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Optimization of electrical conductivity and strength combination by structure design at the nanoscale in Al–Mg–Si alloys

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ABSTRACT

The contribution of ultrafine grains and nanoscaled precipitates has been investigated in the Al–Mg–Si system to optimize the combination of strength and electrical conductivity. A full range of nanoscaled structures was achieved by varying severe plastic deformation and post-processing precipitation treatments. Nanoscaled features, like grain size, solute content of the matrix, precipitate size, density or distribution were quantitatively estimated by analytical transmission electron microscopy and atom probe tomography. Deformation induced precipitation and grain boundary segregations are reported here and the physical origins are discussed. The concomitant grain growth and precipitation mechanisms that occur during post deformation aging treatment have also been investigated. Then, the quantitative data obtained from the nanoscale characterization of ultrafine grain structures allowed adjusting physical models to account both for the mechanical strength and the electrical conductivity. Based on this approach, the range of properties achievable in Al–Mg–Si alloys was estimated.

1. Introduction

The industrial demand for low weight, high strength and high electrical conductivity materials is continuously growing. Due to the low density and the relatively high intrinsic conductivity of aluminum, Al based composites or alloys are certainly among the best candidates. The key issue is to find a proper strategy to keep these properties while increasing the strength. Alloying with a low fraction of Mg and Si is a common approach to manufacture wires for different applications in electrical engineering [1,2]. Standard conductors are typically made of 6101 and 6201 aluminum alloys via the following route: (i) solution treatment; (ii) cold drawing; (iii) artificial aging. Such a process typically leads to an ultimate tensile strength in a range of 250–330 MPa combined with an electrical conductivity in a range of 57–52% IACS (International Annealed Copper Standard) respectively [3–6]. The high strength of these conductors is the result of the combination of strain and precipitation hardening. The strain hardening is provided by the relatively large level of deformation typically achieved by drawing. During the artificial aging, some recovery occurs leading to a decrease in the dislocation density but the precipitation of nanoscaled Mg and Si rich precipitates gives rise to a significant increase in strength. The complex precipitation sequence in artificially aged AlMgSi alloys and the link with the hardening have been investigated down to the atomic scale since a long time [7–10]. In a first step, it involves the formation of Mg and Si clusters, then the precipitation of the β″ phase followed by the β′ and it ends up finally with the β phase (Mg_2Si). At the peak aging, the maximum hardness is achieved mainly thanks to a high density of needle or rod shape β″ and β′ precipitates. They are aligned along the three equivalent ⟨001⟩ directions of the fcc Al matrix, with a diameter of few nanometers and a length ranging from 20 to 100 nm. It is important to note that the final combination of properties achieved in such alloys strongly depends on a number of processing parameters, like the solution treatment temperature, the natural aging period prior and/or following the cold work process, the level of strain applied to the material, the time and the temperature of the final artificial treatment [6]. In a more general way, it has been demonstrated that even low levels of plastic deformation (as low as few%) may significantly accelerate the precipitation kinetics and also modify the precipitation sequence in some Al–Mg–Si alloys [11,12].

To further increase the strength of Al–Mg–Si alloys while keeping a similar (or even higher) electrical conductivity, a number of present authors have proposed recently to combine UltraFine Grained (UFG) structures obtained by severe plastic deformation (SPD) processes and precipitation [13–15]. It should be noted that

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SPD processing of Al–Mg–Si alloys has been the subject of active research in recent years. As is known, the grain refinement achieved by SPD techniques is typically in a range of 1 μm to 100 nm in Al alloys [16], which gives rise to a strong strengthening as predicted by the Hall and Petch law [17–19]. Also, a fine distribution of precipitates in a bulk nanostructured material may increase both the yield stress and the ductility [20,21]. Both Equal Channel Angular Pressing [13,22–27] and HPT [28] processes have been applied to various Al–Mg–Si alloys with post-SPD aging treatments. However, controlling the precipitation in such UFG structures is challenging because of the concomitant recovery, grain growth or even recrystallisation. Besides, heterogeneous precipitation along dislocations and/or grain boundaries is very likely to occur [26,27]. And like in slightly deformed alloys, much faster precipitation kinetics and a modified precipitation sequence have been reported [29–31]. In this context, the application of SPD processes to produce UFG structures with a high density of nanoscaled intragranular precipitates is very challenging. The main issues are: (i) keeping the small grain size during the artificial aging treatment necessary for the nucleation and growth of precipitates; (ii) avoiding heterogeneous precipitation along GBs as it will not provide significant strengthening, (iii) controlling the concentration of alloying elements in solid solution. Another route that has been explored in Al–Mg–Si alloys is to carry out the SPD process at a temperature were the atomic mobility is high enough to promote the precipitation at the same time as grain refinement [14,15,26,32]. In this situation, the precipitate number density, distribution and volume fraction are also relatively difficult to control because of the strong interactions between defects and solute atoms. It has been shown that even for moderate levels of plastic deformation; a finer precipitate distribution can be obtained. However the precipitation kinetics, the final microstructures and the related strength strongly depend on the deformation route [33]. Following these studies, it has been recently demonstrated that such an approach might be effective to obtain the enhanced strength and electrical conductivity of a 6201 alloy using SPD in a temperature range of 100–200 °C where grain refinement and dynamic precipitation occur simultaneously [14,15].

In the present work, this approach has been developed in a systematic way for two aluminum alloys commonly used for electric applications, namely the 6101 and 6201 alloys. Both defects created during SPD (GBs and dislocations) and nanoscaled precipitates increase the strength but also typically decrease the electrical conductivity. But another advantage envisioned through the SPD process, is a possible reduction of the solute content in the matrix as compared to classically treated alloys due to non-equilibrium GB segregations [34,35]. This is of particular importance for the electrical conductivity because, as stated by the Mattisen–Flemming rule, the electrical resistivity of diluted aluminum alloys is directly linked to the solute concentration [36,37]. Thus, the aim of this work was first to investigate and understand the influence of the thermo-mechanical treatments on grain refinement, precipitation and non-equilibrium segregations in both the 6101 and the 6201 alloys processed by SPD. The relationship with the properties provides a direct access to the contribution of these nanostructural features to the strength and the conductivity. Finally, this work provides a background for the development of aluminum-based UFG alloys with enhanced strength and electrical conductivity for advanced conductors in the electrotechnical field.

2. Material and experimental procedures

Two aluminum alloys corresponding to the commercial standard 6101 and 6201 were cast and extruded for the present work with the following composition (wt.%): 0.59Mg, 0.54Si, 0.07Fe, Al balance for the 6101 and 0.81Mg, 0.79Si, 0.09Fe, Al balance for the 6201. Disc-shaped samples (diameter 20 mm, thickness 1.4 mm) were cut out from the extruded rods for SPD processing using High Pressure Torsion (HPT). Materials were always solutionized one hour at 550 °C followed by water quenching immediately before HPT processing. The HPT process was carried out with a pressure of 6 GPa and an anvil revolution speed of one turn per minute. Samples were deformed up to 20 revolutions at various temperatures ranging from room temperature (RT) to 170 °C. The temperature was controlled by heating the anvils prior to deformation, and samples were pressed without shear deformation during 10 min to stabilize their temperature.

The HPT process gives rise to a shear strain gradient along the radius of disc shaped samples, and the shear strain can be estimated as $\gamma = 2\pi rN/t$, where N is the number of revolution, t the sample thickness and r the radius. Therefore, physical properties and microstructures were characterized systematically at a distance of 5 mm from the disc center, corresponding to a shear strain of about 30, 300 and 600 for 1, 10 and 20 revolutions respectively.

The mechanical strength was characterized by hardness measurements. The Vickers hardness (HV) was measured on a Micromet-5101 device with a diamond pyramid indenter, a load of 0.5 kg and a dwell time of 15 s. Data provided in the following sections are the average of at least 10 measurements. The electrical conductivity (ω) was measured at room temperature using an eddy-current electric conductivity meter (VE – 27 NC/4-5). The device was calibrated before each measurement with reference samples providing an accuracy of ±2% on the measured conductivity. Conductivity measurements were performed at a distance of 5 mm from HPT discs centers, in the location of HV measurements and microstructure characterization. The contact area was about 1 mm in diameter, so that the probed zone was about 10 mm in diameter (following the specifications of eddy current measurement procedure). The following relation was used to express ω in IACS units: IACS = ω_{Al}/ω_{Cu} * 100 [%], where ω_{Al} is the conductivity of an Al alloy in MS/m and ω_{Cu} is the conductivity of copper (58.0 MS/m).

Microstructures were characterized by transmission electron microscopy (TEM) to provide information about grain size and precipitates. Atom probe tomography (APT) analyses were also carried out to investigate more precisely the distribution of alloying elements (namely Si and Mg) within the structure with a special emphasis on clusters and segregations and also to determine the solute concentration in the matrix. TEM samples were prepared by jet polishing in a solution of 20% nitric acid and 80% methanol at −30 °C. Observations were carried out on a JEM-2100 microscope operating at 200 kV and on a JEOL ARM-200F probe corrected microscope operating at 200 kV. Scanning TEM (STEM) was performed with a probe size of 0.2 nm and a convergence angle of 34 mrad. STEM Dark Field (STEM-DF) images were recorded with a collection angle in a range of 20–50 mrad. Elemental mapping was performed using Energy Dispersive X-ray Spectroscopy (EDS) with a JED2300 detector. APT samples were also prepared by standard electropolishing techniques [38]. Analyses were performed in ultra high vacuum conditions, using an energy compensated atom probe equipped with an ADLD detector [39] and a reflectron. Samples were field evaporated at 40 K using electric pulses (30 kHz pulse repetition rate and 20% pulse fraction). The amount of Si and Mg in solid solution was directly estimated from regions free of precipitates. When the precipitate number density was large, a filtering procedure based on the local concentration was applied to remove atoms belonging to precipitates (threshold value of the local composition of 1 at.% Mg).

Most of the microstructural data were collected on the 6101 alloy. They provide a basis for the understanding of the fundamental mechanisms operating both during SPD and aging in such