Remarkable improvement in damping capacity of M2052 alloy by step-cooling treatment

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Abstract
In order to efficiently produce high-damping Mn–Cu based alloys, different heat treatment processes were designed in this study, and the influence of the cooling process on the damping property of M2052 alloy was systematically investigated. The results show that the step–cooling process significantly influenced the damping performance of M2052 alloy. Compared with M2052 alloy processed using air-cooling with an internal friction (tan δ) of 0.087 at a strain amplitude $\lambda = 5 \times 10^{-4}$, the damping capacity was improved by more than 49% through the step-cooling treatment. This was mainly because of the increment of manganese content of in the Mn-rich regions, which is caused by the spinodal decomposition on the low temperature side of the miscibility gap. Meanwhile, the step–cooling treatment significantly suppressed the precipitation of $\alpha$-Mn, which reduced the negative effect of $\alpha$-Mn on the damping capacity and kept the Mn content in the Mn-rich regions at a high level. The step–cooling treatment, a novel and effective microstructure control process, provides a new method for the efficient preparation of high-damping M2052 alloy.

1. Introduction
With the development of high speed, high efficiency and automated mechanical equipment, problems with vibration and noise have become increasingly prominent. Mn–Cu based alloys have received extensive attention and research for their good mechanical properties and better high-damping properties than other damping alloys [1–4].

Among the various high-damping Mn–Cu based alloys, M2052 is a twin damping alloy with a high manganese content. When the alloy is subjected to vibration, the inelastic strain caused by the movement of martensite twins or caused by the mutual movement between the twin boundaries and martensitic phase boundaries relaxes the stress and consumes the vibration energy [3]. Because of its excellent mechanical properties, M2052 alloy has broad application prospects for vibration reduction in aerospace, shipbuilding, vehicle, and machinery manufacturing industries. However, the original as-cast M2052 alloy has a low damping capacity, and its martensitic transformation temperature ($Ms$) is lower than room temperature [6]. Many studies have confirmed that spinodal decomposition of Mn–Cu based alloys during miscible gap aging can form Mn-rich and Cu-rich microregions. This increases and the martensitic transformation temperature of the alloy, resulting in the formation of many fine thermoelastic martensitic twins in the alloy, giving it high damping properties [7, 8]. Mn–Cu based damping alloys are twin-type high-damping alloys, whose damping properties are not only related to the density of the twin interface, but also to the mobility of the interface. It is generally believed that the precipitation of $\alpha$-Mn is accompanied by a decrease in the manganese content in Mn-rich regions, which reduce the number of effective Mn-rich regions, and ultimately affects the formation of martensitic transformation twins. At the meantime, the precipitation of $\alpha$-Mn also will hinder the movement of
the twin interface, thus weakening the damping property of the alloy [9]. Ke et al found that the damping capacity of Mn–Cu based alloys decreased greatly due to the formation of \( \alpha \)-Mn after overaging [10]. Therefore, to accurately control the damping properties of the alloy, it is very important to increase the manganese content in the Mn-rich regions and avoid the emergence of \( \alpha \)-Mn, in which the reasonable heat treatment process is an important part.

Previous, research on the aging process of Mn–Cu based alloys has mainly focused on the aging temperature and aging time, while few reports have reported on the effect of the cooling process on the damping properties. Yin et al found that in order to obtain a higher damping capacity, the alloy can be slowly cooled to room temperature after solid-solution treatment, which increases the width of decomposition components [11]. However, this also introduces artificial aging when held at a high temperature for a long time, which accelerates the diffusion of interstitial atoms. It has been reported that the segregation of interstitial atoms at grain boundaries weakens the damping properties of materials during storage at room temperature [6, 12]. Therefore, long-term furnace cooling will likely promote the segregation of interstitial atoms at the interface, accelerate the reduction in the damping capacity and shorten the service life of the alloy. As mentioned above, optimizing the cooling process to improve the atomic distribution width of Mn–Cu based damping alloys during spinodal decomposition is important for improving the damping properties of Mn–Cu based damping alloys, but the mechanism is unclear.

Due to this, our research group use a the step-cooling (SC) process for M2052 alloy [13]. We then characterized and analyzed the precipitated phases and martensite of the M2052 alloy after a treatment method involving step-cooling, furnace cooling (FC), and air cooling (AC). The effect of the cooling process on the microstructure and damping properties of M2052 alloy and its mechanism were systematically studied.

### 2. Experimental

The ingots of M2052 alloy were produced in a vacuum smelting furnace in which the molten alloy was poured into the prefabricated sand mold to obtain M2052 alloy ingots with a certain shape. The alloy ingots specimens were treated with the same solid solution treatment: 900 °C for 1 h, and water cooling. Table 1 shows the details of three different aging cooling processes after solution treatment for each type of specimen, in which specimens AC-1#, AC-2#, AC-3# were aged at 435 °C for different times and then cooled in the air; specimens SC-1#, SC-2#, and SC-3# were aged at 435 °C for 4 h and then air-cooled after cooling to the designed temperature in the furnace; specimen FC-1# was aged at 435 °C for 4 h and then cooled in the furnace.

Dynamic mechanical analyzer (model Mettler Toledo 861, DMA) was used to measure the damping capacity and martensitic transformation temperature \( (M_a) \). These measurements were made in the dual cantilever mode. The DMA specimens were initially prepared with an electric spark cutter, and then thinned and polished with sandpaper, to obtain specimens with a final size of 1 mm × 10 mm × 35 mm. In the strain measurement mode, the range of the applied strain amplitude was from \( 1 \times 10^{-3} \) to \( 5.5 \times 10^{-4} \), the vibration frequency was 1 Hz, and the test temperature was 25 °C. In the temperature sweep mode, the test frequency and applied strain amplitude were 1 Hz and 2.4 × \( 10^{-4} \), and the temperature ranged from 230 to \(-120\) °C, the cooling rate was 5 °C/min. The damping capacity was evaluated by tan \( \delta \), where \( \delta \) is the lag angle between the applied strain and response stress.

X-ray diffractometer (model D8 ADVANCE, XRD) was used to measure the lattice distortion of FCT phases. Each XRD specimen possessed a dimension of 1 mm × 10 mm × 10 mm, and the surface strain of specimens was removed carefully. The Co K\( \alpha \) radiation was used in the test, the working voltage was 35 kV and the current was 40 mA. All diffraction peaks were obtained in continuous mode of 2°/min.

Transmission electron microscopy (model G2 F20, TEM) was used to observe martensitic and precipitates with an operating voltage of 200 kV. Before TEM observation, the 0.5 mm-thick foil was ground into 50 \( \mu \)m and

| Specimen | Solution       | Aging                                      |
|----------|----------------|--------------------------------------------|
| AC-1#    | 900 °C × 1 h, water cooling | 435 °C × 4 h, air cooling                  |
| AC-2#    | 900 °C × 6 h, air cooling     | 435 °C × 8 h, air cooling                  |
| AC-3#    | 435 °C × 4 h, air cooling     | 435 °C × 4 h, and then air cooling after cooling to 320 °C in furnace |
| SC-1#    | 435 °C × 4 h, and then air cooling after cooling to 210 °C in the furnace | 435 °C × 4 h, and then air cooling after cooling to 100 °C in the furnace |
| SC-2#    | 435 °C × 4 h, and then air cooling after cooling to 100 °C in the furnace | 435 °C × 4 h, furnace cooling              |
| SC-3#    | 435 °C × 4 h, and then air cooling after cooling to 100 °C in the furnace | 435 °C × 4 h, furnace cooling              |
| FC-1#    | 435 °C × 4 h, furnace cooling | 435 °C × 4 h, furnace cooling              |
then punched into a round foil with a diameter of 3 mm. Finally, the thin area was further thinned by ion-thinning at \(-20^\circ\text{C}\).

3. Results and discussion

The variation curves of \(\tan\delta\) with the strain amplitude of different specimens are shown in figure 1(a). The results show that \(\tan\delta\) increases rapidly upon increasing the strain amplitude at first and then stabilizes, which is typical characteristic of the variation of \(\tan\delta\) with strain amplitude in Mn–Cu alloys. When the strain amplitude is \(5 \times 10^{-4}\), the \(\tan\delta\) of the AC-1# specimen is the lowest (0.087) and the \(\tan\delta\) of the FC-1# specimen was 3.4% higher than the AC-1# specimen. Compared with the FC-1# and AC-1# specimens, the \(\tan\delta\) of the SC specimens was much higher, and the \(\tan\delta\) of the SC-1# was the highest (0.130), which was 49.4% higher than that of the AC-1# specimen. Thus, it can be concluded that the cooling process during aging process was strongly related to the damping capacity, and the SC treatment can significantly improve the damping performance compared with the FC and the AC treatments. Figure 1(b) compares the damping properties of the SC-1# specimen with those Mn–Cu alloys reported in other articles [3, 14–17]. It indicates that the step-cooling process can significantly improve the damping capacity of Mn–Cu alloys.

The XRD patterns of specimens after different heat treatments are shown in figure 2, which shows that all specimens were composed of \(\gamma\) phase, \(\gamma\)' phase, and \(\alpha\)-Mn phases. Generally, the height of the XRD diffraction peak is related to the content of the precipitated phase. Figure 2(b) is the local magnification of the (411) diffraction peak. Based on the height of the (411) diffraction peak, it can be inferred that compared with the SC-1# sample, more \(\alpha\)-Mn phase was precipitated in the AC-1# and FC-1# samples, which indicates that SC treatment can inhibited the precipitation of \(\alpha\)-Mn. In addition, the \(\alpha\)-Mn phase is paramagnetic, which does not improve the damping capacity, but decreases the enrichment of Mn in the Mn-rich regions.

In addition, the (220) diffraction peak shows obvious splitting and the diffraction peak of (202) \(\gamma\)' appeared, which indicates that fcc→fct transformation occurred during aging [18, 19]. Figure 2(c) is the local magnification of the (220) diffraction peak, which shows that the width and height of the (220) diffraction peaks of SC-1# and FC-1# specimens were markedly larger than those of AC-1# specimen. According to the split peak position of the (220) peak and equation (1), the degree of lattice distortion \((\alpha/\alpha−1)\) can be calculated, and the detailed results are listed in table 2.

\[
d_{\text{fct}} = \frac{1}{\sqrt{h^2+k^2+1/4l^2}}
\]

Where \(a\) and \(c\) are the lattice parameters, whose the values can be estimated by the split peak positions of the \{220\} peak in the XRD patterns, and \(d\) is the interplanar distance of the \((hkl)\) plane of \(\gamma\) phase. It is reported that if the fcc→fct transformation can occur at room temperature, the Mn concentration \((C_{Mn})\) in the Mn-rich regions should be more than 83.4%. There is a quantitative relationship between \(C_{Mn}\) and \(\alpha/\alpha\), as shown in equation (2) [17, 20]:

\[
\frac{\alpha}{\alpha} = 1.618C_{Mn}^2 - 3.317C_{Mn} + 2.638
\]
Combined with the results of table 2 and equation (2), it can be inferred that the order of \( C_{\text{Mn}} \) in the Mn-rich regions of three kinds of specimens is FC-1# > SC-1# > AC-1#. Theoretically, after spinodal decomposition, the increased segregation of manganese atoms in the alloy will increase the lattice distortion, which improves the damping properties; however, previous results have shown that this is not the case. Therefore, the degree of distortion may not fully reflect \( C_{\text{Mn}} \) in the Mn-rich regions of Mn–Cu alloys, so it is not necessarily accurate to infer the damping capacity by the degree of distortion.

Figure 3 shows the \( \tan \delta \) and storage modulus curves of the SC-1# and the FC-1# specimens as a function of temperature. Figure 3(a) shows that, upon decreasing the temperature, the \( \tan \delta \) increases first and then decreases slowly. When the temperature was lower than 75°C, the value of \( \tan \delta \) of SC-1# was much higher than that of FC-1#, which is consistent with the result of figure 1(a). However, in contrast to the results in figure 1, the value of \( \tan \delta \) at room temperature in figure 3(a) was slightly higher, which may be related to the different heating process of measurement process.

The martensitic transformation of Mn–Cu based alloys is accompanied by modulus softening, so \( M_S \) can be determined by measuring the lowest modulus during the modulus softening process. As can be seen from figure 3(b), the \( M_S \) of SC-1# and FC-1# specimens were 76.4°C and 60.1°C, respectively. As shown in table 2, a larger lattice distortion was observed after furnace cooling.

Generally, the degree of lattice distortion caused by fcc-fct phase transformation depends on the Mn concentration in the Mn-rich regions. The lattice distortion increases upon increasing the Mn content and the fcc-fct phase transition temperature (\( M_S \)) increases; thus, the damping property and service temperature of the alloy will be higher [3, 21]. According to K Tuchiya et al., increases in the phase transition temperature are caused by spinodal decomposition into Mn-rich and Cu-rich phases [22]. In light of this, it is easy to infer that the

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Table 2. Calculated lattice parameters and lattice distortion of M2050 alloy specimens under different heat treatment conditions.

| Specimen | \( d_{220} \) | \( d_{022,202} \) | \( a \) | \( c \) | Lattice distortion (\( a/c-1 \)) |
|----------|--------------|----------------|-------|-------|------------------|
| AC-1#    | 1.3229       | 1.3135         | 3.7417| 3.6891| 0.0143           |
| SC-1#    | 1.3226       | 1.3106         | 3.7409| 3.6740| 0.0183           |
| FC-1#    | 1.3231       | 1.3100         | 3.7423| 3.6693| 0.0199           |
concentration of Mn in the Mn-rich regions of SC-1 # is the highest, so that alloy has a better damping property; however, the FC-1 # showed the largest lattice distortion, and the Ms of the furnace-cooled specimen was not the highest. Therefore, it is necessary to further analyze the microstructure of the specimens.

Generally, α-Mn forms in Mn–Cu alloys due to the excessive enrichment of Mn atoms, and the lattice distortion increases upon increasing the degree of manganese enrichment. The XRD results show that the α-Mn content was the largest in the AC-1 # specimen, but its lattice distortion was the smallest, that is, it had the lowest degree of manganese segregation. This indicates that the precipitation of α-Mn was not directly related to the degree of segregation of Mn atoms in the Mn-rich regions. So, there must be other factors leading to the precipitation of α-Mn. Thus, changes in the precipitated phase appear to be related to the cooling process.

Figures 4 and 5 show the TEM micrographs of FC-1 # and AC-1 # specimens, respectively. It can be seen from figure 4(a) that the substructure of the martensitic lath in the FC-1 # specimen contained dense stacking faults with dislocation accumulation. Related studies have shown that the deformation zone caused by a high density of dislocations may provide nucleation sites for the formation of α-Mn phase during aging [5]. Furthermore, figure 4(b) shows that there are many fine bar-shaped precipitated phases with sizes of 100 ~ 200 nm in the FC-1 # specimen. The α-Mn phase also exists with a size of 300 ~ 1000 nm in the AC-1 # specimen. For Mn–Cu based alloys, α-Mn will hinder the movement of the interface and reduce the number of effective the Mn-rich regions, thus weakening the damping capacity. In addition, as mentioned in the introduction, the segregation of interstitial atoms caused by a slow cooling process can also worsen the damping capacity.

The TEM results further confirmed that although the manganese content in the Mn-rich regions was relatively small after AC treatment, α-Mn easily nucleated and grew, which indicated that the ability to hold Mn atoms in the Mn-rich region of AC-1 # was poor, and α-Mn precipitated in the Mn-rich region with a lower concentration. Among the three cooling processes, the air cooling treatment had the fastest cooling rate. The greater the free energy difference between the new and old phases, the faster the growth rate of the new phase. Accordingly, although the enrichment of manganese atoms in the AC-1 # specimen was not high, the air cooling...
process created huge undercooling and provided a sufficient driving force for the nucleation and growth of α-Mn during cooling. In contrast, the enrichment degree of manganese atoms in the microstructure of the FC-1# specimen was higher, but the size of α-Mn was smaller. Thus, it can be inferred that the FC-1# specimen had a greater ability to hold Mn atoms in its Mn-rich region than the AC-1# specimen.

Figure 6 shows the TEM micrographs of the microstructure of the SC-1# specimen, which indicates that large amounts of lamellar martensite and laths were formed by dense stacking faults from figure 6(a). It has been reported that stacking faults can not only promote the formation of martensite but also serve as a source of damping [23, 24]. In addition, there are fine SF interfaces in the SC-1# specimen, and a similar phenomenon was observed by Yin and was considered to be a reason for the improved damping capacity [25]. It is worth mentioning that in the SC-1# microstructure, no α-Mn was observed, which shows that α-Mn did not easily form and grow during the SC process, which improved the segregation degree of manganese atoms and the damping capacity.

The temperature range of the miscibility gap is from 350 °C to 550 °C in Mn–Cu alloys. The first cooling stage ranges from 435 °C to 330 °C for the SC-1# specimen, which is just on the low-temperature side of the miscibility gap. Combining Mn–Cu binary phase diagram and lever theorem, the lower the aging temperature is, the higher the manganese atom holding capacity of the manganese rich region formed by spinodal decomposition is, thus, the higher the C_{Mn} of Mn-rich regions is. According to the designed heat treating regimen, the step-cooling treatment is equivalent to aging on the low temperature side, so the Mn-rich regions with a high C_{Mn} formed by the spinodal decomposition of SC-1# shows excellent damping properties. In addition, figure 1(a) shows that the damping properties of AC-2# and AC-3# samples were lower than those of SC-1# sample, which confirms that the higher C_{Mn} in the Mn-rich regions of SC-1# was not due to prolonging the aging time. Therefore, through the step-cooling process, the manganese content in the Mn-rich regions was increased, and the holding capacity of manganese atoms in the Mn-rich regions was also improved. Because the precipitation of α-Mn is the result of excessive enrichment in the Mn-rich regions, the Mn-rich regions can

Figure 5. (a), (b) TEM micrographs of precipitates in the AC-1# specimen; (c) SEM-EDS analysis of the precipitate phase.

Figure 6. (a), (b) TEM micrographs of the SC-1# specimen.
more strongly hold manganese atoms, which reduce the precipitation tendency of $\alpha$-Mn phase. This also explains why although the enrichment degree of manganese in air-cooled samples was low, $\alpha$-Mn easily precipitated and grew.

The XRD results show that the lattice distortion degree of the FC-1# sample was the highest, but it did not display the highest damping capacity. The combined XRD and TEM results show that $\alpha$-Mn precipitates existed in the microstructure of both AC-1# and FC-1# samples. As discussed in the introduction, the existence of $\alpha$-Mn inhibits the movement of the interface, and more importantly, the formation of $\alpha$-Mn will consume the Mn-rich regions. This decreases the manganese content in the manganese-rich regions, which deteriorates the damping capacity. According to the study of Bacon G E and Shimizu K, the antiferromagnetic transformation and martensitic transformation of Mn-Cu alloys with high Mn content are two independent processes. Antiferromagnetic transformation is accompanied by fcc-fct structural transformation, resulting in lattice distortion and martensitic transformation induced by lattice distortion $[26, 27]$. At the same time, according to the study of Song Zhang et al, the formation of $\alpha$-Mn can slow down the fcc-fct phase transition and the formation of $\{110\}$ twins. According to the TEM observations, there is no obvious dislocation accumulation in the SC-1# sample compared with AC-1# and FC-1# samples, and fine twins were formed in the microstructure, indicating that the distortion caused by antiferromagnetic transformation during step-cooling treatment was more likely to be released due to the higher martensitic transformation temperature, which corresponds the previous calculation results of lattice distortion. Generally, the formation of $\alpha$-Mn consumes the content of Mn in the Mn-rich regions and slows down the martensitic transformation and twin formation, and consumes the lattice distortion energy to form twins during the martensitic transformation. This may be why the lattice distortion of the SC-1# sample is smaller than that of the FC-1# sample, but why the damping property of the AC-1# sample is better than that of the FC-1# sample.

Figure 7 shows a schematic diagram of the microstructure evolution of M2052 alloy with three different cooling processes. Among the three cooling processes, the manganese content in the Mn-rich regions of the sample treated by air cooling was the lowest and $\alpha$-Mn phases were precipitated at the same time. The manganese content in the Mn-rich regions of the sample treated by staged cooling was slightly higher than that of the sample treated by furnace cooling, and $\alpha$-Mn phases were not precipitated in the Mn-rich regions because of its strong ability to hold Mn atoms. On the contrary, $\alpha$-Mn phases were also precipitated in the furnace cooling process. At the same time, segregation of interstitial atoms may occur with long furnace cooling treatment times. The precipitation of $\alpha$-Mn and the segregation of interstitial atoms will hinder the movement of the interface and worsen the damping capacity.

Combining above results and discussion, it can be concluded that under the comprehensive influence of the increased manganese content in the Mn-rich regions and the excellent interfacial movement performance, the SC-1# specimen exhibited an excellent damping capacity.
4. Conclusions

The effect of different cooling processes on the damping capacity of M2052 alloy has been systematically investigated in this research. Based on the experimental results, the following conclusions can be drawn:

(1) Step-cooling can significantly improve the damping capacity of M2052 alloy. Compared to the M2052 alloy using the air cooling process with an internal friction (tan δ) of 0.087 at a strain amplitude $\lambda = 5 \times 10^{-4}$, the damping capacity of the step-cooling specimen was more than 49% higher. Moreover, prolonging aging time had no obvious effect on the high damping capacity.

(2) The mechanism for the improved damping capacity by the step-cooling treatment was attributed to the synergistic effect of low-temperature spinodal decomposition and the suppression of $\alpha$-Mn precipitation. Those concurrently help maintain the manganese content in the Mn-rich regions and ensure the mobility of the interfacial movement.

(3) In addition to the excessive enrichment of manganese atoms in the Mn-rich regions, the precipitation of $\alpha$-Mn also occurred due to the poor ability of holding Mn atoms in the Mn-rich region and the faster cooling rate in the miscibility gap.

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Conflict of interest

The authors declare no competing financial interests.

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