APPLICATION OF DOPPLER BROADENING SPECTROSCOPY FOR DETECTION OF VACANCY DEFECTS

V. Sabelová[1], M. Petriska[1], J. Veterníková[1], V. Slugeň[1], S. Sojak[1]

[1]Institute of Nuclear and Physical Engineering, Slovak University of Technology in Bratislava, Ilkovičova 3, 812 19 Bratislava, Slovakia

E-mail: veronika.sabelova@stuba.sk

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Abstract

Positron Annihilation Coincidence Doppler Broadening Spectroscopy (PA CDBS) has been used for detecting of structural defects in alloys with different content of chromium in “pure” and in implanted state. Defects were created and therefore microstructure of materials was changed by different implantations levels of He and H ions using linear accelerator at the Slovak University of Technology. Displacement Per Atom values profiles of materials were calculated using SRIM 2011.

Momentum distribution densities profiles of annihilation electron-positron pairs in studied materials were obtained using CDBS technique. Quantitative sensitivity of geometry of positron annihilation set-up was proved from results.

1. Introduction

The aim of this study is to prove specific behaviour of helium and hydrogen in implanted alloys. This improvement is possible to reach by comparison CDBS results of pure material and material with damages caused by ion implantation.

Doppler Broadening Spectroscopy (DBS)

DBS is non-destructive technique that can be used to characterize the material microstructure at the atomic scale. When the positron is injected into a sample, it quickly loses its energy in the thermalization process and consequently, having just a thermal energy, it diffuses through the lattice until annihilation.

When a positron annihilates with an electron, two photons are emitted with a total energy of $2m_0c^2=E_B$ with $m_0.c^2$ the electron rest mass energy and E_B the binding energy of the electron. Energy of one photon is increased by energy of $p_L.c/2$, thereby the energy of the other is decreased by the same amount, where p_L is the longitudinal component of the electron–positron momentum along the direction of the photon emission [1].

Coincidence Doppler Broadening Spectroscopy equipment which uses two-detector system was used for measurements. More precise selection of measured data with elimination of background is possible to reach with this apparatuses engagement [2].

Studied alloys

Detailed chemical composition of studied alloy can be seen in the Tab. 1. After casting, obtained ingots were cold worked under protective atmosphere to fabricate plates of 9 mm in thickness. Further, alloys were treated for 3 hours at 1050°C in high vacuum for austenitisation and stabilization. The duration of the heat treatment was chosen to get rid of any possible precipitation or phase transformation that might have been happening during hot rolling and also to allow a maximum degassing of the alloys. This treatment was then followed by air cooling to room temperature [4].

Thus obtained ferritic steels have been cut to desired dimensions and then carefully polished to mirror-like surfaces before exposure to helium and hydrogen implantation.

Tab. 1 *Chemical composition of studied Fe-Cr alloys (wt.%) [3].*

Alloy	Cr*	O*	N*	C*	Mn	P	Ni	Cu	V
L251	2,36	0,0346	0,0117	0,0081	0,009	0,013	0,044	0,005	0,001
L252	8,39	0,0667	0,0148	0,0211	0,03	0,012	0,07	0,01	0,002
L253	11,62	0,0307	0,0237	0,0278	0,03	0,05	0,09	0,01	0,002

* measured after heat treatment

The samples were implanted by hydrogen and helium ions with using linear accelerator at Slovak University of Technology. Simulations of radiation damage and vacancy defects were created by this implantation. Set of experimental samples with the implanted ion charges is listed in the Tab. 2. Temperature during implantation was under 100°C.

Tab. 2 *List of implanted materials by hydrogen and helium ions; energy of implantation (keV); electric charge surface density of implantation (C/cm^2).*

	Hydrogen	Hydrogen	Helium	Helium
Alloy	0,2 C/cm^2	0,4 C/cm^2	0,2 C/cm^2	0,4 C/cm^2
L251 2,36 wt.%Cr	100 keV	100 keV		
L252 8,39 wt.%Cr	250 keV	250 keV	250 keV	250 keV
L253 11,62 wt.%Cr			250 keV	250 keV

Hydrogen can cause defect formation in the same way as helium. It diffuses into vacancies and dislocations. These defects are mobile in comparison with defects containing helium, which guarantees lower internal stress. As the diameter of hydrogen nuclei (proton) is smaller than alpha particle, the created displacement damage is lower (about 10 times) [4].

SRIM 2011 calculations

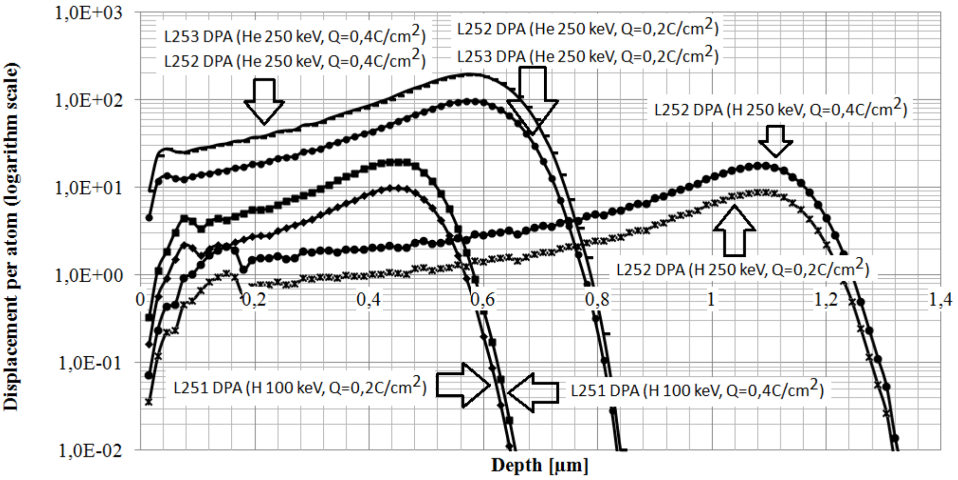

Fig. 1 *Calculated DPA of implanted samples L251 (2,36 wt.%Cr), L252 (8,39 wt.%Cr), L253 (11,62 wt.%Cr).*

Simulations of vacancies creation in alloys were realized during SRIM (The Stopping and Range of Ions in Solids) [5] detailed calculation using hydrogen and helium ions.

Fig. 1 shows DPA (Displacements Per Atom) of all implanted materials. Significant differences between hydrogen and helium implantation charge surface density and between different implantation energy can be seen there. DPA of material with He implantation is more than 10 times higher than DPA value of material with H implantation with same electric charge surface density value. It depends mostly of size of He and H ions. Values of DPA are almost the same for alloys L252 and L253 with same electric charge surface density of helium implantation.

2. Experimental measurements

Coincidence Doppler Broadening Spectroscopy measurements were realized using two HPGe detectors with resolution 1,9keV at 1,33MeV.

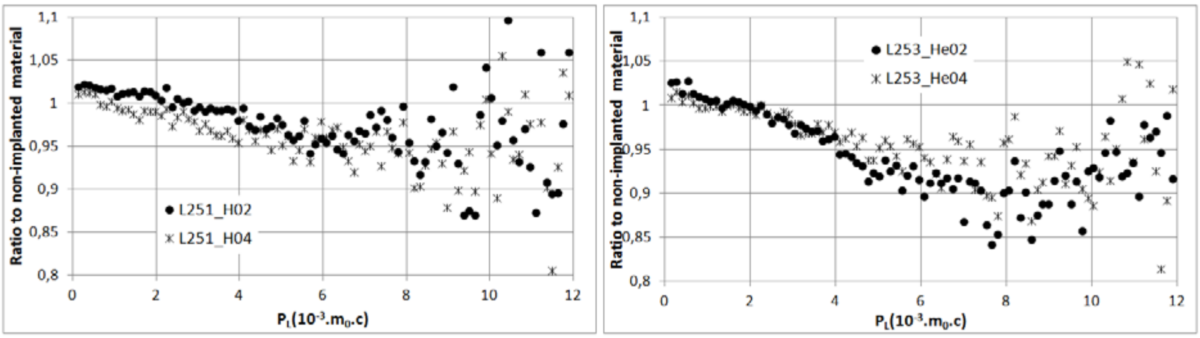

Fig. 2 *1D momentum density distributions, LEFT - L251 (2,36 wt.%Cr), H (100 keV) implanted materials (0,2 C/cm^2; 0,4 C/cm^2); RIGHT - L253 (11,62 wt.%Cr), He (250 keV) implanted materials (0,2 C/cm^2; 0,4 C/cm^2).*

CDB technique measures 3D momentum density of electron-positron pair annihilation; 2D momentum density distribution is obtained from it. Then the 1D projection of momentum density distribution is profiled. It describes longitudinal momentum component of the annihilation photon along the photon-emission direction [6].

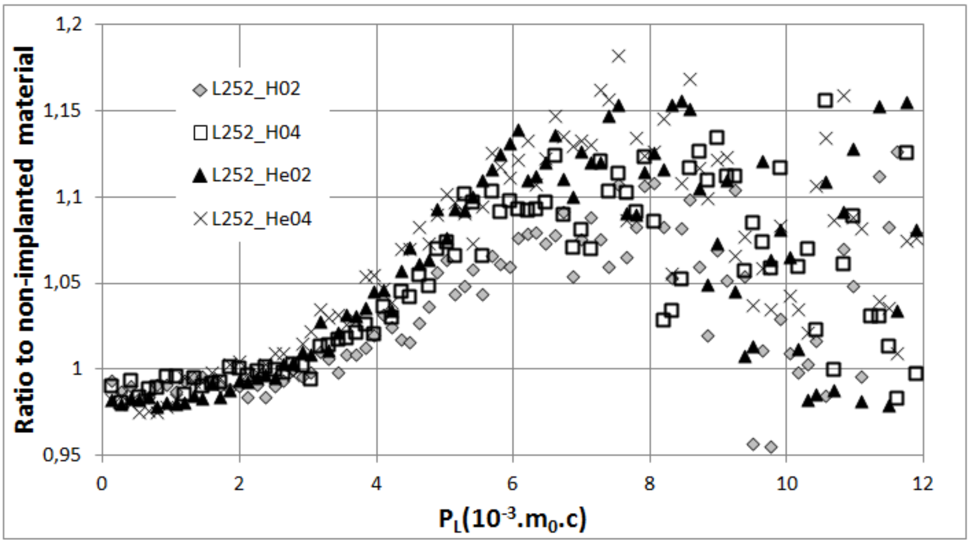

Fig. 3 *1D momentum density distributions, L252 (8,39 wt.%Cr), H and He (250 keV) implanted materials (0,2 C/cm^2; 0,4 C/cm^2).*

1D-profiles of momentum distributions of electron-positron pair annihilations are shown in Fig. 2 and Fig. 3. Annihilations with valence electrons cause lower shift of longitudinal momentum $P_L(10^{-3}.m_0.c)$ values. Annihilations with core electrons characterize wings of spectra; create higher values of PL with significant dispersion.

3. Discussion and conclusion

Experimental results show an analogy to theoretical calculations by SRIM 2011. It was reached higher microstructural damage associated with the formation of vacancies and vacancy clusters with higher electric charge surface density of implantation. That fact was confirmed by CDBS, but in some cases it is important include specific properties of implanted elements. In materials occurs an effect of filling vacancy type of defects by helium and hydrogen atoms, changing chemical environment of microstructure and annihilation with core electrons of present elements. It depends mostly of size of He and H ions, how it was written above, which influences diffusion in material.

Differences of caused damage are obvious by comparison from diversion of ratio value “1”. Different behaviour of hydrogen and helium implantation is shown in Fig. 2 and Fig.3. Hydrogen, as helium, creates open volume defects, but its diffusion in material is larger. Annihilation with core electrons and therefore changing of microstructural environment is not so significant, as in the case of helium. Creation of higher damage is caused by 10 times higher size He ions than H ions and also with higher electric charge surface density of implantation value. But the results are affected due to filling vacancy type of defects (more by helium atoms), changing chemical environment of microstructure and diffusion of present elements.

Acknowledgement

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