Influence of laser hardening on laser induced periodic surface structures on steel substrates

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Abstract. Laser-induced periodic surface structures (LIPSS) are used to structure surfaces for functionalization. Thus, hydrophilic states are generated using LIPSS. However, these nanostructures do not withstand mechanical loads and therefore cannot be used for most tribological applications. Within this work the approach of laser hardening of LIPSS is investigated. It is shown that laser hardening leads to an alteration of prior structured surfaces. That effects the wetting behaviour. The higher the laser power during hardening, the more increases the contact angle of a single droplet on the surface and the more the surface lacks in terms of wetting behaviour. This phenomenon is attributed to changes in LIPSS’ aspect ratio. A high ratio leads to low contact angles and is shifted to low values when the laser power increases resulting in high contact angles. Hence, it is concluded that the thermal load during laser hardening, and it’s influence on the wettability must be taken into account when LIPSS are subjected to laser hardening.

1. Introduction
Laser-induced periodic surface structures (LIPSS) are used to structure surfaces for their functionalization in mechanical applications [1]. These structures enable for example the influence of wetting properties on the one hand and of friction and wear behavior of contact partners on the other and are therefore subjected to many investigations [2–4]. Changing wetting properties becomes evident if conventional lubricants should be substituted with water. And alternatives to conventional lubricants are of great interest from ecological and economical points of view. But the low viscosity and the resulting insufficient film thickness due to poor wettability represent a major obstacle to use water as an effective lubricant [5]. Structuring a surface with laser-induced periodic surface structures which create a hydrophilic surface and therefore increase the wettability, it was shown that this obstacle could be overcome, and water be used as lubricant in a lubricated sliding application [6].

Nevertheless, LIPSS wear out quickly in direct tribological contact even when loads of 1 N (corresponds to 467 MPa) [7] down to 25 mN (corresponds to 218 MPa) [8] are applied. Consequently, for many technical applications, their wear resistance must be significantly increased while simultaneously preserving their functionality. Due to the local applicability, the realizable velocities, the generally achievable high hardness [9-14] and the feature that no quenching medium is needed, which could influence the nanostructures, laser hardening seems to be the tool of choice for processing LIPSS while minimizing substrate distortion [15]. El-Khoury et al. [16] showed the
feasibility of laser hardening to increase the hardness of direct laser interference patterning (DLIP) micro-textures.

The current state of the art shows the beneficial use of LIPSS in mechanical contact situations between two steel substrates. However, since LIPSS are subjected to wear, a treatment is needed to improve their resistance in terms of mechanical contact. Consequently, this work deals with the influence of laser hardening on geometrical and functional properties of laser induced periodic surface structures on X153CrMoV12 steel substrates. Besides contact angle measurements to determine the change in the wetting behavior of LIPSS, confocal laser scanning microscopy (CLSM) and scanning electron microscopy (SEM) are additionally used to characterize the geometrical changes.

2. Methodology

2.1. Substrate Material

The cold work steel X153CrMoV12 is used as substrate material. It has an initial hardness of 250 HV1 and is delivered as flat material. It is cut in substrates of 30 mm x 10 mm x 10 mm in length x width x height. Primary to structuring and hardening, the substrates are polished to an initial surface roughness Sa of 100 nm and then cleaned in an ultrasonic bath with isopropanol for five minutes. The chemical composition of X153CrMoV12 is given in Table 1.

| material number | material designation | C in mass-% | Cr in mass-% | Mo in mass-% | V in mass-% |
|-----------------|----------------------|-------------|--------------|--------------|-------------|
| 1.2379          | X153CrMoV12          | 1.55        | 12.00        | 0.70         | 0.80        |

2.2. Laser Structuring

The applied structures are low spatial frequency LIPSS first order (LSFL-I). These are applied via pico-second laser TruMicro 5050 made by Trumpf using the listed parameters in Table 2. The LIPSS are generated scanning parallel lines with a certain line spacing. The parameters are listed in Table 3.

| wavelength in nm | pulse duration in ps | repetition rate in kHz | spot diameter in µm |
|------------------|----------------------|------------------------|---------------------|
| 1030             | 6                    | 50                     | 46                  |

| pulse energy in µJ | scan velocity in mm/s | line spacing in µm | number of cycles |
|--------------------|-----------------------|-------------------|------------------|
| 200                | 1840                  | 36.8              | 1                |

2.3. Laser Hardening

A continuous wave (cw) disk laser TruDisk 12002 made by Trumpf was used for laser hardening. The laser parameter to harden the substrates with LIPSS on the surface are given in Table 4. The laser spot is rectangular. The energy distribution along the short axis is quasi-Gaussian while it is homogeneous along the long axis. Using a rectangular spot counteracts the increased heat influence of track overlap that would occur with a single, scanning laser spot. Therefore, the substrate is hardened with one cycle.
Table 4. Laser parameter to harden the substrates.

| Parameter                      | Value         |
|--------------------------------|---------------|
| Wavelength in nm               | 1030          |
| Spot size in mm x mm           | 10 x 1.6      |
| Laser power in W               | 250 to 1100   |
| Scan velocity in mm/s          | 10            |
| Shielding gas                  | Argon         |
| Flow rate in L/min             | 20            |

2.4. Sessile-Drop Tests
Alterations in the wetting properties of the LIPSS were determined using contact angle measurements based on the “Sessile-Drop”-method. A detailed description of the basic experimental setup is given in [14]. In the present work a highspeed camera Phantom VEO 410 L was aligned perpendicular to the structured surface to record the development, fall and spreading of a single droplet of deionized water. The droplet is produced by a dosing device model 900 E made by GLT and develops at the tip of a cannula with a diameter of 300 µm which is placed 5 mm above the substrate. The scenery is illuminated using an illumination laser CAVILUX HF system made by CAVITAR with a wavelength of 810 nm. The emitted light cone is directed onto a screen behind the sample and thus created a high contrast shadow image of the droplet. Actuating the dosing unit started the high-speed camera to record enabling an evaluation after a certain period. This was chosen to 80 ms since no state changing dynamic was to be detected after this. The contact angle was derived from the certain image using a self-written software program. An overview of the experimental setup is given in Figure 1.

![Figure 1. Overview of the experimental setup regarding the “Sessile-Drop”-method according to [18].](image)

2.5. Confocal Laser Scanning Microscopy and Scanning Electron Microscopy
The results of structuring and hardening were on the one hand analysed with a confocal laser-scanning microscope (CLSM) of type VK-9700 made by KEYENCE. Its fifty-fold magnification was used to record an overview of the altered surfaces.

On the other hand, the Auriga 40 scanning electron microscope (SEM) built by Zeiss was used for a more detailed investigation of the influence of laser radiation during hardening on the LIPSS in the sub-micrometre range. The parameters used to generate the images can be found in Table 5.

Table 5. SEM parameters for imaging the surface and structure alterations due to laser hardening.

| Parameter                       | 1.19k-fold | 6.29k-fold |
|--------------------------------|------------|------------|
| Magnification                  |            |            |
| Working distance in mm          | 11.2       | 11.2       |
| EHT in kV                       | 10         | 10         |
| Signal                         | SE         | SE         |
| Aperture size in µm             | 30         | 30         |

3. Results
The alteration of the surface and thus of the nanostructures due to laser hardening at different laser powers is shown in Figure 2. There, for the laser powers 500 W and 1100 W, the resulting changes in
the nano structured surfaces compared to the reference without hardening are clearly visible. As the power increases, the structures disappear over a wide area, see in Figure 2 the red marked areas. It is additionally to observe that both the number of these areas and their size increase with increasing laser power.

Figure 2. CLSM images of the substrate depicting the surface alterations due to the laser hardening.

The qualitatively structural reduction of the LIPSS is reflected in the change in their heights. Thus, Figure 3 shows that the width of the nanostructures remains unchanged, while there is a decrease in the structure height with increasing laser power.

Figure 3. Alteration of the LIPSS after being exposed to different laser powers during laser hardening at 1.19k-fold ((a) to (c)) and 6.29k-fold ((d) to (f)) magnification.

The quantitative change in nanostructures is shown in Figure 4. There, the height, and width of the structures are plotted against the laser power used. For comparison, the height and width of the unhardened nanostructures are additionally indicated. While the width shows no change on average, the height shows a decrease on average.

Figure 4. Alteration of the LIPSS after being exposed to different laser powers during laser hardening at 1.19k-fold ((a) to (c)) and 6.29k-fold ((d) to (f)) magnification.
Figure 4. Height and width of LIPSS alteration in dependency of the laser power used.

Contact angle determinations carried out with these geometrical prerequisites led to the result shown in Figure 5. While laser hardening has no effect on unstructured substrates and their wettability, the nanostructures hardened by laser show an increase in the contact angle from a laser power of 700 W. The contact angle converges to the contact angle of a drop on a polished and hardened substrate.

Figure 5. Contact angle in dependency of the operated laser power on structured and hardened steel substrates.

4. Discussion
The literature shows that LIPSS are suitable to influence and even change the surface properties. Thus, surfaces that initially exhibit hydrophobic properties change to hydrophilic properties after structuring with LIPSS. However, the literature also shows that these nanostructures do not withstand mechanical wear. As a result, the surfaces provided with them lose the properties previously set via the LIPSS. To
increase the wear resistance of these structures they must be protected. Therefore, they can be put in lubrication pockets as shown by Rathmann et al. [18], or laser hardening can be used to increase the wear resistance, as El-Khoury et al. [16] demonstrated when they hardened DLIP microstructures via laser. Hence, LIPSS were hardened in this work using laser hardening and at different laser powers. Thereby the wettability decreases with increasing laser power. There is, to our best knowledge, no literature dealing with short term heat treatment of LIPSS. Nevertheless, geometrical changes and chemical changes can impair the wettability. It is known that LIPSS change their wettability over time based on changes in the chemical composition of the structured surface, as Kietzig et al. [19] presented. They generated LIPSS at a fluence of 5.16 J/cm² on substrates of 304L and stored them in different atmospheres (air, nitrogen, water, and carbon dioxide). Within five days, the previously hydrophilic behaviour of the nanostructures changed to hydrophobic. Even if the laser hardening was carried out under inert gas atmosphere, a chemical influence e. g. oxidation of the surface cannot be completely excluded. Further the hight of the LIPSS and therefore the aspect ratio changes. This distinguishes the results presented here from those by El-Khoury et al. [16] where there was no effect on geometry, the DLIP structures present there were in the micrometre range and the LIPSS present here are in the nanometre range. Regarding the shape changes, according to Wenzel [20], the surface enhancement affects the wetting behaviour. For the given results, the LIPSS hight decreases when laser hardened with 500 W without a reduction of wettability. The wettability is unaffected up to a laser power of 600 W. Hence, in this range, the geometric effect seems not to have a dominant effect. The shape changes may be a result of local melting, as they appear like those shown in Gregorcic et al. [21]. Nevertheless, this must be clarified further high-resolution images like transmission electron microscopy. Further investigations must clarify which mechanism if geometrical or the chemical effects are the dominating one for the decreasing wettability.

5. Conclusion
In this work, the influence of laser radiation during laser hardening on laser-induced periodic surface structures was presented. These nanostructures initially have a super-hydrophilic behaviour, which, however, changes to a hydrophobic one, like those of polished specimens, with increasing laser power during hardening. Thereby, the geometrical shape of the LIPSS changes. Hence, it is concluded that the thermal load during laser hardening, and it’s influence on the wettability must be considered when LIPSS are subjected to laser hardening.

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