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Mechanical and functional properties of ultrafine grained Al wires reinforced by nano-Al_2O_3 particles

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ABSTRACT

A powder metallurgy route based on high-energy ball milling, powder consolidation by hot extrusion and cold rolling was used to produce Al composite wires reinforced with Al_2O_3 nanoparticles. The process was capable of preparing fully dense nanocomposites characterized by well dispersed nanoparticles in a ultrafine grained matrix. Ball milling led to the fragmentation of the passivation oxide layer that covers the aluminum particles and of the alumina particle clusters added ex-situ in addition to embedding these nano-sized particles in the Al matrix and thus producing optimal precursors for subsequent consolidation. The nanocomposites showed improved mechanical performances in term of hardness and tensile strength. They also exhibited excellent damping behavior at high temperatures.

1. Introduction

Metal matrix nanocomposites (MMnCs) are considered interesting materials since they show higher strength than the corresponding base metals while retaining a good toughness [1–3]. They are generally made up of a ductile metal matrix reinforced with hard nanoparticles (NPs). Different from precipitates formed in precipitation hardening alloys, the reinforcing NPs are thermodynamically stable, making MMnCs ideal for high temperature applications [1–3]. Such small particles can obstruct the motion of dislocations and are responsible for the formation of geometrically necessary dislocations due to the mismatch in coefficients of thermal expansion and in elastic moduli between the metal matrix and the NPs [4]. Low wettability and a high surface to volume ratio of ceramic nano-particles are the main issue to face to prepare MMnCs. NPs tend to agglomerate and form clusters, losing their capability to effectively obstruct the movement of dislocations. For this reason, they cannot be prepared by conventional casting methods. To overcome this problem several non-conventional manufacturing methods have been proposed, and they can be categorized into two major groups: ex-situ and in-situ synthesis routes [1–3]. The former refers to those processes in which the nano-reinforcement is added to the liquid or powder metal whereas the latter refers to those methods that lead to the formation of nano-sized compounds during the process itself, e.g. through reacting gases. Powder metallurgy routes [5–10], ultrasound assisted casting [11,12], disintegrated melt deposition [13] are some of the processes commonly used to produce MMnCs.

The high strength of MMnCs can be further improved by decreasing the grain size of the matrix down to the sub-micrometer level (Hall–Petch strengthening) [14]. Indeed, ultrafine grained (UFG) materials, processed by severe plastic deformation methods like equal channel angular pressing or high pressure torsion, have attracted growing interest because of their unique physical and mechanical properties [15]. The combination of properties conferred to the aluminum matrix by the combination of an UFG microstructure and hard NPs would be particularly attractive for all those applications requiring low density and high mechanical properties.

In this investigation, a powder metallurgy route to produce Al-based MMnCs reinforced by Al_2O_3 NPs is adopted. An UFG microstructure was conferred to the micro Al particles through high-energy ball milling (BM) and preserved during consolidation thanks to the oxide dispersion. Ball milling has proven to be a suitable technique for breaking the surface oxide layer that covers the aluminum particles into nano-sized fragments (in-situ production of NPs). It also revealed able to embed NPs into the ductile Al matrix. The ball milled powder, after canning, were consolidated via hot extrusion. The extruded rods were cold rolled down into

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wires to verify the formability. The wires were then characterized for mechanical properties (tensile and Vickers hardness) and microstructures including NPs dispersion and grain sizes by electron microscopy analysis.

The damping capacity, or internal friction (IF), was also investigated. IF is a measure of the energy dissipated by a material during imposed mechanical vibration under cyclic loading. IF of the Al/Al_2O_3 MMnC wires was studied to consider these materials as possible candidates for applications in which the combination of good mechanical properties and high internal friction required. Indeed, vibrations generated in response to a dynamic loading are responsible for high noise levels, premature fatigue failure and wear in most of the frequently used structural materials such as steels and Al alloys which exhibit relatively low IF [16,17].

2. Method

A commercial purity Al powder with an average size of 20 μm (supplied by ECKA Granules) and a colloidal solution of alumina particles with an average particle size of 50 nm in isopropyl alcohol (supplied by Sigma Aldrich) were used as starting materials. The aluminum particles were passivated by exposure to air to possess a thin oxide layer. According to previous works [18–23], the thickness of this layer is considered to be in the range of 2–4 nm. The nominal oxygen content in the Al powder was <0.5 wt.% (data supplied by ECKA Granules). A scheme of the starting materials used for MMnCs manufacturing is depicted in Fig. 1.

The Al powder was added to the colloidal solution (with 2 wt.% of ex-situ Al_2O_3 NPs in the composite) and the mixture was stirred in a beaker and then dried at 50 °C. After this operation, the Al particles were covered by the NPs in clusters, as illustrated in Fig. 2.

High energy ball milling was performed on the Al powder or the Al–2 wt.% Al_2O_3 powder by using a Vario-Planetary Mill Pulverisette 4 equipped with steel vials and balls (10 mm in diameter). 1.5 vol.% of ethanol was used as process control agent to avoid excessive cold welding and agglomeration of the particles. To minimize oxidation during attrition, the two vials were back-filled with argon. The milling was performed for 16 h with a ball-to-powder weight ratio of 10:1. An excessive temperature rise was avoided by interrupting the procedure for 10 min after each 30 min of milling. The speed of the main disk was set at 250 rpm clockwise whereas the speed of the two planets at 200 rpm counter-clockwise. After milling, the powders were packed in cylindrical Cu alloy cans (external diameter = 10 mm and thickness = 1 mm). They were closed by means of press-fit plugs and then subjected to hot extrusion at 400 °C, as described in [24]. The extrusion speed was 5 mm/min. The die was heated by an induction coil and the temperature was monitored by a type-K thermocouple. The starting billet diameter was reduced to 4 mm after extrusion. After hot deformation, the can material was stripped off and the rods were cold rolled down to a square section of 1 mm^2 by a caliber rolling mill, with intermediate annealing at 400 °C for 5 min after each area reduction of about 20%.

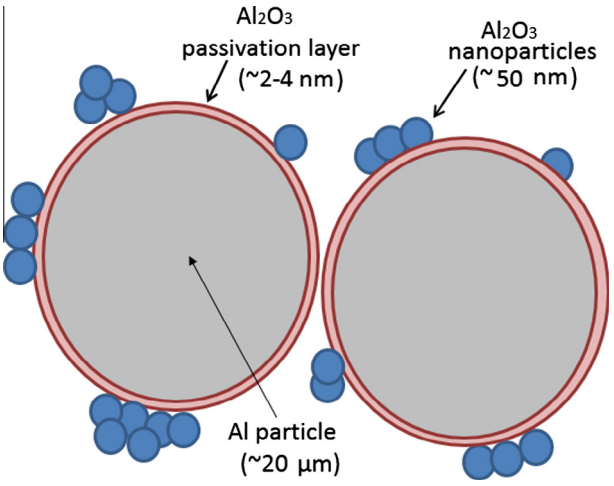

Fig. 2. Schematic of Al particles covered by alumina NPs after mixing and drying.

The density of the materials was estimated based on the Archimedes' principle using polished samples. Five measurement were performed for each type of sample.

Microstructural analysis of the transversal section of the rolled wires was carried out by scanning electron microscopy (SEM) – Zeiss Supra 40 equipped with a high efficiency in-lens SE detector. To obtain better contrast between the NPs and matrix, samples were etched with the Keller's solution. TEM foils with a final thickness of ~100 nm were prepared using a Nova Nanolab 200 focused ion beam (FIB). High angle annular dark field (HAADF) imaging by scanning transmission electron microscopy (STEM) was performed using a FEI Tecnai F20 operating at 200 kV. X-ray diffraction (XRD) was performed using PANalitical X-Pert PRO equipped with a RTMS X'Celerator sensor. Cu Kα (λ = 0.15418 nm) radiation was used. The crystallite size and micro-strain were calculated using the Williamson–Hall analysis [25].

Vickers microhardness (HV) was measured using Future Tech Corp. FM-700, applying a 2 N load for 15 s. The gauge length of the samples was 30 mm and the cross-section was 1 mm^2. Tensile tests were performed with a crosshead speed of 0.5 mm/min ($d\varepsilon/dt = 2.7 \times 10^{-4}\ s^{-1}$). Since the wire samples were too short to adopt special clamps for wires, premature fractures occasionally occurred close to the clamping position at strain levels exceeding about 4.5%. For this reason, the stress–strain curves presented have been are terminated at a strain of 4.5%.

Mechanical spectroscopy [26] is a technique that consists of applying a sinusoidal stress to a material and measuring the strain response. IF is related to the time-dependent elasticity of a material. Metals and alloys respond to an applied load not only by time-independent elastic strain, but also by time-dependent strain that lags behind the applied load. Because of the lag induced by the relaxation, the stress σ and strain ε can be expressed as:

$$\sigma = \sigma_0 \exp(i\omega t) \quad (1)$$

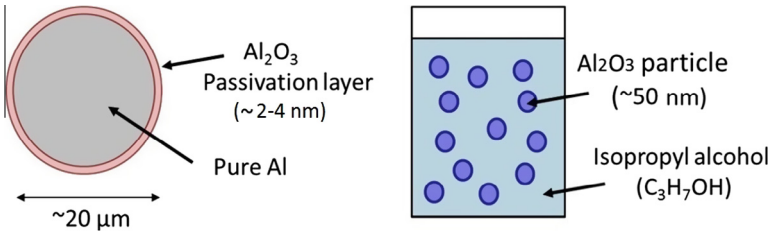

Fig. 1. Schematics of a passivated aluminum particle and the colloidal solution of alumina particles in isopropyl alcohol.