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Mechanical properties of Al/ω-Al-Cu-Fe composites synthesized by the SPS technique

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ABSTRACT

Al/40 vol%ω-Al-Cu-Fe composites were produced from Al powder and i-Al-Cu-Fe quasi-crystalline particles using spark plasma sintering (SPS) technique. The mechanical properties of the composite were evaluated over the temperature range 293 K–823 K by performing compression tests at constant strain rate. The temperature dependence of the $\sigma_{0,2\%}$ yield stress gives evidence of two temperature regimes with a transition in the range 473 K–523 K. The decrease of $\sigma_{0,2\%}$ with increasing temperature, more pronounced in the low temperature regime, indicates that the two temperature regimes correspond to two different thermally activated deformation mechanisms. Based on microstructural analyses of the Al matrix, where plastic deformation takes place, the different strengthening contributions are discussed and the results are finally compared to those obtained for composites produced by hot isostatic pressing (HIP), for which the $\sigma_{0,2\%}$ temperature dependence is similar. In the low temperature regime, the $\sigma_{0.2\%}$ stress of the SPS composites is higher than that of the HIP composites. In this temperature regime, the stress difference is mainly ascribed to the different reinforcement phases present in the Al matrix. In the high temperature regime, the temperature dependence of $\sigma_{0.2\%}$ is comparable for the two composites whatever the processing route: load transfer is thus the main strengthening mechanism, which is similar for the two Al/ω-Al-Cu-Fe composites, the temperature dependence being ascribed to cross slip and climb processes.

1. Introduction

Al-based particle-reinforced metal matrix composites (MMC) have a high potential for technological applications, in particular in transport industry, because they meet the criterion of weight reduction and exhibit mechanical properties that cannot be achieved by any Al-based alloy [1–5]. Two categories of particle-reinforced composites must be distinguished. A first category, *in-situ* composites, where reinforcement is formed in the matrix by chemical reactions during the composite fabrication process; the main advantage being a homogeneous distribution of fine particle in the matrix [6–8]. A second category, where particle reinforcement is obtained by embedding hard particles in a metallic matrix. In this case, powder metallurgy processes are mainly employed, which allow an accurate control of particle content and size and the use of a large variety of reinforcement particles.

In the frame of this second category, quasicrystalline (QC) particles present mechanical properties (high hardness together with high elastic modulus and yield stress) and tribological properties that make them promising candidates as reinforcement particles in MMC. Tsaï et al. [9] were the first to fabricate by powder metallurgy route, an Al-based composite reinforced by icosahedral (i) Al-Cu-Fe quasicrystalline particles. Since then, numerous studies have been dedicated to Al/QC composites using different QC alloy systems. Most of them have been devoted to Al-Cu-Fe reinforcement particles [10–24] and to a less extend to Al-Pd-Mn [22,25], Al-Mn-Ce [25], Al-Cr-Mn-Co-Zr [26] and Al-Fe-Cr [27] reinforcement particles. The main reasons for using Al-Cu-Fe QC particles are that the mechanical properties of this QC phase have been well characterized [28–30], they are easy to produce by conventional casting and milling, the alloying elements ensure a low cost production for industrial applications.

Tsaï et al. [9] have shown that, for an Al matrix, a phase transformation takes place during powder metallurgy processing: when the temperature (T_p) is 873 K, the initial QC $Al_{63.5}Cu_{24.0}Fe_{12.5}$ phase transforms into the crystalline ω-$Al_7Cu_2Fe_{10}$ phase [Bown and Brown

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1956]. For similar composite produced at $T_p = 673$ K, the phase transformation does not occur and the QC phase is preserved. Concerning the mechanical properties, Kaloshkin et al. [18] reported that the yield stress of Al/Al-Cu-Fe composites measured at room temperature was processing temperature dependent. Laplanche et al. [17] compared the mechanical properties of Al/QC-Al-Cu-Fe ($T_p = 673$ K) and Al/ω-Al-Cu-Fe ($T_p = 823$ K) [15] composites, both produced by hot isostatic pressing (HIP) from Al and QC-Al-Cu-Fe powders. By performing compression tests over the temperature range 293 K–823 K, they showed that, below 573 K, the Al/ω-Al-Cu-Fe composites present higher yield stresses than that of the Al/QC-Al-Cu-Fe composites, while they are similar for higher deformation temperatures. Laplanche et al. [31] have also shown that ω-Al-Cu-Fe single-phase and QC-Al-Cu-Fe alloys exhibit similar normalised yield stress in a large temperature range (*i.e.* taking into account of the differences in their elastic coefficients and peritectic temperatures). Therefore, the differences observed between the two composites cannot be ascribed to differences in the mechanical properties of the ω-Al-Cu-Fe and the QC-Al-Cu-Fe phases. In addition, load transfer should be also very similar for both composites, since comparable particles and identical particle volume fraction and size are involved. Thus, the different observed mechanical properties in the temperature range 293 K–573 K may be due to indirect strengthening, that is to different Al-matrix microstructures: the Al-matrix obtained after the phase transformation from QC-$Al_{63.5}Cu_{24.0}Fe_{12.5}$ to ω-$Al_{70}Cu_{20}Fe_{10}$ leading to a more efficient strengthening of the composites.

Most of the studies have used HIP processes. In the present study, the Al/ω-Al-Cu-Fe composites were produced by the SPS (Spark Plasma Sintering) technique, which allows short-time production of large pieces, more suited for industrial applications. Mechanical properties are evaluated by compression test performed at different temperatures. Al-matrix evolutions are analyzed by post-mortem transmission electron microscopy (TEM) observations, that were complemented by *in-situ* heating TEM experiments. The different strengthening contributions are discussed and the results are finally compared to those obtained for composites produced by HIP.

2. Material and Methods

2.1. Synthesis

The production of i-Al-Cu-Fe quasicrystalline material has already been detailed elsewhere [32]. EDS analyses coupled with SEM observations indicate that the i-Al-Cu-Fe phase is predominant and give also evidence of minor phases corresponding to λ-$Al_{13}Fe_4$ and β-$Al_{50}Cu_{50-x}Fe_x$ phases, in good agreement with XRD results [15,33]. The i-$Al_{59\pm2}Cu_{27\pm2}Fe_{13\pm1}$ and commercial Al powders (Alfa Aesar, 99.8 at.% purity, 45–420 μm particle sizes) were used to synthesize the Al/ω-Al-Cu-Fe composites. For this, the i-Al-Cu-Fe powder was obtained by milling a i-Al-Cu-Fe ingot using a planetary ball mill with stainless steel balls and dies. The ball to powder mass ratio is 11 and the rotation speed is 100 rpm. No surfactant was used. Then, i-$Al_{59\pm2}Cu_{27\pm2}Fe_{13\pm1}$ and Al powders were sifted to obtain a particles size smaller than 25 μm and 60 μm, respectively.

To produce Al/40 vol% ω-Al-Cu-Fe composite, Al and 30% vol% of i-Al-Cu-Fe powders [15] were mixed in a Turbula for 30 min. The mixing step allows for the homogenization of the powder distribution. The powder mixtures were then inserted in a graphite die and placed in the Spark Plasma Sintering (SPS) device. They were reactively sintered at $T = 823$ K and a uniaxial pressure P = 100 MPa for 2 min. Commercial Al samples were also densified by SPS using similar parameters. The SPS process has been fully described in [34].

2.2. Microstructural Characterization

The crystallographic structure of the composite material was investigated by X-Ray diffraction (XRD) using a Bruker D8 diffractometer with Cu-$K_{\alpha1}$ radiation. Phase identification was complemented by scanning electron microscopy (SEM) observations using a field-emission gun scanning electron microscope (FESEM, Jeol 7001F-TTLS). The composition was quantified using an energy-dispersive X-ray spectrometer (EDS, Oxford Intruments) with the AZtec software based on PhiRoz correction method. SEM observations were performed using a back-scattered electron (BSE) detector to distinguish the different phase contrasts. The acceleration voltage of the electron beam was set to 15 kV.

The density of the composites has been calculated by accurately measuring the volume and the weight of the cylinder-shape sample.

The local microstructures of the deformed samples were characterized by transmission electron microscopy (TEM) using a Philips CM-20 operating at 200 kV. Slices, with a thickness of about 300 μm, were cut from the bulk sample. They were then pre-thinned up to a thickness of approximately 20 μm by mechanical polishing and glued onto a molybdenum grid. Finally, ion thinning down to electron transparency was performed by means of a Precision Ion Polishing System (Gatan-PIPS). Viguier and Mortensen [35] have shown the effect of the Ar-beam current on precipitate formation due to an excessive sample heating during the preparation of aluminum alloy thin foil. In fact, during ion polishing with an accelerating voltage of 5 kV, an increase of the sample temperature up to 513 K was observed. To reduce this artefact, an operating voltage of 3 kV was first applied followed by a final thinning step at 2.5 kV.

In situ heating treatment was performed on an Al/ω-Al-Cu-Fe composite thin foil using a JEOL-2200FS TEM equipped with a field emission gun (Schottky-FEG) operated at 200 kV and fitted with an in-column Ω-filter. Series of images of the Al/ω-Al-Cu-Fe composite thin foil, using a Gatan Single-Tilt heating holder model 628Ta, were recorded with a Gatan Ultrascan *2 k × 2 k CCD* camera. The absolute temperature of the sample was measured by a thermocouple with an estimated accuracy of ± 10 K. The final temperature, 573 K, was selected by consideration of the mechanical test results. During the heating treatment, the number and size evolutions of precipitates in the Al matrix were monitored. The same area was selected and analyzed at 573 K after 2 min and 1 h20 dwelling times.

2.3. Mechanical Tests

Compression tests were performed using a compression device (Instron 1195), similar to the one described in [36]. Compression samples, whose dimensions are 2.5 × 2.5 × 6 mm^3, were cut with a wire saw. Samples were deformed at a constant strain rate ($\dot{\varepsilon} = 1.4 \times 10^{-4}\,s^{-1}$) at different temperatures ranging between room temperature and 823 K. In order to achieve the thermomechanical stability of the compression set up, when the desired deformation temperature is reached, the sample is then maintained at this temperature for 2 h at least, before starting the compression test. The $\sigma_{0.2\%}$ stress defined at 0.2% plastic strain on the stress-strain curves is used as the characteristic stress for plastic flow. Note that the change in sample cross-section during plastic deformation is taken into account assuming a constant sample volume, so that true stresses and true strains are considered.

3. Results

3.1. Microstructure of As-produced Composites

Representative examples of XRD pattern and SEM micrograph obtained for Al/Al-Cu-Fe composites are shown in Fig. 1a and b, respectively. All diffraction peaks are indexed according to the Al face-centered cubic structure and the ω-Al-Cu-Fe tetragonal structure (Fig. 1a). Based on SEM observations, the Al-Cu-Fe reinforcement particles (grey contrast) are homogeneously distributed into the Al matrix (dark