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Structure and mechanical properties of amorphous $Fe_{84}Nb_7B_9$ alloy during crystallisation

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Materials

ABSTRACT

Purpose: The investigation results of crystallization, in temperature range of 300-700°C in time 1 h, of amorphous $Fe_{84}Nb_7B_9$ alloy. The alloy was obtained in tape form by planar-flow-casting method.
Design/methodology/approach: The structure was characterized by scanning electron microscope (SEM) and X-ray diffraction (XRD) method. The measurement of mechanical properties, like: tensile strength R_m, ductility E and cracking energy E_p, were made. Tensile tests were performed on testing machine INSTRON 1195. Plastic properties of investigated tapes were determined in bending test. Unit cracking energy E_p was evaluated by carrying out the tear test by the use of tensile testing machine INSTRON 1195.
Findings: The changes of mechanical properties and fracture morphology being connected with the structure changes involved by crystallization process have been stated.
Practical implications: The relationship between heat treatments parameters and mechanical properties can be useful for practical application of these alloys.
Originality/value: It has been stated that heat treatment leads to crystallization of two-stage character.
Keywords: Amorphous materials; Mechanical properties; Heat treatment; X-ray diffraction method

1. Introduction

Nanocrystalline materials characterized by very good soft magnetic properties are obtaining in process of controlled crystallization of metallic glasses which have specific chemical composition. Typical alloy used for production of nanocrystalline material is FINEMET, the alloy of chemical composition $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$. This alloy thanks to two-phase structure composed with amorphous matrix and nanocrystals of α-Fe(Si), obtained by heat treatment process of amorphous tapes, has excellent magnetic properties characterized by low coercive field and high permeability. Successful results of investigation with $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$ alloy were an inspiration for further attempts at searching another materials of different chemical composition and better properties [1÷4]. As a result the group of nanocrystalline soft magnetic alloys type Fe-M-B, where M=Zr, Hf, Nb, has been worked out. It has been found that the Fe-M-B alloys consisting of ultrafine bcc α-Fe grains dispersed in an amorphous matrix exhibit much better soft magnetic properties than nanocrystalline $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$ [2,3]. The group of these alloys is called NANOPERM™. This type of alloys have attracted a great deal of attention over the past 10 years. From a viewpoint of industrial application they are very attractive materials especially because of the highest B_S among the nanocrystalline alloys [2-6].

Nanocrystalline materials are obtained on the way of controlled crystallization of metallic alloys involved by heat treatment process. Crystallization of amorphous alloys is the complex phenomenon, depended on chemical composition of alloy and parameters of heat treatment process. Heating can be realized by conventional or impulse methods [7÷9]. The method most often used is isothermal heating at temperature near crystallization temperature T_x in constant time, usually by 1 hour [1÷4,6,10÷13]. Depending on the chemical composition alloy, transition from metastable amorphous structure to crystalline state (equilibrium or metastable) can proceeds in one stage (polymorphic or eutectic crystallization) or as a multistage process (primary crystallization) [14]. Crystallization process results on changes as well physical as mechanical properties [8,12,13,15]. Technical application of nanocrystalline alloys is depended on versatile recognition of their all properties.

However, many works were dedicated to the results of physical properties investigations, there were no published data referring to mechanical properties of these alloys. In particular interesting is the relationship between structure and mechanical properties, also described by changes of fracture morphology, which can be observed during crystallization process.

Investigations of influence of amorphous $Fe_{84}Nb_7B_9$ alloy crystallization on the changes of structure and mechanical properties, especially on tensile strength R_m, ductility ε and cracking energy E_p, determined in tear test, has been undertaken in presented paper. The observed changes of fracture morphology of tapes after decohesion in tensile test which were proceeded with the increase of annealing temperature, have been shown too.

2. Experiments

Experiments were performed on tapes of amorphous $Fe_{84}Nb_7B_9$ alloy obtained by planar flow casting method. Tapes had 0.037 mm of thickness and 5.15 mm of width. Sections of tapes of 120 mm length were annealed in temperature range of 300 to 700°C with gradation every 50°C, with annealing time equal 1 hour. The observed changes of structure and properties were compared with as quenched alloy.

Tapes in as-quenched state have been heat treated in electric chamber furnace Thermolyne type F6020C of working temperature range 100-975°C and with protective argon atmosphere. Tensile tests were performed on testing machine INSTRON 1195 with tension rate 5 mm/min. Plastic properties of investigated tapes were determined in bending test which is the method generally used for amorphous tapes. Tapes were bended of 180° angle, and then the value of ε was determined by used Eq 1:

$$\varepsilon=2g/h, \qquad (1)$$

where: g - sample thickness; h - distance between micrometer jaws in the moment of appearing the fracture.

Unit cracking energy E_p was evaluated by carrying out the tear test by the use of tensile testing machine INSTRON 1195. Registration of force during tear test permits on evaluation the unit cracking energy E_p on the basis of the relationship (2):

$$E_p=E/l\cdot g, \qquad (2)$$

where: E=2Fl - cracking energy; F – tear force; g - tape thickness; l - length of cracking.

There were prepared 10 samples on each state for carrying out the mechanical properties investigations. The results have been worked out with the use of statistic methods.

The changes of tapes structures involved by heat treatment have been investigated by X-ray method. X-ray diffraction phase analysis has been performed by the use of diffractometer DRON 2.0 with filtered Co-Kα radiation.

The morphology of fracture surfaces after decohesion in tensile tests was observed in scanning electron microscope OPTON DSM 940.

3. Results and discussion

The investigated $Fe_{84}Nb_7B_9$ alloy has amorphous structure in as quenched state - Fig. 1a, Table 1. Tensile strength R_m is 732 MPa, unit cracking energy E_p has value 9.8 $J\cdot cm^{-2}$ and plasticity of alloy is high, ε=1 (Table 1). Investigations of fractures after decohesion in tensile test confirmed high plasticity of tapes and revealed their ductile character with vein pattern morphology, typical for amorphous alloys (Fig. 2).

Annealing of the alloy at temperature 300°C for 1 h didn't involve the changes of structure and plastic properties of the samples in compare to as quenched state. However, increase of tensile strength R_m up to value 823 MPa, and unit cracking energy E_p up to value 10 $J\cdot cm^{-2}$ (Table 1) has been observed. So it can be stated that after annealing at temperature 300°C $Fe_{84}Nb_7B_9$ alloy reaches better tensile properties then in as quenched state, and plastic properties are still unchanged (ε=1, Table 1).

Increase of the temperature of annealing up to 350°C involves slight decrease of tensile strength, R_m=769 MPa (Table 1) but the considerable loss of plastic properties of investigated tapes is taking place. Tapes are brittle and decrease of characteristic values describing plasticity is observed (ductlility ε=0.033, cracking energy E_p=4.0 $J\cdot cm^{-2}$ – Table 1). Fracture morphology observations confirmed the changes of plastic properties. Characteristic chevron pattern morphology has been revealed on the fracture surface - Fig. 3. It can be pointed out on the structural relaxation process.

Increase of the annealing temperature up to 400°C leads to further loss of tensile strength, R_m=294 MPa, as well of plasticity, ε=0.028 (Table 1). It was impossible to carrying out the tear test because of significant brittleness of annealed tapes. Structure investigations showed that alloy has still amorphous structure in that state - Fig. 1b.

Heat treatment process in annealing temperature range from 450°C up to 600°C involves the changes of the structures as well as the properties of investigated tapes. With the temperature increase the tensile strength R_m is decreasing from the value of 238 MPa to 43 MPa (Table 1). Loss of plastic properties of the tapes following the increase of annealing temperature has been observed. In the structure beside the amorphous phase, α-Fe crystalline phase has been identified - Fig. 1c. This phase is appearing in structure of tapes annealed at temperature of 550°C (Table 1). Fractographic investigations of failure surfaces after decohesion in tensile test showed, in all tapes annealed in temperature range 450°C to 600°C, the same morphology characterized by smooth fracture - Fig. 4.

Existance of FeB, Fe_2B and Fe_2Nb phases together with α-Fe crystalline phase has been identified on X-ray diffraction pattern for tapes annealed at temperature from 650°C - Fig. 1d, Table 1.

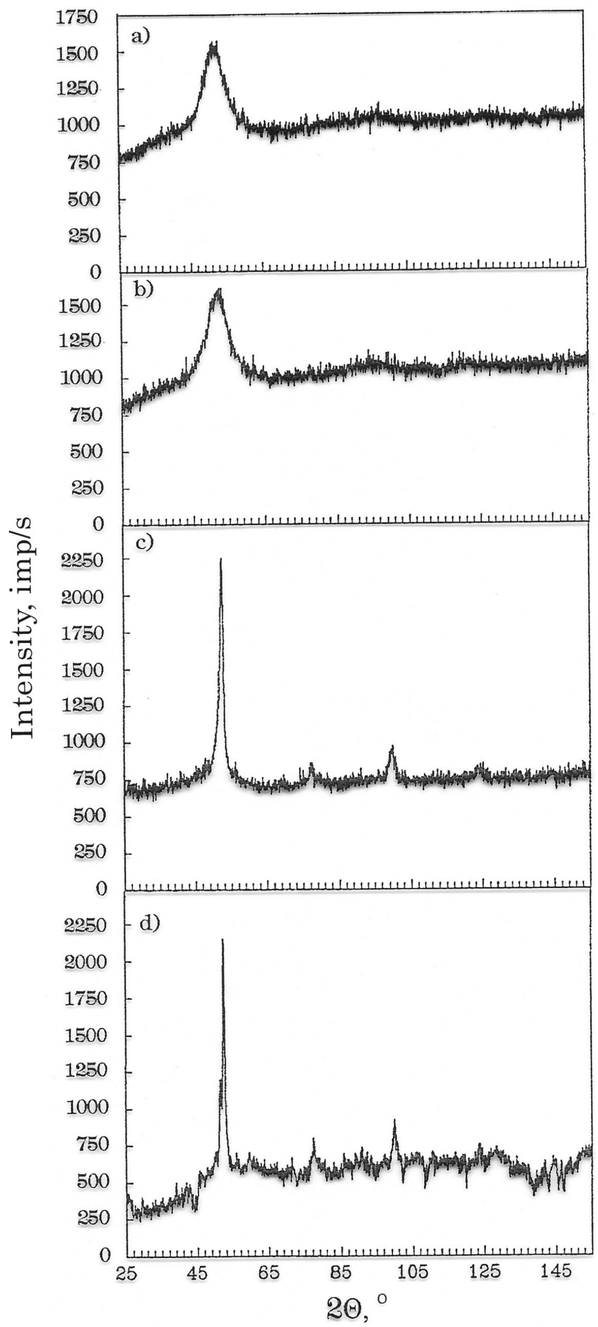

Fig. 1. X-ray diffraction patterns for amorphous $Fe_{84}Nb_7B_9$ alloy: a) in as quenched state; b) after annealing at temperature of 400°C/1h; c) after annealing at temperature of 550°C/1h; d) after annealing at temperature of 650°C/1h

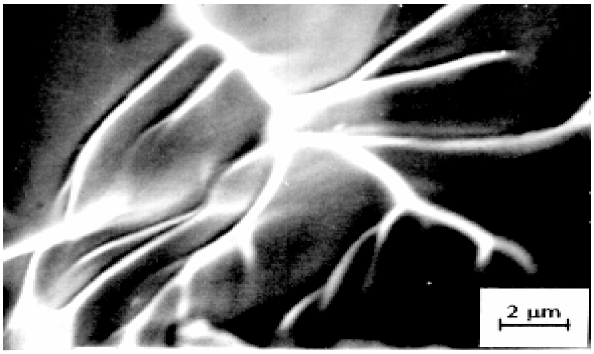

Fig. 2. SEM image of fracture surface of $Fe_{84}Nb_7B_9$ tapes after decohesion in tensile test - as quenched state; vein pattern morphology

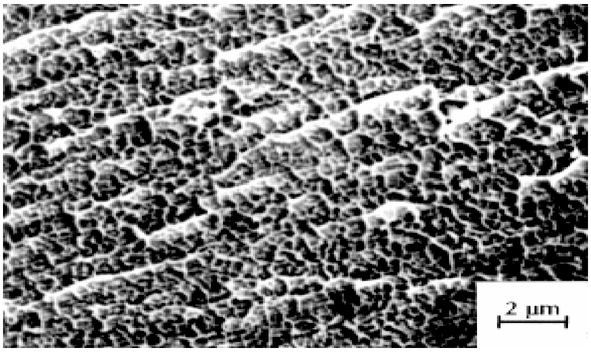

Fig. 3. SEM image of fracture surface of $Fe_{84}Nb_7B_9$ tapes after decohesion in tensile test - annealing temperature 350°C/1h; chevron pattern morphology

The significant changes of fracture morphology of tapes after decohesion are corresponding to the observed structure changes. Morphology is changing from smooth character (Fig. 4) to intercrystalline fracture - Fig. 5.

Table 1.
Influence of heat treatment on mechanical properties (R_m, ε, E_p) and phase composition of amorphous $Fe_{84}Nb_7B_9$ alloy

Heat treatment parameters		Tensile strength	Plastic deformation	Cracking energy	Phase composition
Temperature [°C]	Time [h]	R_m [MPa]	ε	E_p [$J \cdot cm^{-2}$]	
as quenched	1	732	1	9.8	A[1)]
300	1	823	1	10.0	A[1)]
350	1	769	0.033	4.0	A[1)]
400	1	294	0.028	-	A[1)]
450	1	238	0.013	-	A[1)]
500	1	106	0.007	-	A[1)]
550	1	49	0.005	-	A[1)]+α-Fe
600	1	43	0.003	-	A[1)]+α-Fe
650	1	24	0.001	-	α-Fe, FeB,
700	1	20	0	-	Fe_2B, Fe_2Nb

[1)] – amorphous phase

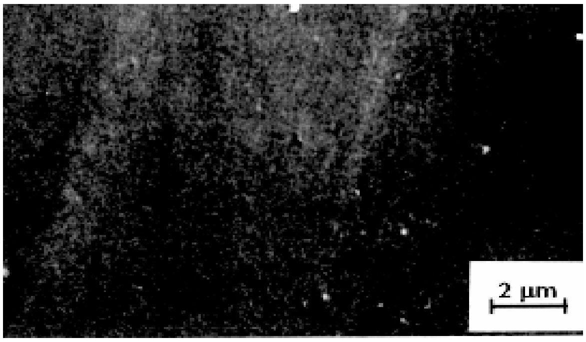

Fig. 4. SEM image of fracture surface of $Fe_{84}Nb_7B_9$ tapes after decohesion in tensile test - annealing temperature 550°C/1h; smooth fracture

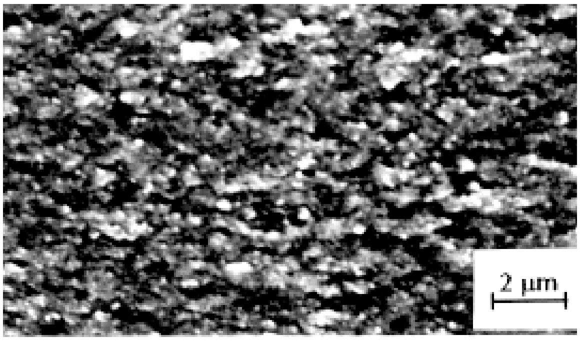

Fig. 5. SEM image of fracture surface of $Fe_{84}Nb_7B_9$ tapes after decohesion in tensile test - annealing temperature 650°C/1h; brittle intercrystalline fracture

4. Conclusions

The worked out investigations have been showed that heat treatment of amorphous $Fe_{84}Nb_7B_9$ alloy in temperature range 300÷700°C involves two-stage crystallization process and leads to radical changes of mechanical properties of tapes.

During annealing process in temperature range from 300°C to 500°C the alloy structure is still amorphous. The relaxation phenomena proceeded in alloy leads only to the changes in mechanical properties and fracture morphology of tapes after decohesion in tensile test.

The increase of annealing temperature up to 550°C causes the beginning of the first stage of crystallization in an amorphous matrix. Crystallization has primary character and is based on precipitation of crystals of α-Fe crystalline phase. In temperature range 550°C to 600°C is existed structure composed of α-Fe crystalline phase and intergranular amorphous phase. The significant decrease of mechanical properties in comparison with as quenched state and characteristic smooth fractures in tapes after decohesion has been observed.

The annealing at temperature over 600°C involves the second stage of crystallization. This stage is characterized by simultanously crystallization of several phases identified as FeB, Fe_2B, Fe_2Nb. This stage of crystallization is responsible for the following loss of mechanical properties. Fracture morphology of tapes has brittle intercrystalline character indicating high dispersion of structure obtained as a result of crystallization process of phases.

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