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New stable icosahedral quasicrystal in the system Al–Cu–Co–Fe

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ABSTRACT

Based on the conceptual Al–Cu–(Fe + Co) phase diagram we found an optimal initial composition and developed a method to grow Al–Cu–Fe–Co single-grain quasicrystals. Our original two-stage cooling process includes (i) fast cooling of the melt down to the quasicrystalline single phase region with rate of ~165 K/h to prevent growing of nonquasicrystalline phases in the melt, and (ii) slow cooling down with rate of ~2–3 K/h to grow large (mm-size) single-grain quasicrystals. As a result a new stable quaternary Al-based icosahedral quasicrystal has been obtained. The chemical composition of the grown quasicrystal determined by both the energy dispersive X-ray analysis and inductively coupled plasma mass spectrometry was $Al_{64.36}Cu_{22.20}Co_{3.05}Fe_{10.39}$. Powder XRD and selected area electron diffraction were carried out for the phase identification and confirmed the icosahedral structure. The temperature dependencies of the electrical resistance measured on the oriented samples in the temperature range of 1.4 K–300 K is typical for icosahedral quasicrystals.

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1. Introduction

The quasicrystalline structure at first was identified in rapidly solidified binary Al-rich Al–Mn alloy [1]. Although thereafter quasicrystals (QCs) were observed in many other binary and ternary alloy systems [2,3], those consisting of aluminum with transition metals (TMs) were studied most extensively, in particular, because they showed the best potential for practical applications [4,5].

Metastable QCs are formed in a wide range of binary alloy systems of Al with the d-transition metals [6]. Stable QCs can be obtained when the third element is added to one of these binary systems. The third element could be one of the transition metals for example, copper [3]. Examples of these stable ternary quasicrystalline systems are icosahedral Al–Cu–Fe and decagonal Al–Cu–Co. Adding of the fourth element, not necessary a transition metal, to these ternary systems may result in a quasicrystalline structure [7,8]. The quasicrystalline alloys contained more than four elements were also obtained [9–11].

Either three-dimensional icosahedral (IQC) or two-dimensional decagonal (DQC) structures are formed in most of the Al-based quasicrystalline alloys [2]. Physical properties of IQC and DQC, in particular, electronic transport strongly differ. In spite of that, there exists a close relationship between the icosahedral and decagonal structures. Thus, for example, direct transformation from IQC to DQC by heat treatment was observed [12].

Investigation of the transition from IQC to DQC by adding the fourth element into a ternary system is an interesting scientific task which may give insights into interrelations between these structures. From this point of view, the quaternary Al–Cu–Co–Fe system is of a particular interest because it contains icosahedral *i*-AlCuFe on one side of its phase diagram and the decagonal *d*-AlCuCo on another side [13,14]. A few attempts to obtain quaternary Al–Cu–Co–Fe quasicrystals have been done [15,16]. For example, metastable Al–Cu–Co–Fe samples which contained both icosahedral and decagonal phases have been produced by melt-spinning [16]. These samples decomposed after heat treatment. Thus, it still remains an open question whether it is possible to obtain stable quaternary Al–Cu–Co–Fe quasicrystals.

The aim of this work was to obtain a stable *i*-AlCuCoFe icosahedral quasicrystal in the form of a single-grain material for further investigation of their physical properties.

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2. Experimental procedures

Two types of Al–Cu–Co–Fe single-grain quasicrystals with low-iron-content (less than 1 at.% Fe) and high-iron-content (more than 10 at.% Fe) have been grown by us from a melt by an original two-stage cooling process as described in Section 3 of this paper.

Chemical compositions of the grown single-grain quasicrystals were determined by both the energy-dispersive X-ray (EDX) analysis and inductively coupled plasma mass spectrometry (ICP-MS). Powder X-ray diffraction (XRD) using Cu K_α radiation and selected area electron diffraction (SAED) were carried out for phase identification.

Oriented samples in the shape of rectangular parallelepipeds were cut out from the obtained quasicrystals. The temperature dependences of the electrical resistance of these oriented samples were measured by the four-probe method in a temperature range of 1.4–300 K.

3. Single-grain quasicrystal growth

Although both decagonal Al–Cu–Co and icosahedral Al–Cu–Fe solidify incongruently, single crystals of these ternary compounds could be directly grown from the melt due to the fact that there are composition regions in Al–Cu–Co and Al–Cu–Fe phase diagrams where these quasicrystalline phases are primarily crystallized phases (D + L region and I + L region respectively) [17–25]. However it could be huge problem to grow quaternary *i*-AlCu(Co,Fe) single crystal directly from the melt because the compositions of D + L and I + L regions strongly differ (if consider that Co just replaced by Fe).

To grow icosahedral *i*-AlCu(Co,Fe) single crystals one has to develop an optimal growth regime and find an appropriate initial composition of elements in an initial mix.

To develop a regime of *i*-AlCu(Co,Fe) preparation we considered the vertical sections of the parent ternary Al–Cu–Co [19] and Al–Cu–Fe [26] phase diagrams. In both diagrams the quasicrystalline single-phase regions are located at 150–200 K below the corresponding temperatures of liquidus. The scheme for the growth of *i*-AlCu(Co,Fe) single-grain quasicrystals proposed here is to heat a specimen with the desirable composition above the temperature of liquidus, then rapidly cool it into the single-phase region, and then slowly cool it down by 200–300 K to grow large single-grain samples. In our quasicrystal growth experiments the high purity elements of Al (99,99%), Co (99,9%), Cu (99,99%) and Fe (99,99%), all powedered, were mixed in required amounts, compacted with press load of 8 t/cm^2, placed in an alumina crucible, sealed into a quartz ampule filled with Ar at partial pressure of approximately 0.5 atm, and then heat treated according to the above mentioned scheme.

To find an appropriate initial composition of the specimen mixes we constructed a hypothetical Al–Cu– (Co + Fe) pseudo-ternary phase diagram (see Fig. 1) using the data reported in Refs. [6,27]. In our diagram Co and Fe together were considered as a single alloying component. Gray areas in the diagram represent the stability regions for decagonal *d*-AlCuCo and icosahedral *i*-AlCuFe phases and are marked D and I, respectively. We suppose that the suitable initial composition for growing the icosahedral *i*-AlCu(Co,Fe) single-grain quasicrystals should be close to the stability region of *i*-AlCuFe or to lay between this region and *d*-AlCuCo regions of stability.

We have started our growth experiments from low-iron-doped (less than 1 at.% Fe) samples to find a regime of single grain quasicrystal growth according to the scheme proposed above and then to adapt it to high-iron-concentration samples. With such Fe content this phase could be consider as ternary. The initial composition for quasicrystals growth was $Al_{65}Cu_{18}(Co+Fe)_{17}$. This composition belongs to the region of the decagonal phase stability in the Al–Cu–(Fe + Co) phase diagram (see Fig. 1).

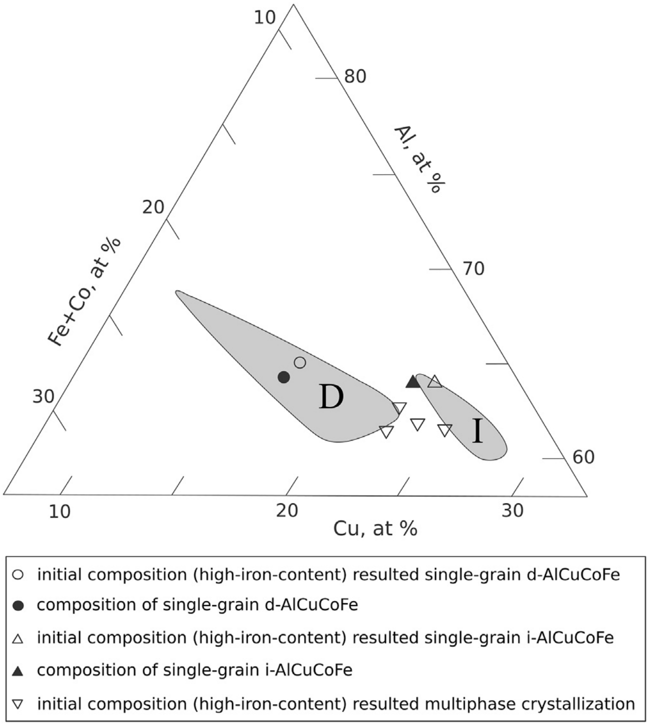

Fig. 1. Al-Cu-(Fe, Co) phase diagram. Stability regions of decagonal Al–Cu–Co and icosahedral Al–Cu–Fe phases [6,27] are shown as grey areas marked D and I, respectively.

The temperature profile for growth of the low-iron-content single-grain quasicrystal was as follows: heating and dwelling for 1 h at 1453 K, fast cooling for 2 h to 1123 K (i.e., at cooling rate ~165 K/h), and then slow cooling to 873 K with cooling rate of 2–3 K/h. As a result single-grain quasicrystals in the shape of elongated decahedral prisms with linear dimensions of up to 3 mm in length and of up to 1.5 mm in the transverse section were obtained (Fig. 2a). The maximum size of the samples obtained by this method is smaller than dimensions of the *d*-AlCuCo samples grown by the slow cooling crystallization from the D + L region [18,28] but big enough to measure resistivity in both the periodic direction and the quasiperiodic plane.

A similar two-stage cooling process was used to grow high-iron-concentration single-grain quasicrystals: heating above the temperature of liquidus, short dwelling, and fast cooling into the single phase region following by slow cooling. The initial compositions of the alloys which we used in our growth experiments are marked by empty triangles in Fig. 1. As noticed above these compositions are located in the Al–Cu–(Fe + Co) diagram either in the region of icosahedral phase stability or in between the icosahedral and decagonal phases. Each symbol in the diagram corresponds to a specific concentration of Al, Cu and (Fe + Co). Of course, compositions with different cobalt to iron ratio (i.e., with different *x* in $Al–Cu–Co_{(1-x)}Fe_x$ notation) are not distinguishable in the diagram. For each of the studied compositions we examined two sets of concentrations of cobalt and iron with $x = 0.5$ and $x = 0.75$. The XRD analysis revealed that at $x = 0.5$ produced alloys contain a small amount of the icosahedral phase, while at $x = 0.75$ the icosahedral material is the major phase. Single-grain samples containing the only icosahedral phase were obtained from the

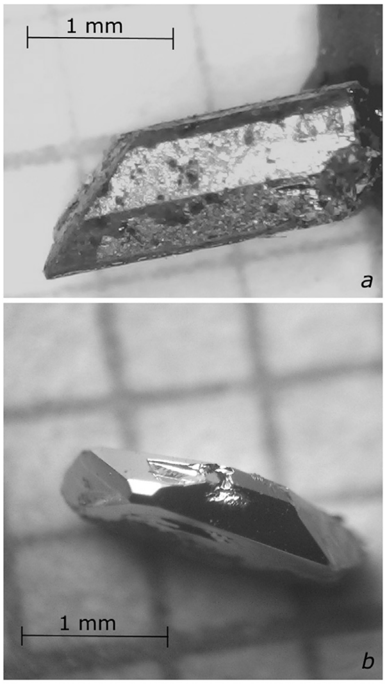

Fig. 2. As-grown single-grain quasicrystals: a) *d*-AlCuCoFe, b) *i*-AlCuCoFe.

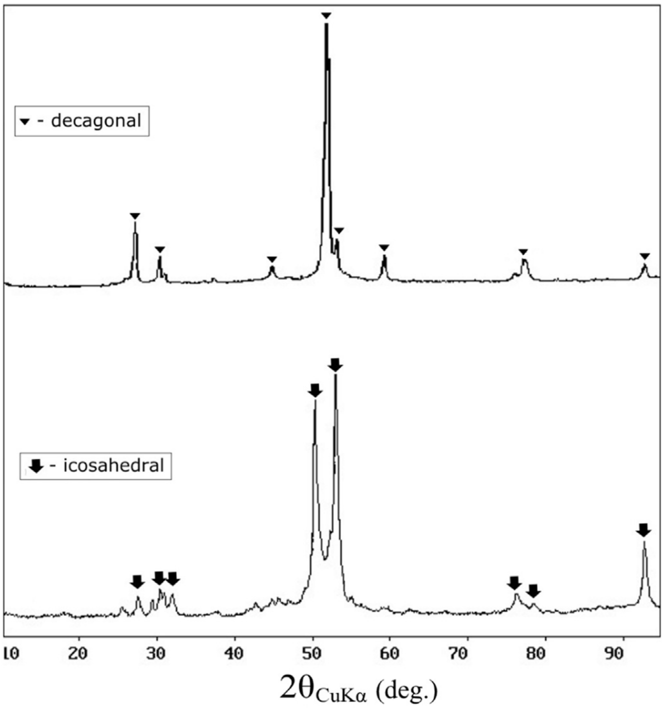

Fig. 3. Powder X-ray diffraction patterns obtained on crushed single-grain *d*-AlCuCoFe (top line) and *i*-AlCuCoFe (bottom line) samples. Triangles and arrows mark diffraction peaks corresponding to decagonal and icosahedral structures, respectively.

alloys with initial composition $Al_{64}Cu_{24}Co_{3}Fe_{9}$ represented in Fig. 1 by an empty pointing up triangle.

The optimal temperature profile of the high-iron-concentration crystals was found to be as follows: heating up to 1423 K, dwelling for 1 h, fast cooling for 1 h to 1073 K, and then slow cooling down to 673 K with cooling rate of 2–3 K/h. Thus, the maximum heating temperature and the final temperature of the fast cooling (i.e., the start temperature of slow cooling) are respectively 30 K and 50 K below than these parameters used for growing low-iron-content crystals.

4. Results and discussion

Grown samples with different iron content have strong morphological differences. If as-grown low-iron-content crystals have the shape of a regular decagonal prism (see Fig. 2a), high-iron-content crystals are distorted elongated icosahedra (see Fig. 2b). Powder X-ray diffraction patterns obtained from crushed single-grain samples are shown in Fig. 3. The X-ray diffraction peaks of low-iron-content and high-iron-content samples corresponding to the decagonal and icosahedral structures were identified and are marked in Fig. 3 by triangles and arrows, respectively. The structure of the crystals was examined using transmission electron microscopy (TEM) under a JEM-2100 microscope with a 200 kV acceleration voltage. SAED patterns of low-iron-content sample (Fig. 4a and b, c) are typical for decagonal symmetry and SAED patterns of high-iron-content samples (Fig. 4d and e, f) are typical for icosahedral symmetry [13,29]. Thus, the X-ray and electron diffraction results are in good agreement.

Chemical compositions of the grown decagonal and icosahedral single-grain quasicrystals were respectively $Al_{64.34}Cu_{17.34}Co_{18.25}Fe_{0.07}$ and $Al_{64.36}Cu_{22.20}Co_{3.05}Fe_{10.39}$, according to the EDX analysis. The resulted compositions are very close to the initial ones. It is known that when it is slowly grown from ternary melt, the quasicrystal composition can vary considerably from the center to its periphery [30]. For this reason, several of the resulting single-grain quasicrystals were also investigated by ICP-MS to determine the average composition of the samples. Within the measurement error both methods EDX and ICP-MS gave the same results. The initial (empty symbols) and final (solid symbols) compositions of the low-iron-content (circles) and high-iron-content (triangles) samples are indicated in Fig. 1. The composition of the grown low-iron-content sample, as expected, is in the region of existence of the decagonal phase of *d*-Al-Cu-Co. The composition of the grown high-iron-content sample is located at the boundary of the region of the icosahedral *i*-Al-Cu-Fe phase existence, on the side facing *d*-Al-Cu-Co.

The temperature dependences of the electrical resistivity ρ(T) of the low-iron-content samples are shown in Fig. 5a for the current flowing parallel to the long axis of the single-grain quasicrystal (according to the SAED pattern this is the ten-fold axis) and for the current flowing in the perpendicular direction. It should be noted that above 50 K the resistivity along the ten-fold axis grows linearly with increasing temperature (see Fig. 5a), while in the perpendicular direction (see Fig. 5 b) ρ(T) is nonlinear with positive derivative $d\rho/dT$ in the whole temperature range. In Fig. 5a inset we show the temperature dependence of the resistivity anisotropy which is defined ordinarily as ρ_q/ρ_p. The anisotropy is near 5.9 at room temperature and increases with temperature decreasing. When temperature reaches about 30 K the anisotropy value saturates near 7.3 and does not change in the temperature range from 30 K to 1.4 K.

Observed linear temperature dependence of the resistivity along the ten-fold axis together with nonlinear dependence of the resistivity in the perpendicular direction are typical for decagonal quasicrystals. Also the resistivity anisotropy value at low temperatures from $\rho_q/\rho_p = 5.5$ to $\rho_q/\rho_p = 12$ is typical for decagonal quasicrystals [28,31–33].

The temperature dependences of the electrical resistivity ρ(T) of high-iron-content sample (see Fig. 5b) differs significantly from

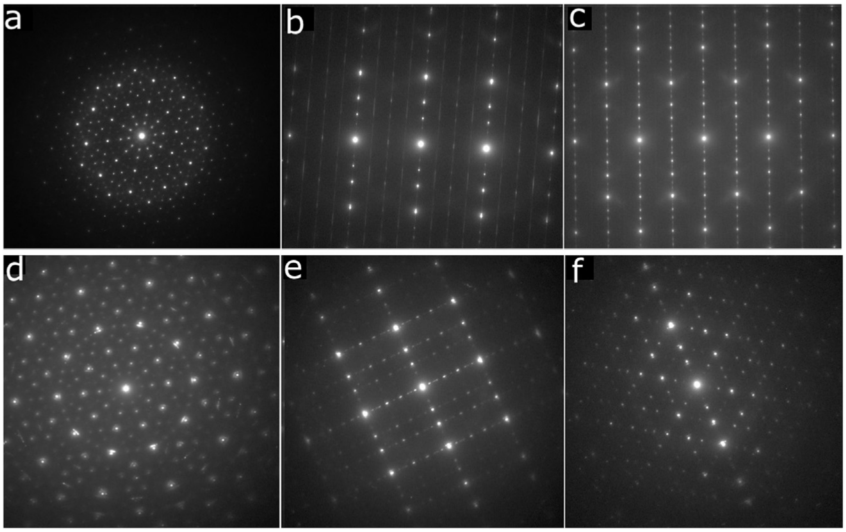

Fig. 4. Selected area electron diffraction (SAED) patterns for *d*-AlCuCoFe (a, b, c) and *i*-AlCuCoFe (d, e, f) quasicrystals.

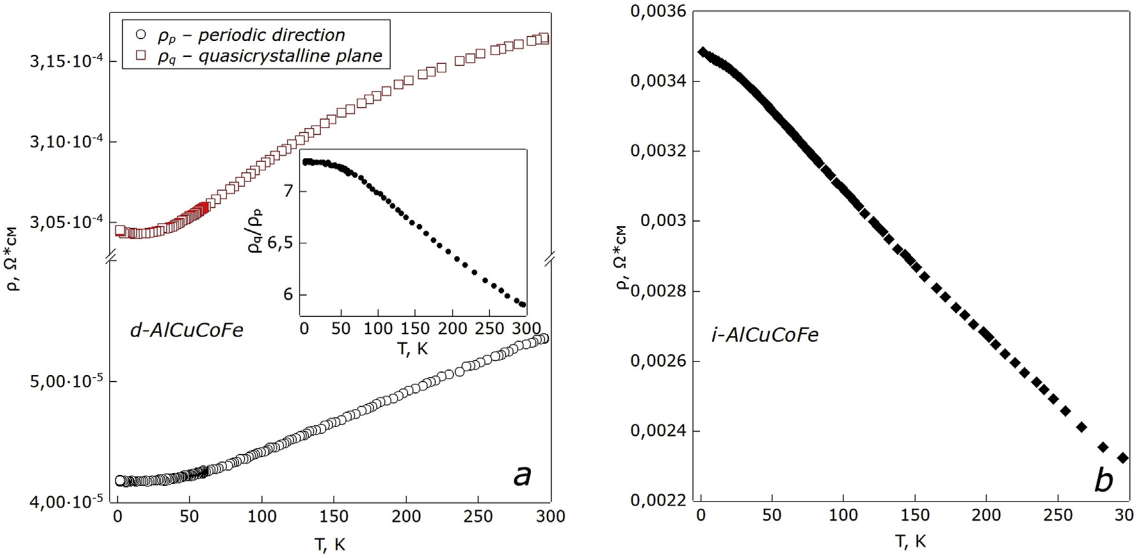

Fig. 5. a - Temperature dependences of electrical resistivity of *d*-AlCuCoFe along the periodic direction and in the quasicrystalline plane, inset: Temperature dependence of resistance anisotropy in *d*-AlCuCoFe; b - Temperature dependence of electrical resistivity in *i*-AlCuCoFe.

that of low-iron-content one. The resistivity increases with temperature decreasing, and this is typical for icosahedral quasicrystals. The resistivity ratio $\rho(4K)/\rho(295K)$ is equal to 1.6 which is close to the value observed by other authors in *i*-AlCuFe [34].

Phase and structural analyses, as well as the resistivity data, allow us to state that our low-iron-content sample is a decagonal quasicrystal while the high-iron-content sample has an icosahedral structure. Thus, at high level of cobalt substitution with iron in the ternary *d*-AlCuCo a quaternary *i*-AlCu(Co,Fe) with icosahedral structure is formed.

Let us compare our results with the data from literature review. Metastable quasicrystals $Al_{65}Cu_{20}Fe_{15\text{-}x}Co_x$ (x = 3; 5; 8) with decagonal and icosahedral structures were studied by Kim et al. [16]. To compare with our results, this equation should be rewritten in the following form: $Al_{65}Cu_{20}(Co_{1\text{-}x}Fe_x)_{15}$ (x = 0.8; 0.67; 0.47). Dendritically grown icosahedral phase with a small amount of B2 phase was found in Ref. [13] in the melt-spun $Al_{65}Cu_{20}Fe_{12}Co_3$ (which corresponds to $Al_{65}Cu_{20}(Co_{1\text{-}x}Fe_x)_{15}$ (x = 0.8)) samples. Coexistence of the icosahedral, decagonal, and B2 phases was observed in the melt-spun $Al_{65}Cu_{20}Fe_{10}Co_5$ (which corresponds to $Al_{65}Cu_{20}(Co_{1\text{-}x}Fe_x)_{15}$ (x = 0.67)) ribbon. These results are in good agreement with the data of the present work: single phase samples with icosahedral structure were obtained from $Al_{65}Cu_{20}(Co_{1\text{-}x}Fe_x)_{15}$ (x = 0.77) melt, while growth from $Al_{65}Cu_{20}(Co_{1\text{-}x}Fe_x)_{15}$ (x = 0.5) melt resulted in multiphase crystallization.

Let us consider now the quaternary Al–Cu–Co–Fe system with a high iron content as a child of the *i*-AlCuFe compound. One may suppose that when iron is replaced by cobalt in *i*-AlCuFe, cobalt occupies position of iron. At the same time, obviously, the icosahedral cluster is deformed. At x = 0.77 (i.e., when about one-fourth of the iron atoms are replaced by cobalt) both XRD and SAED show a structure similar to the pure undoped *i*-AlCuFe. Thus, at such cobalt concentration the cluster is still stable. Further increase of cobalt concentration leads to a significant deformation of the original icosahedron, which in turn leads to the formation of a complex structure containing both the icosahedral and decagonal clusters together with a crystalline structure. It is why our attempts to grow quasicrystals with equal amount of cobalt and iron failed.

Apparently, similar reasoning is also valid if we consider the stability of the decagonal structure of *d*-AlCuCo when cobalt is replaced by iron. Up to a certain concentration of iron, deformation of decagons does not lead to the destruction of the cluster structure, and accordingly, formation of stable quaternary *d*-AlCuCoFe compound with decagonal structure is possible. The development of this approach could be perspective for the studying of the transition from IQC to DQC by adding the fourth element into a ternary system.

5. Summary

Quaternary decagonal *d*-AlCuCoFe and icosahedral *i*-AlCuCoFe single-grain samples were obtained by an original two-step cooling process consisting of fast cooling of a melt with a specific composition into the single phase region followed by slow cooling. Starting compositions of Al, Cu, and (Fe + Co) for single-grain quasicrystals growth were determined from our original Al–Cu–(Fe + Co) phase diagram. Initial Co and Fe content in the icosahedral sample corresponded to $x = 0.75$ according to Al–Cu–$Co_{(1-x)}Fe_x$ formula ($x = 0.77$ in the resulted sample). In the case of $x = 0.5$ multiphase crystallization is observed. The obtained result is in good agreement with Ref. [16] and shows that a boundary of icosahedral structure stability is in between $x = 0.5$ and $x = 0.75$.

Acknowledgments

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