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# Void and cavity determination in micro-PIXE analysis of composed material using binocular detectors: A computational study

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#### ABSTRACT

This study presents a new method to determine the location, size and depth of the pores in the material that is based on the asymmetry in the X-ray yield induced by proton micro-beam acquired in samples by a pair of X-ray detectors.

Most of the samples have cavities within their structures that may affect the quantitative elemental concentration. Although, there are several methods to measure the porosity that are based on a physical fact. Here, we demonstrate a study that is based on the lack of X-ray absorption induced by proton along the void region. In fact, we have different X-ray absorption along the sample which results in asymmetry X-ray yield in two spectrometers positioned at backward of the probing beam. The presented approach introduces an asymmetry factor of the X-ray intensity in each of the detector to obtain an image asymmetry map.

Our calculation was employed on silicon-based devices to estimate the size of the proposed cavity and the localized depth of the hole in composed material in micro-PIXE analysis. It was deduced that the presented approach is sensitive to the depth and the size of the hole in the composed material.

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#### 1. Introduction

The quantitative application of ion beam analysis methods is usually restricted to laterally homogeneous targets or smooth films. Although Ion Beam Analysis (IBA) techniques are not usually employed for the analysis of samples with totally unknown composition or unknown trace impurities. But, many material have finely featured patterns which one should take care about these features that may affect the interpretation of the results. Also, lateral inhomogeneities such as mixed phase layers, discontinuous layer, rough surface, rough interface, void and cavities may mislead to the RBS and PIXE experimental measurement. Somewhat in RBS measurement surprisingly the acquired spectrum within the cavity area behaves identical to the area outside of the cavity since the arriving beam penetrate thorough the whole thickness of the sample.

Electron microscope (SEM) and nuclear microprobe (NMP) have been used to acquire a detailed picture of the void structures in thin film systems [1]. In addition, X-ray u-CT to characterize the internal defect structure has been used for potential structural materials such as foams, composites, light metal alloys and imaging of voids in alloy steels. It has poor resolution to internal defects

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http://dx.doi.org/10.1016/j.nimb.2017.01.066 0168-583X/© 2017 Elsevier B.V. All rights reserved. due to high linear absorption coefficient [2]. However, some technical improvements have been recently carried out to get in-depth information with a few tens of micrometers in size [3,4]. Using simultaneously dual X-ray detector has benefits and gives a binocular view of the sample [5,6]. Occasionally, something goes wrong with one or other of the detection channels, so for every analysis it is a valuable analytical check on system integrity to have this binocular view.

Previously, using a pair of X-ray detectors we reconstructed the 3-D surface topography and the roughness of the surface based on the asymmetry in the X-ray absorption in micro-PIXE set-up [7]. After that, the method was developed to reconstruct the features on metal coins and complex curvature surfaces [8,9]. Here, we study the feasibility of using dual X-ray detector in μ-PIXE set-up to reconstruct the existence of voids or cavities in the composed material.

#### 2. Model

The ions beam are scanned over the sample and loss their energy in collisions with atoms and nucleus of the target. One possible of ion-atom interactions is emission of characteristic X-rays and they would be absorbed in the matter until they are detected. า

When the target has void in the scanned area combination of two different situations for ions and photons occurs.

- I. The bombarded ions are or not hit in the area of the void.
- II. The emitted photons may or not cross the void along their pass to detectors.

The ions are subject to no scattering or energy loss if they are directed in the void area and hence penetrate deeper than the other ions. At the other side, the photons that cross the void on their pass length experience less absorption and obviously have more intensity. A similar argument applies if a sample or film has isolated voids, uniformly porous or has abnormally low density.

To take into account these process in X-rays yield a schematic diagram of a void or cavity with radius of R was located at  $(x_0, y_0, z_0)$  position in the target (Fig. 1). Protons penetrate to the sample until they lose their energy. The energy loss data was taken from SRIM software [10] and the sample was divided to enough number of layer break in such a way that the energy loss is negligible in every layer. Produced X-rays by proton induced [11] may intersect the void at points A and B to reach the left detector mounted on angle  $\alpha$  with respect to the beam direction. Hence, the produced photons at depth d will travel distance  $Z_L$  in the matter until they are detected by the left detector. Indeed, the coordinates of A and B points are the intersection of the line across the detector and the circle with radius of R,

$$\begin{cases} (z - z_0)^2 + (x - x_0)^2 = R^2 \\ z = -x \cot \alpha + d \end{cases} \rightarrow$$

$$\begin{cases} x_A = \frac{(x_0 + (d - z_0) \cot \alpha) - \sqrt{R^2 \sec^2 \alpha - (x_0 \cot \alpha - (d - z_0))^2}}{\sec^2 \alpha} \\ x_B = \frac{(x_0 + (d - z_0) \cot \alpha) + \sqrt{R^2 \sec^2 \alpha - (x_0 \cot \alpha - (d - z_0))^2}}{\sec^2 \alpha} \end{cases}$$
(1)

and 
$$AB = \sqrt{(x_A - x_B)^2 + (z_A - z_B)^2}$$
. Hence,

$$Z_L = z/\cos\alpha - AB \tag{2}$$

*AB* equals to zero if photons not encounter the circle. The same procedure applies for the right detector.

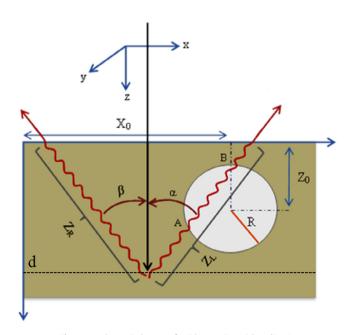


Fig. 1. A schematic layout of void or cavity with radius R.

The accumulated X-ray yield Y(z) at left detector in a given target matrix for element f is given by the expression [12]

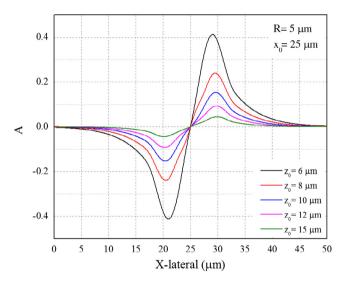
$$Y_{L}^{f} = \frac{N_{p}\varepsilon^{f}\Omega}{4\pi} \int c(z)\sigma^{f}(E)exp\{-\mu^{f}z_{L}\}dz \tag{3}$$

where Np is the incident ion flux,  $\varepsilon$  is the detector efficiency,  $\Omega$  is the solid angle subtended by the detector at the point irradiation, c(z) is trace element concentration,  $\sigma(E)$  is the X-ray production cross section by ions of energy E and  $\mu$  is the X-ray attenuation coefficient of the sample.

The asymmetry of X-rays yield detected by the left and right detectors appears as the following relation

$$A = (Y_L - Y_R)/(Y_L + Y_R)$$
 (4)

This ratio is influenced by no absorption of X-rays through the matter along the symmetric angles of two identical detectors.



**Fig. 2.** The asymmetry profile for various value of localized void depth positioned at  $z_0$  = 6, 8, 10, 12 and 15  $\mu$ m.

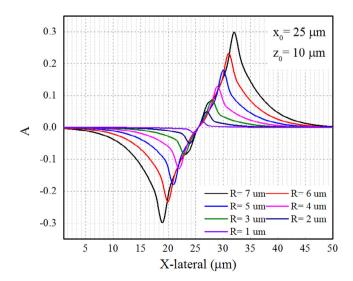


Fig. 3. The asymmetry profile for various void sizes of 7, 6, 5, 4, 3, 2 and 1  $\mu m$  in radius.

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