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Effect of Annealing Time on Structure of $Fe_{72.5}Cu_1Nb_2Mo_2Si_{15.5}B_7$ Alloy

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The process of a primary crystallization of the $Fe_{72.5}Cu_1Nb_2Mo_2Si_{15.5}B_7$ alloys was investigated by differential thermal analysis (DTA), x-ray diffraction (XRD) and transmission electron microscopy (TEM). Amorphous ribbons were isothermally annealed for 0.5, 2, 6, 30 and 150 minutes at 520 °C. Both, the XRD and TEM study showed that the level of devitrification of the sample increases with the annealing time. The above mentioned techniques confirmed the presence of the nanocrystalline grains of the Fe_3Si phase and enable us to study the evolution of the identified phase.

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1. Introduction

The Fe-based amorphous and nanocrystalline alloys have been confirmed as excellent soft magnetic materials for applications in many fields and therefore large interest has been devoted to them [1, 2]. The excellent soft magnetic properties of these alloys are related to their nanostructure [3]. The purpose of this study was to follow the devitrification process of the investigated alloy and to observe the influence of annealing time on the increase of the nanocrystalline phase.

2. Experimental

Amorphous ribbons of the chemical composition $Fe_{72.5}Cu_1Nb_2Mo_2Si_{15.5}B_7$ were prepared by melt spinning technique. The samples were annealed in a conventional vacuum furnace for 0.5, 2, 6, 30 and 150 minutes at temperatures of 520 °C. The choice of the annealing temperature was dictated by the fact that the primary crystallization in FINEMET type alloys takes place around 520 °C [4]. In this work the FINEMET type soft magnetic materials with a small content of Mo were characterized by the DTA, XRD and TEM.

DTA experiments were performed in order to study the influence of annealing temperature on the process of a primary crystallization of the ribbons. The DTA investigations were realized in the 25 – 800 °C temperature range, using experimental equipment Netzsch STA 409PC in atmosphere of argon (50 cm^3/min.), with heating rate of 20 °C/min. The structure of the samples was investigated by XRD and TEM. The XRD was carried out at the beamline P02.1 at the storage ring PETRA III (Hamburg, Germany). The measurements were done in transmission geometry using the wavelength of 0.2073 Å. TEM experiments were realized using JEOL JEM 2100F UHR operating at 200 kV. The sample foils for the TEM observation were prepared by electrolytic thinning.

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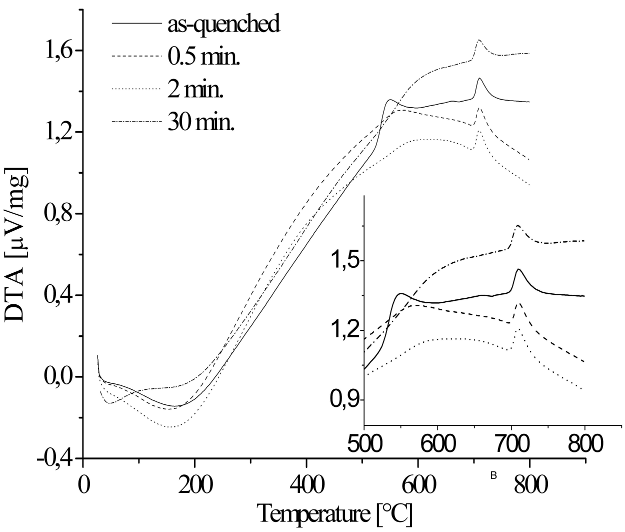

Fig. 1. DTA curves of $Fe_{72.5}Cu_1Nb_2Mo_2Si_{15.5}B_7$ alloy.

3. Results and discussion

Figure 1 illustrates the obtained DTA curves. Two exothermic peaks (with maximums at $T_{x1} = 550$ °C and $T_{x2} = 710$ °C) are seen on the DTA curve for amorphous sample, demonstrating the two-step devitrification process. The first one corresponds to the formation of α-FeSi grains embedded in a still amorphous matrix. The second one corresponds to the creation of boride-type phases. This behaviour has been observed by several earlier studies [5, 6] and was confirmed by our XRD measurements as well (see below). Additionally, DTA measurements show that the first exothermic peak corresponding to the first crystalline temperature diminished with the sample annealing. The position of the second peak was not influenced by the sample annealing as can be seen in Fig. 1.

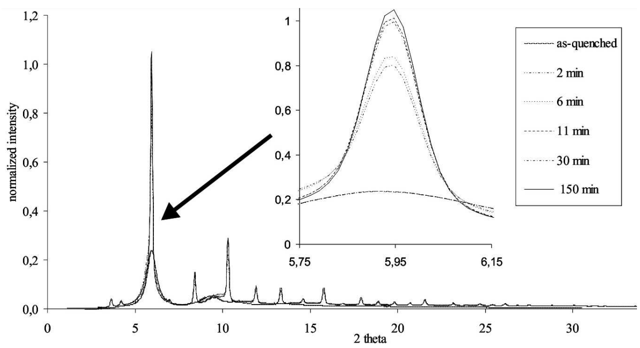

Fig. 2. Measured XRD data of $Fe_{72.5}Cu_1Nb_2Mo_2Si_{15.5}B_7$ alloy. The inset shows detail to the biggest peak.

The XRD profiles (Fig. 2) of the annealed specimens clearly manifest the effect of the annealing time on the investigated alloy. The peaks became sharper with increasing of annealing time, indicating the growth of nanocrystallites (see insert in the Fig. 2). The x-ray data analysis confirmed the presence of Fe_3Si phase. The average particle size was determined from the full width at half maximum (FWHM) of the third diffraction peak using Scherrer's equation (see Table).

TABLE

Average grain size, in nanometers, obtained by XRD and TEM analysis.

	6 min.	30 min.	150 min.
XRD	15.77	16.1	16.17
TEM	13.36	14.11	15.44

STEM (Scanning Transmission Electron Microscopy) images with corresponding electron diffraction patterns and histograms are shown in Fig. 3. The grain size distribution and average grain size were determined by TEM analysis (see in Tab. I). From STEM images it is clear, that the grain size was more homogeneous and average particle size increased with the increasing annealing time.

4. Conclusions

In this paper electron microscopy technique and XRD were used for structure analysis of $Fe_{72.5}Cu_1Nb_2Mo_2Si_{15.5}B_7$. DTA measurements confirmed that the alloy crystallises through two stages. The effect of annealing time on grain growth was confirmed, the average grain size increased from 13 nm to 16 nm with increasing annealing time.

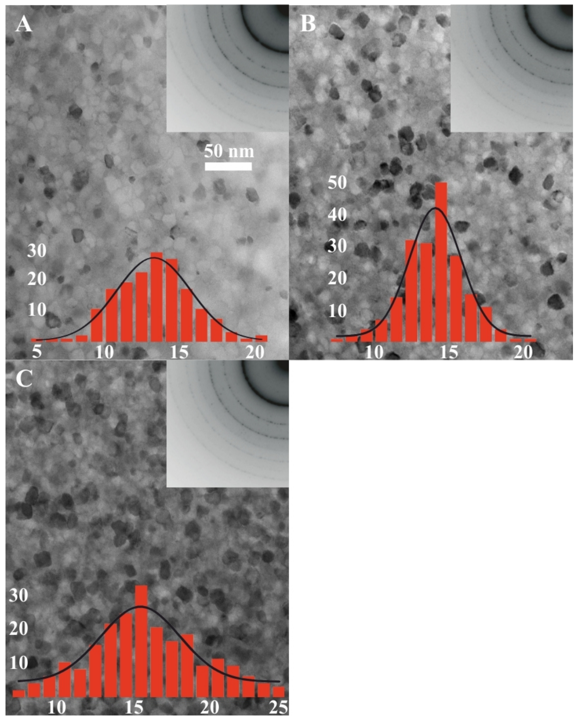

Fig. 3. STEM BF (Scanning Transmission Electron Microscopy Bright Field) images with electron diffraction patterns of nanograins structure. A) 6 minutes, B) 30 minutes, C) 150 minutes. The size distributions of nanograins are presented in imbedded histograms, where x and y axis represent particle diameter in nanometers and counts, respectively.

Acknowledgments

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