Influence of Zn on Structure and Magnetic Properties of Rapid Quenched (Fe–Zn)–Cu–Nb–Si–B Alloys

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The aim of this work was to study the influence of Zn on the formation of nanostructure in Fe\textsubscript{x}Zn\textsubscript{y}Cu\textsubscript{1}Nb\textsubscript{3}Si\textsubscript{13.5}B\textsubscript{9} (x = 1, 3, 5) as-quenched ribbons prepared by melt-spinning technique. X-ray diffraction measurements proved amorphous state of the sample with x = 1. The sample with x = 3 contains a small fraction of Fe\textsubscript{3}Si phase and the sample with x = 5 is in nanocrystalline state with the average grain size of about 25 nm. The reduced radial distribution function G(r) was calculated for local structure investigation. Increasing Zn content enhances crystallization during solidification which leads to increase of magnetocrystalline anisotropy and consequently to increase of coercivity.

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1. Introduction

Nanocrystalline Fe–Cu–Nb–Si–B alloy prepared from amorphous state by annealing at about 550°C has excellent magnetic properties and a lot of potential applications such as magnetic parts of power transfers, magnetic sensors, actuators etc. [1–3]. Manufacturing of these ribbons employing melt-spinning technique. X-ray diffraction (XRD) method. XRD measurements were performed at the BW5 beamline at DESY/HASYLAB (Hamburg, Germany). The samples in form of ribbons with chemical composition Fe\textsubscript{x}Zn\textsubscript{y}Cu\textsubscript{1}Nb\textsubscript{3}Si\textsubscript{13.5}B\textsubscript{9} (x = 1, 3, 5) were prepared by melt spinning technique. The ribbons are 2 mm wide and 50 μm thick at the linear speed of cooling rate 30 m/s. Their structure was investigated by X-ray diffraction (XRD) method. XRD measurements were performed at the BW5 beamline at DESY/HASYLAB (Hamburg, Germany). The samples were illuminated for 20 s by X-rays of wavelength 0.124 Å. XRD patterns were collected in symmetric transmission geometry using a MAR 345 two-dimensional (2D) image plate detector (2300 × 2300 pixels, 150 × 150 μm\textsuperscript{2} pixel size), which was mounted perpendicular to the incident X-ray beam. Radiation energy was determined by fitting a standard reference LaB\textsubscript{6} sample. The saturation magnetization (M\textsubscript{s}) measurements of as-quenched ribbons were performed by vibration sample magnetometer (VSM). Coercivity was determined from hysteresis loops traced with fluxmeter in quasi DC magnetic field.

2. Experimental

The samples in form of ribbons with chemical composition Fe\textsubscript{x}Zn\textsubscript{y}Cu\textsubscript{1}Nb\textsubscript{3}Si\textsubscript{13.5}B\textsubscript{9} (x = 1, 3, 5) were prepared by melt spinning technique. The ribbons are 2 mm wide and 50 μm thick at the linear speed of cooling rate 30 m/s. Their structure was investigated by X-ray diffraction (XRD) method. XRD measurements were performed at the BW5 beamline at DESY/HASYLAB (Hamburg, Germany). The samples were illuminated for 20 s by X-rays of wavelength 0.124 Å. XRD patterns were collected in symmetric transmission geometry using a MAR 345 two-dimensional (2D) image plate detector (2300 × 2300 pixels, 150 × 150 μm\textsuperscript{2} pixel size), which was mounted perpendicular to the incident X-ray beam. Radiation energy was determined by fitting a standard reference LaB\textsubscript{6} sample. The saturation magnetization (M\textsubscript{s}) measurements of as-quenched ribbons were performed by vibration sample magnetometer (VSM). Coercivity was determined from hysteresis loops traced with fluxmeter in quasi DC magnetic field.

3. Results and discussion

The XRD patterns of Fe\textsubscript{x}Zn\textsubscript{y}Cu\textsubscript{1}Nb\textsubscript{3}Si\textsubscript{13.5}B\textsubscript{9} (x = 1, 3, 5) as-quenched ribbons are presented in Fig. 1. The Bragg peaks in XRD pattern of x = 5 belong to cubic Fe\textsubscript{3}Si phase (ICSD 412838, space group: Fm\textsubscript{3}m). The average grain size of Fe\textsubscript{3}Si phase (≈ 25 nm) was determined from the Debye–Scherrer equation [8]:

\[ L = \frac{(0.9)\lambda}{\beta \cos \theta} \]

Here, L is the coherent length, λ is the X-ray wavelength, β is the full-width at half-maximum of the XRD peak and θ is the Bragg angle. Small crystalline fraction Fe\textsubscript{3}Si phase is detectable in x = 3 sample (small Bragg peaks) as well. Only the sample x = 1 is in a fully amorphous state as it is documented by its diffraction profile consisting of two diffuse peaks without any indications of the Bragg peaks.

The Fourier inversion of structural factor S(Q) obtained from diffraction data gives the reduced radial dis-

(790)
with the interatomic distances in the Fe defined maxima, whose positions are in good agreement the other hand, maxima of...

maxima of... can be seen in Fig. 2.

amplitudes and sharpness of... samples are shown in Fig. 2.

Summarization of magnetic parameters for the as-quenched samples (Table) are apparently lower in comparison to the results of [6] (≈ 26 A/m for as-quenched samples). Increasing value of coercivity with higher Zn content can be interpreted as follows: Crystallization of investigated samples (see Fig. 1, Fig. 2) is a result of rapid solidification process which leads to hinder domain wall movement (magnetocrystalline anisotropy increasing) [9], analogous to the results reported in [6]. The decrease of magnetocrystalline anisotropy (magnetic softening) can be reached by annealing process when the nanostructure is created after (or as a consequence) of structural relaxation process [10].

The decrease of \( M_s \) values (listed in Table) is caused by decreasing Fe content.

4. Conclusion

The paper was supported by Slovak Grant Agency VEGA (1/0167/10 and 1/0311/10) and it was done in the frame of the project “Centre of Excellence of progressive materials with nano- and submicron microstructure”, with support of the Operational Program Research and Development financed from European Regional Development Fund.

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The determined \( G(\mathbf{r}) \) functions of Fe\(_{73.5-x}\)Zn\(_x\)Cu\(_1\)Si\(_{13.5}\)B\(_3\)Zn\(_x\) (\( x = 1, 3, 5 \)) as-quenched samples shown in Fig. 2.

With increasing degree of structural ordering amplitudes and sharpness of \( G(\mathbf{r}) \) function increase as it can be seen in Fig. 2. \( G(\mathbf{r})_{x=3} \) shows pronounced and well defined maxima, whose positions are in good agreement with the interatomic distances in the Fe\(_3\)Si structure. On the other hand, maxima of \( G(\mathbf{r})_{x=1} \) and \( G(\mathbf{r})_{x=5} \) are evidently broader than those of \( G(\mathbf{r})_{x=3} \). Additionally it is seen that main peaks (in the interval 2–3.5 Å) of \( G(\mathbf{r})_{x=1} \) and \( G(\mathbf{r})_{x=3} \) are very similar suggesting a similar short local ordering in both, \( x = 1 \) and \( x = 3 \) samples. Deviations from this similarity (marked with inclined arrows in Fig. 2) are clearly visible for distances \( r \) greater than 4.5 Å. These differences reflect the fact that the \( x = 3 \) sample contains small amount of crystalline Fe\(_3\)Si phase in the amorphous matrix compared with the fully amorphous \( x = 1 \) sample (Fig. 2).

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From prepared Fe\(_{73.5-x}\)Zn\(_x\)Cu\(_1\)Si\(_{13.5}\)B\(_3\)Zn\(_x\) (\( x = 1, 3, 5 \)) as-quenched ribbons the \( x = 1 \) sample was in amorphous state. The \( x = 3 \) sample contains a small fraction of Fe\(_3\)Si phase and the sample with \( x = 5 \) is in nanocrystalline state with the average grain size of about 25 nm. The shape of main peaks (in the range 2–3.5 Å) of \( G(\mathbf{r})_{x=1} \) and \( G(\mathbf{r})_{x=3} \) indicate similar short local ordering in both, \( x = 1 \) and \( x = 3 \) samples. Increasing Zn content enhances crystallization during solidification which leads to increase of magnetocrystalline anisotropy and consequently to increasing value of coercivity.

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