Proceedings of the 15th Czech and Slovak Conference on Magnetism, Košice, Slovakia, June 17–21 2013

# Sputtering and Implantation of VV-6025X Surface with Slow Heavy Ions Monitored with PIXE

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In this work the characteristic radiation, emitted during interaction of medium energy (200 keV) ambient heavy ions (Ar) with $Fe_4Co_{66}Si_{12}B_{14}Nb_1Mo_2Cu_1$ (VV-6025X) amorphous alloy, was measured in grazing incident-exit angle geometry and in time sequence, in order to determine dynamics of formation of subsurface region, damaged through implantation, sputtering and interface mixing. It was shown that structure and composition of surface is unstable against heavy ions irradiation due to preferential sputtering and implantation of ions, and recoils, and that the dynamics of such modification can be monitored *in-situ* with particle induced $X$-ray emission (PIXE) method.

DOI: 10.12693/APhysPolA.126.136

PACS: 34.35.+a, 61.80.Lj

## 1. Introduction

Impact of heavy ions (HI) on surface of multicomponent material results in implantation of beam ions, preferential sputtering of surface elements and selective implantation of recoils, thus changing the initial composition of the surface [1]. Extensive kinematic mixing of the surface and interfaces, combined with local thermal heating, caused by the energy deposited in the surface by the stopping HI [2], can be observed. These phenomena determine structural transformations of the materials and influence their electric and magnetic properties [3–5].

Particle Induced $X$-rays Emission (PIXE) is one of the methods which can give an insight into these processes [6–8]. It is based on the analysis of characteristic $X$-rays emitted during the impact of HI with the surface, which provide not only fundamental information on atomic excitation and further recombination processes, but can also give practical information on elemental composition and dynamics of restructuration of the films and subsurface region, measured at the time of irradiation [7–8].

Because PIXE is accompanied and superimposed by continuous radiation coming from accelerated electrons and by other recombination and emission processes, the grazing incidence-exit geometry was used in order to suppress bremsstrahlung and the response from the deeper regions.

We show how to use PIXE to determine the dynamics of composition change for multicomponent amorphous alloys, subjected to prolonged irradiation with ambient HI.

## 2. Experimental setup

The Ar ions beams were accelerated by 300 kV Cockroft-Walton accelerator of the Department of Solid State Physics to an energy of 230 keV. After leaving the acceleration tube, the beam was magnetically analyzed.

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Ion beam current density on the sample, measured by the Faraday cup, was about 0.5 $\mu$A/cm$^2$. 20 $\mu$m thick 6025X foils, after surface cleaning with Ar ion sputtering, were transferred through air to reaction chamber of $10^{-6}$ hPa, fixed on $LN_2$ cryostat and irradiated with Ar ions at various doses. Targets were mounted on a two axis goniometer in double alignment geometry: the incident grazing angle was fixed at at $\varphi_{in} < 5^\circ$ and the exit grazing angle was fixed at $\varphi_{out} < 5^\circ$, after preliminary measurements minimizing the signal from Si background. Ions RBS-scattered from the surface were registered at 90° off the ion beam direction by a Si(Li) detector, cooled down to *ca* -70 °C. PIXE $X$-rays spectra, emitted at 90° off ion beam direction, were measured with Amptek SDD spectrometer [9] (active area 25 mm$^2$, fwhm=120 eV@6.4 keV), placed behind a 25 $\mu$m kapton window and de-convoluted with XRF-FP and GuPIX [9, 10].

## 3. Results and discussion

Simulations of the experiment, performed with SRIM [11] showed for instance, that in the grazing incidence geometry, a 250 keV Ar ion can sputter about 30 Fe atoms (and the same amount of Au atoms). About 55% of incident Ar ions are scattered back above the Fe surface.

If an incident fluence of $10^{15}$ Ar/cm$^2$ at $\varphi_{in} = 5^\circ$ is assumed, a 3 nm thick layer will be sputtered from the Fe surface.

This should be compared with penetration depth of 40 nm and projected range of 100 nm of Ar ions in Fe. This means that in this geometry the measured radiation is emitted mainly from the excited target atoms, leaving the surface, and from the incident ions backscattered in an excited state. The remaining part of incident ions, travelling in surface layer and initializing cascades, excite atoms in the topmost thin films. Radiation from deeper layers and substrate should be fairly suppressed.

In order to apply PIXE to analysis of selective sputtering of multicomponent surface, subjected to pro-

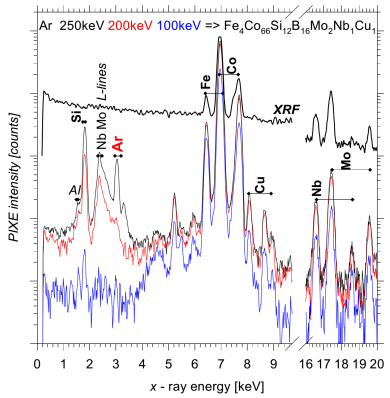


Fig. 1. PIXE spectra emitted from VV-6025X, irradiated with 250/200/100 keV Ar ions and XRF spectrum induced by 122 keV $\gamma$-radiation from $^{57}$Co. SDD spectrometer, resolution 120 eV/6.4 keV.

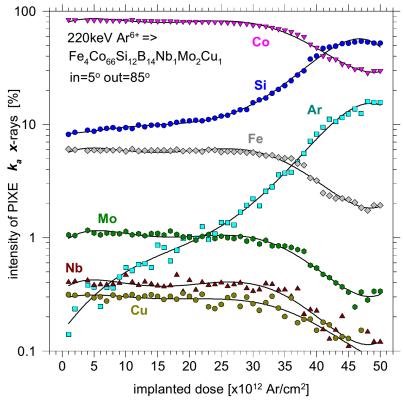


Fig. 2. Intensity of $k_\alpha$ $X$-rays from PIXE spectra, shown in Fig.(1), as functions of the implanted Ar dose.

longed irradiation with heavy ions, the characteristic $X$-ray spectra of elements contained in amorphous alloy $Fe_4Co_{66}Si_{12}B_{14}Nb_1Mo_2Cu_1$ were measured in time sequence. An example of raw spectra, induced by 100-250 keV Ar ions is shown in Fig. 1. $X$-ray lines are clearly shaped and the nominal initial composition can be confirmed with PIXE and XRF-FP package [9, 10]. Ghost lines are not marked in the figure. The corresponding penetration depth, at normal ion incidence, ranges from approximately 50 nm for 100 keV Ar to 100 nm for 200 keV Ar [11]. Normalization of the four spectra to Fe $k_\alpha$ intensity enables us to see, that spectral structure remains constant (except for Ar peak). It can be seen that the dependence on energy is more important. The yield is much higher for 250 keV ions, but the background is lower for 100 keV ions, particularly at low $X$-ray energy.

Intensities of $k_\alpha$ $X$-rays from the surface elements, as functions of the dose of incident 220 keV Ar ions, are shown in Fig. 2. Below $10^{13}$ Ar/cm$^2$ there is a region of slow change of the PIXE intensity with Ar dose, and a region of rapid variation above this limit. Weaker PIXE signal from Co and stronger signal from Si can be interpreted as a result of preferential sputtering, which causes deficiency and abundance in the corresponding elements. The remaining elements, Fe, Nb, Mo and Cu give the PIXE yield which initially weakly depends on the implanted dose, however it subsequently decreases at higher doses. Calculation with SRIM [11] confirms, that Cu, Fe and Co are sputtered at a higher rate, whereas Si, Mo, Nb and B are removed about twice less intensively. PIXE yield from Ar was increasing proportionally to implanted dose, and $5 \times 10^{13}$ Ar/cm$^2$ in Fig. 2 corresponds to 0.05% concentration of Ar within the penetration depth of 100 nm. Simultaneously, nearly 55% of incident Ar fluence is scattered out from the surface.

## 4. Conclusions

PIXE with MeV protons and helium ions is a reliable and well-established method of nondestructive elemental analysis of films and surfaces, supported by many codes which enable quantitative analysis without standards [9, 10].

PIXE produced by low energy heavy ions, despite of destructive effects, benefits from shallow penetration depth, larger $X$-ray production yield and better signal to background ratio. It can be shown that beam induced surface transformations such as: ion implantation, preferential sputtering and interface mixing can be quantitatively monitored with dose-dependent HI PIXE.

Such modification of surface and change of its elemental composition is essential for determining physical and chemical properties of the materials [12–14].

## References

[1] M. Toulemonde, C. Dufour, E. Paumier, *Phys. Rev. B* **46**, 14362 (1992).

[2] M. Antoszewska, M. Wasiak, T. Gwizdałła, P. Sovak, M. Moneta, *J. Therm. Anal Calorim.* **115**, 1381 (2014).

[3] G. Herzer, *IEEE Transactions Magn.* **26**, 1397 (1990).

[4] M. Moneta, B. Pawłowski, *Vacuum* **78**, 467 (2005).

[5] R. Brzozowski, M. Moneta, *Nucl. Instr. Meth. Phys. Res. B* **279**, 208 (2012).

[6] S.A.E. Johansson, J.L. Campbell, K.G. Malmqvist, *Particle Induced X-ray Emission spectrometry*, WILEY 1995.

[7] B. Pawłowski, M. Moneta, *Nucl. Instr. Meth. Phys. Res. B* **279**, 194 (2012).

[8] E.Z. Frštczak, J.E. Prieto, M. Moneta, *J. Alloys Com.* **586**, 375 (2014).

[9] http://www.amptek.com/.

[10] http://pixe.physics.uoguelph.ca/gupix/main/.

[11] J.F. Ziegler, J.P. Biersack, M.D. Ziegler, http://www.SRIM.org *The Stopping and Range of Ions in Solids* (2008).

[12] R. Brzozowski, M. Wasiak, H. Piekarski, P. Sovak, P. Uznaski, M. Moneta, *J. Alloys Comp.* **470**, 5 (2009).

[13] M. Moneta, R. Brzozowski, M. Wasiak, P. Uznański, *Nucl. Instr. Meth. Phys. Res. B* **267**, 411 (2009).

[14] M. Antoszewska, R. Brzozowski, J. Balcerski, K. Dolecki, E. Frątczak, B. Pawłowski, M. Moneta, *Nucl. Instr. Meth. Phys. Res. B* **310**, 27 (2013).