doi:10.1017/S1431927616000404 © Microscopy Society of America 2016

Non-Destructive Surface Analysis of Materials by MeV Ion Beams, Microscopy and Computer Simulation

J. Pacheco de Carvalho[1,2], C. F. R. Pacheco[2] and A. D. Reis[1,2]

[1.] Departamento de Física, Universidade da Beira Interior, Rua Marquês d'Ávila e Bolama, 6201-001 Covilhã, Portugal.
[2.] Unidade de Detecção Remota, Universidade da Beira Interior, Rua Marquês d'Ávila e Bolama, 6201-001 Covilhã, Portugal.

Material analysis, especially surface analysis of materials, has been increasingly important. A wide range of surface analysis techniques is available, involving e.g. ion, electron and photon beams interacting with a solid target. The techniques are, generally, complementary and provide target information for near surface depths. Nuclear techniques, which are essentially non-destructive, provide for analysis over a few microns giving absolute values of concentrations of isotopes and elements. Applications have been given in a variety of areas such as scientific, technologic, industry, arts and medicine, using low energy MeV ion beams [1-7]. Nuclear reactions make it possible tracing of isotopes with high sensitivities. We use ion-ion reactions and the energy analysis method. At a suitably chosen energy of the incident ion beam, an energy spectrum is acquired of ions arising from nuclear events, occurring at successive depths in the target. Θ_L is the laboratory detection angle and Θ_R is the target rotation angle. Such spectra are simulated and compared with experimental data, giving target composition and concentration profile information [4-7]. Elastic scattering is a particular and important case. In this context a computer program has been developed, mainly for flat targets [4-6]. The non-flat target situation arises as an extension.

Successful applications of the method are given using the $^{18}O(p,\alpha_0)^{15}N$ reaction and elastic scattering of $(^4He)^+$ ions for three types of samples. Scanning electron microscopy (SEM) is used as a complementary technique. Experimental details have been given [4]. The samples used for acquisition of charged particle spectra were: 1) S1 was obtained by high temperature oxidation of austenitic steel in $C\,^{18}O_2$ gas. Weight gain measurements had given a 4.2 μm thick oxide. A uniform concentration profile of ^{18}O was expected. SEM has shown a reasonably flat oxide (Fig. 1). 2) S2, a thick flat sample of sapphire (Al_2O_3). Uniform distributions of Al and O were expected in the sapphire substrate. 3) S3, a thick flat sample of zinc sulphide (ZnS). Uniform distributions of Zn and S were expected in the sample substrate. Spectral data were obtained from: 1) S1, using the $^{18}O(p,\alpha_0)^{15}N$ reaction at E_p=1.78 MeV, an energy slightly above the resonance energy at 1.766 MeV in the differential cross section, and $\Theta_L=165^o$. 2) S2, using a $(^4He)^+$ ion beam at E_α=1.5 MeV, $\Theta_L=165^o$. 3) S3, using a $(^4He)^+$ beam at E_α=3.1MeV, $\Theta_L=165^o$.

Published nuclear data, namely for reaction differential cross section and stopping power, were used in the computer predictions. Good fits to experimental data were obtained. For S1, a uniform concentration profile of ^{18}O was found with X_1=4.4 μm. This value is close to the expectation and higher than the determination made by the resonance method of analysis using the 1.766 MeV resonance, as the present method presents a higher depth resolution. Details of the fit are shown in Fig. 2. For S2, uniform concentration profiles were used with X_1 parameters of 0.53 and 0.23 μm for Al and O, respectively. Details of the fit are shown in Fig. 3. For S3, uniform concentration profiles were used with X_1 parameters of 2.5 and 1.5 μm for Zn and S, respectively. Details of the fit are shown in Fig. 4.

https://doi.org/10.1017/S1431927616000404 Published online by Cambridge University Press

The present work shows that the combined use of nuclear techniques and SEM microscopy is a highly powerful analytical tool for surface analysis of materials [8].

References:

[1] "Handbook of Modern Ion Beam Materials Analysis", ed. J.R. Tesmer, M. Nastasi (Materials Research Society, Pittsburgh, PA) 1995.
[2] G. Amsel, G. Battistig, Nucl. Instr. and Meth. B, **240** (2005) p. 1.
[3] J. M. Calvert *et al.*, J. Phys. D: Appl. Phys., **7** (1974) p. 940.
[4] J. A. R. Pacheco de Carvalho, A. D. Reis, Bol. Soc. Esp. Ceram., **47**, 4, (2008) p. 252.
[5] J. A. R. Pacheco de Carvalho *et al.*, Nucl. Instr. and Meth. B, **269**, 24, (2011) p. 3054.
[6] J. Pacheco de Carvalho *et al.*, Microsc. Microanal., **19**, S4, (2013) p. 133.
[7] N.P. Barradas *et al.*, Nucl. Instr. and Meth. B, **262**, (2007) p. 281.
[8] Supports from Universidade da Beira Interior and FCT (Fundação para a Ciência e Tecnologia) /PEst-OE-FIS/UI0524/2014 (Projecto Estratégico-UI524-2014) are acknowledged.

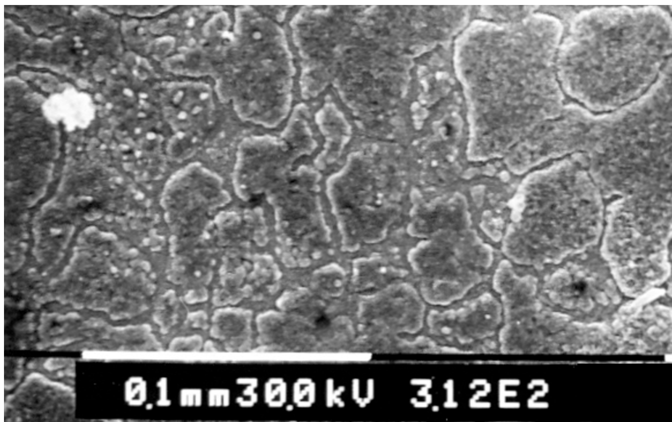

Figure 1. SEM image of the oxidized steel sample (S1).

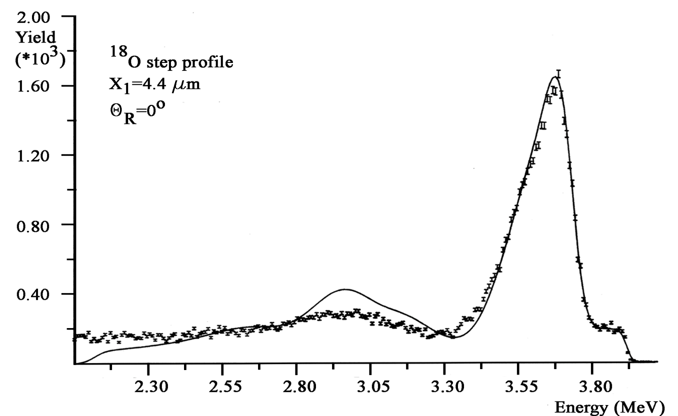

Figure 2. Computed fit to data of the $^{18}O(p,\alpha_0)^{15}N$ reaction from the oxidized steel target, (S1), for E_p=1.78 MeV, Θ_L=165°.

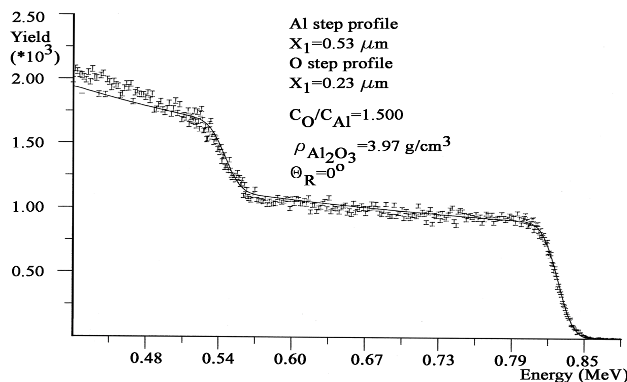

Figure 3. Computed fit to the elastic scattering data from the sapphire target, (S2), for E_α=1.5 MeV, Θ_L=165°.

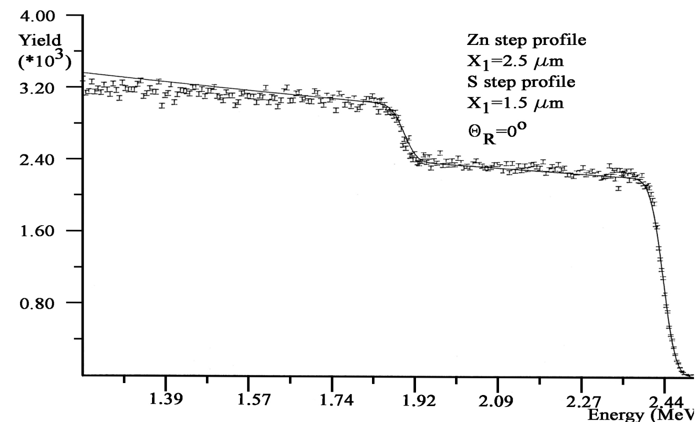

Figure 4. Computed fit to the elastic scattering data from the ZnS target, (S3), for E_α=3.1 MeV, Θ_L=165°.

https://doi.org/10.1017/S1431927616000404 Published online by Cambridge University Press