On the Evolution of Cu-Ni-rich Bridges of Alnico Alloys with Tempering

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Abstract:

Tempering is a critical step in Alnico alloy processing, yet the effects of tempering on microstructure have not been well studied. Here we report these effects, and in particular the effects on the Cu-Ni bridges. Energy-dispersive X-ray spectroscopy (EDS) maps and line scans show that tempering changes the elemental distribution in the Cu-Ni bridges, but not the morphology and distribution of Cu-bridges. The Cu concentration in the Cu-Ni bridges increases after tempering while other element concentrations decrease, especially Ni and Al. Furthermore, tempering sharpens the Cu bridge boundaries. These effects are primarily related to the large $2C_{44}/(C_{11}-C_{12})$ ratio for Cu, largest of all elements in Alnico. In addition, the Ni-Cu loops around the α_1 phases become inconspicuous with tempering. The diffusion of Fe and Co to the α_1 phase during tempering, which increases the difference of saturation magnetization between the α_1 and α_2 phases, is observed by EDS. In summary, α_1 , α_2 and Cu-bridges are concentrated with their major elements during tempering which improves the magnetic properties. The formation of these features formed through elemental diffusion is discussed via energy theories.

Key Words:

Cu-Ni bridges; Permanent magnet; Tempering; Energy dispersive X-ray spectroscopy (EDXS); Diffusion

1. Introduction

Alnico alloys draw intensive attention because of their low cost, acceptable permanent magnetic properties and applicability to high temperature applications (~ 400 °C) [1]. Alnico alloys are composed mainly of Fe, Co, Ni, Al, Ti, Cu and Nb, and are prepared by sintering powders or casting. The permanent magnetic properties of Alnico alloys are attributed to shape anisotropy; the rod-like ferromagnetic α_1 phases, comprised mainly of Fe and Co, are distributed periodically in the non-ferromagnetic α_2 matrix, comprised mainly of Ni, Al and Ti. This essential composite structure is achieved through spinodal decomposition. The α_1 rods are formed with their axes of elongation parallel to the $\langle 100 \rangle$ direction, the easy magnetization direction. The final magnetic properties are obtained through two critical steps, thermomagnetic treatment at 800-850 °C for 10-20 minutes and low temperature tempering at 550-650 °C for 10-20 hours [2].

Spinodal decomposition occurs during thermomagnetic treatment, and the temperature and time of this step have been studied to optimize the size and shape of the α_1 phase formed [1, 3-15]. Additionally, the contents of critical elements, such as Co and Ti, were varied to improve the permanent magnetic properties and thermal stability of Alnico alloys [13]. Of the recent studies, Zhou et al. [16] reported the Cu-rich bridges might play an important role in the final magnetic properties as spacers between adjacent α_1 phases. Subsequently, we reported recently the formation and evolution of Cu-rich bridges during thermomagnetic treatment[17]. We showed that Ti fosters the conditions resulting in the formation of Cu-Ni-rich bridges, which are precipitated to decrease the elastic energy. The coarsening and thus the shape and size of α_1 phases are influenced strongly by a pinning effect due to the presence of the bridges. The Cu-Ni bridges are distributed non-uniformly and the distribution depends on the edges and shape of α_1 phases. Furthermore, Ni-Cu loops are observed around the α_1 phases, and direct contact between the Cu-Ni-rich bridges and Ni-Cu loops is observed rather than direct contact between

the bridges and the α_1 phases. Two types of Cu-Ni-rich bridges are observed, those with more Cu than Ni, and those with more Ni than Cu.

Tempering is the other critical step for final shape anisotropy, yet it has drawn much less attention. This is partly because the shape and size of the α_1 phase in Alnico is well developed during the thermomagnetic treatment, and no microstructural changes were observed during low temperature tempering with the microscopy available at the time of the earlier studies. Therefore, the underlying mechanism of the effects of temperature on Alnico magnetic properties remains unclear. Especially, when the Cu-Ni bridges were observed [16, 17], it was not clear whether the Cu-Ni bridges change during tempering, affecting the final magnetic properties. In this paper, high magnification energy-dispersive X-ray spectroscopy (EDS) is used to observe microstructural changes during tempering, including the evolution of Cu-Ni bridges and the subtle but important changes in the distribution of atoms between the phases and bridges.

2. Experimental procedure

Bulk samples (wt%: Fe32.3-Co36.9-Ni13.5-Al7.2-Ti4.1-Hf2.1-Cu3.0-Nb0.9) were prepared in a partial pressure of argon by non-consumable electrode arc melting. A Ti button was melted before each sample to getter any residual oxygen. Bulk elements (purity>99%) were melted on a water-cooled copper hearth and then flipped and remelted at least thrice to ensure the elements were well mixed. Samples were drop cast into a water-cooled 3-mm-diameter cylindrical copper mold and cut into 3 mm diameter by 3 mm long cylinders. The as-cast samples were heat treated with typical procedures for Alnico. First, the samples were solutionized at 1250 °C followed by water quenching. Then the samples were thermomagnetically treated at 800 °C for 10 minutes in a 3 T magnetic field along the cylindrical axis. The aim of this work is to compare the difference

in microstructures and magnetic properties of samples before and after tempering, so sample A is untempered and sample B is tempered at 650 °C for 4 hours, 600 °C for 6 hours, and finally 550 °C for 10 hours. Note that multi-step tempering improves the magnetic properties more effectively than one step tempering [18].

Magnetization hysteresis measurements were performed in a Quantum Design superconducting quantum interference device (SQUID) magnetometer by sweeping the magnetic field from -3 T to 3 T at room temperature. The coercive magnetic field (H_c), remanent field (B_r) and the maximum of energy product $(BH)_{\max}$ are extracted from the magnetization hysteresis.

Structural and compositional properties were analyzed via transmission electron microscopy (TEM). The TEM foil (rectangular, length $\sim 20 \mu\text{m}$ x width $\sim 10 \mu\text{m}$) was prepared using a focused ion beam (FIB) lift-out technique in a FEI Quanta 3D FEG dual-beam instrument in the transverse orientation. α_1 phases form along the magnetic field direction during thermomagnetic treatment, the [001] direction, so the samples are observed along the magnetic field direction, i.e. the transverse orientation. The FIB cut specimens were first thinned by Ga⁺ ions at 30 keV, followed by 5 keV for final thinning. EDS analysis was performed in an aberration-corrected FEI Titan G2 microscope with an X-FEG source and ChemiSTEM technology, operated in the scanning transmission electron microscopy (STEM) mode at 200 keV. The electron beam probe size is 0.1 nm in a convergent angle of 21 mrad. EDS maps of regions of interest were acquired for 900 s. An annular dark-field detector with 100 mm camera length, corresponding to a collection angle of 77-300 mrad, was used to obtain STEM high-angle annular dark-field (HAADF) images. The image contrast in HAADF is proportional to the atomic number (Z), the material density or the sample thickness, enabling us to detect different phases in the microstructure[19].

3. Results

3.1 Magnetic properties

Table 1 summarizes the magnetic behavior of the two samples, A before tempering and B after tempering, including H_c , B_r and $(BH)_{max}$. With tempering, H_c increases from 1100 Oe (A) to 1300 Oe (B). B_r is relatively low, due to the random grain orientations. If directionally solidified, B_r increases. With this change, $(BH)_{max}$ becomes similar to Alnico 9 [16].

3.2 Microstructure

Figure 1 shows HAADF images of samples A and B observed along the [001] direction, parallel to the magnetic field during thermomagnetic treatment. Three distinguishable areas are observed in each sample: the hard magnetic α_1 phase appears as bright areas of primarily Fe and Co, the soft magnetic α_2 phase is seen as a dark matrix of primarily Ni and Al, and the Cu-rich areas, which are small, bright circles appearing as precipitates bridging adjacent α_1 phases. The average size of the α_1 phase in samples A and B are same, about 26 +/- 4 nm. The distribution of the Cu-rich bridges is non-uniform and depends on the edges and shape of the α_1 phases[17]. No obvious microstructural changes due to tempering are observed at this magnification.

Figure 2 shows high magnification HAADF images and the corresponding EDS compositional maps of samples A and B, focusing on the α_1 phase. Samples A and B have some common features. The main element of α_1 , Fe, has a larger contrast difference between α_1 and α_2 than Co; similarly, the main element of α_2 , Ni, has a larger contrast difference between α_1 and α_2 than Al and Ti. Significant differences between samples A and B are also observed. There are no obvious Ni-Cu loops around the α_1 phases in B, whereas they are present in A. The Cu contrast between α_1 and α_2 in B is smaller than in A, and for the bridge areas, Cu has a larger contrast between the bridges and α_2 in B than in A. Co and Ti form sharper bridges boundaries in B than in A, and Ni

and Al are rare in the bridges in B but are the same as in α_2 in A. Note that the Hf distributions are not considered because the major peaks of Hf and Cu overlap in the EDS spectrum. Electron energy loss spectroscopy (EELS) is also ineffective for verifying the Hf distribution because the composition is too low.

Figure 3 shows EDS line scans from the α_1 to the α_2 phases. Although the compositions of different elements cannot be compared through the line scan intensities, spatial variations of the concentration of a specific element is revealed. The line scans shows that Fe has a larger intensity difference between α_1 and α_2 than Co, and Ni has a larger intensity difference between α_1 and α_2 than Al and Ti in both A and B. This is the evidence for the common features, but it does not reveal the possible changes of elemental distributions in α_1 and α_2 due to tempering. Therefore, quantitative EDS in Table 2 summarizes elemental compositions in α_1 and α_2 phases before and after tempering. For the different features, the small bumps in the Ni and Cu curves in the line scans in B (Figure 3(c,d)), compared to the peaks of Ni and Cu in A (Figure 3(a,b)), is the evidence that the Ni-Cu loops disappear due to tempering. The contrast in Cu between α_1 and α_2 before and after tempering cannot be differentiated in the line scans due to the low intensity difference.

Quantitative EDS in Table 2 summarizes the compositions of each element before and after tempering. Shown are the average values with identically sized areas in each phase along the [001] direction. The TEM foil thickness ensures only one layer of each phase exists along the [001] direction. Table 2 shows that tempering increases the Fe and Co in the α_1 phase and the Al, Ni and Ti in the α_2 phase, so the diffusion direction is from α_2 to α_1 for Fe and Co and from α_1 to α_2 , for Ni, Al and Ti.

Figure 4 the HAADF images and the corresponding EDS line scans at higher magnification to study changes in the Cu-Ni bridges due to tempering. Figure 5 shows the corresponding EDS elemental maps. Again, some features are similar; both samples have similar bridge size and the bridges in both have one preferred direction, as the long axis lies along the connection of two α_1 phases in Figure 4(a, b). Furthermore, the bridges do not contact with two adjacent α_1 phases, but exhibit gaps with a high concentration of Ni between α_1 and the bridges, Figure 4(c,d). There are also some clear differences between samples A and B shown in the line scans along the [100] direction. In the horizontal direction, Ni has two very small peaks along the bridge boundary in B (Figure 4f) whereas there are no obvious Ni peaks in that area of sample A (Figure 4e). In both directions, the Cu concentrations increased significantly with tempering (Figure 4(c-f)). The EDS maps of Cu-Ni bridges shown in Figure 5 also show changes due to tempering. In B, relative to A, the Cu contrast between bridges and α_2 is large and the bridge boundaries are sharper in Co and Ti, and the Ni and Al distributions are smaller.

4. Discussion

The similarity of HAADF images of A and B in Figure 1 indicates that the size and distribution of Cu bridges, formed during the thermomagnetic treatment, and the α_1 and α_2 phases, did not change with tempering. Therefore, the improvement of magnetic properties is due to changes in the morphology or elemental distributions observable only at higher magnification. These were shown in Figures 2-5 via the EDS maps and line scans. The disappearance of the Ni-Cu loops, smaller Cu contrast between α_1 and α_2 , and larger Cu contrast between bridges and α_2 in B indicate that Cu diffuses from α_2 to the bridges during tempering. The larger intensity difference between Cu and other elements in the bridge area, the well-defined Ni peaks around the bridge in sample B (Figure 4), compared with the smaller intensity difference between Cu and other

elements and the undefined Ni peaks in sample A, indicate that tempering facilitates the growth of Cu bridges and the diffusion of Ni from bridges to the bridge boundaries. The small distributions of Ni and Al in the bridges in B (Figure 5) compared to the almost uniform distributions of those in A also provide evidence for these features.

The evolution of Cu-Ni-rich bridges during the tempering needs to be discussed based on these new features in the context of the recent results on the formation and evolution of Cu-Ni-rich bridges during the thermomagnetic treatment[17]. At the stage of the thermomagnetic treatment, Cu-Ni loops form because it is thermodynamically favorable to concentrate Cu in α_2 at the α_1 boundary and because Ni has high solubility in Cu. This also promotes the coarsening of α_1 and spinodal decomposition. When the samples are tempered at 550 °C - 650 °C, the temperature is not sufficiently high to activate the spinodal decomposition, so the α_1 phases cannot elongate, coarsen or coalesce, and the formation of Cu loops is not thermodynamically favored. Thus, the Cu and Ni loops disappear via diffusion from the loops to α_2 during tempering.

During the thermomagnetic treatment, when the distance between adjacent α_1 phases is small enough with the coarsening of α_1 , Cu and Ni in α_2 diffuse to the bridges to decrease the elastic energy[17] and Cu-Ni bridges form. According to Cahn's theory, if $2C_{44}-C_{11}+C_{12}>0$, the waves parallel to the $\langle 100 \rangle$ directions in the $[20]$ planes in cubic crystals are favored because the elastic coefficient Y is smallest [17, 21, 22]. For both Cu and Ni, $2C_{44}/(C_{11}-C_{12})>1$, which is even larger than Fe, so Cu and Ni have the lowest elastic energy in $\langle 100 \rangle$ and decompose most rapidly with this orientation with the smallest wavelength. During tempering, although no magnetic field is applied, the intermediate temperature promotes Cu diffusion from α_2 to the bridges to decrease the elastic energy, because the growth of Cu-Ni bridges is dominated by elastic energy rather than magnetic energy[17]. The sizes of α_1 , α_2 and bridges are fixed during

thermomagnetic treatment as shown in Figure 1, so the Cu concentration in the bridges increases, as shown in Figure 4, with decreasing Cu concentration in Ni-Cu loops and α_2 (Figures 2-3). One special feature is the high ratio of the Cu concentration to the Ni concentration after tempering (Figure 4), as compared to their relatively low ratio after thermomagnetic treatment. One possible reason is the ratio of $2C_{44}/(C_{11}-C_{12})$ for Cu (3.19) is larger than for Ni (2.63)[17], so Cu is more favorable than Ni and dominates in the bridges after the lengthy tempering. The Cu atoms concentrating in the bridges “squeeze” other elements out, resulting in the smaller distribution of Ni and Al in the bridges, and the sharper bridge boundaries in Co and Ti in the EDS maps in Figure 5. The very small Ni peaks in Figure 4(d,f) are due to its thermodynamically favorable concentration in the bridges, even though Cu atoms are squeezing them out. In previous work on thermomagnetic treatment, we found two types of Cu-Ni bridges, those with more Cu than Ni and those with more Ni than Cu. Here we found only one type of bridge, with very high Cu concentration, after tempering, which may be attributed to this predomination of Cu atoms. These bridges with very high Cu concentration and sharp boundaries may be one reason for the better magnetic properties after tempering, because the bridges contain less magnetic elements (Ni and Co) and increase the difference of saturation magnetization between bridges and the other two main phases. This interpretation, however, remains speculative.

5. Conclusion

In this paper, the microstructures of Alnico alloys before and after tempering were compared. Tempering changes the elemental distribution in Cu-rich bridges rather than the morphology and size of these bridges. The tremendous increment of Cu concentration in the bridges indicates that tempering facilitates the growth of the Cu bridges and supplies us the information to discuss the effect of tempering on the Cu-Ni bridges. The predominance of Cu in the bridges is attributed to

the largest ratio of $2C_{44}/(C_{11}-C_{12})$ of all elements present. The loops around α_1 become obscure with diffusion during tempering. Furthermore, both the α_1 and α_2 phases reach their final equilibrium compositions with a maximum saturation magnetic polarization by diffusion during tempering. Although the compositional changes are not large, they are distinguishable and provide evidence for the underlying mechanisms by which tempering improves the magnetic properties of Alnico.

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Table 1: Magnetic properties of samples A and B

Sample	H_c (Oe)	B_r (T)	$(BH)_{\max}$ (kJ/m ³)
A	1100	0.53	12
B	1300	0.58	16

Table 2 Compositions of α_1 and α_2 in untempered (A) and tempered (B) samples

Elements	Wt %	Untempered A		Tempered B	
	Nominal	α_1	α_2	α_1	α_2
Fe	32.3	46.6	12.6	49.1	11.7
Co	36.9	41.8	37.2	42.9	32.2
Ni	13.5	6.1	22.2	4.1	25.8
Al	7.2	2.8	9.5	2.2	13.5
Ti	4.1	0.9	9.2	0.6	8.1
Cu	3.0	1.4	3.5	1.0	4.1
Hf	2.1	0.1	2.6	0.0	1.5
Nb	0.9	0.3	3.2	0.1	3.1