Materials Characterization 121 (2016) 68–75

Contents lists available at ScienceDirect

Materials Characterization

journal homepage: www.elsevier.com/locate/matchar

# Effect of cooling rate and Mg addition on the structural evaluation of rapidly solidified Al-20wt%Cu-12wt%Fe alloy

Ercan Karaköse [a,*], Hakan Çolak [b]

[a] Çankırı Karatekin University, Faculty of Sciences, Department of Physics, 18100 Çankırı, Turkey
[b] Çankırı Karatekin University, Faculty of Sciences, Department of Chemistry, 18100 Çankırı, Turkey

ARTICLE INFO

*Article history:*
Received 13 July 2016
Received in revised form 22 September 2016
Accepted 24 September 2016
Available online 28 September 2016

*Keywords:*
$Al_{20}Cu_{12}Fe$ quasicrystal
Mechanical properties
Microstructure
Rapid solidification

ABSTRACT

The present work examines the effect of Mg contents and cooling rate on the morphology and mechanical properties of $Al_{20}Cu_{12}Fe$ quasicrystalline alloy. The microstructure of the alloys was analyzed by scanning electron microscopy and the phase composition was identified by X-ray diffractometry. The melting characteristics were studied by differential thermal analysis under an Ar atmosphere. The mechanical features of the melt-spun and conventionally solidified alloys were tested by tensile-strength test and Vickers micro-hardness test. It was found that the final microstructure of the $Al_{20}Cu_{12}Fe$ samples mainly depends on the cooling rate and Mg contents, which suggests that different cooling rates and Mg contents produce different microstructures and properties. The average grain sizes of the melt spun samples were about 100–300 nm at 35 m/s. The nanosize, dispersed, different shaped quasicrystal particles possessed a remarkable effect to the mechanical characteristics of the rapidly solidified ribbons. The microhardness values of the melt spun samples were approximately 18% higher than those of the conventionally counterparts.



## 1. Introduction

To achieve the improvement of functional materials it is of significance that they have been manufactured with nominal advantage producing cost and special features such as density, high temperature hardness, stiffness and strength. Success in achieving these features can lead to a reduction in the weight of materials resulting in fuel saving, extended lifetime, etc. The requirement for these developments is a driving force for alloy development and processes in functional aluminum alloys [1]. The transformation from the solid to liquid state in the quick subtraction of thermo power energy in rapid solidification processing (RSP) produced an alloy with superior and unique features, which include a decrease in the particle sizes, increased solid solubility, decreased segregation levels, and, in some instances, the formation of quasicrystals, amorphous and partly-amorphous alloys by using cooling rates exceeding $10^4$ K/s for cooling materials melts [1–3]. Recently, rapid solidification (RS) methods, containing chilling techniques (melt drag and melt spinning, e.g.), atomization techniques (e.g., water, centrifugal atomization and gas), etc., have been commonly used [4–6]. Among these techniques, RS using melt spinning (MS) is most commonly used methods that can clearly development and change the morphology of phases [7]. The superior properties, improved morphological and rising in solubility limits, and chemical homogeneity are some of the refined morphologies provided via MS methods [8,9]. The first discovery of the icosahedral quasicrystalline structure by Shechtman et al. [10] created a new area of study in the atomic structure and crystallography of solids. These icosahedral samples were concerned with developing a category of alloys known as quasicrystals. The icosahedral quasicrystalline phase (IQC) has been described as a construction with disallowed symmetries (e.g. twelvefold, tenfold, eightfold and fivefold rotation axis) and a long-range orientational order [11]. It is clear that the emergence of QC alloys and their potential for utilization have attracted much research interest in recent years [12] and quasicrystal-creating materials have high potential for applications such as those requiring low-friction, wear resistant coatings, and high temperature thermal barriers [13–15], and as catalysts [16] or composite biomaterials [17]. The facility of using a QC structure in alloys for sporting goods and automotive application and researches of QC containing materials, interactions with a constitutional aluminum alloy have also been examined. The microstructure of QC alloys has a major effect on their physical and mechanical properties; for example, particle size is anticipated to be a very major factor for hydrogen storage and catalytic applications [18–19]. Therefore, the working and preparation of QC samples and their effect on the quasi-periodic structure are vital factors in their applications. Recently, the production of QC alloys has become feasible means of a number of distinct production processes making use of powder methods [20–21], rapid solidification methods [13,22–24] and thin film methods [25–26]. RS by melt spinning (MS) has also been found to be one of the methods that can be used to prepare QC alloys [23]. Up to the present day, a lot of alloy systems have been discovered to form IQC phases; but, only a small of number alloy systems established

* Corresponding author.
*E-mail address:* ekarakose@karatekin.edu.tr (E. Karaköse).

http://dx.doi.org/10.1016/j.matchar.2016.09.030
1044-5803/

stable QC phases, and most of them occurred in aluminum alloys. The occurrence of a stable or exact IQC phase was first found in the AlCuFe sample by Tsai et al. [24,27] at a composition in the range of 12 to 13 at.% Fe and 20 to 26 at.% Cu. Until now, former works on MS AlCuFe samples have illustrated that the QC phase structure is attainable by using the MS method [22,24]. However, according to Divakar et al. and Lee et al. [28,29], the formation of the QC phase structure together with multiphase structure is obtainable for Al–Cu–Fe samples by using the MS method. Therefore, researches on the interactions of QC containing materials with a constitutional aluminum alloy have also been examined. These works were devoted to the manufacturing of samples including QC phases by non-equilibrium techniques but the effect of Mg addition on the mechanical properties and morphology of Al-20Cu-12Fe alloy has rarely been examined [12,13,15]. The aim of this study is to contribute to the discussion about the effect of Mg addition and cooling rate on the mechanical and morphological properties of melt-spun Al–Cu–Fe alloys by comparing them with their conventionally solidified (CS) counterparts.

## 2. Experimental procedure

Conventionally cast alloys with nominal compositions of Al–wt% 20Cu–wt% 12Fe, Al–wt% 20Cu–wt% 12Fe–wt% 3Mg and Al–wt% 20Cu–12 wt% Fe–wt% 5 Mg were prepared in a vacuum induction furnace by using 99.9% Al (Alfa Aesar catalog no:10093), 99.99% Cu (Alfa Aesar catalog no:36686), 99.99%Fe (Alfa Aesar catalog no:40500), and 99.9%Mg (Alfa Aesar catalog no:43355). The CS samples with a mass of about 100 g were put into a graphite crucible. The rapidly solidified alloys were prepared using the single-roller melt-spinning method at 25 °C under an Ar atmosphere. The MS operations were applied using a brass wheel with 10, 20, 25, 30 and 35 m/s disc speed. The morphology and phase identification of all samples were analyzed by as ZEISS Ultra Plus Gemini field emission scanning electron microscopy (FESEM) with energy dispersive spectrometry (EDX) and Bruker AXS D8 X-ray diffractometer with device parameters of 160 mA, 40 kV and 10°/min using CuKα radiation. The conducted thermal properties were determined using a Perkin-Elmer DTA-7 differential thermal analyzer (DTA) at a heating rate of 10 °C/min in Ar atmosphere. EDX analysis results were shown in Table 1 and no elemental composition change was observed in the alloys. The Al–Cu–Fe samples were more detailed examined by a JEOL JEM 2100F transmission electron microscope (TEM) operated at 200 kV. The measurement tensile strength tests were made at a strain rate of $1 \times 10^{-3}$ $s^{-1}$with a Shimadzu Universal Testing Instrument (Type AG-10KNG). The samples lengths were chosen $5 \times 10^{-2}$ m for each ribbon. The tensile direction was chosen parallel to the longitudinal direction of a sample and each test represents three measurements at 25 °C. The hardness tests of the alloys were made a Durascan 70 model digital Vickers microhardness tester at 25 °C. This device performed of 0.098, 0.245, 0.49, 0.98, 1.47 and 1.96 N applied loads and a loading period of 10 s.

**Table 1**
Sample codes and chemical compositions with EDX analyses for investigated alloys.

| Sample code | Composition (wt%) | Cu (wt%) | Fe (wt%) | Mg (wt%) | Al (wt%) |
|---|---|---|---|---|---|
| CS0 | Al-20Cu-12Fe | 9.44 | 9.44 | 0 | 90.56 |
| CS3 | Al-20Cu-12Fe–3 Mg | 9.39 | 9.39 | 5.64 | 84.97 |
| CS5 | Al-20Cu-12Fe–5 Mg | 8.54 | 8.54 | 10.02 | 81.44 |
| MS0 | Al-20Cu-12Fe | 7.62 | 7.62 | 0 | 92.38 |
| MS3 | Al-20Cu-12Fe–3 Mg | 7.59 | 7.59 | 4.52 | 87.89 |
| MS5 | Al-20Cu-12Fe–5 Mg | 7.44 | 7.44 | 9.02 | 83.54 |

## 3. Results and discussion

### 3.1. Microstructure and thermal properties of Al-20Cu-12Fe–xMg ($x = 0$, 3 and 5 wt%) alloys

In order to observe the effect of the cooling rate and Mg addition on rapid solidification, CS and MS quasicrystal alloys were analyzed by X-ray diffraction. Fig. 1 illustrates the XRD analysis results taken from the CS Al-20Cu-12Fe–xMg ($x = 0$, 3 and 5 wt%) alloys and from the wheel side of the MS samples at wheel speeds of 15 and 35 m/s. According to the Al-20Cu-12Fe phase diagram [30], four diffraction peaks corresponding to the monoclinic λ-$A_{13}Fe_4$ phase (Bravais lattice: base-centered monoclinic, Space group: *C*2/*m* and a = 1.54 nm, b = 0.80 nm, c = 1.24 nm, β = 107.6°), tetragonal θ-$Al_2Cu$ phase (Bravais lattice: body-centered tetragonal, Space group: *I*4/*mcm* and a = 0.606 nm, c = 0.048), the cubic β-AlFe phase (Bravais lattice: body-centered cubic, Space group: *Pm*3*m* and a = 0.29 nm) and the mainly icosahedral i-$Al_{65}Cu_{20}Fe_{15}$ phase structures are observed in the CS Al-20Cu-12Fe sample, as seen in Fig. 1 (a). After the addition of 3 and 5 wt%Mg, besides the i-$Al_{65}Cu_{20}Fe_{15}$ , λ-$A_{13}Fe_4$, β-AlFe and θ-$Al_2Cu$ diffractions peaks, also diffraction peaks of $Mg_2Si$ intermetallics (crystal system; face-centered cubic, a = 0.6339 nm) are observed in the CS3 and CS5 alloys (Fig. 1 (b)–(c)). Also, such as in the 2θ ≈ 38 peaks have no matching IQC phase in the AlCuFe system, so they could be stems from some unknown intermetallic phases. Similar phase formations have also been reported by Sordelet et al. [31] in their CS Al25Cu12Fe samples. Fig. 2 shows the effects of cooling rate on RS and the phase constituents for MS3 and MS5 samples at 15 and 35 m/s wheel speeds. Two diffraction peaks overlapping the i-$Al_{65}Cu_{20}Fe_{15}$ and β-AlFe peaks are detected at 15 and 35 m/s for MS0 wheel speeds (as seen in Fig. 2 (a)). Also, the β-AlFe and icosahedral i-$Al_{65}Cu_{20}Fe_{15}$ phases are still detected after the content of 3 and 5 wt%Mg at the circumferential wheel velocity of 15 and 35 m/s. Except for β-AlFe and icosahedral i-$Al_{65}Cu_{20}Fe_{15}$, $Mg_2Si$ diffraction peaks are determined for the MS3 and MS5 samples at 15 m/s. Furthermore, at a speed of 35 m/s for the magnesium added MS3 and MS5 samples, β-AlFe and icosahedral i-$Al_{65}Cu_{20}Fe_{15}$ peaks are detected and intermetallic $Mg_2Si$ peaks are largely missing (as seen in Fig. 2 (b) and (c)). These results clearly

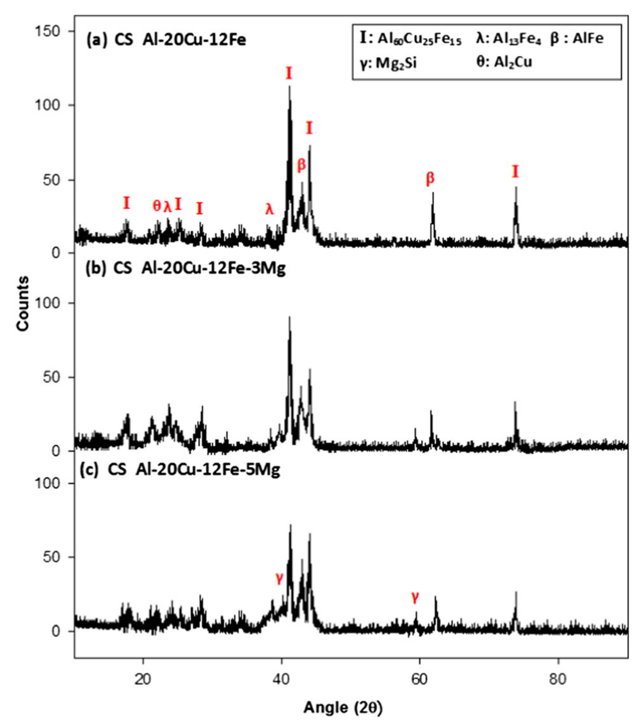


**Fig. 1.** The XRD patterns of conventionally solidified samples: (a) CS0, (b) CS3 and (c) CS5.