

## PHASE TRANSFORMATION IN RAPIDLY SOLIDIFIED Al-Cu-Fe ALLOY<sup>1</sup>

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### ABSTRACT

By means of rapid solidification, two Al<sub>65</sub>Cu<sub>20</sub>Fe<sub>15</sub> powders were prepared with water and liquid N<sub>2</sub> as the respective cooling agent. Both powders are composed of a quasicrystalline icosahedral phase and a crystalline hexagonal phase, with the water-cooled alloy having a higher crystalline phase content. In the isothermal annealing process, the crystalline phase in the water-quenched alloy begins to decrease at 500°C and then disappears at 600~700°C. At about 800°C, new crystalline phases form, and at 900°C, the quasicrystalline phase disappears. Conversely, in the liquid N<sub>2</sub> quenched alloy, the quasicrystalline phase starts to decrease at about 500°C, and the hexagonal phase decomposes into new crystalline phases. At 700~800°C, the quasicrystalline phase disappears. For the water-cooled sample, the quenching at 100~200°C makes the crystalline to quasicrystalline phase transformation start at a lower temperature and the crystallization of the quasicrystal occur at a higher temperature. For the liquid N<sub>2</sub> quenched alloy, the quenching at 100~400°C, did not affect its phase transformation at high temperature.

**Key words:** quasicrystal Al-Cu-Fe alloy rapid solidification phase transformation

## 1 INTRODUCTION

In 1987, Tsai *et al* found a quasicrystalline icosahedral phase (*I* phase) in rapidly solidified Al-Cu-Fe alloy<sup>[1]</sup> and the optimum composition for the formation is Al<sub>65</sub>Cu<sub>20</sub>Fe<sub>15</sub>. Because there was no exothermic peak in the DSC curve, they considered that this *I* phase remains stable until melting. By means of ingot metallurgy, Ishimasa and Mori got millimeter scale quasicrystalline grains<sup>[2]</sup>. This work further confirmed that the *I* phase of Al-Cu-Fe alloys is an equilibrium phase.

Recently, we found that the quasicrystalline phase can transform into crystalline phase and *vice versa*; this transformation is dramatically affected by the alloy preparation

and the annealing procedure. Here we give the details.

## 2 EXPERIMENTAL

Using block Al, Cu, Fe with purity of more than 99.9% as raw materials, three alloys with a nominal composition of Al<sub>65</sub>Cu<sub>20</sub>Fe<sub>15</sub> were produced. The block metals were melted in an electrical furnace. Alloy A is obtained by rapidly solidifying the melt in pure copper plates; alloys B and C are powders produced with the RS device, using water and liquid N<sub>2</sub> respectively as cooling agent<sup>[3]</sup>. The cooling rate of three alloys are estimated to be 10<sup>1</sup>~10<sup>2</sup> K/s, 10<sup>4</sup>~10<sup>5</sup> K/s and 10<sup>5</sup>~10<sup>6</sup> K/s respectively. Chemical analysis indicates that the real compositions are consistent with the

nominal compositions.

Rigaku3014 X-ray Diffractometer and a Hitachi H-800 Transmission Electron Microscope were used to determine the phase constitutions of the alloys. The isothermal annealing of the samples was conducted in a protective atmosphere. The samples were put into furnace at the setting temperature. After the holding stage, the samples were removed from the furnace and cooled in air.

### 3 RESULTS AND DISCUSSION

#### 3.1 Phase Constitution of the Alloys

Fig. 1 illustrates the XRD patterns of the three alloys. Alloy A is basically composed of quasicrystalline *I* phase; both alloys B and C contain *I* phase and an unknown phase, which constitutes a greater fraction of alloy c than does the *I* phase. This means that a higher cooling rate promotes its formation. TEM analysis indicates that in alloy C, besides the *I* phase, there exists a crystalline hexagonal phase (*H* phase) with the lattice parameters of  $a = 0.48$  nm and  $c = 0.58$  nm. The phase spacing  $d$  values of the *H* phase calculated from the  $a$  and  $c$  values are consistent with those of the unknown phase determined in the XRD pattern. Thus, it is concluded that the unknown phase is the *H* phase. Fig. 2 is the electron diffraction pattern of the *H* phase.

Figs. 3 and 4 show the XRD patterns of heat treated alloys B and C. For alloy B, the intensity of the *H* phase peak starts to decrease at 300°C. Meanwhile, obvious diffraction peak widening occurs. The *H* phase still exists at 500°C and disappears at 600~700°C while the alloy contains almost only *I* phase due to the transformation of the *H* phase to the *I* phase. some new crystalline phase appears at 800°C, and the dominant constituent of the alloy is the crystalline phase at 900°C, while the *I* phase disappears.

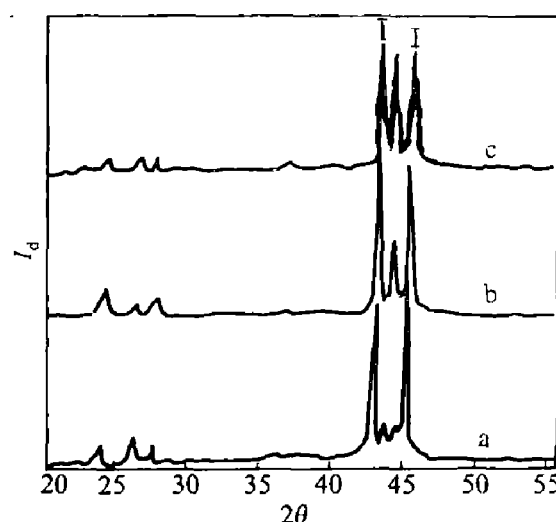


Fig. 1 XRD patterns of three alloys

a—alloy A : b—alloy B: c—alloy C

#### 3.2 The Crystalline and Quasicrystalline Phase Transformation During Annealing

Compared with alloy B, alloy C displays different phase transformation behaviour during annealing. No obvious changes can be seen at 400°C, and the *H* phase peak breaks into two at 500°C, while one of the crystalline peaks becomes higher and the quasicrystalline phase peak lower as the holding time is prolonged. The *I* phase intensity is very weak at 600°C and disappears at higher temperature while the crystalline phase becomes the dominant phase.

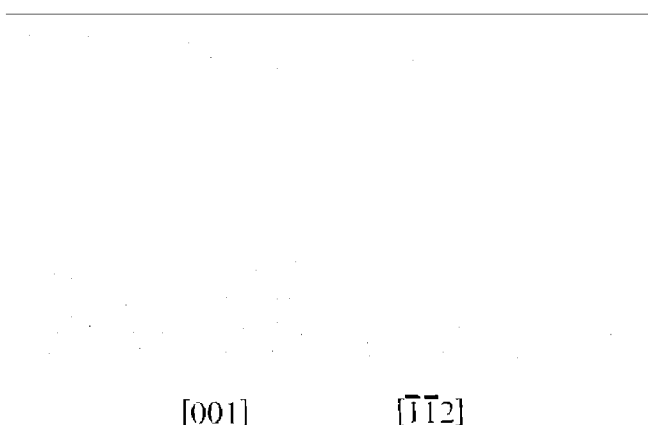


Fig. 2 Electron diffraction pattern of *H* phase

TEM observation revealed that, for the alloy C treated at 600°C for 15 min, it is difficult

to find the *I* phase: the main crystalline phases are  $\text{Al}_{13}\text{Fe}_4$ , *B*<sub>2</sub>-Base phase<sup>[4]</sup> and cubic *C* phase<sup>[5]</sup>.

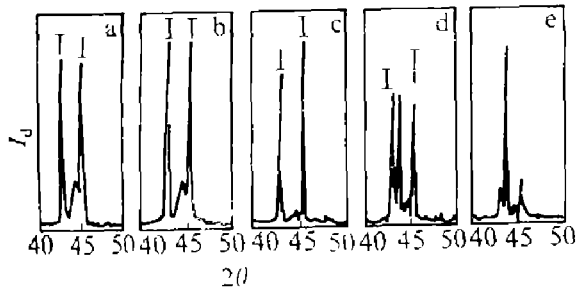


Fig.3 XRD patterns of alloy B isothermally treated at various temperatures

a—300°C; b—500°C; c—700°C; d—800°C;  
e—900°C. Annealing time  $t=15$  min

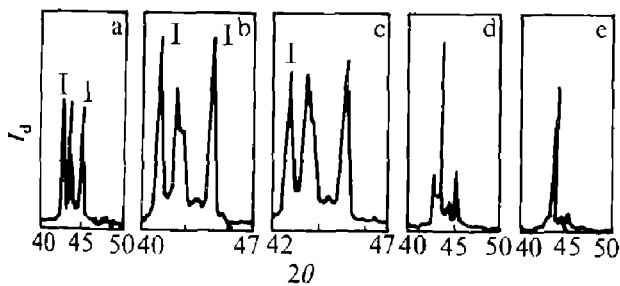


Fig.4 XRD patterns of alloy C isothermally treated at various temperatures

a—400°C, 15 min; b—500°C, 15 min; c—500°C, 30 min;  
d—600°C, 15 min; e—800°C. Annealing time  $t=15$  min

### 3.3 The Effect of Low Temperature Annealing on the High Temperature Phase Transformation of Alloys B and C

As shown in Fig 5, alloy B was first annealed at 100°C, 200°C for 60 min, then isothermally treated at a higher temperature. The XRD patterns of the 100°C treated alloy and the raw powder are not different, the phase transformation at 300~400°C is also similar to that in the raw powder, but the *H* phase disappears at 500°C, the results are similar again at 800°C, and the crystalline phase content is much lower in the pre-treated powder. The *I* phase disappears in both powders at 900°C.

In the XRD pattern of powder pre-treated at 200°C (Fig. 6), an obvious widening of the

peaks of the *H* phase can be seen. This means that some structural change occurred in the *H* phase. In the following treatment at 300~700°C, the phase transformation behaviour is similar to that of powder pre-treated at 100°C. The *H* phase disappears at 800°C. The crystalline phase content in the sample is still very small at 900°C, and this is quite different from what is observed in the raw powder and the 100°C pre-treated powder.

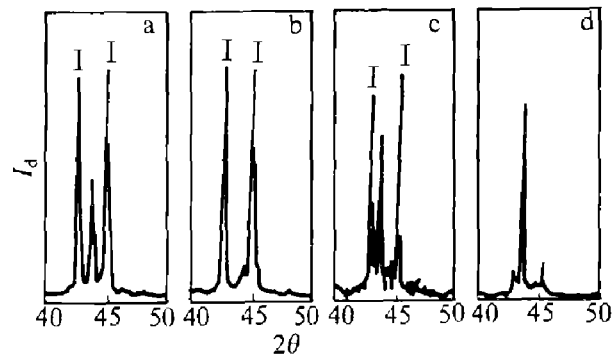


Fig.5 XRD patterns of alloy B annealed at various temperatures

a—100°C, 60 min; b—(100°C, 60 min) + (500°C, 15 min);  
c—(100°C, 60 min) + (800°C, 15 min);  
d—(100°C, 60 min) + (900°C, 15 min)

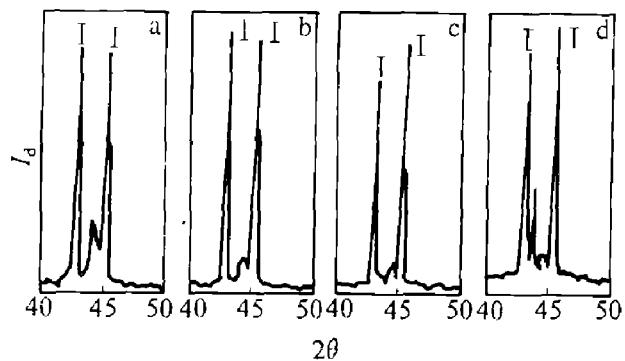


Fig.6 XRD patterns of alloy B annealed at various temperatures

a—200°C, 60 min; b—(200°C, 60 min) + (500°C, 15 min);  
c—(200°C, 60 min) + (800°C, 15 min);  
d—(200°C, 60 min) + (900°C, 15 min)

These facts revealed that the low temperature annealing obviously alters the phase transformations of alloys during high tempera-

ture annealing processes, extending the temperature range in which the *I* phase remains stable, and lowering the *H* phase disappearing temperature. The higher the preannealing temperature, the more pronounced this tendency. In contrast this alloy C, the preannealing at 100~400°C did not affect the phase transformation of the alloy at a higher temperature; the *I* phase still changes into the crystalline phase at 500~800°C.

Even though the *H* phase in alloy C can not transform into the quasicrystalline *I* phase at normal pressures, under a pressure of 6 GPa and at 600~800°C, most of the crystalline phase can transform into the quasicrystalline phase<sup>[6]</sup>.

Although both alloys B and C are composed of *I* phase and *H* phase, they exhibit totally different phase transformation behaviours in the annealing process. This means that there are some differences in the relative phase stabilities of the two alloys. This may proceed from the cooling rate differences in the alloy preparation methods, so many researchers have obtained different results<sup>[1,5]</sup>.

For alloy B, some structural relaxations may take place during the low temperature annealing. This process adjusted the phase structure formed in the quenching process. Therefore, adjusted the relative thermodynamic potential of the *I* phase and *H* phase. Despite the fact that a difference in the XRD patterns of alloy B before and after the 100°C 1 h treatment cannot be observed, some structural defects caused by rapid solidification have been eliminated. The thermodynamic status of the phases has been changed. After the treatment at 200°C 1 h, a change in the XRD pattern is observable. These changes result in the phase transformation behaviour difference.

For alloy C, the *H* phase has higher energy for its higher forming cooling rate. The low

temperature annealing only eliminates a small fraction of the structural defects; it can not alter the relative thermodynamic status of the phases. Thus it did not affect the high temperature phase transformation.

#### 4 CONCLUSIONS

(1) By means of rapid solidification with water and liquid N<sub>2</sub> as coolant, the two Al<sub>65</sub>Cu<sub>20</sub>Fe<sub>15</sub> powders obtained are composed of a quasicrystalline *I* phase and a crystalline hexagonal phase. The crystalline phase content in the former powder is obviously lower compared to the latter:

(2) In the water-quenched powder, the crystalline to quasicrystalline phase transformation begins at 500°C; the crystalline phase disappears at 600~700°C, while the crystalline phase content starts to increase until the powder almost only contains the crystalline phase. In the liquid N<sub>2</sub> quenched powder, the quasicrystalline phase starts to transform into the crystalline phase at about 500°C and finally disappears at temperatures above 600°C:

(3) 100°C, 200°C annealing extends the phase stable temperature range of the *I* phase in water quenched powder. 100~400°C annealing did not affect the transformation behaviour of the quasicrystalline and crystalline phases.

#### REFERENCES

- 1 Tsai, A. P. *et al.*, JPN J Appl Phys, 1987, 26, L1505.
- 2 Ishimasa, T. *et al.*, In: Proc 2nd China-Japan Seminar on Quasicrystals, Beijing, China, 1990
- 3 Chen, Zhenhua *et al.*, CHN Patent 8821237, 5 Feb., 1988.
- 4 Zhang, Z. *et al.*, Scripta Metallurgica & Materialia, 1990, 24(10), 329.
- 5 Dong, C. *et al.*, J Phys Condens Matter, 1990, 2, 6331.
- 6 Jiang, Xiangyang *et al.*, Scripta Metallurgica & Materialia, 1992, 26(10).