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Anisotropy of the residual strain in Mg–SiCp composites

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Abstract

Residual strains have been determined by neutron diffraction in pure magnesium matrix composites. In the reinforcing phase, they are hydrostatic, compressive and increase with increasing reinforcement volume fraction. Both the unreinforced alloy and the composites present anisotropic matrix residual strains. They are hydrostatic and compressive for grain with their basal pole almost perpendicular to the extrusion direction (''basal oriented grains'') and tensile and deviatoric for some other orientations (''non-basal grains''). This can be explained modeling the material as a three-phase composite, where both the ''basal oriented grains'' and the SiC particles are hard phases and the rest of the Mg grains are the soft phase.

Keywords: A. Metal matrix composites (MMC); C. Residual stress; D. Neutron diffraction; A. Hexagonal lattices; B. Elastic–plastic anisotropy

1. Introduction

Magnesium alloys present a great potential as structural materials in the aerospace and automobile industries mainly because of their low density and high specific strength. However, because of their rapid loss of strength at temperatures above ambient and their poor creep resistance at elevated temperatures, they are rarely used above 190 °C (half the melting point), unlike aluminium alloys with a similar melting temperature. The addition of ceramic particles improves the creep resistance at intermediate and high temperatures. A second harder phase leads generally to an increase in the tensile strength, Young's modulus and hardness, particularly at room temperature, and to a reduction of the coefficient of thermal expansion (CTE).

The advantages of magnesium alloys prepared by powder metallurgy stem from the fine grain size, the increase in solid solubility of alloying element and near net shape production. Moreover, it is possible to control the final microstructure, which leads to optimum mechanical properties, by means of the extrusion temperature.

An important aspect of magnesium alloys is the anisotropy of their physical properties. The mechanical properties of these materials are strongly dependent on their texture, especially at low temperature. Wrought magnesium alloys develop a strong texture with the basal pole (c direction) parallel to the stress direction and their mechanical properties depend not only on the stress direction but also on the stress sign (tension or compression) [1–3]. The tension/compression asymmetry at room temperature is a well-known phenomenon in wrought magnesium alloys and is related to their texture, which determines the slip systems controlling the plastic flow [4–9]. The slip system most easily activated at room temperature is the basal $(0001)\langle 11\bar{2}0\rangle$ system. Moreover, magnesium can also deform by twinning. The more common twinning system is the $\{10\bar{1}2\}\langle 10\bar{1}1\rangle$. However, the basal slip system is inhibited when the material is deformed in the extrusion or rolling direction and the $\{10\bar{1}2\}\langle 10\bar{1}1\rangle$ twinning system is active in compression but not in tension [3–9]. For magnesium matrix composites produced by powder metallurgy, the tension/compression asymmetry (the strength differential effect, SDE) decreases with increasing volume fraction

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of ceramic particles, due to the decrease of the fiber texture intensity [10,11]. Moreover, for higher reinforcement volume fraction the tension/compression asymmetry reverses and the composites become stronger in compression than in tension, which is the usual behaviour of many other composites [12]. In aluminium matrix composites, a positive SDE (higher strength in *compression*) has been related to the presence of *tensile* residual stresses (RS) in the matrix [13]. Some diffraction work has been carried out to track applied stresses in magnesium alloys, especially in welding and highly deformed materials [14–17], but very little is available on *Residual* Stress.

Residual microstrains and microstresses also evolve as a consequence of deformation or texture formation (e.g. because of extrusion). Recently, Agnew et al. [17] have studied the evolution of the internal strains of some crystallographic orientations during plastic deformation in a AZ31 alloy. They showed that there is an extended elasto–plastic transition lasting up to at least 10% macroscopic strain. The apparent strain hardening is actually the result of a composite-like load sharing between soft- and hard crystallite orientations. Moreover, due to the plastic anisotropy, after deformation it was also observed that the formation of residual strains depends on the relative crystallographic orientation of each grain [16,17]. These observations match very well with theoretical predictions.

Both the crystallographic elastic and plastic anisotropy and the volume fraction of the reinforcement phase heavily influence the intergranular stresses [18]. These residual microstresses are mainly local but their magnitude can reach that of the macro-stress. Thus, they often have a strong impact on the fatigue and the corrosion resistance of heavily deformed materials. This study aimed at casting light on the effect of the reinforcement content on the residual stress (RS) in magnesium composites produced by powder metallurgy. In our case, pure magnesium matrix composites reinforced with different volume fraction of SiC particles were used to avoid any possible influence of matrix precipitations, often found in Mg alloys.

2. Experimental procedure

Powders of pure magnesium with less than 45 μm particle size, were blended with 5% and 13% volume of SiC particles with particle size around 5 μm (aspect ratio ~1). Homogenization of the blends was conducted in a high speed impeller at 110 rpm during 4 h. After homogenization, the powders were cold compacted by slowly increasing pressure up to 340 MPa in a special die designed for this propose. The resulting compacts were extruded at 400 °C employing an extrusion ratio of 18:1.

Microstructural characterization of the composites was carried out through optical microscopy. Metallographical preparation consisted in mechanical polishing and etching in a solution of 5 g of picric acid, 0.5 ml of acetic acid, 5 ml of water and 25 ml of ethanol.

Laboratory X-ray diffraction (XRD) was used for texture measurements. The texture analysis was carried out by the Schulz reflection method, using a SIEMENS™ Kristalloflex D5000 diffractometer equipped with a full circle Eulerian cradle. The X-radiation used was β-filtered Cu Kα. The orientation distribution functions (ODFs) and the inverse pole figures(IPFs) were computed from the measurement of (0002), $(10\bar{1}1)$, $(10\bar{1}2)$, $(10\bar{1}3)$ and $(11\bar{2}0)$ pole figures and using the series expansion method [19].

Cylindrical samples were machined from the extruded bar with their axes parallel to the extrusion (Z) direction. The final sample dimensions were: diameter 9 mm and length 18 mm.

The residual stress analysis was carried out by neutron diffraction. The neutron diffraction measurements were done on the beamline D1A [20] at the Institut Laue-Langevin (ILL), Grenoble, France. Monochromatic neutrons with wavelength $\lambda = 1.91$ Å were used. A gauge volume of approximately $4 \times 4 \times 1$ mm^3 was defined by a primary slit and a secondary radial collimator. The gauge volume was positioned in the centre of the specimens under investigation.

The $\sin^2\psi$ technique was used in the ω-mode, see [21] for a thorough description. The tilt angle ψ was defined as the angle between the specimen axis (extrusion direction) and the scattering vector **Q** (bisecting the angle between the incident and diffracted beam directions). The specimens were tilted in the scattering plane from the axial $\psi = 0$ to the radial $\psi = \pm 90°$ direction. Up to nine ψ tilts were used, but at some ψ angles peaks without a reasonable signal-to-noise ratio were detected. As commented in the introduction, extruded bars develop a strong fiber texture with the basal planes parallel to the extrusion direction and therefore we only found intense peaks at $\psi \sim \pm 90°$ for the $10\bar{1}3$ near-basal peak. Consequently, the use of different diffraction peaks was crucial to obtain a signal in the whole ψ range from $\psi = 0$ to $\psi = \pm 90°$. In fact, the $(11\bar{2}2)$ and $(20\bar{2}1)$ magnesium lattice planes have also been used for the residual stress analysis. In spite of that and because of texture, the counting times for each acquisition varied from 20 min to more than 1.5 h, depending on ψ. In the case of the SiC phase, the 311 diffraction peak was used for the analysis.

A position-sensitive detector collected the diffracted neutrons in a ~4° wide 2θ-range around 80.8° for the Mg $10\bar{1}3$ peak, 89.0° for the Mg $11\bar{2}2$ and $20\bar{2}1$ peaks and 93.2° for the SiC 311peak. The diffraction peaks were fitted by a Gaussian curve with constant background. Magnesium and silicon carbide powders canned in vanadium were used as reference specimens.

For each of the two phases, the elastic residual strains in the principal direction i can be calculated by the shift on the position of the diffraction peak

$$\varepsilon_i = \frac{d - d_0}{d_0} = -(\theta_i - \theta_0)\operatorname{cotan}\theta_0 \quad (1)$$