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Fabrication of four-level hierarchical topographies through the combination of LIPSS and direct laser interference pattering on near-beta titanium alloy

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Complex repetitive periodic surface patterns were produced on a near-beta Ti-13Nb-13Zr alloy, using two-beam Direct Laser Interference Patterning (DLIP) employing a picosecond-pulsed laser source with wavelengths of 355 nm, 532 nm and 1064 nm. Different types of Laser-induced periodic surface structures (LIPSS) are produced, including low and high spatial frequency LIPSS, which are observed frequently on top of the line-like DLIP microstructures, as well as quasi-periodic microstructures with periods greater than the laser wavelength. The feature size of the fabricated LIPSS features could be tuned as function of the utilized laser process parameters.

1. Introduction

Periodic surface microstructures are widely used to tailor the properties of functional surfaces, such as enhancing the biocompatibility of materials. Among a large variety of texturing methods, laser based approaches have emerged as a versatile technique for directly functionalizing metallic surfaces in a one-step process. A well-established laser technique involves the fabrication of laser-induced periodic surface structures (LIPSS), which present different morphologies depending on the used material, laser wavelength, process parameters as well as on the pulse-width [1]. These microstructures are commonly generated when ultra-short laser pulses are applied (USP), and the most popular types have been categorized into low spatial frequency LIPSS (LSFL), with periods close to the laser wavelength (Λ_{LSFL} λ), and high spatial frequency LIPSS (HSFL), having periods smaller than half the wavelength ($\Lambda_{HSFL} < \lambda/2$) [1]. LIPSS with feature sizes larger than the laser wavelength have also been reported in the literature and are usually referred to as grooves [2].

A more flexible method for generating periodic and deterministic microstructures is Direct Laser Interference Patterning (DLIP). Through the coherent overlapping of multiple laser beams, an interference pattern is created within the overlapping volume, allowing to ablate the substrate material at the interference maxima positions. In the case that two laser beams are employed, a line-like interference pattern is created and its period (Λ) depends on the intercepting angle (θ) and the laser wavelength (λ) by $\Lambda = \lambda/2\sin(\theta/2)$.

When DLIP is combined with USP lasers, the most common types of LIPSS (LSFL and HSFL) are typically also observed covering the DLIP microstructures [3,4]. For instance, it has been demonstrated that a tailored combination of LIPSS and DLIP can lead to the formation of 2D-structures composed of LSFL perpendicular to the line-like DLIP structure [5], exhibiting anti-bacterial functionality. However, to the best of our knowledge, a combination of supra-wavelength LIPSS with DLIP features has not yet been described in the literature, as well as how the different feature sizes are affected when using the DLIP method.

In this work, we report on the fabrication of a four-level hierarchical structure on near-beta Ti-13Nb-13Zr titanium alloy by utilizing DLIP method with a USP laser source. In particular, the influence of the laser wavelength (355 nm, 532 nm and 1064 nm) and cumulated fluence on the feature sizes of the produced LSFL, HSFL and groove texture is systematically analyzed.

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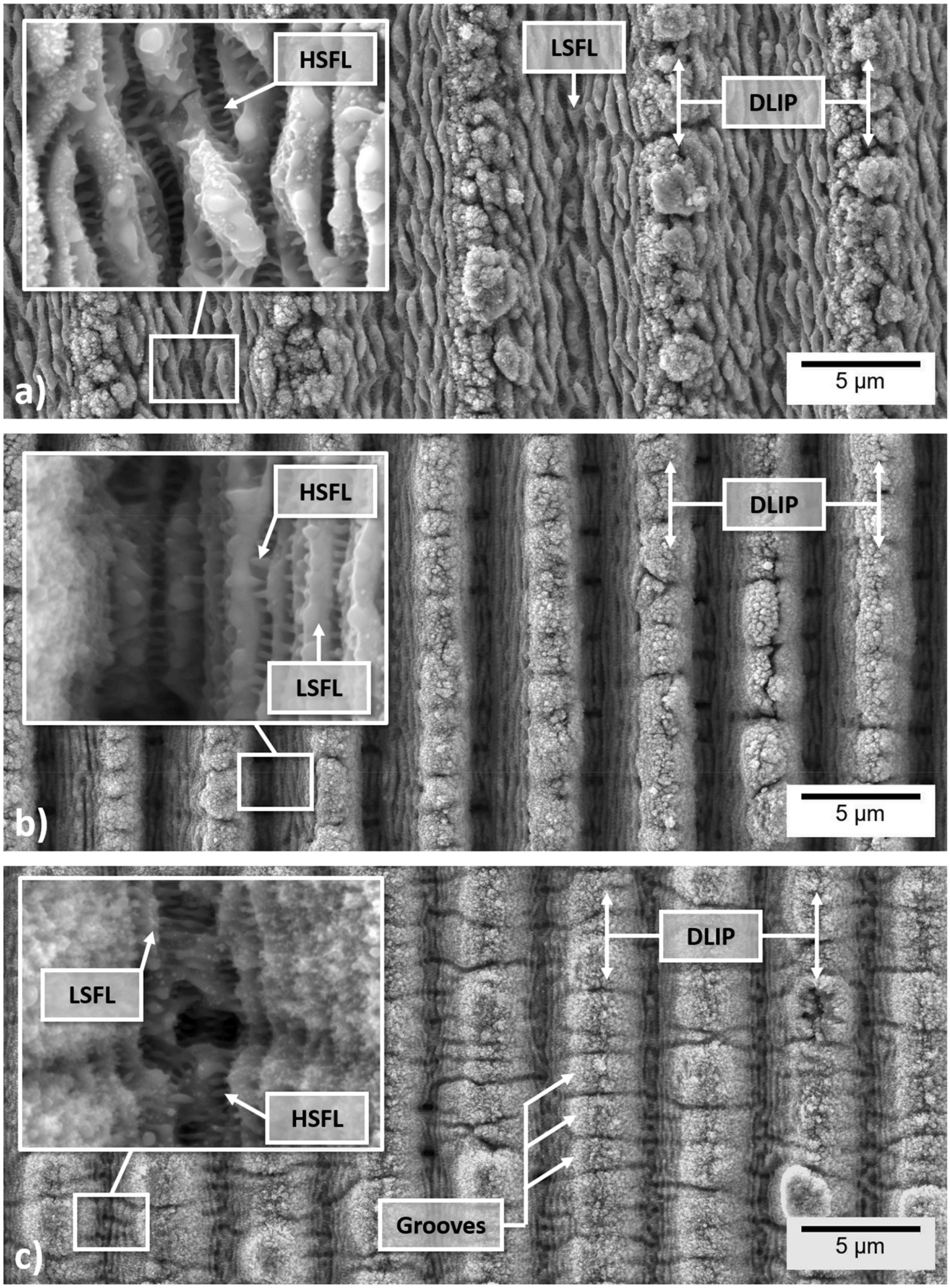

Fig. 1. Top-view SEM images of selected microstructures produced with a) $\lambda = 1064$nm, $\Lambda = 8.5\mu$m, $\Phi_{cum} = 92.3$J/cm^2, b) $\lambda = 532$nm, $\Lambda = 5\mu$m, $\Phi_{cum} = 215$J/cm2, c) $\lambda = 355$nm, $\Lambda = 5\mu$m, $\Phi_{cum} = 87.6$J/cm2.

2. Material and methods

The experiments were performed on near-beta Ti-13Nb-13Zr discs (fabricated through laser powder bed fusion process) with a thickness of 1 mm. The surface was prepared using standard metallographic techniques to reach a surface roughness (Sa) of 0.21 ± 0.05 µm.

The microstructuring experiments were carried out using a pulsed solid-state Q-switched laser source (Nd:YV04, Innoslab PX-200, Edgewave) generating 12 ps pulses at wavelengths of 355 nm, 532 nm or 1064 nm with a TEM_{00} profile ($M^2 < 1.2$). The DLIP-setup consists of a diffractive optical element (which splits the incoming linearly polarized beam into two sub-beams), a prism (which parallelizes the sub-beams) and a converging lens (for overlapping and focusing the beams on the substrate surface). Large areas are treated by translating the sample, using high precision linear stages (Aerotech PRO165-300, USA). The material surface was scanned line-wise, overlapping individual pulses by a freely choosable amount in the scanning direction and laterally superposing lines so that the interference maxima positions are aligned. For the purpose of this work, the term cumulated fluence (Φ_{cum}) was used, which considers the total energy per unit of area provided by the

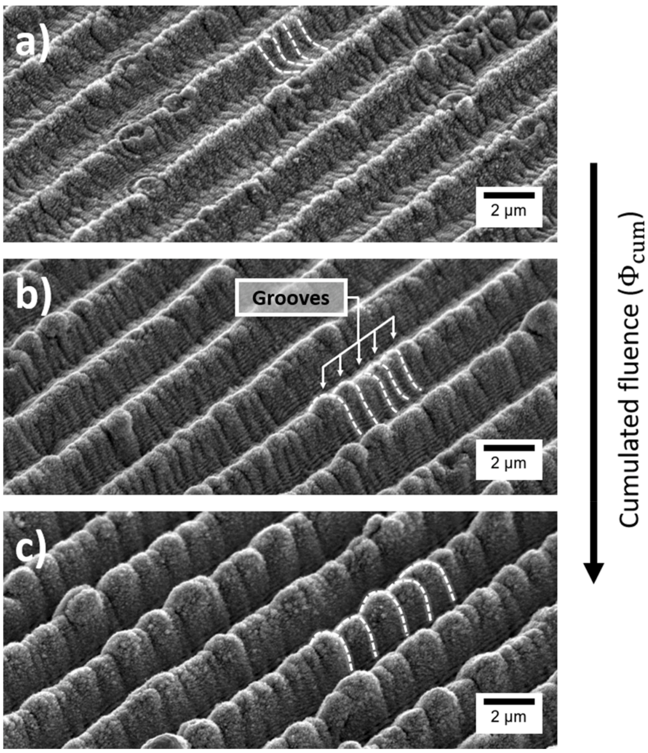

Fig. 2. Tilted SEM images of DLIP microstructures with grooves fabricated with $\lambda = 355$nm, $\Lambda = 5.0\mu m$ and a) $\Phi_{cum} = 32.1$J/cm2, b) $\Phi_{cum} = 59.1$J/cm2, c) $\Phi_{cum} = 98.7$J/cm2.

amount of applied laser pulses to irradiate a certain position.

The sample morphology was characterized using a scanning electron microscope (SEM) operating at 10 kV (7800F, JEOL). The spatial period of the microstructures was measured using the software ImageJ, averaging a minimum of 10 individual measurements.

3. Results and discussion

For the first set of experiments, a broad texturing screening was performed to investigate the variation of the structure morphology with the employed laser wavelengths (355 nm, 532 nm and 1064 nm) and process parameters (laser fluence, pulse overlap and period). Fig. 1a shows line-like textures with a characteristic period of 8.5 μm, fabricated with a wavelength of 1064 nm. The regions within the interference maxima are covered with LSFL, and the regions between the LSFL in turn exhibit HSFL which are perpendicular to them. In particular, the LSFL have a period of ~ 600 nm and are perpendicular to the polarization of the laser beam whereas the HSFL show a period of ~ 200 nm, which is in agreement with reported values in the literature [6]. Fig. 1b and c show surfaces manufactured with a DLIP period of 5.0 μm at wavelengths of 532 nm and 355 nm, respectively. The period of the LSFL features was in this case 336 ± 43 and 268 ± 28 nm, for 532 nm and 355 nm wavelengths, respectively (Fig. 1b, c). In addition, Fig. 1c shows a different type of quasi-periodic structures located at the interference maxima positions, which can neither be classified as HSFL nor as LSFL. These structures are oriented parallel to the laser beam polarization and exhibit periods significantly larger than the observed LSFL features and the used wavelength (355 nm). Furthermore, their average size (1.6 ± 0.4 μm) does not match the pulse-to-pulse distance (4 μm) used in the experiments. Hence, it can be excluded that they are an artifact of pulse overlapping, and they can be classified as supra-wavelength grooves.

Fig. 2 shows different regimes of groove formation on textures fabricated with $\lambda = 355$nm and increasing cumulated fluence (from 32.1 to 98.7 J/cm^2). The grooves are firstly formed in the valleys of the DLIP structure (corresponding to the interference maxima positions) when low cumulated fluences are applied (Fig. 2a). For higher fluences, the grooves are also produced at the peaks of the DLIP pattern (interference minima) and their size increases. The groove features were observed only on DLIP textures fabricated with wavelengths of 355 nm and 532 nm. This can be explained since the needed threshold fluence for groove formation is lower for shorter wavelengths as already reported [7].

Finally, the feature size of all types of produced periodic structures was measured from SEM images (top-view). Plotting the variation of the structure period as a function of the cumulated fluence (Fig. 3), the different characteristics of the produced patterns become visible. While the DLIP-microstructure period remains constant (since it is defined by the intercepting angle θ between the beams, see Fig. 3a), the LSFL and HSFL structures show a dependence on the laser wavelength (Fig. 3b and 3c). Shorter wavelengths induce smaller LSFL and HSFL, which is in good agreement with the literature [6]. In addition, their period is independent of the cumulated fluence. In contrast, the groove structures exhibit a linear dependence with the cumulated fluence, increasing their size from 0.67 ± 0.11 μm at 13.67 J/cm^2 to 3.12 ± 0.81 μm at 175.0 J/cm^2 (Fig. 3a).

The range of groove sizes here reported are in agreement with values found in the literature [7,8]. The increase of the groove period with higher cumulated fluences was, for instance, reported by Allahyari et al., who observed larger features when the number of applied pulses or the laser fluence was increased [7]. The origin of the groove formation is not yet fully understood. Some authors attribute their formation to the aggregation of nanoparticles resulting from the ablation process while others attribute it to inhomogeneous energy absorption [2,8]. However, in our case we can suggest that the increase of the grooves' period with the cumulated fluence can be explained by the bonding of the hills (in our case parallel to the DLIP lines), as has been reported for pure titanium in [9].

4. Conclusions

In this work, multiscale hierarchical periodic surface structures were produced on Ti-13Nb-13Zr samples through DLIP, employing a picosecond laser. LSFL and HSFL features were also observed. In particular, their period depended on the used laser wavelength and did not show any significant dependency on the cumulated fluence. In case of the samples treated with a wavelength of 355 nm and 532 nm, also supra-wavelength grooves were found occurring in parallel to the polarization of the radiation. Their size increased with the cumulated fluence, making this type of LIPSS a potential candidate for fabricating tunable hierarchical structures in combination with DLIP without needing to vary the laser wavelength.

CRediT authorship contribution statement

Frederic Schell: Conceptualization, Investigation, Formal analysis, Visualization, Writing – original draft. **Sabri Alamri:** Investigation, Methodology, Writing – review & editing. **Avinash Hariharan:** Resources, Writing – review & editing. **Annett Gebert:** Supervision, Project administration, Funding acquisition, Writing – review & editing. **Andrés Fabián Lasagni:** Supervision, Methodology, Conceptualization, Writing – review & editing. **Tim Kunze:** Conceptualization, Supervision, Project administration, Funding acquisition, Writing – review & editing.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

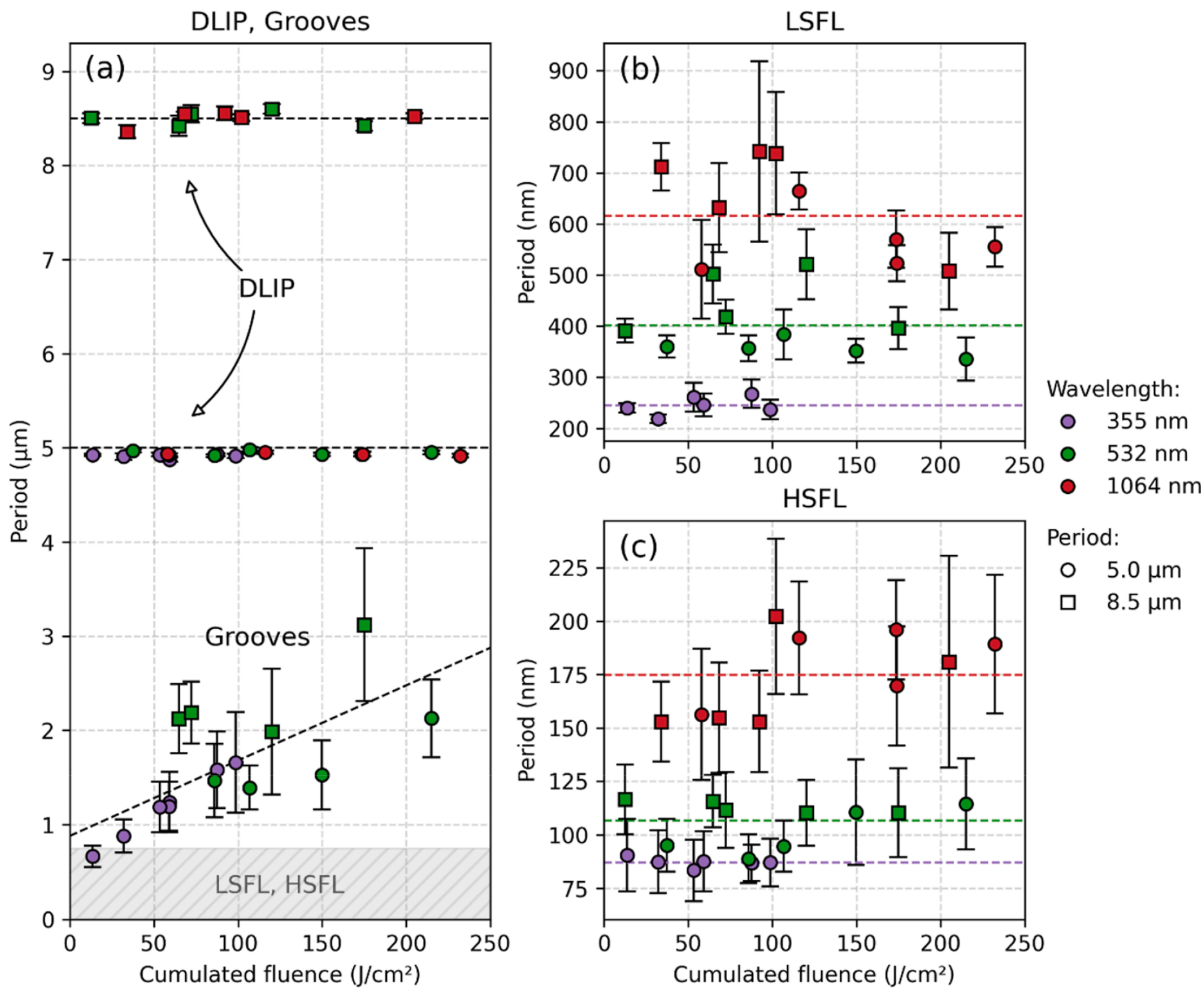

Fig. 3. Periods of different features as a function of the cumulated fluence for a) DLIP and grooves, b) LSFL and c) HSFL. The dots in the different diagrams for the same cumulated fluence correspond to the same surface.

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