THICKNESS DETERMINATION OF Al FILMS ON Si
BY A MONTE CARLO CODE INCLUDING A
SECONDARY FLUORESCENCE CORRECTION
A. Armigliato, A. Desalvo, A. Garulli, R. Rosa

To cite this version:
A. Armigliato, A. Desalvo, A. Garulli, R. Rosa. THICKNESS DETERMINATION OF Al FILMS ON Si BY A MONTE CARLO CODE INCLUDING A SECONDARY FLUORESCENCE CORRECTION. Journal de Physique Colloques, 1984, 45 (C2), pp.C2-29-C2-32. <10.1051/jphyscol:1984207>. <jpa-00223763>

HAL Id: jpa-00223763
https://hal.archives-ouvertes.fr/jpa-00223763
Submitted on 1 Jan 1984

HAL is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L’archive ouverte pluridisciplinaire HAL, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d’enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.
THICKNESS DETERMINATION OF Al FILMS ON Si BY A MONTE CARLO CODE INCLUDING A SECONDARY FLUORESCENCE CORRECTION

A. Armigliato*, A. Desalvo**, A. Garulli* and R. Rosa*

*CNR, Istituto LAMEL, Via Castagnoli, I, 40126 Bologna, Italy
**Istituto Chimico, Facoltà d'Ingegneria, Università di Bologna, Italy

Résumé - On a introduit dans notre programme Monte Carlo une correction pour le rayonnement de fluorescence X dû aux raies caractéristiques du substrat dans une couche mince. Cette correction a été appliquée avec succès pour évaluer avec une meilleure précision l'épaisseur de couches d'Al sur Si.

Abstract - A correction for x-ray characteristic fluorescence in thin films on substrates has been successfully applied to Al films on Si in order to get a more accurate evaluation of their thicknesses.

INTRODUCTION

Corrections for characteristic fluorescence in electron probe microanalysis are often disregarded by computer programmes dealing with thin unsupported and supported films. Analytical treatments as applied to unsupported films and film/substrate systems were put forward by Nockolds et al. /1/ and Cox et al. /2/ respectively. To account for such an effect we improved our Monte Carlo computer programme (CARLONE) as described elsewhere /3/. In this paper we applied the code to the determination of the mass thickness of Al/Si films. The results were compared with the ones obtained by Rutherford backscattering spectrometry (RBS).

EXPERIMENTAL

By electron beam evaporation we prepared a number of Al films of different thicknesses up to ~50 μg/cm², onto Si and boron nitride (BN) substrates. The latter was chosen for RBS measurements, because, as it is known, accurate values of thickness can be obtained by the above technique only if the atomic number of the substrate is lower than that of the film. X-ray microanalysis measurements were performed with two different instruments: i) an ARL-SEMQ fully automated electron microprobe (take-off angle Ψ = 52.5°) equipped with WDS spectrometers; ii) a Cambridge Stereoscan 150 Mark II SEM (Ψ = 30°) equipped with a Tracor-Northern EDS Spectrometer. The accelerating voltages were 10, 20 and 30 kV in both instruments. The intensity of the AlKα line was measured on both Al/Si and Al/BN samples, as well as on bulk standards of Al, in order to deduce the corresponding k-ratios. RBS analyses were performed with the 2 MeV Van de Graaff accelerator of the Laboratori Nazionali di Legnaro. The incident beam was 1.8 MeV 4He and the backscattered ions were detected at θlab = 150° by a Si surface barrier detector.

MONTE CARLO PROGRAMME

According to /3/ the primary x-ray intensity emitted by the volume element of thickness d(ρz) (s stands for "substrate") around point P is calculated (see Fig. 1). After that the programme computes the x-ray intensity absorbed by the annular volume element of thickness d(ρz) (f stands for "film") centered on point Q (see also /4/). The x-ray fluorescence intensity at this point is obtained from the
absorbed one by the usual procedure, taking into account the jump ratio and the fluorescent yield. The x-ray characteristic fluorescence intensity $dI_F$ to be added to the primary radiation $dI_p$, as generated in the layer $d(qz)_f$, is obtained by performing the integration over the angle $\psi$ from 0 to $\pi/2$ and over the layer $d(qz)_s$ from infinity to 0. The result is given by the formula:

$$dI_F = \frac{r_i - 1}{r_i} \frac{p_i w_i}{2} \left[ \int_0^{\pi/2} \phi(qz)_S d(qz)_S \right] \left[ \int_0^\infty \phi(qz)_S d(qz)_S \right] E_1 \left[ \int_0^\infty (qz)_S + \int_0^\infty (qz)_f \right]

where $r_i$ is the jump ratio for the particular x-ray, $w_i$ is the fluorescent yield, $p_i$ is the relative intensity of the analyzed x-ray in the series and $E_1(x)$ the exponential integral. The function $\phi(qz)_S$ is obtained by the Monte Carlo simulation and the integration is performed numerically. The resulting total intensity $dI_{tot} = dI_p + dI_F$, as emitted from the film layer $d(qz)_f$, can be handled by CARLONE for any value of film thickness, thus removing the limitation to a thin layer inherent in the analytical approach by Cox et al. /2/. Notice that in evaluating the k-ratio, some constants in the fluorescence term do not cancel out, giving however a factor very close to unity. For sake of completeness we took into account also the fluorescence due to the SiK$\beta$ radiation, which turns out to be very small (some percent of that due to SiK$\alpha$).

Fig. 1 - Computational scheme of x-ray fluorescence emission for the case of a film over a substrate.

RESULTS AND DISCUSSION

By Monte Carlo simulation we have constructed calibration curves of $k_{Al}$ vs. $qz$ for the Al/Si case, both neglecting and taking into account the fluorescence effect. The results, for the case of the ARL microprobe ($\Phi = 52.5^\circ$) at the three operating energies, are displayed in Fig. 2. It is worthwhile to notice that the simulations at a given energy were undertaken using the correlated sampling, i.e. with the same random numbers sequence, both with and without fluorescence. Thus the statistical uncertainty due to the random draws does not overshadow the effect of the fluorescence itself. From Fig. 2 it appears that, while at 10 keV the fluorescence effect is negligible (the curves are practically superimposed) at 20 and 30 keV it is of increasing significance. The experimental k-ratios of four Al films of increasing thickness are reported in Tab. 1 for both electron microprobe and SEM/EDS. It is important to remind that, due to the inherent lower energy resolution of this latter technique, the net peak intensities of the AlKa peaks are obtained by a multiple least square fitting procedure applied to the overlapped Al-Si peaks /5/. Instead, in the case of microprobe, the AlKa and SiKa peaks are very well separated and hence it is only necessary to subtract the background under the peak, as obtained by a linear interpolation. Therefore, particularly at higher accelerating voltages, when the x-ray intensity generated in the film is lower, a
better accuracy in the determination of k-ratios is expected in case of the micro-probe. From the k-ratios reported in Tab. 1 it is possible to deduce the film thickness by comparison with the calibration curves reported in Fig. 2.

![Fig. 2 - k-ratio for the AlKα line vs. film thickness for 10(a), 20(b) and 30 keV (c) incident electrons ( \( \Phi = 52.5^\circ \)). Full curve, with fluorescence; broken curve, without fluorescence.](image)

The results are shown in Tab. 2, together with the corresponding values obtained by RBS analysis; the method employed to determine the film thickness from a RBS spectrum is reported elsewhere /6/. An inspection of Tab. 2 allows one to draw the following conclusions: i) the thickness data obtained with the microprobe are generally in good agreement with those obtained by RBS technique, whereas the SEM/EDS results are less accurate, as previously discussed. ii) the difference between the curves reported in Fig. 2 corresponds to a 10% variation in the k-ratios with and without secondary fluorescence. Neglecting the fluorescence correction results in a worsening of the overall agreement with the nuclear backscattering data.

In conclusion, this work demonstrates that in the Al/Si case, which is of interest in silicon technology, to obtain an accurate evaluation of the film thickness, it is necessary to have available a thickness dependent fluorescence correction. If this correction is inserted into a Monte Carlo code, it is also possible to predict for what thicknesses and accelerating voltages such a correction is significant.

Tab. 1 - Experimental k-ratios \((x10^{-2})\) of four Al films analysed at three accelerating voltages \((E_0 = 10, 20 \text{ and } 30 \text{ keV})\) by both electron microprobe and SEM/EDS.

<table>
<thead>
<tr>
<th>Film</th>
<th>MICROPBRE</th>
<th>SEM/EDS</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>10</td>
<td>20</td>
</tr>
<tr>
<td>Al/Si1</td>
<td>6.32±0.06</td>
<td>1.76±0.06</td>
</tr>
<tr>
<td>Al/Si2</td>
<td>11.08±0.10</td>
<td>2.95±0.12</td>
</tr>
<tr>
<td>Al/Si3</td>
<td>16.63±0.15</td>
<td>4.22±0.20</td>
</tr>
<tr>
<td>Al/Si4</td>
<td>22.54±0.25</td>
<td>5.93±0.12</td>
</tr>
</tbody>
</table>
Tab. 2 - Mass thickness (μg/cm²) of the films reported in Tab. 1, as obtained by x-ray microanalysis (electron microprobe and SEM/EDS) through the Monte Carlo calibration curves. The corresponding RBS results are also shown for comparison.

<table>
<thead>
<tr>
<th>Film</th>
<th>MICROPORBE</th>
<th>SEM/EDS</th>
<th>RBS</th>
</tr>
</thead>
<tbody>
<tr>
<td>E₀</td>
<td>10</td>
<td>20</td>
<td>30</td>
</tr>
<tr>
<td>Al/Si1</td>
<td>13.0±0.2</td>
<td>14.0±0.6</td>
<td>14.4±0.2</td>
</tr>
<tr>
<td>Al/Si2</td>
<td>22.4±0.2</td>
<td>23.4±0.7</td>
<td>24.8±0.2</td>
</tr>
<tr>
<td>Al/Si3</td>
<td>31.6±0.4</td>
<td>32.8±1.6</td>
<td>34.4±0.8</td>
</tr>
<tr>
<td>Al/Si4</td>
<td>41.0±0.4</td>
<td>44.8±0.8</td>
<td>45.6±0.8</td>
</tr>
</tbody>
</table>

ACKNOWLEDGEMENTS

The authors are indebted to S.Guerri for preparation of the films, to R.Rinaldi for the use of electron microprobe facility of the University of Modena, and to M. Berti and A.V.Drigo for the RBS measurements.

REFERENCES