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Aging characteristics and properties of Fe-16Cr-2.5Mo-1.0Cu damping alloy

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ABSTRACT

The precipitating behavior of Cu-riched particles during isothermally aging and its effect on hardness and damping capacity of Fe-16Cr-2.5Mo-1.0Cu damping alloy have been investigated by atom probe tomography (APT) and scanning transmission electron microscopy (STEM). The results show that aging at temperature range from 400 °C to 700 °C has minor influence on grain size. During aging at temperature below 400 °C, the supersaturated Cu dose not precipitate out. While aging temperature is 450 °C, a relatively small number of Cu-riched particles can be observed and the particle radius is small (1.5 ± 0.56 nm). With a further increasing aging temperature, the Cu-riched particles increase to 3.21 ± 0.52 nm at 550 °C and further grow to 12.88 ± 2.02 nm at 700 °C. As compared with 450 °C, the particle number increases significantly at 550 °C, and then decreases at 700 °C. Due to the stable grain size and no precipitate, the hardness keeps constant below 400 °C. With a further increasing aging temperature, the hardness increases gradually and reaches peak value aging at 600 °C due to the increase of Cu-riched particle number. The damping capacity significantly depends on particle. Once there appear Cu-riched particles, the damping capacity decreases obviously. During aging, both hardness and damping capacity of the alloy can keep stable when temperature is below 400 °C.

1. Introduction

As a typical magnetic-mechanical damping alloy, Fe-Cr based ferritic alloys have attracted much attentions in recent years [1–6]. This kind alloys have not only the high damping capacity due to the irreversible mobility of magnetic domain wall [7–10], but also the relative high strength and corrosion resistance induced by the solid solution strengthening of high Cr content (13–25 wt%) [11,12]. Therefore, Fe-Cr based damping alloys are expected to be used for reducing vibration and noise in many engineering fields [13,14]. Generally, high temperature annealing at 1000–1200 °C for 0.5–1 h is necessary for magnetic-mechanical damping alloy to obtain the high damping capacity, while its strength is reduced simultaneously owing to the coarsening of ferrite grains [8,15]. Hence, researches [11,12,15] to develop high damping and high strength Fe-Cr based damping alloys have arisen great attention. Addition of Mo or Al in Fe-Cr damping alloy has been found to increase both damping capacity and strength, and the Fe-Cr-Mo and Fe-Cr-Al ternary damping alloys have been developed [8,16]. In order to further improve their performance, quaternary alloys such as Fe-13Cr-4Al-0.5Mo and Fe-13Cr-5.5Al-0.5Cu alloys have also been developed [11,12]. Recently, Hu et al. [15,17] found that damping capacity and strength for Fe-16Cr-2.5Mo alloy with 0.5–1 wt% Cu are improved compared with the Fe-16Cr-2.5Mo ternary alloy. The possible reasons are attributed to the modification of magnetic domain structure and the grain refinement induced by Cu addition.

As is well-known, Cu has limited solubility in the ferritic alloys and can be used to increase their strength by precipitation strengthening [18,19]. During isothermally aging, nanoscale Cu-riched particles precipitate in the ferritic matrix and can exhibit different structure types from bcc Cu to fcc ε-Cu with rise of aging temperature and time [20]. In our previous study [15,17], it is found that Cu is supersaturated in as-annealed Fe-16Cr-2.5Mo-(0.5–1.0)Cu damping alloy. As a structural-function material, this quaternary alloy may be used in the service environment with a certain temperature. Therefore, it is necessary to investigate the influence of aging temperature on the stability of microstructure and properties, including damping capacity and strength. Since Cu-riched particles is small and coherent with ferritic matrix [21], it is difficult for TEM to observe these particles. Atom probe tomography (APT) is a unique powerful tool and is used in this investigation to analyze the morphology, size, number density and compositions of Cu-riched particles with a sub-nanoscale spatial resolution [22,23]. It is of both fundamental and practical importance to elucidate the correlation between precipitating of Cu-riched particles and properties for Fe-16Cr-2.5Mo-1.0Cu damping alloy.

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Table 1
Chemical compositions of Fe-16Cr-2.5Mo-1.0Cu alloy.

	Fe	Cr	Mo	Cu	C	N	O	S	P
wt%	80.53	15.92	2.52	1.01	0.006	0.003	0.003	0.002	0.006
at%	80.57	17.10	1.47	0.89	0.030	0.01	0.01	0.0003	0.01

2. Material and experimental procedures

A 25 kg ingot of Fe-16Cr-2.5Mo-1.0Cu alloy was prepared by vacuum induction melting and was hot forged, and then hot rolled to 12 mm thick plate. The chemical compositions of the alloy are listed in Table 1. The impurity elements such as S, P, O and N are controlled as low as possible. The as-rolled plate was annealed at 1000 °C for 1 h followed by air-cooling at room temperature and then were isothermally aged at different temperatures from 350 °C to 700 °C for 2 h. TA Q800 dynamic mechanical analyzer (DMA) with dual cantilever vibration mode was used to measure damping capacity. The damping specimens with dimensions of 60 mm × 8 mm × 0.8 mm were cut by electric sparking machine, and then ground carefully on each side with SiC paper through 600 grit. The damping curves were tested at temperature of 30 °C and vibration frequency of 1 Hz. Hardness was measured as an indicator for strength. Considering larger grain size caused by high annealing temperature, Brinell hardness tests were performed under a 3000 kg load with a loading duration of 12 s by HB-3000B tester with the 10 mm diameter. Each reported hardness value is an average of at least five measurements on each specimen.

The optical microstructure on the section perpendicular to the rolling direction was carried out on Olympus optical microscope (GX51). The sample was polished to a final surface finish of 1 µm SiC and etched with a solution containing 5 g copper sulfate, 20 mL hydrochloric acid and 20 mL distilled water. Scanning transmission electron microscopy (STEM) observation was performed on JEOL JEM-2100F microscope with energy dispersive spectroscopy (EDS). The STEM specimens were cut from aged samples and then ground to ~ 45 µm in thickness. The disk 3 mm in diameter were punched and twin-jet electropolished using a solution 10 vol% perchloric acid in ethanol. Atom probe tomography (APT) was used to determine the size, number density and compositions of Cu-riched particles. APT experiments were performed using a Cameca local-electrode atom-probe (LEAP) 5000XR. A two-step electropolishing procedure was used to prepare the APT samples [18,21]. The tip blank with dimensions of 0.5 mm × 0.5 mm × 20 mm was first electrically polished with 25 vol% perchloric acid in acetic acid solution using a direct current voltage of 10–20 V and the final polishing was done with 5 vol% perchloric acid in butoxyethanol acid solution at 9–15 Vdc. The APT samples were analyzed in laser mode with a specimen temperature 50 K under ultrahigh vacuum, a pulse repetition rate of 200 kHz, an ion collection rate of 1% ions per field evaporation pulse and a pulse energy of 40 pJ. The software IVAS™ 3.8 was used for 3D reconstruction, composition analysis, creation of isoconcentration surfaces and NND (Nearest neighbor distribution) analysis [24,25]. The spherical volume equivalent radius R_V and number density N_V of Cu-riched particles [19,26] is given by: $R_V = \left(\frac{3N_{atoms}}{4\pi\rho_{th}\eta}\right)^{1/3}$ and $N_V = \frac{N\rho_{th}\eta}{N_{tot}}$, where N_{atoms} is the number of atoms detected within the analyzed particle, ρ_{th} is the theoretical atomic density of the particle, which is equal to 84.3 atoms nm^{-3} for this alloy, and $\eta = 0.52$ is the estimated detection efficiency, N is the total number of particles identified in the reconstructed volume and N_{tot} is the total number of atoms detected in the reconstructed volume.

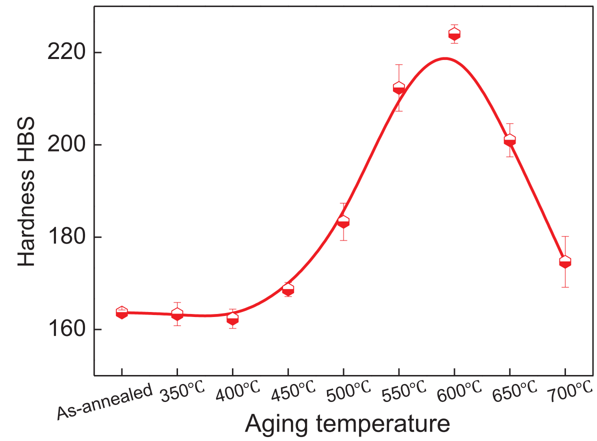

Fig. 1. Variation of hardness with aging temperature for Fe-16Cr-2.5Mo-1.0Cu damping alloy.

3. Results

3.1. Hardness and damping capacity

Hardness measurement was conducted to evaluate the aging precipitate response of experimental alloy. The Brinell hardness at different aging temperature is shown in Fig. 1. The Fe-16Cr-2.5Mo-1.0Cu alloy shows a hardness of ~ 164 HBS in the as-annealed condition. When aging temperature is below 400 °C, the hardness keeps steady, which indicates that there is no change in the microstructure. After aging at 450 °C, the hardness increases slightly to ~ 169 HBS, which suggests that Cu-riched particles begin to precipitate in the matrix. With the rise of aging temperature, the hardness increases fast and reaches the peak value ~ 224 HBS at 600 °C. The hardness declines sharply with further increasing of aging temperature and reaches to ~ 175 HBS at 700 °C.

The damping curves were measured by DMA to investigate the damping behavior of the experimental alloy. The as-annealed sample exhibits the classical magnetic-mechanical damping behavior and its maximum damping value Q^{-1}_{max} is 0.050 as shown in Fig. 2a. For the sample aged at 550 °C, the damping capacity obviously reduces and Q^{-1}_{max} decreases to 0.031. This result suggests that the aging has significant influence on the damping behavior. Fig. 2b shows the variation of maximum damping value Q^{-1}_{max} of aging samples with temperature from 350 °C to 700 °C, where Q^{-1}_{max} is an average of three measurements. As compared with as-annealed sample, it can be seen that there is no change of the maximum damping value Q^{-1}_{max} for samples aged below 400 °C. With further increasing aging temperature, the maximum damping capacity Q^{-1}_{max} drops sharply until to the low platform value at 550 °C aging. It is interesting to note that the onset of decrease for Q^{-1}_{max} at 450 °C is corresponding to the initial increase of hardness (Fig. 1), which may be related to the precipitation of Cu-riched particles in the matrix.

3.2. Microstructure observation

After isothermally aging, the Fe-16Cr-2.5Mo-1.0Cu alloys are fully ferrite with polygonal shape as shown in Fig. 3. Fig. 3d shows that the grain size is almost independent of aging temperature in the range of 64–71 µm. Fig. 4 shows STEM images of Fe-16Cr-2.5Mo-1.0Cu alloy aged at different temperatures and EDS analysis of particles aging at 700 °C. No second phase is observed at 400 °C aging. Aging at 550 °C has introduced a lot of particles in the matrix as shown in Fig. 4b. Their size is small and difficult to distinguish, but from the enlarged view as shown with arrow in the insert of Fig. 4b it can be see that many particles are uniformly distributed in the α-Fe matrix. As continuing to