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Microstructures and mechanical properties of conventionally solidified $Al_{63}Cu_{25}Fe_{12}$ alloy

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ABSTRACT

In this study, the microstructures and mechanical properties of conventionally solidified $Al_{63}Cu_{25}Fe_{12}$ alloy after different heat-treatments were investigated. The microstructures of the as-cast and subsequently heat-treated samples were investigated by X-ray diffraction (XRD), scanning electron microscopy (SEM) and differential thermal analysis (DTA). The XRD results showed the presence of quasicrystalline icosahedral phase (i-phase) together with crystalline phases corresponding to β-AlFe(Cu) solid solution phase (β-phase) and τ-AlCu(Fe) solid solution phase (τ-phase). The SEM investigations clearly showed the formation of i-phase with pentagonal dodecahedra structure. However, the i-phase together with β-phase was also observed in the heat-treated samples and the peak intensity of the β-phase decreased with increasing heat-treatment temperature. From the DTA curves, the melting point of i-phase was determined as 890 °C for this alloy composition. Mechanical properties of the as-cast and subsequently heat-treated samples were measured by a Vickers indenter. Results showed that the microhardness (*HV*) and the elastic modulus (*E*) of the as-cast sample were around 598 kg fmm^{-2} (5.86 GPa) and 104 GPa, respectively. In addition, the characteristic of material plasticity (δ_H) value was calculated to be 0.54.
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1. Introduction

Since the production of the first quasicrystalline phase in the Al–Mn system by a rapid solidification technique [1], a number of studies have been carried out on the formation, structure and properties of the quasicrystalline alloys. Unique structure and unusual properties of these materials make them attractive materials in potential industrial applications, such as hydrogen storage, solar power, thermal insulation, surface engineering, etc. [2,3]. Up to now, the i-phase has been obtained in over 100 different alloy systems, and the majority of them have consisted of Al-based alloys. Among them, Al–Cu–Fe alloys have great importance because of their nontoxicity, easy availability, thermal stability and the favourable costs of their alloying elements [3,4].

It has been reported that Al–Cu–Fe quasicrystal materials can be prepared by conventional solidification, rapid solidification, mechanical alloying, laser- or electron-beam superficial fusion and electron irradiation [5–7]. However, in the $Al_{65}Cu_{20}Fe_{15}$ alloy thermodynamically stable i-phase was obtained by conventional solidification technique [8]. Therefore, the discovery of thermodynamically stable i-phase in Al–Cu–Fe alloy has opened a new way for its experimental investigations. Moreover, it has been reported that the i-phase in $Al_{63}Cu_{25}Fe_{12}$ alloy mostly co-exists with τ-phase which is a metastable phase with a lower melting temperature [9]. On further heat-treatments, the metastable τ-phase disappears and the amount of the i-phase increases.

Although there have been so many investigations on the formation, microstructure, surface morphology, thermal stability and mechanical properties of Al–Cu–Fe quasicrystals, the conventionally solidified $Al_{63}Cu_{25}Fe_{12}$ alloy has not yet been discussed in detail as a function of the heat-treatments. The aim of the present work is to investigate the formation of the i-phase and the mechanical properties during heat-treatments. Microstructures, thermal behaviours and mechanical properties of both as-cast and heat-treated samples were examined by combination of X-ray diffraction (XRD), scanning electron microscopy (SEM), differential thermal analysis (DTA) and Vickers microhardness techniques.

2. Experimental

A master alloy with a nominal composition of $Al_{63}Cu_{25}Fe_{12}$ (in atomic percent) was prepared by induction melting a mixture of high purity (99.99%) Al, Cu, Fe under a dynamic purified argon atmosphere. The master alloy was cut into suitably shaped pieces for the subsequent heat-treatments. The alloy ingots were heat-treated at 200, 400 and 600 °C for up to 4 h followed by slow cooling to room temperature under air atmosphere. Structural characterization of all these samples was done by X-ray diffraction (XRD) using a Philips X'Pert powder diffractometer with Cu Kα radiation and by scanning electron microscopy (SEM) using a JEOL 5400 with an accelerating voltage of 15 kV. Thermal events were investigated for both master

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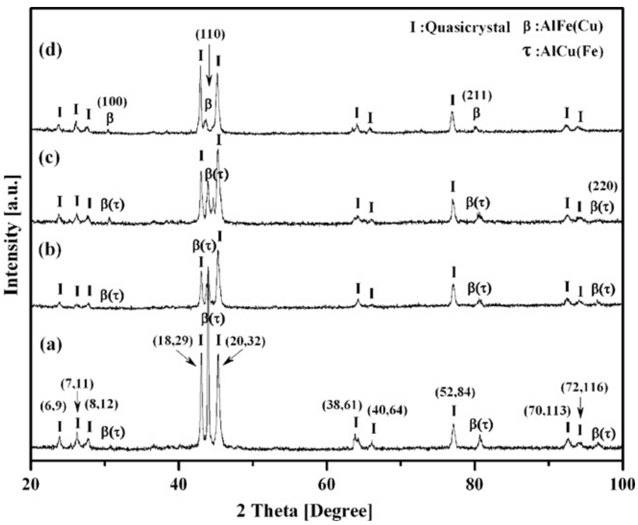

Fig. 1. XRD patterns for conventionally solidified $Al_{63}Cu_{25}Fe_{12}$ alloy before and after heat-treatments: (a) as-cast, (b) 200 °C, (c) 400 °C, and (d) 600 °C.

alloy and heat-treated alloys by differential thermal analysis (DTA) using a Perkin-Elmer Diamond TG/DTA at a heating rate of 20 °C min^{-1} under dynamic nitrogen atmosphere. Mechanical properties of the as-cast and the heat-treated alloys were measured by a Vickers indenter. The Vickers indenter was used in a dynamic ultra microhardness tester (Shimadzu, DUH-W201S). Vickers microhardness measurements were done by using a 0.98 N load and the loading rate was 23.5 mN s^{-1}. For a particular load at least five indentation tests were made and the experimental errors were also analysed.

3. Results and discussion

Fig. 1 shows the X-ray diffraction patterns for conventionally solidified $Al_{63}Cu_{25}Fe_{12}$ alloy before and after heat-treatments at 200, 400 and 600 °C for up to 4 h. The XRD result for the as-cast $Al_{63}Cu_{25}Fe_{12}$ alloy shows the presence of icosahedral quasicrystalline phase (i-phase) together with crystalline phases. The peaks corresponding to the i-phase and crystalline phases were completely indexed using Cahn indices (N,M) [10] and Miller indices, respectively. The crystalline phases were identified as cubic β-AlFe(Cu) solid solution phase (β-phase) and cubic τ-AlCu(Fe) solid solution phase (τ-phase). As shown in Fig. 1, the diffraction peaks of the τ-phase exactly superimpose on the peaks of the β-phase. Therefore, τ-phase was also denoted with parenthesis besides each β-phase. However, the XRD patterns are not suitable to discriminate the τ-phase from the β-phase since owing to same crystal structure of CsCl type cubic and very similar lattice parameter (a = 0.2910 nm) in the solidification product [11–13]. In the as-cast sample the peak intensity of the β(τ)-phase (1 1 0) at observed around $2\theta = 44°$ is the strongest. Also, the lattice parameter of the β(τ)-(1 1 0) peak was found to be 0.2912 nm. During the heat-treatments at 200, 400 and 600 °C, the intensity of the β(τ)-(1 1 0) peak, at observed around $2\theta = 44°$, significantly decreased with increasing heat-treatment temperature. Similarly, the intensity of the β(τ)-(2 2 0) peak, at observed around $2\theta = 96°$, decreased with the heat-treatment temperature and disappeared after 600 °C for 4 h. However, no significant difference was observed in the peak intensities of the i-phase during heat-treatments. Therefore, this is an indication that the i-phase is the stable phase for this composition under the processing conditions studied. From three most intense diffraction peaks of i-phase ((18,29), (20,32) and (52,84)), the six-dimensional quasilattice parameter (a_{6D}) was calculated and optimised to be 0.630 nm.

On the other hand, it has been reported that the τ-phase is a metastable phase with a lower melting temperature and the β-phase is a stable phase, containing more amount of Fe content, with a higher melting temperature [11]. Thus, after heat-treatment at 600 °C, the metastable τ-phase disappeared and probably transformed into the i-phase and the β-phase. Therefore, XRD pattern of the $Al_{63}Cu_{25}Fe_{12}$ alloy after heat-treated at 600 °C for 4 h consists of i-phase and β-phase. Also using a peak-fitting program with the Pearson VII function, volume fraction of the i-phase was calculated by the ratio of sum of integrated areas of all diffraction peaks for the i-phase [14]. The resultant volume fraction of the i-phase was calculated to be around 62% in the as-cast sample and 72%, 73% and 88% in the heat-treated samples at 200, 400 and 600 °C for 4 h, respectively. These results indicate that as the heat-treatment temperature increased, the amount of the i-phase increased, corresponding with the relative decrease of the τ- and β-phases. This is in good agreement with other results [11–14].

In order to understand the microstructural evolution depending on the heat-treatment in conventionally solidified $Al_{63}Cu_{25}Fe_{12}$ alloy, the samples were examined by SEM. Fig. 2 shows the SEM micrographs for the as-cast and the heat-treated samples. These micrographs show similar morphologies. All micrographs revealed the formation of pentagonal dodecahedra in the quasicrystalline phase. These results confirm the XRD analysis. This observation is also in agreement with the earlier reported study for the conventionally solidified $Al_{65}Cu_{20}Fe_{15}$ by Tsai et al. [8].

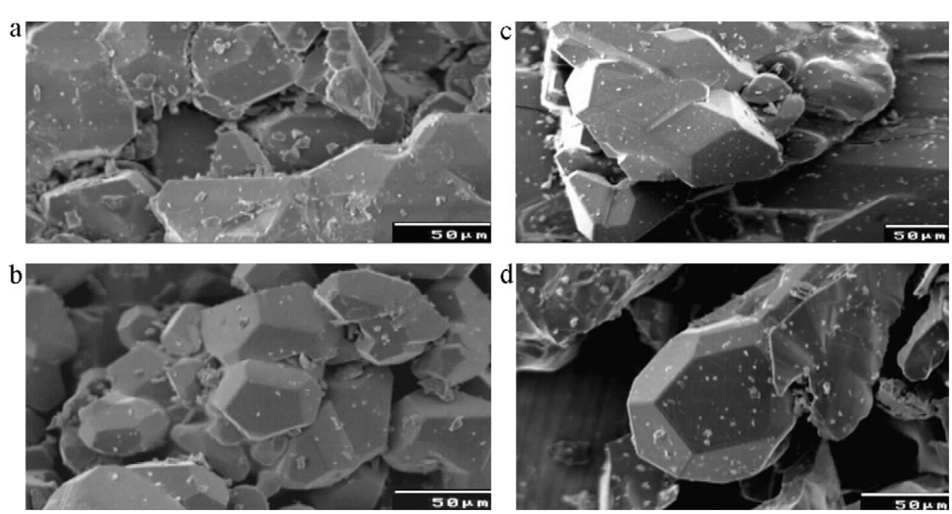

Fig. 2. SEM micrographs for conventionally solidified $Al_{63}Cu_{25}Fe_{12}$ alloy before and after heat-treatments: (a) as-cast, (b) 200 °C, (c) 400 °C, and (d) 600 °C.

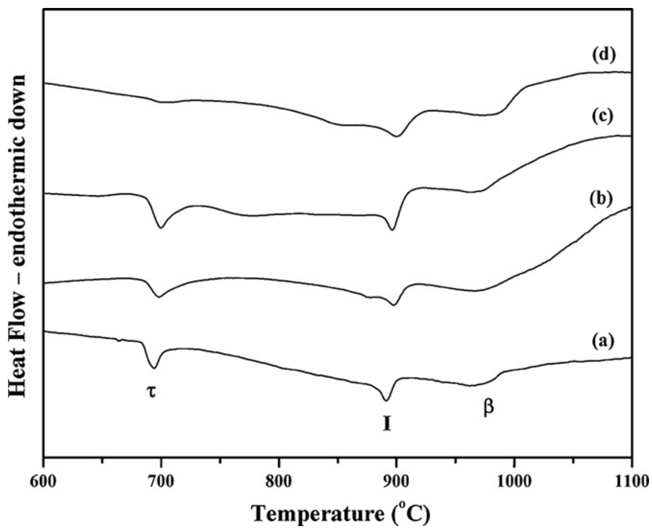

Fig. 3. DTA curves for conventionally solidified $Al_{63}Cu_{25}Fe_{12}$ alloy before and after heat-treatments: (a) as-cast, (b) 200 °C, (c) 400 °C, and (d) 600 °C.

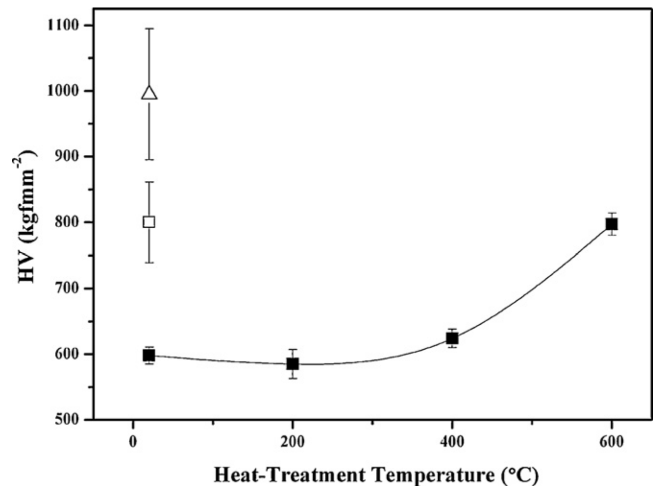

Fig. 4. Variation of Vickers microhardness during heat-treatments.

Fig. 3 shows the DTA curves of the as-cast and the heat-treated $Al_{63}Cu_{25}Fe_{12}$ ingots during heating from 600 to 1100 °C with a heating rate of 20 °C min^{-1}. DTA curves for all samples showed two major and one minor endothermic peaks at around 700, 890 and 980 °C, respectively. The first major endothermic peak at around 700 °C is considered to correspond to the dissolution of the τ-phase. Recently it is reported that the τ-phase is a metastable phase with a lower melting temperature [11]. As it shown in Fig. 3(d), the intensity of the first endothermic peak is decreased with the heat-treatment at 600 °C for 4 h. This result is consisted with the XRD observations. As mentioned above, the XRD results of the $Al_{63}Cu_{25}Fe_{12}$ alloy indicate the decreasing of β(τ)-phase during heat-treatments. The second sharp endothermic peak at 890 °C for all samples is related to the fusion of i-phase. Therefore, this result indicates that the quasicrystal phase has the melting point at 890 °C for this alloy composition. This is again consistent with the XRD results. As seen in Fig. 1, XRD patterns of the $Al_{63}Cu_{25}Fe_{12}$ alloy heat-treated up to 600 °C have shown no significant difference even in the peak position and peak intensity of the i-phase. This DTA result is quite similar to the earlier reported study for the $Al_{65}Cu_{20}Fe_{15}$ by Tsai et al. [8]. The third broad endothermic effect is related with the melting of the β-phase. Above 1000 °C, the as-cast and the heat-treated samples were completely melted. These results are supported by the earlier report of Lee et al. [11].

Mechanical properties of the as-cast and subsequently heat-treated samples were measured by a Vickers indenter. Fig. 4 shows the Vickers microhardness (*HV*) results, where solid square symbols represent the microhardness values of the as-cast and subsequently heat-treated samples. For comparison, open triangle and open square symbols represent, respectively, the microhardness values at room temperature for i-phases in Al–Cu–Fe alloys previously reported by Köster et al. [15] and Giacometti et al. [16]. For the as-cast and the subsequently heat-treated samples, the *HV* values gradually increase with the increasing temperature. This increase in the *HV* values corresponds to the increasing volume fraction of i-phase during heat-treatments, as mentioned in the XRD results. The present result is consistent with the hardness variation dependence of the i-phase amount in the alloy [17]. In this research the microhardness of the as-cast sample was found to be 598 kg fmm^{-2} (5.86 GPa). However, the highest microhardness value in the heat-treated samples was found to be 797 ± 17 kg fmm^{-2} at 600 °C, which is close to that reported by Lee et al. [11], Giacometti et al. [16] and Dubois et al. [18]. But it is lower than about 200 kg fmm^{-2} for single i-phase alloy obtained by Köster et al. [15]. Here, it is worth noting that the volume fraction of the i-phase was found to be around 88% in the heat-treated sample having the highest microhardness value. Also, as seen in Fig. 4, the volume fraction of the i-phase was around 95% in the Al–Cu–Fe alloy reported by Giacometti et al. [16]. Comparing the microhardness values, it was found that the HV value for single i-phase alloy obtained by Köster et al. [15] is the highest because of the containing more i-phase.

In the present study the elastic modulus (E) of the as-cast sample was found to be 104 ± 5 GPa at room temperature. It is noted that the elastic modulus (E) can be determined using an empirical relation, defined as $E = 0.123T_m - 34$, applicable for Al-based alloys [19]. Here, T_m is the melting temperature, which was determined as 1163 K by the DTA analysis. Thus, E was estimated to be about 109 GPa, which is very close to the experimentally measured E value (104 ± 5 GPa). This value matches closely with that of $Al_{64}Cu_{22}Fe_{14}$ alloy [15], and significantly larger than for conventional Al-alloys (about 70 GPa) [20].

Furthermore, the plasticity factor (δ_H) which is qualifying the brittleness of a material was calculated using the expression; $\delta_H = 1 - 14.3(1 - \upsilon - 2\upsilon^2)HV/E$ [21], where υ is the Poisson coefficient, *HV* is the microhardness and E is the elastic modulus. For the as-cast sample, $HV = 5.86$ GPa, $E = 104$ GPa and $\upsilon = 0.28$ in agreement with the measured elastic constants of Al–Pd–Mn quasicrystal [22], and the plasticity factor (δ_H) was calculated to be about 0.54. According to previously reported by Milman et al. [21], the conventionally brittle materials always have a value of $\delta_H \leq 0.9$. Thus, conventionally solidified $Al_{63}Cu_{25}Fe_{12}$ alloy is identified to be brittle material at room temperature. This is also in good agreement with other results [23,24].

4. Conclusions

In the present study, the formation of icosahedral quasicrystalline and crystalline phases in conventionally solidified Al–Cu–Fe alloys with heat-treatments up to 600 °C were investigated and the results were summarized as follows.

The structure of the as-cast $Al_{63}Cu_{25}Fe_{12}$ alloy is consisted of a mixture of quasicrystalline i-phase and crystalline β(τ)-phase. During the heat-treatments at 200, 400 and 600 °C, the intensity of the β(τ)-phase significantly decreased with increasing heat-treatment temperature.

SEM micrographs for both as-cast and heat-treated samples revealed the formation of pentagonal dodecahedra in the quasicrystalline phase.

From the DTA curves, the melting point of i-phase was determined as 890 °C for the as-cast and heat-treated $Al_{63}Cu_{25}Fe_{12}$ alloys.

The microhardness (*HV*), the elastic modulus (*E*) and the plasticity factor (δ_H) of the as-cast sample were found to be 598 kg fmm^{-2} (5.86 GPa), 104 GPa and 0.54, respectively.

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