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# Boiling effect in crater development on magnesium surface induced by laser melting



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#### ABSTRACT

This paper demonstrated that micron-craters could be fabricated on both magnesium and Mg alloy surfaces by nanosecond pulsed laser processing. Experimental results revealed that the craters with broad distribution of dimension occurred after laser melting. Morphological difference at the irradiated surface between Mg and Mg alloy indicated that thermal properties, alloying elements and microstructure of irradiated materials were key factors responsible for crater formation. It was proposed that the craters were formed by combined effect of explosive volume boiling and generation of cavitation bubbles during laser melting. Thermal effect of nanosecond pulsed laser processing on Mg and Mg alloy as well as progressive formation of laser-induced crater was further discussed. Such phenomenon extended potential applications of Mg materials, which offered the potential for developing new types of Mg-based biomedical devices.

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#### 1. Introduction

Thermal effect in laser processing is responsible for precision and quality of produced structures on materials. In the past decades, compared to conventional continuous wave laser, pulsed lasers have been developed rapidly due to peak power performances [1–8]. Nanosecond pulsed laser processing with high efficiency and productivity has been considered as promising method in manufacturing industries [2,3].

When a short-pulse laser beam interacts with metals, thermalization in the electron subsystem is very fast, and electrons are heated to high temperature [1,2,5–10]. Correspondingly, surface temperature of irradiated materials increases rapidly. Referring to governing equation in heat transfer and fluid flow model of laser–material interaction [1, 9–11], it is assumed that thermal conductivity for the lattice subsystem is neglected, and electrons can be heated to very high transient temperature. According to the most used models including one-dimensional two-temperature-component model for heat transport inside the metal [2–5] and three-dimensional heat balance equations for temperature distribution as well as threshold energy density calculation [9–11], the main source of energy losses during nanosecond pulse duration is heat diffusion into the irradiated substrate. Therefore, thermal diffusivity of materials is a key factor responsible for melting and solidification

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processes. Various structures can be produced at the surface for wide applications.

Magnesium and Mg alloy have been increasingly used in the industries and biomaterial fields due to low density, high specific strength and biodegradability [12–14]. Unfortunately, inferior surface performance is a major factor that limits their actual applications [12,13,15]. Previous researchers show significant enhancement of surface properties including wear and corrosion resistance on laser-irradiated Mg alloy, mainly because laser can produce surface layers with fine microstructure that reduce the size of galvanic couples and expand solid solution range of alloying elements [11,15–18].

In this paper, we firstly report the capability of nanosecond pulsed laser to achieve local boiling phenomenon and micro-sized crater formation at Mg surface without chemical composition change. The laser used has a top-hat beam profiler with scanning mode, which is suitable for large area melting due to high power density and uniform energy distribution. We demonstrate how thermal effect occur over a large area of irradiated surface and discuss the influence of materials with laser processing on morphology as well as dimension of the crater. We conclude by explaining the mechanism of crater formation during laser–Mg interaction, and indicate how it applies for potential applications.

#### 2. Experimental procedures

The materials studied were as-cast Mg and 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 dimension of 30 mm by 30 mm by 3 mm were extracted from the ingot, ground with progressively finer SiC paper (180, 400, 800, 1200, 2400 and 4000 grit) to minimize the effect of incidence angle, and cleaned with alcohol.

A nanosecond pulsed Nd:YAG laser (ROFIN DQ  $\times$  50 S, wavelength 1064 nm, pulse duration 43 ns, repetition rate 12 000 Hz) was used in this study. The average power density was 2.15  $\times$  10<sup>8</sup> W/cm<sup>2</sup> (heat flux 1.85 J/cm<sup>2</sup> per pulse). The laser beam was delivered normal to the specimen surface through an optical fiber and galvanometer scanner. The square beam spot size was focused as 0.6 mm side length at the sample surface. The laser scanning speed was varied as 100 mm/s, 500 mm/s and 1000 mm/s to study the effect of laser energy on surface evolution of irradiated materials, and the corresponding laser pulse number for single spot is 72, 7.2, 3.6, respectively. The irradiated area was 10  $\times$  10 mm<sup>2</sup> in square using hatched scanning mode with 50% overlapping in the program. When laser was turned on, the specimen was placed in a well-sealed chamber using 2.0 bar Ar (2.0  $\times$  10<sup>5</sup> Pa) gas flow to minimize oxidation, as shown in Fig. 1.

After laser irradiation, surface topography of irradiated area was measured using Talysurf stylus profilometer, and microstructural features were examined by Scanning Electron Microscope (JEOL 5600 LV) equipped with Energy Dispersive Spectroscopy. The EDS measurements provided information on the chemical composition.

#### 3. Results

Fig. 2 displays effect of nanosecond pulsed Nd:YAG laser melting on Mg and Mg alloy surfaces. Various craters and their development occur at both surfaces. Diameter evolution of the craters with the decreasing laser scanning speed is in the range of  $10-50 \,\mu\text{m}$  for Mg alloy and  $10-25 \,\mu\text{m}$  for Mg, respectively. It also shows that typical shape of the crater is a combination of conical profile with ripples at the wall of conical crater. Tiny pores can be found at the wall of the crater on Mg alloy surface at a slow scanning speed. Moreover, molten materials surrounding the craters were observed at the surface, and it increased with the decreasing laser scanning speed significantly. It has been suggested that micron-sized droplets are resulted from hydrodynamic instability of the molten liquid layer during laser melting [10], and the molten mass is pushed radially outward from the irradiated area. This is in good agreement with our results, as shown in high magnification figures of the craters in Fig. 2.

Surface topography of the craters was further investigated. The diameter of Mg craters is 10–25  $\mu$ m and the diameter of Mg alloy craters is 10–50  $\mu$ m, which is the same as SEM results in Fig. 2. Depth range of the craters was found to be 10–150  $\mu$ m for Mg alloy and 5–100  $\mu$ m for Mg, respectively. The results of surface topography measurement reveal that Mg alloy surface melt further than that of Mg irradiated by incident laser light.

Cross-sectional views of laser-irradiated surface were also examined. Fig. 3(a) presents that melt depth is around 20  $\mu$ m for Mg alloy at fast scanning speed as 1000 mm/s, which is much shallower than 150  $\mu$ m

#### Laser beam through optical fiber



Fig. 1. Schematic diagram of experimental setup.

induced by millisecond pulse Nd:YAG laser according to previous study [11,18,19]. Few cellular/dendrite microstructures were found in both the melt layers. Fig. 3(b) shows that the molten materials can be seen clearly inside the crater, and Fig. 3(c) reveals big pores at the wall of the crater. Fig. 3(d) observes a large amount of melt drop on Mg at slow scanning speed as 100 mm/s, and no obvious phase change as well as pores was found in the microstructure. Melt depth for Mg and Mg alloy in Fig. 3(c) and (d) is around 100 µm.

Quantitative analyses of elementary compositions based on EDS found that Al content in all melted layers of Mg alloy was in the range of 10.2–11.1 wt.%, which was slightly higher than the average value of 9.0 wt.% in the untreated substrate. This was due to relatively more Mg element vaporization during laser irradiation [11,18,19]. According to experimental work, the combined effect of various craters and less solidification microstructure as well as Al concentration in the melt layer resulted in no improvement of surface properties of AZ91D alloy following nanosecond pulse Nd:YAG laser irradiation. In addition, no change of chemical composition was observed for Mg samples.

#### 4. Discussions

#### 4.1. Thermal effect

At nanosecond pulse duration, excited electrons created by incident laser radiation transfer energy to phonons during electron–phonon relaxation, and the energy will be redistributed through lattice vibrations of metal. Consequently, heat is conducted into the irradiated substrate [1, 2,5]. As mentioned in the introduction, thermal diffusivity of the material is crucial for producing structures. Thermal diffusivity of Mg and AZ91D alloy is calculated as  $8.468 \times 10^{-5}$  m<sup>2</sup>/s and  $2.894 \times 10^{-5}$  m<sup>2</sup>/s, respectively [12,13], while melting point of Mg and AZ91D alloy is 650 °C and 595 °C [12,13], respectively. Thereby, more molten materials occur at the irradiated surface of AZ91D alloy compared to that of Mg under the same laser fluence, as shown in Fig. 2.

Temperature distribution curve for single laser pulse can be calculated from transient heat balance equation based on the heat flow model [9–11]. Results show that surface temperature of irradiated Mg and AZ91D alloy increases rapidly from room temperature to peak temperature near to 4000 °C within pulse duration of 43 ns. Such rapid heating will lead to explosive boiling because liquid Mg can be heated above its normal boiling temperature (boiling point of Mg and AZ91D alloy is 1091 °C and 1107 °C [12,13], respectively) and become highly superheated. According to the foundation on explosive boiling established by Martynyuk and Kelly [20,21], superheated liquid metal experiences large density fluctuations, and these fluctuations can generate vapor bubbles in the entire superheated liquid layer. Once bubbles of size exceed critical radius, they undergo rapid transition into a mixture of vapor and liquid droplets. Such process is termed as explosive boiling.

Threshold energy density for explosive boiling on Mg and AZ91D alloy can also be estimated from the heat flow model [11,22,23]. Critical temperature of Mg was assumed at 5000 K as average value for common metals due to lack of available data. Based on the value for optical and thermal properties of Mg and AZ91D alloy in the literature [12,13,24], the calculated threshold value on both surfaces would be  $2.41 \times 10^8$  W/cm<sup>2</sup> and  $1.48 \times 10^8$  W/cm<sup>2</sup>, respectively. Due to the presence of a well-sealed chamber with 2.0 bar Ar gas flow, plasma shielding effect decreases significantly compared to laser radiation in atmosphere environment [22,25]. Therefore, current average power density of  $2.15 \times 10^8$  W/cm<sup>2</sup> is sufficient to induce explosive boiling.

#### 4.2. Crater formation

The development of crater formation is illustrated in Fig. 4. During nanosecond pulsed laser irradiation, incident light at high fluence created a large population of heat at the surface of materials. The conducted

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