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CRYSTALLIZATION OF SOME FeNbCuSiB - TYPE ALLOYS[1]

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The influence of Si and B on the formation of nanocrystalline structure was studied by Mössbauer spectroscopy. The kinetics of crystallization was investigated during isothermal annealing at the temperature of 550° C. Samples containing different concentrations of Si and B were compared. Higher amount of boron (14%) stabilised the amorphous structure. At lower content of Si (< 10%), the formation of a-FeSi solid solution is observed.

The kinetics of crystallization and the structure of nanocrystalline materials have been extensively studied [1,2,3]. The majority of these works deals with the optimalization of the thermal treatment. Rather few works have been devoted to the clarification of the role of copper and niobium for the formation of ultrafine grains [4,5]. In order to understand the correlation between nanocrystalline structure and physical properties, a variety of methods have been used [6,7].

In the present work we focus an attention on the influence of Si and B on the crystallization kinetic.

Amorphous $Fe_{74}Cu_1Nb_3Si_{13.5}B_{9.5}$ (I), $Fe_{78}Cu_1Nb_3Si_4B_{14}$ (II), and $Fe_{72.5}Cu_1Nb_{4.5}Si_{10}B_{12}$ (III) have been prepared by the method of planar flow casting. Samples were annealed at 550° C in a vacuum furnace ($p \cong 10^{-3}$ Pa) for up to 16 hours. Transmission ^{57}Fe Mössbauer spectra were taken at room temperature using a ^{57}Co(Rh) source. Spectra were evaluated by the NORMOS DIST program [8] supposing the presence of crystalline and amorphous phase.

Mössbauer spectra of all samples in the as-cast and in the nanocrystalline state are shown in Figs. 1,2 and 3.

The Mössbauer spectra of the as-cast samples are described with a broad hyperfine field distribution which is typical of the amorphous state. Mössbauer parameters are given in Tab. 1. The evolution of the average hyperfine field is proportional to the iron concentration. Samples differ in the relative area D23 of lines 2 and 5 with respect to lines 3 and 4. Taking the parameter D23 into account, we conclude that for higher Nb content (4.5%), sample (III), the net magnetic moments are oriented more out of the

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Table 1. Mössbauer parameters of the as-cast samples. $< H >$ is average field, D_{23} relative area, σ_H standard deviation

Sample	$< H >$ (T)	D_{23}	σ_H (T)
I	20.8	2.5	4.8
II	21.4	2.6	4.6
III	19.1	1.9	5.1

Table 2. Mössbauer parameters of the annealed samples. 1 hour annealing - I(1h), II(1h), III(1h); 8 hours and 14 hours - I(8h), II(8h) and III(14h); $< H_{AM} >$ - average hyperfine field; AM - relative amount of the amorphous phase derived from the area of the spectrum; A1 - A6 - relative amount of the crystalline component derived from the area of the spectrum

Parameters	Sample					
	I(1h)	I(8h)	II(1h)	II(8h)	III(1h)	III(14h)
$< H_{AM} >$ (T)	11.2	6.7	12.7	11.9	18.5	9.3
AM (%)	16	12	20	17	75	27
H1 (T)	31.4	31.4	33.1	33.1	32.0	31.0
A1 (%)	22	26	27	28	35	48
H2 (T)	28.5	28.5	30.1	30.1	30.3	27.8
A2 (%)	14	14	31	33	25	15
H3 (T)	24.3	24.3	25.7	25.8	27.8	25.2
A3 (%)	30	31	13	12	7	4
H4 (T)	19.3	19.4	20.9	20.9	21.5	23.1
A4 (%)	34	29	28	27	5	14
H5 (T)					33.1	33.1
A5 (%)					25	12
H6 (T)					34.1	19.7
A6 (%)					3	7

ribbon plane. We suppose, that the higher content of Nb causes changes in the magnetic structure of the as-cast specimens. The increase of the standard deviation σ_H of the P(H) distributions with increasing the Nb content confirms changes in the local short range order. Comparing the samples with 3 at% of Nb and different concentration of Si and B, samples (I,II), we conclude that in the as-cast state the D23 and σ_H parameters change only slightly. Probably Si and B have not significant influence on the magnetic moment orientation.

After one hour of isothermal annealing Mössbauer spectrum of $Fe_{74}Cu_1Nb_3Si_{13.5}B_{9.5}$ (I) gives evidence for a nanocrystalline structure. Besides the remaining amorphous phase, the additional sharp lines can be assigned to FeSi -DO_3 structure and are fitted with four sextets (Table 2).

The Mössbauer spectrum of $Fe_{78}Cu_1Nb_3Si_4B_{14}$ (II) annealed one hour differs from

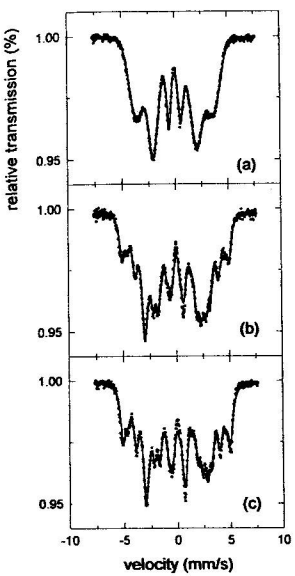

Fig.1. Mössbauer spectra of sample I
a) as-cast
b) 1 hour annealing
c) 8 hours annealing

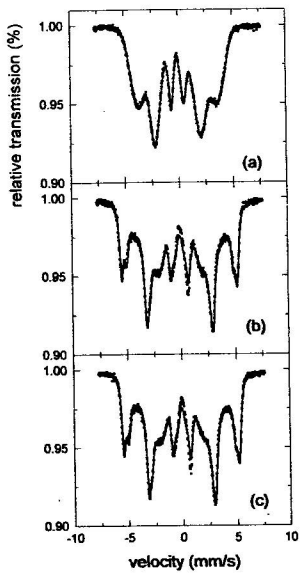

Fig.2. Mössbauer spectra of sample II
a) as-cast
b) 1 hour annealing
c) 8 hours annealing

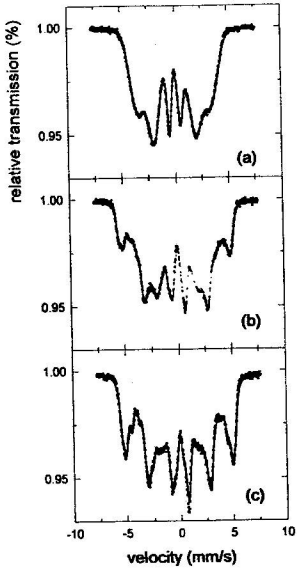

Fig.3. Mössbauer spectra of sample III
a) as-cast
b) 1 hour annealing
c) 14 hours annealing

that of sample (I) after the same annealing time. We observed a higher amount of the remaining amorphous phase with higher values of the average hyperfine field. The crystalline phase is consistent with four sextets with parameters different from those of sample (I). The magnetic hyperfine field values extend over 30 T. We suppose that Fe and Si form a disordered alloy corresponding to a low Si concentration. In a such system the number of Fe atoms with 8 Fe nearest neighbours is reduced and the mean hyperfine magnetic field decreases wit increasing Si content. Therefore, the observed crystalline phase could correspond to a FeSi solid solution.

The Mössbauer spectrum of $Fe_{72.5}Cu_1Nb_{4.5}Si_{10}B_{12}$ (III) after one hour annealing is rather complex. However, the sample contains high amount of remaining amorphous phase. According to our opinion the crystalline phase consists for one part of FeSi solid solution and for a second part of FeSi - DO_3 structure.

After 8 hour heating the Mössbauer spectrum of sample (I) exhibits a typical behaviour of nanocrystalline structure. The amount of the remaining amorphous phase decreases and the crystalline one increases. The value of the average hyperfine field also decreases roughly down to 50 % of the value observed after one hour of annealing.

In sample (II) after 8 hours annealing the average hyperfine field and the amount

of remaining amorphous phase decreases only slightly. The parameters of the crystalline phase exhibit also a very small change. We suppose that a higher content of B contributes to the stabilisation of the amorphous structure. The crystalline FeSi solid solution remains stable.

In sample (III) after 14 hours annealing the amount of the amorphous phase significantly falls. The crystalline phase appears as a mixture of FeSi solid solution and FeSi - DO_3 phase.

The amount and composition of crystalline and amorphous phases created during the isothermal heat treatment of amorphous alloy depend on the concentration of constituent elements. Low content of Si leads to the creation of Fe- Si solid solution as a crystalline phase. Long time annealing up to 8 hours does not cause a noticeable change in the crystalline and amorphous phase. This may be also due to higher content of B which stabilise the amorphous structure. This behaviour differs from that of nanocrystalline samples containing higher content of Si and lower one of B in which FeSi-DO_3 structure was created. In these samples significant changes in parameters take place with prolonged annealing.

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