



# Microstructure, texture, and mechanical properties of friction stir spot welded rare-earth containing ZEK100 magnesium alloy sheets



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## ABSTRACT

The effect of friction stir spot welding process parameters on the microstructure, texture, and mechanical properties of ZEK100 (Mg–1.0 wt% Zn–0.5 wt% RE–0.5 wt% Zr) Mg alloy was investigated. Lap-shear joints were prepared using two different tool rotational speeds (1500 and 2250 rpm) and three different shoulder plunge depths (0.0, 0.2, 0.6 mm). Microstructure analysis revealed significant grain refinement in the stir zone, when compared to the base material. Electron backscatter diffraction analysis revealed a strong texture development in the keyhole periphery and adjacent regions despite the presence of RE-elements, however, no significant texture variation was observed within the process parameters. These results suggest that the ultimate failure of the weld is more attributed to macroscopic features such as the bond width and upper sheet thickness rather than texture development.

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## 1. Introduction

The increasing demand for fuel-efficient vehicles has driven the transportation industry to investigate the use of lightweight materials. Due to their low density and high strength, magnesium alloys are attractive materials for drastically reducing the weight of structural components. However, due to the high-energy consumption, solidification cracking, porosity, and high residual stresses [1–4], conventional fusion joining techniques such as resistance spot welding (RSW) are generally impractical for joining Mg alloys. Among the alternatives, friction stir welding (FSW) and friction stir spot welding (FSSW) are excellent joining techniques for Mg alloys.

Despite their great potential and attractive properties, the application of wrought Mg alloys has been limited in use compared to other alloys (e.g. aluminum and steel). This limited use has been mainly attributed to the low ductility and formability of Mg alloys at room temperature [5–7]. Mg alloys consist of a hexagonal close-packed structure, where there is a limited number of deformation mechanisms that can be activated at room temperature (e.g. basal slip, tensile twinning) [5]. Furthermore, the

limited ductility at room temperature has been attributed to the formation of strong textures produced after plastic deformation. Such textures contain the majority of the grains oriented with their basal plane (0001) aligned parallel to the rolling plane, thus, making the material difficult to deform [5,8,9].

Recently, improvement in the formability of Mg alloys at room temperature has been demonstrated by the addition of rare-earth (RE) elements, such as cerium (Ce) and lanthanum (La) [5,6,9–11]. Among the commercially available RE containing Mg alloys, there is the recently developed ZEK100. This alloy containing Zn, Zr and RE-elements (nominal composition Mg–1.0 wt% Zn–0.5 wt% RE–0.5 wt% Zr [9]), is known to have enhanced room-temperature formability compared to other Mg alloys [5,6,9–12]. The addition of RE-elements in the ZEK series is known to weaken the texture, where the orientation spread of the basal planes is broader towards the sheet transverse direction (TD) [12]. In fact, the improved formability at room temperature is attributed to the weakened texture. On the other hand, the addition of Zr is known to serve as a grain refiner [11], whereas the Zn increases the strength via solution hardening [6,11,13,14].

Multiple studies have been conducted to characterize and understand the effects of the addition of RE-elements in Mg alloys [6,10,11,15]. In all of them, a weak texture is developed to commensurate with an improved ductility and a decreased anisotropy after warm extrusion and annealing. Ball and Prangnell [16]

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attributed the development of a weak and randomized texture to particle-stimulated nucleation (PSN), whereas Stanford et al. [17] attributed it to a combination of shear band nucleation (SBN) and retardation of recrystallization. However, the debate on the specific mechanisms that originate the RE-texture is still ongoing [18,19].

Additional studies have been conducted to characterize the mechanical performance of ZEK100 and similar alloys. For example, Min and Hector et al. [20] derived the forming limit diagrams for the ZEK100. Aslam and Li et al. [21] studied the performance of ZEK100 sheet during a three point bending test, revealing a certain degree of anisotropy despite the weakened texture. On the other hand, Kurukuri and Worswick et al. [22] studied the constitutive mechanical response of this alloy by conducting testing at different strain rates.

Evidently, there have been multiple studies regarding the mechanisms of texture development and the formability and mechanical performance of Mg alloys containing RE-elements, however there is very limited information regarding the effects of the process parameter of existing joining methods and their applicability to these alloys. Recently, Rao and coworkers [23] successfully demonstrated to use of FSSW for the welding of ZEK100 Mg alloy sheets. Their study included an analysis of the macroscopic features of the welded region such as hook formation, effective sheet thickness and bond width. However, the effect of the RE-elements on the texture and microstructure in FSSW of Mg alloys was not investigated. As such, the aim of this paper is to study the texture and microstructure development on the ZEK100 Mg alloy sheets joined by FSSW. To the best of the author's knowledge, this paper is the first to examine the microstructure and texture development in a ZEK100 joined by FSSW.

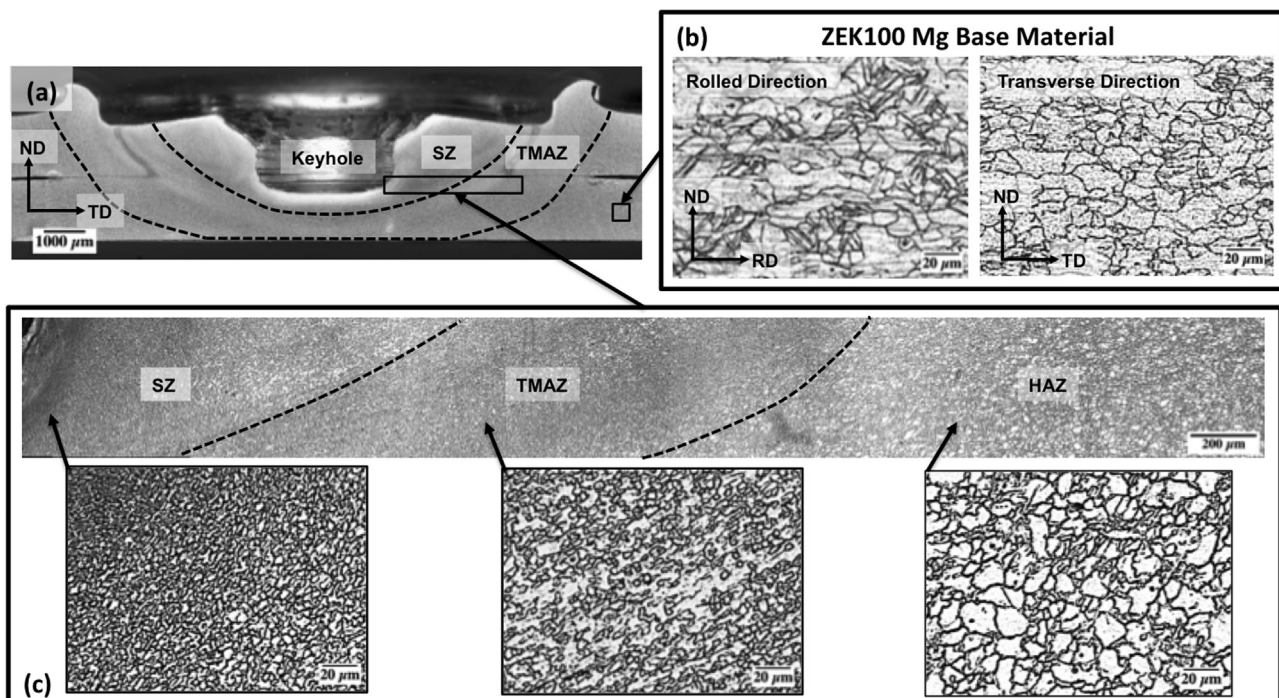
## 2. Experimental methods

FSSW lap-joints were fabricated using as-rolled ZEK100 sheets ( $100 \times 35 \text{ mm}^2$  coupons) having a thickness of 1.4 mm. Samples were made at two different tool rotational rates of 1500 and 2250 revolution per minute (rpm) and three different shoulder plunge depths varying

from 0 to 0.6 mm. A tool with a 12 mm concave shoulder and 1.8 mm long cylindrical pin was employed for the current study.

In order to characterize the effects of welding parameters on the microstructure, FSSWed samples were sectioned through the center of welded region and then cold mounted in epoxy resin. Subsequently, samples were mechanically polished using abrasive papers and diamond slurry suspensions down to  $1 \mu\text{m}$ , followed by a fine polishing step using a  $0.05 \mu\text{m}$  colloidal silica and ethylene glycol. Samples for metallographic study were etched using a solution of 4.2 g picric acid, 10 ml acetic acid, 10 ml  $\text{H}_2\text{O}$  and 70 ml ethanol. Microstructural features in different regions were characterized and quantified using a Keyence VHX-100 optical microscope. The average grain size was measured using the circular intercept method. Microhardness measurements were performed in the transverse cross-section of the FSSWed samples using  $0.5 \text{ mm}$  spacing, a load of 100 gf and a dwell time of 5 s.

Texture characterization at various regions of the weld, as a function of the welding parameters, was conducted using the JEOL 7000 scanning electron microscope (SEM) equipped with a detector for electron backscatter diffraction (EBSD). EBSD samples were electro-polished at 3 V for 20 s using  $\text{H}_3\text{PO}_4$  diluted in ethanol (3:5 ratio). The texture analysis was conducted using 20 kV beam voltage in  $0.5 \mu\text{m}$  steps. The EBSD data was acquired using the AZTEC software from Oxford Instruments, post processing was done using the HKL Channel 5 package. A FEI Tecnai F-20 transmission electron microscope (TEM) was used to characterize the precipitate formation in the base material as well as in the keyhole periphery. TEM samples were prepared using a FEI focus ion beam (FIB). Samples were machined and polished into a  $5 \times 10 \mu\text{m}^2$  and 100 nm foils. X-ray energy dispersive spectroscopy (EDS) was conducted for compositional evaluation of the developed phases. Monotonic lap-shear testing was performed on the FSSWed coupons at room temperature using an electromechanical tensile load frame in displacement control mode at a rate of 1 mm/minute. In order to characterize the lap-shear failure mode, fractography analysis was performed using a SEM.



**Fig. 1.** (a) Transverse cross-section of the FSSW ZEK100 Mg alloy sheets at 1500 rpm and 0.2 mm plunge depth. (b) Optical micrographs of the base material. (c) Microstructural features at various locations such as SZ, TMAZ and HAZ.

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