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Surface structure formation in WE54 Mg alloy subjected to ultrafast laser texturing

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Pico- and femtosecond laser surface structuring of WE54 Mg alloy was investigated in this paper. In particular, the effective response of rare earth elements during laser structuring and the surface structure formation mechanisms were studied. A 10 W picosecond laser, having a wavelength of 1064 nm, and a 1.5 W femtosecond laser, having a wavelength of 795 nm, were used to irradiate WE54 surface to change the surface morphology. A number of pulses varying from 100 to 1500 were applied to create a crater at single and multiple points on the WE54 alloy surface in an air medium. Taylor Hobson profilometer and energy dispersive x-ray spectroscopy (EDS) techniques were employed in order to analyze the modification in surface morphology and chemical composition, respectively. A crater depth around 1.1 μm was measured by the profilometer. It was observed that a compound of yttrium and neodymium was formed during the process of rapid thermalization, which is investigated by x-ray diffraction. The surface portion containing yttrium was converted into a white crystal-like structure as observed by optical and scanning electron microscope (SEM). The white color compound of rare earth element is turned into gray, then dark, and is finally ablated from the surface with increasing scanning number and number of laser pulses. The change in color represents the change in optical properties of WE54. SEM analysis also revealed that no cracks were present on the laser structured surface; hence, stress effect on the surface can be neglected. Explosive vaporization of the substrate was observed in pico- and femtosecond laser structuring. The investigation on ultrafast laser structuring suggests that the surface properties created by laser structuring of WE54 can be controlled if proper laser processing is applied. © 2016 Laser Institute of America. [<http://dx.doi.org/10.2351/1.4944447>]

Key words: laser texturing, WE54 alloy, surface morphology, picosecond and femtosecond laser, pulse number

I. INTRODUCTION

In the field of laser material processing, the laser treatment technology becomes more advanced because of the development of ultrafast lasers. Ultrafast lasers are capable to fabricate structures on material surface from simple to complex micro/nano 3D structures by the use of optimum value of the parameters such as laser fluence, number of pulses, wavelength, scanning speed, depth of focus, beam polarization, and operating medium. Ultrafast laser energy is absorbed by the conduction band electrons of metals and energy is transferred to the material lattice. Ions or lattice will be rapidly heated to very high temperature because electrons are driven to a very high temperature. The material will be vaporizing immediately after the short interaction with high temperature phenomena.¹ The use of very short laser pulses strongly diminished the plasma shielding. Therefore, before the expansion of plasma plume, the short laser pulses allow strong material excitation and are capable of precise ablation.² The small amount of heat produced by each pulse

has plenty of time to disappear before the next pulse arrives, thus minimizes the heat affected zone (HAZ). Ultrafast laser has unique features which can be quoted as “small pulses of intense laser energy.” Nonstationary, nonlinear optical, and nonequilibrium processes have a vital role during femtosecond and picosecond laser processing.

Ultrafast lasers have provided significant contributions in the various fields such as surface cleaning, microstructuring, thin film deposition, ablation, cutting, drilling, etc. Surface functionalization of wide range of materials is achieved by modifying tribological, chemical, mechanical, wetting, optical, and electrical properties of materials by ultrafast lasers. The unique micro/nanosurface structures produced by femtosecond laser have open new potential areas for application of material in the different fields. Ultrafast lasers, with pulses ranging from picosecond to femtosecond, have the ability to ablate any material with reduced side effects. Ultrafast lasers are adjustable with gas, liquid, and vacuum medium, and these medium can be chosen according to the situation to avoid the influence of the surrounding environment. The

investigation on ultrafast laser material interaction has been done in different metals most commonly aluminum, copper, nickel, stainless steel, gold, and titanium. Particularly on metals, study on ablation threshold, effect of pulses, ablation efficiency and random/periodic/groove/columnar structures have been reported. Semerok *et al.* have studied the ablation efficiency of pure metals. They mentioned that the ablation rate increases with the pulse duration if the pulse duration is larger than the relaxation time.³

Ultrafast laser structures can be categorized as laser irradiated structures and self-organized structures.⁴ Laser irradiated structures consist of micro/nano structures where, nanostructures have random and periodic patterns whereas microstructures have undulating grooves, columnar, hole, and maze structures. Nayak and Gupta have reported self-organized conical micro/nanostructure on Ti sheets by direct laser texturing.⁵ They mentioned that the conical microstructure and the intercone distance was highly dependent on the laser fluence. Optical, hydrodynamic, and plasmonic effect could be the key factors to create the conical surface structure. Laser induced periodic surface structures (LIPSS) are mainly two types: low spatial frequency LIPSS (LSFL) and high spatial frequency LIPSS (HSFL). The LSFL periodic surface structures, which are perpendicular to the polarization of the incident laser beam, have a spatial period close to the laser wavelength. The HSFL periodic surface structures, which are either perpendicular or parallel to the polarization of the beam, have a spatial period much smaller than the laser wavelength. The formation of periodic structures depends upon the spatial energy distribution. The interference between the incident laser beam and scattered optical wave at the surface of metals leads to the periodic distribution.^{6,7} Metal colorization in different metal due to the femtosecond laser structuring has been reported.^{8,9} Vorobyev and Guo reported changing of highly reflective metals to highly absorptive due to the color change by laser irradiation.⁹

In the case of magnesium alloys, numerous researches have been reported on laser surface melting^{10,11} whereas very little research has been reported recently in ultrafast laser structuring.¹² Guan *et al.* first reported the nano and microripples formation on femtosecond laser irradiation on AZ31B Mg alloy.⁸ They mentioned that the formation of microripples on AZ31 was due to the combined effect of surface roughness and near field interference. The formation of the nanoripples on the AZ31 surface was due to the interference between incident light and surface plasmons. Ultrafast laser texturing on WE54 Mg alloy has not been reported. Mg and its alloys are very useful because of their properties such as low density, high specific strength, stiffness good castability, and damping capacity. Their application fields include automobile, aerospace, microelectronics, and they are also potential biomaterials. However, high reactivity of Mg alloys makes them inferior in different application fields. Laser surface texturing can be one of the techniques to widen Mg alloys application. The structured Mg alloys can be used in structural application. In this paper, the surface structure formation by single scanning picosecond and femtosecond laser has been reported. The random and periodic structures with change in the concentration of alloying element were investigated. X-ray

diffraction analysis also suggested the change in phases after laser surface texturing. Threshold pulse energy was investigated by using crater formation parameters.

II. EXPERIMENTAL DETAILS

Extruded WE54 magnesium alloy with a composition of 5.5% Y, 1.7% Nd, 0.4% Zr, 0.01% Mn, and 1.2% HRE by weight percentage was used for ultrafast laser texturing. The specimen having 3 cm diameter was cut into 3 mm thickness. Prior to laser surface texturing, the specimens were grounded from 320 to 2400 grit SiC paper followed by ethanol cleaning and air flow drying.

Laser irradiation was carried out by using a 1.5 W QUANTRONIX Integra-C femtosecond laser and 10 W DUETTO time bandwidth picosecond laser. 100, 500, 1000, and 1500 fs laser pulses were used progressively to create a single and multiple craters in WE54 Mg alloy. The threshold pulse energy was calculated by using a craters diameter and a pulse energy relationship. The calculated threshold fluence was used to scan the WE54 surface. Single scan was performed on WE54 Mg alloy surface. A femtosecond laser parameters was used to scan a (10×10) mm² surface area in open atmosphere environment with the following parameters: 795 nm wavelength, pulse duration 130 fs, repetition rate 1 kHz, scanning speed 200 mm/s, focal length 163 mm, and hatching density 0.01 mm. The focus was maintained by using a camera. Similar experimental procedure was applied for picosecond laser texturing. A (10×10) mm² surface area of WE54 alloy was textured by the picosecond laser under the following parameters: 1064 nm wavelength, pulse energy 50.2 μ J, repetition rate 50 kHz, pulse duration 10 ps, focal length 100 mm, scanning speed 200 mm/s, and hatching density 0.01 mm. The picosecond laser experiment was performed in a closed chamber with a vacuum machine provided to remove dust. Both femtosecond and picosecond laser surface irradiation were performed at focused beam condition. After laser surface irradiation, the surface was cleaned using ethanol. The single and multiple craters were created at different pulse energy and number of pulses to obtain a threshold value. Figure 1 represents the experimental setup of the laser.

Laser textured structures were examined by JEOL 5600 LV Scanning electron microscope (SEM) and optical microscope (OM). Electron dispersive spectroscopy (EDS) was used to investigate the concentration of alloying elements on the laser textured surface. PANALYTICAL x-ray diffraction system with CuK α was used to examine the phases of laser textured surface. Taylor Hobson Profilometer was used to observe the surface morphology. Cross section profile of laser textured surface, surface roughness, and particle size distribution was obtained from the profilometry.

III. RESULTS AND DISCUSSION

A. Crater formation

The single and multiple row craters were created by the femtosecond laser to understand the relation between the crater depth and laser fluence. Threshold fluence was investigated by single shot crater formation. Different laser pulse

FIG. 1. Schematic diagram of laser experiment setup used in this study.

energies were applied to ablate the material. The threshold energy was analytically calculated by using the following equation:¹³

$$D^2 = 2W_0^2 \ln(E/E_{th}), \quad (1)$$

where D is the diameter of the crater which can be measured by profilometer. W_0 is the radius of beam measured at $(1/e^2)$. E is the applied laser energy during experiment. E_{th} is the threshold energy, which is the energy required to form the crater. Laser pulses from 100 to 1500 were applied to create the single and multiple craters. Figure 2(a) represents the effect of laser pulses at $28 \mu\text{J}$. The surface did not ablate in that case. The crater shown in Fig. 2(b) was ablated by the pulse energy $84 \mu\text{J}$ and 500 pulses. The deposition of ablated material was seen around the crater surface. The deposited material around the surface was highly enriched with MgO. Figure 2(c) shows the effect of number of pulses on the surface of WE54. When 1500 pulses were applied to the surface

of WE54, the effects were seen around the crater as circularly, which is shown in Fig. 2(c). Stress effect was seen with the increasing number of pulses. It represents that the increasing number of pulses affects the surrounding area of metal surface. The ablation depth can be measured by the surface profilometer or it can be calculated by using the following equation:¹⁴

$$d = 1/\alpha_{eff} \ln(E/E_{th}). \quad (2)$$

This equation is valid for single photon absorption process. α_{eff} represents the effective absorption coefficient. The threshold pulse energy and threshold fluence for femtosecond laser and picosecond laser used in this study were ($84 \mu\text{J}$, 11.39 J/cm^2) and ($50.2 \mu\text{J}$, 13.21 J/cm^2), respectively, if the surface is in a focused position. The crater depth at the threshold fluence is nearly $1.1 \mu\text{m}$. From the similar procedure, the crater depth of picosecond laser ablated surface was found to be approximately $2 \mu\text{m}$. Multiple craters were created at the threshold energy and the variation in depth was analyzed. Figure 2(d) shows the multiple craters at the threshold pulse energy, which demonstrates the consistent ablation of craters.

B. Surface structuring with picosecond/femtosecond laser

Single pass scan was performed on WE54 Mg alloy surface. Figure 3(a) represents the microstructuring of WE54 alloy by picosecond single passes line scanning. A periodic structure was observed at the microscale with a gray appearance when viewed from the naked eye. The surface was seen to be an arrangement of crests and troughs. The enlarged view, which represents the white portion of Fig. 3(a), was

FIG. 2. (a) Laser shot at energy lower than threshold value, (b) crater formation at threshold value ($84 \mu\text{J}$, 11.39 J/cm^2), (c) crater at 1500 pulses and threshold energy showing stress effect, and (d) multiple craters at threshold energy.

FIG. 3. (a) Laser texturing by picosecond laser at threshold pulse energy $50.2 \mu\text{J}$, (b) femtosecond laser texturing of WE54 at threshold pulse energy $84 \mu\text{J}$, both are single scan.

enriched with Y element. The element was detected by the EDS analysis. The total elements that have been identified on the laser textured surface were Y, Nd, and Mg. These elements can form different stages of $(\text{Mg}_a\text{Y}_b, \text{Nd}_c)$ phase, where a , b , and c are variables. It has been reported that evaporation of Mg occurs due to its low melting and boiling points compared to other elements in Mg alloy during laser surface melting.¹⁰ In LST with picosecond and femtosecond laser, the change in element concentration has been observed. The concentration of Y was increased from 5.5% to 11% after texturing with picosecond laser whereas the concentration of Nd was increased by only 0.5%. The Mg element in WE54 was highly affected by the high temperature which leads to quicker vaporization compared to the other elements. It indicates that the thermodynamic properties of material such as heat capacity, heat of vaporization, heat of fusion, and optical properties play an important role during ultrafast laser irradiation. Further, scanning number was increased to five to check the change in element concentration. After five times line scanning, the surface was changed into a dark color when viewed from the naked eye. This is due to the light trapping properties of metal.⁵ The active interaction of rare earth elements has been observed. The white portion was minimized, reducing the concentration of Y element with increasing number of scanning and pulses.

Random microstructure was observed when surface was textured by the femtosecond laser at the threshold pulse

energy of $84 \mu\text{J}$. The surface consists of buds like structure when viewed in less magnified mode as shown in Fig. 3(b). Gray and slightly yellowish colors were seen when the surface was viewed from the different angles. Guan *et al.* reported the term iridescent effect, which occurs because of laser irradiated nanoripples acts as the diffraction grating and eventually induces color on the surface.⁸ The gray color surface indicates that the optical properties of surface have changed after texturing. Similarly, the change in concentration of Y element from 5.5% to 8% was observed in femtosecond laser texturing. Selective vaporization occurred during laser material interaction. The Mg element vaporized directly whereas yttrium element was gradually ablated with increasing number of laser scanning. The micro/nanostructure with colored appearance can be used in photovoltaics, artificial colorization, and bio-

FIG. 4. (a) and (b) represent surface morphology created by femtosecond and picosecond laser, respectively. (c) Cross section profile of femtosecond laser, scale $3 \mu\text{m}$ and (d) cross section picosecond laser textured surface, scale $10 \mu\text{m}$.

FIG. 5. Size distribution of irradiated structure at threshold pulse energy: (a) picosecond laser and (b) femtosecond laser textured surface.

optical devices. Single and multicolors surface can be obtained by use of proper laser parameters.¹⁵ The periodic structure formed from the laser texturing can act as the diffraction grating. The initial single scanning observation suggested the change in optical properties of WE54 alloy.

C. Surface morphology

Profilometer was used for the quantitative analysis of the textured surface. An irregular rough topography was observed in single scanning mode. A periodic and random (buds like) structures were found in the microscale. Orientation of microstructure term as a microripples could be parallel to the laser polarization direction. Figures 4(a) and 4(b) show the 3D view of laser textured surface by picosecond and femtosecond laser, respectively. The height of the microscale structure ranged from 0.3 to 1 μm in case of the femtosecond laser textured surfaces, whereas the height variation of peaks and valleys ranged from the 0.3 to 2 μm in picosecond laser textured surfaces. The variation in height of surface is shown in the surface profile of Figs. 4(c) and 4(d), respectively.

The average three dimensional surface roughness (S_a) of femtosecond and picosecond laser textured surface were 0.535 ± 0.034 and 0.858 ± 0.145 μm, respectively. The surface roughness (S_a) of as-received WE54 used for laser texturing was 37.82 ± 2.182 nm. Figure 5 shows the cluster distribution on the picosecond and femtosecond laser

textured surface. The structure is highly depending upon the wavelength and laser fluence. The number of peaks per mm² was higher in picosecond laser textured surface as compared to femtosecond laser textured surface at threshold fluence. The cluster distribution reached up to 1800 in picosecond laser, whereas the maximum number in femtosecond laser textured surface was 800 as shown in Figs. 5(a) and 5(b). Large particles were found on picosecond laser textured surface at threshold pulse energy.

D. X-ray diffraction analysis

In as-received WE54 alloy, the grain boundaries were enriched in Y and Nd elements as observed by electron

FIG. 6. X-ray diffraction pattern of WE54 Mg alloy, (a) as-received WE54, (b) picosecond laser textured surface, and (c) femtosecond laser textured surface, showing different phases.

dispersive spectroscopy (EDS). Generally, the compounds are formed from the precipitates of Nd and Y along the grain boundaries. The microstructure of WE54 consists of α -Mg matrix and secondary phase as Y and Nd precipitates. Figure 6 shows the x-ray diffraction graphs of as-received and laser textured surface of WE54 alloy. In Fig. 6, A represents the α -Mg phase, B represents Mg_{24}Y_5 and C represents Mg_4Nd_5 phase which was analysed by Panalytical X'Pert HIGHSCORE Plus software and compared with the standard data. X-ray diffraction analysis showed that the intensity of peaks was decreased after laser surface texturing. The elements present in the WE54 also play a vital role during laser texturing. The intensity ratio of Mg- α phase was highly decreased to 0.075 as compared to as received WE54 alloy. The Mg- α phase was eliminated from the some portion of surface which was shown in XRD pattern of femtosecond and picosecond textured surface.

IV. CONCLUSION

The single scanning of WE54 surface by picosecond and femtosecond laser created the periodic and random (buds like) structures in a microscale, respectively. The maximum depth of crater at threshold pulse energy was observed to be nearly $1.1\text{ }\mu\text{m}$ in femtosecond laser texturing, whereas $2\text{ }\mu\text{m}$ in picosecond laser texturing. Laser changes the optical reflectance properties of metal by changing mirror like surface to gray color. The surface structured mechanism can be considered as the rapid vaporization of elements of WE54 because the concentration of Mg was decreased. The formation of structures at microlevel is possibly because of the interference between the surface plasmons and laser light. In this study, the structure formation mainly includes the effect of optical properties, vaporization of element, and plasmonic effect. The multiscanning at threshold fluence is necessary to understand the nanoripples formation and multiple coloration on surface as suggested by the literature.

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Indira Khadka received her Bachelor's degree in Mechanical Engineering from Tribhuvan University, Nepal, in 2011. She worked as a design engineer for modeling of honda automotive parts in E & T Pvt Ltd. She started her Ph.D. study in NTU since August 2012. Her research interests include laser material processing, material characterization and laser additive manufacturing. Currently, she is working on laser surface processing of rare earth magnesium alloys.

Dr. Sylvie Castagne is an Assistant Professor in the School of Mechanical and Aerospace Engineering since July 2008. She received her Ph.D. in Applied Sciences from the University of Liege, Belgium. She also graduated with a M.Sc. in Applied Physics Engineering and a DEA in Applied Sciences from the same University, which encompass a broad range of physics, materials, and engineering areas. Before joining NTU, Sylvie worked in Singapore as a Senior Research Engineer at SIMTech (Singapore Institute of Manufacturing Technology) and as an Assistant Professor at the University of New South Wales Asia.

Dr. Wang Zhongke is a research scientist in SIMTech since November in 2007. He specializes in laser micro- and nanoprocessing. Before joining SIMTech, he was with AIST as a JSPS Fellow in Japan, ETH-Zurich as a Simon Fellow in Switzerland and REKIN as a contract researcher in Japan, from 2001 to 2007. He studied in three different universities in China. He has published over 80 scientific papers (including conference proceedings), two book chapters (in USA and in UK, respectively), and a number of patents including in China, Japan, Singapore, and international.

Dr. Zheng gained his B.Eng. in mechanical engineering from Tsinghua University Beijing in 1985 and Ph.D. in laser materials processing from Imperial College London in 1990. He has gained working experience as an engineer, academic, and scientist in Singapore. He has published over 80 journal and conference papers in laser processing and has filed eight patents and technology disclosures.