



# Porosity estimation of alumina samples based on resonant backscattering spectrometry



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## ARTICLE INFO

### Article history:

Received 23 November 2015

Received in revised form 29 February 2016

Accepted 8 March 2016

Available online 17 March 2016

### Keywords:

Porosity  
Porous alumina  
Structure  
Characterization  
Resonance  
Backscattering

## ABSTRACT

In this work, columnar porous alumina samples were investigated using the  $^{16}\text{O}(\alpha,\alpha)^{16}\text{O}$  resonance scattering at 3.045 MeV. If the incident energy is slightly above the resonance energy, a resonance peak appears in the energy spectra of the backscattered ions. The position and width of this peak for non-porous samples are mainly determined by the experimental setup, whilst for porous materials, the peak position shifts towards higher energies under certain conditions. This effect can be explained by the lower amount of material which the ions encounter along the backscattered trajectories. The energy shift of the resonance peak towards higher energies was revealed experimentally and discussed theoretically. The estimated porosities of the samples based on this energy shift were compared with those evaluated from the graphical analysis of the images obtained by field emission scanning electron microscopy.

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## 1. Introduction

In recent years, porous alumina samples have attracted great attention due to their potential applications in a wide range of fields, such as micro- and optoelectronics, bionanotechnology, chemical sensing, gas separation, catalysis, purifications, etc. [1,2]. The most common techniques for pore characterization include: mercury porosimetry [3], adsorption and BET analysis [4], Ellipsometric Porosimetry (EP) [5], Transmission Neutron and X-ray Scattering (TNS and TXS) [6], and visualization techniques such as Transmission Electron Microscopy (TEM), Scanning Tunneling Microscopy (STM) and Atomic Force Microscopy (AFM) [7].

Ion beam analysis (IBA) is a fast, non-destructive, and standard-less technique, which can be used for characterization of porous materials by means of collecting energy spectra of charged particles [8–13]. Since ions neither lose energy nor scatter in hollow cavities of porous materials, the backscattering spectrometry cannot be sensitive to gain information on the porous structure [14]. However, it has been shown that in the energy spectra of the backscattered particles, an extra “structure induced energy spread” appears due to fluctuating amount of material crossed by the individual ions on their way in and out. This extra “energy spread” which is easily observed in resonant backscattering measurements can be used to gain information on the structure and morphologi-

cal details of porous samples. In other words, any peak width of the resonant backscattering spectrum depends on the structure of the porous materials. Therefore, it is possible to characterize the pore-structure by performing resonant backscattering measurements [14–18].

In this study, samples of anodic porous alumina having a self-ordered honeycomb structure with cylindrical pores were analyzed. Anodizing of aluminum generates a porous alumina layer containing cylindrical parallel pores extending essentially perpendicular to the substrate [19]. In order to investigate porosity, the resonant elastic scattering peak of the  $^{16}\text{O}(\alpha,\alpha)^{16}\text{O}$  at the energy of 3.045 MeV was examined. The  $^4\text{He}^+$  backscattering spectra collected in porous and non-porous alumina samples under the same experimental conditions were compared.

It is well known that columnar structure and extra “structure induced energy spread” could affect the resonance backscattering spectra. Using this fact, the porosity of alumina samples was estimated based on resonant backscattering spectrometry. Finally, the results are compared to the surface porosities estimated from the images obtained by Field Emission Scanning Electron Microscopy (FESEM).

## 2. Theoretical approach

To approach the subject, we consider  $^4\text{He}^+$  beam irradiation of porous and non-porous alumina samples under the same

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experimental conditions. In this method, the narrow resonance ( $\sim 10$  keV) of  $\alpha$  scattering from  $^{16}\text{O}$  at 3.045 MeV is used [20]. In fact, the incident beam with an excess energy of  $\Delta E$  above the resonance energy ( $E_r$ ) crosses inward the sample, slows down to the energy  $E_r$  and then backscatters with high probability. The observed differences between the resonant peak in the spectra of porous and non-porous samples will be used to obtain information on the porosity of the porous sample. The following theoretical consideration can help to estimate the porosity of alumina samples based on the resonant backscattering spectrometry:

The ions irradiated porous sample with cylindrical pores, perpendicular to the sample surface. The individual ions, however, can cross various amount of material travelling in pores and pore walls to reach a given geometrical depth; or while they lose a well defined energy, as necessary in this resonance case, the ion will reach various geometric depths, as indicated in Fig. 1. In this case, the amount of crossed material will depend on the angle between the ion direction and pore direction.

Let us consider if the ions reach the sample in perpendicular incidence ( $\alpha = 0^\circ$ , parallel to the pores) and the ions impinging to the pore walls. In this case the ions will travel only pore wall in the inward path while they reach the resonance energy, therefore the resonance event take place at the same depth for porous and non-porous materials. They reach the resonance energy,  $E_r$  at the geometry depth  $X$ :

$$X = \frac{(E_0 - E_r) \cos \alpha}{S_1} \quad (1)$$

where  $E_0$  is the incident ion energy,  $\alpha$  is the incident angle,  $S_1$  is the stopping power of He in alumina at energy of  $E_0$ .

The detected energy for the porous sample,  $E_{dp}$  is:

$$E_{dp} = kE_r - \frac{XS_2(1-P)}{\cos \beta} \quad (2)$$

where  $k$  is the kinematic factor of scattering,  $\beta$  is the exit angle,  $S_2$  is the stopping power of He in alumina at energy of  $kE_r$  and  $P$  is the sample porosity (the volume fraction of the pores). In the latter equation, the homogeneous approximation is used, because the scattered ions cross several pores and walls in their outgoing way toward the detector placed to a scattering angle of  $165^\circ$ .

For the non-porous sample,  $P$  is equal to zero. Therefore, the detected energy for the non-porous sample,  $E_{dnon}$  is:

$$E_{dnon} = kE_r - \frac{XS_2}{\cos \beta} \quad (3)$$

In an ideal columnar porous sample, where the columns are thoroughly straight and parallel and the pore walls are smooth; if the beam is irradiated parallel to the columns, the ions in the pores will be travelling deeply without interactions, so they will not contribute to the resonance peak. Of course, the columns should be long enough, so the scattered ions at the end of the pores will not be able to reach the detector at the resonant peak energies. On the other hand, the incoming ions in the walls slow down to the resonance energy without crossing the pores. As a result, the depth ions travel and slow down to the resonance energy,  $X$ , for both porous and non-porous samples (in Eqs. (2) and (3)) are the same. By combining the Eqs. (2) and (3), the following expression can be easily derived:

$$P = \frac{E_{dp} - E_{dnon}}{kE_r - E_{dnon}} \quad (4)$$

Based on this equation, the porosity can be estimated from the difference between detected peak energies in porous and non-porous samples. Therefore, this theoretical approach can be followed for estimation of the porosity of a columnar porous sample when the incident ions remain in the walls in the inward path, reaching  $E_r$  at a well-defined depth, and passing several pores and walls at outgoing the material.

### 3. Experiments, results and discussions

In this work, to demonstrate our methodology, three of the investigated samples are illustrated including: two porous alumina films prepared by DC anodization of pure aluminum and a non-porous alumina sample.

The non-porous alumina sample was prepared by pulsed unipolar plasma electrolytic oxidation (PEO) in an electrolyte containing  $10 \text{ gL}^{-1}$   $\text{NaAlO}_2$  and  $1 \text{ gL}^{-1}$   $\text{KOH}$  in deionized water. A current density of  $0.1 \text{ A cm}^{-2}$  was applied for 20 min. The temperature of the bath was kept at  $30^\circ \text{C}$  during the electrooxidation. The frequency and duty cycle of the pulses were 50 Hz and 80%, respectively.

The low porosity anodized alumina (sample 1) was grown on high purity (99.999%) aluminum foil via a typical "mild anodization" process. First, a 0.3 mm-thick Al foil was degreased by multiple washing with acetone/ethanol. It was then electropolished in a stirring mixture of perchloric acid and ethanol (1:4) at  $0^\circ \text{C}$ . The electropolishing step was carried out at 20 V constant potential and was continued for 60 s until a clean shiny surface was obtained. Finally, the anodization was performed in a 0.3 M oxalic acid ( $\text{H}_2\text{C}_2\text{O}_4$ ) solution at 40 V for 10 h. The higher porosity sample (sample 2) was prepared by a two-step anodization process [21]. The aluminum foil was electropolished (as described above) prior to the first anodization which was performed in 0.3 M oxalic acid at 40 V and  $17^\circ \text{C}$  for 5 h. The oxide layer grown in the first step was stripped off in a solution containing 0.2 M  $\text{H}_2\text{Cr}_2\text{O}_4$  and 0.5 M  $\text{H}_3\text{PO}_4$  by mild heating at  $60^\circ \text{C}$  for 10 h. The second anodization was then performed under the same conditions as the first for 3 h. Finally, the sample was dipped into a 0.3 M phosphoric acid ( $\text{H}_3\text{PO}_4$ ) at  $30^\circ \text{C}$  for 40 min to widen the pores.

The porous aluminum oxide samples were studied by Elastic Backscattering Spectroscopy (EBS) technique using  $^4\text{He}^+$  ions accelerated by the 3 MV Van de Graaff accelerator of Nuclear Science and Technology Research Institute (NSTRI) in Tehran. All the three samples were irradiated by 3.195 MeV  $^4\text{He}^+$  ions at normal incidence with a beam current of about 10 nA under the same experimental conditions. The experimental chamber with an inner diameter of 440 mm and height of 300 mm was evacuated to the order of  $10^{-6}$  torr using the combination of a rotary backing pump

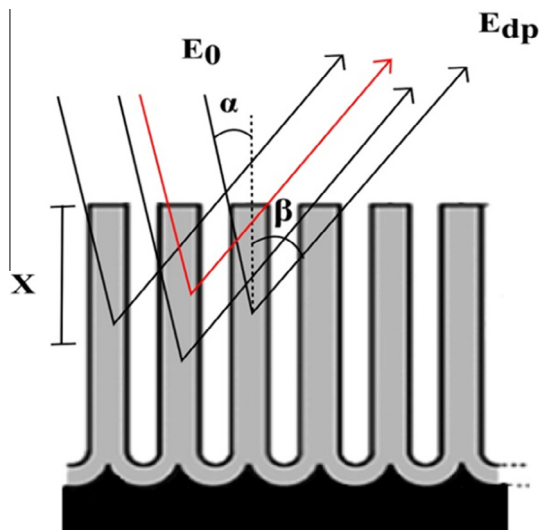


Fig. 1. A schematic representation of columnar porous material, with individual ions at incident angle  $\alpha$ , exit angle  $\beta$ , and the average resonance depth  $X$ .

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