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Driving forces of redistribution of elements during quasicrystalline phase formation under heating of mechanically alloyed Al$_{65}$Cu$_{23}$Fe$_{12}$ powder

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Abstract. Al$_{65}$Cu$_{23}$Fe$_{12}$ alloys were prepared by ball milling of the elemental powders mixture. Phase and structural transformations at heating of as-milled powders were investigated by X-ray diffraction analysis. Precision analysis of Mössbauer spectra was performed to check the adequacy of the fitting of X-ray diffraction patterns. The results were compared with the data of differential scanning and solution calorimetry, as well as with the thermodynamic literature data, in order to estimate the driving forces of redistribution of elements that preceded the formation of single-phase quasicrystalline structure. The heat of elements mixing, which is positive for Cu-Fe system and negative for Al-Fe and Al-Cu systems, was supposed to be a decisive factor for phase transformations during heating of the alloy. The correlation between sequence of phase transformations during heating and the thermodynamic data was discussed and the scheme describing phase transformations observed was proposed.

1. Introduction
In the recent 20 years, quasicrystalline (QC) materials have become an object of intensive study due to their unusual properties and a considerable promise for possible applications. Similarly to the evaluation of amorphous alloys, the evaluation of QC alloys resulted in an overthrow of the established view of structures of solids. Since this first work [1], a number of stable and metastable quasicrystals were obtained in several binary and multicomponent systems [2 – 8]. Commonly, QC phases form in systems based on Al, Mg, Zn, Zr and Ti.

The peculiarities of the electron configuration of QC phases cause their unusual properties, which may be useful for industrial application. The most important are the mechanical properties, such as high strength, hardness and wear resistance [9]. Young’s modulus of quasicrystals was measured to be 100 – 200 GPa. Hardness of Al-Cu-Fe quasicrystals is about 10% of the Young’s modulus, which is close to the ratio obtained for diamonds and some ceramics. An increase in temperature results in a significant decrease in the quasicrystal’s hardness [10].
QC phases are expected to possess high antifriction properties, because they are very hard and, simultaneously, have very low friction coefficient. Such a low friction coefficient associated with low surface energy of QC (28 mJ/m$^2$) [11]. High brittleness and, consequently, low deformability are the main disadvantages of QC phase as a material for industrial application in bulk shape. At the same time, quasicrystals offer promise as a reinforcing phase for composite materials. For instance, QC were suggested as a filling phase for Al matrix [12, 13]; also QC-filled maraging steel (yield stress of about 3 GPa) was elaborated for surgical application [14]. Recently [15, 16], Al-Cu-Fe quasicrystals have been suggested as fillers of ultra-high molecular weight polyethylene for acetabular cup prosthesis [17].

There are some other shortcomings, which may prevent a wide application of QC alloys. These shortcomings are caused by obstacles, associated with the QC producing. Stable quasicrystalline phases usually have narrow compositional range of existence: no more than 2-3 at. % for each component even in a high-temperature range of phase diagram. The restriction on the concentration ranges causes the rigid requirements on the engineering parameters and environment of the production process. One more shortcoming is caused by a significant difference in the densities of parent element (Mg, Al, Ti, i.e. lower than 4.5 g/cm$^3$) and alloying elements (transition and rare-earth metals, 7.5 g/cm$^3$ and more). These disadvantages strongly restrict the use of classic metallurgical techniques. Therefore, even in the case of stable QC production, it is necessary to apply special techniques, such as rapid quenching from the melt, roller casting, atomization of the melt by inert gas, mechanical alloying, etc, to overcome a zonal segregation.

The use of mechanical alloying (MA) method offers several advantages, which are of special importance for QC preparation. MA leads to the broadening of the existence range (phase fields) of phases, making the preparation of quasicrystals technologically easier [18, 19]. Moreover, all reactions proceed in solid state, which helps to overcome the problems of complex solidification process during conventional casting.

It has been widely reported [20 – 34] that in various systems QC phases can be obtained by means of MA. The Al-Cu-Fe system has attracted particular attention [30 – 34] because of the presence of thermodynamically stable QC phase in this system. In our earlier papers we described the phase transformations in quasicrystals obtained by MA of Al-Cu-Fe powder mixture [35 - 39]. It was found that combination of MA and subsequent heating results in the single-phase icosahedral quasicrystalline structure formation. The aim of the present study is to compare the effects of various routes of MA on the QC formation in the Al-Cu-Fe system.

2. Experimental

Powders of carbonyl iron annealed in hydrogen (99.95 % purity, particle size 5-10 µm), copper (99.9 % purity, particle size 10-25 µm) and aluminium (99.0 % purity, particle size 40-80 µm) were used as starting materials. An AGO-2U planetary ball mill with cylindrical vials (volume 160 cm$^3$ each) of hardened carbon-chromium steel and steel balls of 6 mm in diameter was used at carrier rotational velocities of 685 and 1235 rpm. The ball-to-powder mass ratio was 10:1, the milling was carried out in Ar atmosphere.

Thermal behavior of as-milled powder was examined by DSC using a DSM-2M calorimeter at the heating rate of 16 K/min in purified argon atmosphere. Annealing of samples at various temperatures was performed by means of heating the samples at a rate of 16 K/min up to the required temperature and then cooling at the rate of 64 K/min.

The powders in the as-milled state and after annealing were analyzed by X-ray diffraction using a computer controlled DRON-4 diffractometer under monochromatized CoK$\alpha$ radiation. Phase analysis was carried out using a reduced Rietveld refinement. Room temperature Mossbauer measurements were carried out by means of a constant acceleration spectrometer with a $^{57}$Co:Rh source. The isomer shifts were calculated with respect to the centroid of the $\alpha$-Fe spectrum. Current minimization routines were used to obtain the best fit of the spectra.
3. Results
A mixture of elemental powders with composition Al$_6$Cu$_3$Fe$_{12}$ was processed using the a rotational velocity of 685 rpm for two and four hours. Figure 1 shows the X-ray diffraction patterns of test samples, the results of phase analysis are shown in figure 2. As-milled for 2 h alloy contains intermetallic phases: metastable Al(CuFe) cubic phase with bcc structure A2, and a small amount of Al$_2$Cu phase. Also considerable amounts of remained Al, Cu and Fe were observed in the sample milled for 2 h. Milling for 4 hours leads to a decrease of the remained Al and Fe content, pure Cu was not detected at all in this case. The content of A2 phase increased after 4 h of milling, and a certain amount of ternary phase with D0.3-superstructure of bcc, having AlFe$_3$ as prototype, formed.

Figure 3 gives the DSC traces for these samples. The curves show a large exothermic effect with several broad peaks. To identify the nature of different steps observed in the DSC traces, samples for XRD study were heated in the calorimeter (a heating rate of 16 K/min, without exposure) up to the prescribed temperatures marked by the arrows in figure 3; heating up to the temperatures higher than 500 °C was performed using the tube furnace. In general, similar transformations occur in the samples milled for 2 and 4 h. However, the temperatures of all transformations for the samples milled for 4 h are lower than for 2 h. The powder milled for 2 h contains intermetallic phases, namely, a metastable Al(Cu,Fe) cubic phase with A2 structure (bcc), and a small amount of Al$_2$Cu phase. In addition, significant amounts of remaining unreacted Al, Cu and Fe were observed for this sample. Milling for 4 hours leads to a decrease in the contents of residual unreacted elements. In particular, no residual Cu was observed for this sample. The content of the bcc Al(Cu,Fe) phase increased after 4 h of milling, and another intermetallic phase with D0.3-fcc-type superstructure, having AlFe$_3$ as prototype, forms.

![Figure 1. X-ray diffraction patterns of Al$_6$Cu$_3$Fe$_{12}$ samples milled for (a) 2 and (b) 4 hours, annealed at the temperatures indicated.](image-url)
In both cases, during the first step of heating, the transformation of A2 structure into a B2-type phase and the formation of intermetallic compounds was observed. Furthermore, the residual pure elements dissolve in the intermetallic matrix and about 10-15 at.% of Al$_2$Cu phase form in this temperature range. At higher temperatures (350 - 500 °C for 2 h and 290 - 350 °C for 4 h), the transformation of D0.3 structure into the D8.3 structure, which has the Al$_4$Cu$_9$ phase as a prototype, was observed. A weak peak, which can be related to the Al$_9$Fe$_2$ phase with D8d structure, was observed in the diffraction patterns of the alloy, which was milled for 2 hours, after heating to 440 °C. At the temperatures between 350 °C and 440 °C, the ternary intermetallic compound Al$_7$Cu$_2$Fe forms, which is a stable phase in the concentration range close to the initial Al$_65$Cu$_23$Fe$_{12}$ composition. The process of QC phase formation starts at temperatures higher than 500 °C for 2 h and 350 °C for 4 h of milling.

At the temperatures higher than 700 °C (sample milled for 2 h) and 600 °C (sample milled for 4 h), only quasicrystalline phase pattern was identified by X-ray diffraction. Some weak peaks, which do not correspond to the known phases, also were found in the X-ray patterns of these samples. We suppose that these peaks correspond to the presence of small amounts of one of “approximant” phases, which were observed in this system [40].

Figure 4 shows the Mössbauer spectra of the samples milled for 4 and annealed at the temperatures lower than 350 °C. These spectra consist of the central component (doublet), and the sextet with the hyperfine effective magnetic field of H$_{eff} = 330$ kOe, which corresponds to residual α-Fe. Mössbauer data shows, that α-Fe remained up to 350 °C. The broad shape of X-ray patterns in Fig. 1 prevents accurate determination of the amount of α-Fe, so we used the Mössbauer data for this purpose. Spectra of samples, annealed at temperatures of 400 °C and higher display no component of...
residual iron.

Figure 5 shows the central component of Mössbauer spectra of these samples. Following the approach of [41], we fit the central component of spectra with one doublet to obtain the “effective” values of isomer shift $\delta$, quadrupole splitting $\Delta$ and linewidth $\Gamma$. The as-milled sample has a defected crystalline structure due to a high intensity of milling. Its spectrum is centered at the same position and is slightly broader than that of crystalline alloy of this composition ($\delta = 0.23$ mm/s and $\Delta = 0.41$ mm/s) [42].

![Mössbauer spectra of Al$_65$Cu$_{23}$Fe$_{12}$ samples milled for 4 hours, annealed at the temperatures indicated.](image1)

**Figure 4** Mössbauer spectra of Al$_{65}$Cu$_{23}$Fe$_{12}$ samples milled for 4 hours, annealed at the temperatures indicated.

![Central component of Mössbauer spectra of Al$_{65}$Cu$_{23}$Fe$_{12}$ samples milled for 4 hours, annealed at the temperatures indicated.](image2)

**Figure 5.** Central component of Mössbauer spectra of Al$_{65}$Cu$_{23}$Fe$_{12}$ samples milled for 4 hours, annealed at the temperatures indicated.

Heating of samples, as it was mentioned above, results in sequent transformations of phase with B2 structure into phase with D0.3, and, then, with D8.3 structures. Known data about the crystallography of these phases allows to propose that the local atomic configurations in these phase are similar, that is why no significant changes in the Mössbauer parameters of the central component were observed at heating.

As shown in Figure 6, the effective isomer shift fluctuates in the range 0.22–0.24 mm/s, while the effective quadrupole splitting decreases significantly with the temperature in the range up to 400 °C. The values known for quasicrystalline alloys of this composition ($\delta = 0.24$ mm/s and $\Delta = 0.38$ mm/s) [41 - 43] are reached only upon the annealing at 800 °C. For the samples annealed at 600 °C and 700 °C, which also possess the quasicrystalline structure according to the XRD data, these parameters are essentially different.

Analysis of distribution of quadrupole splittings $P(\Delta)$ for quasicrystalline alloys shows that the description of Mössbauer spectra by solely doublet is a too rough approximation of the experimental data [38, 44]. We fit the central component of Mössbauer spectra for all samples, staring with the as-milled one, to obtain best routines using physically correct value of linewidth $\Gamma$, which, according to
data [45], is of 20 - 23 mm/s. For as-milled sample the best fit was obtained using three doublets. Mössbauer spectra of samples, annealed at 290 and 350 °C, were also fitted by three doublets, but only one of them has the values of δ and Δ equal to the observed for as-milled sample. Comparing this data with the results of X-ray diffraction, we propose, that doublet with δ = 0.22 mm/s and Δ = 0.44 mm/s relates to the phase with D0.3 structure; correspondingly, other two doubles can be associated with the phase with A2 or B2 structure, which represents, correspondingly, disordered and ordered α-Fe based solid solution [46]. Parameters of Mössbauer spectra fitting are presented in table 1. Analyzing the literature data we observed that values of both δ and Δ are very sensitive to the chemical composition: for ordered equiatomic AlFe samples Mössbauer spectrum represents a singlet with δ = 0.28 mm/s [47], but for the solution containing 50 at. % of Al and 25 at. % of Cu, appearance of the doublet with δ = 0.33 mm/s and Δ = 0.25 mm/s was observed [48]. In our case, we found two doublets probably related to this phase. We propose that these two components relates to the ordered and disordered area of this solid solution, i.e. the A2/B2 phase, obtained by ball milling is partially disordered.

Figure 6 illustrates the changes of Mössbauer spectra parameters for coexisting phases at heating. For the doublet related to D0.3 phase, annealing temperature has no effect on δ and Δ values, whereas for D8.3 phase appearance of QC phase at 440 °C results in significant changes of δ for both doublets. It means that transformation of this phase into QC one accompanies with noticeable change of its chemical composition.

Table 1. Parameters of Mossbauer spectra components, related to coexisting phases.

| Phase | Doublet 1 | Doublet 2 |
|-------|-----------|-----------|
|       | δ, mm/s   | Δ, mm/s   | δ, mm/s   | Δ, mm/s   |
| A1/B1 | 0.44      | 0.27      | -0.02     | 0.27      |
| D0.3  | 0.22      | 0.41-0.43 | -         | -         |
| D8.3  | 0.35-0.41 | 0.42-0.44 | 0.04-0.10 | 0.37-0.44 |
| pQC (?) | 0.24 | 0.50 | 0.23 | 0.33 |
| iQC   | 0.22-0.24 | 0.42-0.49 | 0.21-0.22 | 0.18-0.24 |

Figure 6. Effect of the heating temperature on the isomer shift δ, quadrupole splitting Δ and the linewidth Γ in the case of fitting one doublet to the central component of the Mössbauer spectra of Al_{65}Cu_{23}Fe_{12} alloy, prepared by MA for 4 h.

Figure 7. Effect of the heating temperature on the isomer shift δ, quadrupole splitting Δ and the linewidth Γ in the case of fitting several components to the central component of the Mössbauer spectra of Al_{65}Cu_{23}Fe_{12} alloy, prepared by MA for 4 h.
QC phase, which appears at 440 °C, following the analysis [42], was fitted by two symmetric doublets with close isomer shifts and various quadrupole splittings. Only one doublet, related to the QC phase, possess δ and Δ values close to those obtained for stable QC phase [43]. Second doublet possesses the Δ value significantly higher than it was observed for this doublet for stable QC phase (0.33 vs. 0.24 mm/s). We propose, that in sample heated up to 440 °C, the metastable primitive cubic icosahedral quasicrystalline phase (pQC), which was found previously [36] in mechanically alloyed samples, is formed. As it was shown in [48], metastable precursor phases has a total (i.e. calculated by spectra fitting with one doublet) value of Δ = 0.41 - 0.43 mm/s, which is significantly higher than the total value for stable face-centered cubic icosahedral quasicrystal (Δ = 0.37 - 0.38 mm/s). This data confirms our assumption about pQC phase formation in this sample. Further heating of alloy up to 500 °C results to the decreasing of Δ for the second doublet to value chouse to those for stable QC phase, i.e. pQC phase transformed into the stable one.

It is noticeable, than for samples, annealed at 440 and 500 °C, no singlet, related to Al$_7$Cu$_2$Fe, was found, whereas about 10 vol. % of this phase was found by X-ray diffraction. It is associated with the very small amount of the Fe atoms, associated with this phase, to the total Fe content in alloy. In this case the X-ray diffraction is more sensitive technique.

Knowing the area of the subspectra related to each phase, we can calculate the relatively amount of Fe atoms, associated with every Fe-containing phases. Figure 8 shows the dependencies of Fe atoms amount, bonded in various phase, on the heating temperature. Unfortunately, as we do not know exactly the chemical composition of A2/B2, D0.3 and D8.3 phases, the correct quantitative comparing of the phase composition data obtained by X-ray diffraction and Mössbauer spectroscopy is not possible; nevertheless, the phase content dependencies on temperature, obtained by both method, shows the same trends, see Figs. 2 and 8. There is one more important conclusion we made: D8.3 phase, which, as it was shown on the base of Mössbauer spectra analysis, contains significant amount of iron. It means that the chemical composition of this phase is very far from its prototype Al$_4$Cu$_9$ phase. We propose, that D8.3 phase, as well as D0.3 one, formed by further ordering of A2/B2 phase, and their chemical composition changes gradually and is enough close to those for initial A2 solid solution.

For samples, heated up to 600, 700 and 800 °C, which show, according the X-ray data, the single-phase quasicrystalline structure we also fit the Mössbauer spectra with two doublet. Figure 9 shows the evolution of parameters of these doublets with the temperature. After increase of heating temperature from 700 to 800 °C, a noticeable increase in quadrupole splittings and isomer shifts was observed for both doublets. Figure 9 also shows the data, obtained in [43] for icosahedral Al-Cu-Fe quasicrystals. Comparison of the our data with literature one shows that only after heating up to 800 °C we obtain the values of hyperfine parameters close to those for well-ordered single-phase Al-Cu-Fe quasicrystal. Thus, results of precision analysis of Mössbauer spectra confirm that any structure evolution proceeds in the samples even after the single-phase quasicrystalline state is achieved according to X-ray data.

According to [40], several “approximant” phases to quasicrystalline state exist. As an approximant phase, it is supposed any structure, not necessarily periodic, whose diffraction pattern is close, regarding peak locations and intensities, to that of parent high-symmetry quasicrystal. These phases are hardly distinguished in a current X-ray diffraction pattern (only very weak peaks, which cannot be identified, were observed for the samples annealed at 600 and 700 °C), whereas Mössbauer spectroscopy is more sensitive. In our samples small amount of “approximant” phases may exist at annealing temperature higher than 600 °C. For this temperatures the XRD patterns indicate the unique presence of the quasicrystalline phase, but rather different shape of the p(Δ) distributions [38] suggests a structural inhomogeneity probably due to the short annealing time used in this case.
Thus, summarizing the dependences presented in Figs. 6, 7 and 9, we conclude that rather high chemical and structural homogeneity of as-milled powder leaves only relatively few atomic rearrangements to be accomplished to reach the final structure. Little variations measured for $\delta$ suggest that such atomic rearrangements in the entire temperature range considered produce no significant changes in spherical distribution of electrons. The observed significant changes (Fig. 6) of both $\Delta$ and $\Gamma$ above about 400 $^\circ$C reveal the passage from a disordered complex bcc structure to a structural arrangement, which is very close to the quasicrystalline state. The rise of both $\delta$ and $\Delta$ at 800 $^\circ$C may be related to the transformation of residual approximant phases into the stable icosahedral one.

4. Discussion

On the base of above-described studies, the general regularities, governing the sequence of phase transformations at heating of MA Al-Cu-Fe alloys were determined. Comparing the DSC curves (Fig. 3) with the phase evolution diagrams (Figs. 2 and 9) one can see that it is of little confidence to associate the observed DSC maximums with any particular phase transformation using the X-ray data only. Each DSC maximum corresponds to simultaneous progress of more than one phase transformations. In fact, these DSC peaks just reflect the relative maximums of solid-state reactions total rate. The formation of ternary QC alloys is accompanied by a pronounced thermal effect, the total exothermal effect determined from the DSC curves was $\sim -20$ kJ/mole, which is in good accordance with data of solution calorimetry $\sim -22$ kJ/mole. At the same time the characters of interaction in corresponding binary systems are essentially different. The Al-Cu and Al-Fe systems are characterised by negative heats of intermetallic compounds formation (-25.2 kJ/mole for AlFe, -12.2 kJ/mole for Al$_2$ Cu, and -19.9 for AlCu [50]), whereas the Fe-Cu system possesses significant positive heat of mixing ($\sim +15$ kJ/mole for equiatomic alloy [51, 52]). The forces of repulsion between Cu and Fe have a pronounced effect on the elements distributions over the phases during the transformations caused by heating.

Figure 10 shows the schemes of phase transformations for both cases considered. At the initial stage of transformations copper and iron tend to separate as much as possible, the phases, formed at these stages are based on either Cu (Al$_2$Cu) or Fe (A2/B2 or $\alpha$-Fe). Then during subsequent heating.

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**Figure 8.** Dependencies of Fe atoms amount, bonded in various phase, on the heating temperature of Al$_{65}$Cu$_{23}$Fe$_{12}$ alloy, prepared by MA for 4 h.

**Figure 9.** Effect of the heating temperature on the isomer shift $\delta$ and quadrupole splitting $\Delta$ in the case of fitting two doublets to the central component of the Mössbauer spectra of Al$_{65}$Cu$_{23}$Fe$_{12}$ alloy, prepared by MA for 4 h.
chemical composition of the phases gradually approaches an average chemical composition of the alloy. The DSC curves at the temperatures higher than 400 °C show that ternary phases are formed from the binary Al-based phases with exothermal effect. This means that, in spite of chemical repulsion of Cu and Fe atoms, the Al-based ternary phases appear to be more thermodynamically stable than the mixture of binary intermetallics. This, in turn, points to the strong atomic bonds in such ternary structures, including QC phase.

Figure 10. Schemes of phase transformations during heating of mechanically alloyed Al_{65}Cu_{23}Fe_{12} compositions.

The compositions of Al_{2}Cu_{2}Fe and D8.3 phases, which are homogenized with respect to Cu and Fe and are very close to the total chemical composition of the alloy. Transformation of these phases into the QC one is a final step of phase transformations in the system considered.

Taking into account the thermodynamic driving forces allows us also to analyze the results of quantitative phase analysis. During ball milling and subsequent heating of the elemental powder mixture (Fig. 2), the binary Al-based phases, enriched in Fe (such as A2/B2 or D0.3) forms first, and the contents of these phases is rather high, whereas the Cu-enriched phase, namely Al_{2}Cu, forms later, and amount of this phase is rather small. This is associated with the above-mentioned difference in the formation heat of Al-Fe and Al-Cu intermetallics. The formation enthalpy of equiatomic AlFe phase, which nearly corresponds to A2/B2 phase, is two times lower than the Al_{2}Cu phase formation enthalpy. Moreover, the ternary α-Fe based solid solution with high Al content possess the formation enthalpy lower than binary AlFe phase [49]. Such difference provides the predominant formation of the most energy favourable phases based on α-Fe based solid solution.

5. Conclusions

Annealing of mechanically alloyed Al-Cu-Fe powders gives rise to a complex transformation and finally provides the formation of a quasicrystalline structure. The transformations proceed at lower temperatures if samples are milled for longer times. The sequence of phase transformations during heating, which proceed from the Al-based binary phases, which are separated with respect to Cu and Fe, to the chemically homogeneous QC phase, is determined by the thermodynamics of elements interaction.

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