Phase precipitation and isothermal crystallization kinetics of FeZrB amorphous alloy

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Received: 27 April 2013 / Accepted: 26 June 2013 / Published online: 26 July 2013
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Abstract The crystallization process of Fe₇₈Zr₇B₁₅ (at%) amorphous ribbon was investigated by X-ray diffraction (XRD), differential scanning calorimetry and scanning electron microscopy (SEM). The fully amorphous structure of as-quenched (Aq) ribbons was confirmed by XRD pattern. The saturation magnetization (M_s) and Curie temperature of the Aq ribbon were measured as 124.3 (A·m²)/kg and 305 °C with vibrating sample magnetometer (VSM), respectively. When the ribbons was annealed at 550 °C near the first onset temperature ($T_{x1} = 564.9$ °C), the M_s was increased by 17 %, which was caused by the formation of a dual phase structure. The isothermal crystallization kinetics and crystallization mechanism of primary α -Fe phase in the dual phase structure were studied by Arrhenius and Johnson-Mehl-Avrami-Kolmogorov equations respectively. The results showed that the crystallization of α -Fe phase was a diffusion-controlled surface nucleation growth process, and the nucleation rate decreased with longer crystallization time.

Keywords Crystallization · Dual phase structure · Activation energy · Avrami exponent

1 Introduction

In contrast to Co-based alloys with excellent magnetic properties [1], Fe-based soft magnetic alloys usually do not exhibit a good magnetic property [2, 3]. Considering the extensive applications of Fe-based soft magnetic alloys in certain fields such as transformer core and magnetic sensor [4–6], abundant endeavors were still done to explore the effective methods to improve their magnetic properties. One method is to introduce an α -Fe phase into the amorphous phase matrix. To achieve this α -Fe/amorphous dual phase structure, annealing of the amorphous precursors is a useful and easy way [4, 5]. During annealing, the crystallization process of Fe-based alloys generally proceeds in two distinct steps, i.e., a primary crystallization of α -Fe phase followed by the precipitation of Fe-B(Zr) compounds from the residual amorphous matrix [6, 7]. According to Herzer's model [8], the interaction between α -Fe grains and amorphous matrix can average out the magneto-crystalline anisotropy, promoting the magnetic properties of the Fe-based alloys. On the other hand, the existence of Fe-B(Zr) phase can increase the magneto-crystalline anisotropy and correspondingly deteriorate the soft magnetic properties [9, 10]. Therefore it is critical to achieve excellent soft magnetic properties by enhancing α -Fe amorphous dual phase structure formed in the primary crystallization and strictly prohibiting the precipitation of Fe-B(Zr) phase. Furthermore Suzuki et al. [11] found that the coercivity (H_c) sharply increased with the growth of α -Fe grain and built up a dependence of the cubic grain size (d^3) on H_c . As such, precise control of the α -Fe phase precipitation during the primary crystallization is important to optimize the soft magnetic properties in the Fe-based alloys. Besides, the understanding of the crystallization kinetics and crystallization mechanism of primary α -Fe

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phase can provide a significant guidance to choose an appropriate annealing process and therefore achieve the optimized soft magnetic properties.

In particular, as-annealed Nanoperm alloy with dual phase structure is widely used [12, 13]. Since 1990s, efforts have been done on improving the soft magnetic properties of FeMB system (M standing for the transition elements, such as Zr, Nb, Mo, etc.) with high iron content by doping elements [14–17] and optimizing the annealing process [18–20]. Generally, two kinds of annealing processes have been employed to obtain the dual phase structure: i.e., continuously heating process and isothermal process. Johnson-Mehl-Avrami-Kolmogorov (JMAK) equation and its extension have also been developed to evaluate the isothermal and non-isothermal kinetics [21–23]. Our previous study [24] has discussed the non-isothermal crystallization kinetics of $\text{Fe}_{78}\text{Zr}_{15}\text{B}_{15}$ alloy with high stability and high Curie temperature (T_c) in the amorphous state. To compare the crystallization kinetics of the annealing process and its corresponding effects on the phase precipitation, the isothermal crystallization kinetics of $\text{Fe}_{78}\text{Zr}_{15}\text{B}_{15}$ alloy was investigated in this paper. Besides, the local activation energy (E_c) and Avrami exponent (n) of primary α -Fe phase were calculated to illustrate the precipitation mechanism of α -Fe phase.

2 Experimental process

Master alloy ingots with nominal composition of $\text{Fe}_{78}\text{Zr}_{15}\text{B}_{15}$ (at%) were prepared in an arc furnace under argon atmosphere. The ingots were melted at least five times to ensure the homogeneity. Amorphous ribbons with a cross section from $0.025 \text{ mm} \times 4 \text{ mm}$ to $0.045 \text{ mm} \times 4 \text{ mm}$ were prepared by a single roller melt-spinning method in argon atmosphere at a linear speed of 34.5 m/s. Heat treatments were carried out in a vacuum furnace with the vacuum of $2.6 \times 10^{-3} \text{ Pa}$. The ribbons were cut into small strips with length of 20 mm and annealed at different temperatures (T_a) for 20 min.

Differential scanning calorimetry (Netzsch DSC 404) was used to investigate the thermal behavior of the as-quenched (Aq) ribbons at a heating rate of $20^\circ\text{C}/\text{min}$. The structures of the Aq and annealed ribbons were examined by X-ray diffraction (XRD, DImax-2200) with $\text{Cu K}\alpha$ radiation. The isothermal DSC experiments were performed in a DSC (Perkin-Elmer Diamond 3000). The M_s and T_c were measured by vibrating sample magnetometer (VSM, LakeShore 7407) under a magnetic field with a maximum of 0.5 T. The morphology of the precipitated phases was observed by scanning electron microscopy (SEM, JSM-6700F).

3 Results and discussion

3.1 Crystallization process

The DSC curve of Aq ribbon (see Fig. 1) exhibited two distinct steps. The crystallization characteristic parameters, including the glass transition temperature T_g , onset crystallization temperatures of the first and the second exothermic peak, T_{x1} and T_{x2} , and the peak temperatures of the first and the second exothermic peak, T_{p1} and T_{p2} , were listed in Table 1. Normally it was believed that the crystallization started at T_{x1} [25]. However the T_{x1} was not constant and shifted to a higher temperature with increasing the heating rate due to the thermodynamic effect. Therefore the crystallization actually occurred at a lower temperature than T_{x1} . Considering this thermodynamic effect, three different annealing temperatures (T_a) were chosen, i.e., (i) $T_a = 450^\circ\text{C} < T_g$, (ii) $T_g < T_a = 550^\circ\text{C} < T_{x1}$, (iii) $T_{x1} < T_a = 720^\circ\text{C} < T_{x2}$, to explore the possible precipitation phases at different crystallization steps.

The XRD patterns of the Aq and annealed ribbons were shown in Fig. 2. A broad peak at around $2\theta = 44^\circ$ confirmed the fully amorphous nature of Aq ribbons. It did not significantly change the amorphous structure because the XRD pattern of the Aq ribbon annealed at 450°C was almost the same to that in Aq state (see Fig. 2). When $T_a = 550^\circ\text{C}$, namely $T_g < T_a < T_{x1}$, the Aq ribbon exhibited a sharp diffraction peak (corresponding to an α -Fe phase in Fig. 2) overlapping the broad peak, indicating an occurrence of crystallization. When T_a increased to 720°C slightly below T_{x2} , the broad peak completely disappeared and the relative intensity of α -Fe phase diffraction peak increased, implying that much more α -Fe phase precipitated. Meanwhile, the diffraction peaks corresponding to Fe_2B could be observed with low height.

Fig. 1 DSC curves of Aq $\text{Fe}_{78}\text{Zr}_{15}\text{B}_{15}$ ribbons at the heating rate of $20^\circ\text{C}/\text{min}$

Table 1 Thermal parameters of the Fe₇₈Zr₇B₁₅ ribbons obtained from the DSC curve at the heating rate of 20 °C/min

Parameter	T_g	T_{x1}	T_{p1}	T_{x2}	T_{p2}
Temperature/°C	533.0	564.9	575.8	735.9	749.4

Based on the aforementioned discussion, the crystallization process in the whole range could be described as $\text{Am} \rightarrow \alpha\text{-Fe} + \text{Am}^* \rightarrow \alpha\text{-Fe} + \text{Fe}_2\text{B}$ with Am^* representing the residual amorphous matrix.

3.2 Crystallization mechanism of primary $\alpha\text{-Fe}$ phase

The M_s and T_c of the Aq ribbons measured by VSM were 124.3 (A·m²)/kg and 305 °C, respectively, which were higher than those of Nanoperm alloy with high iron content [12, 13]. Some researchers pointed out that the structural relaxation could attribute to the decrease of the M_s [26]. The same phenomenon was observed in the present study that the M_s reduced to 120.7 (A·m²)/kg though the ribbon was still amorphous when the Aq ribbon was annealed at 450 °C. When annealed at a lower temperature, the alloy was transformed into a stable state from the metastable amorphous state [27]. It was deemed that the structure of the alloy slightly changed in this process. However the atomic configuration and local atomic structure were different from those in Aq state, which consequently resulted in the decrease of M_s . After being annealed at 550 °C, M_s was increased to 145.6 (A·m²)/kg, which was approximately 17 % higher than that of the Aq ribbons. It was obvious that the precipitation of $\alpha\text{-Fe}$ phase in the amorphous phase matrix namely the formation of dual phase structure was favorable to the improvement of M_s . Once Fe₂B phase was precipitated from the residual amorphous matrix after the ribbons being annealed at 720 °C, the soft magnetic properties were deteriorated [8]. Hence, the

Fig. 2 XRD patterns of the Aq and annealed ribbons **Fig. 3** SEM image of Fe₇₈Zr₇B₁₅ ribbons annealed at 550 °C

annealing temperature must be centered at T_{x1} , which can avoid the deterioration of the soft magnetic properties. Figure 3 showed the precipitated dual phase structure of the FeZrB ribbons annealed at 550 °C, revealing that the nano-sized grains were embedded in the amorphous matrix. The crystallized volume fraction of $\alpha\text{-Fe}$ phase increased with annealing time, and M_s was closely related to the crystallized volume fraction of $\alpha\text{-Fe}$ phase. To shed much light on their relationship, the isothermal annealing was carried out in DSC at temperatures of 540, 550, 556, 562, 565 and 572 °C, respectively.

The isothermal DSC traces of ribbons at different temperatures were shown in Fig. 4. At 540 °C, it was found that the exothermic peak spread out gently and covered a large span along the x -axis. With increasing the isothermal annealing temperature, the exothermic peak got sharper and the time span become shortened, reflecting the occurrence of the violent reaction. The isothermal crystallization at 540 °C was so weak that the DSC curve lost smoothness in the amplification condition, and this result was neglected in the following discussion.

According to the isothermal DSC curves, the temperature–time–transformation (TTT) curves were plotted in Fig. 5. The time consisted of the onset crystallization time (t_s), the peak crystallization time (t_p) and the end crystallization time (t_f), respectively. It could be observed that the incubation time (τ) was getting longer with decreasing annealing temperature, indicating that the reaction was slowed down at lower temperature. To further probe the crystallization mechanism, the crystallization kinetics was discussed as follows.

Integrating the isothermal DSC curves up to time t , the crystallized volume fraction x as a function of time t was plotted in Fig. 6. The VSM measurement suggested that after annealed at 550 °C for 10 and 20 min the M_s values of

Fig. 4 Isothermal DSC curves of the $\text{Fe}_{78}\text{Zr}_7\text{B}_{15}$ ribbons at different temperatures

Fig. 5 TTT curves of the first-step crystallization

the Aq ribbon were 138.2 and 145.6 ($\text{A}\cdot\text{m}^2$)/kg respectively, implying that the value of M_s depends on the crystallized volume fraction of α -Fe phase. Herzer [9] described this relationship as the following formula:

$$M_s = xM_{\alpha\text{-Fe}} + (1 - x)M_{\text{amor}}, \quad (1)$$

where x is the volume fraction of α -Fe phase, and $M_{\alpha\text{-Fe}}$ and M_{amor} are saturation magnetizations of α -Fe phase and the amorphous ribbon, respectively. The volume fraction-time curves showed a sigmoid shape (S-type), as shown in Fig. 6. In these S-type curves, the largest slope occurred in the middle range, which was marked by arrow line. In this middle range, the volume fraction was more sensitive to the annealing time as compared to that in other time ranges, suggesting that the M_s was also more sensitive to the annealing time based on Eq. (1). Hence, the annealing time should be located in this range to control the volume fraction of α -Fe phase to optimize the soft magnetic properties. It was worth noting that although the t_p shifted to the short time with increasing the annealing temperature, the volume fraction at the annealing time of t_p remained

Fig. 6 Plots of crystallized volume fraction (x) versus time (t) of the $\text{Fe}_{78}\text{Zr}_7\text{B}_{15}$ ribbons isothermally heated at different temperatures: a 572 °C; b 565 °C; c 562 °C; d 556 °C; e 550 °C

constant at approximately 33 %, which was marked by two dashed lines parallel to x -axis in Fig. 6. To further characterize the sensitivity of the crystallization rate to temperature, activation energy $E_c(x)$ of crystallization was evaluated based on Arrhenius equation [28]:

$$t(x) = t_0 \exp(E_c(x)/(RT)), \quad (2)$$

where $t(x)$ the time-consuming consistent with x , t_0 a time constant, $E_c(x)$ the local activation energy, R the universal gas constant, and T the isothermal temperature. The plot of $\ln t(x)$ against $1/(RT)$ yields a straight line with a slope of $E_c(x)$. Then the relationship between the local activation energy and crystallized volume fraction was formulated in Fig. 7.

Figure 7 showed that $E_c(x)$ decreased with increasing the crystallized volume fraction, which could be classified as three stages: $x < 10$ %, $10 \% < x < 90$ % and $x > 90$ %. In the initial stage with $x < 10$ %, the higher activation energy suggested a higher energy barrier of crystallization. Once the crystallization was initiated, the activation energy was dropped rapidly. $E_c(x)$ was slightly decreased at the second stage with $10 \% < x < 90$ %, implying that the crystallization proceeded stably in this region. Referring to Fig. 7, the stable decrease of the activation energy was corresponding to the dramatically increasing of the crystallized volume fraction. When $x > 90$ % at the last stage, the energy barrier dropped rapidly. However the variation of sigmoid curves in Fig. 6 was not consistent with this tendency. The linear slope of sigmoid curves became flat again and the reaction slowed down at the end of the crystallization process. It was known that the amorphous structure was in a metastable high energy state. During the crystallization process, the structure became more stable [27]. It became more difficult for the following reaction in the residual amorphous matrix. Hence, the reaction slowed down in the final crystallization

Fig. 7 Relationship between local activation energy $E_c(x)$ and volume fraction x of the first-step crystallization

stage, and the nucleation transformed into growth gradually.

The crystallization mechanism can be reflected by Avrami exponent n [29]. The value of n reflects the nucleation and growth modes during the crystallization process. Different nucleation and growth modes can result in distinct crystalline structures. As a result, the change of M_s is not only related to the volume fraction of α -Fe phase, but also to the crystalline structure. The n value could be deduced from the JMAK equation, which is based on the following assumptions [29]:

- (i) Nucleation and growth occurs in isothermal crystallization conditions.
- (ii) Homogenous nucleation or heterogeneous nucleation occurs at randomly dispersed particles of the second phase.
- (iii) Growth rate of new phases is only controlled by temperature and independent of time.
- (iv) Growing crystals possess low anisotropy.

JMAK equation was given by [30, 31]:

$$x(t) = 1 - \exp(-k(t - \tau)^n), \quad (3)$$

where $x(t)$ the crystallized volume fraction, τ the logarithmic time, n the Avrami exponent, and k a kinetic coefficient which is influenced by the temperature, given by [32]:

$$k = k_0 \exp(-E_c(x)/(RT)), \quad (4)$$

where k_0 is a constant, and $E_c(x)$ is the local activation energy which decreases with increasing volume fraction. According to Eq. (4), when the temperature is kept constant, the crystallization is a process with a decreasing reaction rate and the maximum rate occurs at the very beginning. As a function of the temperature, k is generally taken as a fixed value at certain T and x . Accordingly, JMAK equation can be rewritten as [30, 33]:

$$\ln(-\ln(1 - x(t))) = n \ln(t - \tau) + \text{const.} \quad (5)$$

The plot of $\ln(-\ln(1 - x(t)))$ against $\ln(t - \tau)$ will provide the n values from the slope of curves, as shown in Fig. 8. It is found that the slope of curve mainly keeps constant in the whole crystallization process until the last stage, which indicates that the nucleation and growth of primary α -Fe phase holds the same way in the crystallization process. Furthermore, local Avrami exponent $n(x)$ is applied to characterize the nucleation and growth behavior, which is given by [30]:

$$\ln(x) = \frac{\partial \ln(-\ln(1 - x(t)))}{\partial \ln(t - \tau)}. \quad (6)$$

Figure 9 shows the relationship between the $n(x)$ and the crystallized volume fraction x . It shows that the n values of the first crystallization are in the range of 1.5–2.5 in isothermal crystallization. In non-isothermal crystallization these values are in the range of 1.6–3, which reflects the

Fig. 8 JMAK plots of $\ln(-\ln(1 - x(t)))$ versus $\ln(t - \tau)$ at different temperatures of the first-step crystallization

Fig. 9 Local Avrami exponent $n(x)$ versus crystallized volume fraction x at different temperatures of the first-step crystallization

different crystallization mechanism in the various annealing processes [24]. At the annealing temperature of 550 °C, the $n(x)$ value keeps constant at 2. It clearly reveals that the crystallization mechanism is a diffusion-controlled surface crystallization mode. During this process, nucleation rate decreases with annealing time. When x increases to 98 %, the JMAK equation does not work because the above-mentioned assumptions are no longer satisfied. Since the crystallization mechanism does not change at 550 °C during the whole crystallization process, the higher M_s of the ribbon annealed for 20 min than that annealed for 10 min can be attributed to the increasing volume fraction of α -Fe phase.

The $n(x)$ values at 556 °C were similar to those at 550 °C in the initial stage. But for other annealing temperatures, the values of $n(x)$ were slightly higher than that at 550 °C, which implied that the nucleation rate was higher at higher annealing temperatures. With increasing crystallized volume fraction, the $n(x)$ values decreased gently, suggesting a reduction of nucleation rate. Although the mechanism of nucleation and growth remained unchanged, the nucleation rate decreased rapidly at higher temperatures. When x approached approximately 60 %, the local Avrami exponents at different temperatures intersected at the same value point (see Fig. 9). This might indicate that the nucleation rate kept the same. The maximal reduction of $n(x)$ was achieved at 572 °C. The value of $n(x)$ was lower than 1.5 when x was around 75 %, reflecting that the nucleation rate was equal to zero. As discussed above, $n(x)$ decreased with increasing isothermal annealing temperature; the crystallization process was slowed down in the final stage; the crystallization mode was changed from nucleation to growth gradually. Due to the lack of free point position for nucleation, the energy mainly supports grain growing. Therefore, the heat treatment should be carried out at lower temperature nearby T_{x1} . Meanwhile, the annealing time should be correspondingly shortened when the temperature increases.

4 Conclusion

The crystallization process of amorphous ribbon $\text{Fe}_{78}\text{Zr}_{17}\text{B}_{15}$ was investigated with XRD and DSC. The crystallization mechanism of primary α -Fe phase was studied by DSC at isothermal conditions. The main results are summarized as follows:

- (i) The M_s and T_c of Aq ribbon were 124.3 (A·m²)/kg and 305 °C, respectively. The crystallization proceeds in two distinct steps: amorphous (Am) $\rightarrow$ α -Fe + residual amorphous (Am*) $\rightarrow$ α -Fe + Fe₂B. Annealing at 550 °C resulted in the increase of M_s by 17 % due to the formation of a dual phase structure.
- (ii) The local activation energy $E_c(x)$ decreased with the increase of crystallized volume fraction, implying a stronger crystallization tendency as soon as the initial crystallization occurred.
- (iii) The crystallization of primary α -Fe phase was dominated by diffusion. It was initiated by surface nucleation and its growth began with a decreasing nucleation rate with a local Avrami exponent $n(x)$ in the range of 1.5–2.5. In order to obtain the dual phase structure with excellent soft magnetic properties, the heat treatment should be carried out at a temperature about 20 °C lower than T_{x1} and the annealing time should be correspondingly shortened while the annealing temperature increased.

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