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# Characterization of the icosahedral phase in as-cast quasicrystalline $Al_{65}Cu_{20}Fe_{15}$ alloy

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**Abstract**

Morphology features and microstructures of a quasicrystalline $Al_{65}Cu_{20}Fe_{15}$ alloy were studied using X-ray diffraction, scanning electron microscopy (SEM), electron probe microanalysis, electron backscattered diffraction (EBSD), and transmission electron microscopy (TEM) techniques. A typical layer dendritic microstructure of the as-cast quasicrystalline alloy consisted of four phases: an $Al_{71}Cu_5Fe_{24}$ phase as a core of the dendritic structure, which was surrounded by a quasicrystalline $Al_{60}Cu_{26}Fe_{14}$ phase and a crystalline $Al_{50}Cu_{45}Fe_5$ phase being in the interdendritic regions, and a Cu-rich $Al_{44}Cu_{54}Fe_2$ phase. The quasicrystalline phase was characterized of three symmetries: five-, three-, and twofold. The Kikuchi diffraction patterns obtained from both SEM and TEM are very similar. It is shown that without the need to prepare thin film specimen, EBSD is an alternative method to characterize quasicrystals. © 2001 Published by Elsevier Science Inc.

*Keywords:* EBSD; Phase identification; SEM; Quasicrystals

## 1. Introduction

Quasicrystals are materials that exhibit noncrystallographic symmetries (5-, 8-, 10-, and 12-fold) but not a periodic structure. The unexpected structure of the quasicrystals was discovered, for the first time, in rapidly solidified Al–Mn alloy using transmission electron microscopy (TEM) by Shechtman et al. [1]. Since then, several binary quasicrystalline phases have been found in Al–TM (TM = Cr, Fe, Mo, Co) alloys and are characterized mainly by TEM.

Kikuchi lines can be produced by backscattered electrons. In scanning electron microscopy (SEM), these patterns are known as electron backscattered diffraction (EBSD) [2]. It is possible to map out rapidly the texture of polycrystalline materials using these patterns without thinning the sample. A similar technique is available for TEM Kikuchi maps, known as convergent beam electron diffraction (CBED). Although this technique cannot be automated, TEM can give the interface plane much more accurately so that these two techniques are complementary.

In the present paper, the morphological features and the microstructures of as-cast specimens of the

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quasicrystal $Al_{65}Cu_{20}Fe_{15}$ alloy were examined by both TEM and EBSD techniques.

## 2. Experimental procedures

The nominal composition used in this study was $Al_{65}Cu_{20}Fe_{15}$ (in atomic percentage). The alloys were prepared from Al (99.99%), Cu (99.99%), and Fe (99.9%). The bulk pure metals were arc melted in a copper mould in a high vacuum chamber. In order to reach a high homogeneity, the ingots were remelted at least three times. The total mass loss was less than 0.1%.

Phase identification and microstructural examinations of the as-cast alloy specimen were carried out using X-ray diffraction (XRD), SEM, electron probe microanalysis (EPMA), EBSD, and TEM techniques. The XRD examination was performed on a diffractometer with nickel-filtered Cu K$\alpha$ radiation, scanning at a speed of $1^\circ$ $min^{-1}$, the characteristic peaks of the XRD being collected within a diffraction angle ($2\theta$) ranging from 20° to 80°. Microstructural analysis of the sample was performed with a Cambridge Stereoscan 360 scanning electron microscope equipped for composition analysis with an energy dispersive X-ray spectroscopy (EDX) apparatus.

The specimen preparation for EBSD is straightforward, often similar to that for optical microscopy. Specimens were polished with diamond paste from 6 to 0.25 μm. Since AlCuFe quasicrystal is of extremely high hardness [3], it is not easily deformed by diamond polishing. Specimens are required to be ultrasonically cleaned prior to SEM examination to remove the diamond paste and oil. These kinds of contaminates will obscure the EBSD results. EBSD measurements were obtained using commercially available software (supplied by the Company HKL Technology APS) linked to a Stereoscan SEM operating at an accelerating voltage of 20 keV with the specimen tilted to 70°. Thin film samples for TEM examination were prepared by mechanical polishing and ion beam milling. The TEM investigation was carried out in a Jeol 2000 FX transmission electron microscope operating at 120 kV.

## 3. Results and discussion

### 3.1. Morphology feature of quasicrystalline $Al_{65}Cu_{20}Fe_{15}$ alloy

The SEM examination of the fractured surface of the as-cast alloy is shown in Fig. 1a and b. An array

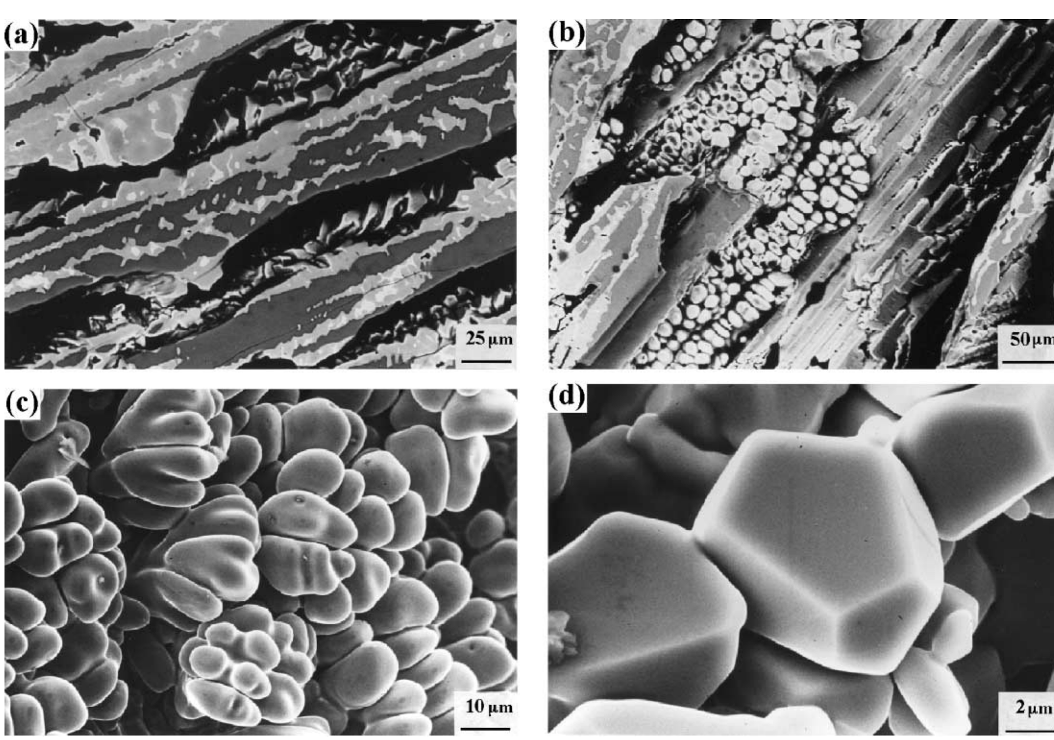


Fig. 1. Morphology features of as-cast $Al_{65}Cu_{20}Fe_{15}$ alloy.

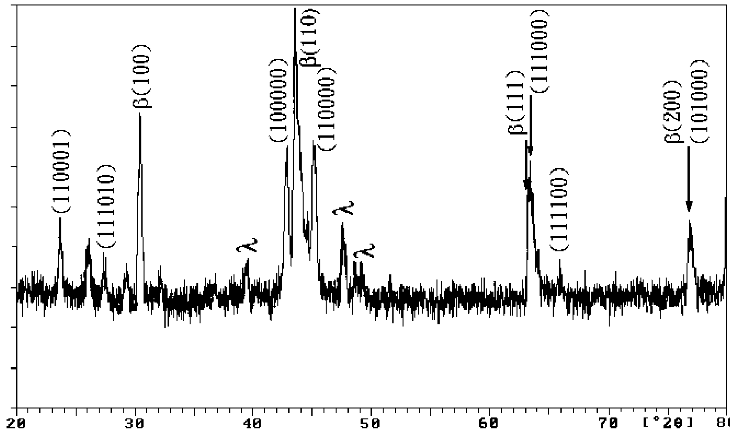


Fig. 2. X-ray diffraction pattern of as-cast $Al_{65}Cu_{20}Fe_{15}$ alloy.

of pentagonal dodecahedral crystals in the shape of a cauliflower is shown in Fig. 1c and d. The polished surface of the as-cast specimen exhibits a featureless flat surface resulting from cleavage fracture. The fracture surface looks rough, being of a quasicleavage nature rather than cleavage. The quasicrystal-forming alloys often exhibit novel microstructures. The nucleation-dominated formation of icosahedral quasicrystals sometimes leads to a banded-growth morphology in two phase alloys due to the formation of fine-grained quasicrystals ahead of the moving crystalline interface [4]. The quasicrystalline phase is also reflected in characteristic cauliflower-like morphologies with protuberances, as shown in Fig. 1c.

High magnification reveals the formation of a pentagonal dodecahedron in the quasicrystalline phase with an edge size of about 5 μm as shown in Fig. 1d. The icosahedral quasicrystal particles in the aluminum matrix of the AlMnCrSi alloy neither have the shapes of dodecahedra nor triacontahedra such as in the Al–Li–Cu system [5,6].

The morphological evolution of a quasicrystal during growth depends upon the stability of the growing interface and the atomistic mechanism of growth. While the former controls the growth shapes like cells and dendrites, the atomistic mechanism is often reflected in the nature of the interface, for example, faceted or nonfaceted. Often, the growth morphology is dictated by the growth temperature. If growth occurs below the roughening transition temperature, the cell and dendrites grow with a faceted shape excepting at very high growth rates when a dynamic roughening takes place. In the latter case, the dendrites and cells are again surrounded by a macroscopically smooth, continuously curved interface. As pointed out earlier, the quasicrystal is not expected to have a true roughening transition and hence the growth morphology of cells and dendrites should be faceted. The smooth quasicrystalline dendrites can be

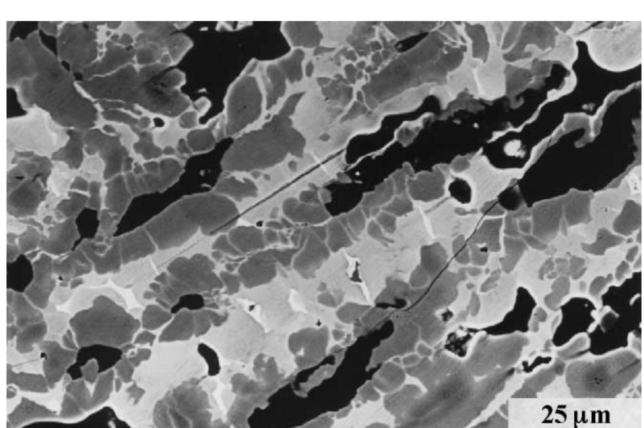


Fig. 3. Typical SEM micrograph of as-cast $Al_{65}Cu_{20}Fe_{15}$ alloy.

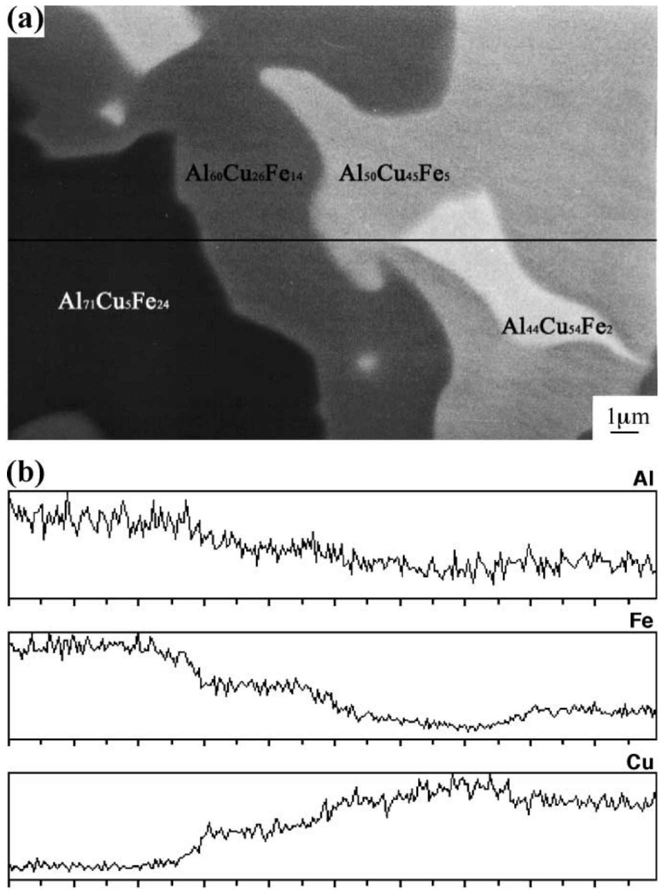


Fig. 4. SEM micrograph of the as-cast quasicrystalline $Al_{65}Cu_{20}Fe_{15}$ alloy (a) and SEM X-ray line scanning showing distribution of elements Al, Cu, and Fe (b) in the microstructure shown in (a).

observed only at very high undercooling, when the growth rate is very high. It was reported that dendrites also reflected the inherent icosahedral symmetry and the quasicrystal could also twin during the growth process [4].

### *3.2. Microstructure of as-cast quasicrystalline $Al_{65}Cu_{20}Fe_{15}$ alloy*

The X-ray diffractogram of the as-cast $Al_{65}Cu_{20}Fe_{15}$ quasicrystal alloy specimen is shown in Fig. 2. The i-phase and two crystalline phases were found in the as-cast sample. Most of the peaks can be indexed for the face-centered icosahedral (fci) phase [7]. The second phases include β-Al(Cu,Fe) and λ-$Al_{13}Fe_4$ phases as indicated in Fig. 2.

A backscattered SEM micrograph of the as-cast quasicrystal alloy is shown in Fig. 3. A typical dendritic layer structure is clearly observed. A black imaged phase solidified first as a core of the dendritic structure, then it was surrounded by a grey imaged phase and a light imaged phase; the nonfaceted grain size of the grey phase ranged from 3 to 20 μm. A white imaged phase solidified finally in the interdendritic regions.

From the SEM examination, the as-cast quasicrystalline alloy consisted of four phases, as shown in Figs. 3 and 4a.

Distribution profiles of three elements Al, Fe, and Cu in the four phases of the alloy were produced using SEM X-ray line scanning, as shown in Fig. 4b. A distinct contrast of the four phases is shown in the

Table 1
Analyzed compositions of the four phases in the quasicrystal alloy

| Phases | Analyzed compositions (in atomic percentage) |
|---|---|
| $Al_{65}Cu_{20}Fe_{15}$ | $Al_{60}Cu_{26}Fe_{14}$ |
| Al(Cu,Fe) | $Al_{50}Cu_{45}Fe_5$ |
| $Al_{13}Fe_4$ | $Al_{71}Cu_5Fe_{24}$ |
| Cu-rich | $Al_{44}Cu_{54}Fe_2$ |

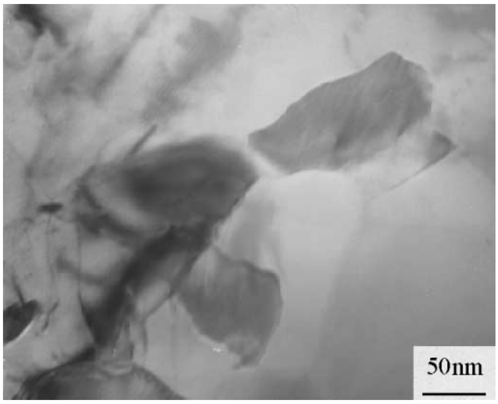


Fig. 5. TEM bright field micrograph of as-cast $Al_{65}Cu_{20}Fe_{15}$ alloy.

SEM photograph (Figs. 3 and 4a). According to the atomic contrast, the $Al_{71}Cu_5Fe_{24}$ phase was referred to as the Al-rich phase and the $Al_{44}Cu_{54}Fe_2$ phase was referred to as the Cu-rich phase.

According to EPMA, the average chemical composition of the grey region was $Al_{60}Cu_{26}Fe_{14}$ (in atomic percentage). The light grey phase was $Al_{50}Cu_{45}Fe_5$ phase, and the black phase was $Al_{71}Cu_5Fe_{24}$. The white phase in the interdendritic region was $Al_{44}Cu_{54}Fe_2$ phase (in atomic percentage). The analyzed compositions of the four phases in the quasicrystal alloy are shown in Table 1.

Only three phases: the fci-quasicrystalline $Al_{60}Cu_{26}Fe_{14}$ phase, the $Al_{50}Cu_{45}Fe_5$, and the $Al_{71}Cu_5Fe_{24}$ phases, were identified in the as-cast quasicrystalline alloy using XRD, as shown in Fig. 2. This was probably because of either the small volume fraction of the Cu-rich phase or the decomposition of this undetected phase.

The grey quasicrystalline grains of about 4 μm in diameter were detected in the as-cast alloy in the TEM bright field image, as shown in Fig. 5. This morphology of the quasicrystalline phase was different from that observed in the previous studies. Various types of morphology of the quasicrystalline phase, such as spherically shaped particles with

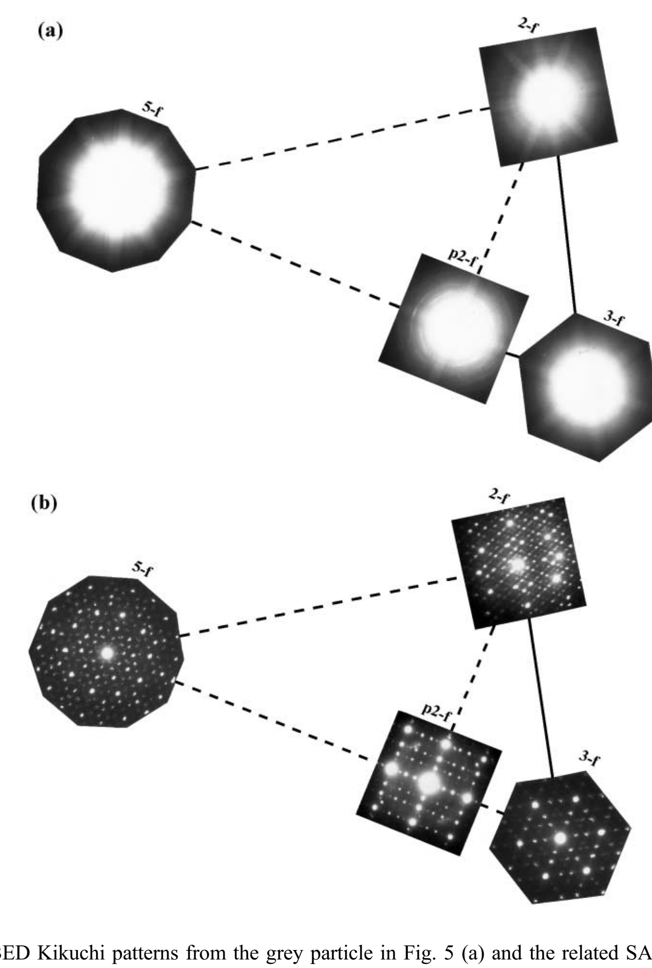


Fig. 6. CBED Kikuchi patterns from the grey particle in Fig. 5 (a) and the related SAD patterns (b).

deeply indented radial streaks or coral-like dendritic shape with mottled internal contrast, were reported depending upon the composition and the quenching [1,8,9].

Kikuchi diffraction patterns from the grey particles in the TEM bright field image were produced using CBED in TEM, as shown in Fig. 6a. The Kikuchi band is symmetrical and the camera length used in the CBED was 30 cm. The selected area diffraction (SAD) patterns were taken along the tracks of the Kikuchi bands, with the incident beams parallel to the five-, three-, and twofold axes, respectively. The camera length of the SAD was 120 cm. Typical SAD patterns with three symmetries of five-, three-, and twofold are shown in Fig. 6b. The three symmetries of the Kikuchi diffraction patterns were characteristic of the quasicrystalline phase.

EBSD appears as an effective method in identifying the icosahedral quasicrystals directly from a bulk specimen on SEM. However, there are no predetermined lattice parameters stored in the computer database and no associated automatic Kikuchi pattern indexing for quasicrystals because of their complex and nonperiodic structure. Therefore, the EBSD patterns for quasicrystal can usually be indexed by visual appraisal or by comparison with standard Kikuchi maps. From EBSD examination, the typical three symmetries of the Kikuchi diffraction patterns were produced from the grey imaged phase ($Al_{60}Cu_{26}Fe_{14}$) as shown in Fig. 7. Obviously, the grey imaged phase is the quasicrystalline phase in the alloy being tested.

Despite the differences between the methods of producing and detecting Kikuchi-type patterns in TEM (Fig. 6a) and SEM (Fig. 7), the patterns are geometrically similar. Each set of diffracting planes

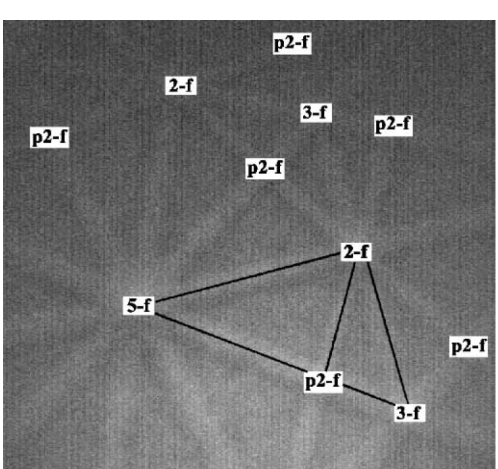


Fig. 7. EBSD Kikuchi patterns from the grey imaged phase ($Al_{60}Cu_{26}Fe_{14}$).

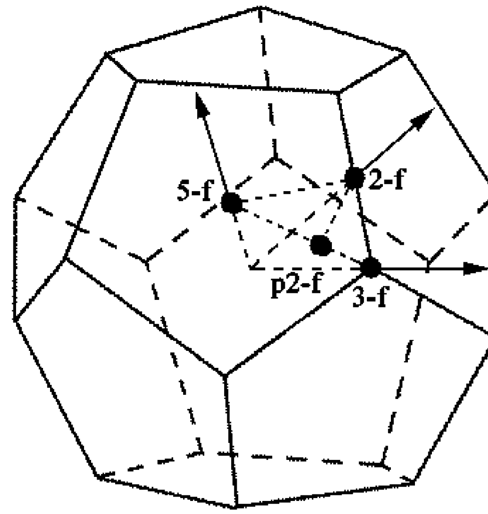


Fig. 8. Icosahedral unit cell in the cubic reference frame of as-cast $Al_{65}Cu_{20}Fe_{15}$ alloy.

thus produces a pair of K lines separated by twice the Bragg angle and the intersection of the crystal planes with the photographic plate lies halfway between its K-line pair.

Based on the Kikuchi diffraction patterns, a structural model of the fci-quasicrystalline phase was proposed as shown in Fig. 8. The solid lines and the dashed lines in Fig. 6 are in correspondence with that in Fig. 8.

## 4. Conclusions

The as-cast quasicrystalline $Al_{65}Cu_{20}Fe_{15}$ alloy is of a typical layer dendritic microstructure and consists of four phases: $Al_{60}Cu_{26}Fe_{14}$, $Al_{50}Cu_{45}Fe_5$, $Al_{71}Cu_5Fe_{24}$, and $Al_{44}Cu_{54}Fe_2$ phase (in atomic percentage). The $Al_{60}Cu_{26}Fe_{14}$ phase, which is of cauliflower-like morphology, is the quasicrystalline phase. This phase is identified using TEM and EBSD, which shows the characteristics of three symmetries: five-, three-, and twofold. EBSD is considered to be an effective method in identifying the icosahedral quasicrystals directly from a bulk specimen.

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