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Laser-induced microstructural development and phase evolution in magnesium alloy

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ABSTRACT

Secondary phase plays an important role in determining microstructures and properties of magnesium alloys. This paper focuses on laser-induced microstructure development and secondary phase evolution in AZ91D Mg alloy studied by SEM, TEM and EDS analyses. Compared to bulk shape and lamellar structure of the secondary phase in as-received cast material, rapid-solidified microstructures with various morphologies including nano-precipitates were observed in laser melt zone. Formation mechanisms of microstructural evolution and effect of phase development on surface properties were further discussed.

1. Introduction

Driven by energy and environmental concerns for lightweight structural as well as functional parts, magnesium alloys have been increasingly used in automotive, aerospace, and electronic industries due to their low densities and high specific strengths [1–5]. Unfortunately, practical application of Mg alloys has been largely limited due to poor surface-related properties [1,2,5]. Secondary phase in the microstructure normally helps to improve surface properties of the Mg alloys, but the effectiveness of such phase in improving surface performance depends on its amount and distribution in the matrix [1,3]. It is believed that fine and homogeneous secondary phase provides an effective barrier for performance enhancement; however, the presence of coarse and non-homogeneously distributed microstructure could deteriorate surface performance [1,2,5].

In recent years, laser processing of metallic materials including Mg alloys has attracted considerable attentions because the surface properties of laser-treated layer can be enhanced significantly [6–10]. As one of the most widely used Mg alloys AZ91D, previous studies show that wear and corrosion resistance of laser-melt AZ91D Mg alloy is improved significantly due to refined β-$Mg_{17}Al_{12}$ secondary phase and enriched alloying element concentration in the laser melt zone [9–12]. However, direct observation of laser irradiation on microstructural evolution of Mg alloy, in terms of phase transformation during rapid solidification, has not been fully reported, although it is well known that faster cooling rates produce finer microstructures. The formation mechanism of microstructure development is also open for debate. The objective of this study is to observe microstructural evolution of the β-$Mg_{17}Al_{12}$ secondary phase in AZ91D Mg alloy irradiated by a millisecond pulse Nd:YAG laser. Special attention is paid to understand effect of rapid solidification on phase transformation and phase precipitation induced by laser surface melting.

2. Experimental procedures

The material studied was an as-cast AZ91D Mg alloy with the following chemical composition (wt.%): Al 8.97, Zn 0.78, Mn 0.31, Si 0.023, Cu 0.002, Ni 0.0005 and Mg balance. The specimens of dimensions of 20 mm by 30 mm by 3 mm were extracted from an ingot, ground with progressively finer SiC papers (180, 400, 800, 1200, 2400 and 4000 grit), cleaned with alcohol, and then irradiated under Ar gas protection with an Nd:YAG laser system (wavelength of 1064 nm) using the following parameters: power density 3.82×10^4 W/cm^2, scanning speed 10 mm/s, repetition rate 100 Hz, pulse duration 1.0 ms, and spot overlap 50%. The laser was operated in a near TEM_{00} mode, and the beam was defocused to 1 mm in diameter.

Microstructural features of as-received and laser-melted specimens were studied using a JEOL 5600 LV SEM and a JEOL 2010 TEM equipped with EDS. A selected area electron diffraction technique (SAD) in TEM was used to extract crystallographic information. The TEM thin foils were obtained first by polishing mechanically on one side of substrate until the samples were less than 30 μm thick. Disks of 3 mm diameter were punched from the foils for subsequent jet electro-polishing in a solution composed of an electrolyte of 750 ml methanol, 150 ml butoxyethanol, 16.74 g magnesium perchlorate, and 7.95 g lithium chloride at −20 °C and an applied voltage of 40 V. Finally, ion beam was used on a Gatan precision ion polishing

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system under conditions of 5.0 kV and an incident angle of 4–5°. An X-ray diffractometer (Philips PW1830 series) was used to identify phases in the as-received alloy and in the laser-melt zone.

3. Results

Fig. 1 shows typical microstructural evolution of AZ91D Mg alloy before and after laser treatment. In the as-received microstructure, β-$Mg_{17}Al_{12}$ secondary phase indicated lamellar and bulk growth from the grain boundaries into α-Mg grain interior, as shown in Fig. 1(a). After laser irradiation, the microstructure of AZ91D Mg alloy was much refined, as shown in Fig. 1(b) and (c). Typical cellular/dendrite structure was observed in the melt zone, and line-shape structure was also found. XRD analyses indicated that the solidification microstructure of laser melted AZ91D Mg alloy consisted of refined α-Mg matrix phase and β-$Mg_{17}Al_{12}$ secondary phase [11,12].

The solidification microstructure in the laser-melted zone was further investigated by TEM, as shown in Figs. 2–4. Fig. 2(a) and (b) shows the cellular/dendrite structures of β-$Mg_{17}Al_{12}$ secondary phase. Fig. 2(b) reveals that many individual precipitates were distributed randomly inside the matrix, and the sizes of these precipitates were in the range of 50–200 nm. SAD patterns show that these particles exhibit BCC crystal structure. Quantitative elementary compositions in the particles were analyzed by EDS, including 63.11 wt% Mg, 32.83 wt% Al, 3.48 wt% Zn and 0.68 wt% Mn. Therefore, these nano-scale precipitates were identified as β-$Mg_{17}Al_{12}$ secondary phase. It should be noted that various types of Mg–Al, Mg–Mn, and Mg–Ca precipitates are generated in sizes ranging from nanometers to microns in Mg alloys processed by different methods [13,14]. However, rapid solidification induced by laser treatment results in a more homogeneous distribution as well as a relatively more refined scale.

Fig. 3 shows that the typical orientation relationship (OR) between the individual nano-particle and the matrix. The OR between the plate-shape β-$Mg_{17}Al_{12}$ secondary phase and the α-Mg matrix was found to be Burgers OR ($(0001)\alpha \| (110)\beta$), as shown in Fig. 3(a). It has been known that Burgers OR was the predominant Burgers OR of β-$Mg_{17}Al_{12}$ precipitation in AZ91D Mg alloy [15–17]. Moire fringes were observed in the precipitates due to overlapping effect between the nano-crystal and α-Mg matrix [18]. The OR between the spherical-shaped β-$Mg_{17}Al_{12}$ secondary phase and the α-Mg matrix was found to be $(10\bar{1}2)_m//(113)_p$, as shown in Fig. 3(b).

A large amount of cluster-shaped precipitates were observed in the overlapped region of laser-melt AZ91D Mg alloy, as shown in Fig. 4. Fig. 4(a) shows cluster-shaped precipitates together with individual tiny precipitates in the matrix. The size of cluster-shaped particles was in the range of 200–400 nm, while the size of individual particles was in the range of 50–100 nm. Fig. 4(b) shows the morphology of one of these cluster-shaped precipitates, and it can be found that the nucleation and development of small particles above the existing particles. SAD pattern and EDS result confirmed that such cluster-shaped particles were β-$Mg_{17}Al_{12}$ precipitation. However, it should be noted that further effort needs to study the specific OR among each crystal and the OR between single nano-crystal and the matrix.

4. Discussion

Generally, solidification reaction sequence in AZ91D Mg alloy can be described qualitatively at initiation of solidification using projection of liquidus valleys for Mg-rich region in Mg–Al–Zn system [19,20], as shown in Fig. 5. The first solid to form would be α-Mg matrix due to combined effect of thermodynamics and kinetics [21,22]. On further cooling, depending on prevalent solidification condition, liquid composition in enriched Al region, such as grain boundary where supersaturated α-Mg region with high Al concentration [15–17], could intersect the line of twofold saturation between α-Mg phase and β-$Mg_{17}Al_{12}$ secondary phase. At this stage (represented by point P and Q, respectively in Fig. 5), the solubility limit of primary solidification phase, α-Mg matrix, would be exceeded, and secondary solidification constituents $\alpha + \beta$ would

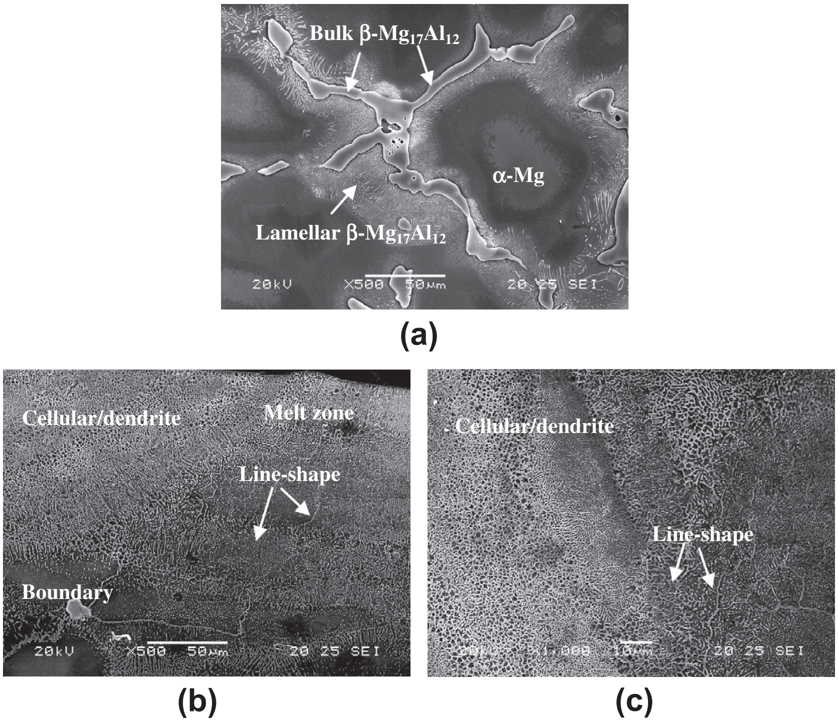

Fig. 1. Typical microstructure of AZ91D Mg alloy before and after laser treatment: (a) as-received microstructure, showing bulk and lamellar β-$Mg_{17}Al_{12}$ phase distributed in the α-Mg matrix; (b) laser-melt microstructure, showing longitude view of the melted zone and (c) laser-melt microstructure, showing cross-section view of the melt zone.