NANOCRYSTALLINE ALLOYS AFTER Cu IONS IMPLANTATION

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1. Introduction

In the recent years, nanocrystalline alloys have become attractive for many applications. The most prominent FINEMET, NANOPERM and HITPERM-type alloys have been frequently investigated because they exhibit excellent soft magnetic properties. In recent time a new alloy NANOMET is in the centre of interest.

The crystallization process of the amorphous precursor is known to be controlled by thermal annealing. It has been already shown that nanocrystalline structure could be more or less realized or affected by external factors [1,2] such a neutron and electron irradiation, applied magnetic field [3,4] and ion implantation [5].

Changes in the orientation of the average magnetic moment were observed by Mössbauer spectroscopy in different particle irradiated metallic glasses and nanocrystals [6]. The particle bombardment produces defects that may cause a realignment of magnetic domains implying a reorientation of the magnetic moments. Changes in the local neighbourhoods of the atoms affect the average hyperfine magnetic field as well as the shape of the hyperfine field distributions. In the case of nanocrystalline alloys, which consist of crystalline nanograins embedded in an amorphous intergranular matrix, irradiation by 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. Such a mechanism of particle interaction with nanocrystalline alloys was experimentally demonstrated by the radiation damage of these alloys by neutrons [6].

Effect of electron irradiation on the crystallization of NANOPERM alloys also was examinated [4].In low B concentration of Fe- Zr- B alloy induced crystallization was observed by irradiation but in high B concentration alloy formation of nanocrystalline structure was not realized. Ribbons of amorphous $Fe_{74}Cu_1Nb_3Si_{16}B_6$ alloy were irradiated by 593 MeV Au ions to characterize the irradiation-induced structural changes. Visible deviations in the Mössbauer spectra acquired at room temperature can be observed even in transmission geometry [7]. Results at amorphous and nanocrystalline (Fe_{1-x}Ni_x)8₁Nb₇B₁₂ (x = 0, 0.25, 0.5, 0.75) alloys indicated that weak external magnetic field has influence on the orientation of the net magnetic moment which is reflected in parameters of Mössbauer spectra [8]. Last study of the same alloy after Cu ions implantation indicated at the possible modification these alloys due to heavy ions implantation [5].

2. Experimental

The samples for Mössbauer experiment were studied in form of ribbon produced by planar flow casting of the melt at the Physical Institute SAS. Nanocrystalline samples were prepared by thermal annealing by 400 °C during 0.5 hour. Thickness of the samples was a 25

microns with composition of $(Fe_{64}Co_{21}B_{15})_{95}P_4$ and $(Fe_{64}Co_{21}B_{15})_{95}P_4Cu_1$. Irradiation with Cu ions was perform in accelerator at Material-technological faculty STU. The Mössbauer spectra were measured at room temperature using a Wissel Mössbauer spectrometer with the $Co^{57}(Rh)$ source in transmission geometry. The hyperfine parameters of the spectra including relative component area (Arel), isomer shift (IS), quadrupole splitting (QS), as well as internal hyperfine magnetic field (B_{hf}). Spectra were evaluated by CONFIT program [9] allowing simultaneous treatment of crystalline components and residual amorphous phases using individual lines and distributions of hyperfine parameters.

3. Results and discussion

Mössbauer spectra of all samples were evaluated preferably using a model comprising two distributions of sextets representing the amorphous rest and two sextets describing the crystalline phase. After irradiation, changes in the orientation of the net magnetic moment, in the value of the average hyperfine magnetic field of the amorphous and crystalline components and of their volumetric fraction took place. Orientation of the net magnetic moment is reflect by the ratio of the second and fifth line intensities of a Mössbauer spectrum (A₂₃). This parameter achieve its maximum value if the direction of the net magnetic moment lay in sample plane and its minimum value, when oriented perpendicular to the sample plane. According to the previous measurements, significant structural damage manifests also via changes of other Mössbauer parameters on the radiation damaged nanocrystalline alloys [1,4]. Changes in the volumetric fraction of the constituent phases were found (i.e. a part of the crystalline component was amorphised and amorphous component was also partly damaged) after neutron irradiation. If the structure is only modified by irradiation, the values of the internal magnetic fields and the relative amounts of the amorphous and crystalline components relatively changes usually very low. The most sensitive is A₂₃ parameter and shape of distribution of internal magnetic field P(B) of amorphous component.

Mössbauer spectrum of amorphous $Fe_{64}Co_{21}B_{15})_{95}P_4$ is shown in Fig.1 and parameters are given in Tab.1. Sample consists of two components with relative amount of approximately 50 %. This sample was heated after short time (a few second) increasing temperature at 400 °C during 30 minuts. After heating the low field component increased at 66 % and high field component decreased at 34 %. Then, Cu ions were implanted into $Fe_{64}Co_{21}B_{15})_{95}P_4$ alloy which caused qualitative change of the spectra. By side amorphous component a new crystalline component was created. Mössbauer spectrum of this alloy is given on Fig.2.



Fig.1. Mössbauer spectra of amorphous $Fe_{64}Co_{21}B_{15})_{95}P_4$ alloy

All spectra of this alloy were compared with spectra of the alloy containing Cu. Addition of Cu change morphology of the structure as was shown in detail in [10] and is reflected in A_{23} parameter and internal magnetic field as is sown in Tab.1.We observed even in amorphous state different relative amount of high and low field component in the alloy containing Cu.

Table 1. Parameters of Mössbauer spectra: A_{23} -direction of net magnetic moment, **B**- magnetic induction of internal magnetic field, **A**-relative amount of the components; amorphous high – amorphous high magnetic component; amorphous low - amorphous low magnetic component; crystalline-crystalline component

sample	component	A ₂₃	В	Α	
			(T)	(%)	
$\begin{array}{c} Fe_{64}Co_{21}B_{15})_{95}P_4\\ amorphous\end{array}$	Amorphous high	2.47	27.95	49	
	amorphous low	3.76	25.31	51	
Fe ₆₄ Co ₂₁ B ₁₅) ₉₅ P ₄ short time incr.temp.	amorphous high	2.42	28.57	34	
	amorphous low	3.62	26.47	66	
$Fe_{64}Co_{21}B_{15})_{95}P_4$ nanocrystalline, Cu impl.	amorphous high	2.05	26.65	29	
	amorphous low	3.40	25.04	51	
	crystalline 1	2.82	36.90	14	
	crystalline 2	2.78	35.34	6	
$\begin{array}{c} Fe_{64}Co_{21}B_{15})_{95}P_4Cu_1\\ amorphous\end{array}$	amorphous high	2.14	28.06	36	
	amorphous low	3.71	26.03	64	
Fe ₆₄ Co ₂₁ B ₁₅) ₉₅ P ₄ Cu ₁ nanocrystalline,	amorphous high	2.51	27,95	32	
	amorphous low	3.20	25.84	53	
	crystalline 1	3.52	36.70	12	
	crystalline 2	2.00	35.30	3	



Fig.2. Mössbauer spectra of $Fe_{64}Co_{21}B_{15})_{95}P_4$ alloy after Cu implantation

Results of amorphous $Fe_{64}Co_{21}B_{15})_{95}P_4Cu_1$ measurements show that its parameters are closed to the results of sample without Cu after short increasing heating temperature as is shown in Tab.1. Sample $Fe_{64}Co_{21}B_{15})_{95}P_4Cu_1$ was undertaken to long time increasing (a few minutes)

temperature heating at 400 $^{\circ}$ C for 30 minutes and as a results amorphous and crystalline components were created. Parameter of such a prepared sample are very similar to parameters of implanted samples. It means that for alloy without Cu after short time increasing heating temperature and following Cu implantation we can prepare identical sample as by long increasing time heating temperature of alloy containing Cu.



Fig 3. Mössbauer spectra of nanocrystalline $Fe_{64}Co_{21}B_{15})_{95}P_4Cu_1$ alloy

Conclusion

Our results show that nanocrystalline alloy could be prepare not only by heating from amorphous precursor but also by implantation of ions and probably some kinds of alloys could be modified by irradiation.

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