



## Mechanical and fracture properties of an AZ91 Magnesium alloy reinforced by Si and SiC particles

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### ABSTRACT

A commercial AZ91 magnesium alloy (nominal composition Mg–9%Al; 1%Zn; 0.3%Mn, balance Mg in weight percent) reinforced with SiC particles and modified by the addition of Si has been used in this study. Formation of an “in situ” composite (Mg–Mg<sub>2</sub>Si) results in strong bonding between Mg<sub>2</sub>Si and the matrix interface. Samples were deformed in compression in the temperature interval from room temperature up to 300 °C. Stress relaxation tests were performed with the aim to reveal the thermally activated processes. Reinforcing effect of SiC and Mg<sub>2</sub>Si particles decreases with increasing temperature. The estimated values of the activation volume as well as the activation enthalpy indicate that the main thermally activated process is connected with a rapid decrease of the internal stress. Fracture properties were studied in impact tests at various temperatures. A ductility enhancement was found at 200 °C and temperatures above 200 °C.

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### 1. Introduction

Mechanical properties of magnesium alloys decrease rapidly with increasing temperature. Remarkable improvement of mechanical properties can be reached by the addition of reinforcing phases (fibres or particles) which may improve stiffness, wear resistance, thermal expansion coefficient (CTE) and deformation properties at elevated temperatures [1–7].

AZ91 magnesium alloy is the most popular commercially available magnesium alloy. This alloy shows superior castability and good mechanical properties combined with good corrosion resistance for the high purity versions of the alloy. Typical applications include transmission casings, valve covers, intake manifolds, brackets, pumps, etc. Mg–Al–Zn based alloys like AZ31, AZ61, AZ80 and AZ91 are, in general, utilized for applications at ambient or slightly elevated temperatures primarily in the automotive and electronic industry. They successfully combine acceptable mechanical properties with good castability and relatively low production costs.

Solid solubility of Al in Mg is about 2.1 wt.% (1.9 at.%) at ambient temperature [8]. Exceeding of solid solubility of Al gives rise to Mg<sub>17</sub>Al<sub>12</sub> intermetallic phase in the lamellar form. An addition of Zn, (its solubility limit in Mg is about 0.8 at.% at RT [9]) further improves the mechanical properties of the Mg–Al system. The Si additions lead to the improvement of mechanical properties at elevated temperatures (up to about 150 °C). The result of negligible solubility of Si in Mg matrix is the formation of the Mg<sub>2</sub>Si intermetallic phase [10]. By this way it is possible to obtain relatively cheap alloys with acceptable mechanical properties for some thermally and mechanically exposed parts in the transport industry [11].

It is generally accepted that during deformation of a composite, deformation process occurs in the matrix and short ceramic fibres deform only elastically. An increase in the flow stress in the composite is caused mainly by the following four reasons:

- Load transfer from the matrix to fibres.
- Direct impact of fibres as impenetrable obstacles for the dislocation motion.
- Influence of fibres on the development of the dislocation substructure in the matrix.
- Influence of fibres on the microstructure formation.

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While the intrinsic mechanical and physical properties of a reinforcement (stiffness, strength, and thermal expansion) are dictated by its chemical nature, the geometric and topological parameters of the reinforcement (shape, size, volume fraction, spatial orientation, and distribution) can be altered during processing.

According to the dislocation theory, the applied stress  $\sigma$  necessary for deformation of any polycrystalline material can be divided into two components: the internal (athermal) stress  $\sigma_i$  and the effective stress  $\sigma^*$ , i.e. one can write

$$\sigma = \sigma_i + \sigma^* \quad (1)$$

The internal stress  $\sigma_i$ , resulting from long-range internal stresses impeding the plastic flow can be written as

$$\sigma = \alpha_1 G b \rho_t^{1/2}, \quad (2)$$

where  $G$  is the shear modulus,  $\alpha_1$  is a constant describing interaction between dislocations,  $b$  is the Burgers vector of dislocations and  $\rho_t$  is the total dislocation density. The effective shear stress  $\sigma^*$  acts on dislocations during their thermally activated motion when they overcome short range obstacles. The mean velocity of dislocations  $v$  is connected with the plastic strain rate by the Orowan equation:

$$\dot{\epsilon} = (1/\psi) \rho_m b v, \quad (3)$$

where  $\rho_m$  is the density of mobile dislocations and  $\psi$  is Taylor orientation factor. The most common equation used in describing the average dislocation velocity as a function of the effective stress is an Arrhenius type. The plastic strain rate  $\dot{\epsilon}$  for a single thermally activated process can be expressed as:

$$\dot{\epsilon} = \dot{\epsilon}_0 \exp[-\Delta G(\sigma^*)/kT], \quad (4)$$

where  $\dot{\epsilon}_0$  is a pre-exponential factor containing the mobile dislocation density, the average area covered by the dislocations in every activation act, the Burgers vector, the vibration frequency of the dislocation line, and the geometric factor.  $T$  is the absolute temperature and  $k$  is the Boltzmann constant.  $\Delta G(\sigma^*)$  is the change in the Gibbs free enthalpy depending on the effective stress  $\sigma^* = \sigma - \sigma_i$  and the simplest form is

$$\Delta G(\sigma^*) = \Delta G_0 - V\sigma^* = \Delta G_0 - V(\sigma - \sigma_i). \quad (5)$$

Here  $\Delta G_0$  is the Gibbs free enthalpy necessary for overcoming a short range obstacle without the stress and  $V = bdL_c$  is the activation volume where  $d$  is the obstacle width and  $L_c$  is the mean length of dislocation segments between obstacles.

The stress relaxation technique is very useful method to study the thermally activated processes and to reveal the dominant process occurring during plastic deformation. In a stress relaxation (SR) test, the specimen is deformed to a certain stress,  $\sigma_0$ , then the machine is stopped and the stress is allowed to relax. The stress decreases with the time  $t$ . The specimen can be again reloaded to a higher stress (load) and the SR test may be repeated. The time derivative  $\dot{\sigma} = d\sigma/dt$  is the stress relaxation rate and  $\sigma = \sigma(t)$  is the flow stress at time  $t$  during the SR. The stress relaxation tests are very often analysed under the assumption that the SR rate is proportional to the strain rate  $\dot{\epsilon}$ , according to [12], as:

$$\dot{\epsilon} = -\dot{\sigma}/M, \quad (6)$$

where  $M$  is the combined modulus of the specimen–machine set. The stress decrease with the time during the SR can be described by the well known Feltham equation [13]:

$$\Delta\sigma(t) = \sigma(0) - \sigma(t) = \alpha \ln(\beta t + 1), \quad (7)$$

where  $\sigma(0) \equiv \sigma_0$  is the stress at the beginning of the stress relaxation at time  $t = 0$ ,

$$\alpha = \frac{kT}{V}, \quad (8)$$

$$\beta = \frac{M\dot{\epsilon}_0 V}{kT} \exp\left[-\frac{\Delta G_0 - V\sigma^*(0)}{kT}\right] = \frac{M\dot{\epsilon}(0)}{\alpha}, \quad (9)$$

where  $\dot{\epsilon}(0)$  the plastic strain rate at the beginning of the relaxation.

In the present work, the microstructure, mechanical and fracture properties of the AZ91 alloy-based composite consisting of the intermetallic phase ( $\text{Mg}_2\text{Si}$ ), formed during the manufacture process and reinforcing SiC particles were studied.

## 2. Experimental procedure

A commercially available AZ91 magnesium alloy (the nominal composition Mg–9%Al; 1%Zn; 0.3%Mn; balance Mg in weight percent) was reinforced with 10 vol.% of SiC particles (SiCp) and an addition of 3 wt.% of Si. The size of the SiCp exhibited about 20  $\mu\text{m}$ . Composite was prepared by the squeeze cast technology. The preform was infiltrated by the liquid alloy using two-stage application of the pressure. The samples were subjected to a standard T6 thermal treatment (annealing for 18 h at 413 °C in the protective argon atmosphere, then ageing for 8 h at 168 °C [11]) Compression tests were carried out at an initial strain rate of  $3.3 \times 10^{-5} \text{ s}^{-1}$  over a wide temperature range from room temperature (RT) to 300 °C. Dimensions of the samples used in compression tests were  $5 \times 5 \times 10 \text{ mm}^3$ .

Sequential stress relaxation tests were performed at increasing stress along a stress–strain curve. Duration of the SR was 300 s. Ductility of the alloy at room temperature is very low and therefore the SR tests were performed at elevated temperatures. On the other hand, recovery during the SR test was observed at 300 °C and hence, the results obtained at this temperature were not taken into account because the above given equations describing the SR were derived under an assumption that the internal stress  $\sigma_i$  is constant during the SR. Only in the first SR test at 300 °C, recovery may be considered to be negligible, i.e.  $\sigma_i$  is constant.

Components of the applied stress ( $\sigma_i$ ,  $\sigma^*$ ) were estimated using Li's method [14,15]. The SR curves were fitted to the power law function in the form:

$$\sigma - \sigma_i = [a(m-1)]^{\frac{1}{1-m}} (t + t_0)^{\frac{1}{1-m}}, \quad (10)$$

where  $a$ ,  $t_0$  and  $m$  are fitting parameters.

The impact tests were performed by the Charpy pendulum in the temperature range from RT to 300 °C. The samples with dimensions of  $10 \times 10 \times 55 \text{ mm}^3$  were machined from the billet using standard tools. The U-type notch has been created by the electro erosive cutting to 2 mm depth. High temperature tests were conducted with the preheated samples in an electric furnace. The fracture surfaces were investigated immediately after the impact tests using a scanning electron microscope (SEM) in order to prevent possible oxidation.

Samples for the microstructure analysis were prepared by the conventional mechanical polishing and etching using glycol solution (1 ml  $\text{HNO}_3$ , 24 ml water, 75 ml ethylene glycol). In order to obtain detailed three-dimensional morphology the surfaces of samples were deeply etched using a mixture of 36 ml phosphoric acid ( $\text{H}_3\text{PO}_4$ ) and 64 ml ethyl alcohol. The samples were etched for 30–60 s by an electrolytic method at the current  $I = 0.05 \text{ mA cm}^{-2}$  and the voltage of 0.7 V. For the phase identification, a SEM analysis was performed on the samples that undergone both etching and deep etching procedure.

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