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Effect of grain refinement on phase transformation behavior and mechanical properties of Cu-based alloy

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Abstract: A small amount of misch metal was added to Cu-Zn-Al alloy in order to study its effect on grain refinement, mechanical properties, phase transformation behavior and stabilization of martensite. It is found that the addition of misch metal is very effective for reducing the grain size. The coarse grains over 1 000 μ m are refined to the size of 30 μ m by the addition of 0.43% (mass fraction) misch metal. The grain size of thermo-mechanically treated alloys is barely affected by cold working. The fracture strength and ductility increase significantly with the increase of misch metal content when tensile test is carried out below $M_{\rm f}$ temperature. Also, the fracture strength is larger in the case of post-quench ageing treatment than that in the case of direct quench ageing treatment. The fracture mode is changed from transgranular brittle fracture to ductile fracture with void formation and coalescence by the addition of misch metal.

Key words: mechanical properties; grain refinement; post-quench ageing; transgranular fracture

1 Introduction

Cu-Zn-Al alloy is known to have a short fatigue life and problems of poor mechanical properties such as fracture and fatigue strengths and elongation by coarse grain size, because it contains a large variation of transformation temperature with composition and high elastic anisotropy. In order to solve these problems[1–4], it is required to enhance the mechanical properties of the Cu-Zn-Al alloy and to avert the boundary fracture[5]. As to the protection methods of boundary fracture, stabilization with ageing[6–8], control of texture by preferred orientation and grain refinement method of the grain size are known to be effective. Especially, it is reported[1, 4] that the latter method is more effective in the protection of boundary fracture.

One of the refining methods [2-3, 9] is adding alloying elements, with which it allows the increment of nucleation site. It then also provides the formation and solidification [10] of fine compound in β -phase in the matrix. The short fatigue life due to boundary fracture and reduced ductility cause the fragility of the boundary itself, as the precipitation of alloy elements becomes segregated into the grain boundaries. In this study, misch metal, which is known to show low solidification and to be effective refining material, is added to examine the effect on mechanical properties. Through the review of the result, basic data are collected and provided for further utilization of the Cu based alloy system.

2 Experimental

2.1 Melting and heat treatment

Cu, Zn, Al of high purity and small amount of misch metal were melted in the induction melting furnace. The nominal compositions for the experiment are shown in Table 1.

The cast ingot was homogenized at 850 °C for 24 h. Then, it was hot rolled to the thickness of 1 mm and continued to the thickness of 0.5 mm by cold rolling at the same temperature. The specimens for examining tensile properties, hardness and X-ray diffraction were prepared through water cooling in the iced water after maintaining for 10 min at 850 °C of β -phase region.

2.2 Mechanical test and fracture surface observation

After the process of melting and heat treatment,

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Alloy	Cu	Zn	Al	Misch metal
А	70	26	4.0	
В	70	26	4.0	0.10
С	70	26	4.0	0.27
D	70	26	4.0	0.43

Table 1 Nominal compositions of used alloy (mass fraction, %)

tensile tests for the specimen in the martensite phase were carried out to study the effect on the mechanical properties for the variations of adding amount of alloying elements. And also, in order to survey the effect of post-quench ageing treatment on tensile property, the test was carried out after ageing treatment for 60 min at the temperature above M_s point following solution heat treatment. The fracture surface was observed using SEM. The Rockwell hardness was measured in order to survey the hardness change with ageing temperature and refinement of grain size.

2.3 X-ray diffraction analysis

To survey the martensite stabilization and crystal structure change with refinement of grain size and ageing temperature, X-ray diffraction analysis was performed. The specimen for X-ray diffraction analysis was prepared through ageing at 150 °C for 1 h below $M_{\rm f}$ (martensite finish) point in the martensite phase and at 200 °C for 1 h above $M_{\rm s}$ (martensite start) point after solution heat treatment, respectively.

3 Result and discussion

3.1 Optical microscope observation

Fig.1 shows the microstructure of the quenched specimen observed through optical microscope. In the case of alloy A, the magnitude of the grain size shows the coarse one around 1 000 μ m, but it becomes finer with increasing the addition of alloying elements. It is noted that the magnitude of grain size of alloy D is around 30 μ m. This implies that the particles of the second phase are scattered and thus the grain growth velocities are restrained in the grain boundary and intergrains. It is anticipated that the scattered particles work as nucleation sites and promote nucleation of β -phase, which eventually causes the grain refinement.

3.2 Mechanical properties and fracture observation

3.2.1 Tensile transformation behavior

When the grain size of the Cu based alloy is refined, the occurrence of crack increases and propagation stress becomes larger. This means that the fracture strength and strain are increased. Fig.2 shows the stress-strain curves of specimen after post-quench ageing and quenching only, with varying the addition of alloying elements. It is seen that the fracture strength and elongation are increased with decreasing the grain size. Alloy D shows the increasing of yield and fracture strengths and elongation. In the state of quenching, alloy A shows fracture strength and elongation of 206 MPa and 5.6%,



Fig.1 Optical microstructures showing change of grain size by addition of misch metal: (a) Alloy A; (b) Alloy B; (c) Alloy C; (d) Alloy D



Fig.2 Stress-strain curves of alloys A, B, C and D post-quench aged and as-quenched state

respectively. In the case of alloy D, the fracture strength and elongation become 408 MPa and 8.9%, respectively. Alloy A shows the lowest fracture strain of 5.6% and lower fracture stress, because of its coarse grain size and easy occurrence of boundary fracture. It is considered that the crack occurs at the triple point of grain boundary with the largest stress concentration, then propagates along the grain boundary, and finally causes the fracture.

The case of alloy B does not show the typical stress-strain curve, except the so-called 3-steps feature of the curve. Alloys C and D do not even show such features. The specimen after post-quench ageing appears to have increased fracture and yield strengths, but slightly

decreased elongation compared with the quenching ones, after solid-solution treatment. This phenomenon is considered to occur due to the rapid cooling during the post-quench ageing, which results in the rearrangement and regularity of the atoms. It is also considered that the increase of yield and fracture strengths is due to the distribution of refined compound, refinement of grain size and ensuing reduction of martensite plate thickness. 3.2.2 Fracture surface observation

Fig.3 shows the fracture surface of tensile specimen by SEM. Alloy A shows the trend of intergranular fracture of propagated crack with typical facet due to stress concentration at the triple point of grain boundary. And it shows the trend of dimple type transgranular fracture with the addition of alloying elements. In case of alloy D, the crack does not necessarily occur at the grain boundary and therefore it shows typical ductile fracture surface.

3.2.3 Variation of hardness

Fig.4 shows the variation of hardness of the aged specimens at 0-350 °C with quenching in the icedwater of 0 °C after maintaining for 10 min at 850 °C. The hardness increases with increasing the addition of alloying elements. The increasement of hardness is not large when aged at the iced-water of 0 °C and below 150 °C, but the hardness becomes nearly constant maximum one after ageing at 200 °C and 250 °C. And then, the hardness decreases after ageing at 300 and 350 °C. Fig.5 shows the hardness variation of specimen with ageing treatment for 60 min after quenching in the



Fig.3 SEM images of tensile fracture surface metal: (a) Alloy A; (b) Alloy B; (c) Alloy C; (d) Alloy D



Fig.4 Variation of hardness of alloy A, B, C and D aged at 0-350 °C after quenching in ice water



Fig.5 Variation of hardness of alloy A, B, C and D aged at 150 -350 °C after step-quenching in salt-bath

salt bath at 150, 200, 250, 300, and 350 °C, after maintaining 10 min at 850 °C. The hardness increases with adding the alloying elements and becomes maximum after aged at 200 and 250 °C, which shows the same tendency with ageing treatment in martensite phase.

3.2.4 Analysis of X-ray diffraction

In order to survey the characteristics of phase transformation along the variations of heat treatment conditions and crystal grain size, the results of X-ray diffraction after ageing at room temperature and 200 °C were analysed.

Fig.6 shows the results of quenching state alloy A and B and aged specimen C and D. It is found that the matrix, showing the peak of $(0018)_M$, is the martensite with transformed 18R structure from D0₃ type[11–12] β -phase of parent phase. But, the peak of $(1214)_M$ disappears in case of ageing at above A_f point and, instead,



Fig.6 X-ray diffraction patterns of alloys A, B(as-quench) and C, D(aged-specimen)

the peak of $(200)_{\beta}$ appears. This means that a part of martensite appears to be β -phase with inverse transformation.

It is anticipated that the stabilization of martensite makes it difficult for inverse transformation to occur. A part of martensite is inversely transformed into α and β phases at the ageing temperature of 200 °C. It is believed that these α and β phases show effects on the mechanical properties such as tensile strength and hardness.

4 Conclusions

1) Fracture and yield strengths and elongations are increased with the refined grain size and the addition of alloying elements. Fracture surface shows the trend of transgranular fracture from intergranular fracture. The specimen after ageing treatment shows increased fracture and yield strengths, but decreased elongation than those with quenching only.

2) The hardness increases with refining the grain size. The maximum of hardness is obtained in the speamen aged at 200 $^{\circ}$ C and 250 $^{\circ}$ C. It is anticipated that high temperature ageing causes a part of the martensite to the state of stabilization.

3) A_s point increases with ageing time in the martensite and M_s point, given by the solid solution treatment time, decreases with refining the grain size.

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