Materials Science & Engineering A 660 (2016) 77–83

Contents lists available at ScienceDirect

Materials Science & Engineering A

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

Low weight steel-magnesium composites achieved by powder compaction

Julien Nguyen [a,*], Frédéric Douville [a], Olivier Bouaziz [b], Xavier Sauvage [a]

[a] Groupe de Physique des Matériaux, CNRS UMR 6634, Université et INSA de Rouen, St-Etienne-du-Rouvray, France
[b] Laboratoire d'Etude des Microstructures et de Mécanique des Matériaux (LEM3), CNRS UMR 7239, Université de Lorraine, Metz, France

ARTICLE INFO

Article history:
Received 1 December 2015
Received in revised form
25 February 2016
Accepted 27 February 2016
Available online 28 February 2016

Keywords:
Steel
Magnesium
Metal matrix composite
Co-deformation
Strength
Size effect

ABSTRACT

Low weight steel-magnesium composites with various volume fractions of Mg were achieved by a multi-step co-extrusion process. Hollow tubes filed with Mg powders were extruded, annealed, cut and re-stacked in hollow tubes before subsequent extrusion. This way, fully dense composites with various architectures and length scales were achieved. Microstructure observations revealed that both phases undergo an axisymmetric deformation in the early stage of the process. At higher extrusion ratio, Mg/Fe interfaces become wavy and irregular which is attributed to a strong crystallographic texture. In the as-extruded states, the composites exhibit peculiar elastic to plastic transition with an unusually high strain hardening. Tensile tests performed after annealing prove that this behavior is directly linked to large internal stresses.

1. Introduction

There is nowadays a strong demand in the automotive industry for the design of low weight structures. Motivations are led by energy savings and CO_2 emission reductions. There is therefore a need for new materials with enhanced specific strength combined with good formability, high stiffness and toughness. One possible way is to create composites in which steel is combined with a light metal like aluminum or titanium [1–5]. Magnesium and its alloys also belong to the category of light metals and are therefore potentially interesting for the design of such composite materials [6]. It is important to note that the mutual solubility of iron and magnesium is extremely low and that no intermetallic phases are reported in the Fe–Mg phase diagram [7]. The melting point of Fe is significantly higher than that of Mg; therefore the most realistic way to achieve Fe–Mg composites is probably to use a mechanical process as proposed by Russel et al. [8]. Using a co-deformation extrusion process, they managed indeed to significantly increase the strength of magnesium by inserting up to 20% volume fraction of steel fibers. But one limitation of such process is the low intrinsic ductility of magnesium owing to its hexagonal structure (it exhibits a typical elongation-to-failure lower than 10% [6]). However, it is interesting to note that earlier studies have shown that grain refinement could lead to a significant enhancement of the ductility [9] and that severe plastic deformation processes, such as co-drawing or co-extrusion, may significantly refine microstructures. Interphase boundaries play a significant role in this evolution [8] and it may additionally lead to a significant increase of the material strength [10].

In a previous study, we have demonstrated that a composite made of a steel matrix with magnesium fibers (24% volume fraction) can be successfully achieved by co-extrusion [11]. The limited ductility of Mg resulting from the original texture of the extruded rods was however a limiting factor for the design of fine scaled structures. To overcome this issue, we propose in the present work a new approach based on a multi-step co-extrusion process where hollow tubes are filed with Mg powders, then extruded, annealed, cut and restacked in other hollow tubes before another extrusion step. This way, various volume fractions of Mg, with different length scales and architectures have been obtained to investigate the relationships between composite structures and properties.

2. Experimental

2.1. Materials and fabrication procedure of the composites

Materials used in this study were magnesium powder (purity 99.8%, powder size ≤ 250 μm) purchased from Goodfellow and low carbon steel tubes with an outside and inside diameters of 9 and 7 mm, respectively. To get a fully recrystallized microstructure as received steel tubes were annealed at 600 °C for 10 h in a primary

* Corresponding author.
E-mail address: julien.nguyen@etu.unilim.fr (J. Nguyen).

http://dx.doi.org/10.1016/j.msea.2016.02.083
0921-5093/

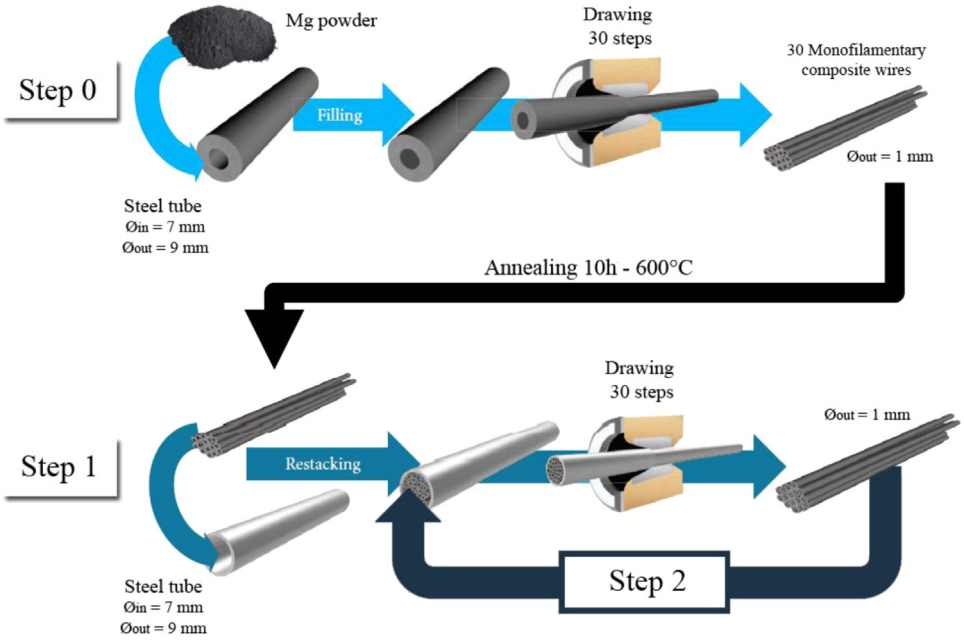

Fig. 1. Schematic illustration of the co-extrusion process used for the fabrication of the composite steel-magnesium wires.

vacuum (ca. 12 Pa) and cleaned by sandblasting.

Plastic deformation was achieved with a 50 kN capacity drawing bench using a set of 30 tungsten carbide dies. At each pass, a reduction ratio between 10% and 20% was applied. At first, magnesium powder was manually introduced in a 33 cm long steel tube. Thus, the initial theoretical volume fraction of magnesium was 60%. Then, both ends of the so-filled tube were closed by pointing to avoid any magnesium loss during drawing. A schematic representation of the process is shown in Fig. 1. At the step 0, the magnesium-filled tube was cold drawn down to a diameter of 1 mm in 30 passes with an intermediate annealing treatment (2 h at 600 °C) performed at 45% of reduction to recover some ductility. After this first drawing step, the material was annealed again (10 h at 600 °C), cleaned and cut into 30 pieces that were stacked in another steel tube (outer diameter 9 mm, thickness 1 mm, see Fig. 1). Then, at the next step, similar to the first one, the composite was cold drawn again down to 1 mm in 30 passes, with two intermediate annealing treatments (10 h at 600 °C). Finally, an additional step (step 2), similar to step 1 (see Fig. 1), was done to achieve a composite (1 mm in diameter) with a number of Mg filaments of close to 1000 and a Mg volume fraction near 10%. Composites investigated in the present work have been listed in Table 1.

2.2. Microstructural observations and mechanical characterization

Composite microstructures have been observed by optical microscopy at steps 0, 1 and 2 with an Olympus BX51M microscope.

Table 1
The three fabrication steps of Fe–Mg composites.

	Step 0	Step 1	Step 2
Bundling	Magnesium powder filled in tube	30 wires of step 1 in tube	31 wires of step 2 in tube
Theoretical volume fraction of Mg (%)	60	30	10
Theoretical final diameter of Mg filaments (μm)	770	86	9.6

Additional Scanning Electron Microscopy (SEM) observations have been also performed for step 3 with a ZEISS LEO 1530 XB microscope. Samples were mounted and mirror polished with 6 and 1 μm diamond pastes before observation without chemical etching. Structural characterization was also performed using X-ray diffraction (XRD) with a Siemens D8 diffractometer (Co K_α radiation, $\lambda = 1.7889$ Å), within the $2\theta = 35–90°$ range, with steps of 0.03° and an exposure time of 1 s. X-ray patterns were indexed with the DIFFRAC+EVA software (Bruker AXS) containing the PDF database. In order to characterize the mechanical behavior, tensile tests have been performed at the end of each step (wire diameter 1 mm) in the as-extruded and annealed states. Measurements were repeated 10 times on different samples using an Instron testing machine with a 5 kN load cell. Measurements were carried out at room temperature with a 15 mm gauge length and at a strain rate of $1.11 \times 10^{-3}\ s^{-1}$.

3. Results and discussion

3.1. Composite microstructures

On the optical micrograph taken at the end of step 0, in the composite center (i.e. in the Mg core of the composite), some dark lines are clearly visible (Fig. 2). They are attributed to boundaries between Mg powder grains that have been elongated and compacted during drawing. The cross sectional diameter of these powder grains is in a range of 10–40 μm, corresponding to a reduction by a factor of ten as compared to the original powder grain size (about 250 μm). This reduction ratio is close to the composite diameter reduction ratio performed during the step 0, indicating that both phases undergoes an axisymmetric plastic deformation. Only few porosities remains, they appear as larger dark areas on Fig. 2, up to 10 μm in size. The Mg core could however be considered as nearly fully dense. Indeed, the volume fraction of Mg at the end of step 0 is down to 50%, significantly lower than the original free volume of the steel tube filed with Mg powder in the initial stage (60%). This clearly indicates that most of porosities have disappeared during drawing as a result of the powder compaction.