

# ANALYSIS OF RADIATION DAMAGED NANOCRYSTALS

*<sup>1</sup>Sitek Jozef, <sup>1</sup>Dekan Július, <sup>1</sup>Sedlačková Katarína, <sup>1,2</sup>Šagátová Andrea*

*<sup>1</sup> Institute of Nuclear and Physical Engineering, Faculty of Electrical Engineering and Information Technology, Slovak University of Technology, Slovakia*

*<sup>1,2</sup>UCEA, Slovak Medical University, Ku Kyselke 497, 91106 Trenčín,*

*E-mail:jozef.sitek@stuba.sk*

*Received 06 May 2014; accepted 17 May 2014*

## 1. Introduction

In the recent years was shown [1], [2] that some physical properties of nanocrystalline materials are more or less affected by neutron irradiation. Neutron radiation damage produces defects that may cause a realignment of magnetic domains implying a reorientation of magnetic moments. Change in the local neighbourhoods of atoms affect the average value of internal hyperfine magnetic field as well as the shape of hyperfine field distributions. Similar structure changes are created after charge particle irradiation.

In the case of nanocrystalline alloys, which consists of crystalline nanograins embedded in an amorphous intergranular matrix [3], irradiation by neutrons and charges particles will lead to redistribution of atoms in the amorphous matrix, disturbance of regular atomic ordering of the crystal lattice and atom exchange between the amorphous and crystalline component. The mechanism of the radiation damage also dependent on the constituent elements and their concentration.

Using Mössbauer spectroscopy the interest is oriented on changes in the orientation of the net magnetic moment, in the value of the magnetic hyperfine field as well as the volumetric fraction of the crystalline and the amorphous component of irradiated samples.

## 2. Experimental Details

Ribbon-shaped specimens of the master alloy were prepared by planar flow casting. The ribbons with nominal composition of  $(\text{Fe}_{1-x}\text{Ni}_x)_{81}\text{Nb}_7\text{B}_{12}$  ( $x = 0, 0.25, 0.5, 0.75$ ) were about 25  $\mu\text{m}$  thick and 10 mm wide. To achieve nanocrystalline state, the amorphous ribbons were annealed in vacuum at the temperature of 550 °C for 1 hour. Samples were irradiated by neutrons in nuclear reactor with fluence of  $10^{16}$  n/cm<sup>2</sup> and  $10^{17}$  n/cm<sup>2</sup>. and by electrons in linear accelerator with dose 1MGy at the Slovak Medical University.

Mössbauer spectra were collected in transmission geometry by a conventional constant-acceleration spectrometer with a <sup>57</sup>Co(Rh) source. All spectra were measured at room temperature and evaluated by the CONFIT program [4], which allows simultaneous treatment of crystalline and residual amorphous phase by means of individual lines and distribution of hyperfine components.

## 3. Results and Discussion

We observed from Mössbauer spectra of all nanocrystalline samples, one example is shown in Fig.1, that after neutron irradiation, changes in the orientation of net magnetic moment, in the value of the average hyperfine magnetic field of amorphouse and crystalline

components and also volumetric fraction take place as is shown in Tab.1. Orientation of net magnetic moment is reflect in relatively intensity of second and fifth line of Mössbauer spectrum ( $A_{23}$ ). This parameter achieve maximum value if the direction of net magnetic moment lay in sample surface and minimum value, if it is perpendicular on the sample surface. Change in the volumetric fraction of the amorphous component can indicate a movement of Fe atoms from the amorphous matrix to the crystalline part, contributing to slight decrease of the internal magnetic field after irradiation. We suppose that free Fe atoms originate either from the neutron capture by boron or from recoil atoms displacement. We cannot exclude that part of crystalline iron got damaged, meaning that it contains vacancies and interstitial atoms.

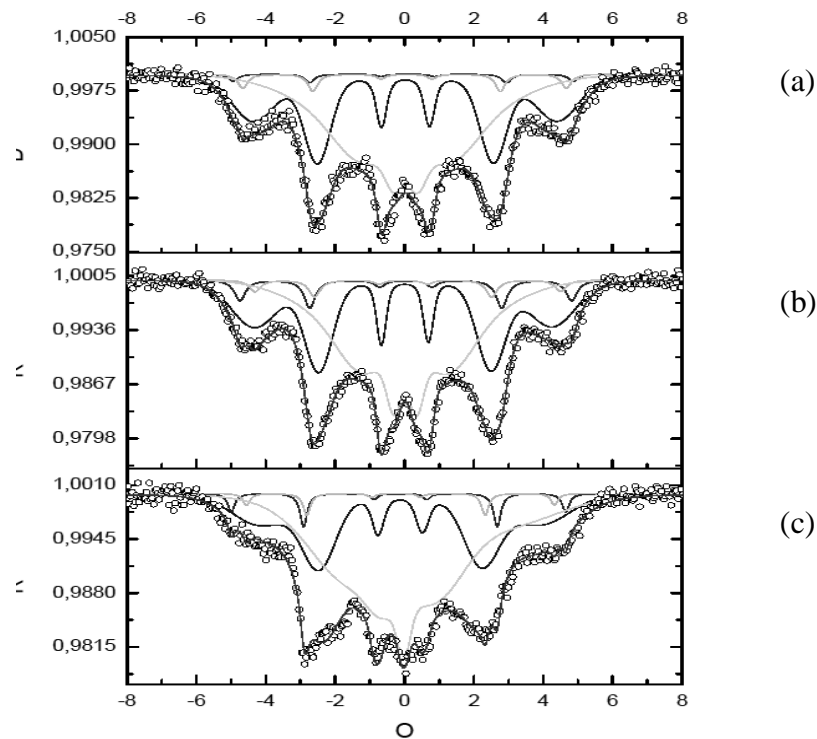


Fig. 1: Mössbauer spectra of the nanocrystalline  $(Fe_{0.5}Ni_{0.5})_{81}Nb_7B_{12}$  alloy: Non-irradiated sample (a), sample irradiated with the neutron fluence of  $1 \times 10^{16}$  n/cm<sup>2</sup> (b), sample irradiated with the neutron fluence of  $1 \times 10^{17}$  n/cm<sup>2</sup> (c).

Mössbauer spectra of irradiated samples by electrons of dose 1 MGy (Fig.2) exhibit similar changes of parameters as the sample irradiated by neutrons as is shown in Tab.1. In both cases, the most sensitive parameter is direction of net magnetic moment  $A_{23}$ . Changes in volume fraction and in internal magnetic field are in the frame of the errors.

Tab. 1. Parameters of Mössbauer spectra.  $A_{23am}$  –parameter of amorphous component,  $B_{am}$  – average value of magnetic induction of internal magnetic field of amorphous component,  $A_{am}$  – volumetric fraction of amorphous component:  $A_{23cr}$  parameter of crystalline component,  $B_{cr}$  - magnetic induction of internal magnetic field of crystalline component,  $A_{cr}$  - volumetric fraction of crystalline component

Specimen	Parameters	non-irr	$10^{16}$ n/cm <sup>2</sup>	$10^{17}$ n/cm <sup>2</sup>	1 MGy el.
$(Fe_{0.5}Ni_{0.5})_{81}Nb_7B_{12}$	$A_{23m}$	1.28	3.60	2.72	3.7
	$B_{am}$ (T)	27	26	25	27
	$A_{am}$ (%)	94	90	93	95
	$A_{23cr}$	4.00	4.00	4.00	3.9
	$B_{cr}$ (T)	30	29	29	31
	$A_{cr}$ (%)	6	10	7	5

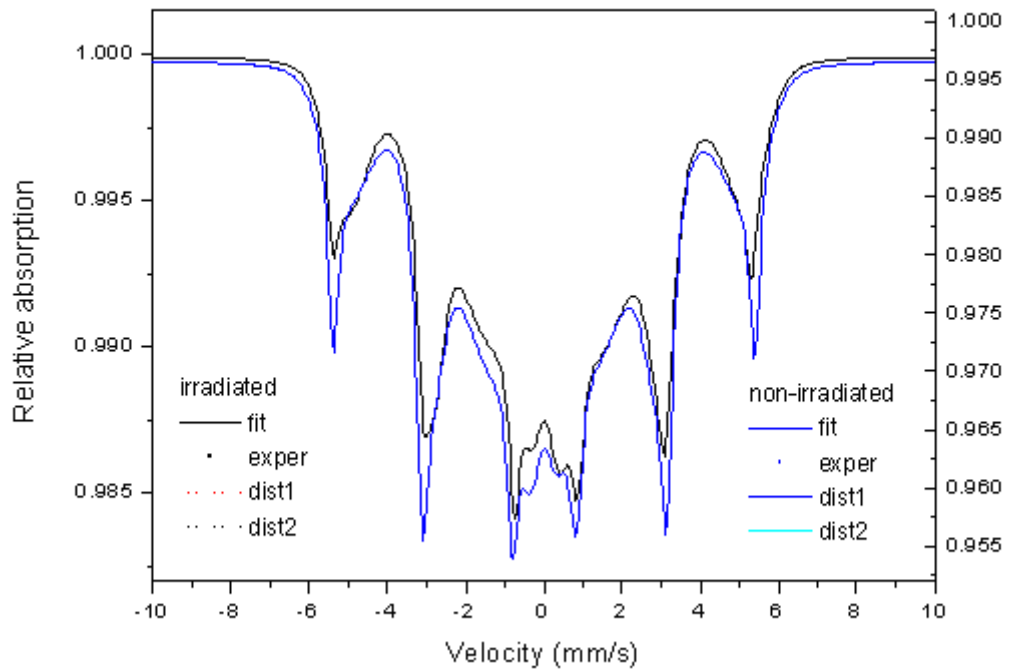


Fig. 2: Mössbauer spectra of the nanocrystalline  $(Fe_{0.5}Ni_{0.5})_{81}Nb_7B_{12}$  alloy: Non-irradiated and irradiated by electrons of dose 1 MGy,

#### 4. Conclusion

After summarizing all obtained results, the fluence  $10^{16}$  n/cm<sup>2</sup> is still not sufficiently high significantly damage amorphous and crystalline structure. This fluence more or less modify the structure than damage. After fluence  $10^{17}$ n/cm<sup>2</sup> we observed beginning of the. structural damage. Our results show, that high electron dose also modify the structure of nanocrystalline

alloys. In further study of this alloy it would be necessary to find the limit of electron dose under that the alloy is resistant against electron's damage. From point of view Mössbauer spectroscopy the most sensitive parameter is direction of net magnetic moment.

### Acknowledgement

This work was financially supported by grant of Scientific Grant Agency of the Ministry of Education of Slovak Republic and the Slovak Academy of Sciences No. VEGA-1/0286/12.

### References:

- [1] F. P. Schimansky, R. Gerling, and R. Wagner, "Irradiation-induced defects in amorphous  $\text{Fe}_{40}\text{Ni}_{40}\text{P}_{20}$ ", *Mater. Sci. Eng.*, vol. 97, pp. 173-176, 1988.
- [2] J. Degmová, J. Sitek, and J. M. Grenèche, "Neutron Irradiation Effect on Fe-Based Alloys Studied by Mössbauer Spectrometry", in *Properties and Applications of Nanocrystalline Alloys from Amorphous Precursors*, B. Idzikowski, P. Švec, M. Miglierini, Eds., Kluwer Academic Publishers, Dordrecht, 2005, pp. 219-228H..
- [3] P. Švec, M. Miglierini, J. Dekan, J. Turčanová, G. Vlasák, I. Škorvánek, D. Janičkovič, and P. Švec, Sr., "Influence of Structure Evolution on Magnetic Properties of Fe-Ni-Nb-B System", *IEEE Trans. Magn.*, vol. 46, pp. 412-415, 2010.
- [4] T. Žák and Y. Jirásková, "CONFIT: Mössbauer spectra fitting program", *Surf. Interface Anal.*, 38, 710-714, 2006