

Influence of Polishing Orientation on the Generation of LIPSS on Stainless Steel

Florian Preusch, Stefan Rung and Ralf Hellmann

*University of Applied Sciences Aschaffenburg, Wuerzburger Straße 45, D-63743 Aschaffenburg,
Germany
E-mail: stefan.rung@h-ab.de*

We report on the influence of different angles between the electrical field of the impinging laser and the polishing direction of linearly polished surfaces on the generation of low spatial frequency LIPSS on stainless steel. The electrical field is rotated in a range of 0° to 90° with respect to the polishing direction and its effect on the orientation and homogeneity of the LIPSS is determined. In addition, the influences of the initial surface roughness and laser parameters such as the laser fluence on the generation of LIPSS are investigated. It can be shown, that the formation of LIPSS is driven by the initial surface roughness. The experimental results lead to the assumption that LIPSS were attracted by the linear grooves caused by polishing. Depending on the used parameter set, the orientation of the generated LSFL formation derived up to a value of 45° against the common predictions. Furthermore, a dependency of the required fluence for LSFL on surface roughness and polishing direction is demonstrated. Particularly, LSFL generated with a low fluence are more attracted by the surface polishing. Continuatively, the results may contribute to a further understanding of the underlying mechanisms involved in the generation of LIPSS. Moreover the results can be useful for producing LIPSS in large-scale for possible applications.

DOI: 10.2961/jlmn.2016.01.0025

Keywords: LIPSS, ripples, LSFL, surface structure, surface influence, stainless steel, polishing, roughness

1. Introduction

Upon irradiation by ultrashort laser pulses the generation of laser induced periodic surface structures (LIPSS or referred to as ripples) has been reported for several materials such as metals [[1][2][3]], semiconductors [[1][4][5]] or dielectrics [[1][6][7]]. Depending on the laser parameters, these self-organized nano structures occur with a periodicity near the used laser wavelength (low-spatial-frequency-LIPSS, LSFL) or with a smaller spatial period of approximately $\lambda/10$ (high-spatial-frequency-LIPSS, HSFL). The orientation of LIPSS is mainly determined by the polarisation of the employed laser, with LSFL commonly being oriented perpendicular to the polarisation of the electric field and HSFL being parallel to the polarisation [1].

The formation of LIPSS has a high potential in various mechanical, optical or medical application. For example, LIPSS affect the tribological properties of surfaces [8][9], alter the wetting behavior of surfaces (hydrophobic and hydrophilic characteristics) [3] or manipulate the cell expansion and cell adhesion for medical applications [10]. These manifold application demand for a profound understanding of the influences of system and laser processing parameters on the growth of LIPSS.

While LSFL can be described by a commonly accepted theory, there is no established model for HSFL. Since the first documentation of LIPSS on a semiconductor surface by Birnbaum [11] in 1965, several models were developed, most of which being based on an interference effect. By taking into account the influence of the laser wavelength, the incident angle and polarisation of the electric field,

Emmony et al. [12] laid the fundament of todays accepted theories. They describe the generation of periodic surface structures as a result of optical interference between the incident laser beam and the laser pulse generated surface electromagnetic waves (SEW). Sipe et al. [13] developed a mathematical description of the inhomogeneous energy deposition to the irradiate substrate: $A(\vec{k}) \propto \eta(\vec{k}, \vec{k}_i) \times |b(\vec{k})|$, where A describes the inhomogeneous absorption, the efficacy factor η is the response function describing the efficacy with which the interaction of the laser beam and the material leads to inhomogeneous absorption and b represents the Fourier component of the surface roughness. The latter is an important factor to stimulate the inhomogeneous absorption. A normal incident laser beam wouldn't be able to generate surface plasmon polaritons (SPP) without surface roughness [1]. To simplify the surface description, $b(\vec{k})$ is given by an isotropic approximation of equal spherical structures, described by a filling factor F and a shape factor s [14] [15]. Due to the high homogeneity of the surface described by this approximation, the Fourier component $b(\vec{k})$ is a slow varying function without a spatial effect on the LIPSS growth.

Contrary to that, real surfaces have roughnesses deviating from the ideal model. Due to their individual manufacturing processes or pretreatment, surfaces typically exhibit anisotropic surface roughness properties. As a consequence, it becomes apparent that the orientation of LIPSS may not only be governed by the polarisation of the

laser but also by $b(\vec{k})$. It has previously been shown that surface defects like scratches or debris have an influence on the orientation of the laser induced periodic surface structures [16]. Furthermore, Ardrion et al. [17] recently demonstrated the influence of surface finishing on the homogeneity of LSFL formation, in turn offering a further possibility to influence the selective generation of LIPSS. In turn, these findings in conjunction with the previous discourse on the role of $b(\vec{k})$ clearly demand for a further investigation on the influence of surface finish on the LIPSS formation.

2. Experimental Section

2.1 The Laser-System

The used beam source is an ultra short pulsed laser (Pharos 10-600-PP, Light Conversion) with an adjustable pulse duration between 250 fs and 15 ps, an emission wavelength of 1030 nm and a repetition rate of 300 kHz, respectively.

In figure 1, the experimental setup for the surface treatment is shown. The energy of the laser beam is adjusted by an external attenuator. To decrease the focal spot size, a beam expander telescope increases the beam waist. With a half wave plate in front of the focusing unit, the linear polarisation of the laser beam is rotated parallel to the onwards used scanning direction. A galvo head (RTA AR800 2G+, Newson) is used in combination with a telecentric lens ($f = 100$ mm) to focus the beam on the sample.

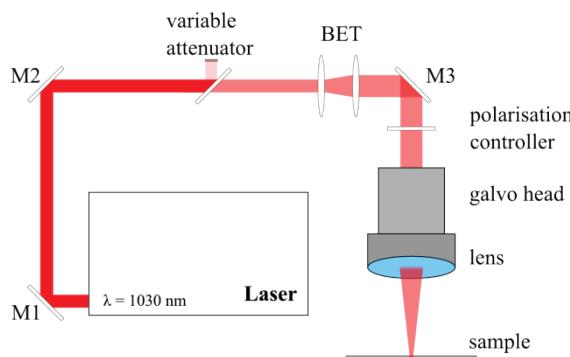


Figure 1 Experimental setup for surface treatment: M1-M3 are mirrors, BET is a beam expander telescope, and polarisation control is obtained by a half wave plate.

2.2 Method

To investigate the influence of the surface finish on the generation of LSFL, stainless steel samples were pretreated by linear polishing with different abrasives. The used grain size is between 65 μm (abrasive sheets, grit grade 220) and 10 μm (abrasive sheets, grit grade 2500) or suspension at 3 μm grid size, respectively. Upon this process, samples with a surface roughness between $R_z = 1.261 \mu\text{m}$ and $R_a = 0.063 \mu\text{m}$ are prepared (R_z being determined by a laser scanning microscope and describe the peak-to-valley amplitude). At table 1 the detailed used abrasive and the determined R_z is shown.

Table 1 used abrasives and the measured R_z

grain size	grit grade	R_z
220	65 μm	1.261 μm
320	65 μm	0.634 μm
600	26 μm	0.529 μm
1000	18 μm	0.489 μm
2500	10 μm	0.187 μm
suspension	3 μm	0.063 μm

The laser fluence is adjusted between 0.15 J/cm² and 0.60 J/cm² and the pulse duration is set by 300 fs. The lateral scanning speed is chosen to achieve a pulse overlap of 95 % at a repetition rate of 300 kHz. The angle between the polarisation of the electrical field with respect to the polishing direction is rotated in a range of 0° to 90° by rotating the sample. Throughout this paper, the rotation angle is abbreviated by RA. On stainless steel without a pretreatment, i.e. an undefined surface structure, the LSFL orientation is perpendicular to the polarisation of the incident laser beam in accordance to literature [18][19][20]. The influence of the orientation of the linear surface finish on the LSFL formation is determined by the angels α and β , as defined in figure 2. The angle α describes the rotation between the polishing direction and the scanning direction. At the initial position of the sample (rotation 0°), the scanning direction is perpendicular to the polishing direction. Hence, the angle α is 90°. With increasing rotation angle, α is decreasing. The angle β describes the orientation of the LSFL with respect to the scanning direction. Due to the commonly used model, LSFL formation on stainless steel is always perpendicular to the polarisation; thus the angle β should generally be 90°, as long as there is no other influence on the generation direction. In addition, the difference Δ between α and β should match the rotation angle without any influence to the periodic surface structure formation.

$$\Delta = |\alpha - \beta| \quad (2)$$

A deviation of the difference Δ from the rotation angle indicates a disturbing influence on the LSFL orientation.

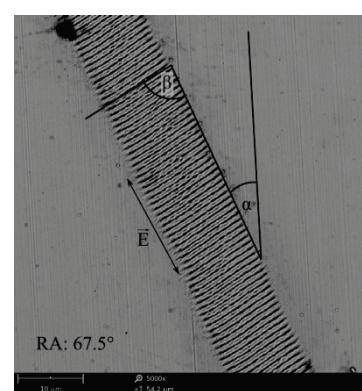


Figure 2 Method to measure the influence of linear surface polishing on the LSFL orientation

3. Results and Discussion

The investigation was carried out with different parameter sets consisting of combinations of rotation angle, surface roughness and laser fluence. In a first step of the in-

vestigation, it can be shown that a linear polishing can enhance the homogeneity of the LSFL formations. Thereto, the polishing direction is set perpendicular to the laser polarisation.

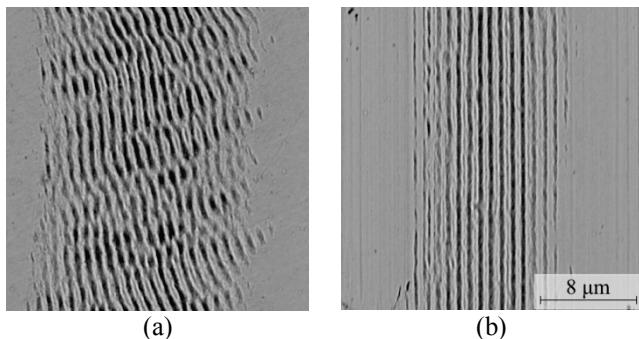


Figure 3 Periodic surface structures on a) undefined b) linear (vertical) polished surface

Figure 3 shows both LIPSS generated on an undefined polished surface (a) and on a linear polished (vertical) surface (b) as measured by a scanning electron microscope. Comparing both structures, the roughness apparently has a positive effect on the uniformity of the generated laser induced periodic surface structures.

Based on this perception, the influence of the polishing direction with respect to the rotation angle RA is investigated. During the evaluation of the experimental results, three different regimes of LSFL formations can be observed which is shown in an example in figure 4.

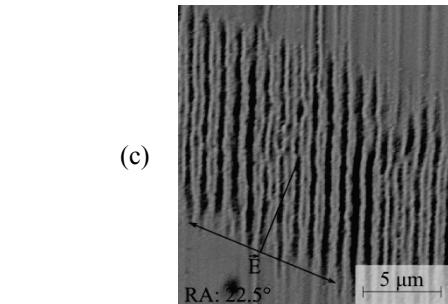
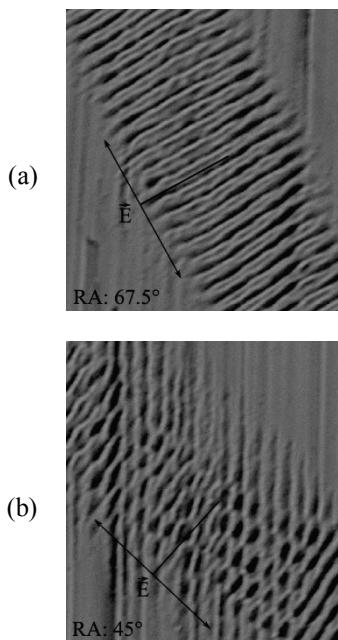


Figure 4 Different regimes during the LSFL generation on linear polished surfaces. The double arrow indicates the polarisation direction of the incident laser (a) for RA = 67.5° LSFL follow the normal of the polarisation, (b) for RA = 45° irregular LSFL formation and (c) for RA = 22.5° LSFL follow the polishing direction. All structures are generated on stainless steel with a surface roughness of $R_a = 1.261 \mu\text{m}$ with a fluence of 0.5 J/cm^2 .

(I) The typical behavior of LIPSS formation can be seen in figure 4a for a rotation angle RA of 67.5°. There is no influence of the polishing direction on the orientation of the periodic structures and according to the common models, the orientation of LIPSS is orthogonal to the polarisation of the incident laser beam. (II) However, the results shown in figure 4b clearly reveal that for a lower RA of 45° the polishing direction influences the linear homogeneity of the LIPSS and LSFL structures propagate in an irregular mode. Some structures follow the normal of the polarisation whereas other ripples were forced by the surface roughness into the direction of the linear polishing. (III) Finally, figure 4c highlights a behavior contrary to the common expectations for LSFL formation. The LIPSS orientation follows entirely the polishing direction of the stainless steel workpiece.

3.1 LSFL follow the normal of the polarisation

To quantitatively evaluate the effect of surface roughness and laser fluence on the LIPSS formation, the angle difference Δ introduced in section 2.2 is examined as a function of the rotation angle for different levels of surface roughness. According to the definition of Δ in section 2.2, Δ equals the rotation angle (RA) in case the orientation of the LIPSS is governed by the direction of the laser polarisation. In contrast, any deviant behavior of Δ from the rotation angle indicates a disturbing influence on the LSFL orientation. Figure 5 depicts the measured results of the determined angle difference Δ (given by the measured difference between α and β) as a function of rotation angle RA for a surface roughness R_a in the range of $0.063 \mu\text{m}$ to $0.187 \mu\text{m}$ and varying laser fluence.

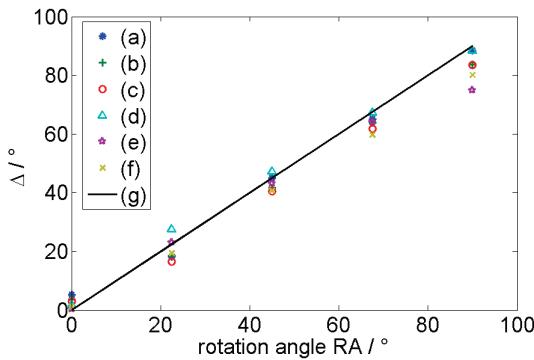


Figure 5 Difference Δ equals the rotation angle RA

- | | |
|--|--|
| (a) $F = 0.30 \text{ J/cm}^2, R_z = 0.187 \mu\text{m}$ | (b) $F = 0.45 \text{ J/cm}^2, R_z = 0.187 \mu\text{m}$ |
| (c) $F = 0.60 \text{ J/cm}^2, R_z = 0.187 \mu\text{m}$ | (d) $F = 0.30 \text{ J/cm}^2, R_z = 0.063 \mu\text{m}$ |
| (e) $F = 0.45 \text{ J/cm}^2, R_z = 0.063 \mu\text{m}$ | (f) $F = 0.60 \text{ J/cm}^2, R_z = 0.063 \mu\text{m}$ |
| (g) normal line with $\Delta = RA$ | |

Apparently, the calculated differences Δ by trend follow the ideal behavior with a slope of 1 independently of the applied laser fluence ($F=0.30 \text{ J/cm}^2$ to 0.6 J/cm^2) up to small deviations at high RA. In this regime of low surface roughness, R_z obviously has no influence on the orientation of periodic surface structures. They are oriented exclusively by the polarisation, i.e. orthogonal to the electrical field vector as expected from theory.

3.2 LSFL follow the polishing direction

In a regime of higher surface roughness, R_z being in the range between $1.261 \mu\text{m}$ and $0.489 \mu\text{m}$, LSFL orientation shows a behavior deviating from the common model. In addition, the degree of this discrepancy scales with the laser fluence. As figure 6 and 7 show, in certain parameter areas the generated LSFL are orientated along the linear polishing profile instead of being perpendicular of the polarisation. In this case, the difference Δ drops to 0 because α and β are alternate interior angles. Depending on the laser fluence, two sub regimes are determined for the case of LSFL following the polishing direction, which are referred to as a regime of a strong influence of the polishing direction (laser fluence below 0.30 J/cm^2) and to a regime of a weak influence of the polishing direction (laser fluence between 0.30 J/cm^2 and 0.60 J/cm^2).

In figure 6, Δ is plotted versus RA for a laser fluence of up to 0.30 J/cm^2 , showing that Δ is zero for rotation angles of up to 45° , i.e. the LSFL are orientated along the linear polishing profile (regime of strong influence).

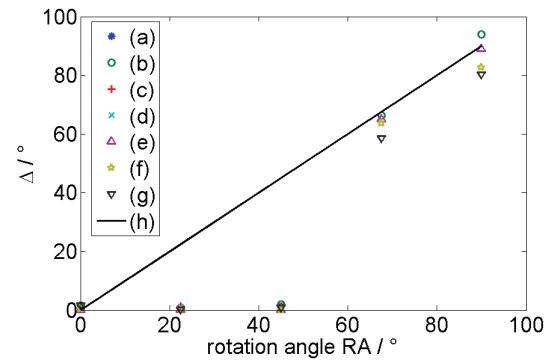


Figure 6 Difference Δ versus the rotation angle in the regime for LSFL following the polishing direction

- | | |
|--|--|
| (a) $F = 0.15 \text{ J/cm}^2, R_z = 1.261 \mu\text{m}$ | (b) $F = 0.30 \text{ J/cm}^2, R_z = 1.261 \mu\text{m}$ |
| (c) $F = 0.15 \text{ J/cm}^2, R_z = 0.634 \mu\text{m}$ | (d) $F = 0.15 \text{ J/cm}^2, R_z = 0.529 \mu\text{m}$ |
| (e) $F = 0.30 \text{ J/cm}^2, R_z = 0.529 \mu\text{m}$ | (f) $F = 0.30 \text{ J/cm}^2, R_z = 0.489 \mu\text{m}$ |
| (g) $F = 0.45 \text{ J/cm}^2, R_z = 0.489 \mu\text{m}$ | (h) normal line $\Delta = RA$ |

For higher laser fluence in the range between 0.30 J/cm^2 and 0.60 J/cm^2 , the rotation angle up to which the LSFL orientation is governed by the polishing direction drops to about 22.5° (figure 7) and for larger RA the LSFL again follow the polarisation of the laser ($\Delta = RA$): regime of weak influence of the polishing direction. Hence, for increasing laser fluence (higher amplitude of the electric field vector of the incident beam) the impact of the surface roughness decreases and the well-known influence of the polarisation direction dominates the LSFL generation process.

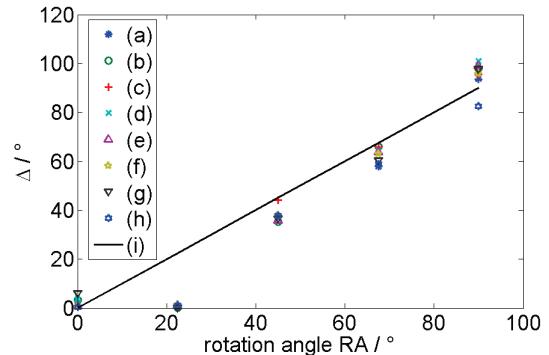


Figure 7 Difference Δ as a function of RA in the regime of a weak influence of polishing direction

- | | |
|--|--|
| (a) $F = 0.45 \text{ J/cm}^2, R_z = 1.261 \mu\text{m}$ | (b) $F = 0.60 \text{ J/cm}^2, R_z = 1.261 \mu\text{m}$ |
| (c) $F = 0.30 \text{ J/cm}^2, R_z = 0.634 \mu\text{m}$ | (d) $F = 0.45 \text{ J/cm}^2, R_z = 0.634 \mu\text{m}$ |
| (e) $F = 0.60 \text{ J/cm}^2, R_z = 0.634 \mu\text{m}$ | (f) $F = 0.45 \text{ J/cm}^2, R_z = 0.529 \mu\text{m}$ |
| (g) $F = 0.60 \text{ J/cm}^2, R_z = 0.529 \mu\text{m}$ | (h) $F = 0.60 \text{ J/cm}^2, R_z = 0.489 \mu\text{m}$ |
| (i) normal line $\Delta = RA$ | |

Please note, that experiments with a further increased fluence are prohibited since beyond the ablation threshold material is removed without significant LSFL generation. To confirm that the observed structures are LSFL and not periodic structures generated by polishing, a spectral analysis by using a fast Fourier transform (FFT) of the structures is done and can see in figure 8.

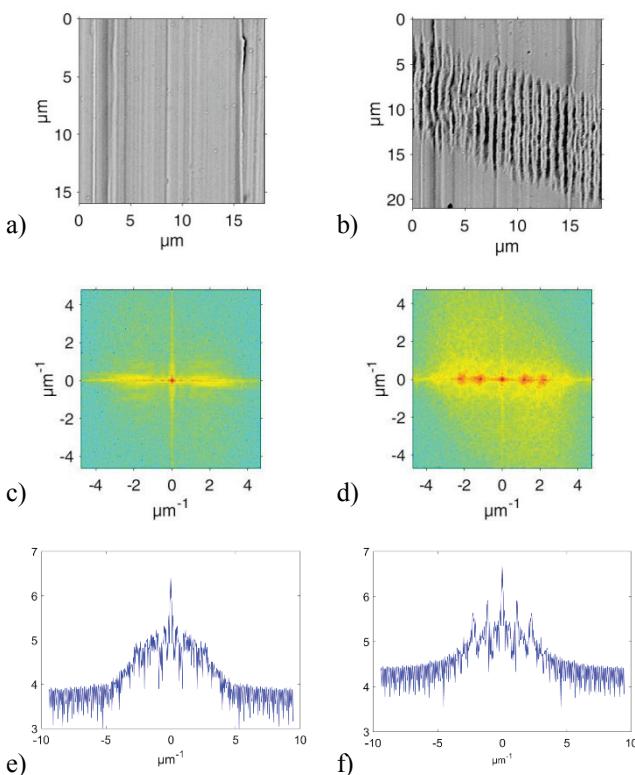


Figure 8 a) polished surface without LSFL; b) polished surface with LSFL; c) FFT picture of polished surface without LSFL; d) FFT of polished surface with LSFL; e) cross-section of FFT at polished surface without LSFL; f) cross-section of FFT at polished surface with LSFL

At figure 8 first line the polished structure is shown without a) and with LSFL b). At the spectral analysis on the second line it can be see that the polished structures c) have no LSFL-like periods. At figure 8 d) there are clearly peridical formation. The investigation of the cross-section shows that the posihed sureface e) has no periodical peak. In contrast at figure 8f) there are periods about 874nm and 437nm. That are LSFL and a half of the periods are the pits at the middle of the amplitude which could also describe at literature [21][22]. This behavior can be explain by contributions of Fresnel diffraction which have an influence of the orientation of the LIPSS [23].

3.3 Undetermined LSFL formation

In a transition region between those observations described in sections 3.1 and 3.2, a third classified regime of LSFL generation is observed. At the transition from LSFL following the polarisation to LSFL following the polishing direction, an undefined ripple growth is noticed (see figure 4b). This effect occurs at every executed test with an observed orientation change. A controlled LSFL generation is not possible with the combination out of a rough surface ($R_z > 0.187 \mu\text{m}$), a rotation angle between 22.5° to 45° and a low laser fluence.

3.4 Required fluence depending on surface roughness

In addition, an influence on the required laser fluence for the LSFL generation can be found. Samples with a rough surface ($R_z > 0.489 \mu\text{m}$) have a smaller LSFL threshold fluence if the rotation angel is below 45° . According to

Bonse et al. this can be attributed to a feedback mechanism of already generated LSFL formations to the inhomogeneous energy absorption of a rough surface [14]. In turn, this observation supports the conclusion that linear polishing enhances the growth of LSFL.

4. Conclusions

We have shown that the surface roughness of linear polished stainless steel has a strong impact on the generation of low-spatial-frequency-LIPSS. By studying the influence of the angle between the polishing direction and the polarisation of the incident laser on the LSFL orientation, we have shown that for smooth surfaces ($R_z \leq 0.187 \mu\text{m}$) the polishing direction has no influence on the LSFL orientation. In contrast, for a surface roughness $R_z > 0.187 \mu\text{m}$ the LSFL direction is driven by the polishing direction. This deviation from the common model additionally depends on the applied laser fluence. The orientation of LSFLs generated with laser fluence between 0.45 J/cm^2 and 0.6 J/cm^2 are influenced by the polishing up to a rotation angle of 22.5° . For smaller laser fluences (0.15 J/cm^2 - 0.3 J/cm^2) the influence of the polishing direction increases with the LSFL following the initial surface structure up to a rotation angel of 45° , this we could confirm by a FFT. Beyond this angle LSFL again follow the laser polarisation. In a transition area between these regimes the LSFL exhibit an irregular orientation, i.e. for a controlled LSFL generation this transition area should be avoided.

From an application point of view, the reported results are of significant importance as they clearly reveal that highly polished surfaces are not preferential for the generation of large scale uniform LSFL, i.e. elaborate and costly surface finishing processes can be avoided. Moreover, from a fundamental point of view, the presented experimental results may stimulate a further understanding of the generation of periodic surface structures.

References

- [1] J. Bonse, J. Krüger, S. Höhm and A. Rosenfeld: J. Laser Appl., 24, (2012) 042006
- [2] S. Bashir, M. S. Rafique, A. Ajami and W. Husinsky: Appl. Phys. A, 113, (2013) 673
- [3] B. Wu, M. Zhou, J. Li, X. Ye, G. Li and L. Cai: Appl. Surf. Sci., 256, (2009) 61
- [4] P. Fauchet and A. Siegman: Appl. Phys. Lett., 40, (1982) 824
- [5] J. F. Young, J. S. Preston, H. M. Van Driel and J. E. Sipe: Phys. Rev. B, 27 (1983) 1155
- [6] J. Heitz, B. Reisinger, M. Fahrner, C. Romanin, J. Siegel and V. Svorcik: 14en Int. Symp. on Transparent Optical Networks, Coventry, (2012) p. 1
- [7] G. Seifert, M. Kaempfe, F. Syrowatka, C. Harnagea, D. Hesse and H. Graener: Appl. Phys. A, 81, (2005) 799
- [8] C. Y. Chen, B. H. Wu, C. J. Chung, W. L. Li, C. W. Chien, P. H. Wu and C. W. Cheng: Tribol. Lett., 51, (2013) 127
- [9] J. Bonse, R. Koter, M. Hartelt, D. Spaltmann, S. Pentzien, S. Höhm, A. Rosenfeld and J. Krüger: Appl. Surf. Sci., (2014), <http://dx.doi.org/10.1016/j.apsusc.2014.08.111>

- [10] K. Wallat, D. Dörr, R. Le Harzic, F. Stracke, D. Sauer, M. Neumeier, A. Kovtun, H. Zimmermann and M. Epple: J. Laser Appl., 24 (2012) 42016
- [11] M. Birnbaum: J. Appl. Phys., 36, (1965) 3688
- [12] D. Emmony, R. Howson and L. Willis: Appl. Phys. Lett., 23, (1973) 598
- [13] J. Sipe, J. Young, J. Preston and H. Van Driel: Phys. Rev. B, 27, (1983) 1141
- [14] J. Bonse, M. Munz and H. Sturm: J. Appl. Phys., 97, (2005) 013538
- [15] J. Z. P. Skolski, G. R. B. E. Römer, A. J. Huis in't Veld, V. S. Mitko, J. V. Obona, V. Ocelik and J. T. M. De Hosson: J. Laser Micro/Nanoengineering., 5, (2010) 263
- [16] J. Gottmann, D. Wortmann and R. Wagner: Adv. Laser Techn., 7022 (2007) 702202-1
- [17] M. Ardron, N. Weston and D. Hand: Appl. Surf. Sci., 313, (2014) 123
- [18] J. Bonse, A. Rosenfeld and J. Krüger: J. Appl. Phys., 106 (2009) 104910
- [19] V. S. Mitko, G. R. B. E. Römer, A. J. Huis in 't Veld, J. Z. P. Skolski, J. V. Obona, V. Ocelík and J. T. M. De Hosson: Phys. Procedia 12, (2011) 99
- [20] S. Graef and F. A. Mueller: Appl. Surf. Sci., 331, (2015) 150
- [21] S. Hou, Y. Huo, P. Xiong, Y. Zhang, S. Zhang, T. Jia, Z. Sun, J. Qiu and Z. Xu: J. of Phys. D: Appl. Phys. Bd. 44, Nr. 50, (2011) 505401
- [22] J. W. Yao, C. Y. Zhang, H. Y. Liu, Q. F. Dai, L. J. Wu, S. Lan, A. V. Gopal, V. Trofimov und T. M. Lysak: Opti. express 20/2, (2012) 905
- [23] R. D. Murphy, B. Torralva, D. P. Adams, and S. M. Yalisove: Appl. Phys. Lett., 104 (2014) 231117

(Received: May 26, 2015, Accepted: February 22, 2016)