Magnetothermal Analysis of Fe-Cu-Nb-V-Si-B Alloy

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Thermomagnetic curves ($\sigma_s$–$T$ curve) of Fe$_{72.7}$Cu$_{1}$Nb$_2$V$_{1.8}$Si$_{13.3}$B$_9$ alloy annealed at different temperatures (490–600°C) for 1h were investigated by magnetic analysis. Based on the $\sigma_s^{\alpha}$–$T$ curve and X-ray diffraction analysis, the changes in volume fraction, saturation magnetization and Curie temperature of $\alpha$-Fe(Si) phase and residual amorphous phase with annealing temperatures as well as the correlation between them were determined. The dependence of magnetic coupling between $\alpha$-Fe(Si) grains on volume fractions of each phase was also discussed.

Key words: Fe-Cu-Nb-V-Si-B alloy, $\alpha$-Fe(Si) phase, residual amorphous phase, saturation magnetization, Curie temperature

1. Introduction

Fe-based nanocrystalline alloys founded by Yoshizawa et al.\(^1\) have excellent soft magnetic properties (both high saturation induction like Fe-based metallic glasses and high permeability like Co-based metallic glasses and Permalloy). Fe$_{73.5}$Cu$_{1}$Nb$_{2}$Si$_{13.3}$B$_{9}$ is a typical example of the alloys, because it exhibits the best soft magnetic properties. However the high brittleness of as-quenched alloy is a major hindrance to the wide-spread applications of the alloy. It has been shown that the partial replacement of Nb by V in Fe-Cu-Nb-Si-B alloys could suppress the as-quenched embrittlement\(^2\) and better soft magnetic properties can also be obtained by appropriate nanocrystallization annealing\(^3\). X-ray diffraction has shown that the structure of nanocrystalline alloy annealed at 490–600°C is similar to Fe$_{73.5}$Cu$_{1}$Nb$_2$Si$_{13.3}$B$_9$ nanocrystalline alloy, composed mainly of b.c.c $\alpha$-Fe(Si) solid solution and residual amorphous phase. For such a dual-phase alloy, the magnetic properties of it should be determined not only by the magnetic properties of each phase but also by the volume fraction of each phase.

Hence determination of the relation between annealing temperature during crystallization and the magnetic properties of each phase as well as its volume fraction is helpful not only to understand deeply the origin of excellent soft magnetic properties but also to determin the optimum heat treatment conditions.

2. Experimental detail

Amorphous alloy strip of Fe$_{72.7}$Cu$_{1}$Nb$_2$V$_{1.8}$Si$_{13.3}$B$_9$ was prepared in air by single-roller melt spinning method. The as-quenched samples were annealed at different temperatures for 1h in protecting argon atmosphere and then measured for $\sigma_s$–$T$ curve by automatic recorded Farady balance. At the same time, the lattice parameters and the average grain size of $\alpha$-Fe(Si) phase were measured by X-ray diffraction. According to the data of $\sigma_s$–$T$ curve, the $\sigma_s^{\alpha}$–$T$ curve may be drawn\(^4\), which is composed of two straight lines $I_1$ and $I_2$ with different slope as shown in Fig.1. The crosspoint of the two straight lines corresponds to the Curie temperature of amorphous phase, $T_c^\alpha$. The straight line $I_1$, which is below $T_c^\alpha$, represents the changes in saturation magnetization of the alloy, $\sigma_s$, with temperature; Above the $T_c^\alpha$ the amorphous

![Fig.1 Plot of $\sigma_s^{\alpha}$–$T$ for Fe$_{72.7}$Cu$_{1}$Nb$_2$V$_{1.8}$Si$_{13.3}$B$_9$ alloy with $\beta$=0.36, $\sigma_s$: Am$^2$/kg](image-url)

phase becomes paramagnetic, so the straight line \( l_2 \) represents the changes in saturation magnetization of \( \alpha\)-Fe(Si) with temperature. The crosspoint of \( l_2 \) and horizontal ordinate represents the Curie temperature of \( \alpha\)-Fe(Si) phase, \( T_a^\alpha \). The stretching of \( l_2 \) toward ordinate can determine the product \( f_0 \alpha \sigma_0^\alpha \) of volume fraction \( f_0 \alpha \) and saturation magnetization \( \sigma_0^\alpha \) at room temperature in \( \alpha\)-Fe(Si) phase. \( \sigma_0^\alpha \) can be determined from the curve of Si content and \( \sigma_0^\alpha \) in \( \alpha\)-Fe(Si) alloys. And, in turn, the Si content in \( \alpha\)-Fe(Si) is determined by the curve of Si content and lattice parameters in \( \alpha\)-Fe(Si) alloys. From this, the volume fraction of \( \alpha\)-Fe(Si) phase, \( f_0^\alpha \), and that of amorphous phase, \( f_0^A = 1 - f_0^\alpha \), can be determined. Since the saturation magnetization of alloy, \( \sigma_0 \), can be expressed as \( \sigma_0 = f_0^\alpha \sigma_0^\alpha + f_0^A \sigma_0^A \), the saturation magnetization of amorphous phase, \( \sigma_0^A \), can be determined.

3. Experimental results and analysis

3.1 Changes of saturation magnetization with annealing temperature

Fig. 2 shows the changes in saturation magnetization of alloy, \( \sigma_0 \), and that of \( \alpha\)-Fe(Si) phase and residual amorphous phase, \( \sigma_0^\alpha \) and \( \sigma_0^A \), with annealing temperature \( T_a \). The \( \sigma_0 \) is basically unchangeable. However, \( \sigma_0^\alpha \) and \( \sigma_0^A \) are both changeable with \( T_a \). The \( \sigma_0^\alpha \) decreases with increasing of \( T_a \) up to 550°C and then keeps an unchangeable value above \( T_a \geq 550°C \). The \( \sigma_0^A \) decreases with \( T_a \) increasing in the whole annealing process. The decreasing of \( \sigma_0^\alpha \) is ascribed to the increasing of Si content in \( \alpha\)-Fe solid solution. On the other hand, the volume fraction of \( \alpha\)-Fe phase increases with \( T_a \) (see 3.2), but the B and Nb are insoluble in \( \alpha\)-Fe, so the content of B and Nb in residual amorphous phase increases, which makes \( \sigma_0^A \) decrease.

3.2 Changes of volume fraction and Curie temperature with annealing temperature

Fig. 3 shows the changes in volume fraction of \( \alpha\)-Fe(Si) phase and residual amorphous phase, \( f_0^\alpha \) and \( f_0^A \), with annealing temperature \( T_a \). It can be seen that the volume in \( \alpha\)-Fe solid solution. On the other hand, the volume fraction of \( \alpha\)-Fe(Si) phase, \( f_0^\alpha \), increases with \( T_a \) and volume fraction of residual amorphous phase, \( f_0^A \), correspondingly decreases with \( T_a \) increasing. When \( T_a = 510°C \), the proportion of volume fraction of \( \alpha\)-Fe(Si) phase and residual amorphous phase is about 1:1. When \( T_a = 550-570°C \), the volume fraction of \( \alpha\)-Fe(Si) phase, \( f_0^\alpha \), is about 62-68. The magnetic measurements have shown that the softest magnetic behavior was obtained for the alloy annealed at this temperature range.

Fig. 4 shows the Changes in Curie temperature of \( \alpha\)-Fe(Si) phase and the residual amorphous phase, \( T_a^\alpha \) and \( T_a^A \), with \( T_a \). The \( T_a^\alpha \) decreases a little with \( T_a \) increasing up to 550°C and then keeps an invariable value. However, \( T_a^A \) decreases with \( T_a \) increasing in the whole annealing process. This may be due to the fact that \( f_0^A \) increases with \( T_a \) which makes the Nb content of the residual amorphous phase increase.
Fig. 4 The Curie temperatures of $\alpha$-Fe(Si) phase $T_C^\alpha$ and residual amorphous phase $T_C^r$ vs annealing temperature for Fe$_{72.7}$Cu$_1$Nb$_2$V$_{1.8}$Si$_{13.3}$B$_9$ alloy

4. Discussion

According to Herzer's theory$^9$, the effective magnetic anisotropy of Fe-Cu-Nb-V-Si-B nanocrystalline alloy, $<K>$, is connected with the grain size of $\alpha$-Fe(Si) phase D. When D is less than the ferromagnetic exchange length, $L_{EX}$, $<K>$ can be expressed as

$$<K> \approx D^6$$

(1)

It can be seen from eq.(1) that the finer nanometer grain size will result in smaller $<K>$. But the promise of eq.(1) is that there must be a strong ferromagnetic coupling among these finer nanometer grains. For dual-phase Fe$_{72.7}$Cu$_1$Nb$_2$V$_{1.8}$Si$_{13.3}$B$_9$ nanocrystalline alloy, the strong ferromagnetic coupling of nanometer grains is produced via the residual amorphous phase. Apparently the volume fraction of residual amorphous phase would play an important role in the intergranular ferromagnetic coupling. Because the lower $f^s$ corresponds to a higher $f^r$, in which the distance of nanometer grains become larger, the ferromagnetic coupling of grains becomes weaker. On the other hand, the higher $f^s$ corresponds to a short distance of nanometer grains which makes the ferromagnetic coupling of grains stronger. For example$^9$, when annealed at lower temperature(490°C), the finer nanometer grain size(9nm) can be obtained for Fe$_{72.7}$Cu$_1$Nb$_2$V$_{1.8}$Si$_{13.3}$B$_9$ alloy but $f^s$ is also small, so the permeability of alloy is not high. With $T_a$ increasing $f^s$ increases which makes the ferromagnetic coupling stronger, so the permeability also increases. But further increasing of $T_a$ will make the grain size larger which, according to eq.(1), results in the $<K>$ increase, so the permeability will decrease. Our experimental results show that when $f^s$ increases D also increases, but $f^r$ does not have a linear corresponding with D. This suggests that by appropriate nanocrystallizing annealing for adjusting D and $f^r$, the optimum ferromagnetic coupling (both larger $f^r$ and finer D) can be obtained which corresponds to the optimum magnetic properties. The idea have been confirmed by quick heating nanocrystallization$^9$.

5. Conclusions

(1) The saturation magnetization of Fe$_{72.7}$Cu$_1$Nb$_2$V$_{1.8}$Si$_{13.3}$B$_9$ alloy after isothermal annealing at 490–600°C is basically invariable, however, saturation magnetization of $\alpha$-Fe(Si) phase and residual amorphous phase, $\sigma^r$ and $\sigma^s$, are all changeable with $T_a$. The $\sigma^r$ decreases with $T_a$ increasing up to 550°C and then keeps an invariable value at $T_a$=550–600°C. The $\sigma^s$ decreases with $T_a$ increasing in the whole annealing process.

(2) The volume fraction of $\alpha$-Fe(Si) phase increases with $T_a$ and that of residual amorphous phase correspondingly decreases.

(3) The Curie temperature of $\alpha$-Fe(Si) phase decreases with $T_a$ increasing up to 550°C and then keeps an invariable value at $T_a$ $\geq$ 550°C; the Curie temperature of residual amorphous phase decreases monotonously with $T_a$ increasing in the whole annealing process.

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Reference