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Study on aging strengthening and nano precipitates of Cu–Ni–Mn–Fe alloy

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Abstract

This paper presents a systematic study of the precipitation strengthening mechanism in polybasic Cu–Ni–Mn–Fe alloy. The effect of aging treatment on the microstructure, morphologies and phase transition of the Cu–Ni–Mn–Fe alloy were analyzed and discussed using scanning electron microscope (SEM), transmission electron microscope (TEM), X-ray diffraction (XRD) and differential scanning calorimeter (DSC), respectively. The θ-MnNi precipitates with a size of 50–100 nm, mainly formed during aging, were considered as the source of the aging strengthening. In addition, there is a semi-coherent between the plane (111) of the θ-MnNi precipitates and the plane (200) of the a-Cu matrix. The density of θ-MnNi precipitates increases with the increase of the aging time. The hardness, yield strength and ultimate tensile strength of the aged alloy increase from 127 to 366HB, 217 to 788 MPa, and 433 to 842 MPa, respectively, which is attributed to precipitation strengthening.

1. Introduction

Cu–Ni–Mn–Fe alloys are a kind of polybasic copper alloy exhibit a good combination of properties such as high elasticity, high strength and excellent heat resistant performance. Moreover, it still remains high hardness and strength and possesses good wear property, corrosion resistance and good mechanical properties even at 400 °C. The Cu–Ni–Mn–Fe alloys have a great potential for application in special machinery fields owing to their un-conducted magnetic properties [1–4]. Among these properties, strength and hardness are the most important in the engineering application of Cu–Ni–Mn–Fe alloys and are related to the precipitation phenomenon. Thus, the strength and hardness of the Cu–Ni–Mn–Fe alloys are emphasized in this article. The strengthening effect of Cu–Ni–Mn–Fe alloy is mainly due to precipitation of solute atoms during aging treatment [5, 6]. High-density of precipitates, which are mainly formed during the aging treatment, have been investigated by many researchers. However, the microstructure, morphologies and formation mechanism of the precipitates have not been clearly studied yet.

At present, numerous articles have been published on the methods and precipitation behavior during the aging process of alloys such as Cu–Ti–Ni [7–10], Cu–Cr–Zr [11–15], Cu–Ni–Zn–Al [16], Cu–Be [17, 18]. During the aging process, the precipitates will preferentially precipitate along certain crystal planes of the matrix or some place where there are high dislocation density and high solute concentration. The precipitates greatly affect the microstructure, fracture modes and mechanical properties [19, 20] as well as photoelectric, magnetic and other physical properties in the alloys [21].

In this work, the θ-MnNi precipitates were characterized by transmission electron microscope (TEM), X-ray diffraction (XRD) and the forming condition was investitived using differential scanning calorimeter (DSC). Moreover, the strengthening effect of θ-MnNi precipitates on the Cu–Ni–Mn–Fe alloys was also discussed, it can help to inform further modify and design for strengthening and toughening of Cu–Ni–Mn–Fe alloy.
2. Materials and methods

The Cu–Ni–Mn–Fe alloy with a nominal composition of Cu–20 wt% Ni–20 wt% Mn–5 wt% Fe was melted by using electrolytic copper (purity of 99.99%), pure nickel (purity of 99.99%), pure manganese (purity of 99.99%) and pure iron (purity of 99.99%) in a vacuum induction melting furnace under argon atmosphere. The temperature was set at 1250 °C. After the alloy was completely melted, the molten was heated and stirred for about 20 min, and then poured into a water-cooled copper mold to obtain the initial ingot.

The thermal analysis of the alloy was performed using a 204F1 differential scanning calorimeter (DSC). The temperature of the compact was increased from room temperature to 1200 °C at a heating rate of 10 °C/min. Moreover, the suitable heat treatment temperature was chosen according to the results of DSC. The heat treatment was carried out in the box type resistance furnace (SX-12-10), and the solid solution treatment at 520 °C for 50 min followed by water quenching, and aging treatment at 430 °C for various times.

X-ray diffraction detection were carried out using a 7000 X-ray diffractometer of Shimadzu with Kα diffraction rays of Cu target at an accelerating voltage of 40 kV, current of 40 mA and step-scanning rate of 10° min⁻¹. The morphology and microstructures of the precipitated phases were characterized by JSM-6700F field emission scanning electron microscope (SEM), JEM-3010 transmission electron microscope (TEM). The TEM specimens sliced from aged specimens were mechanically grinded into thin foils with the thickness of 70 ~ 80 μm. Then, several disks with a 3 mm diameter punched out from the foils were double-jet electropolished in a mixture of 67% methanol and 33% hydrogen nitrate under −30 °C. The size of the precipitate was evaluated using Image-Pro Plus. The hardness of the alloy was tested using HB-3000 type brinell hardness tester which is developed in Shenzhen, China. The measuring range of this hardness testing machine is 8 ~ 650HBW. The test force range is 187.5 kgf ~ 3000 kgf, and the pressure holding time is 30 s ~ 60 s. Tensile tests were carried out on HT-10 universal testing machine which is developed in Taiwan, China to obtain tensile strength. The maximum force value of this tensile testing machine is 100KN. The tensile speed during the tensile test is 0.5 mm min⁻¹. The size of the tensile specimen [22] is shown in figure 1.

3. Results and discussion

3.1. Microstructure of the as-cast Cu–Ni–Mn–Fe alloy

Figure 2 shows the microstructure of as-cast Cu–Ni–Mn–Fe alloy. Coarse dendrite grains can be observed (figures 2(a') and (c)) as a result of continuous grain growth during solidification. There are mainly two types of primary precipitates in the as-cast Cu–Ni–Mn–Fe alloys. Figure 2(a) shows the first type of primary precipitate in the as-cast Cu–Ni–Mn–Fe alloys. These granular black precipitates within 5 nm, which were produced during solidification, are mainly distributed inside the grains. It is worth noting that these primary precipitates could be re-dissolved into the matrix of α-Cu in the subsequent solid solution treatment.

The crystal structure of α-Cu matrix was determined from the analysis of selected area electron diffraction patterns (SAED) (figure 2(b)). Remarkably, the Ni, Mn elements, which have different atomic radius, entered into the Cu lattice, and thus inevitably caused the lattice constant changed. It can be deduced that the phase of the matrix is the copper-based solid solution with a face-centered cubic (FCC) crystal structure in the as-cast Cu–Ni–Mn–Fe alloy. The Cu–Ni–Mn–Fe alloys with FCC crystal structure, which has 12 slip systems and 12 slip directions, present good elongation but low strength. As reported in Ref. [5, 6], the hardness and tensile strength of Cu–Ni–Mn–Fe alloy can be greatly improved during aging process. So, it is necessary to further study the mechanism of strengthening effect in the Cu–Ni–Mn–Fe alloy during aging treatment.

Figure 3 shows the second type of primary precipitate of as-cast Cu–Ni–Mn–Fe alloys. Square particles with a size about 1 μm can be obviously seen from figure 3(a). Figure 3(b) is the SAED pattern of the precipitates.
According to the result of the standard PDF card, it can be confirmed as the FeMn$_4$ precipitate. Due to the highest melting point of Fe and low solubility of Fe in copper, Fe and Mn formed the FeMn$_4$ precipitates during the cooling process. These FeMn$_4$ precipitates are distributed among the dendrites.
3.2. Differential thermal analysis of as-cast Cu–Ni–Mn–Fe alloy

Cu–Ni–Mn–Fe alloy as a precipitation-strengthening alloy, the solid solution and precipitation behavior are directly related to the temperature [1]. At high temperature, the Cu and Ni in Cu–Ni–Mn–Fe alloy can be miscible with each other to form infinite solid solution, a large number of Mn can be dissolved in Cu. Fe and Ni can be infinitely soluble form a continuous solid solution. However, as the cooling temperature decreases, the solubility of the solute element decreases, and Ni, Mn, Fe solute atoms precipitate from the supersaturated solution and formed precipitates dispersed in the α–Cu matrix during the aging treatment and thus increased the strength and hardness of the Cu–Ni–Mn–Fe alloy [5]. Hence, appropriate aging conditions can be determined to promote the precipitation of the precipitates effectively and thus can increase the strength of the Cu–Ni–Mn–Fe alloy. Figure 4 is the DSC curve of the as-cast Cu–Ni–Mn–Fe alloy during the heating and cooling process. As can be shown, there are two endothermic peaks of the Cu–Ni–Mn–Fe alloy during the heating process from 300 °C to 1200 °C. The initial temperature of first peak is 450 °C, a termination temperature is 580 °C, and a peak temperature is 530 °C, indicating that there is a phase transformation during this endothermic process [23–25]. As reported in Ref. [26, 27], the aging treatment can greatly improve the strength and hardness of Cu–Ni–Mn alloys. When alloys were aged at 450 °C for 40 h, the strength and hardness show the best performance in the Cu-Ni-Mn alloys. And it can be explained that a large amount of θ–MnNi precipitates were precipitated from the Cu matrix under the aging treatment at 450 °C. Especially, when the aging time reached 40 h, the content of the precipitate reached it’s maximum. According to the DSC curves, it can be judged that the precipitates be likely generated when the temperature is below 450 °C during the heating process in the Cu–Ni–Mn–Fe alloy, while the type of precipitate needs to be clarified. When the temperature is at 450 °C ~ 580 °C, the precipitates could be re-dissolved into the Cu matrix. In order to study the heat treatment process of Cu–Ni–Mn–Fe alloy, a large number of preliminary experiments were carried out. According to results, it was found that when the alloys was solution treated at 520 °C for 50 min, the primary precipitates inside the grains were almost completely re-dissolved into the Cu matrix, and the size of grains did not become big significantly. What’s more, when the aging temperature is 430 °C and the aging time is 72 h, the hardness and strength of the Cu–Ni–Mn–Fe alloy reached it’s maximum. The aging temperature of Cu–Ni–Mn–Fe alloys is close to the aging temperature of the Cu–Ni–Mn alloys. The results of the hardness and strength tensile test are shown in figures 7 and 8, respectively. Moreover, the second endothermic peak with a large area has an initial temperature of 990 °C, a termination temperature of 1130 °C, a peak temperature of 1055 °C. With the temperature increasing, the volume fraction gradually increase, indicating that the Cu–Ni–Mn–Fe alloy melted at the high temperature, which requires the absorption of large amounts of heat, and the alloy liquidus is approximately 1130 °C.

In addition, an upward exothermic peak appears only at the high-temperature during the cooling process of Cu–Ni–Mn–Fe alloy, indicating that there is a solidification process. In addition, there is no exothermic peak (seen from the red curve) in the Cu–Ni–Mn–Fe alloy during the rapid cooling process and this can be explained that the precipitates can hardly precipitate from the solid solution in a limited time due to the high cooling rate during the cooling process.
3.3. Aging Cu–Ni–Mn–Fe alloy

3.3.1. Microstructure and precipitate evolution

According to the result of differential thermal analysis in section 3.2, a reasonable heat treatment condition was determined in combination with the experiment. The suitable aging temperature was determined to be 430 °C.

Figure 5 shows the microstructure of the Cu–Ni–Mn–Fe alloy subjected to aging at 430 °C for different aging times after the solution treatment. It should be noted that the microstructure are dendrites that are not significantly different from the as-cast Cu–Ni–Mn–Fe alloy, suggesting that the aging process only evolves solute atoms with temperature and time, failing to change the morphology of alloys. Remarkably, with the increase of aging time, the density of aging precipitates in the α-Cu matrix increases, as shown in figure 5. These precipitates are distributed within grains. Generally, the initial precipitation of aging precipitates is a decomposition process of solid solution which highly depends on the solute atom diffusivity and driving force. Meanwhile, the driving force for the nucleation of aging precipitates generally depends on the solute and vacancy concentrations [28]. In the Cu–Ni–Mn–Fe alloys, the high concentration of solute atoms can accelerate precipitates. Furthermore, the water quenched Cu–Ni–Mn–Fe alloy is rich in vacancies, which supply the heterogeneous nucleation sites for aging precipitates, and the clustering of solute atoms occurs around vacancies during the early stage of decomposition. Due to the interaction between solute atoms and vacancies, accompanied by thermal diffusion, nucleation and growth of solute atoms occur in the segregation region, and thus can accelerate the precipitation [28, 29]. Obviously, with the prolongation of aging time, the migration speed and activity of solute atoms will be accelerated, resulting in the increases of the number of precipitates inside the grains. Moreover, as shown in figure 5, it can be observed that large precipitated particles were distributed in the α-Cu matrix at the early stage of aging. The precipitated particles became finer and the distribution became much more dispersive after reaching the peak aging.

Figure 6 shows TEM micrograph and SAED pattern of Cu–Ni–Mn–Fe alloy after aging treatment. Figure 6(a) shows that the precipitates are uniformly distributed in the α-Cu matrix. The precipitates presents a granular particle, with the grain size of about 50–100 nm. Generally, the type of aging precipitates can be distinguished by the diffraction patterns. Figure 6(b) is the corresponding SAED pattern of the precipitates. There are diffraction spots of crystal plane group {100}, {110}, {111}, and the direction of the incident parallel to the [011] axis. TEM analysis and similar morphology reported for this precipitates in the same alloy system, can prove the existence of θ-MnNi precipitates [26, 27].

Figure 5. Evolution of the microstructure and precipitates with the aging time: (a) 0 h, (b) 24 h, (c) 48 h, (d) 72 h.
3.3.2. Mechanical properties

Figure 7 shows the evolution of the Brinell hardness as a function of aging time at 430 °C for the Cu–Ni–Mn–Fe alloy. The hardness of the aged alloy increases from 127 HB at 0 h to a peak value of 366 HB at 72 h. In the range of 0–24 h, the increase of hardness is significant. This is mainly due to the fact that the solute atoms dissolved into the matrix and formed supersaturation solution before the aging treatment. The high concentration of solute atoms can provide the dynamic conditions for the precipitation of the precipitates. When the aging time exceeds 24 h, since the amount of solute atoms in the α-Cu matrix decreases with the increase of precipitate fraction and thus result in the decreases of the speed of the precipitates. With the aging time reaching 48 h, the hardness curve tends to be flat, and the hardness of the alloy reaches its peak after aging for 72 h.

Generally, the strength of the alloy is relate to the density of precipitates, since the more precipitates will shorten the interspacing of them. Thus, the dislocations are more difficult to pass them. Therefore, the effect of precipitation strengthening will be significantly increased with the increase of ⁰-MnNi precipitates density. The tensile properties of Cu–Ni–Mn–Fe alloy samples aged at 430 °C for different times are shown in figure 8. It can be seen from the curve that the ultimate tensile strength increases from 433 MPa to 842 MPa and yield strength from 217 MPa to 788 MPa of Cu–Ni–Mn–Fe alloy with the aging time prolonging from 0 to 72 h. Compared with the evolution of the density of precipitates in the microstructure of the alloy after aging treatment in figure 5, it can be seen that the increase in hardness and strength during the strengthening of the alloy is mainly dependent on the density of precipitates. Due to the pinning effect of precipitates, the hardness of the alloy increases by about 188% compared with the as-cast state. A significant increase in yield stress and ultimate tensile strength can be observed from figure 8(b).
3.4. X-ray diffraction

As shown in figure 9, it can be seen that three diffraction peaks from the X-ray diffraction patterns after solid solution and aging treatment of as-cast Cu–Ni–Mn–Fe alloy. The diffraction angles of Cu–Ni–Mn–Fe alloy after solid solution treatment peaks at 42.79°, 49.63°, and 73.14°, among which the diffraction angle of 42.79° corresponds to the highest diffraction peak intensity. By the analysis, it is confirmed that the phase is a copper-based solid solution, which is consistent with the result in figure 2. As the isothermal aging treatment progresses, the relative peak intensities of the (200) crystal plane of the α-Cu matrix are gradually increasing. From XRD analysis, it is confirmed that the diffraction angle of the (111) crystal plane of the θ-MnNi precipitate is close to the diffraction angle of the (200) crystal plane of the α-Cu matrix. Thus, it is confirmed that θ-MnNi precipitates are precipitated in the Cu–Ni–Mn–Fe alloy during the isothermal aging treatment. In addition, the mismatch \( d \) between the (200) crystal plane of the α-Cu matrix and the (111) crystal plane of the θ-MnNi precipitates was estimated from formula (1):

\[
\delta = \frac{d_{(200)_{\alpha}} - d_{(111)_{\theta}}}{d_{(200)_{\alpha}}}
\]

Where \( d_{(200)_{\alpha}} \) is the interplanar spacing of the (200) crystal plane of the α-Cu matrix; \( d_{(111)_{\theta}} \) is the interplanar spacing of the (111) crystal plane of the θ-MnNi precipitate; \( \delta \) is the mismatch between the (200) crystal plane of the α-Cu matrix and the (111) crystal plane of the θ-MnNi precipitates.

Based on the X-ray diffraction spectra, the interplanar spacing was calculated to be 1.834 Å for the (200) plane and 1.633 Å for the (111) plane. The mismatch \( \delta \) between the (200) crystal plane of the α-Cu matrix and the (111) crystal plane of the θ-MnNi precipitates would be approximately 10.96%. Thus, according to the numerical range of phase interface mismatch, there is a semi-coherent interface between α-Cu matrix and the θ-MnNi precipitates [30]. These findings indicate that the precipitation strengthening effect of the Cu–Ni–Mn–Fe

![Figure 8. The mechanical properties of aged Cu–Ni–Mn–Fe alloy with different aging times: (a) Stress-strain curve; (b) Ultimate tensile strength and yield strength curve.](image)

![Figure 9. XRD spectra of the Cu–Ni–Mn–Fe alloy in various processing conditions.](image)
alloy during aging treatment is mainly due to the precipitation of semi-coherent θ-MnNi precipitates along the α-Cu matrix.

4. Conclusions

1. The aging strengthening effect of the alloy is mainly derived from the θ-MnNi precipitates with a size of 50-100 nm in the α-Cu matrix.

2. There is a semi-coherent between the plane (111) of the θ-MnNi precipitates and the plane (200) of the α-Cu matrix.

3. The density of θ-MnNi precipitates increases with the increase of the aging time. The hardness, yield strength and ultimate tensile strength of the aged alloy increase from 127 to 366HB, 217 to 788 MPa, and 433 to 842 MPa, respectively, which is attributed to precipitation strengthening.

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Author contributions

Conceptualization and data curation, J Z and H S; funding acquisition, J Z; methodology, S L; writing–original draft, L S and H S; writing, review and editing, Q F and L S

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Conflicts of interest

The authors declare no conflict of interest.

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