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# Properties of High-Frequency Sub-Wavelength Ripples on Stainless Steel 304L under Ultra Short Pulse Laser Irradiation

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**Abstract**

The paper concentrates on surface texturing on sub-micro meter scale with ultra short laser pulses that has several applications, e.g. changing the hydrophilic/hydrophobic performance, optical or tribological properties of materials. In general, the formations of wavy structures, or ripples on a surface irradiated by short pulse lasers has been observed experimentally since 1965, and are usually referred to as Laser Induced Periodic Surface Structures (LIPSS). Generally Low Spatial Frequency LIPSS (LSFL) and High Spatial Frequency LIPSS (HSFL) are observed. The existing theoretical models do not describe the origin, nor growth of the ripples satisfactorily. That is why the experimental approach still plays a leading role in the investigation of ripple formation. In this paper we study the development of HSFL and LSFL as a result of picosecond laser pulses on a surface of stainless steel. Influences of number of pulses and pulse overlap on ripples growth were examined.

*Keywords*: ripples, ultrashort, pulses, laser

## 1. Introduction

Laser Induced Periodic Surface Structures (LIPSS) have been discovered in 1965 by Birnbaum [1]. LIPSS were observed on a wide range of materials such as metals, semiconductors and dielectrics [2 – 5] and have dimensions in (periodicity and amplitude) which are comparable to, or smaller than the wavelength of the laser radiation. These wave-like surface structures, also called nano-ripples, can be divided into two groups: Low Spatial Frequency LIPSS (LSFL) and High Spatial Frequency LIPSS (HSFL). Usually, the direction of LSFL is perpendicular to the

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polarization of incident laser light and direction of HSFL is parallel to the polarization direction. The most popular theory that describes LSFL is an interference model [2]. This theory interprets the development of ripples as a result of interference between refracted laser beam and surface-scattered waves. However, the interference model cannot give a complete explanation of HSFL. In particular we explored how HSFL evolve into LSFL under influence of applied processing parameters.

## 2. Experimental set-up

Figure1 shows the experimental setup used in this study. An Ytterbium-doped YAG (Yb:YAG) laser source (TruMicro 5050 of Trumpf, Germany) with a central wavelength at 1030 nm was used for generation of the laser pulses with a maximum average power of 50 W. The pulse duration of this laser source is fixed at about 7 ps. The power density distribution of the laser beam is Gaussian. The IR (1030 nm) radiation was converted into green (515 nm) by a Second Harmonic Generator (SHG) unit. The laser beam was linearly polarized. A combination of a rotary λ/2 wave plate and a beam splitting cube served as a power attenuator. In all experiments, the processing direction was perpendicular to the polarization direction and at normal incidence to the surface. Scanning of the beam over the sample was accomplished by a two mirror galvo-scanner system (Intelliscan 14 of ScanLab, Germany). A telecentric f-theta lens (Ronar of Linos, Germany) with focal distance of 80 mm focused the beam to an almost circular spot with diameter of about $d$=18 µm, according to the ISO11146 standard measured by a MicroSpotMonitor (of Primes, Germany). The average power was measured at the exit of the scanner system by a power meter, and set to 10 mW. This results in 200 nJ pulse energy of the laser spot on the surface of the sample for these all experiments. This fluence is slightly above the ablation threshold of the material.

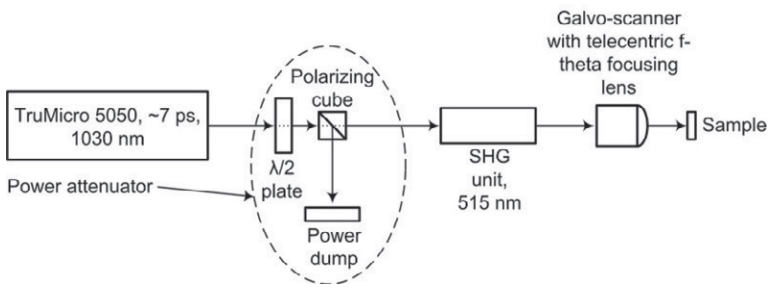


Figure 1. Sketch of the experimental set-up

The pulse frequency of the source was set to 50 kHz and fixed in all experiments. The scan velocity of the scanner was tuned to study the influence of overlap (OL) between subsequent laser pulses. We defined this OL as:

$$OL = \left(1 - \frac{v}{f.d}\right).100\%$$

where d [m] denotes the beam diameter, f [Hz] the pulse frequency and v [m/s] the velocity of the focal spot relative to the material. Hence, for velocities v > f·d, it was assumed that the Gaussian laser spots do not overlap.

The material, stainless steel (AISI304L), was chosen because of its wide use in industry. First the surface of the steel sample was mechanically polished with sandpaper having a grit size of 2500 grooves/mm and subsequently fine polished with a silicon suspension. After that the sample was cleaned by ethanol in an ultrasonic bath. The chemical composition of the material is shown in Table 1:

Table 1. Composition of stainless steel AISI 304 L sample in weight percentage [6]

| AISI 304 L | C% | Mn% | Si% | P% | S% | Cr% | Ni% | N% |
|---|---|---|---|---|---|---|---|---|
| | 0.03 | 2.0 | 0.75 | 0.045 | 0.030 | 18-20 | 8-12 | 0.1 |

The parameter space of the experiments is summarized in Figure 2. Data on the horizontal axis correspond to different scanning speeds, or overlap. For the conditions of our experiments at low scanning speed, the machining results in strongly overlapping laser spots. The increase of the scanning speed leads to no overlap for velocities over 900mm/s. We varied the number of overscans, i.e. multiple scanning of the same laser track, from 1 to 1000 times.

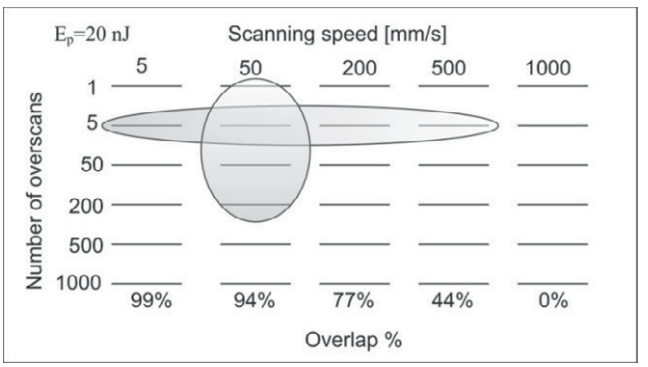


Figure 2. The process window of the experiment

The most interesting results with respect to the initiation and growth of LIPSS were observed in the range of machining conditions marked by the two ellipses in Figure 2. After laser machining the surface of the sample was qualitatively inspected with a Scanning Electron Microscope (SEM), Philips XL 30S FEG. A confocal microscope (Keyence VK-9700K/VK-8700K) was applied for quantitative analysis of the dimensions of LSFL. The most pronounced wavelength (periodicity) of LSFL was derived by 2D Fourier analysis (FFT) of the data obtained with the SEM microscope. However, the periodicity of HSFL was obtained from SEM images, not by 2D FFT, but by averaging the "manually" measured periodicity values.

## 3. Results and Discussion

Figure 3 shows SEM images of ripples observed for different overlap conditions. At an overlap of 44% (Figure 3a) the analysis showed that the periodicity of HSFL is about 120 nm. The predominant direction of HSFL coincided with the polarization direction of laser light. On the fringes of HSFL bubble like structures can be observed, see also [4]. At the overlap of 77% (Figure 3b) there is a coexistence of HSFL (period 120 nm) and LSFL (period 360 nm). LSFL directed perpendicular to the polarization of light. Finally, at high overlap of 99% well developed LSFL were observed. The amplitude of the structures grew with increasing number of overlaps (evidenced by Confocal Microscopy).


Figure 3. The evolution of LIPSS as a function of increasing overlap. The experiment was performed with 5 overscans. Overlap between consequent pulses was: a) 44%; b) 77%; c) 99%. The arrow indicates the polarization direction of the incident laser light.

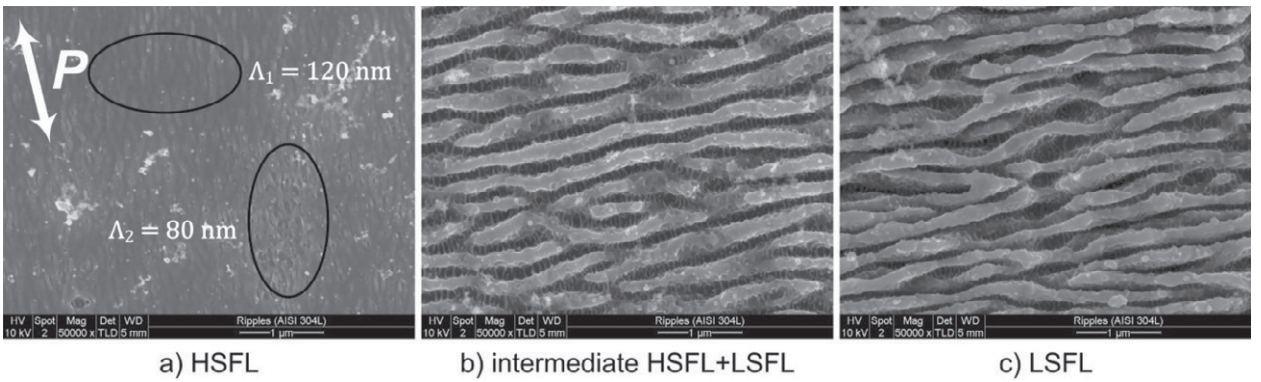


Figure 4. The evolution of ripples as a function of increasing number of overscans is presented. The scanning speed was 50 mm/s implying an overlap of 94%. The number of overscans was: a) 1(ellipses indicate different periodicity (Λ) of HSFL); b) 5; c) 50

Figure4 illustrates the evolution of ripples when the overlap was kept constant (94%) and the number of overscans was varied. After the first overscan, HSFL with a period of about 140 nm was observed (Figure4a). By careful inspection, HSFL with different periodicities can be distinguished, see Figure4a, b. Exposing the sample to 5 overscans results in the formation of LSFL with a typical period of about 360 nm (Figure 4b), which is not surprising because the number of overscans and degree of overlap are about the same as in Figure3c. This corresponds to the intersection area of process ellipses in Figure2. As in Figure3a-c, the direction of the HSFL was the same as the polarization of the incident laser light and the direction of LSFL was orthogonal to it. By careful inspection of the Figure 3b, one can distinguish HSFL among LSFL. After 50 overscans, micro-channels start to occur. With a further increasing number of overscans (more than 100) LSFL structures are destroyed and deep channels are formed.

Generally, our observations are in agreement with earlier experimental findings. The polarization dependencies for HSFL and LSFL are the same [4, 7]. The predominant orientation of HSFL is collinear with the polarization vector, while the orientation of LSFL is perpendicular. For example the alloy 800H sample in [4] was etched and highly polished before the laser machining and irradiated with femtosecond pulses at wavelength 800 nm. The sample of stainless steel 304 industrial BA surface finish was investigated in [7]. In our experiments the surface preparation, laser pulse duration and wavelength were essentially different but we observed the same polarization dependency of LSFL.

In our experiments we found that the presence of surface defects, i.e. scratches indicated by arrows in the Figure 5, ignores the dependence of HSFL direction on the polarization as observed on smooth surfaces. The HSFL are oriented perpendicular to the scratch, independently of the polarization of incident light [8].

From Figure4b it can be observed that intermediate ripples between LSFL are not driven by polarization direction of the incident light [8]. Instead of following the polarization direction they were perpendicular to the sides of LSFL. As it follows from the results of the previous investigations, the absorption of the incident light increases with increasing roughness value of the irradiated surface [9-11]. Each new incoming pulse makes the surface rougher. Thus, it suggests that within a certain range of fluence, LSFL with period comparable with the wavelength of irradiation light are produced. These LSFL start to play a role of scratches. In this intermediate regime, they behave as a micro scratch in Figure 5 determining the direction of HSFL.

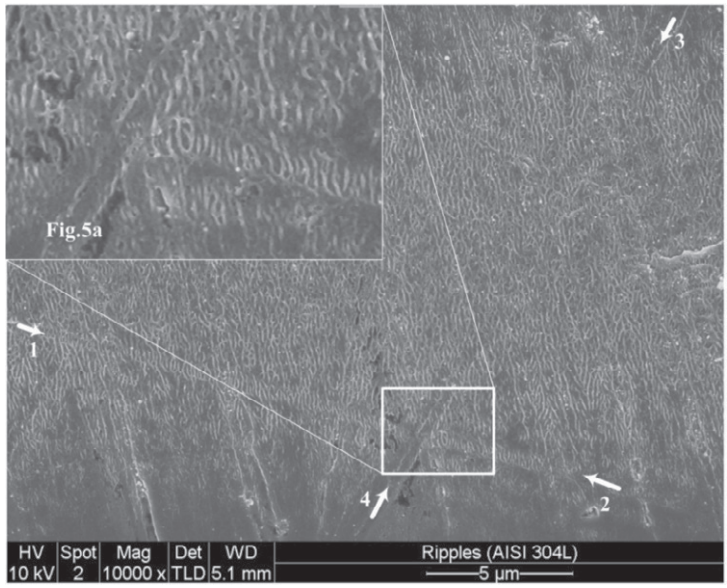


Figure 5 shows stainless steel 304L sample with scratches irradiated by ps-laser pulses with 1030 nm. Energy per pulse was 1 µJ. Arrows (1, 2), (3, 4) indicate beginning and end of scratches respectively. Figure5a presents the magnified area from Figure 5.

If we compare two types of structures that are shown in Figure3a, 4a and 4b one can observe striking differences. The periodicity of HSFL in Figure 3a, 4a is about 120-150 nm and the width of a ripple of about 80 nm. In the case of intermediate ripples the periodicity is about 100 nm and the width is 50 nm. Further increasing the number of overscans leads to channel forming. This appearance can also be addressed to the higher energy deposition with each subsequent overscan of the surface. The inhomogeneity of energy deposition is presented in Figure 4a. Even within the one area shown in the SEM image, HSFL have two periodicities: one about 120 (top ellipse) and other of 80 (bottom ellipse) nm.

It is widely accepted [4, 7, 12] that the periodicity and the height of ripples are changing with increasing number of overscans. However, in our experiments the periodicity of LSFL showed to be independent of number of overscans once they were produced. This will be addressed in future studies. An overview of the measured periodicity is presented in Table 2:

Table 2 Periodicity of ripples under different irradiation conditions

| Overlap [%] | | | | |
|---|---|---|---|---|
| | 44 | 77 | 94 | 99 |
| Over scans | | | | |
| 1 | Cleaning effect | Cleaning effect | HSFL Λ = 140 nm | |
| 5 | HSFL Λ = 120 nm | Transition point<br>LSFL Λ = 360 nm<br>HSFL Λ = 120 nm | LSFL Λ = 360 nm<br>HSFL Λ = 110 nm<br>(intermediate regime) | LSFL Λ = 360 nm |
| 50 | LSFL Λ = 360 nm | LSFL Λ =360 nm | LSFL Λ = 360 nm<br>Channels | Deep groove along the laser track |
| 200 | LSFL Λ = 360 nm | LSFL Λ = 360 nm<br>Channels forming | Deep groove along the laser track | Deep groove along the laser track |

## 4. Conclusion

- LSFL and HSFL have a more or less fixed periodicity; irrespective of the number of overscans applied. The periodicities measured are about 120 nm for HSFL and 360 nm for LSFL, the periodicity does not change with varying number of overscans for the fluence applied. The height of ripples grows with increase of delivered energy.
- Increasing the overlap between subsequent pulses results in the formation of LSFL. Small pulse overlap provides beneficial conditions for generating HSFL. There is a coexistence of HSFL and LSFL when the pulse overlap is in the range of 77 to 94%.
- It seems probable that the direction of HSFL is governed by a different mechanism (scratching) in the area of coexistence with LSFL.

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