Micro and sub-micron surface structuring of AZ31 by laser re-melting and dimpling

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Abstract

In this work, the use of ns-pulsed fibre laser for surface structuring of AZ31 Mg alloy is investigated. Surface re-melting was employed to change surface morphology, especially in terms of surface roughness. Dimpling was employed to investigate microdrilling of the material with controlled geometry. With surface remelting mono-directional and homogeneous surfaces were obtained with $F_{1} < 500$ J/cm$^2$. Above 500 J/cm$^2$ particle generation was observed, which induced sub-micron structure growth with nano-fibrous features. Moreover, surface roughness could be controlled below the initial value and much higher. With dimpling, transformation from gentle to strong ablation was observed at $F=10.3$ J/cm$^2$. XRD analysis was employed to link oxide growth to the surface morphology. Tensile tests were carried out to assess the damage on the mechanical properties after surface structuring.

Keywords: Surface roughness, nano fibrils, MgO

1. Introduction

Laser surface structuring can improve key properties of components, such as tribological behaviour [1], wettability [2], biological performance [3], optical reflectivity [4], and adhesion stability [5]. Depending on the used laser type and machining strategy surface patterning in micrometric to nanometre scale is achievable [6]. For different surface functions the control over micro and sub-micron surface patterns or their combinations becomes highly appealing. The common approach for laser surface patterning is based on direct
writing, which makes use of scanning heads. The machining resolution is comparable to the beam size and is generally micrometric. The sub-micron and nano structures obtained in direct writing are the consequence of non-linear interaction occurring on the material surface. Most commonly the nano structures are obtained with the use of ultra fast lasers, which generate ripple formation [7]. This mechanism is based on a polarized beam that interferes with its reflected portion on the material surface and generates periodic sub-micron surface structures. More recently the use of interfering beams has also been proposed as a solution for nanometric surface patterns [8].

Magnesium and its alloys are highly appealing materials for low weight structures in automotive and aerospace sectors mainly due to their low density and high specific strength [9]. The biocompatibility and biodegradability properties render Mg alloys also desirable for biomedical applications [10]. Moreover, its oxide (magnesia, MgO) is a structural ceramic, which is used a refractory material with high thermal and chemical stability [11]. Magnesia is also used in thin-film semi-conductors, in making crucibles where high corrosion resistance is required [12], and as catalyst support in reforming bioethanol for hydrogen production [13]. Laser surface treatments of Mg alloys have also received attention regarding mostly their corrosion properties [14-17]. The majority of the attention has been on surface melting with CW or long pulsed lasers to reduce the high corrosion rate of Mg alloys for their use in mechanical, aerospace and lightweight structure applications. In more recent works on Mg alloys, the modified optical properties with excimer [18] and ultrafast laser [19], controllable wetting behaviour with a ns-pulsed fibre laser [20], and laser shock peening for bone ingrowth for biomedical implants [21] have been proposed. Despite the numerous works in the literature, the processing strategies to achieve surface structures of different dimensional scale seem to be missing on Mg alloys. The structured Mg surfaces can be applied to many different application fields. The micro dimples can be used in biomedical implants for bone ingrowth and for drug delivery. Lowered surface roughness is for surface polishing of mechanical components [22]. The nano-fibrous structures can be exploited for their optical properties [23], the gecko effect seen in nano hairs [24] or as scaffold for tissue growth [25].
In this work, two different laser surface structuring methods are proposed to study micro and sub-micron structures obtainable with a ns-pulsed fibre laser. In the laser re-melting strategy, a superficial layer of material is melted and re-deposited through high speed thermal cycles, forming different surface structures. The change in surface morphology with surface re-melting is categorically analysed and processing conditions generating particle growth with nano fibrous structures are identified. Moreover the dependence of surface roughness to the laser process parameters is identified. In laser dimpling, microdrilling conditions for controlled hole geometry are studied and different ablation conditions identified. Finally the surface integrity is analysed through chemical analyses, cross-sections and tensile tests.

2. Materials and Methods

2.1. Materials

Cold rolled 0.4mm-thick AZ31 magnesium alloy sheets were used throughout the work. The alloy compounds are measured as 3.08 wt% Al, 0.98 wt% Zn and bal. Mg. The initial surface roughness was measured as $R_a=0.26\pm0.01\ \mu m$ and $R_z=1.77\pm0.04\ \mu m$.

2.2. Laser surface structuring

Laser surface structuring was realized by a nanosecond pulsed fiber laser source (IPG Photonics YLP 1/100/50/50). Laser source characteristics are summarized in Table 1. Beam manipulation was achieved by a scanner head (TSH 8310 by Sunny Technology, Beijing, China) equipped by an f-theta lens with 100 mm focal length.

2.2.1. Laser surface texturing by re-melting

Surface re-melting aims to change surface topography by applying fast heat cycles that minimize material loss due to vaporization. Re-molten areas manifest when processing with nanosecond pulsed lasers around the tails of the Gaussian energy distribution of the beam [26]. This indicates that lowering the intensity peak of the beam below the ablation threshold, it is possible to have a melting dominant process. In this condition, material removal would occur only due to vaporization from melt, hence would be limited. Accordingly, processing
was applied under defocused conditions to lower the high intensity of the pulsed laser beam. The beam diameter at a given position \((d(z))\) from the focal lens can be estimated by the following set of equations:

\[
d_0 = \frac{4M^2\lambda f}{\pi d_c}
\]

\[
d(z) = \sqrt{d_0^2 + h_f^2\theta^2}
\]

where \(d_0\) is the beam diameter at focal plane, \(M^2\) is the beam quality factor, \(f\) is the focal distance of the focusing lens, \(d_c\) is the collimated beam diameter, \(h_f\) is the focal height that is the relative distance to the focal plane and \(\theta\) is the divergence angle of the beam, with \(\theta = d_c/f\). With the present optical arrangement the laser beam diameter at focal plane is 39 µm. After initial experiments, suitable focal height value was found to be 2 mm above the surface. Thus, in surface re-melting the beam diameter \((d_c)\) was 124 µm on the material surface. In order to study the effect of different thermal cycles applied to the material, laser energy \((E)\), pulse frequency \((\text{PRR})\) and scan speed \((\nu)\) were varied. Moreover number of passes was varied along with the scan direction. Three different strategies were used, which varied the scan direction along with the scan number. The axis perpendicular to the rolling direction of the sheets was set as \(0^\circ\) and single passes were applied at this direction \((S=1)\). Two passes \((S=2)\) were applied at \(0^\circ\) and \(90^\circ\). Four passes \((S=4)\) were applied at \(0^\circ\), \(90^\circ\), \(45^\circ\) and \(135^\circ\). In all conditions pitch \((p)\), i.e. the distance between scanned lines, was kept constant at 10 µm. All experiments were carried out in ambient atmosphere.

Surface morphology of the experimental conditions was characterized via SEM (EVO-50, from Carl Zeiss, Oberkochen, Germany). Surface roughness was measured with an optical 3D profilometer based on focus variation microscopy (InfiniteFocus from Alicona Imaging GmbH, Graz, Austria). The acquisition area was 1.3 mm x 0.2 mm. Five lines along the long axis were taken, on which average surface roughness \((R_a)\) and average distance between the highest peak and lowest valley \((R_z)\) were calculated. Experimental conditions and measured variable in re-melting study are summarized in Table 3.
2.2.2. **Laser surface structuring by dimpling**

Laser dimpling is based on ablation, hence material removal. The main parameters for designing surface pattern are dimple diameter (D), depth (h) and pitch (p). While pitch is a parameter controlled by the beam manipulation system, diameter and depth depend on the laser processing parameters. In order to evaluate the processing conditions of AZ31 Mg alloy, dimpling was employed with percussion drilling. Particularly, a ramped train of pulses was used. The ramped emission was controlled via the modulation duration ($t_{\text{mod}}$), which is the gate signal given to the laser source for emission. If the modulation duration is set below the initial transitory of the laser emission, a burst of ramped emission is obtained. This control scheme is discussed in detail in another work [27]. In particular, within this study the modulation duration was varied between 35 and 80 µs, with pumping current (PI) at 100% and pulse repetition rate at 50 kHz. The emission profiles belonging to intermediate values of modulation duration are shown in Figure 1. In this configuration the energy of the highest pulse in the burst train was estimated as 313 µJ for $t_{\text{mod}}$=80 µs and less than 40 µJ for $t_{\text{mod}}$=35 µs. Dimpling was applied on AZ31 sheets with fixed pitch at 100 µm. Modulation duration was varied between 35 and 80 µs with 5 µs steps of increase. Focal position was kept on material surface. All experiments were carried out in ambient atmosphere. Dimple depth and diameter were measured with a focus variation microscopy system (Alciona InfiniteFocus). Experimental conditions and measured variables in dimpling study are summarized in Table 3.

2.3. **Analysis of surface oxidation and mechanical properties**

Candidate surfaces were defined from the two surface structuring strategies for further analysis. Five surfaces for re-melting with $R_s$=0.22-4.33 µm were chosen and characterized. The effect of laser treatment over the surface chemistry was evaluated through XRD analysis. SEM images of the surface cross sections were made to estimate the layer thickness.

In order to test surface integrity tensile tests were conducted. One representative condition from re-melting and dimpling conditions was tested and compared to non-structured AZ31 sheet. Flat test specimens, with dog
bone geometry, were prepared and texturized on both faces. Specimens were 0.8 mm of width in grip section and 4 mm in reduced section with 50 mm total length. All conditions were replicated 3 times.

3. Results and discussion

3.1. Control over surface roughness by surface re-melting

Figure 2 reports the SEM images of the surfaces obtained with different experimental conditions. It can be seen that the obtained surfaces vary greatly in surface geometry within the experimental region. The diverse surfaces can be grouped in three main categories regarding their geometry:

1) Surfaces with mono-directional surface structures,
2) Homogenous surfaces,
3) Surfaces with particle growth.

The mono-directional surfaces are generated with low energy (E=0.10 mJ) levels and low number of passes (S=1-2). The surface structure follows the direction of rolling. The first pass is applied parallel to the rolling direction, this preferential growth can be linked to relief of the stress generated during the rolling process due to the thermal effect of the laser. Previous work on MgO growth in air also reports preferential sites for oxide growth were the intersections of grain boundaries with the AZ91 alloy surface, which is coherent with the present observation [28]. The homogenous surfaces require medium to high energy levels (E=0.2-0.25 mJ) and are observed with 2 or 4 passes. Surfaces with particle growth are visible especially when high repetition rate (PRR=70 kHz) and low scan speed (v=25 mm/s) are employed. An attempt to describe the presence of different surface structures can be carried out using line fluence ($F_l$) described as:

$$F_l = \frac{E \cdot PRR \cdot S}{v \cdot d_x}$$  \hspace{1cm} (3)

In Figure 3 the surface types in their occurrence order are plotted as a function of line fluence. It can be seen that mono-directional and homogeneous surfaces exist in an overlapped zones. The main distinction between the two is related to the energy level. On the other hand, the generation of particle growth on AZ31 surface can be observed above 500 J/cm². The generation of such particles can be associated to surface oxidation.
Above the critical linear fluence value the surface oxidation may give rise to the generation of complex surface structures.

Close observations of the surfaces reveal the generation of sub-micrometric surface structures especially above 500 J/cm², hence on surfaces with particle growth. The mono-directional surface shown in Figure 4a is characterized by microgranular particles, with diameters in the order of a few µm (F_l=258 J/cm²). The surface shown in Figure 4b is obtained with higher energetic conditions (F_l=645 J/cm²) and depicts the generation of microglobular structures partially covering the surface. The higher magnification image in Figure 4f confirms existence of a nanofibrous structure, hence a micro - sub-micron hierarchical surface structure. As the energetic conditions increase (F_l=903 J/cm²) surface is covered by fractal micro and sub-micron structures as seen in Figure 4g. The increasing magnifications seen in Figure 4h and 4i confirm the fractal structure and nanofibrous surface structure. This behaviour is connected to magnesium oxidation process at high temperature. As reported by Czerwinski [28], at high temperatures, magnesium oxidation starts with a linear growth and changes its kinetics to a non-linear trend. The results are the growth in different nucleation sites and generation of complex and porous structures, with a typical cauliflower shape. The laser treatment in air accelerates the oxidation process. Within the process, material temperature is increased very rapidly and vaporization is expected to occur in the particle growth region, which generates similar sub-micron surface structures.

In the experimental range the surface roughness could be changed between R_a=0.22 µm and 4.50 µm and R_z=1.30 µm - 22 µm. Regression equations were fitted for both R_a and R_z roughness parameters. Both the equations were found to be in the same mathematical form with different coefficients of the regressors, as expressed in Eq.(4) and Eq.(5).

\[
\ln(R_a \ [nm]) = 15.59 \cdot E [mj] - 1.745 \cdot \ln(E [mj]) + 0.0486 \cdot PRR[kHz] - 0.00667 \cdot v [mm/s] + 0.3330 \cdot S + 0.02158 \cdot \ln(E [mj]) \cdot PRR[kHz]
\]  
\text{(4)}

\[
\ln(R_z \ [nm]) = 17.86 \cdot E [mj] - 2.512 \cdot \ln(E [mj]) + 0.0538 \cdot PRR[kHz] - 0.00605 \cdot v [mm/s] + 0.3181 \cdot S + 0.02467 \cdot \ln(E [mj]) \cdot PRR[kHz]
\]  
\text{(5)}

Both of the regression equations fitted the data well as represented by the high \( R^2_{adj} \) and \( R^2_{pred} \) values. The equations depict that all the process parameters are significant and an interaction between the energy (E) and...
the pulse repetition rate parameters (PRR) exists. The plots of the fitted regression models are presented in Figure 5 and 6. The effect of energy balance is evident in the plotted graph. The highest roughness parameters lie on the highest (E=0.25 mJ, S=4, v=25 mm/s, PRR=70 kHz), whereas the lowest roughness is achieved with lowest (E=0.10 mJ, S=1, v=100 mm/s, PRR=70 kHz) energetic conditions.

3.2. Geometrical characteristics of the dimples

In Figure 7 the measured dimple depths (h) and diameters (D) are reported. The dimple diameter increase can be very well described by Liu’s model as [29]:

\[ D^2 = 2w_0^2 \ln \left( \frac{F_0}{F_{th}} \right) \]

(6)

where \( w_0 \) is the beam radius, \( F_0 \) is the ablation threshold and \( F_0 \) is the fluence that is calculated as:

\[ F_0 = \frac{2E}{\pi w_0^2} \]

(7)

The control parameter \( t_{mod} \) that regulates the energy content of the ramped pulse trains substitutes fluence in Eq.(6). Accordingly, the fitted non-linear regression equation becomes:

\[ D^2 = -8866 + 2620 \cdot ln(t_{mod}) \]

(8)

This fact confirms that the increase of \( t_{mod} \), increases the energy content of the highest peak, thus increases the fluence of the single pulse with highest energy. The dimple diameter is determined by the most energetic pulse, therefore the diameter increase follows the same trend with fluence increase when \( t_{mod} \) is employed. On the other hand the depth increase is very much linked to the whole pulse train. Two regions can be in accordance to the identified [30]:

i) Gentle ablation (\( t_{mod}<50 \) µs): Ablation is predominantly in form of vaporization and is accompanied by spallation. The generated dimples are shallow and depth variation is limited.

ii) Strong ablation (\( t_{mod}\geq50 \) µs): Ablation is in the form of phase explosion and melt expulsion. The dimple depth variation increases.

The energy of the strongest pulse was estimated as 61 µJ for \( t_{mod}=50 \) µs. Resultantly, the threshold fluence between gentle and strong ablation was calculated at \( F_0=10.3 \) J/cm². The change of ablation characteristics is
shown in Figure 8. The shallow dimples obtained in gentle ablation conditions show material build up around the hole entrance and uneven shape (Figure 8c). The example conditions show the increase in dross formation and surface oxidation in strong ablation conditions (Figure 8b).

3.3. Effect of laser surface structuring to surface chemistry and bulk material integrity

The chosen surface types for further analysis on surface and bulk material integrity are reported in Table 4. The surfaces were chosen to represent different surface roughness conditions in re-melting and a representative case in dimpling.

Figure 9 shows the cross sections of the structured surfaces with laser re-melting. The images registered in back scattered emission mode show the change in chemical composition as well. As the surface roughness increases the extent of the darker regions depicting oxidized zones increases. This is directly linked to the laser energy conditions, as the SEM images are also in the order of increasing energetic conditions. For the surfaces showing the highest Rₐ at 3.02 µm and 4.33 µm, the structured surface exhibits porous structure. Both of these surfaces are obtained with F_l > 500 J/cm², thus this confirms that the surfaces with particle growth are also likely to be porous. In Figure 10.a, the XRD signals belonging to the different surfaces types are shown around the most significant MgO peak around 2θ=43°. The signal intensity clearly increases moving towards higher surface roughness. In Figure 10.b, the peak intensity of the XRD data are plotted against average surface roughness, along with the structured surface thickness measured from the SEM images. The plot shows a clear correlation between MgO peak intensity and structured layer thickness. It can be therefore implied that the obtained surface geometry is highly linked to the surface chemistry. The gas environment is an essential parameter for surface chemistry, but is also linked to surface morphology, because it can change also induce changes in the beam propagation path [31-33]. The laser processing in this work was carried out in ambient atmosphere. Due to the high reactivity of Mg, MgO forms easily on the surface and its extent increases as a function of the energetic parameters. More interestingly, the generation of the sub-micrometric surfaces structures with nano-fibrils is accompanied by the presence of MgO formation. Similarly, Guan et al demonstrated the effect of Ar use in the ablation of AZ31B with a KrF laser, and showed that ablation in air produced finer surface structures [18]. The authors linked the generation of these structures to the condensation
of oxide particles on the surface. However, the nano-fibrils obtained in the sub-micrometric surface structure of this work are much finer in dimension. Czerwinski reported similar sub-micron surface structures on AZ91 alloy after air exposure at 745 K for 1 h [28]. On the surface loose, powder-like nodules of MgO formed that showed geometry much similar to the obtained nano-fibrous structured in this work. The laser process carried out in ambient atmosphere generates similar effects with a more compact surface structure and allows complete surface transformation under the irradiation area. Nanofibrous surface structures were also obtained on grade 2 Ti by Tavangar et al [34]. After processing the Ti samples in ambient atmosphere with fs laser pulses, the nanofibrous structures were obtained and accompanied by rutile and anatase TiO₂ as well as TiO. Thus, it can be implied that the formation of the nano-fibrous structures is linked to the chemical reaction kinetics that forms the MgO.

The change in surface morphology as well as the generation of MgO layer on the material can be problematic regarding the mechanical properties. In the case of dimpling, the generated dimples can induce notch effects and reduce the effective section of the material. In the case of re-melting, the surface may also induce internal stresses due to the thermal process. For comparison purposes moderate conditions for both surface re-melting with Rₐ=1.06 µm and dimpling with average hole depth at 15.3 µm were chosen. In Figure 11, stress-strain curves of different surface conditions are shown. It can be seen that the surface treatments do not induce any remarkable difference compared to the non-structured alloy. The main parameters obtained from the tensile tests are also reported in Table 5. The small reduction in the UTS and elongation at fracture can be attributed to generated fragile MgO structure as well as induced thermal stresses.

4. Conclusion
This work demonstrated the use of a fibre laser operating with ns pulses for surface structuring of AZ31 Mg alloy. Two distinct strategies, namely surface re-melting and dimpling, were introduced to generate different micro and sub-micron structures. It is demonstrated that the control over the key laser process parameters allows flexible manipulation of the surface roughness. In particular Rₐ could be decreased below the initial value resulting a polished surface and could be increased up to 4 µm. More interestingly, mono-directional, homogenous surfaces as well as surfaces with particle growth were observed. The surfaces with particle growth showed nano-fibrous structures. These sub-micron structures were obtained with a single step laser process
when $F_l > 500 \text{ J/cm}^2$ in ambient atmosphere. The growth of the particles and nano-fibrous structures was linked to the oxidation kinetics. The dimpling strategy showed that the Mg alloy could be ablated in both gentle and strong ablation modes, while the transition occurred around $F_0 = 10.3 \text{ J/cm}^2$. Tensile tests also confirmed that the surface structures did not alter the mechanical performance of the alloy significantly under static load. However, the effect on fatigue resistance would require more attention.
References


[28] Czerwinski F. The oxidation behaviour of an AZ91D magnesium alloy at high temperatures. *Acta Mater*
2002;50:2639-2654


List of tables

Table 1. General characteristics of the pulsed laser source.

<p>| | | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Wavelength</td>
<td>$\lambda$</td>
<td>1064 nm</td>
</tr>
<tr>
<td>Max. average power</td>
<td>$P_{\text{avg}}$</td>
<td>50 W</td>
</tr>
<tr>
<td>Pulse duration</td>
<td>$\tau$</td>
<td>250 ns</td>
</tr>
<tr>
<td>Pulse repetition rate</td>
<td>PRR</td>
<td>20-80 kHz</td>
</tr>
<tr>
<td>Max. pulse energy</td>
<td>$E$</td>
<td>1 mJ</td>
</tr>
<tr>
<td>Beam</td>
<td>$M^2$</td>
<td>1.7</td>
</tr>
<tr>
<td>Collimated beam diameter</td>
<td>$d_c$</td>
<td>5.9 mm</td>
</tr>
<tr>
<td>Focal length</td>
<td>$f$</td>
<td>100 mm</td>
</tr>
<tr>
<td>Focused beam diameter</td>
<td>$d_0$</td>
<td>39 $\mu$m</td>
</tr>
<tr>
<td>Beam diameter at $h_f$=-2 mm</td>
<td>$d_s$</td>
<td>124 $\mu$m</td>
</tr>
</tbody>
</table>

Table 2. Processing conditions used for laser surface structuring by re-melting.

<table>
<thead>
<tr>
<th>Fixed parameters</th>
<th>Levels</th>
</tr>
</thead>
<tbody>
<tr>
<td>Focal height</td>
<td>$h_f$ [mm]</td>
</tr>
<tr>
<td>Pitch</td>
<td>$p$ [µm]</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Varied parameter</th>
<th>Levels</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pulse energy</td>
<td>$E$ [mJ]</td>
</tr>
<tr>
<td>Pulse repetition rate</td>
<td>PRR [kHz]</td>
</tr>
<tr>
<td>Scan speed</td>
<td>$v$ [mm/s]</td>
</tr>
<tr>
<td>Number and direction of passes</td>
<td>S</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Measured variables</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Average surface roughness</td>
<td>$R_a$ [µm]</td>
</tr>
<tr>
<td>Average distance between five the highest peaks and five lowest valleys</td>
<td>$R_z$ [µm]</td>
</tr>
</tbody>
</table>
Table 3. Processing conditions used for laser surface structuring by dimpling.

<table>
<thead>
<tr>
<th>Fixed parameters</th>
<th>Level</th>
</tr>
</thead>
<tbody>
<tr>
<td>Jump delay t(_{\text{delay}}) [(\mu)s]</td>
<td>8000 (\mu)s</td>
</tr>
<tr>
<td>Pump current PI</td>
<td>100%</td>
</tr>
<tr>
<td>Pulse repetition rate PRR [kHz]</td>
<td>50</td>
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<tr>
<td>Focal height h ([\text{mm}])</td>
<td>0</td>
</tr>
<tr>
<td>Pitch (p) [(\mu)m]</td>
<td>100</td>
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<table>
<thead>
<tr>
<th>Varied parameter</th>
<th>Levels</th>
</tr>
</thead>
<tbody>
<tr>
<td>Modulation duration (t_{\text{mod}}) [(\mu)s]</td>
<td>From 35 to 80 with 5 steps</td>
</tr>
</tbody>
</table>

Measured variables

<table>
<thead>
<tr>
<th>Dimple diameter (d) [(\mu)m]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dimple depth (h) [(\mu)m]</td>
</tr>
</tbody>
</table>

Table 4. Process parameters belonging to the chosen surface conditions..

<table>
<thead>
<tr>
<th>Surface type</th>
<th>Re-melting</th>
<th>Dimpling</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>(R_a=0.22) (\mu)m</td>
<td>(R_a=0.36) (\mu)m</td>
</tr>
<tr>
<td>Pulse energy, (E)</td>
<td>0.10 mJ</td>
<td>0.10 mJ</td>
</tr>
<tr>
<td>Modulation duration, (t_{\text{mod}})</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Pulse repetition rate, PRR</td>
<td>20 kHz</td>
<td>20 kHz</td>
</tr>
<tr>
<td>Scan speed, (v)</td>
<td>100 mm/s</td>
<td>100 mm/s</td>
</tr>
<tr>
<td>Number of passes, (S)</td>
<td>1</td>
<td>4</td>
</tr>
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</table>

Table 5. Mechanical properties of the non-structured and laser structured AZ31 alloy.

<table>
<thead>
<tr>
<th>Type</th>
<th>UTS [MPa]</th>
<th>YS [MPa]</th>
<th>Fracture elong. (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Non-structured</td>
<td>277±1</td>
<td>161±17</td>
<td>18±0.5</td>
</tr>
<tr>
<td>Dimpling</td>
<td>277±3</td>
<td>185±3</td>
<td>18±0.5</td>
</tr>
<tr>
<td>Re-melting ((R_a=1.07) (\mu)m)</td>
<td>268±3</td>
<td>180±14</td>
<td>15±5.5</td>
</tr>
</tbody>
</table>
List of figures

Figure 1. Ramped emission profiles obtained with the change in modulation duration.

Figure 2. SEM images of the surfaces obtained in different processing conditions (v↓=25 mm/s, v↑=100 mm/s). The different surface structures are highlighted by different colours (blue: mono-directional surfaces, green: homogeneous surfaces, pink: surfaces with particle growth).

Figure 3. Generation of different surface structures as a function of line fluence (MD: mono-directional surfaces, HS: homogeneous surfaces, PG: Surfaces with particle growth).

Figure 4. Evolution of micro and nano structure growth on AZ31 Mg alloy surface: a), b), c) micro globular surface structures obtained with E=0.10 mJ, PRR=20 kHz, S=4, v=25 mm/s; d), e), f) surface partially covered with microglobular and nanofibrous structures obtained with E=0.25 mJ, PRR=20 kHz, S=4, v=25 mm/s; g), h), i) complete surface coverage with fractal micro and sub-micron structures obtained with E=0.20 mJ, PRR=70 kHz, S=2, v=25.

Figure 5. Regression model of average surface roughness $R_a$ as a function of laser surface re-melting process parameters ($R^2_{adj}= 99.4\%$; $R^2_{pred}= 99.3\%$). Response variable $R_a$ is plotted on the z axis, whereas process parameters S and E are plotted in x and y axes respectively. The colour scale shows the v level and two levels of PRR are shown in separate graphs.

Figure 6. Regression model of distance between the highest peak and lowest valley $R_z$ as a function of laser surface re-melting process parameters ($R^2_{adj}= 99.7\%$; $R^2_{pred}= 99.7\%$). Response variable $R_z$ is plotted on the z axis, whereas process parameters S and E are plotted in x and y axes respectively. The colour scale shows the v level and two levels of PRR are shown in separate graphs.

Figure 7. Dimple diameter and depth as a function of modulation time. The error bars indicate standard deviation of the measurements.

Figure 8. Dimples obtained in different ablation regimes shown in optical microscope and 3D images in false colours: a),c) gentle ablation obtained with $t_{mod}=45 \mu$s; b),d) strong ablation obtained with $t_{mod}=80 \mu$s.

Figure 9. Cross sections of the laser structured surfaces by re-melting. The back scattered emission SEM images depict changes in surface chemistry: a) $R_a=0.22 \mu$m, b) $R_a=0.36 \mu$m, c) $R_a=1.07 \mu$m, c) $R_a=3.02 \mu$m, d) $R_a=4.33 \mu$m

Figure 10. Oxide growth on laser structured surfaces by re-melting: a) XRD signal intensity at $2\theta=43^\circ$ depicting MgO formation, b) layer thickness and MgO intensity as a function of surface roughness.

Figure 11. Stress-strain curves belonging to non-structured specimens and representative cases of laser structured surfaces.
Figure 2a

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Figure 7

Dimple diameter, D
Dimpler depth, h

\[ D = (-8866 + 2620 \ln(t_{\text{mod}}[\mu s]))^{0.5} \]

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Figure 10

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