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## Al-based metal matrix composites reinforced with Al-Cu-Fe quasicrystalline particles: Strengthening by interfacial reaction



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#### ABSTRACT

The interfacial reaction between the Al matrix and the  $Al_{62.5}Cu_{25}Fe_{12.5}$  quasicrystalline (QC) reinforcing particles to form the  $Al_7Cu_2Fe$   $\omega$ -phase has been used to further enhance the strength of the Al/QC composites. The QC-to- $\omega$  phase transformation during heating was studied by in situ X-ray diffraction using a high-energy monochromatic synchrotron beam, which permits to follow the structural evolution and to correlate it with the mechanical properties of the composites. The mechanical behavior of these transformation-strengthened composites is remarkably improved as the QC-to- $\omega$  phase transformation progresses: the yield strength increases from 195 MPa for the starting material reinforced exclusively with QC particles to 400 MPa for the material where the QC-to- $\omega$  reaction is complete. The reduction of the matrix ligament size resulting from the increased volume fraction of the reinforcing phase during the transformation can account for most of the observed improvement in strength, whereas the additional strengthening can be ascribed to the possible presence of nanosized  $\omega$ -phase particles as well as to the improved interfacial bonding between matrix and particles caused by the compressive stresses arising in the matrix.

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#### 1. Introduction

Composites are complex engineering systems in which the constituent materials are generally not in thermodynamic equilibrium during initial fabrication, during production of the components or in use [1]. However, diffusion and phase transformations at interfaces can occur at any of these stages. These interfacial reactions between the matrix and the reinforcement, which depend on the system temperature, play a crucial role in determining the properties of metal matrix composites. By the use of thermodynamic and kinetic concepts, changes in the interface morphology in composite systems can be predicted and thus controlled [2]. Powder metallurgy processing employs lower and controllable temperatures compared to the liquid phase processing and, therefore, it offers improved regulation of interface reaction kinetics [2]. The main features, which govern interfacial reactions, compositions, phases

and structures are: (1) surface energy effects at interfaces, including nucleation, and (2) stress effects accompanying diffusion at interfaces [1,3]. Reactions between the matrix and reinforcement generally deteriorate the properties of the composites. For example, in  $Mg/Al_2O_3$  composites, the interfacial reaction between Mg and the oxide creates a brittle interface that decreases the strength of composites beyond acceptable limits [1] and, therefore, it should be avoided.

Interfacial reaction between the matrix and reinforcement is not always detrimental. For example, in the Al/Al–Cu–Fe quasicrystal (QC) composite system, the interfacial reaction between the Al matrix and the QC reinforcement has been used to enhance the room temperature strength of the composites through the formation of a new microstructure consisting of the Al matrix reinforced with Al<sub>7</sub>Cu<sub>2</sub>Fe  $\omega$ -phase particles [4]. The  $\omega$ -phase (space group *P4/mnc*) has tetragonal structure with cell parameters a = 0.6336 nm and c = 1.4870 nm, and 40 atoms in the unit cell; therefore, it belongs to the family of complex metallic alloys. The Al/ $\omega$  composites are not only stronger at room temperature but also exhibit

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higher yield strength with respect to the Al/QC composites at temperatures below 570 K [5].

According to the Al–Cu–Fe phase diagram [6,7], the tetragonal  $Al_7Cu_2Fe$   $\omega$ -phase exists between the icosahedral phase and aluminum at room temperature as well as at elevated temperatures [7–9]. Therefore, during processing (e.g. powder consolidation) at temperatures above 723 K, atomic diffusion takes place at the Al/Al–Cu–Fe QC interface leading to the transformation from the quasicrystalline to the  $\omega$ -phase having higher Al content [4,10].

In the present work, the reaction between the Al matrix and the Al $_{62.5}$ Cu $_{25}$ Fe $_{12.5}$  reinforcement, for the formation of the Al $_{7}$ Cu $_{2}$ Fe  $\omega$ -phase, and its effect on the mechanical behavior of the composites are analyzed. The QC-to- $\omega$  phase transition during heating was studied by in situ high-energy X-ray diffraction in order to correlate any structural modification induced by heating with the observed variations of the mechanical properties of the composites consolidated at different temperatures.

#### 2. Experimental

Metal matrix composites consisting of commercially pure Aluminum (purity >99.5 wt.%) blended with 40 vol.% of Al<sub>62.5</sub>Cu<sub>25</sub>Fe<sub>12.5</sub> quasicrystalline particles were synthesized by uni-axial hot pressing followed by hot extrusion under argon atmosphere (for further details on sample preparation see Refs. [11,12]). Hot pressing was done at 523 K and 637 MPa for 10 min, whereas, hot extrusion was carried out at 693, 743, 753, 773 and 848 K using a pressure of 530 MPa. The extrusion ratio was 4:1. The structure of the composites was analyzed by X-ray diffraction (XRD) in reflection configuration using a Philips PW 1050 diffractometer (Co Kα radiation,  $\lambda$  = 0.17889 nm). The structural evolution during heating of the composite extruded at 693 K was analyzed by XRD in transmission configuration using a high-energy monochromatic synchrotron beam ( $\lambda = 0.01304 \text{ nm}$ ) at the ID11 beam line of the European Synchrotron Radiation Facilities (ESRF). The sample was induction-heated to about 1070 K and X-ray patterns were recorded in situ every 2 s. Diffraction data were collected at a constant heating rate of 20 K/min. The microstructure was investigated by scanning electron microscopy (SEM) using a Gemini 1530 microscope equipped with an energy dispersive X-ray spectroscopy (EDX). The matrix ligament size  $(\lambda)$  was measured by superimposing random lines on the microstructures of the composites and its value was determined from the number of matrix region intercepts per unit length of the test line. N. and the total length fell into the matrix (L) as  $\lambda = L/N$  [13]. Ten random lines were superimposed on each micrograph and a minimum of three micrographs was used per composite.

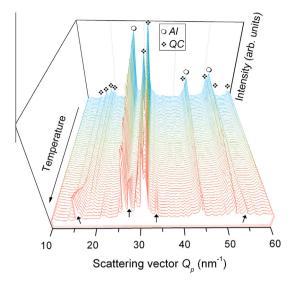
Length to diameter ratio of 2.0 was kept in cylindrical specimens (9 mm length and 4.5 mm diameter) prepared for compression test out of the extruded samples. Polishing was done to make both ends of the specimens parallel to each other prior to the test. INSTRON 8562 testing facility was used to test the specimens in compression under quasistatic loading (strain rate of  $8\times 10^{-4}\,\mathrm{s}^{-1}$ ) at room temperature. A Fiedler laser-extensometer was utilized to measure the strain directly on the specimen during compression.

#### 3. Results and discussion

#### 3.1. Structure evolution during heating

The phase evolution during heating of Al/QC composite extruded at 693 K was investigated by high-energy X-ray diffraction at temperatures between 300 and 925 K. The results are shown in Fig. 1, where the diffraction intensities are plotted against the scattering vector  $Q_p = 4\pi \sin\theta/\lambda$  in the range between 10 and  $60 \text{ nm}^{-1}$ . The pattern taken at room temperature (corresponding to the as-extruded sample) shows the presence of Al along with the QC phase. The Al/QC composite is stable within a very large temperature regime. Only at high temperatures the patterns clearly show the emergence of new peaks (see for example the peaks at about 16, 27.6, 33.5 and 53 nm<sup>-1</sup>, indicated by arrows in Fig. 1), which implies that a phase transformation occurs in this temperature range.

To highlight the structural changes occurring during heating, selected patterns taken at temperatures between 675 and 925 K are presented in Fig. 2. The structure does not show any new diffraction peak up to 740 K (Fig. 2(a)). Above this temperature, new peaks corresponding to the tetragonal  $Al_7Cu_2Fe$   $\omega$ -phase can



**Fig. 1.** XRD patterns ( $\lambda$  = 0.013 nm) as a function of temperature for the Al matrix composite reinforced with 40 vol.% Al<sub>62.5</sub>Cu<sub>25</sub>Fe<sub>12.5</sub> quasicrystalline particles extruded at 693 K, summarizing the phase evolution during heating.

be observed (see for example the peaks at about 27.6 and 30.5 nm<sup>-1</sup> in Fig. 2(b)). From this point onwards, the intensity of the ω peaks increases while that corresponding to the OC phase decreases with increasing temperature. At 925 K the OC peaks can no longer be detected and fcc-Al and the  $\omega$ -phase are the only phases visible. These results are in good agreement with the work of Kenzari et al. [10] for a pure Al matrix reinforced with Al-Cu-Fe-B quasicrystals, where the QC-to-ω transformation was observed at temperatures exceeding 723 K. Similar findings have also been reported for composites reinforced with Al-Cu-Fe quasicrystals produced by powder consolidation at temperatures ranging between 823 and 873 K [14-16]. All these results conform to the phase diagram of the Al-Cu-Fe system, which shows that pure Al and the QC phase do not exist in equilibrium and further indicate that the reaction between pure Al and the reinforcing QC particles leads to the formation of the  $Al_7Cu_2Fe \omega$ -phase [17–19].

The amount of the different phases during heating the Al/QC composite was evaluated from the XRD measurements using relative peak areas; and the results are shown in Fig. 3 for the temperature range between 730 and 950 K. The reaction Al + QC  $\rightarrow$   $\omega$  starts at 755 K at a slow rate and then it considerably accelerates above 800 K. The reaction is completed at about 925 K, where no QC phase can be detected. Due to the higher molar volume of the  $\omega$ -phase [20], the relative amount of matrix/reinforcement changes, finally leading to a composite with a  $\omega$  reinforcement content of about 53 vol.%, therefore, increased with respect to the original QC-reinforced composite (40 vol.%).

In order to study the effect of the Al + QC  $\rightarrow \omega$  reaction on the mechanical behavior of the material, composites reinforced with 40 vol.% Al<sub>62.5</sub>Cu<sub>25</sub>Fe<sub>12.5</sub> QC particles were extruded at different temperatures and then tested at room temperature in compression. Fig. 4 shows the diffraction pattern of the extruded samples. The sample extruded at 693 K shows only two phases: namely QC and Al. On the other hand, the pattern of the sample extruded at 733 K reveals the formation of the  $\omega$ -phase along with Al and QC phases. The amount of the  $\omega$ -phase increases with increasing extrusion temperature and the composite extruded at 773 K shows the presence of pure Al and  $\omega$ -phase only, in agreement with the XRD results in Fig. 2. This indicates that, even during extrusion, the Al + QC  $\rightarrow \omega$  reaction proceeds and the formation of the low density  $\omega$ -phase is not suppressed by the applied load. This is in accordance with Tsai et al. [16], who reported the formation of

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