Project LaserImplant

D2.1 Images hierarchical spikes and ripples

Reporting period	**from**	**01.01.2021**	**to**	**31.12.2021**
Report completed and released		**30.06.2021** **Jörn Bonse, Martina Muck, Johannes Heitz**		

1. Goals

The deliverable **D2.1** provides a collection of SEM images of hierarchical spikes and ripples on cylindrical Ti-based samples published on the **LaserImplant** web-site (www.laserimplant.eu).

2. Detailed Description

Introduction

There are different ways to structure the side walls of cylindrical metal samples with a laser beam – one of the experimental tasks in the **LaserImplant** project. The most simple and straightforward way is scan with a focused laser beam across the cylinder sample. However, for a cylinder radius exceeding the Rayleigh-length of the focused laser beam, this results in a sample, where at maximum the upper half of the cylinder is laser-structured, while the non-transparent metal cylinder itself shades the laser radiation from the lower part of the cylinder. Moreover, in this approach, the angle of incidence of the incident laser radiation locally varies – depending on the lateral position of the laser beam focus on the cylinder wall – and may result in locally varying spatial periods of the laser-induced periodic surface structures (LIPSS, ripples) or orientation of the hierarchical spikes.

BAM

This problem can be overcome by stepwise rotating the cylinder between different laser-processing sequences. Figure 1 provides an example of a Ti6Al4V titanium alloy rod (grade 5, 6 mm diameter) that was polished in first step in order to reduce the sample roughness. In a second step, the polished surface was processed at **BAM** by a focussed Yb-based disk laser beam (wavelength λ = 1030 nm, pulse duration τ = 925 fs, pulse repetition frequency f = 100 kHz, focus diameter $2w_0$ ~ 35 µm, scan velocity v_{scan} = 700 mm/s, line distance ΔS = 5 µm) at a peak fluence F_0 = 0.5 J/cm^2 and with the linear polarization either parallel or perpendicular to the cylinder axis. The corresponding laser-processed areas indicate the presence of submicrometric LIPSS through their colorized appearance arising from optical diffraction at the grating-like surface ripples.

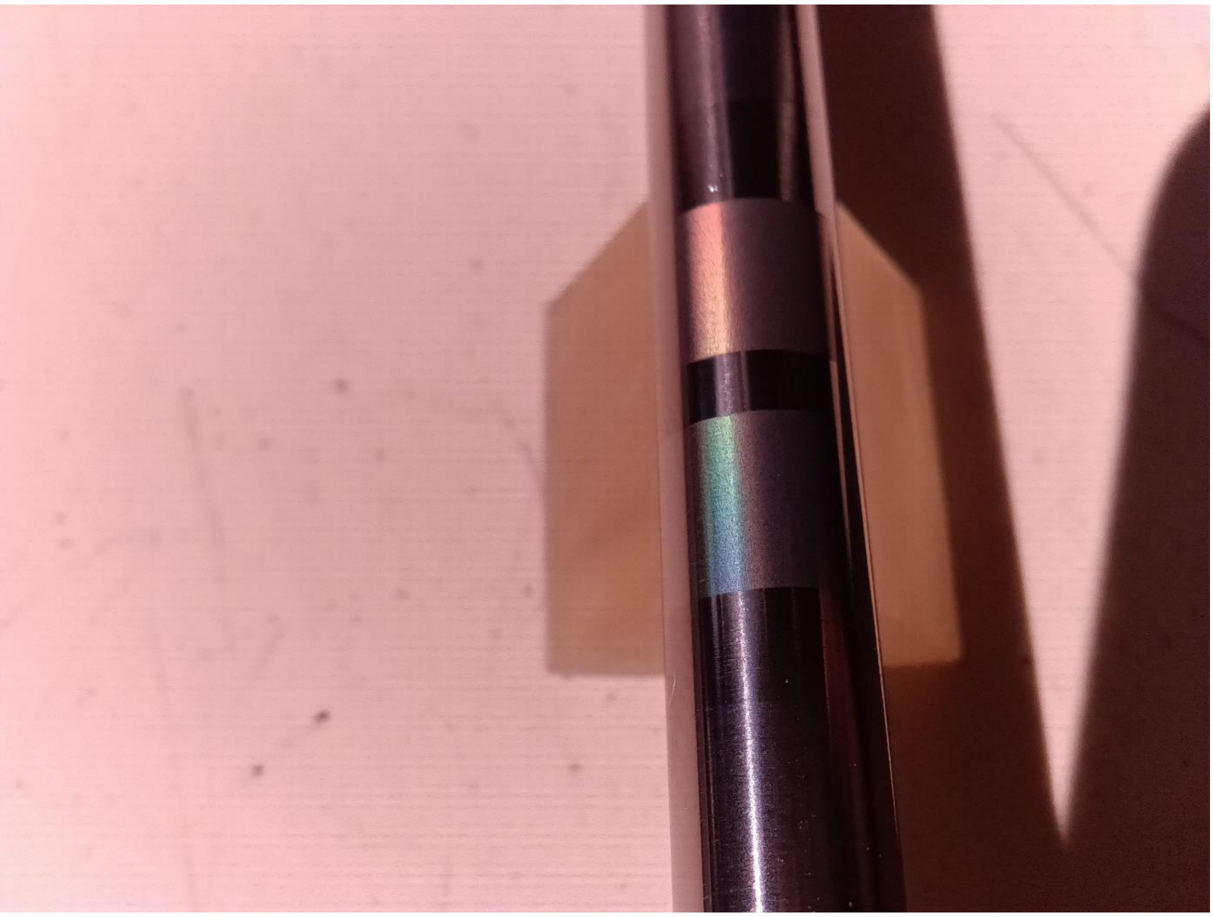

Fig. 1: Photograph of a polished 6-mm diameter Ti6Al4V rod that was laser-processed at two different areas covered by LIPSS (ripples) featuring spatial periods of ~800 nm. The color shade arises from optical diffraction of the illuminating natural sun-light at the surface ripple structures.

For visualizing the ripples, high-resolution optical microscopy was employed in dark-field illumination mode using a long working distance 100× microscope objective. For overcoming the problem of the very limited depth-of-focus in high-resolution microscopic imaging (~1 µm for the given condition), a series of differently focused micrographs (“z-stack”) was acquired and used to process a high-resolution optical micrograph of extended depth of focus, see Fig. 2.

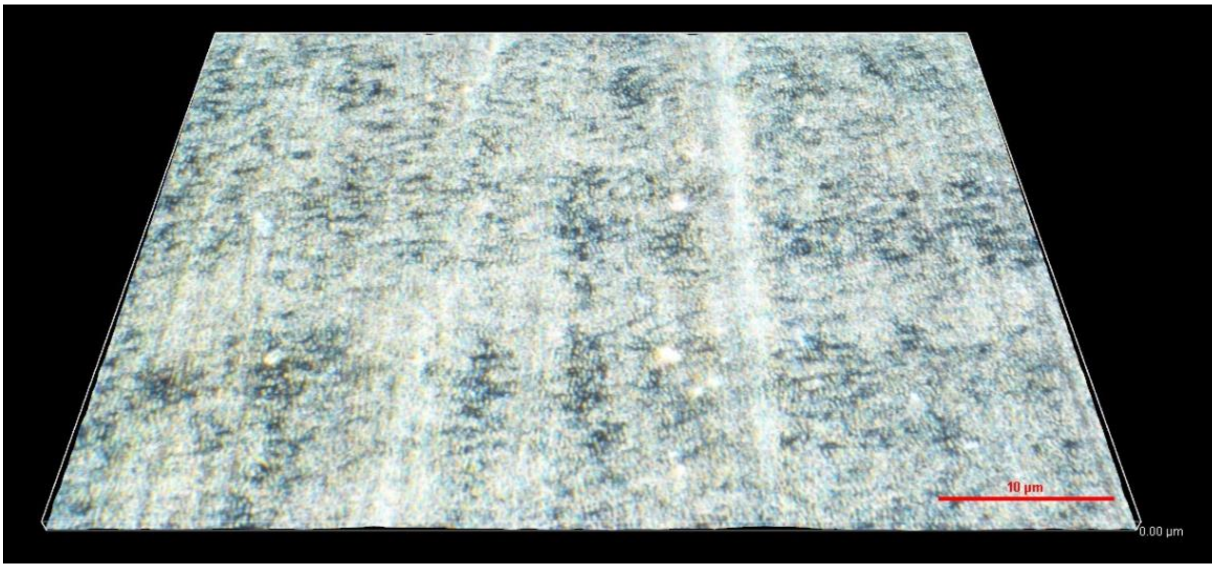

Fig. 2: 3D-view of a z-stacked series of high-resolution optical micrographs of a laser-processed Ti-rod surface previously shown in Fig. 1 featuring LIPSS (vertical) that are oriented perpendicular to the laser beam polarization. The cylinder axis is horizontal. The curvature of the cylinder surface can be recognized at the left/right borders of the bounding box.

Although already close to the optical diffraction limit of the optical microscope, the (nano)ripples are clearly resolved as vertical line pattern, while also the cylinder surface curvature becomes visible at the left/right borders of the bounding box in Fig. 2.

JKU

For the images in this deliverable **D2.1**, **JKU** uses a Yb-based femtosecond laser set-up (Spirit 1040-16 HE, Spectra Physics, wavelength λ = 1040 nm, pulse duration τ = 350 fs) to produce a laser beam that is guided through a system of five mirrors and focused by a lens (100 mm focus length) onto the sample stage, which can be moved linearly in two dimensions. The diameter of the focused laser beam is $2w_0$ = 50 µm ± 2 µm. To be able to produce conical structures with superimposed nanoripples (LIPSS), a parameter set (peak fluence F_0, scanning velocity v_{scan} and line distance ΔS) is determined beforehand based on parameters such as laser type (wavelength and pulse duration), focused beam diameter $2w_0$ and the desired laser frequency f.

For cylindrical Ti-based samples, a continuous rotation during laser structuring was chosen, which was investigated only on titanium cylinders of 8 mm diameter until now, but can be adapted to other cylindrical samples such as bone screws and dental implants. A schematic of the spiral approach for area structuring on cylindrical sample is shown in Fig. 3. While rotating, the sample is moved along the y-axis with a velocity v_{scan} that leads to the desired line distance ΔS. Pulse repetition rate f and y-axis velocity can be chosen according to the desired rotation speed.

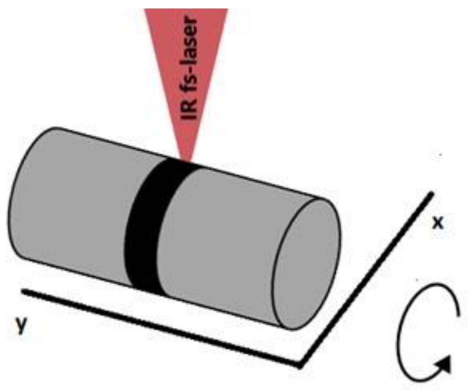

Fig. 3: The left image shows the schematics of continuous laser processing of cylindrical samples. The laser is focused onto the highest point of the cylindrical sample. For continuous laser processing, the sample is irradiated while being rotated and moved along the *y*-axis, leading to a spiral around the cylinder. The right image shows a bone-screw (length 17 mm, diameter 2.5 mm) with a laser-processed ring in black (the blue color of the screw results from the pre-anodization). Figure adapted from [1].

For testing, a rotation speed of 2500 ms/round trip was chosen. Several tests revealed the necessity of a pulse repetition rate f = 33 kHz and a peak fluence F_0 = 4.3 J/cm^2 to produce conical microstructures with superimposed nanoripples. For continuously structuring the sample, a velocity v_{scan} = 6 µm/s along the *y*-axis was chosen to achieve areas with laser line distances of ΔS = 15 µm. Scanning electron microscopy (SEM) images of aforementioned ring on a titanium cylinder are shown in the following Figs. 4 and 5.

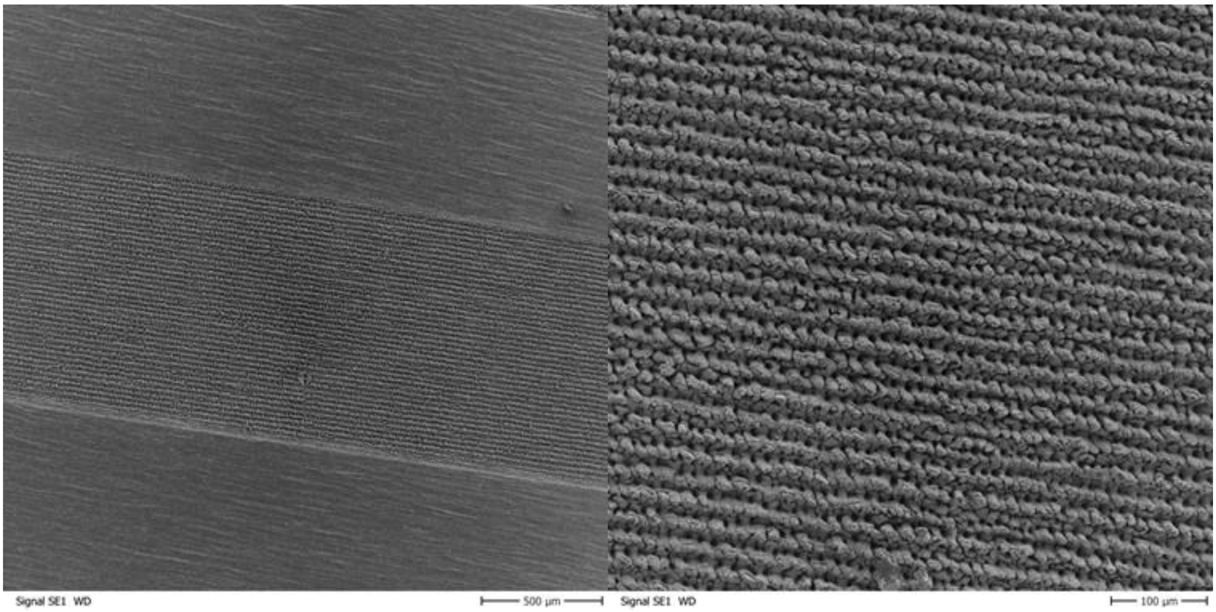

Fig. 4: Overview SEM image of a ring produced by ultrafast laser structuring in a spiral approach (left). Close-up of ring with densely-packed microstructures (right).

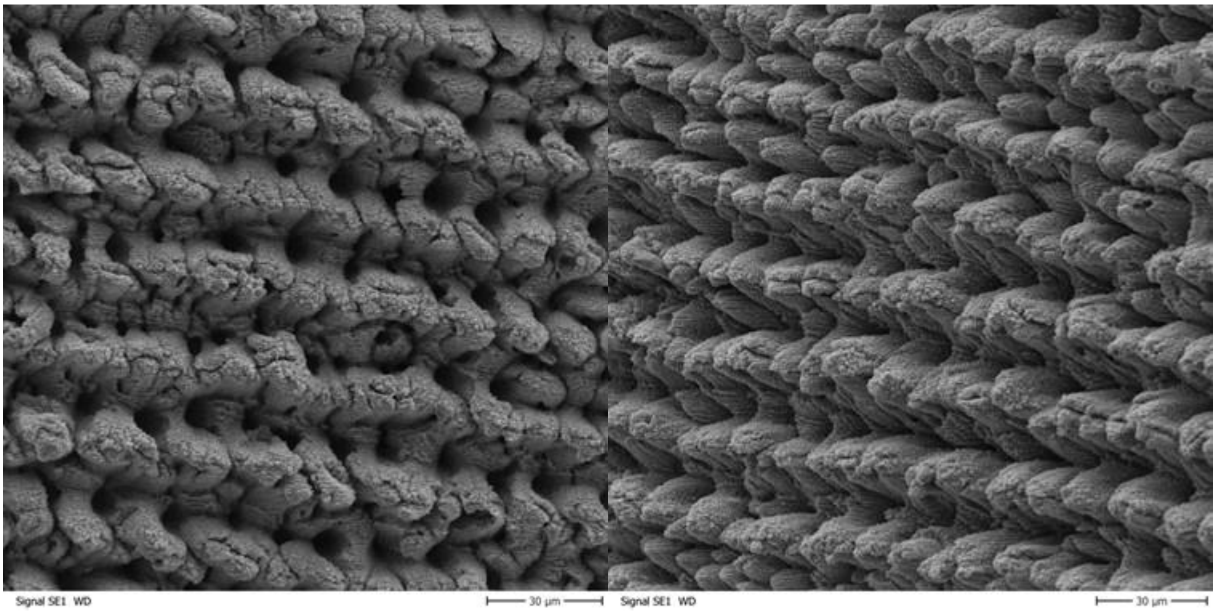

Fig. 5: Magnified view SEM images of microstructures with superimposed nanoripples (left). Corresponding side view of the hierarchical micro-/nanostructures on cylindrical sample (right).

Additionally to cylinders, also pre-anodized Ti-based bone screws (from the product line of **HOFER**) were processed line-by-line (with many pulses per area) with the Yb-based femtosecond laser of **JKU**. Figure 6 shows SEM images of a 1 mm wide ring with peak fluence $F_0 \approx 2.6$ J/cm^2, writing speed of 350 µm/s, a line-to-line distance of 30 µm, and a pulse repetition rate f = 1 kHz. More details can be found in Ref. [1].

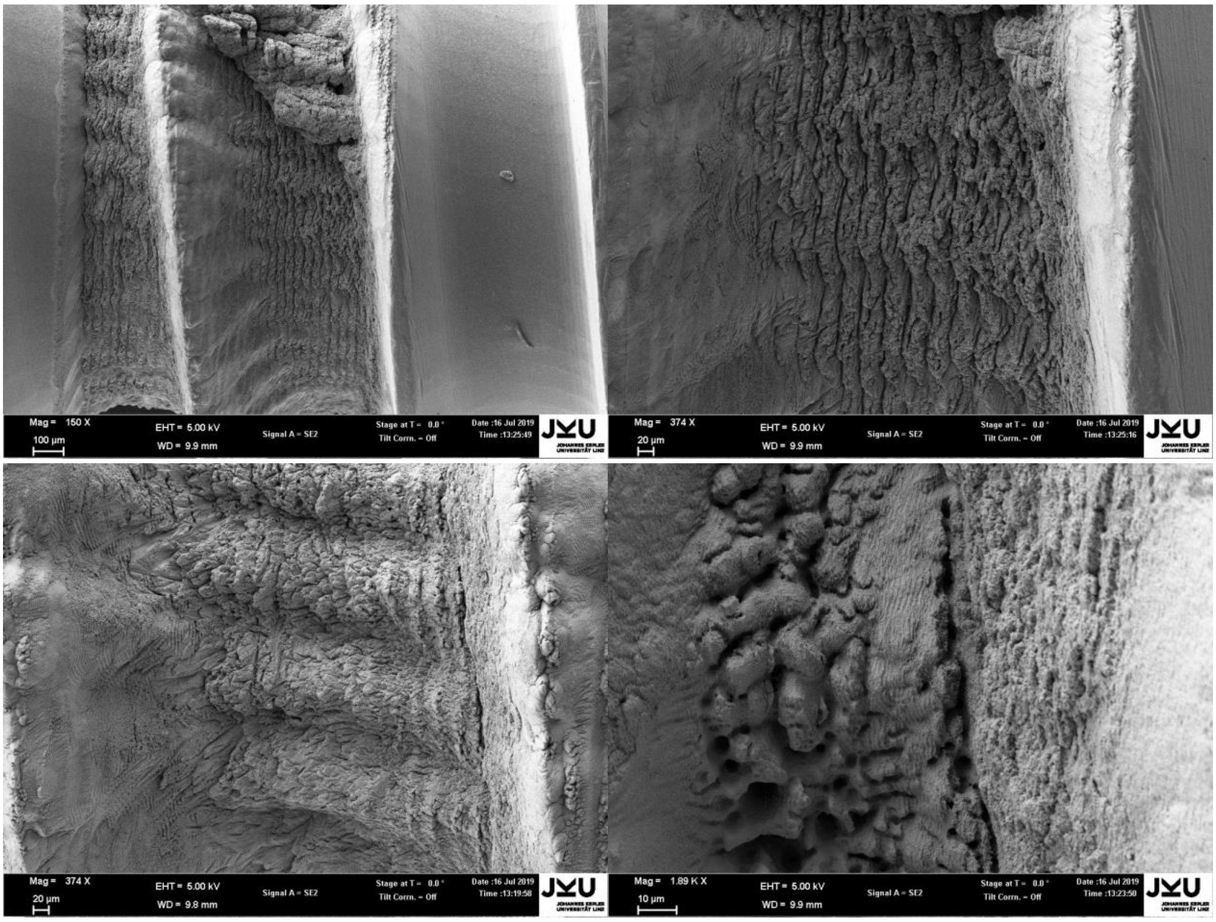

Fig. 6: SEM images of magnified microstructures with superimposed nanoripples on a laser-processed ring on a bone screw. The screw is covered by inhomogeneous structures, as the windings of the screw partially block the laser surface irradiation.

UJM?
[Please add if you can contribute to this deliverable already.]

References

[1] M. Muck, B. Wolfsjäger, K. Seibert, C. Maier, S. Ali Lone, A.W. Hassel, W. Baumgartner, J. Heitz: *„Femtosecond Laser-Processing of Pre-Anodized Ti-Based Bone Implants for Cell-Repellent Functionalization“*, Nanomaterials **11** (2021), 1342.
https://doi.org/10.3390/nano11051342
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3. Evaluation of Goals and Resulting Actions

The deliverable **D2.1 Images hierarchical spikes and ripples** was finalized in time by m6. A link to this report was implemented into the Dissemination the **LaserImplant** web-site (www.laserimplant.eu). A screenshot is shown below.

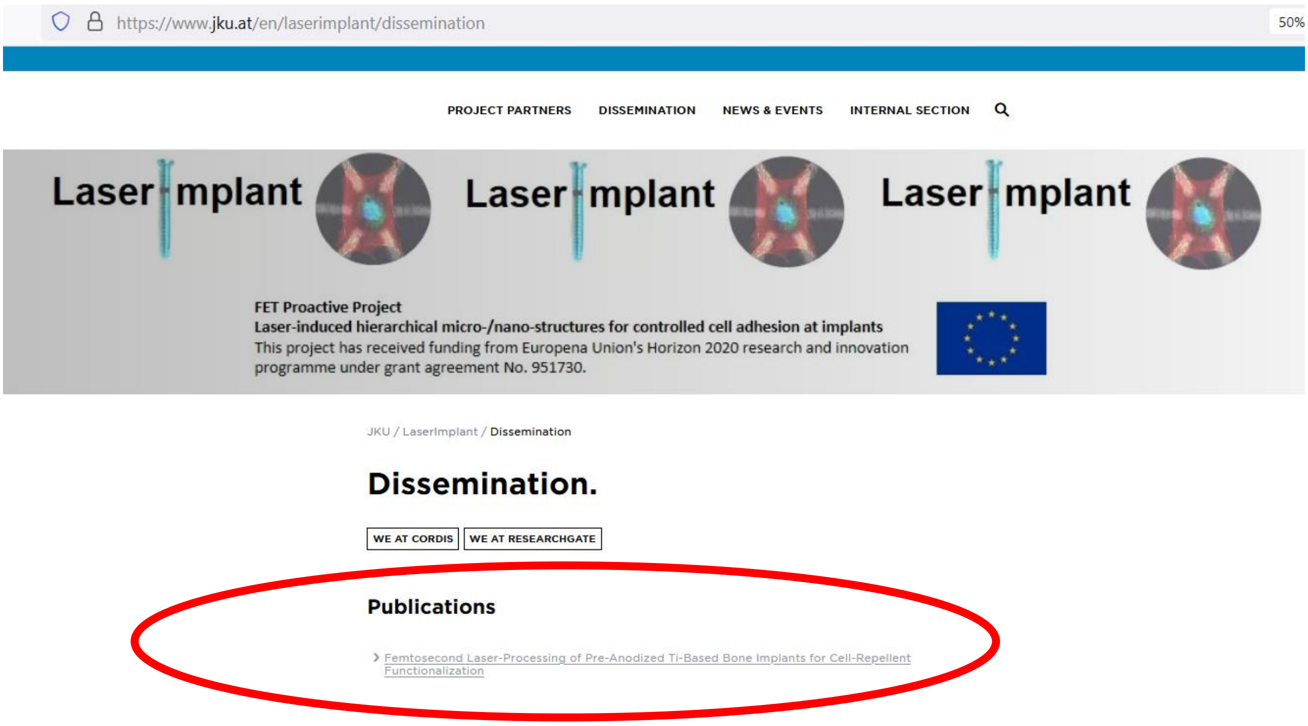

Fig. xxx: Screenshot of the Dissemination section of **LaserImplant** web-site taken on June xx, 2021 {has to be up-dated}.