



# Interfacial design and strengthening mechanisms of AZ91 alloy reinforced with in-situ reduced graphene oxide

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## ABSTRACT

This work highlights to develop an in-situ process to disperse uniformly graphene possessing good interfacial bonds with the matrix of Mg alloy. A homogeneous distribution of graphene in the matrix has been successfully obtained through in-situ reduction of graphene oxide (RGO) by sintering process after the graphene oxide (GO) was uniformly dispersed in AZ91 powders. The interface product nano-sized MgO can significantly improve the interfacial bonding strength between RGO and  $\alpha$ -Mg with analysis by transmission electron microscopy. The orientation relationships of  $(200)_{\text{MgO}}//(\bar{1}102)_{\alpha\text{-Mg}}$  and  $[011]_{\text{MgO}}//[242\bar{3}]_{\alpha\text{-Mg}}$  with the semi-coherent interface characteristics were clarified for the first time at the interface of MgO/ $\alpha$ -Mg, suggesting a good lattice space matching. By employing such process, a composite containing 0.5 wt% of GO shows an 85.7% and 61.4% increase in yield strength and elongation, respectively, over unreinforced AZ91 alloy. The underlying strengthening mechanisms of in-situ reduced graphene oxide are discussed in detail.

## 1. Introduction

The unique performance of graphene, including excellent tensile strength, stiffness, and attractive functional properties represented by high thermal and electrical conductivities, make it a promising candidate for metal-reinforcement [1–3]. Although, graphene as the reinforcement have been widely used in the polymer matrix composites [4,5], there remain some big challenges in fabricating graphene reinforced metal matrix composites (GRMMCs). Strong van der Waals forces between the graphene sheets and their tendency to agglomerate in metal matrix are the key problems in the synthesis of GRMMCs with high performance [6–9]. Ball milling has the potential to realize a uniform dispersion of graphene in GRMMCs [10–13]. However, the original structures of graphene can be seriously damaged during ball milling. Other techniques like, molecular-level mixing [8], layer-by-layer assembly [14], etc. are time taking and complex, which were not suitable for bulk production. Graphene oxide (GO) possesses excellent dispersion ability in water or suitable organic solvents due to the plenty of oxygen-containing groups in GO. This makes GO uniformly disperse within the Al powders, which is beneficial to a good dispersion of graphene in the Al matrix [15,16]. However, the Al powders were easily oxidized during the thermal reduction of graphene oxide (RGO) in

the composite powders, causing to a poor interfacial bonding with the Al matrix. In addition, many of the resulting RGO in the composite powders were found to be seriously wrinkled or folded, leading to a negative effect on the strength of the composite [17]. Therefore, to the best use of GO as the precursor of graphene in the fabrication of graphene-Mg (magnesium) composite with high performance, a novel process should be developed to disperse uniformly graphene possessing good interfacial bonds with the Mg matrix.

Recently, Sohn et al. [18] reported that the Mg metal is highly effective in reducing GO not only because it can accelerate oxygen desorption but also because it can eliminate carbonyl defects to recover perfect graphene lattice. Theoretically, the alkalis, such as Ca and Mg can directly react with the oxygen groups of GO above 600 K, and the interface reaction product is CaO and MgO, respectively [19]. Additionally, nano-sized MgO can effectively refine the grains of Mg-Al series alloy due to the formation of semi-coherent interface of MgO/ $\alpha$ -Mg in the composite [20,21], which were identified to be a good reinforcement in strengthening the performance of Mg-based composites [22]. Direct graphene growth on the cubic MgO substrate suggests that a strong interfacial bonding between graphene and MgO have been formed due to the strong interaction of the carbon-oxygen bonds [23]. The residual oxygen in graphene nanosheets (GNS) is positive to

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increase the interfacial bonding between GNS and the Mg matrix by MgO nanoparticles owing to the formation of semi-coherent interface of MgO/ $\alpha$ -Mg and the distortion area bonding interface of GNS/MgO [24]. They have also shown that (i) GO and Mg reacts to produce graphene and MgO, and (ii) the as-formed MgO is beneficial to improve the interfacial bonding between graphene and the Mg matrix. However, the mechanism of the formation of interfacial bonds between graphene, MgO, and the Mg matrix is still indefinite. The purpose of this work is to ascertain whether GO and the as-formed MgO have contributed to homogeneous disperse graphene with good interfacial bonding in the Mg matrix.

In this work, AZ91 alloy composites reinforced by in-situ reduced graphene oxide were synthesized by powder technology with following hot extrusion and T4 treatment. Microstructure of AZ91-GO mixtures and AZ91-RGO composites was characterized. The experimental results are used to determine a fundamental insight into the interface structure and the strengthening mechanism that underlying in the developed composites.

## 2. Experimental Details

### 2.1. Materials Synthesis

Commercial AZ91 alloy powders with 99.9% in purity and 70  $\mu\text{m}$  in average particle size were used as the alloy matrix, which were purchased from General Research Institute for Nonferrous Metals, China. The starting reinforcement, graphite oxide was 99% in purity, which supplied by Nanchang Taiyang Nanocrystal company, China.

Fig. 1 illustrates the synthesis procedure of AZ91-RGO composites. As shown in the figure, graphite oxide was dispersed into ethanol by ultrasonication for 2 h to obtain the GO ethanol solution. AZ91 alloy powders were added into ethanol under Ar atmosphere and stirred by a mechanical stirrer to form AZ91 suspension. The two suspensions were then mixed together by simultaneously mechanical stirring and ultrasonication for an hour to form the composite slurry. After that, the composite slurry was filtered and dried at 313 K under vacuum for 6 h to obtain the composite mixtures. Five composite mixtures with 0.1,

0.3, 0.5, 0.8 and 1.2 wt% of GO were prepared using the method stated above, respectively.

The as-obtained mixtures were compacted in a cylinder mold with a diameter of 40 mm and height of 200 mm under 120 MPa. The compacts were then sintered at 873 K for 2 h under Ar atmosphere, during which the in-situ reduction of GO took place. Because the Mg metal is efficient to remove oxygen directly from the oxidized carbons of GO due to Mg can act as an electron-donating source for the reduction of GO [18]. The as-sintered compacts were finally hot extruded with following T4 treatment. The hot-extrusion temperature, ram speed and diameter of extruding rod is 673 K, 0.3 mm/min and 12 mm, respectively (shown in Fig. 1). T4 treatment was performed at 686 K and held for 18 h, followed by quenching in air. For comparison, AZ91 alloy was also synthesized by the same procedures above.

### 2.2. Characterization

Scanning electron microscopy (SEM) (Quanta-200F, FEI, Holland) and transmission electron microscopy (TEM), (JEM-2100, JEOL Ltd., Japan) were applied to study the microstructure and phase component of GO, AZ91-GO mixtures and the as-synthesized composites. Elemental analysis was employed by energy-dispersive X-ray spectrometer (EDS) (Genesis-2000X, EDAX In., USA) and energy dispersive X-ray spectroscopy (EDX) (Inca100, Oxford Instruments, UK). Optical Microscopy (OM) (XJG-05, Jiangnan Novel Optics Co., Ltd., China) and X-ray diffraction (XRD) (D/MAX-1200, Rigaku Corporation, Japan) were used to characterize the microstructure of the composites. Vickers hardness (HV) of AZ91 composites was measured by an automatic hardness tester (HVS-1000A, Bimu Instrument Co., Ltd., China) with an applied load of 100 g for 15 s. Uniaxial tensile tests were carried out with a strain rate of  $10^{-3} \text{ s}^{-1}$  along the extrusion direction (UTM-5105, Zhongte Testing Machine Co., Ltd., China). The size of tensile specimens is shown in Fig. 1.

In order to reveal the mechanism of interface reaction between GO and the Mg matrix during sintering, RGO was extracted from AZ91-1.2 wt% GO composite by using hydrochloric acid, and characterized by XRD, Fourier transformed spectroscopy (FT-IR) (Nicolet-5700, Thermo

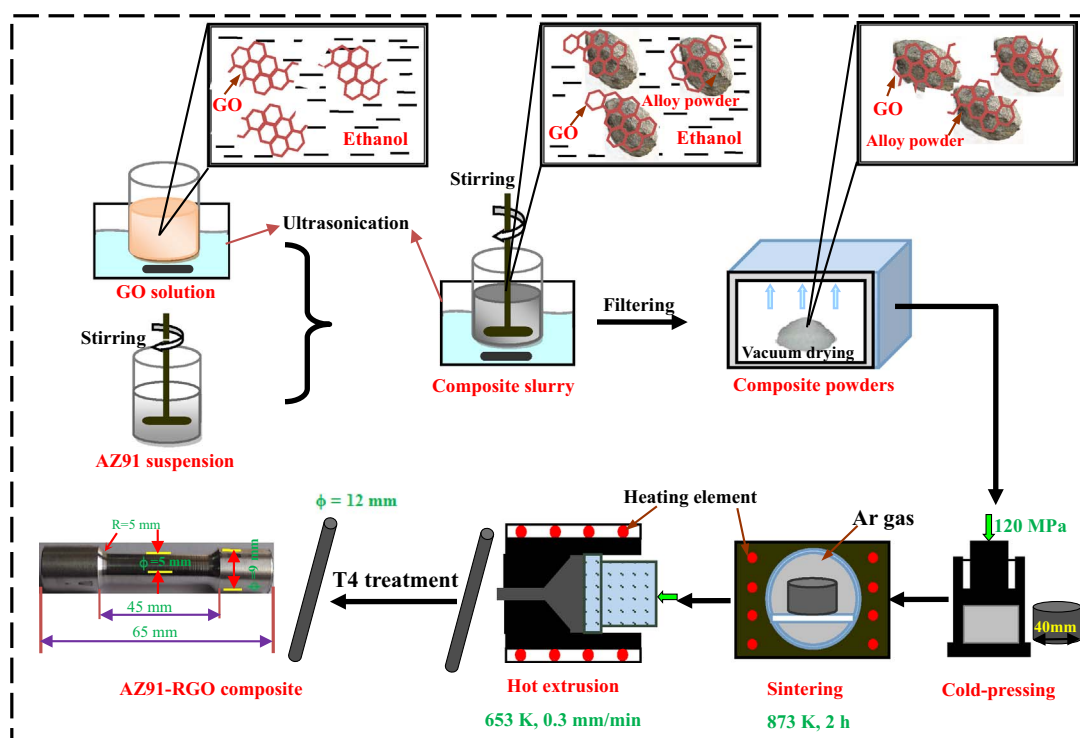


Fig. 1. Schematic illustration of the fabrication process of AZ91-RGO composites.

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