FORMATION OF MICROSTRUCTURE OF AL-CU-FE ALLOY WITH QUASICRYSTALLINE PHASES DURING HEAT TREATMENT AND MECHANICAL MILLING

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Abstract

Stable quasicrystals Al-Cu-Fe can be subjected to high energy treatment in order to grind them up and then use as nano-reinforcers of aluminum composite materials. Mechanical milling of alloy Al - 40 wt.% Cu - 17 wt.% Fe were carried out in a planetary ball mill. Granules with an average size of 35-40 μm and the fine internal microstructure are formed. Mechanical milling after preliminary heat treatment at temperatures 750 ^{0}C leads to the fact that the quasicrystalline phase is ground more than in the case of mechanical milling of cast alloy. Heat treatment leads to a more perfect quasicrystalline structure of the phase and the partial formation of quasicrystalline phase of another composition.

1 Introduction

Quasicrystals are the solids with an ordered structure, symmetry banned in crystallography, and non-periodic translational order. In recent years the great interest in the study of quasicrystals, their structure, properties and possible applications is observed. Quasicrystalline phases of Al-Cu-Fe are in particular interest, primarily because of the availability of alloying elements. Icosahedral quasicrystals Al-Cu-Fe due to unusual internal structure have a number of promising properties: high strength, wear resistance, low coefficient of friction, etc. [1]. Therefore, quasicrystals Al-Cu-Fe are considered as a possible material for wear-resistant powder coatings [1-2] and as a particle reinforcers in dispersion-strengthened composite materials [3-4].
The main methods of obtaining quasi-crystalline phases are the creation of the metastable state of metallic materials: rapid crystallization of powders from the melt or mechanical alloying of elementary powders mixture [1]. Formed metastable quasicrystalline phases are sensitive to subsequent treatments (thermal and mechanical). Apart is the method of obtaining the stable quasicrystals by conventional crystallization of massive castings [1, 5]. Stable quasicrystals Al-Cu-Fe can be subjected to high energy treatment in order to grind them up and then use as nano-reinforcers of aluminum composite materials.

2. Materials and Testing Methods

Ingots of alloy Al - 40 wt.% Cu - 17 wt.% Fe (Al - 25 at.% Cu - 12,5 at.% Fe) 200x30x15 mm in size were obtained by casting in a graphite mold with temperature of 1250 ^{0}C. The initial materials were aluminum (99.99% purity), copper (99.90%) and iron (99.90 % purity). Melting was carried in Nabertherm TOP45 furnace in graphite-fireclay crucible in air atmosphere. Heat treatment was carried out in electrical furnaces also in air atmosphere.

Diffractometric analysis was performed on X-ray diffractometer Bruker D8 ADVANCE (Germany) using a monochromator on diffracted beam and the characteristic radiation of CuK_{α} (λ= 0,154178 nm). Survey was carried out in step mode with step of $2\theta = 0{,}05^0$, time in step was 5 s. Calculation of diffraction patterns and phase identification were carried out using software «EVA» and database of powder diffraction patterns PDF2. The size of coherent scattering regions (CSR) was estimated by Williamson-Hall method.

Metallographic examination was performed with a scanning electron microscope Quanta 200 3D. Metallographic samples were prepared by standard mechanical polishing. To identify the microstructure the Keller solution (5% HF + 5% HCl + 5% HNO_3 in water) was used.

Mechanical milling was carried out in planetary ball mill Retsch PM400 in argon atmosphere.

3. Results and Discussion

3.1. Microstructure in as-cast condition and after heat treatment

Figure 1 shows the diffraction patterns of alloy powder Al - 40 wt. % Cu - 17 wt. % Fe in as-cast condition. At present the main diffraction reflections from two phases, the icosahedral quasicrystalline phase $Al_{65}Cu_{20}Fe_{15}$ (Q) and the cubic β-Al(Cu,Fe), as well as small peaks of monoclinic phase λ-$Al_{13}Fe_4$.

The microstructure of the sample in as-cast condition (figure 2a) shows three phase regions (marked 1, 2 and 3). Based on X-ray diffractometry, the region 2, which occupies a large area, can be identified as the quasicrystalline phase $Al_{65}Cu_{20}Fe_{15}$. The phase of Al(Cu,Fe) with small amount of dissolved iron (region 1) and a small quantity of phase $Al_{13}Fe_4$ (Region 3) also presents in the structure. Electron diffraction pattern of phase 2 (Figure 2b) shows the presence of five-fold symmetry, which confirms the presence of the icosahedral phase $Al_{65}Cu_{20}Fe_{15}$.

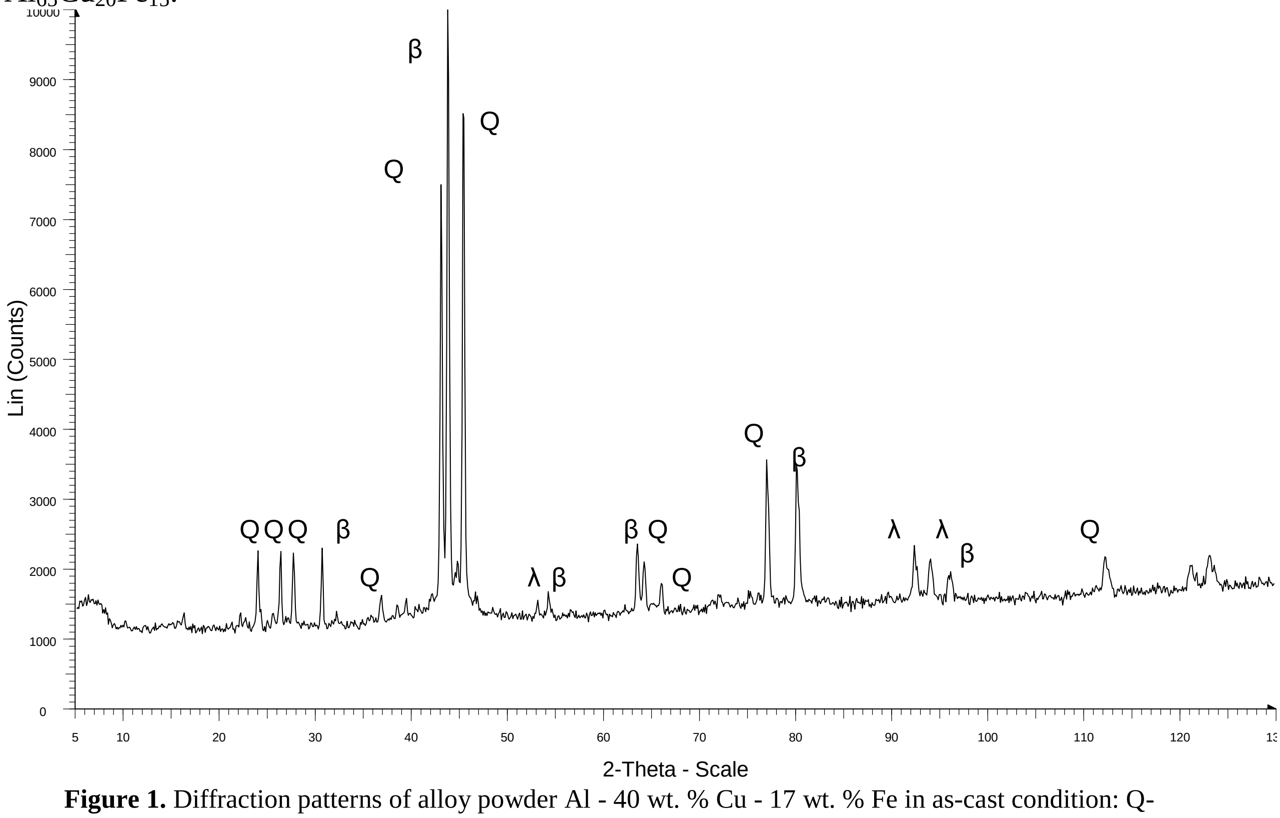
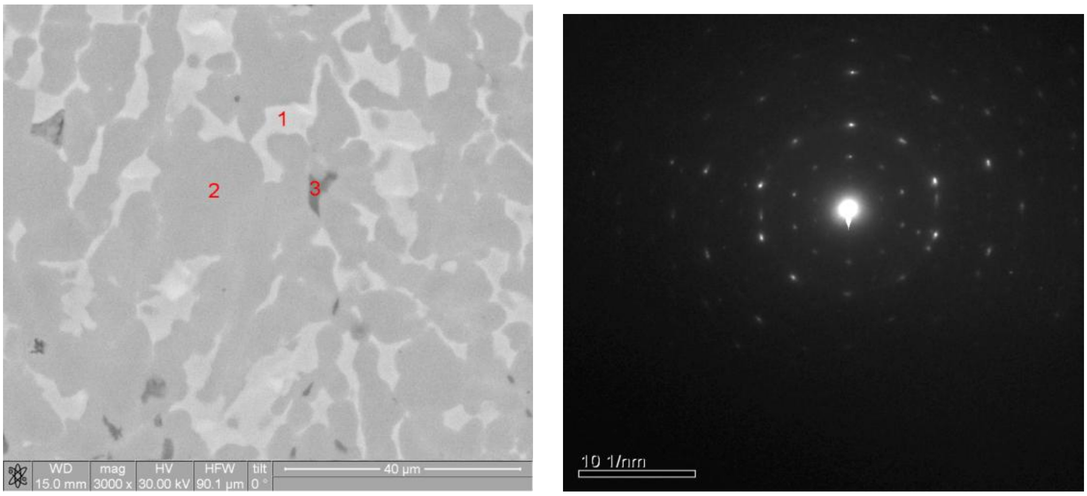


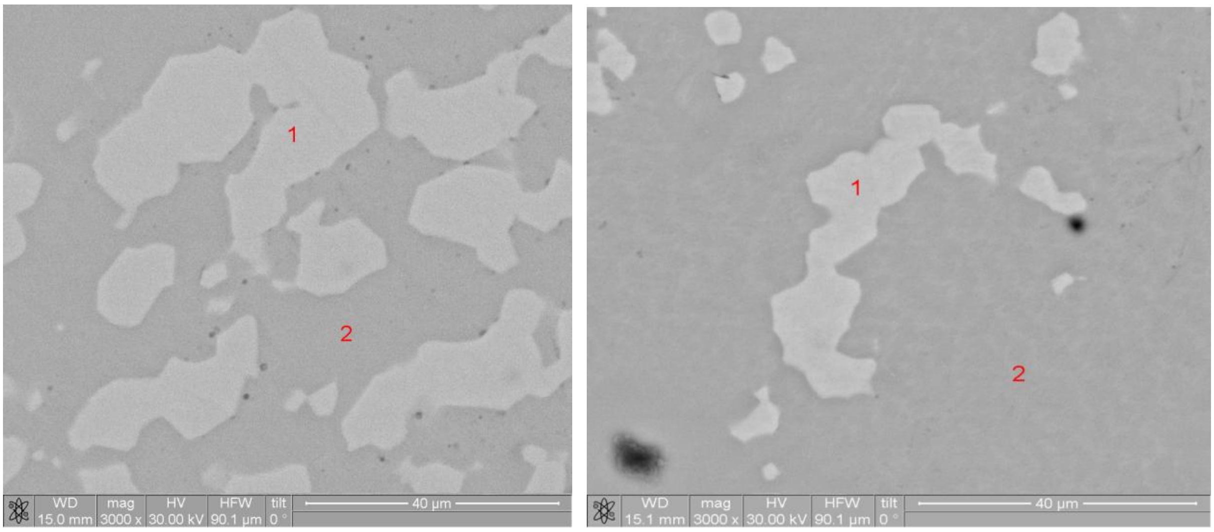
Figure 1. Diffraction patterns of alloy powder Al - 40 wt. % Cu - 17 wt. % Fe in as-cast condition: Q-$Al_{65}Cu_{20}Fe_{15}$, β-AlCu(Fe), λ- $Al_{13}Fe_4$



a b

Figure 2. SEM image of structure of alloy Al - 40 wt. % Cu - 17 wt. % Fe in as-cast condition (a) and electron diffraction pattern of region 2 (b)

Annealing of the original alloy at 750 ^{0}C for 5 h leads to complete dissolution of the phase $Al_{13}Fe_4$ (Figure 3a). Increase of holding time up to 15 h leads to partial dissolution of the phase Al(Cu,Fe) (Region 1), but the formation of a single-phase quasicrystalline structure is not occur(Figure 3b).



a b

Figure 3. SEM image of structure of alloy Al - 40 wt. % Cu - 17 wt. % Fe after annealing at 750 ^{0}C for 5 (a) and 15 (b) hours.

Fig. 4 shows the part of diffraction pattern with the most intense diffraction peaks taken after annealing at 750 ^{0}C for 15 h with air cooling and furnace cooling. After slow furnace cooling from the annealing temperature the quasicrystalline phase $Al_{65}Cu_{20}Fe_{15}$ is transformed into quasicrystalline phase $Al_{13}Cu_4Fe_3$. Diffraction pattern of $Al_{13}Cu_4Fe_3$ phase characterized by large number of reflections in comparison with the phase $Al_{65}Cu_{20}Fe_{15}$, in particular there is a distinct peak at 43.2^0. But the major peaks for both phases coincide (at $2\theta = 43.0^0$ and 45.3^0). This can be explained by the fact that there is a quasicrystalline module which is the same for both phases, but the translation of this quasicrystalline module is different. After slow cooling, i.e. in more equilibrium conditions, $Al_{13}Cu_4Fe_3$ phase with more complicated lattice is formed.

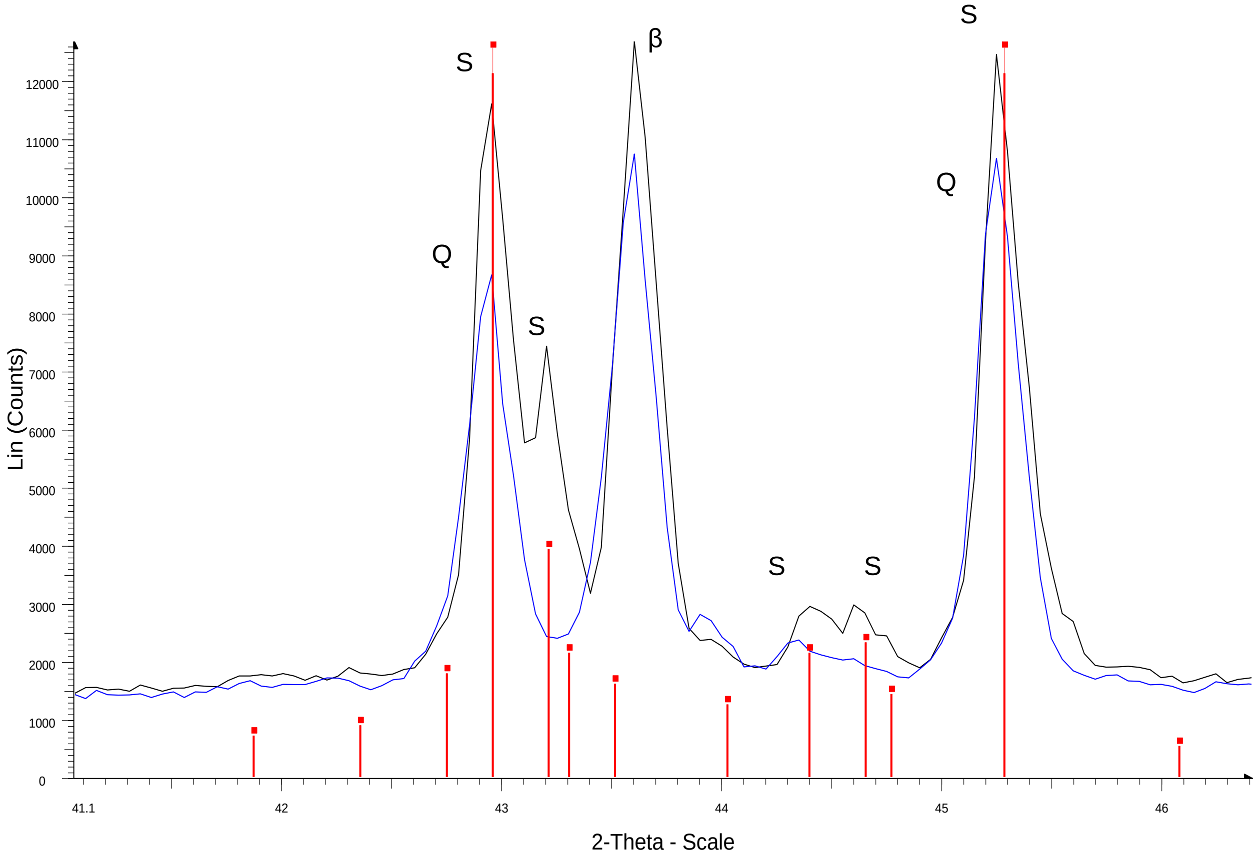
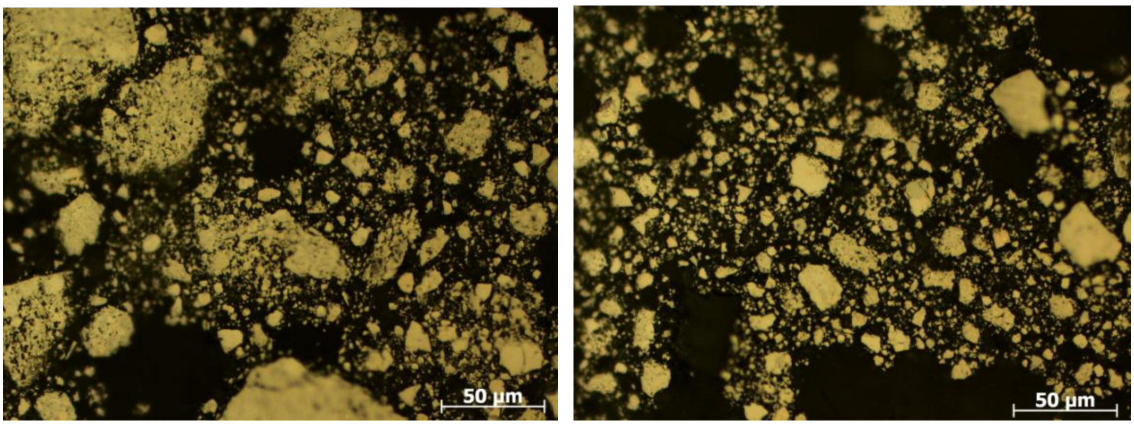


Figure 4. Diffraction patterns after annealing at 750 ^{0}C with subsequent air cooling (blue curve) and furnace cooling (black curve): Q- $Al_{65}Cu_{20}Fe_{15}$,S- $Al_{13}Cu_4Fe_3$, β-AlCu(Fe)

3.2. Mechanical milling

Mechanical milling of alloy particles Al - 40 wt. % Cu - 17 wt. % Fe in the cast state and after annealing at 750 ^{0}C with slow cooling was carried out in planetary ball mill Retsch PM400 in an argon atmosphere. Granules morphology is shown in fig. 5. As a result of mechanical milling granules with an average size of 35-40 μm and fine internal microstructure are formed.



a b

Figure 5. Granules of Al - 40 wt. % Cu - 17 wt. % Fe after mechanical milling for 3 hours: a) cast material; b) after preliminary heat treatment.

The refinement of phase components can be estimated by the change of the size of coherent scattering regions (table 1). After mechanical milling the size of the CSR of the two phases (crystalline and quasicrystalline) decreases compared to initial. For material without preliminary heat treatment prior to mechanical milling the size of the CSR of $Al_{65}Cu_{20}Fe_{15}$ quasicrystalline phase is more than the CSR size of the AlCu(Fe) phase, as well as in the initial state. With increasing time of mechanical milling this dependence does not change. After 2 h of mechanical alloying of the CSR size of $Al_{65}Cu_{20}Fe_{15}$ phase is approximately 25 nm, and after 3 h - 19 nm.

State	Phase	SCR size, nm
As-cast	AlCu(Fe)	62
	$Al_{65}Cu_{20}Fe_{15}$	90
Heat treatment 750 ^{0}C	AlCu(Fe)	114
	$Al_{65}Cu_{20}Fe_{15}$	105
Mechanical milling, cast		
2 h	AlCu(Fe)	15
3 h		11
2 h	$Al_{65}Cu_{20}Fe_{15}$	26
3 h		19
Mechanical milling, heat treatment 750 ^{0}C		
2 h	AlCu(Fe)	17
3 h		11
2 h	$Al_{65}Cu_{20}Fe_{15}$	15
3 h		8

Table 1. CSR size of AlCu(Fe) and $Al_{65}Cu_{20}Fe_{15}$ phases

Mechanical milling after preliminary heat treatment allows much refines the quasicrystalline phase than in the case of mechanical milling of cast alloy. The SCR size of the quasicrystalline phase after mechanical milling for 2 h is 15 nm, for 3 hours - 8 nm. As shown above, heat treatment leads to the formation of a more perfect structure in the quasicrystalline phase and the partial formation of a quasicrystalline phase of different composition $Al_{13}Cu_4Fe_3$. As we know, the dislocation glide is almost impossible in quasicrystals, therefore, strongly reduced the capacity for plastic deformation. Therefore, most of the energy of mechanical milling goes to the grinding of the quasicrystalline phase.

Thus, the preliminary heat treatment of alloy Al - 40 wt. % Cu - 17 wt. % Fe in order to transform the quasicrystalline phases allows to accelerate the process of fining during mechanical milling.

References

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