier (1978). Best fit lines and ninety-five percent confidence intervals were determined by a least squares analysis. The precision in thickness measurements achieved here was about five percent, which is similar to that reported by Castro-Fernandez, et al.(1985).

RESULTS AND DISCUSSION:

Both the Kelly and the Ackermann methods use a result from two-beam dynamical theory that relates minima in intensity oscillations to the foil thickness,t

$$(s_i^2 + 1/\xi_g^2)t^2 = n_k^2 \tag{1}$$

where s_i is the deviation of the i-th minimum from the exact Bragg position (center of CBED disc), ξ_g is the extinction distance and n_i is a whole number. Equation (1) was first derived by MacGillavry (1940). It was used to determine foil thickness by Ackermann (1948) who noted that the foil thickness t and the extinction distance could be obtained from the slope and intercept, respectively of a plot of s_i^2 versus n_k^2. The Kelly method uses a different form of the same equation; the thickness and extinction distance are obtained from the intercept and slope, respectively, of a plot of $(s_i/n_k)^2$ against $(1/n_k)^2$. In these calculations, the value of k is given by $k=i+j$, where j is the largest whole number less than t/ξ_g. If the characteristic equation is modified suitably, the accuracy of the linear regression can be improved by including intensity maxima as well as minima (Allen, 1981).

Systematic errors in the two-beam dynamical approximation and random errors in the measurements of s_i influence the thickness values obtained using either method of analysis. The largest systematic error associated with the two-beam dynamical approximation is the upward shift in the s_i values due to anomalous absorption. The displacement in s_i decreases rapidly with increasing s, so the largest error is associated with the first minimum (Kelly, et al., 1975). However, the maxima are shifted toward $s=0$, so the error is significantly lessened if both types of extrema are considered (Blake, et al., 1978). The measurement error is also greatest for the first minimum, since its absolute value is small. Figure 1 shows the effect of altering the value of s_1 on thickness determinations by both methods for silicon, $g = 02\bar{2}$ using intensity maxima only. Clearly, the Ackermann method is less sensitive to variations in s_1. To quantify this observation, the thickness from the Kelly method would be altered by about 2.5% for a worst case error of about 25% in s_i (15% due to absorption, 10% due to error in measuring the first fringe) while the Ackermann method value would change by less than one percent. The effect is smaller if of both types of extrema are used.

A practical problem with both methods is the determination of the correct value of j given by t/ξ_g. The Ackermann method requires knowledge of ξ_g from other sources, since the lines are straight for all j. In the Kelly method, the correct choice of j is indicated by a straight line on the plot, but in practice this choice can be difficult since it may be possible to fit the data for more than one curve with a straight line. One indication of thr correct choice is the size of the 95% confidence interval for y on x for each best-fit line determined by regression analysis. The 'straight' line is generally the one for which the relative 95% confidence interval, the ratio of the confidence interval to the y-intercept $1/t^2$, is a minimum. However, the confidence interval is a measure of the size of the deviation of a set of points from a straight line and gives no indication of curvature. Comparison of the calculated apparent extinction distance with theoretically determined values often clarifies the situation. However, this method does not always work and the approach suggested by Castro-Fernandez et al., i.e. comparison of experimental and two-beam calculation intensity profiles appears to be the most reliable method.

The two-beam approximation fails when the effect of multi-beam interactions becomes significant. Although theory predicts that the dark field rocking curve is symmetric, Allen (1981) suggested that this effect would be smaller on the $s<0$ side of the CBED disc and proposed that θ_i measurements be made only on that side. This effect was specifically investigated by Castro-Fernandez et al. for a large number of samples and orientations. They did not see a statistically significant effect in their experiments. However, an asymmetry in the CBED fringes was observed in this work which could cause significant errors in the thickness measurements. The difference in thickness results for measurements taken on the $s>0$ and $s<0$ sides of the disc for an iron sample in the [301] orientation is illustrated in Figure 2. The error introduced by using data for $s>0$ was approximately 13% in this case. We put our data forward mainly to suggest that significant errors may result if the sample is wedged or if the probe crossover is not precisely in the specimen plane.

On the basis of the difference in sensitivity to measurement, and anomalous absorption errors, we suggest that the Ackermann method is more accurate than the Kelly method. The correct choice of j may be found using the Kelly method or the approach suggested by Castro-Fernandez et al. prior to making the final calculation with the Ackermann method. However, the difference in the thicknesses determined by the two methods (for $s<0$ data) was never greater than two to three percent. Unless other errors, such as thickness averaging across the probe, can be reduced below this level the methods are roughly equivalent.

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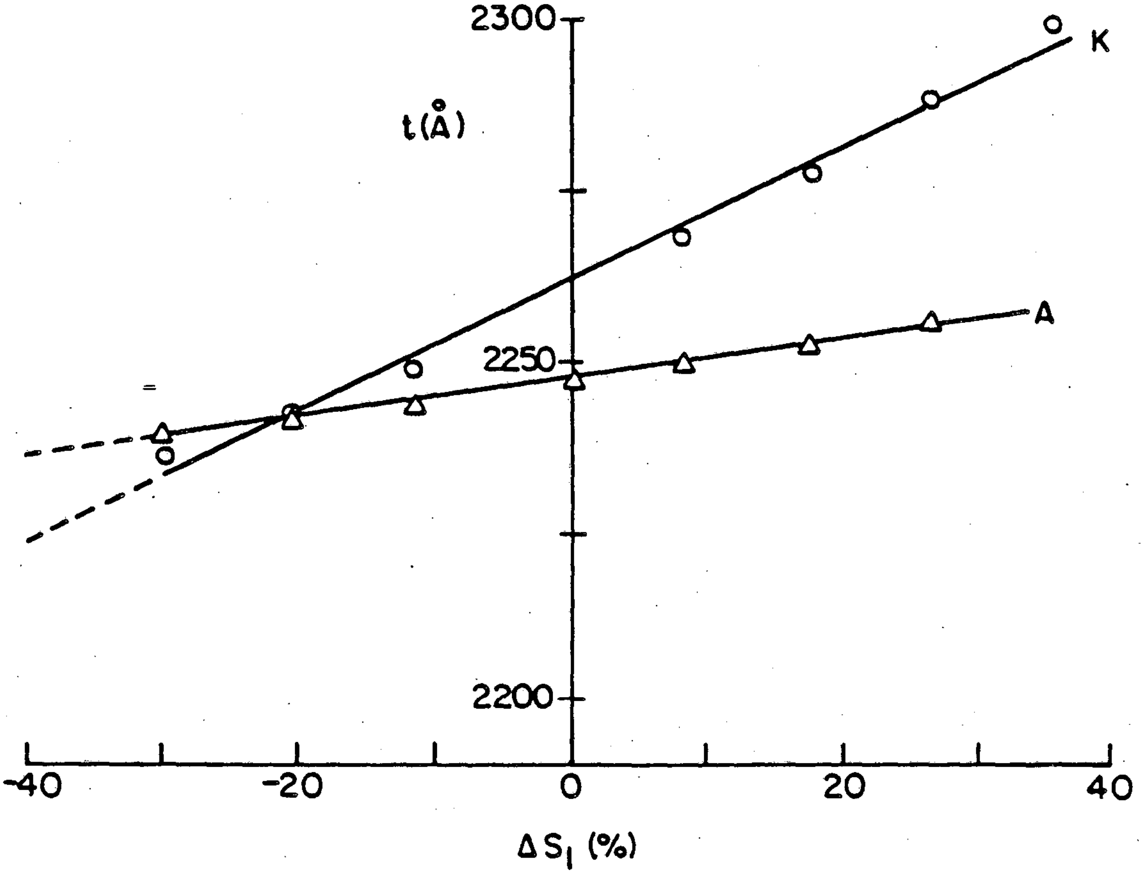

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Figure 1. Comparison of the effect of variation of s_1 on the thickness calculated by the Kelly and Ackermann methods for silicon, $g=02\bar{2}$, maxima and minima.

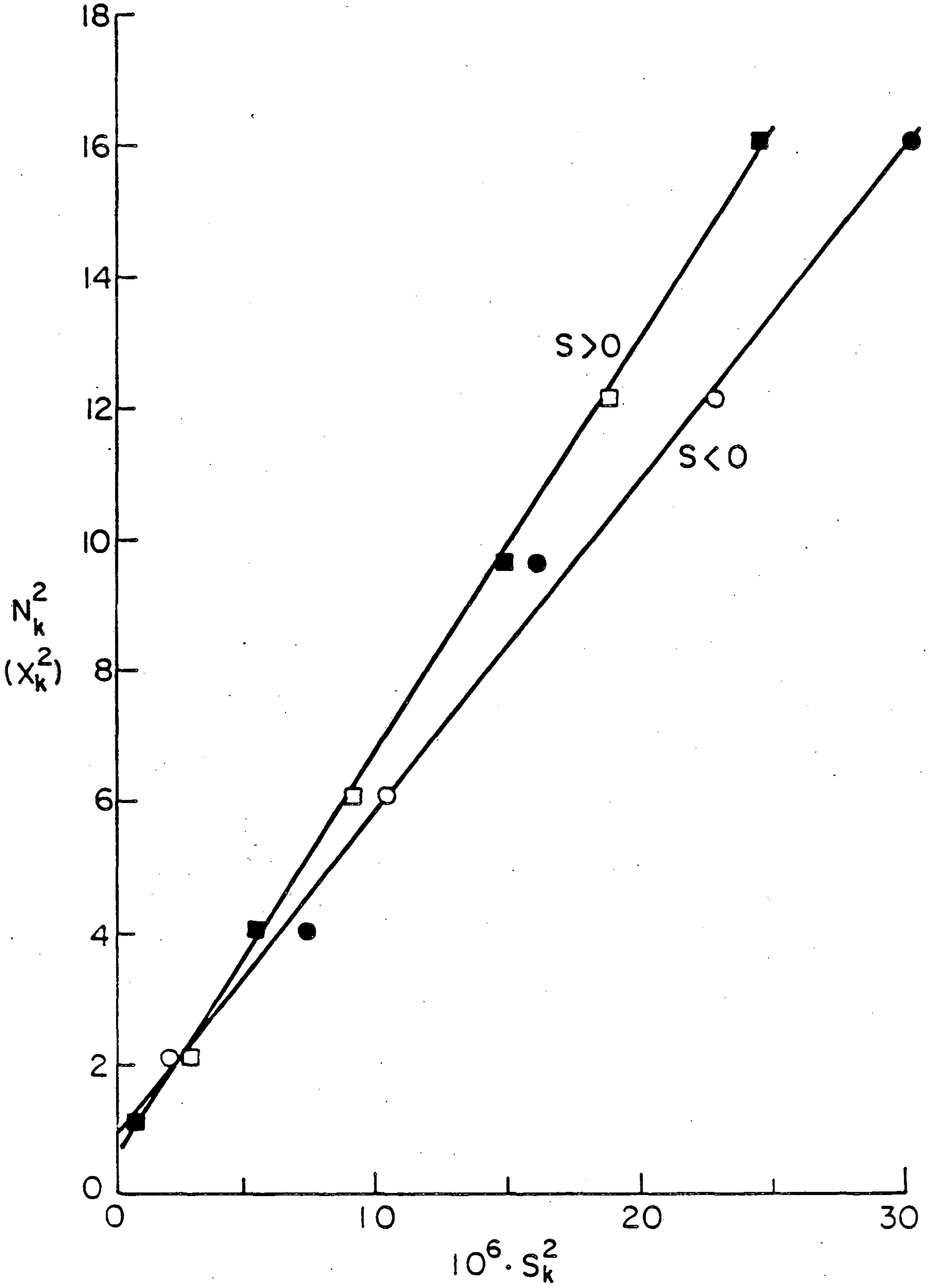

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Figure 2. Illustration of variation in calculated thickness due to asymmetry in intensity distribution for s>0 (squares) and s<0 (circles) in the convergent beam disc. Ackermann method for iron, g=301. Open points are data points for intensity maxima, dark points for intensity minima.