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October 1972

Prepared for the U. S. Atomic Energy Commission under Contract W-7405-ENG-48

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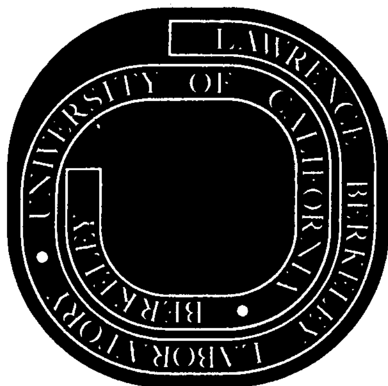

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Specimen Thickness Determination in Transmission Electron Microscopy in the General Case

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2 Figures, 1 Table

(Short title: Thickness Determination)

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The techniques to determine foil thicknesses as treated in some textbooks (Hirsch et. al., 1965; Thomas, 1962; Reimer, 1967; Schimmel, 1969, von Heimendahl, 1970) require special image features such as either traces (from dislocations or precipitates) or wedge fringes or bend contours. However, often there are none of these features in the particular area of interest. One method of thickness determination for this general case using extinction contours under "divergent beam" conditions in the diffraction pattern was developed by Bell and Thomas (1969) (first introduced by Amelinckx, 1964). Sadhukhan (1970) suggested the use of latex balls to be applied on both sides of the foil. Its thickness t is derived from the changes of the projected ball distances after tilting the specimen through large angles. Additional cross shadowing is necessary to distinguish on which of the two surfaces the latex balls were. Also Vingsbo (1970) used a high angle tilting technique together with inherent features extending from one foil surface to the other.

The method described here can be used in the most general case of a transparent foil, i.e. no particular image features and no complicated shadow castings are necessary. This method may be considered as an independent alternative to the one of Bell and Thomas (1969).

Experimental

Latex balls as commercially available (diameter D = 2340 ± 26 Å) are applied to the foil surfaces by simple dipping into a clean alcoholic suspension. The concentration of this latex suspension has to be tried out such that about the required density of latex balls comes onto the surface, ideally just one ball on each side in the field of view (one drop of the latex milk per 5 cm^3 alcohol for first trial). Let the specimen dry by careful setting on filter paper in an oblique manner

such that the suspension is allowed to flow down gently from either side. Fig. 1 shows an example, in this case a disc specimen of an Al-4%Cu alloy obtained by twin-jet electropolishing in a commercial polisher (E. A. Fischione, Verona, Pa.). The sample was aged to produce large platelike Θ'-precipitates for an independent thickness determination by trace analysis. Fig. 1 was taken at 100 kV in a Philips EM 300 microscope fitted with goniometer stage which provides easy and accurate tilting in both directions up to $\pm$ 60°.

Method of Thickness Determination

In Fig. 2 the three black dots should mark the centers of latex balls (which themselves are usually in the order of magnitude of the foil thickness t). The following treatment therefore yields $t' = t + D$, the figure thus giving a cross section of a hypothetical foil, resulting from the real foil t plus twice the latex ball radius. At first the primary beam P is considered to be perpendicular to the foil in the untilted condition (deviations ω from this condition are discussed later.)

First, one has to distinguish whether two balls needed for the analysis are - as required - on different sides. This may be easily performed as follows: two particles on the same side always reduce their projected widths d on the screen during tilting in both directions, whereas particles on different sides increase their d-value while tilting to one direction ($\alpha > 0$ in Fig. 2) and decrease d when tilting in the other direction ($\alpha < 0$).

Let us first suppose the foil is tilted in the positive direction as shown in Fig. 2 from which follows $\cos \beta = t'/\ell$, $\sin \beta = d_1/\ell$, $\sin (\alpha + \beta) = d_2/\ell$. Eliminating ℓ from the last two equations and applying

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the additive formula for sin $(\alpha + \beta)$ yields $d_1(\sin\alpha \cot\beta + \cos\alpha) = d_2$. With $\cot\beta = t'/d_1$ the foil thickness t results as follows:

$$t = t' - D = \frac{d_2 - d_1 \cos\alpha}{\sin\alpha} - D \qquad (1)$$

If the foil is tilted into the other direction the projected width becomes <u>smaller</u> and is denoted as d_3 after tilt for better distinction. Taking the absolute value of α, an analysis analogous to Fig. 2 yields in this case:

$$t = t' - D = \frac{d_1 \cos\alpha - d_3}{\sin\alpha} - D \qquad (2)$$

The d's may be measured as center - to center distances of the latex balls, measured in the direction perpendicular to the tilt axis, or more accurately as the corresponding edge distances as indicated in Fig. 1. With $d_1 = 3.7$ mm, $d_2 = 9.2$ mm, $D = 6.0$ mm on the plate and $\alpha = 24°$, formula (1) yields $t = 8.3$ mm or - divided by the magnification 25,600 times - a foil thickness of 3240 Å. For comparison, thickness determination by conventional trace analysis (see the textbooks mentioned above) using the Θ'-plates in Fig. 1 before and after tilt yields 3470 Å.

<u>Accuracy of the Method</u>

Although this technique is generally applicable, its accuracy is of course limited as that of any physical measurement. The following sources of error have been treated:

(a) uncertainty of d_1-measurement. Magnifying glasses or photometers are used; although normally better, an error of 0.3 mm on the negative or original print is taken into account using

a magnification of 50,000 X;

(b) the same for d_2;

(c) The influence of an error of 1° in α is considered. In crystalline samples α can be measured much more accurately by use of Kikuchi line shifts (Thomas (1970); von Heimendahl (1971)); however, the method described here is also applicable to non-crystalline specimens like carbon films etc.

(d) usually it is unknown whether P is exactly parallel to the foil normal in the start position (d_1-measurement) as assumed in the above derivation. A slight deviation $\omega < 0$ is considered in Fig. 2 as follows: the foil is assumed to be fixed in the specimen holder forming ω between P′ and the foil normal. Then the appearing (wrong) projected widths are seen not along P, but along the deviating direction (Fig. 2) and can be worked out graphically or better numerically this way. $\omega = -5°$ and $-10°$ were taken as examples.

In all cases equation (1) was applied and the resulting thicknesses infected with the errors are called t*. It is of particular interest to know these errors as a function of β since in practice one has often a choice between particle pairs more or less close together. Table 1, therefore, lists the results of (a) - (d) for $\beta = 0°$, 12° and 40°.

It can be seen from Table 1 that the errors (a) to (c) are small to medium, do not depend strongly on the choice of β and cancel out each other partially. Larger errors may appear only from $\omega \neq 0$ <u>if</u> β is large. From this point of view, it is therefore recommended to <u>work with small β</u>. Unfortunately, an $\omega \neq 0$ cannot be identified by using the two different tilt directions: for a given $\omega > 0$ in both

cases, using $\alpha > 0$ and < 0, t^* comes out too large, and for $\omega < 0$ in both cases too small. Also a $\omega \neq 0$ condition cannot be checked by using two particles on one side of the foil: although basically these should have a maximum distance while tilting at $\omega = 0$, this effect is much too small for practical use due to the extremely slow variation of the cos-function near zero. It is one more advantage of disc specimens as obtained from electrolytic jet stream polishing that they usually have only a negligibly small deviation parameter ω.

The dependence on the assumed foil thickness in Table 1 is twofold and easily understood: first, with larger thickness, the projected widths become also larger and therefore the relative d_1 and d_2-accuracy becomes better, keeping the absolute width accuracy constant (0.3mm on the plate). This is in agreement with similar observation of Vingsbo; second, a larger thickness t means generally a better relative accuracy since in (1) and (2) the constant diameter D has to be subtracted (which is in the order of t). Therefore, also the relative errors from α- and ω- deviations in Table 1 become smaller the thicker the foil.

Four or five measurements with different tilt angles α are usually possible under good contrast conditions. In practice, by averaging these independent measurements it was always possible to determine t with a standard deviation of less than 4%. Largest deviation of a single measurement was 8%. This is about in agreement with the calculations of Table 1.

Strongly wedge-shaped specimens need a special geometric treatment which is not intended to be dealt with in this paper. The derivation of equation (1) requires a plain-parallel specimen which is usually provided within the small area of view also in electropolished thin metal foils.

The comparison with trace analysis value for t taken from the Θ'-platelets as calculated above for the example of Fig. 1 yielded agreement within 7%. However, trace analysis results in the current investigation were less accurate due to irregular plate edges resp. poor resolution of the intersection between plate and surface. For reliable and accurate results it is therefore preferably to use the method described in this paper.

Summary

A technique is described to determine the foil thickness t of transparent specimens as used in electron microscopy. t is gained from the changes of the projected widths of pairs of latex balls on both specimen surfaces during tilting (large angles). The accuracy of the method is investigated critically in dependence of several sources of errors. In routine work, a standard deviation of less than 4% is obtainable.

Acknowledgements

The author would like to thank Dr. K. Schneider and Dipl. Phys. H.-J. Hausselt, both Erlangen, for fruitful discussions and Professor G. Thomas, Berkeley for critical reading of this manuscript. This work was partially supported by a grant of the Max-Kade Foundation, New York, which is gratefully appreciated; and partially supported under the auspices of the U.S. Atomic Energy Commission.

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Table I: Relative error = (t* - t)/t of determined foil thickness in % as function of different error sources. Calculations are performed for two thicknesses t: 0.3 and 1.0μ; the tilt angle is assumed to be 20°.

	$\beta = 0$		$\beta = 12°$		$\beta = 40°$	
	Foil thickness t					
	0.3	1.0	0.3	1.0	0.3	1.0μ
(a) d_1 assumed to be 0.3 mm too large measured on the plate (50,000 x Magnification)	-5.5	-1.6	-5.5	-1.6	-5.5	-1.6
(b) d_2 assumed to be 0.3 mm too large measured on the plate (50,000 x Magnification)	+5.9	+1.8	+5.9	+1.8	+5.9	+1.8
(c) α assumed to be 1° too large measured (21° instead of 20°)	-8.1	-5.6	-7.5	-5.2	-5.6	-3.9
(d) $\omega = -5°$	-0.7	-0.5	-4.0	-2.7	-13.7	-9.5
(d) $\omega = -10°$	-2.7	-1.9	-9.3	-6.4	-28.6	-19.8

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Figure Captions

Fig. 1 Electrolytically thinned specimen of an Al-4%Cu alloy with applied latex balls. Foil thickness t from (1) 3240 Å ± 4%. 25,600 X.

Fig. 2 Cross section through a hypothetic foil of thickness t' = real foil thickness t + diameter D of latex balls. Tilt axis perpendicular to drawing plane, α = tilt angle. ℓ = projection of ball distance upon drawing plane, d_1 and d_2 are the projections of ℓ on the plate before resp. after tilt, P = primary electron beam, ω see text.

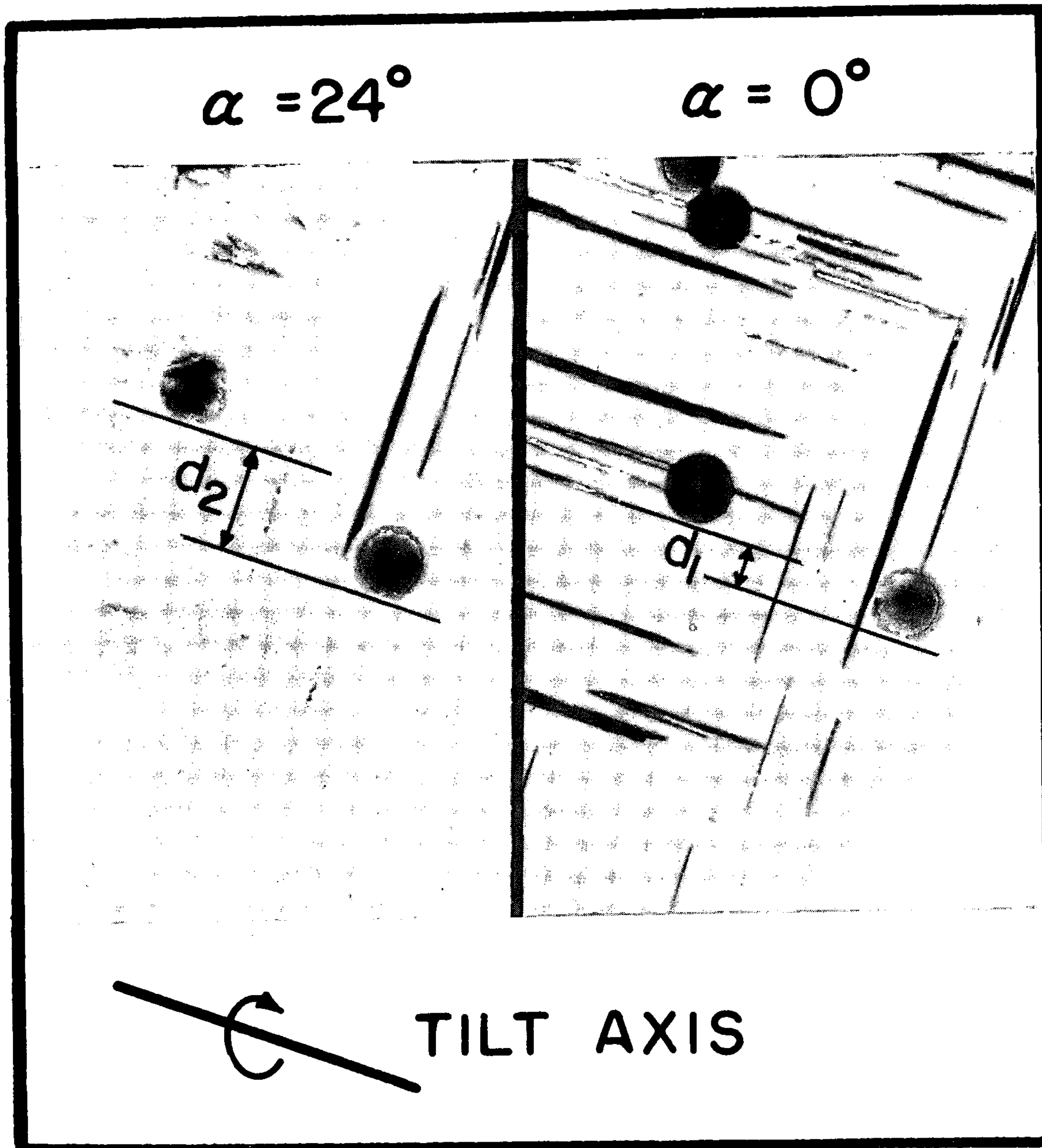

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Fig. 1.

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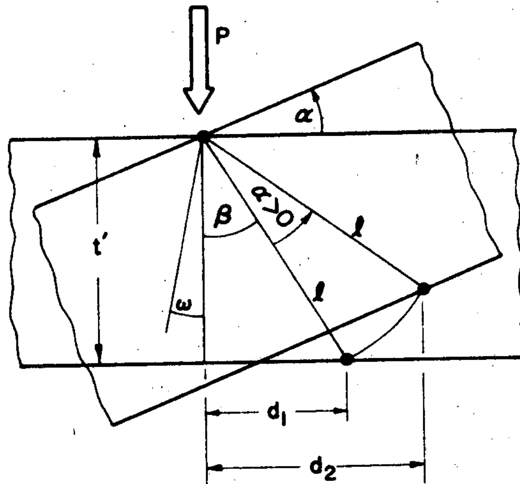

Fig. 2.

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