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AN EXPERIMENTAL TEST OF A ZAF CORRECTION PROGRAM FOR TILTED SPECIMENS AND ENERGY DISPERSIVE SPECTROMETRY

B. Lödding and L. Reimer

Physikalisches Institut, Universität Münster, Domagkstrasse 75, D-4400 Münster, F.R.G.

Résumé - On propose et teste une méthode ZAF modifiée dans laquelle on évalue le facteur R et la correction d'absorption pour le cas d'un objet incliné.

Abstract - A modified ZAF correction program is proposed and tested which considers the R-factor and the absorption correction for tilted specimens.

In many scanning electron microscopes with a plane lower pole-piece of the probe-forming lens, the Si(Li) energy-dispersive x-ray detector works with a take-off direction normal to the electron beam and the specimen has to be tilted by an angle \( \theta \) between electron beam and surface normal. The backscattering coefficient and the mean energy of backscattered electrons increase with increasing \( \theta \) resulting in a decreased production of characteristic x-ray quanta as compared to normal incidence. Otherwise, the absorption decreases with increasing \( \theta \) due to the decrease of the absorption path length and the production of x-rays nearer to the specimen surface. This results in a maximum of recorded x-ray quanta at a tilt angle \( \theta_{\text{max}} \) (Fig.1). This maximum will also be the optimum tilt for a quantitative x-ray microanalysis which, however, needs modifications in the backscatter and absorption correction factors /1/.

The backscatter factor \( k_R \) is defined as \( k_R = 1/R \) where \( R = (n - \Delta n)/n \) and \( n \) denotes the total intensity without backscatter correction and \( \Delta n \) the fraction not excited due to backscattering. The backscatter factor \( R \) can be measured with a special specimen /2/. A thin 1-2 \( \mu \)m foil with a hole of 50 \( \mu \)m is mounted on top of a bulk specimen (Fig.2). We measure either \( n = n - \Delta n \) or \( n^2 = n \) because the backscattered electrons are absorbed in the foil and generate x-ray quanta. The recorded signal \( n^2 \) has to be corrected for the absorption of x-rays in the foil. Fig.3 shows experimental \( R(\theta) \) values for Cu Ka and different overvoltage ratios \( U \).

The loss \( \Delta n \) of x-ray excitation can also be calculated from the energy distribution \( dn/dW \) of the backscattered electrons where \( W = E_{\text{BSE}}/E_0 \):

\[
R = 1 - \int \frac{dn}{dW} \frac{Q_R(E)}{S} dE dW / \int \frac{E_{\text{BSE}}}{E_0} \frac{Q_R(E)}{S} dE.
\]

\( E \) denotes the ionisation energy of the corresponding shell. When calculating \( dn/dW \) by the Monte Carlo method /3/, \( R \) can be calculated by numerical integration. Experiments and calculations are plotted in Fig.4 and are compared with the corresponding formula proposed by Love /4/. Good agreement can be found for \( \theta \leq 45^\circ \).

The absorption correction can be modified by substituting \( \chi \cos \theta \) or \( \chi (1 - 0.5 \sin^2 \theta) \) for \( \chi /5\). If using the complete Philibert /6/ formula, the surface ionisation \( \Phi \) can be considered which is normally neglected. Our experiments showed that \( \Phi(\theta) \propto \sec \theta \). This results in a more reasonable depth distribution function \( \Phi(\rho z) \) of x-ray emission (Fig.5). However, the influence on the absorption correction factor \( f(\chi) \) will...
normally be small, so that the simple formula can be used for $x \leq 1 \text{ cm}^2/\text{g}$.

For testing the modifications, we measured the x-ray counts from alloys of known concentration and from the pure element standards over a large range of tilt and azimuth angles of the surface normal. The concentrations were calculated from the k-ratios using a FRAME program with the modifications discussed above. Figure 6 shows typical examples for Cu-Al and Cu-Au alloys. The measurements were done with different take-off directions of the Si(Li) detector (see insets in Fig.6). The concentrations calculated with the modified A- and R-corrections agree with the real concentration over a large range of tilt angles when using the detector with a take-off angle of $45^\circ$ relative to the electron beam in a SEM with a conically-shaped pole-piece. However, agreement only exists in a range $\phi=30^\circ-60^\circ$ when working with a take-off angle of $90^\circ$ in a SEM with a plane lower pole-piece. The smallest error can be observed at $\phi=45^\circ$ which is identical with $\phi_{\text{max}}$ for this geometry. The exact position of $\phi_{\text{max}}$ depends, however, on the composition of the specimen and on the ratio of backscatter and absorption corrections.

**Fig.1 - X-ray intensity versus tilt**

**Fig.2.** Experiment for measuring R angle $\phi$.

**Fig.3 - Experimental R-values of the backscatter correction of Al, Cu and Au as a function of specimen tilt $\phi$ for different overvoltage ratios $U$.**
Fig. 4 - Comparison of the dependence of \( R \) on overvoltage ratio \( U \) at tilt angles \( \theta = 0^\circ, 45^\circ \) and \( 60^\circ \), (○) Monte Carlo calculations (●) Experiments
Dashed curves: formula of Love et al. /4/.

Fig. 5 - Comparison of depth distribution of Cu Kα excited by 20 keV electrons. a) Monte Carlo calculation, b) modified Philibert formula:
\[
\Phi(pz) = c R_\infty \exp(-\alpha pz \sec \theta) - (1-R_\infty \sec \theta/R_\infty) \exp(-\sigma(1+\frac{1}{n})pz \sec \theta).\]
Fig. 6 - Influence of tilt angle $\phi$ on the x-ray analysis of Cu-Al (a,c) and Cu-Au (b,d) alloys for take-off angles of $90^\circ$ (a,b) and $45^\circ$ (c,d) of the Si(Li) detector. Dash-dotted curves: uncorrected ratio $k=N/N_s$ ($N_s$ counts from pure element standard). Dashed curve: concentrations calculated with an unmodified FRAME program for normal incidence ($\phi=0$). Full curves: concentrations by the program containing the tilt correction.

References