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Application of massive laser shock processing for improvement of mechanical and tribological properties

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14 15 16 17 18 19	*Corresponding author: Assist. Prof. Uroš Trdan, PhD. Email: <u>uros.trdan@fs.uni-lj.si</u> Tel.: +386 1 477 1432 Submitted to: <i>Surface and Coatings Technology</i> Date: November 15 2017
20	Abstract
21	The present paper aims to investigate topographical, microstructural, mechanical and
22	tribological behaviour of precipitation hardened Al alloy subjected to massive laser shock
23	processing (LSP) without protective coating at 2500 pulses/cm ² , using three beam diameters.
24	Wear tests under dry sliding conditions resulted in severe wear, whereas the main wear
25	mechanisms were adhesion accompanied by abrasive wear. Nevertheless, LSP with optimal
26	processing parameters reduce the friction coefficient and wear rate with lower degrees of
27	adhesion and abrasion inside the wear track in comparison to the untreated sample. The
28	enhanced tribological performance is attributed to the positive influence of LSP induced
29	surface topography, surface compressive residual stresses (RS) and dense dislocation

30 arrangements, as the result of high-pressure shock waves. Nonetheless, due to the narrow

window of optimal parameters reduced wear resistance as a consequence of undesired

32 thermal/softening effect due to laser ablation and melting was detected with non-optimal

33 processing parameters.

- Keywords: Laser shock processing; Aluminium alloy; Wear, Microhardness; Residual stress;
 Microstructure.
- 37

38 **1. Introduction**

39 Despite the fact that laser processing technologies belong to a green manufacturing branch 40 and that aluminium and its alloys are the third most commonly used commercial engineering 41 materials, a constant demand towards higher efficient surfaces, lower and cleaner production 42 costs with lower waste gas emissions remains. Among the products within the 6xxx series 43 aluminium alloys, AA6082 is regarded as a high (medium-high) strength alloy, which 44 contains high numbers of intermetallic second-phase particles, ranging up to 10 µm, i.e. 45 spherical α -Al₁₂(Fe, Mn)₃Si, β -Al₅FeSi, and β -Mg₂Si in the form of plates or cubes [1]. 46 However, the predominant nano-precipitate in the peak-aged condition contributing the most 47 to the increase in material hardness and strength is the β'' phase (Mg₅Si₆) [2]. Nevertheless, 48 despite the fact that the age hardenability of Al-Mg-Si alloys is high due to excess amounts 49 of silicon and magnesium, which enhances the precipitation kinetics during heat treatment, 50 the major disadvantage is insufficient wear resistance [3].

51 According to Sánchez-Santana et al. [4] wear can be regarded (along with fatigue and 52 corrosion) as one of the three most common problems found in industry, leading to the 53 replacement of industrial parts and components, due to reduced operating efficiency, 54 increased loss of power, oil consumption, etc. Ductile materials, such as aluminium alloys 55 under dry sliding conditions, usually experience severe wear; however, it is far from clear 56 which aluminium alloy would offer the best wear resistance [5]. In fact, as Ghazali et al. [6] suggested it is not clear if the wear resistance scales with the starting hardness of the alloy, 57 58 which would suggest that a precipitation-hardened matrix would be optimum, or whether it is 59 the work-hardening characteristics that are more important.

60 Over the previous two decades laser shock processing (LSP) has been recognized as an 61 advanced, effective, fast emerging severe plastic deformation (SPD) technology which has 62 been successfully applied to various materials to impart compressive residual stresses, various 63 high-density dislocation configurations, grain refinement, improved fatigue, corrosion and 64 wear resistance [7][8][9][10][11][12][13][14]. Authors [7][10] have confirmed 65 nanocrystalline structures with refined grains, dense dislocation walls and dislocation cells in 66 the material surface as a consequence of laser-induced shock waves propagating into material. 67 Moreover, it has been shown [11] that LSP with a sacrificial protective layer is a reliable and 68 precise surface texturing technique for the fabrication of surface microdents, which may act as 69 lubricant reservoirs to reduce friction and wear in contact applications. Kumar et al. [12] have 70 also confirmed that LSP with optimized laser fluence can improve wear resistance by as much 71 as 91 % compared to an untreated sample. On the contrary, Hatamleh et al. [14] reported only 72 marginal improvement in wear of laser shock processed stir-welded 2195 Al alloy.

LSP also possess important environmental benefit over conventional SP process, with lower material/energy consumption during the peening process, with as much as 55% lower environmental impacts [15]. Moreover, it was pointed out that LSP has close-to-zero particulate emissions, hence greatly improves the indoor air quality and, thus reduces occupational health risks. However, the same authors argued that with the 'coated' LSP regime the consumption of protective opaque, i.e. aluminium foil present the dominant contributor of energy and material losses across all impact categories.

It should be noted that, according to the availability of different laser sources providing different pulse times and different laser energy over different treatment areas, two main processing regimes for LSP treatments exist, which can be applied either with or without an ablative/protective layer [10]. In the so-called 'high energy + long pulse' regime, pulsed lasers with energy in excess of several tens of joules and interaction times of up to several tens of ns, deliver their energy to broad surface areas (in excess of 10 mm²). In such way high thermo86 mechanical impulse able to originate the desired residual stress fields on a single pulse-by-87 pulse basis is provided. This approach demands an additional protective/sacrificial overlay 88 (paint, metallic tape, etc.) at the interaction zone prior to the laser application. The absorbing 89 protective coating enhances the laser radiation absorption and, in turn, prevents thermal 90 effects caused by the relatively long time of contact between the plasma and the treated 91 material.

92 In contrast to the coated LSP regime, the so-called 'low energy + short pulse' regime was 93 developed in 1995 for nuclear power plants since the process requires neither surface 94 preparation under radiation environment, nor drainage of water in a reactor vessel [16] [17]. 95 In this regime, pulsed lasers with interaction times in the range of several ns and with only 96 mJ-J of energy are applied to smaller surface areas in order to maintain the required threshold 97 energy for the LSP effect. In this case, large areas are covered by a controlled pulse 98 overlapping strategy. At each location of pulse incidence, the effect of pulse overlapping can 99 produce a deep (around or over 1 mm) field of compressive residual stress with very good 100 degree of uniformity and control [18]. In this case (with a comparatively short pulse 101 interaction time), the resulting mechanical and thermal waves applied to the treated material 102 are temporarily uncoupled. With the mechanical wave being applied faster, the residual 103 effects of the subsequent thermal wave are comparatively very small and limited to a narrow 104 zone close to the material's external surface. Hence, the effect of shock waves prevails, 105 producing compressive residual stresses [19].

This is an important feature to be taken into account as it is possible to eliminate the coating layer used in the 'high-energy' approach without any appreciable loss of final surface quality. Moreover, a quality factor due to the stress state uniformity in the component being treated can be provided. Further, protective overlay is a time-consuming affair and it must be applied at the interaction zone prior to the application to prevent the surface from being damaged by the high power laser irradiation. Also, the overlay becomes damaged severely 112 during the LSP process, requiring frequent replacement hence making it slow, less efficient 113 and expensive in industrial applications [20]. It should be noted that the handling system with 114 the low energy, uncoated LSP process to access the target component is simpler since there is 115 no reactive force against laser irradiation, which has confirmed this process to be very 116 practical not only in nuclear facilities but also in other harsh environments necessitating full-117 remote operation [19]. In view of this, low energy, short pulse, laser shock processing without 118 protective coating controlled by a predefined massive pulse overlapping strategy can be 119 regarded as a very promising, cost-effective and cleaner surface treatment technology.

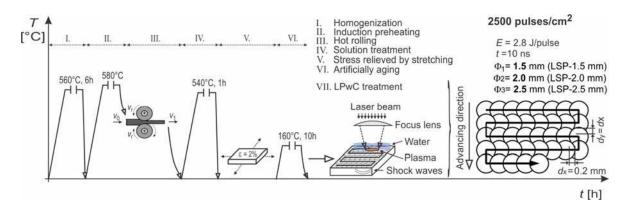
120 With this in mind and since there is limited reports on the possible detrimental effect of 121 non-optimal LSP process parameters on wear behaviour, the present work describes the 122 investigation of possible improvements of the surface morphology and tribological behaviour 123 of A1 6082 alloy by massive, uncoated low energy LSP with 2500 pulses/cm², using three 124 beam diameters, i.e. three laser intensities. Tribological behaviour was evaluated using a ball-125 on-disc tribometer and the wear tracks characterized using a scanning electron microscope 126 (SEM). In addition, the influence of laser shock processing on the surface morphology was 127 characterized using a 3D confocal laser scanning microscope (CLSM) and transmission 128 electron microscope (TEM), whereas the mechanical state was evaluated by microhardness 129 and residual stress by XRD and hole-drilling measurements.

130

131 **2. Experimental design**

132 2.1 Material and sample preparation

Test samples were sectioned from a 10 mm thick rolled plate of 6082 Al alloy, using a
water jet process. The chemical composition (in wt. %) of the material used in this study was
0.87 Si, 0.72 Mg, 0.42 Mn, 0.35 Fe, 0.15 minor elements (Cu, Cr, Ni, Zn, Ti) and Al the rest.
The overall heat treatment procedure, T651 (homogenization, solution treatment, aging, etc.),
including the subsequent LSP is schematically presented in Fig. 1.

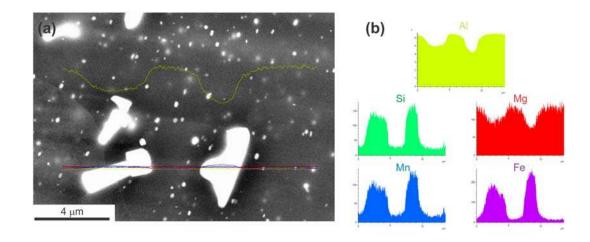


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Fig. 1. Schematic presentation of the overall heat treatment and subsequent LSP treatment.

141 SEM/EDS line analysis (Fig. 2) confirmed the basic Al matrix with fine distribution of 142 intermetallic phases. Results of our previous research [10] confirmed various intermetallic 143 particles in these alloys, i.e. smaller β -Mg₂Si precipitates in the form of plates or cubes and 144 larger α -Al₁₂(Fe,Mn)₃Si intermetallic dispersoids, which are in the length of ~ 4µm.

145



146

147 Fig. 2. (a) SEM/BEI microstructure of the base material; (b) EDS line analysis results marked
148 on (a).

Prior to LSP, no additional machining of the samples was carried out. In order to ensure surface uniformity and proper laser laser-beam interactions with the sample surface all samples were thoroughly degreased with acetone and rinsed with de-ionised water, before performing laser processing.

155 2.2 Laser shock processing

Laser shock processing (LSP) was carried out without any ablative/protective coating in a water confinement regime; whereas laser pulses were overlapped and scanned in a raster-type x-y pattern to completely cover the treated area (Fig. 1). A water jet set up was employed to create a thin water layer with a constant thickness (1–2 mm) and maintain a bubble-free, uniform confinement layer. This enabled plasma confinement during laser-target interaction as well as the replacement of laser ablated particles, ensuring a constant pure laser-matter interaction.

163 A Spectra-Physics Q-switched Nd:YAG laser source with an irradiation wavelength of 164 1064 nm, producing 10 ns duration pulses (FWHM), with the maximum laser beam energy of 165 2.8 J/pulse was used for LSP. The effects of a massive laser shock treatment was investigated 166 at a pulse density of 2500 pulses/cm², with a unified overlapping distance between 167 consecutive pulses of 0.2 mm, while the laser beam was constantly perpendicular to the 168 sample surface. Three kinds of beam diameters were chosen, i.e. 1.5 mm (LSP-1.5 mm), 169 2.0 mm (LSP–2.0 mm), and 2.5 mm (LSP–2.5 mm), which modified surface layers differently 170 due to different peak power density and coverage factors, as shown in Fig. 3.

For each parameter set of the experimental run, two samples were prepared for further analysis; (i) tribological characterization, and (ii) evaluation of surface modifications. In order to investigate the effects of massive LSP on surface integrity, i.e. laser-shock induced surface craters, dislocation configuration, and mechanical properties and its effect on dry-contact wear behaviour, no additional grinding of the treated surface was employed since it would not reflect the real surface condition induced by the laser treatment.

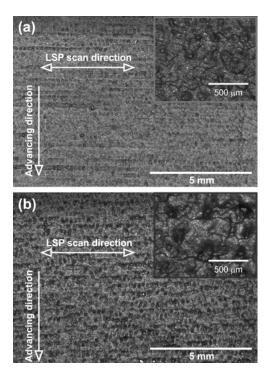




Fig. 3. Surface appearance after LSP with different beam diameters; (a) LSP–1.5 mm and (b)
LSP–2.5 mm.

182 *2.3 Surface topography*

Topographical analyses were performed using a confocal laser scanning microscope (CLSM) at a wavelength of 405 nm (Zeiss LSM700) to study the influence of massive LSP on the spatial characteristics of the treated surface. Determinations of surface roughness and waviness were obtained using a unified high-pass and low-pass filter with ($\lambda_c = 200 \mu m$).

187

188 2.4 Residual stresses and microhardness

Surface transverse σ_{xx} and longitudinal σ_{yy} residual stresses (RS) in regard to the LSP direction (Fig. 1) were determined by a Proto iXRD system with Cr-K α X-rays (2.291 Å) from the Al{222} diffraction peak located at the angle $2\Theta = 156.31^{\circ}$ [21][22]. The side inclination method with 9 beta angles with 5° oscillation for each measurement point was adopt, and the sin² ψ method with a Gaussian profile fitting was applied for the residual stress analysis. The X-ray elastic constant $S_2/2$ used was $18.56 \times 10^{-6} \text{ MPa}^{-1}$. The focused X-ray beam diameter was set to 2.0 mm.

196 Depth-resolved RS were measured with a Hole-Drilling measurement equipment and the 197 strain gage CEA-06-062UM-120 (Vishay Intertechnology Inc., Malvern, PA, USA) in 198 accordance with ASTM E 837-08 standard [23] and Vishay Tech Note TN-503-10 [24]. 199 Diameter of the blind hole was measured using Alicona G4 3D Infinite focus measuring 200 (IFM) device, with an optical lateral and vertical resolution of 800 nm and 100 nm. 201 respectively. The final RS in depth-distribution was calculated with the integral method, using 202 H-Drill software, whereas the average standard deviation of the errors in the measured strain 203 values was 4.7 µE. Both, hole-drilling technique, its operation principles and parameters of the 204 IFM device have been described in detail in ref. [18].

Near surface micro-hardness measurements prior to and after LSP were carried out on a
Vickers Hardness (HV) tester at a constant 200 g load and 20 s load time at the depth 75 μm
below the surface, whereas in average five separate measurements for each data point were
performed.

209

210 2.5 Microstructural observations

Worn surfaces of samples were identified with a Hitachi S-3000N scanning electron microscope (SEM) attached to an Energy Dispersive X-ray spectroscope (EDX). Microstructures of base material (BM) and LSP-treated samples were characterized in the cross-sectional direction using a Jeol 2000-FX transmission electron microscope (TEM), operated at 200 kV. Detailed TEM procedure TEM was described previously in ref. [10].

216

217 2.6 Friction and wear behaviour

Tribological behaviour under dry contact conditions was studied in air at an ambient temperature of 23 °C with a Microtest MT/30/NI/LIN tribometer using a ball-on-disc configuration with a rolling ball of AISI 52100 steel (diameter 3 mm). Test parameters were
chosen according to the ASTM G99-04 standard [25] as follows: sliding tangential speed
0.0785 m/s, sliding distance 1000 m and a normal load of 5 N, which corresponds to a
maximum Hertzian contact pressure of 1.22 GPa (mean value 0.81 GPa).

During the experiments friction coefficient were recorded. Afterwards, additional measurements of the cross-sectional areas for the calculation of the worn volume (area multiplied by the average length of the footprint) were assessed by means of a Leica ICM1000 confocal laser scanning microscope (CLSM) with a wavelength of 635 nm. For each condition, four measurements of the cross-sectional area at different locations of the groove were taken and averaged and specific wear rate K was calculated based on the worn volume.

Moreover, to obtain a further insight about massive LSP effect on wear behaviour additional surface profile, height-difference measurements, i.e. cross-sectional correlations (maximum height & max. depth) measurements and roughness analyses [26][27][28] of the wear track. All measurements were performed using Alicona G4 3D IFM device (using the same parameters as described above), whereas determinations of surface roughness were obtained using a unified high-pass filtration ($\lambda_c = 250 \ \mu m$).

237

238 **3. Results and discussion**

239 *3.1 Surface topography*

Fig. 4 and 5 show the topographic characteristic of the BM and the LSP sample surfaces, respectively. Since all laser treated samples showed similar topography as the result of massive laser shock processing at 2500 pulses/cm², the sample LSP-1.5 mm (Fig. 5) was chosen as the representative one, whereas the topography results of all samples are given in Table 1. In order to obtain a proper insight about LSP effects on surface topography, analyses were performed only on the laser processed region.

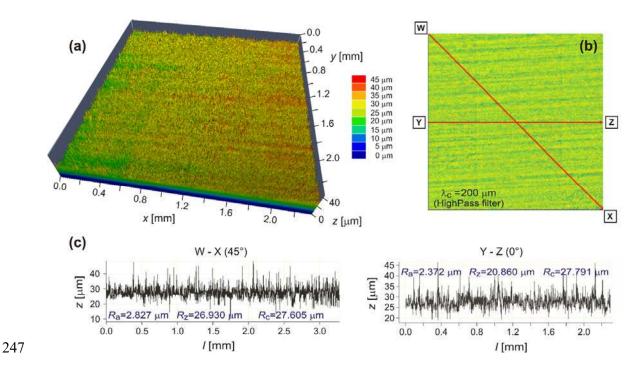
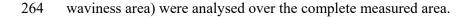


Fig. 4. CLSM topographical analysis of BM sample; (a) 3D image, (b) 2D image after highpass filtration and (c) topographical line profiles marked in (b).

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251 Topography comparison among these two samples confirms a major influence of 252 preliminary laser treatment, with higher waviness and roughness with the LSP sample. 253 Moreover, the roughness profile values with the BM sample in the directions of 0° and 45° 254 (marked on Fig. 4b) reveals very small differences as a consequence of the rolling 255 $(R_a=2.372 \ \mu m \ vs. \ R_a=2.827 \ \mu m)$. However, after LSP (Fig. 5), the significant anisotropy effect 256 in surface roughness can be seen as a collateral effect of the raster-type laser scan pattern (see 257 Fig. 1 and 3) Here, the R_a value is higher by as much as 43 % in the longitudinal (0°) in 258 comparison to the diagonal (45°) direction, i.e. 3.983 µm vs. 2.784 µm.

In order to obtain further surface texture information and comparison among BM and LSPtreated samples, three 3D amplitude roughness parameters (S_c – mean height of the roughness area; S_a – arithmetical mean deviation of the roughness area and S_z – averaged peak to valley of the roughness area) and three waviness parameters (W_c – mean height of the waviness area; 263 $W_{\rm a}$ – arithmetical mean deviation of the waviness area and $W_{\rm z}$ – averaged peak to valley of the



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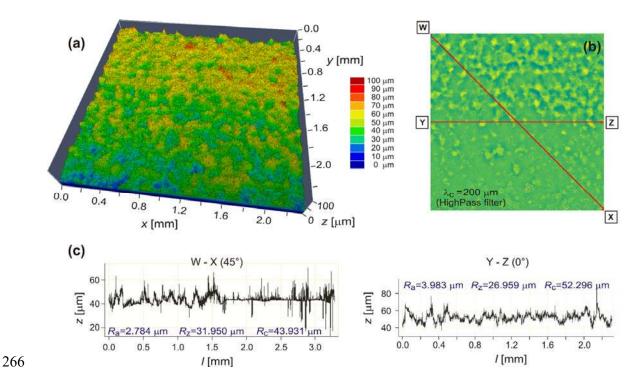
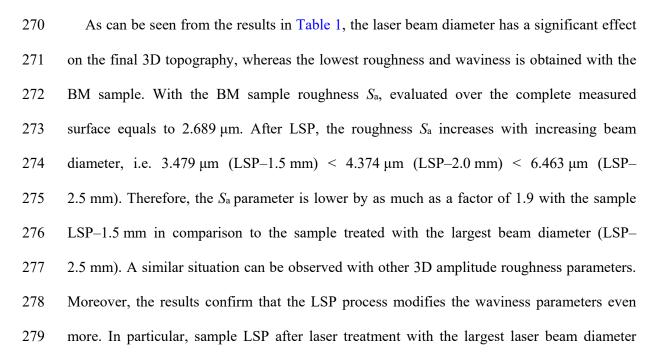


Fig. 5. CLSM analysis of LSP–1.5 mm sample. (a) 3D image, (b) 2D image after high-pass
filtration and (c) topographical line profiles marked in (b).



reveals a high increase of waviness, exhibiting an almost 35-times higher averaged peak to valley parameter W_z in comparison to the BM sample (68.704 µm vs. 1.986 µm) and a 10times higher value in comparison to the LSP–1.5 mm sample (W_z =6.646 µm). Hence, the obtained results indicate that the surface topography is indeed sensitive to the laser beam diameter and overlap ratio, expressing a lower degree of asperities (smaller roughness and waviness) with the lowest overlap ratio with the smallest laser beam diameter, and vice versa.

Sample	Sc (µm)	$S_a(\mu m)$	$S_{z}(\mu m)$	$W_{\rm c}(\mu m)$	$W_{a}(\mu m)$	$W_z(\mu m)$
BM	27.860	2.689	48.629	5.240	1.098	1.986
LSP-1.5 mm	48.369	3.479	76.577	9.047	3.011	6.646
LSP-2.0 mm	66.905	4.374	85.514	21.351	8.955	14.334
LSP-2.5 mm	85.777	6.463	94.140	77.388	21.398	68.704

Table 1. 3D CLSM topography (roughness and waviness) results.

288

289 During LSP, expressive surface craters were generated due to numerous laser-beam 290 interactions with the sample surface, producing local surface ablation and plastic deformation 291 induced by multiple laser-induced shock waves at ultra-high strain rates, which can exceed 292 10^7 s^{-1} [29]. Similar trend of the increased surface topography was reported previously by 293 Yakimets et al. [30], whereas only the changes of wave parameters with unchanged roughness 294 were confirmed. Nevertheless, it should be noted that in their investigation, an ablative 295 protective coating was used, which caused only a mechanical effect due to shock wave 296 propagation.

297

298 *3.2 Residual stresses and microhardness*

In this section residual stresses were investigated since it exist in almost all materials and arise whenever inelastic processes occur [31]. Moreover, the actual sign of residual stresses (tensile/compressive) and their location plays a crucial role on material performance in engineering applications, thus, demands the precise knowledge and control of residual stresses in various applications [32]. Although, recently intensive research efforts have been devoted

304 on downscaling of stress relaxation measurement techniques to a local-/micro-scale by slit 305 milling method using a combination of SEM imaging, FIB milling, and DIC image analysis 306 [31][32][33][34], in this study residual stresses were analysed using a combination of 307 standardized XRD and hole drilling method.

Fig. 6a and b illustrates in-depth residual stress distribution in x (σ_{xx}) and y-direction (σ_{yy}) obtained using the hole drilling technique. Examination of the holes after the drilling (Fig. 6c) confirms good accuracy and stability of the hole drilling equipment even at larger depths, with the average blind-hole diameter of 2.05 ± 0.03 mm.



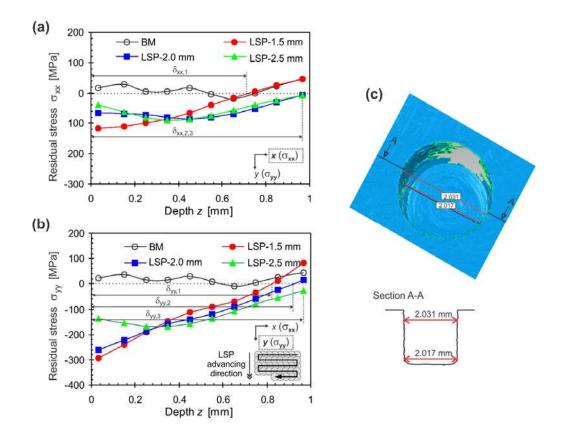




Fig. 6. (a,b) In-depth residual stresses and (c) 3D presentation of the drilled blind-hole and its
cross section (*Note*: blank spaces in Fig.6c represent the un-measured points).

316

The results of the residual stress measurements indicate close to zero stress state with the BM sample. On the contrary, all LSP samples indicate compressive stresses with significantly 319 higher values in the advancing, y-direction (σ_{yy}). Results depict that the magnitude of 320 compressive RS correlates with the magnitude of peak power density and is in the following 321 order: LSP-1.5 mm (-292 MPa) < LSP-2.0 mm (-261 MPa) < LSP-2.5 mm (-138 MPa). As 322 can be seen from Fig. 6b a relatively steep stress gradient is observed where the compressive 323 RS within the first 350 μ m increases to ~ -170 MPa. After this region, similar RS distribution 324 can be seen as with the other two LSP samples. However, with larger beam diameter, the penetration depth of compressive RS is higher. Hence, the lowest ($\delta_{xx,1}$ =725 µm) and the 325 highest ($\delta_{xx,3}$ >967 µm) penetration depths of compression in x-direction are obtained with 326 327 laser beam diameters of 1.5 mm and 2.5 mm, respectively. 328 It should be noted that compressive RS of -292 MPa and -261 MPa obtained with the 329 samples treated with beam diameter of 1.5 and 2.0 mm in y-direction are very close to the 330 yield strength of the material (320 MPa), indicating that RS are probably overestimated and

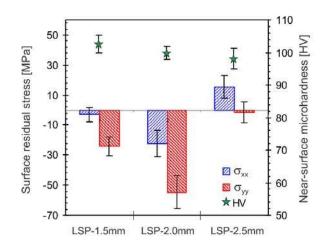
332 qualitative information about in-depth residual stress distribution [35][36].

331

Another important factor to be noted from Fig. 6 is quite large differences among RS in the specific direction (σ_{xx} and σ_{yy}) as the collateral effect of the raster laser scan pattern [37]. Nevertheless, it is worth noting that although differences among RS components in a specific direction exist, both stress components are of a compressive character, which could effectively enhance wear resistance.

the actual RS should be lower. Nonetheless, hole-drilling technique provides useable

In order to investigate the possible thermal/softening effect on the very top surface produced during massive uncoated LSP regime, additional XRD RS measurements were performed. Fig. 7 shows a comparison between top-surface RS and near-surface ($z = 75 \mu m$) Vickers micro-hardness. Surprisingly, the highest compressive RS are achieved with the sample LSP–2.0 mm treated with middle laser beam diameter ($\sigma_{yy} = -55 \pm 11$ MPa and $\sigma_{xx} = -$ 22 ± 9 MPa), followed by sample LSP–1.5 mm and LSP–2.5 mm. Results depict larger compressive RS in the y-direction compared to the stresses in x-direction, which is in 345 accordance with the hole-drilling RS results. Further, with sample treated with 1.5 and 346 2.0 mm laser beam diameter compressive RS are achieved in both directions, indicating 347 sufficient shock waves and work hardening effect, which prevailed over the thermal effect 348 due to laser ablation and melting.





350

Fig. 7. Comparison of surface residual stress and near-surface micro-hardness.

351

352 However, with LSP–2.5 mm sample tensile stresses in the x-direction ($\sigma_{xx} = 15 \pm 8$ MPa) 353 and near zero RS in the y-direction ($\sigma_{yy} = -1 \pm 7$ MPa) are obtained. Such results indicate that 354 here the softening/thermal effect prevailed over insufficient mechanical shock wave loading 355 due to low power density and high coverage factor as a consequence of the largest laser spot 356 diameter. Similar results were reported by Gill et al. [20] who investigated the effects of laser 357 shock processing with and without protective coating, where high compressive RS (-358 550 MPa) with a protective overlay and much smaller compressive stresses (-50 MPa) 359 without a protective coating were confirmed. However, their results depicted tensile RS from 360 the depth of $\sim 5-80 \,\mu\text{m}$ below the surface which afterwards changed to a compressive state. 361 Our results (Fig. 6 and 7) did not demonstrate this, indicating the important influence of the 362 LSP parameters.

363 Micro-hardness of untreated material was approximately 94 HV, whereas the highest 364 micro-hardness increase of about + 9% is obtained with the sample LSP-1.5 mm (103 \pm 3 HV 365 vs. 94 \pm 2 HV). The micro-hardness of other two LSP samples is 100 \pm 2 HV and 98 \pm 3 HV, 366 respectively. These results indicate correlation with the magnitude of compressive RS in Fig. 367 6 at the depth of 75 μ m, where microhardness was measured. Although, it would be expected 368 that large compressive RS would affect the microhardness on the larger scale, our results 369 indicate only minor increase. Such results are consistent with our previous study [38] and 370 with results reported by Peyre et al. [39], who also reported little improvement in the micro-371 hardness properties for 7075 Al alloy after laser shock processing compared to shot peened 372 material.

373

374 3.3 TEM analysis

Fig. 8 shows the TEM bright field images of the surface layers of untreated and LSP samples. It can be distinctly observed from Fig. 8a that the dislocation density in BM sample is moderately low, with two sets of nano intermetallic particles inside the matrix.

378 On the contrary, Fig. 8b-d confirms high-density dislocations introduced by laser shock 379 processing. Fig. 8b and c show dense dislocation walls, dislocation tangles and cells having 380 relatively thin walls. Here, only a few dislocation lines were observed, but dislocation pileups are formed in the vicinity of the different particles, which seems to act as effective 381 382 blockers of dislocation movement. Fig. 8b indicates that severe plastic deformation induced 383 by massive LSP impacts promoted in considerable increase in dislocation density, as well as 384 in the formation of dislocation networks and dislocation cell structures, which can lead to 385 refined and eventually to ultra-fine and nano-grains [40]. Results of our previous study [10] 386 indicate that dislocation slip is the main factor of the grain refinement mechanism during LSP 387 as a result of repetitive laser-induced shock waves at the treated surface. Based on the 388 comprehensive TEM analysis and dislocation density evaluation, the grain refinement 389 mechanism has also been proposed therein.

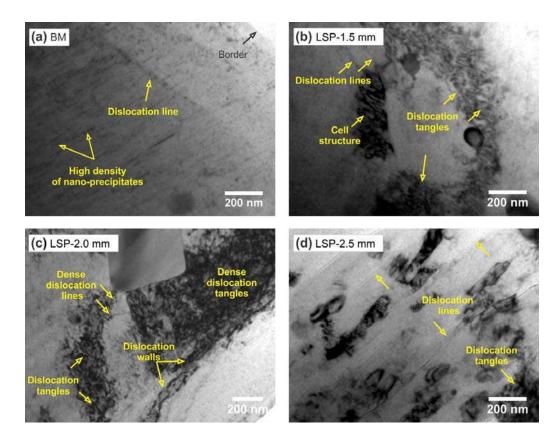


Fig. 8. TEM bright field observations of various samples: (a) BM, (b-d) LSP samples treated
with different laser beam diameters.

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395 Moreover, current and previous TEM observations revealed no effect of LSP on 396 precipitation kinetics, its distribution and size which is common with conventional shot 397 peening process. For example, Noordhuis and De Hosson [41] report obvious very fine 398 dispersion of silicon in aluminium of laser melted and shot peened eutectic aluminium-silicon 399 (Al-12Si) alloy with formation of small silicon precipitates, which lead to a further hardness 400 increase. In contrast, our results did not demonstrate this, which is in good correlation with 401 obtained hardness results, since the nano-size β'' phase (Mg₅Si₆) precipitates contributes the 402 most to the increasing strength of Al-Mg-Si alloys [2][42]. Hence, finer or denser distribution 403 of β'' nano-size precipitates reflects in greater hardness and strength of such Al-alloy [10]. 404 Fabbro et al. [43] attributed this differences (SP vs. LSP) to three interactive factors: (1) shock 405 duration is very small, (2) compared to the SP process, no contact deformation or Hertzian

406 loading occurs, and (3) impact pressures are usually much lower than those from the SP 407 process. Nevertheless, the same authors confirmed more than two times higher fatigue life 408 after laser shock processing compared to the shot peening, in which higher surface 409 embrittlement and surface roughening promoted more rapid crack development.

410 However, in the case of the LSP-2.5 mm sample (Fig. 8d) the situation is different. Here, 411 TEM analysis shows only the presence of dislocation lines and the presence of nano-particles, 412 of which many have planar defects as a result of thermal effect, producing tensile RS (Fig. 7). 413 This phenomenon is most likely associated with nano-particles deposition on top of the 414 underlying matrix during LSP treatment, where after surface ablation shock waves re-deposit 415 these particles on the matrix [20]. However, this is consistent only with LSP–2.5 mm sample 416 treated with the largest beam diameter and the highest overlapping ratio and is not the case 417 with other two LSP samples.

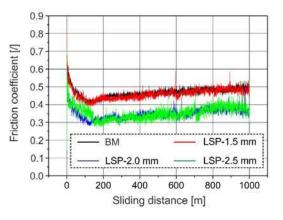
418

419 *3.4 Friction and wear*

Fig. 9 shows the real time friction coefficient variation of BM and LSP samples during wear tests with a contact force of 5N and speed of 0.0785 m/s. From the global friction coefficient evolution for the pair of 6082 Al alloy–AISI 52100 stainless steel ball (Fig. 9a), it can be noted that all samples exhibited a similar trend, with a certain degree of oscillation.

424 By examining the diagram of the friction coefficient vs. sliding distance, it can be seen that 425 the friction coefficient stabilizes quickly after the start (running-in-period). Afterwards, active 426 friction is observed showing the approximately linear function of the sliding distance. At the 427 beginning of the steady state period (~ 100m), the friction coefficient was found to be 428 approximately 0.4 and 0.3 for the BM and LSP samples, respectively. With further sliding 429 distance, the friction coefficient show a constant increase with a certain degree of oscillations, 430 associated with a stick-slip phenomenon as adhesion wear takes place, due to junction 431 formation and interlocking of asperities between mating surfaces and progressive degradation

- 432 of the sample surface. Wear particles which are generated begin to plough into the surface,
- 433 indicating steady weight loss of the tribosystem.



435

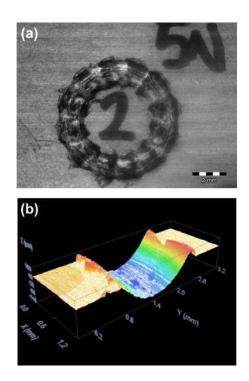
Fig. 9. Friction coefficient as a function of sliding distance.

436

437 Nevertheless, Fig. 9 shows that, in comparison to the BM, LSP produce a lowering of the 438 global friction coefficient under dry contact conditions. After ~700 m, all samples show 439 almost constant friction, indicating that wear particles inside the wear track are balanced by 440 wear particles leaving the wear track. The average friction coefficient at the end of the test at 441 1000 m sliding distance was 0.51 for the BM and in the range of 0.36 - 0.48 for LSP samples. 442 LSP-1.5 mm sample shows similar friction to the BM sample most likely due to the lower 443 surface roughness and waviness compared to the other two LSP sample (Table 1), which 444 reflect in less contact points of asperities resulting in increased contact pressure and 445 coefficient of friction.

Fig. 10 shows an example of the typical wear pattern and an obtained 3D CLSM profile, which was employed to determine worn volumes. Visual inspection revealed no visible damage on the AISI52100 ball surface, due to its much higher hardness. Thus, the wear of the ball was neglected. In order to obtain accurate readings measurements of the wear volume were taken at four different locations along the wear track, and the average value along with the standard deviation was calculated. After determination of the worn volumes, specific wear

- 452 rates of the samples were calculated, and wear scars were evaluated via SEM/SEI analysis to
- 453 determine whether severe or mild wear occurred.
- 454



456

Fig. 10. Typical wear scar (a) and 3D CLSM wear scar profile (b).

457

458 Measured wear volume and calculated values of the specific wear K rate (worn volume (mm³) divided by the product of load (N) and total sliding distance (m)) of untreated and LSP 459 460 samples are given in Table 2. The specific wear rate of BM sample $(4.08 \pm 0.83 \times 10^{-4})$ mm³/Nm) is 12 % higher in comparison to the sample LSP-2.0 mm $(3.64 \pm 0.59 \times 10^{-1})$ 461 462 ⁴ mm³/Nm), which exhibited the lowest wear among all samples. Nonetheless, it should be 463 noted that values of specific wear rate greater than 2×10^{-4} mm³/Nm indicate that sever wear 464 occurred with all tested samples [44][45]. As expected, the highest wear rate ($K=4.09 \pm 0.86 \times 10^{-4} \text{ mm}^3/\text{Nm}$) was achieved with LSP-2.5 mm sample. 465

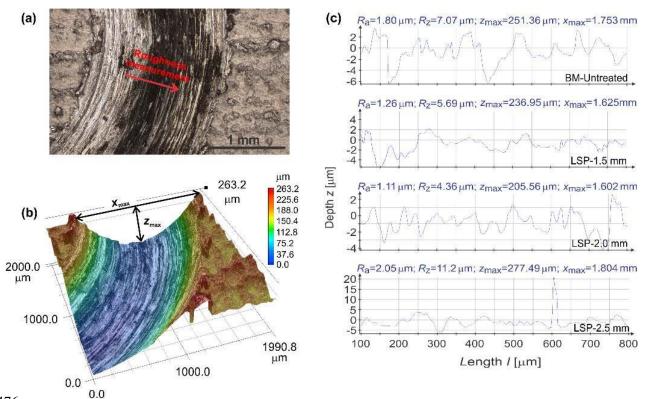
467 **Table 2.** Wear volume (W_v) and specific wear rate (K) results.

		-r		
	$W_{ m v}$	σ	Κ	σ
Sample	(mm ³)	(mm^3)	(mm ³ /N m)	(mm ³ /N m)

Untreated	2.041E-03	4.165E-4	4.083E-4	8.331E-5
LSP-1.5 mm	1.959E-03	3.199E-4	3.918E-4	6.398E-5
LSP-2.0 mm	1.820E-03	2.952E-4	3.640E-4	5.904E-5
LSP-2.5 mm	2.045E-03	4.289E-4	4.090E-4	8.578E-5

468

The higher specific wear rate of the sample LSP–2.5 mm treated with the largest spot diameter is directly associated with the treatment itself, due to the softening effect during laser shock processing. As TEM analysis confirmed local thermal effects with this sample caused the lowest dislocation density inside the Al matrix (Fig. 8d) and tensile surface RS (Fig. 7), which restrained the protective properties above the critical pressure during the wear test as soon as the asperities induced by LSP were worn off.





477 Fig. 11. (a) Schematic presentation of roughness measurement and (b) determination of
478 maximum width and height of the cross sectional profiles of the wear track. (c) surface
479 profiles inside the wear track with given values obtained form the topographical analyses.
480

481 Topographical features of the wear scars, i.e. surface profile, cross-sectional measurements 482 maximum height and roughness analyses were additionally studied by IFM microscopy 483 (Fig.11). From the cross sectional profiles (Fig.11b) maximum width (x_{max}) and height (z_{max}) 484 of the wear track were determined. Results from Fig 11c depict perfect correlation with the 485 Wear volume (W_v) and specific wear rate (K) results. Moreover, it is clearly revealed that the 486 largest x_{max} (~1804 µm) and z_{max} (~277 µm) of the wear track was obtained with LSP–2.5 mm 487 sample, which exhibit almost 35% higher maximum height in comparison to the LSP-2.0 mm 488 sample (and $z_{max} = \sim 206 \ \mu m$) due to occurrence of higher degree of abrasive wear, mainly by 489 a ploughing mechanism. This is additionally confirmed by the roughness results (Fig.11b and 490 c), whereas LSP-2.5 mm sample achieved the highest surface roughness inside the wear 491 track. Results depicts that the roughness R_a inside the wear track is in the following order: 492 LSP-2.0 mm $(1.11 \ \mu\text{m}) < \text{LSP}-1.5 \ \text{mm} (1.26 \ \mu\text{m}) < \text{BM} (1.80 \ \mu\text{m}) < \text{LSP}-2.5 \ \text{mm} (2.05 \ \mu\text{m}).$ 493 Although, in the case of hard, tough coating (e.g. TiC/a-C nanocomposite coatings) almost 494 linear correlation among wear rate and rate of roughness decrease exists [26], that does not 495 hold true in the case of soft materials. It should be noted that in such tribo-systems (soft Al 496 alloy-hard tool steel) surface asperities are worn off almost instantly during wear at such high 497 maximum Hertzian loading ($p_{\text{max}} = 1.22$ GPa). Hence, compressive residual stresses and 498 refined microstructure seemingly plays a crucial role on wear behaviour.

499 The above findings are additionally confirmed by SEM/SEI images of the worn surfaces in 500 Fig. 12. It is clear from Figs. 12a and b that the governing wear mechanism with the BM 501 sample is primarily adhesion and delamination by adhesion due to the occurrence of the stick-502 slip phenomenon resulting in progressive degradation of the surface. SEM analysis of the 503 worn surfaces at higher magnification (Fig. 12b and d) also confirm abrasive wear with many 504 scratches or grooves parallel to each other in the sliding direction in the worn track on both 505 the treated and untreated samples. However, visual inspection in Fig. 12b shows extensive 506 surface damage accompanied by deeper abrasion groove marks on the BM sample, as a result 507 of the agglomeration and compaction of the third-body wear debris generated during the test

508 [45] [46].

509

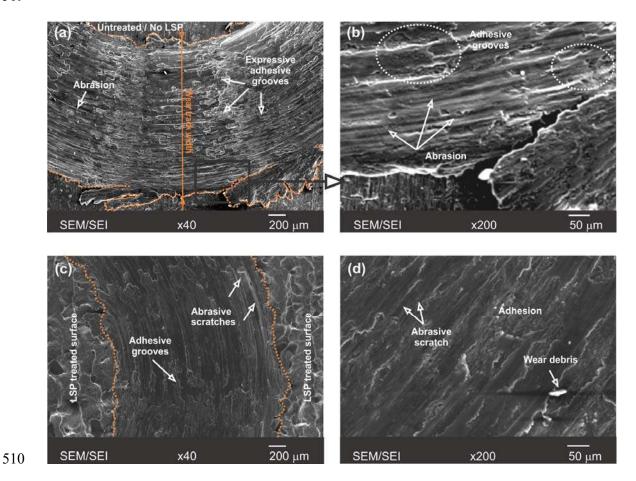
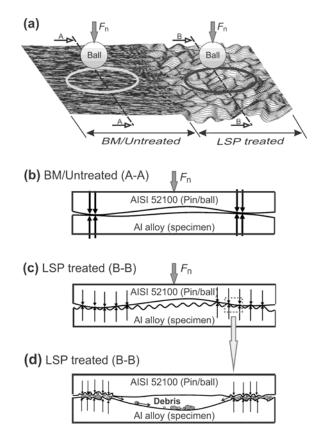


Fig. 12. SEM/SEI images of wear scars; (a-b) Untreated and (c-d) LSP sample treated with
2.0 mm laser beam diameter.

In contrast, after LSP, a smoother surface is obtained, with shallower abrasive marks, with a lower degree of plastic deformation due to high compressive residual stresses induced by the shock waves. Furthermore, the surface condition inside and near the wear track in Fig. 12c shows that the asperities formed during LSP were all removed during the test. Our results are consistent with those reported by Kumar et al. [12], which once again confirm the predominant effect of compressive residual stresses and high density of dislocations induced

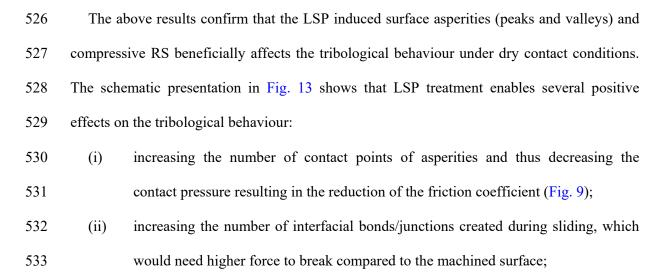
- 520 by the LSP process as soon as the surface asperities due to intense local plastic deformation
- 521 are worn off.



523 Fig. 13. Schematic presentation of surface topography effect on tribological behaviour under

dry contact condition.

524



(iii) neutralizing the degradation process by collecting the wear debris in the valleys of
LSP-induced craters, which is consistent with Fig. 12;

(iv) lowering the frictional energy as the asperities are worn off due to high nearsurface compressive residual stresses and high density of dislocations, which is
consistent with results in Figs. 7-9.

539

Furthermore, a modified, thicker, and more stable passive oxide film layer generated 540 541 during LSP treatment could also have contributed to the lowering of friction coefficient and 542 wear. In our previous work [13], XPS analysis confirmed that plasma ablation and shock 543 waves propagation during LSP treatment transforms Al₂O₃ into a more stable oxide form, with higher binding energy, contributing to 7-times higher polarisation resistance $R_{\rm p}$ and a 544 545 lower value of double layer capacitance (C_{d}) than BM sample. Moreover, the deformation 546 response of the asperities peaks in contact before the worn-out also plays a crucial role. 547 Although hardness has a high influence on the surface deformation mode, it seems that 548 surface compressive residual stresses contribute the most. Since the stress originating from 549 sliding is opposed to RS induced by LSP the frictional energy needed for sliding at the beginning of the running-in-period is lower, which is consistent with diagrams in Fig. 9. Our 550 551 results indicate that surface compressive residual stress plays a crucial role and prevails over 552 surface hardness, indicating higher elastic response of the asperities of the LSP surface.

In addition, under lubricated contact, the positive influence of LSP is even more considerable
because surface valleys act as lubricant reservoirs, which help to lubricate contact areas.
Hence, it contributes to the generation of hydrodynamic pressure and accordingly separation
of materials in contact [47].

557

558 **5.** Conclusions

559 Dry sliding wear characteristics of BM and LSP samples treated with 2500 pulses/cm², 560 have been investigated. Based on the research conducted, for this specific parameter window 561 used on 6082 Al alloy, the following conclusion can be given:

- Massive LSP can result in surface and in-depth compressive residual stresses with
 associated high density dislocation structures, accompanied by low, almost negligible
 increase of surface micro-hardness.
- 565 To minimize surface topography, a smaller laser beam diameter producing a lower 566 overlapping ratio is preferred.
- 567 Massive low energy, LSP was found to be effective in lowering wear rate and friction 568 coefficient by up to 29 %. However, LSP parameters play a crucial role on the 569 tribological properties, which can be even worse than with the BM if the parameters 570 are not optimal and the thermal effect prevails.
- 571 Predominate effect in reducing wear rate are compressive surface RS and high-density
 572 dislocation configurations.
- Maximal surface compressive RS were not achieved with the smallest laser beam diameter, i.e. highest power density. Results indicate optimal condition with LSP sample treated with the middle (2.0 mm) laser beam diameter. Here, the highest surface compressive RS, smallest friction coefficient, specific wear rate and wear volume with lowest roughness inside the wear track were achieved, despite lower microhardness compared to the LSP–1.5 mm sample treated with (highest power density).
- 580 The prevailing wear mechanism consists of adhesion, accompanied with abrasion on 581 both the untreated and LSP-treated samples. However, the latter showed superior 582 morphology, with a lower degree of adhesion and abrasion inside the wear track.
- 583

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27/35

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 592 measurements.
- 593

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- 721

722 Tables

Sample	<i>S</i> _c (μm)	$S_a(\mu m)$	$S_z(\mu m)$	$W_{c}(\mu m)$	W _a (µm)	$W_{\rm z}(\mu{\rm m})$
BM	27.860	2.689	48.629	5.240	1.098	1.986
LSP-2.5 mm	48.369	3.479	76.577	9.047	3.011	6.646
LSP-2.0 mm	66.905	4.374	85.514	21.351	8.955	14.334
LSP-2.5 mm	85.777	6.463	94.140	77.388	21.398	68.704

Table 1. 3D CLSM topography (roughness and waviness) results.

Table 2. Wear volume (W_v) and specific wear rate (K) results.

	$W_{ m V}$	σ	K	σ
Sample	(mm ³)	(mm ³)	(mm ³ /N m)	(mm ³ /N m)
Untreated	2.041E-03	4.165E-4	4.083E-4	8.331E-5
LSP-1.5 mm	1.959E-03	3.199E-4	3.918E-4	6.398E-5
LSP-2.0 mm	1.820E-03	2.952E-4	3.640E-4	5.904E-5
LSP-2.5 mm	2.045E-03	4.289E-4	4.090E-4	8.578E-5

729 Figure captions

- 730 Fig. 1. Schematic presentation of the overall heat treatment and subsequent LSP treatment.
- Fig. 2. (a) SEM/BEI microstructure of the base material; (b) EDS line analysis results marked

732 on (a).

Fig. 3. Surface appearance after LSP with different beam diameters; (a) LSP–1.5 mm and (b)

734 LSP–2.5 mm.

- Fig. 4. CLSM topographical analysis of BM sample; (a) 3D image, (b) 2D image after high-
- pass filtration and (c) topographical line profiles marked in (b).
- Fig. 5. CLSM analysis of LSP-1.5 mm sample. (a) 3D image, (b) 2D image after high-pass
- filtration and (c) topographical line profiles marked in (b).
- 739 Fig. 6. (a,b) In-depth residual stresses and (c) 3D presentation of the drilled blind-hole and its
- ross section (*Note*: blank spaces in Fig.6c represent the un-measured points).
- 741 Fig. 7. Comparison of surface residual stress and near-surface micro-hardness.
- 742 Fig. 8. TEM bright field observations of various samples: (a) BM, (b-d) LSP samples treated
- 743 with different laser beam diameters.
- 744 **Fig. 9.** Friction coefficient as a function of sliding distance.
- Fig. 10. Typical wear scar (a) and 3D CLSM wear scar profile (b).
- Fig. 11. (a) Schematic presentation of roughness measurement, (b) determination of
- 747 maximum width and height of the cross sectional profiles of the wear track and (c) surface
- profiles inside the wear track with given values obtained form the topographiyal analyses.
- 749 Fig. 12. SEM/SEI images of wear scars; (a-b) Untreated and (c-d) LSP sample treated with
- 750 2.0 mm laser beam diameter.
- 751 **Fig. 13.** Schematic presentation of surface topography effect on tribological behaviour under
- 752 dry contact condition.