



Synergistic strengthening mechanism of cryogenic treatment for mechanical properties and damping characteristics of Mn–Cu alloy

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ARTICLE INFO

Keywords:

Mn–Cu damping alloy
Cryogenic treatment
Mechanical property
Damping performance
Strengthening mechanisms

ABSTRACT

Mn–Cu alloys are widely used as shock absorption and noise reduction materials in engineering fields. However, the strengthening of their mechanical properties often leads to a decrease in damping performance, which limits the further application of these alloys. To overcome this limitation, this paper systematically investigated the effects of different cryogenic treatment times (–196 °C) (0 h, 12 h, 24 h, 36 h) on the microstructure, mechanical properties and damping behavior of the Mn–Cu alloy. The results show that cryogenic treatment can effectively increase the internal dislocation density of the alloy, promote the refinement of the α -Mn phase, increase the proportion of the Mn-rich region, and induce the FCC→FCT martensitic phase transformation to improve the formation of twinning. As the cryogenic time increases, the tensile strength and hardness of the alloy increases, but the elongation decreases slightly. Cryogenic treatment introduces a large number of dislocations in the alloy, which makes the damping performance of the alloy exhibit a synergistic strengthening effect of twinning damping and dislocation damping mechanisms at different temperatures, providing a new method for the development of high-performance of Mn–Cu alloys.

1. Introduction

Damping alloys play an indispensable role in modern engineering. They can effectively absorb and dissipate vibration energy, which not only helps to reduce noise but also significantly extends the service life of mechanical structures [1,2]. Among various damping alloys, the Mn–Cu alloy has attracted much attention due to its excellent mechanical properties and damping performance [3,4]. However, the damping performance and mechanical properties usually show an inverse trend [5,6]. The interrelationship between these properties significantly restricts its wider application in engineering.

To overcome the above performance limitations and fully exploit the application potential of Mn–Cu alloys, the development of new heat treatment processes is particularly important. Li et al. [7] pointed out that, compared with Mn–20Cu–5Ni–2Fe(M2052) alloy processed using air-cooling, 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 α -Mn, which reduced the negative effect of α -Mn on the damping capacity and kept the Mn content in the Mn-rich regions at a high level. Dong et al. [8] conducted a comparative analysis of Mn–Cu alloys subjected to various heat treatment processes and found that the cryogenic treatment promoted the formation of twin boundaries, and improved damping capacity. In addition, cryogenic treatment promoted grain refinement, increased dislocation density, improved strength, and facilitated the improvement of mechanical properties. In the previous work of our research group [9], the response surface method (RSM) was employed to investigate the effects of cryogenic treatment combined with aging treatment on the microstructure, mechanical properties and damping performance of M2052 alloy. It was found that cryogenic treatment induced the generation of a large number of dislocations, and the subsequent aging process promoted the formation of precipitated phases and grain refinement. Moreover, the synergistic effect of cryogenic treatment and aging promoted the

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<https://doi.org/10.1016/j.jmrt.2026.02.056>

Received 12 October 2025; Received in revised form 4 February 2026; Accepted 6 February 2026

Available online 6 February 2026

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FCC-FCT martensitic transformation in the Mn-rich region, inducing the generation of more {101} twins. These microstructural changes were achieved through the synergistic effects of precipitation strengthening, fine-grain strengthening and twinning strengthening, which broke through the bottleneck of the inverse correlation between the mechanical properties and damping performance of Mn–Cu-based alloys, providing a new technical path and theoretical support for the simultaneous improvement of the comprehensive performance of such alloys.

Cryogenic treatment, as an extension of traditional heat treatment technology, not only possesses the advantages of being clean, environmentally friendly, and economically efficient, but also significantly enhances the mechanical properties and service performance of alloys [10]. Compared with traditional heat treatment [11,12], cryogenic treatment can improve the comprehensive performance of alloys through various mechanisms such as refining grains, inducing phase transformation, increasing dislocation density, optimizing the morphology of strengthening phases, and promoting twinning formation. However, there are few reports on the mechanism of the effect of cryogenic treatment on the mechanical properties and damping characteristics of Mn–Cu alloys. In this study, the Mn–Cu alloy specimens were subjected to cryogenic treatment using the immersion method. The influence of different cryogenic treatment times on the microstructure and properties of the alloy was systematically investigated, aiming to enhance the mechanical properties of the alloy while maintaining or improving the damping performance of the material.

2. Experiments

The Mn–Cu alloy used was provided by Jiangsu Tianqi New Materials Application Research Institute Co., Ltd. The 80 mm diameter ingots were prepared using a vacuum induction melting furnace, and their chemical composition is shown in Table 1. The samples used in the experiment were obtained from the ingot by wire cutting. The cryogenic treatment was carried out using a YDS-10-80 liquid nitrogen tank, where the room temperature samples were directly immersed in liquid nitrogen at -196°C for a certain period of time, then taken out and air-cooled to room temperature. The test sample numbers and processing procedures are shown in Table 2.

The DDL100 type electronic universal testing machine was used for tensile tests. The hardness was tested using the FM-ARS900 type microhardness tester. The damping characteristics were tested using the TA-Q800 dynamic thermal mechanical analyzer (DMA) at the frequency of 10 Hz and the strain amplitude of 5×10^{-4} . The specimen size was $60 \times 5 \times 2$ mm, the fixture type was three-point bending, the test temperature was -100 to 200°C and the heating rate was $5^{\circ}\text{C}/\text{min}$. The damping performance is represented by the loss factor ($\tan\delta$) of the ratio of the loss modulus (E'') to the storage modulus (E'). When $\tan\delta > 0.01$, it is considered that the alloy has high damping performance. Additionally, the temperature corresponding to the maximum value of the curve in the $dE'/dT \sim T$ graph is defined as the antiferromagnetic transition point (T_N) [13].

The microstructure was analyzed using the FEI Nova Nano 450 field emission scanning electron microscope (SEM) and TF20 transmission electron microscope (TEM). The area proportion of Mn-rich region dendrites in the samples and the particle size of α -Mn phase were calculated using the ImagePro Plus software. Three images were taken for each sample, and the average value was taken as the result. The phase analysis of the samples was conducted using the SmartLab X-ray diffraction instrument (XRD), with a scanning range of 40° to 90° and a scanning speed of $5^{\circ}/\text{min}$.

Table 1

The chemical compositions of the raw material Mn–Cu alloy (wt.%).

Element	Cu	Ni	Fe	Si	C	S	P	Mn
Mass fraction%	20.01	4.87	1.98	0.015	0.0045	<0.02	<0.05	Bal.

Table 2

Cryogenic treatment process for each sample.

Sample	As-casting	DCT12	DCT24	DCT36
Process	Cryogenic treatment for 0 h	Cryogenic treatment for 12 h	Cryogenic treatment for 24 h	Cryogenic treatment for 36 h

3. Results

3.1. Mechanical properties

Fig. 1 shows the mechanical properties of the Mn–Cu alloy after different cryogenic treatment. As the cryogenic treatment time increases, the tensile strength and hardness of the Mn–Cu alloy generally show an upward trend, while the elongation rate shows a downward trend. After 36 h of cryogenic treatment, the tensile strength and hardness of the alloy increased to 385.5 MPa and 128.77 HV, respectively, which were 7.5% and 4.9% higher than those of the as-cast alloy; while the elongation rate became 30.56%, which was 6.9% lower than that of the as-cast alloy. It is worth noting that the elongation of the alloy increased slightly after 12 h of cryogenic treatment. Studies have indicated [14–16]: As the duration of cryogenic treatment increases, on the one hand, the grains gradually become finer and more evenly distributed; on the other hand, it causes the residual tensile stress within the alloy to be transformed into compressive stress, thereby effectively inhibiting crack formation and enhancing the material's ductility. This is consistent with the results obtained in the present study.

3.2. Damping properties

Fig. 2 shows the damping performance of the Mn–Cu alloy after different cryogenic treatment times. As can be seen from Fig. 2(a), within the low temperature range below -50°C and the high-temperature range of 50°C to 200°C , the $\tan\delta$ values of the cryogenic treated samples were generally higher than those of the as-cast sample, moreover, as the cryogenic treatment time increases, the $\tan\delta$ value gradually increases. When the temperature ranges from -50 to 50°C , the $\tan\delta$ value of the cryogenic treated samples is lower than that of the as-cast sample. The above results indicate that cryogenic treatment can enhance the damping performance of the alloy in both low and high temperature ranges, but to some extent, it sacrifices the damping performance in the medium temperature range. The specific reasons will be analyzed in the section Discussion.

The characteristic temperature, namely the antiferromagnetic transition point (Neel temperature, T_N), can be defined as the maximum value of the derivative of the storage modulus with respect to temperature (dE'/dT), as shown in Fig. 2(b). The T_N values of the as-cast sample and the samples subjected to cryogenic treatment for 12 h, 24 h and 36 h were 150.35°C , 152.05°C , 152.35°C and 152.56°C , respectively, indicating that T_N gradually increased with the extension of cryogenic treatment time. When the alloy cools down to the T_N point, lattice distortion occurs, which subsequently triggers the subsequent FCC $\rightarrow$ FCT martensitic phase transformation [17] and that {101} twins were formed, which is one of the important energy dissipation sources in the Mn–Cu alloy. A higher T_N value is conducive to maintaining better damping performance of the alloy at higher temperatures, which is also the main reason for the improvement of the damping performance of the alloy in the high-temperature range.

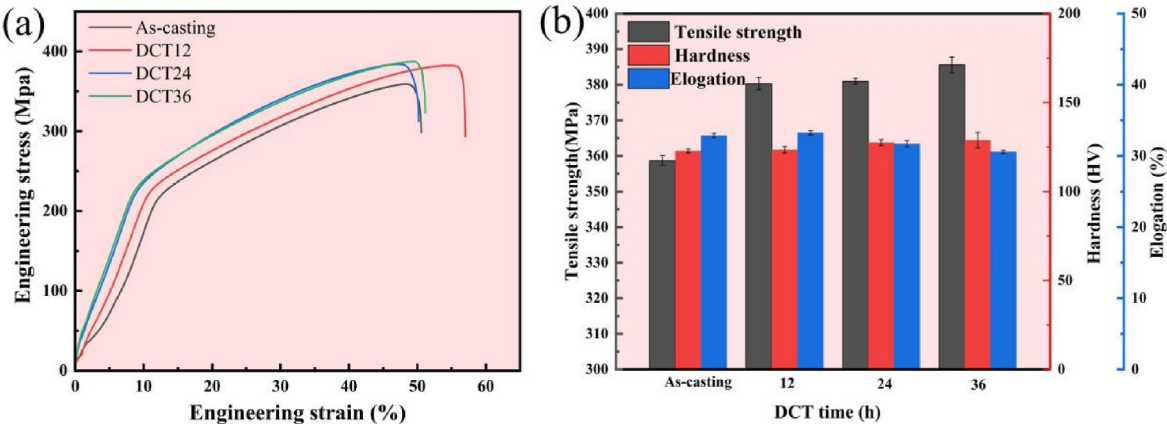


Fig. 1. Effects of cryogenic treatment on the mechanical properties of Mn-Cu alloy: (a) Stress-strain curves; (b) Mechanical properties.

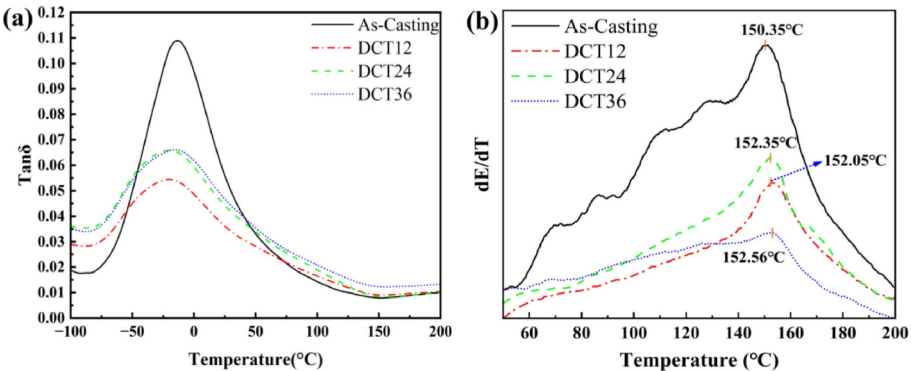


Fig. 2. The influence of cryogenic treatment on the damping performance of Mn-Cu alloy (a) Internal friction (IF) curves; (b) The characteristic temperature.

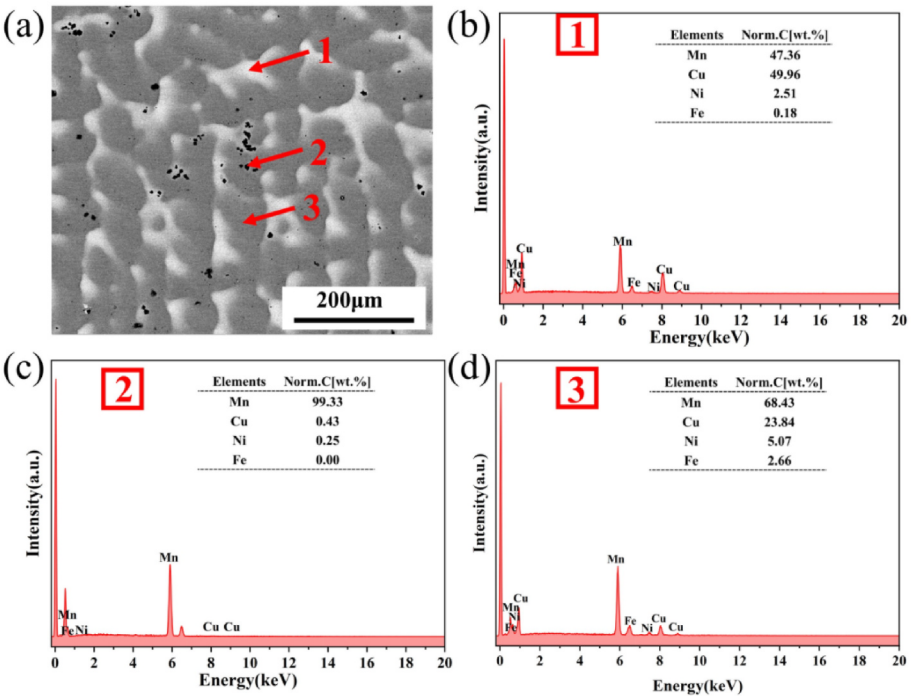


Fig. 3. SEM image of the as-cast sample and point-scan data for each location (a) SEM image of as-cast sample; (b) Energy spectrum at position 1; (c) Energy spectrum at position 2; (d) Energy spectrum at position 3.

3.3. Microstructure analysis

Fig. 3 shows the scanning electron microscope (SEM) image and energy dispersive spectroscopy (EDS) analysis of the microstructure of the as-cast Mn–Cu alloy. The microstructure has obvious macroscopic segregation, which is characterized by a dendrite structure. According to the EDS analysis shown in Fig. 3(b) and (d), the dark areas (position 3) are the Mn-rich region, and the light areas (position 1) are the Cu-rich region. That's because Mn is evidently predominant in the initially solidified dendritic zone compared to terminally solidified interdendritic zone due to the higher melting temperature of Mn than Cu. In addition, a small amount of black granular α -Mn phase (position 2) can also be observed, indicated from Fig. 3(c). The shape and size of α -Mn phase will have an impact on both the mechanical properties and damping performance of the alloy [18,19].

Fig. 4 shows the microstructures of the Mn–Cu alloy after cryogenic treatment at different times. After cryogenic treatment, the microstructure morphology of each sample still maintains the typical dendritic morphology, indicating that cryogenic treatment does not change the dendritic structure of the alloy. The ImageProPlus software was used to statistically analyze and measure three microstructure images of each sample to explore the effect of cryogenic treatment on the Mn-rich region and α -Mn phase of the alloy. As the cryogenic treatment time increases, the particle size of the α -Mn phase gradually decreases from 7.94 μm in the as-cast state to 7.25 μm , 6.38 μm and 6.27 μm after 12 h,

24 h and 36 h of cryogenic treatment, respectively, as shown in Fig. 4(b), (e), (h), and (k). This is because the contraction stress generated at low temperatures promotes the refinement of the α -Mn phase [20,21] which not only helps to reduce local stress concentration but also contributes to improving the mechanical strength of the alloy. However, an increase in the α -Mn phase will decrease the enrichment of Mn in the Mn-rich region, which is not conducive to the damping performance. Regarding the changes in the Mn-rich region, as shown in Fig. 4(c), (f), (i), and (l), the proportions of the Mn-rich region in the as-cast sample and the samples after cryogenic treatment for 12 h, 24 h, and 36 h are 53.07%, 53.87%, 54.48%, and 56.27%, respectively. That is, with the increase in the cryogenic treatment time, the proportion of the Mn-rich region gradually increases. It has been reported [22,23] that the mother-phase twinning is induced by the antiferromagnetic transformation of the Mn-rich dendrites during the cryogenic process. Therefore, an increase in the proportion of the Mn-rich region is beneficial for the formation of the mother-phase twinning, thereby enhancing the damping performance of the alloy.

Fig. 5 shows the TEM analysis of the Mn–Cu alloy. In the microstructure of the as-cast sample (see Fig. 5(a) and (b)), there are only a few relatively ordered dislocations distributed. The selected area electron diffraction pattern shows the $[-5, -2, 1]$ crystal zone axis, with clear diffraction spots and good crystal integrity. In the samples subjected to cryogenic treatment for 36 h (see Fig. 5(c) and (d)), the dislocation density significantly increased, and severe dislocation

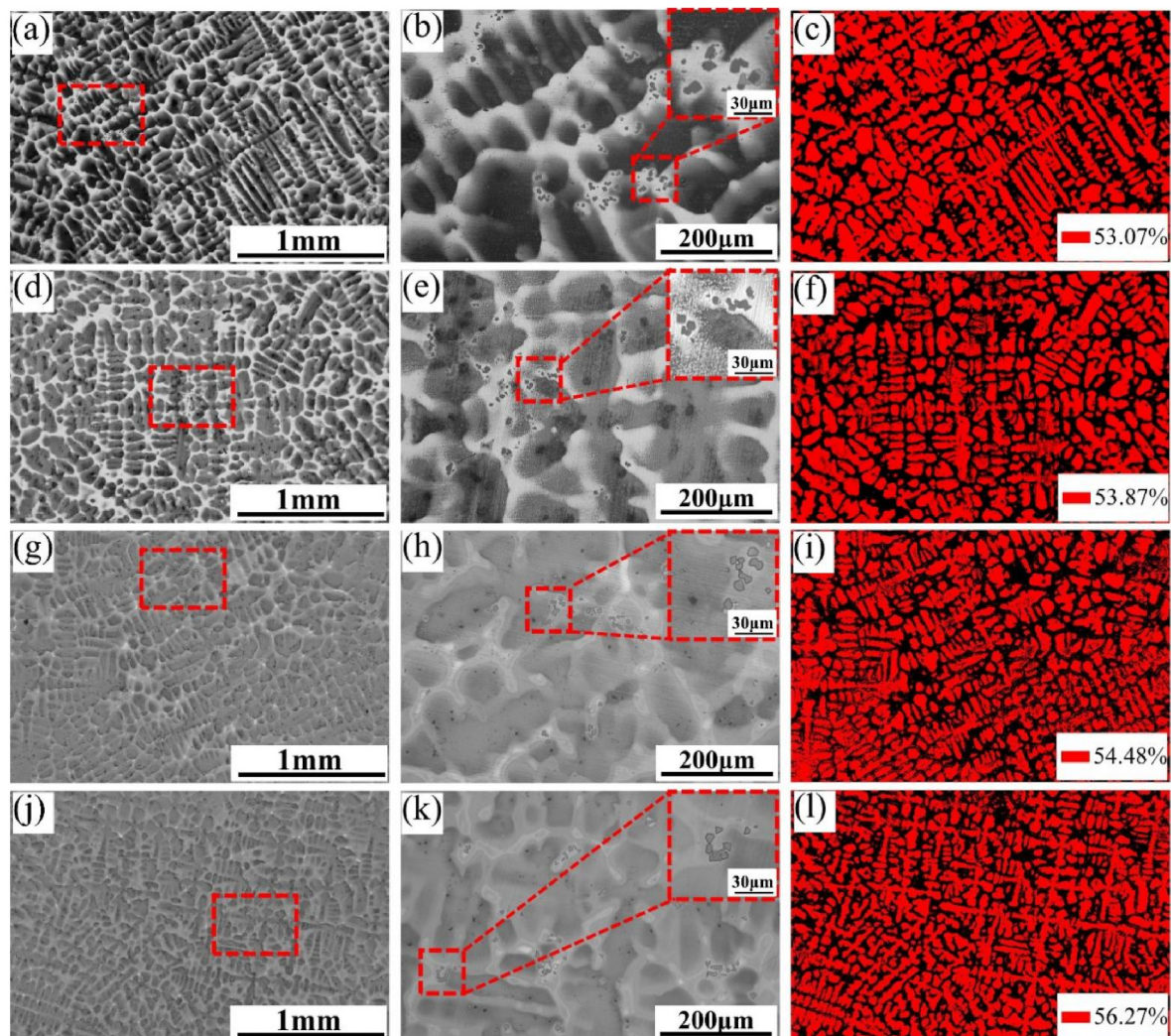


Fig. 4. Microstructures of Mn–Cu alloys after cryogenic treatment for different times: (a,b,c) As-casting; (d,e,f) DCT12; (g,h,i) DCT24; (j,k,l) DCT36.

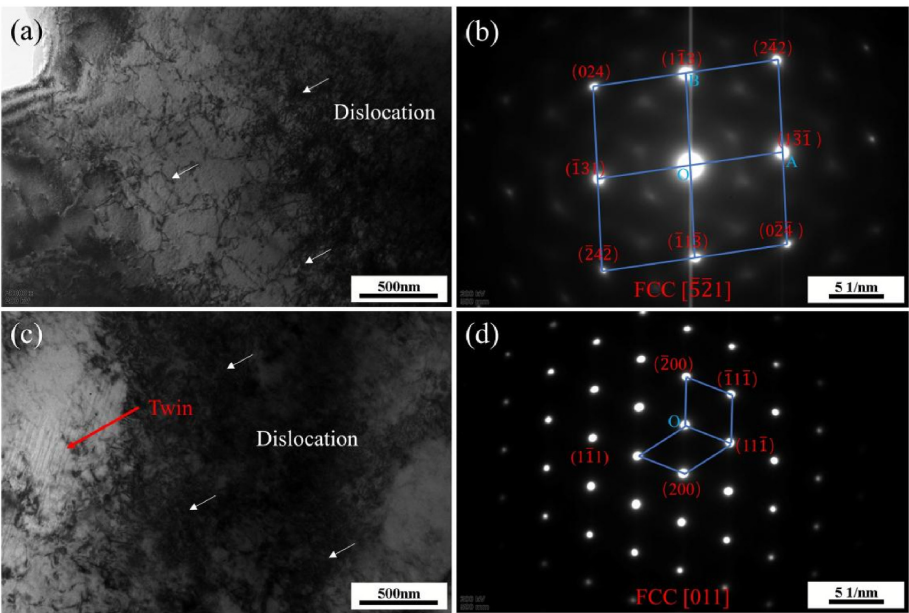


Fig. 5. TEM images and corresponding SADPs of different Mn–Cu alloy samples: (a), (b): As-cast sample; (c), (d): DCT36 h.

entanglement occurred. Twinning structures were also observed, with the twin interface being straight, and the twins are parallel to each other. Therefore, cryogenic treatment can significantly increase the dislocation density within the alloy, resulting in an increase in hardness and tensile strength, and promoting the formation of twinned crystals to improve the damping performance of the alloy [24].

Fig. 6(a) shows the XRD patterns of Mn–Cu alloys with different cryogenic treatment times. It is observed that all the samples have the characteristic diffraction peaks of the single-phase γ solid solution corresponding to the four face-centered cubic lattices (FCC) of (111), (200), (220), and (311), but the intensities of the diffraction peaks are different. Among them, (311) peak strength gradually increased with the extension of cryogenic treatment time. It indicates that the cryogenic treatment leads to a significant increase in the dislocation density of the alloy, accompanied by lattice distortion and even changes in grain orientation (see Table 3). This phenomenon of grain distortion and even grain orientation change occurring after cryogenic treatment has been observed in both aluminum alloys and titanium alloys [25–27]. Secondly, it was found that the (220) crystal plane diffraction peaks of all samples exhibited varying degrees of splitting, as shown in Fig. 6(b). When the cryogenic time reached 36 h, the (220) diffraction peak shows obvious splitting and the diffraction peak of (202) γ' appeared, which

Table 3
Calculated lattice parameters and lattice distortion of Mn–Cu alloy at different cryogenic treatment times.

	$d_{220}(\text{\AA})$	$d_{202}(\text{\AA})$	$a(\text{\AA})$	$c(\text{\AA})$	$a/c-1$
As-casting	1.320952	1.308433	3.736216	3.666384	0.0190465
DCT12	1.321172	1.307661	3.736839	3.661555	0.0205606
DCT24	1.321402	1.307832	3.737490	3.661882	0.0206472
DCT36	1.321429	1.307497	3.737567	3.659977	0.0211996

indicates that FCC→FCT transformation occurred and {101} twins formed during cryogenic treatment. Under the action of stress, the twins will have relaxation movement, which promotes the mobility of interfaces and twin boundaries, leading to a high damping capacity of the Mn–Cu based alloy [9].

The martensitic phase transformation from FCC to FCT involves a change in lattice parameters: the c-axis of the γ' phase lattice will be compressed, while the a-axis will be elongated. The changes in the c-axis and a-axis can be represented by lattice distortion ($a/c-1$). According to the split peak position of the peak {220} and Eqs. (1) and (2), the degree of lattice distortion ($a/c-1$) can be calculated, and the detailed results are listed in Table 3.

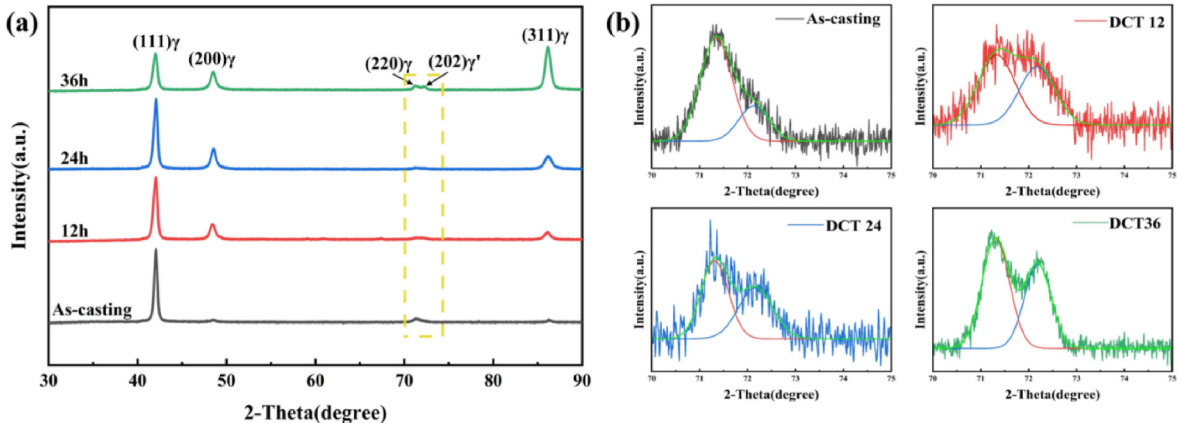


Fig. 6. (a)XRD patterns of samples after different cryogenic treatment times; (b) Local magnification of characteristic diffraction peaks of (220).

$$n\lambda = 2d\sin\theta \quad (1)$$

where n represents the diffraction order (usually taken as 1); λ denotes the wavelength of X-rays, which is 0.15406 nm here; d refers to the interplanar spacing; θ represents the diffraction angle.

$$\frac{1}{d^2} = \frac{H^2 + K^2}{a^2} + \frac{L^2}{c^2} \quad (2)$$

where H , K , and L represent Miller indices; d refers to the interplanar distance between crystal planes with Miller indices (H , K , L); a and c denote lattice parameters.

As seen in Table 3, the degree of lattice distortion varies after different cryogenic treatment times. The degree of lattice distortion conforms to the thermal elastic martensitic transformation characteristics of the Mn–Cu alloy from FCC- γ to FCT- γ' : $0 < a/c-1 < 0.05$ [28]. The increase in lattice distortion not only enhances the resistance to dislocation movement but also plays a significant role in improving the mechanical properties of the alloy. On the other hand, the magnitude of lattice distortion also depends on the degree of martensitic transformation. When the cryogenic treatment lasts for 36 h, the degree of lattice distortion is the greatest, indicating that the degree of martensitic phase transformation is also the highest. As mentioned above, after cryogenic treatment, the alloy contains more Mn-rich regions, and the α -Mn particle becomes finer, which is more conducive to the occurrence of martensitic transformation. The greater lattice distortion reflects more {101} twins caused by FCC-FCT transformation and thus higher damping capacity of the alloy.

4. Discussion

The martensite structure of M2052 alloy has a direct bearing on its damping properties. The movement of different interfaces, such as austenitic and martensitic interfaces, as well as twin interfaces can cause energy dissipation [7]. Additionally, the hysteretic motion of dislocations also plays a role in energy dissipation, thereby effectively mitigating the materials' mechanical vibration [29].

Relevant studies have indicated [30,31] that the M2052 alloy exhibits a broad damping peak, and the internal friction ($\tan\delta$) reaches its maximum value, after which it decreases as the temperature rises. The influence of cryogenic treatment on the damping mechanism of the alloy is shown in Fig. 7. Mn–Cu alloys are known for their thermoelastic martensitic phase transformation, which is reversible and characterized by minimal hysteresis as temperature rise and fall. Our previous study [9] showed that the martensitic transformation temperature (M_s) of the as-cast sample is around 18 °C. Above M_s , the inverse martensitic transformation (FCT-FCC) occurs, causing the twinning to gradually disappear and the damping performance to decline accordingly. As the temperature continues to rise, martensite completely transforms into austenite, and the twinning effect completely disappears. The damping

performance could only rely on the movement of dislocations as the damping source. In this case, the lattice structure of the cryogenic treated samples undergoes a contraction effect due to the rapid drop in temperature. The resulting lattice distortion forms high-density dislocations, which can serve as damping sources, leading to a superior damping performance compared to that of the as-cast sample at high temperatures. Below M_s , the twinning of FCT martensite becomes dominant over dislocation slip [32]. The high-density dislocations within the cryogenic treated samples will hinder the movement of twinning, resulting in a poorer damping performance compared to the as-cast sample [33]. As the temperature continues to drop, the movement of twinning is restricted, and the twinning damping effect gradually decreases. The damping mechanism of the cryogenic treated samples changes from a single damping mechanism dominated by “twinning” to a composite mechanism of “twinning + dislocations” that is dominated by dislocations, resulting in a slightly better damping performance compared to the as-cast sample.

Dong et al. [34] carried out solution and aging treatment on the as-cast Mn–Cu alloy. The damping performance was tested at room temperature, with a testing frequency of 1 Hz, and a strain amplitude gradually increased from 0 to 1×10^{-3} . The results indicated that the alloy underwent spinodal decomposition after aging treatment, forming Mn segregation regions. As the aging time increases, the Mn content in the Mn-rich regions gradually increases. The tweed microstructure formed by the spinodal decomposition promotes the martensitic phase transformation, resulting in twin boundaries, and the twin damping enhances the damping performance. However, when the Mn-rich regions aggregate to form the α -Mn phase, resulting in increased lattice distortion and more frequent dislocation movement, the damping performance will be affected. Chen et al. [35] investigated the damping characteristics of the cold-spray additively manufactured M2052 alloy. The strain amplitude ranged from 4×10^{-6} to 1×10^{-3} , with a vibration frequency of 1 Hz at a test temperature of 25 °C. For the cold-sprayed sample, the influence of dislocation damping on its overall damping performance was significant. This is because the severe plastic deformation-induced work hardening effect causes dislocations to become entangled with each other and may even be pinned and piled up, resulting in degraded damping performance. After solution treatment and aging, the sample undergoes recrystallization and an increase in Mn content in the Mn-rich regions, which promotes the formation and increase in the number of twinned crystals. As a result, the damping performance is improved under different strain amplitudes, indicating that the effect of twinning damping on the improvement of the alloy's damping performance is greater than that of dislocation damping.

In summary, for the Mn–Cu alloy, its twinning and dislocation damping is in a state of dynamic change and closely related to temperature and frequency. Cryogenic treatment significantly increases the dislocation density of the Mn–Cu alloy. Affected by the martensitic phase transformation, at different temperatures, there will be coupling or competition between twinning damping and dislocation damping,

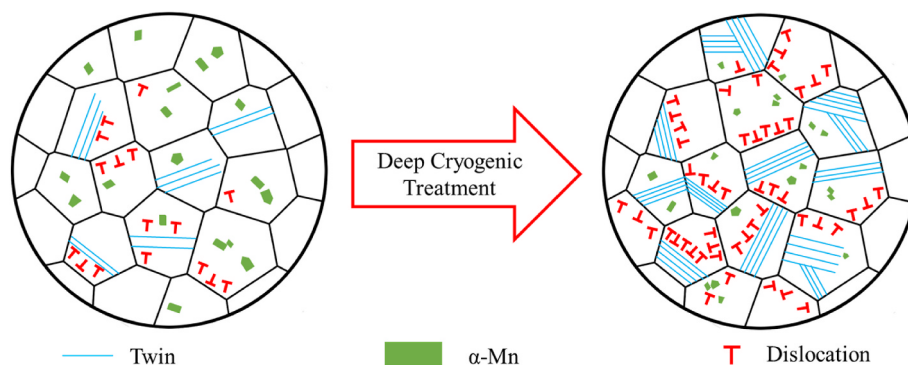


Fig. 7. Mechanistic diagram illustrating effects of cryogenic treatment on Mn–Cu alloy.

directly influencing its mechanical properties and damping behavior.

5. Conclusions

- (1) Cryogenic treatment significantly enhances the mechanical properties of the Mn–Cu alloy. When subjected to cryogenic treatment for 36 h, its tensile strength and hardness increase to 385.5 MPa and 128.77 HV respectively. The improvement in mechanical properties is mainly attributed to the increase in dislocation density and the refinement of the α -Mn phase.
- (2) With the extension of the cryogenic treatment time, the α -Mn phase in the Mn–Cu alloy becomes finer, the proportion of the Mn-rich region increases, and at the same time, the FCC→FCT martensitic phase transformation is induced, and the degree of lattice distortion also increases.
- (3) Under high-temperature or low-temperature conditions, the twinning damping efficiency of M2052 alloy gradually decreases as the temperature rises or falls. Cryogenic treatment introduces a large number of dislocations into the alloy. Through the synergistic mechanism of dislocation damping and twinning damping, the damping performance is effectively enhanced.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgements

The project was supported by the National Natural Science Foundation of China (52175410), the Natural Science Research Program for Higher Educational Institutions in Jiangsu Province (18KJB430009), and Zhenjiang Basic Research Special Project (JC2024010).

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2026-03

Elsevier

<https://doi.org/10.1016/j.jmrt.2026.02.056>

Ding R, Liu G-L, Tan G-P, et al., (2026) Synergistic strengthening mechanism of cryogenic treatment for mechanical properties and damping characteristics of Mn–Cu alloy. *Journal of Materials Research and Technology*, Volume 41, March-April 2026, pp. 4732-4738