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Innovative method to enhance the control over the fabrication of LIPSS on metallic surfaces

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Abstract

LIPSS are an emergent method of fabricating functional surfaces. A high repetition rate ultrashort pulsed laser was used to generate LIPSS on metallic surfaces. Different combinations of the processing parameters were tested to study their effect on the period of the LIPSS. Additionally, the influence of the repetition rate (laser frequency) on the LIPSS period was also studied when the same number of pulses, polarization and peak fluences are applied. Finally, a chemical analysis was performed in order to establish a correlation between the laser processing conditions and the resulting chemical modifications (especially oxidation) in the ultrashort pulse regime. As expected, the analysis shows a decrease in the period of the LIPSS as the number of pulses increases, while the other processing parameters affect the period slightly. Regarding the laser frequency, variations in the repetition rate –while the rest of the parameters are kept constant- only slightly affect the period and morphology of the LIPSS. In the case of the chemical variations, two different regimes were found, with Low Spatial Frequency LIPSS exhibiting no oxidation for low accumulated fluences and High Spatial Frequency LIPSS presenting increased oxidation as the fluences accumulate.

Keywords: LIPSS, femtosecond laser, period, repetition rate

1. Introduction

In the last decades, numerous and intensive studies have been focused on the interaction between ultrashort laser pulses and solid surfaces to create micro- and nanostructures in metals to change their surface properties. In particular, Laser Induced Periodic Surface Structures (LIPSS) have been widely explored to produce surface corrugations for this purpose. LIPSS are an emergent and one-step method of fabricating functional surfaces. Although widely studied in recent years, a fine control of the period of the LIPSS is not achievable yet, as period variations are usually accompanied by other morphology changes. Until now, the most widespread method to obtain LIPSS with different periods while at the same time maintaining the desired morphology has been to change the wavelength, because as reported in bibliography these two features are strongly linked [1]. In this work, we present a different approach to control the fabrication of LIPSS and change their period.

Apart from the aforementioned topographical micro- and/or nano-modifications, most of the available studies report a laser induced modification of the material composition, together with an associated effect on the surface functionalities -either by increasing the wettability [2] or by improving cell adhesion [3] among others-. However, little efforts have been made in order to establish a correlation between the laser processing conditions and the resulting chemical modifications in the ultrashort pulse regime. In this work, we present an EDX and XRD analysis of the oxide formation on Ti6Al4V surfaces as a function of the laser irradiation parameters.

2. Materials and methods

An Amplitude Satsuma HP diode-pumped fiber amplified laser was used to irradiate metallic surfaces in order to create LIPSS on their surface. The laser delivers 280 fs pulses at a central wavelength of 1030 nm (IR), with a maximum repetition rate of 500 kHz, and it is integrated in a station manufactured by Lasea that includes a 3-axis precision stage and a 2D scanner. Processing parameters, such as number of pulses, polarization or peak fluence have been reported to affect the morphology of the LIPSS. Therefore, different combinations of these processing parameters were tested to establish their direct relationship with the period of the LIPSS. Additionally, the effect of the repetition rate was also studied.

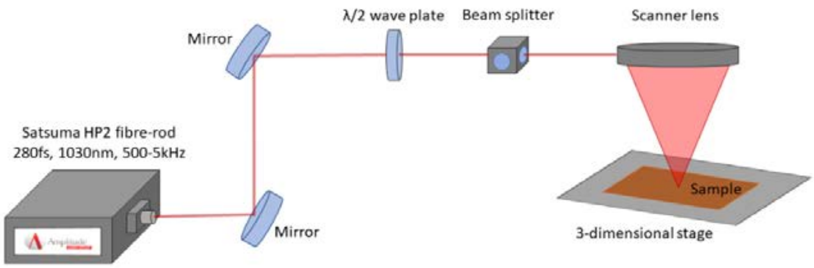

Fig. 1. Setup used in the processing of the samples

2.1. Fabrication process and characterization

Circular polished titanium samples with a diameter of 10 mm were used as the irradiated material. Both before and after the process the samples were cleansed in an acetone and IPA ultrasound baths in order to remove any material and particles generated during the process.

The samples were moved under the laser beam using the 3-axis precision stage, and this was combined with the displacement of the laser beam using the 2D scanner. The samples were irradiated with a minimum fluence of 0.8 mJ/cm^2 and a maximum one of 4.3 mJ/cm^2, while the combined speed of the beam was varied between 10 and 4000 mm/s. To study the influence of the repetition rate, the base frequency was 50 kHz and it was gradually increased up to 500 kHz.

The period of the resulting structures was calculated by means of 2D Fast Fourier Transforms (2D-FFT) from SEM micrographs using a program developed in MATLAB. Regarding the chemical analysis, it was performed with Energy-dispersive X-ray spectroscopy.

3. Results and discussion

3.1. Effect of the variation parameters

Results obtained modifying the fluence and the speed are summarized in the next table. In this case, the frequency has been kept constant at 500 kHz. The most important conclusion that can be drawn from this table is that, when the number of effective pulses per surface unit reaches a certain value, the material is ablated and no LIPPS are formed on it. On the other side, when the number of pulses is too low, no LIPPS are formed and instead a different morphology is obtained, called here “nanobubbles” to differentiate it.

Table 1: Speed vs fluence (f=500kHz)

		SPEED (mm/s)									
		10	20	40	80	160	320	640	1280	2560	4000
FLUENCE (mJ/cm2)	4,30	ABLATION	ABLATION	ABLATION	ABLATION	ABLATION	LIPSS	LIPSS	LIPSS	NANOBUBBLES	NANOBUBBLES
	3,73	ABLATION	ABLATION	ABLATION	ABLATION	ABLATION	LIPSS	LIPSS	LIPSS	NANOBUBBLES	NANOBUBBLES
	3,15	ABLATION	ABLATION	ABLATION	ABLATION	ABLATION	LIPSS	LIPSS	LIPSS	NANOBUBBLES	NANOBUBBLES
	2,58	ABLATION	ABLATION	ABLATION	ABLATION	ABLATION	LIPSS	LIPSS	LIPSS	NANOBUBBLES	NANOBUBBLES
	2,01	ABLATION	ABLATION	ABLATION	ABLATION	ABLATION	LIPSS	LIPSS	LIPSS	NANOBUBBLES	NANOBUBBLES
	1,43	ABLATION	ABLATION	ABLATION	ABLATION	ABLATION	LIPSS	LIPSS	LIPSS	NANOBUBBLES	NANOBUBBLES
	0,85	ABLATION	ABLATION	ABLATION	ABLATION	LIPSS	LIPSS	LIPSS	LIPSS	NANOBUBBLES	NANOBUBBLES
	0,28	ABLATION	ABLATION	ABLATION	ABLATION	LIPSS	LIPSS	LIPSS	NANOBUBBLES	NANOBUBBLES	NANOBUBBLES

In the following images the different results (ablation, LIPSS and “nanobubbles”) can be appreciated.

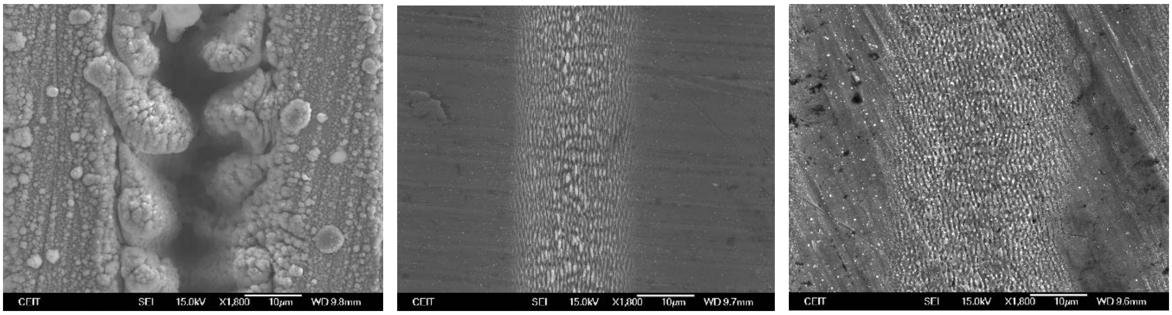

Fig. 2: Different morphologies obtained depending on the number of pulses: ablation (left), LIPSS (middle) and nanobubbles (right).

Focusing on the samples on which LIPSS have been formed, we studied the effect of the number of pulses on the period. The results for the two higher fluences were discarded due to their irregular morphology. The results are displayed in Figure 3.

The graph shows that, when the number of pulses increases, the period of the LIPSS tends to decrease. This is an interesting feature, because it means that this parameter can be used in order to tune the period.

Regarding the effect of the repetition rate (laser frequency), no significant variation has been observed on the period, with the values varying around 30 nm.

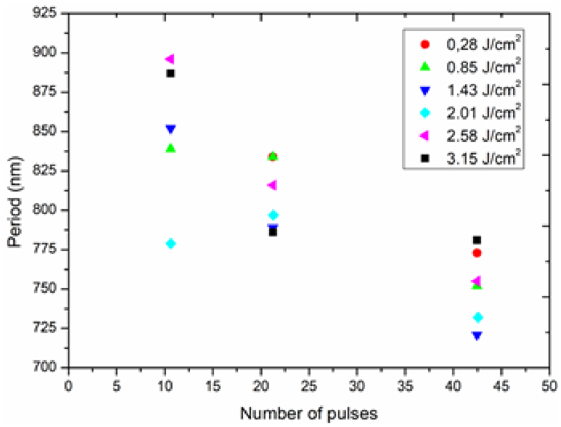

Fig. 3: Effect of the number of pulses on the period of the LIPSS.

3.2. Oxidation

After performing the EDX and XRD analysis onto the LIPPS formed on the surface of the samples, different results were obtained. Two different regimes were found: Low Spatial Frequency LIPSS with no oxide formation for low accumulated fluences and High Spatial Frequency LIPSS with an increasing oxygen content for higher accumulated fluences.

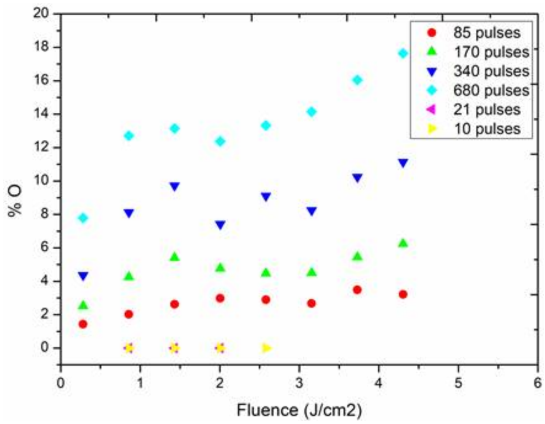

Fig. 4: Effect of the number of pulses on the oxidation.

As observed in the previous figure, at low number of pulses no oxygen is detected on the surface sample, which means that there has not been any oxidation process. At this regime the accumulated fluence is low, and as a result the morphology obtained is formed by Low Spatial LIPSS. On the other side, when a higher number of pulses and, thus, higher accumulated fluences are used, the oxygen content rises significantly. It must be noted that this effect is perceived, albeit in lesser magnitude, even using a low fluence per pulse. This means that, in the cases where oxidation must be avoided, special care must be taken when choosing the processing parameters, because there is a threshold in the number of pulses above which oxidation will occur.

4. Conclusion

In this work we have proven that, modifying the number of pulses when fabricating LIPSS on the surface of metallic materials, their period can be controlled. The periods obtained in this case, for a wavelength of 1030 nm, vary between 725 nm and 900 nm, which means that this parameter can be tuned for different applications in which it is a key.

Regarding oxidation, there is a threshold in the number of pulses above which oxidation occurs. Furthermore, once this value is reached, the increase in the number of pulses increases the oxidation, reaching 18% for high fluences and high number of pulses. Therefore, in applications where changes in surface chemical behavior must be avoided, the method of increasing the number of pulses to vary the period is limited.

References

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