Materials Physics and Mechanics 9 (2010) 85-89 Received: March 17, 2010

STUDY OF CRYSTALLIZATION OF $Fe_{95}Si_5$ AMORPHOUS ALLOY USING XRD AND DSC

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Abstract. Crystallization of amorphous metallic alloy $Fe_{95}Si_5$ (at. %) was investigated using X-ray diffraction measurements performed *in-situ* during isothermal annealing. $Fe_{95}Si_5$ ribbons, prepared by melt-spinning, have been analyzed by differential scanning calorimetry (DSC). Their nanostructural evolution has been studied by combining heat treatments with conventional X-ray diffraction. The objective of the experiment was to determine changes in the structural parameters during crystallization of the examined alloy. The crystalline diameter and the lattice constant of the crystallizing phase were used as parameters to evaluate structural changes in the material.

1. Introduction

It is well known that amorphous alloys are materials with interesting magnetic and mechanical proprieties. The physical properties of the amorphous iron based alloys have been investigated intensively during the last 30 years, because these alloys are very promising as soft magnetic materials. These materials have high saturation, high permeability, and low coercivity magnetization. The materials are already in use in the constructions of cores of distribution transformers, antitheft security systems, recording heads and electronic devices. Further modification of the magnetic properties of these alloys may be achieved by heat treatment.

The optimum magnetic properties can be obtained in the case of metallic glass partly crystallized. For instance, it has been shown that the nanocrystalline structure presented in Fe-Cu-Nb-Si-B alloys after isothermal annealing is responsible for their excellent soft-magnetic properties [1]. Crystallization of amorphous solids has been found to produce nanometer-sized polycrystalline materials in various alloy systems. The primary crystallization can be used to obtain partially crystallized alloys with nanocrystallites embedded in the residual amorphous matrix which display novel magnetic properties. One of the heat treatment methods to obtain the nanocrystalline structure is an isothermal annealing.

In this paper, we present a study of the primary crystallization of the $Fe_{95}Si_5$ amorphous alloy performed under both an isothermal and a continuous heating regime. Kinetic information is obtained from X-ray diffraction and DSC measurements. The article reports experimental data obtained during the microstructural transformations that lead to a nanostructured alloy with BCC – Fe(α) nanocrystals embedded in the amorphous matrix.

2. Experimental procedure

The amorphous samples were obtained by rapid solidification from the melt using the melt-spinning method. The resulting ribbons were about 20 mm wide and 0.03 mm thick, with

chemical composition of $Fe_{95}Si_5$. The calorimetric experiments were performed in a NETZSCH DSC 204. Thermal data were measured at the heating rate of 5, 10, 20 and 30K/min. In the second heat treatment specimens of this material were annealed isothermally in the temperature range of 653÷803 K for 30, 60 and 120 minutes.

The structural characterization was carried out by combining heat treatments with conventional X-ray diffraction (XRD). Conventional XRD experiments were carried out with the help of DRON 4 diffractometer using the monochromatic $CuK\alpha$ radiation, over the 2Θ angle range of 35÷105 degrees with a step of 0.05 degrees/min. The voltage was set at 31 kV and the current set at 21 mA.

3. Experimental results and discussion

Figure 1 shows DSC curves for $Fe_{95}Si_5$ amorphous metallic alloys, obtained at the heating rate of 5, 10, 20 and 30 K/min. Samples of the alloy present two distinct exothermal peaks, and this behavior is independent of the applied heating rate, suggesting a crystallization process with two distinct exothermal reactions. This corresponds to the results reported in [2]. The crystallization parameters are shown in Table 1. X-ray analysis of the annealing process shows that the product of primary crystallization is Fe_α as described below. From DSC curves it is concluded that the temperature of crystallization T_x of alloy depends on heating rate [3].

Figure 2 shows conversion rates of primary crystallization (first peak of DSC) as a function of time for the $Fe_{95}Si_5$ amorphous alloy. The crystallization of $Fe_{95}Si_5$ samples follows different kinetics according to the applied heating rate.

Diffraction pattern of a selected alloy sample after isothermal annealing is presented in Figs. 3 and 4. The visible reflection indicates (Fig. 3.) the beginning of an alloy crystallization process. The reflections in the XRD patterns were identified as Fe_α phase. An analysis of the changes in the values of the lattice constant, leads to conclusion that in the case of lower annealing temperature (T_a =693 and 753 K) the lattice constant a is lower than the lattice constant of pure Fe_α ($a = 2.8663$ Å). This is probably related to the fact that at lower annealing temperature the phase Fe_α(Si) is obtained. This phase is a substitutional solid solution of elements of smaller diameter (Si) in elements of larger diameter (Fe) that is in agreement with [3].

Table 1. Primary crystallization parameters of $Fe_{95}Si_5$ measured by DSC (first peak)

Heating rate	5K/min	10K/min	20K/min	30K/min
Temperature max. Peak [K]	718,6	730,6	742,9	750,2
Enthalpy phases transformation	43,61J/g	41,95 J/g	42,54J/g	41,42 J/g
Onset (T_x) [K]	699,6	722,6	725,5	807,5
End [K]	737,9	746,7	757,9	818,1

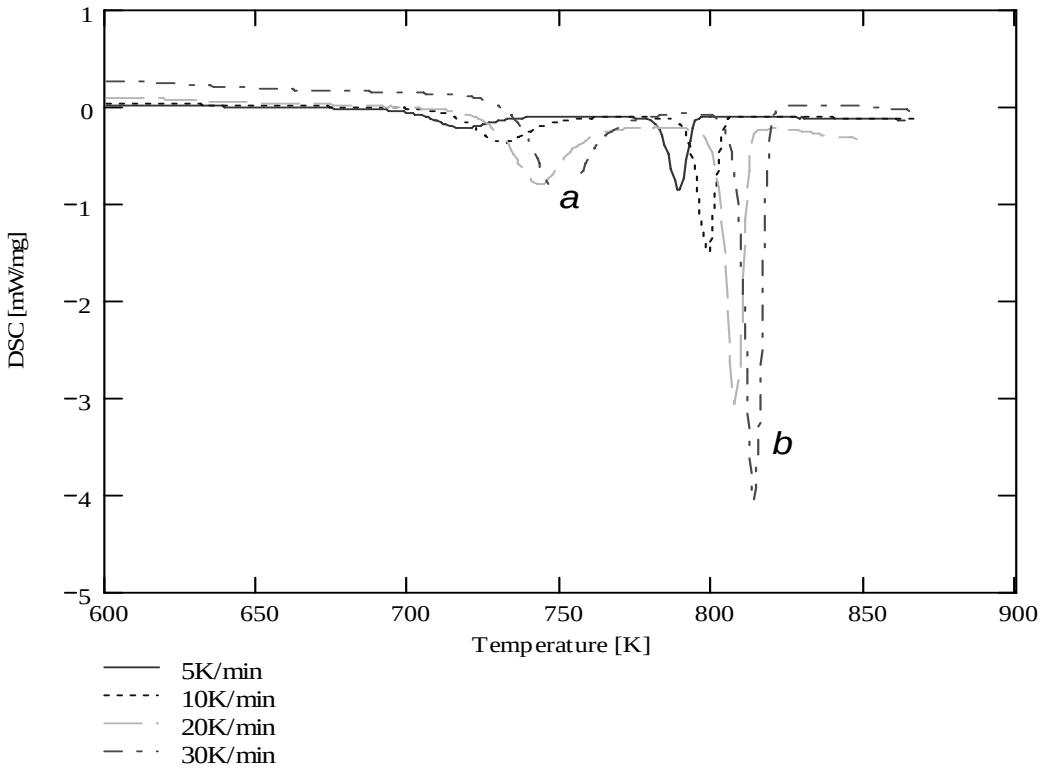

Figure. 1. DSC curves of $Fe_{95}Si_5$ for several heating rates showing the primary crystallization process (a) and secondary crystallization (*b*).

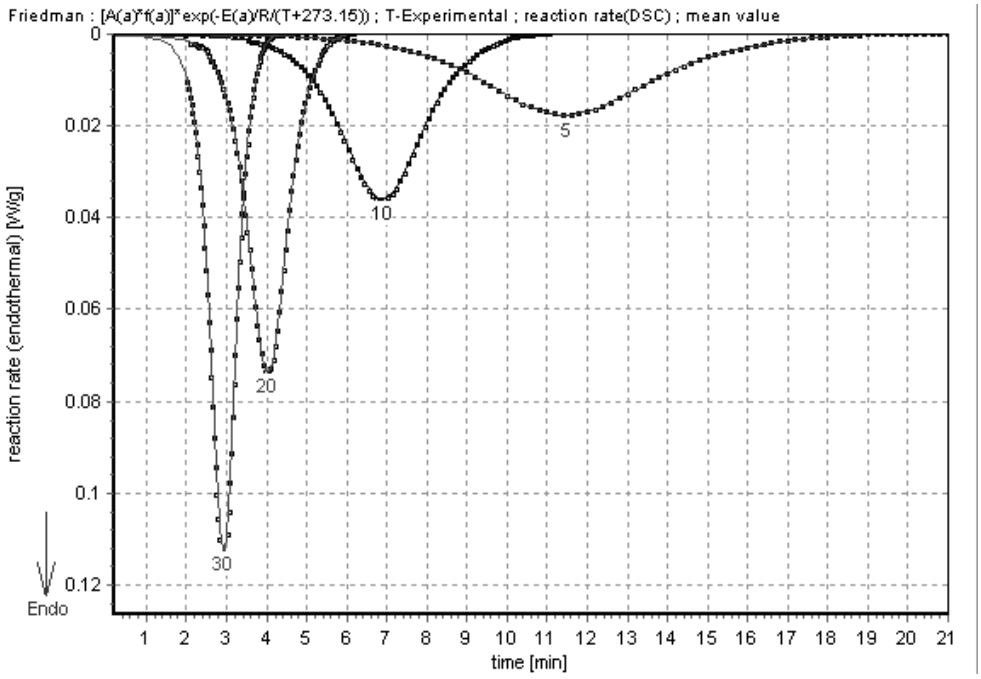

Figure. 2. Conversion rate of primary crystallization (first peak of DSC) as a function of time for $Fe_{95}Si_5$ amorphous alloy.

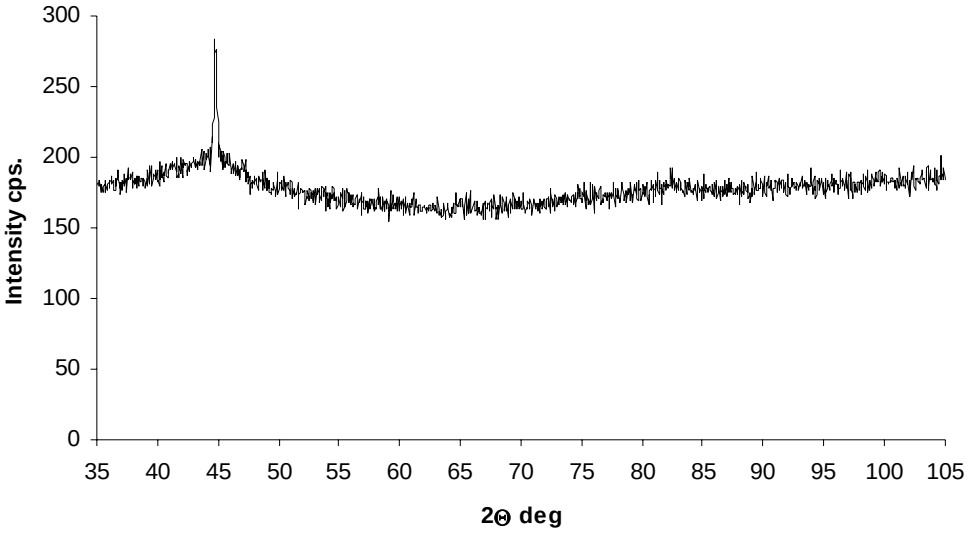

Figure. 3. XRD pattern for the samples annealed at *Ta=653K*. The annealing time is 60 min. Bulk crystallization of the amorphous alloy was observed at higher temperatures.

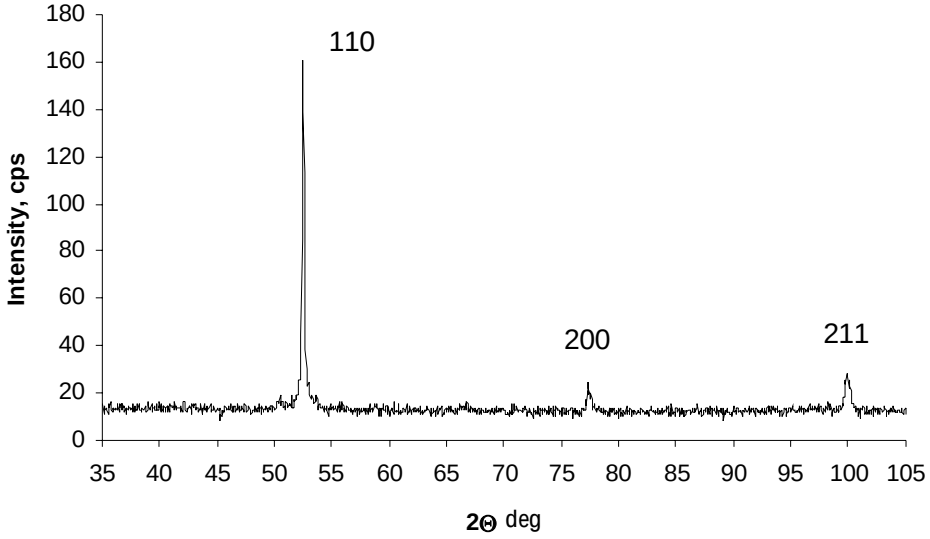

Figure. 4. XRD pattern for the samples annealed at T_a=693K. The annealing time is 60 min.

Figure 5 shows the evolutions of the grain size of the α-iron (bcc lattice) caused by isothermal annealing at T_a =653, 693, 753 and 803K. The average grain size was calculated from the full width at half of the maximum of the 110 Fe_α reflection using the Scherrer formula.

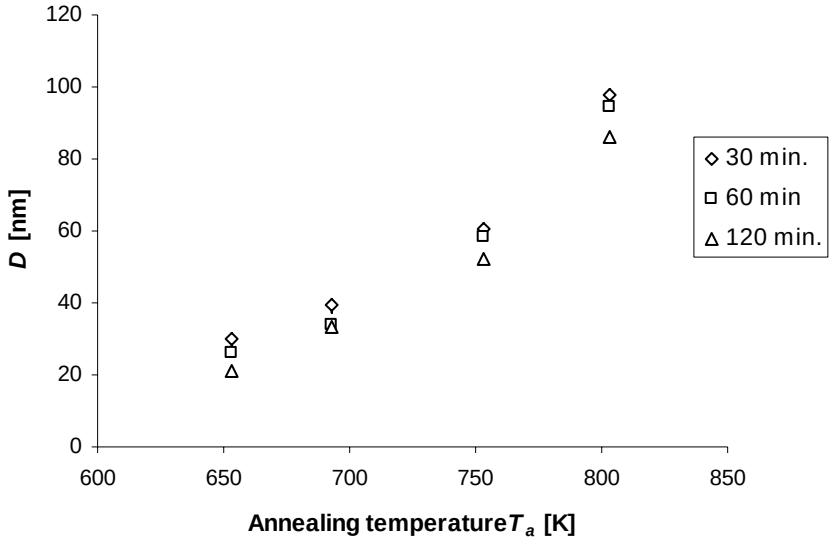

Figure. 5. Changes of domain sizes during isothermal treatments at T_a= 653, 693, 753 and 803K.

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