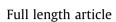
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# Influence of processing parameters on surface texture homogeneity using Direct Laser Interference Patterning

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### ABSTRACT

Surface functionalities in the field of tribology, wettability, biocompatibility and holographic marking introduced by well-defined surface structures strongly depend on the surface texture homogeneity and quality. This work presents strategies for the fabrication of homogeneous periodic surface microstructures employing the Direct Laser Interference Patterning (DLIP) technology with the fundamental transverse mode (TEM00) emitted from a nanosecond laser source. Ti6Al4V substrates are structured using line-like patterns with spatial periods of 7.20 µm, 5.82 µm and 4.31 µm. The impact of various DLIP process parameters such as laser fluence, pulse overlap, hatch distance and spatial period on the produced surface microstructures is introduced and the consequences on the surface texture homogeneity are discussed. Large-area analysis of micro structures is carried out through white light interferometry and scanning electron microscopy. A quantitative measurement scheme of the pattern homogeneity, based on topographical properties such as kurtosis, standard deviation and mean structure height was introduced. Furthermore, the influence of a second modulation arising from the employed hatch distance has been identified. A quantitative parameter, the surface error percentage, has been introduced and employed for the characterization of pattern homogeneity. It was found that specially for larger spatial periods and surfaces treated at high laser fluence, pulse-to-pulse overlaps and a short hatch distance, the overall surface texture homogeneity could be improved up to ~80-90%.

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## 1. Introduction

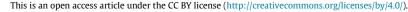
Laser texturing is an effective method for the fabrication of surface structures in a single-step process for a large range of applications [1]. In general, microstructures fabricated by direct laser writing (DLW) on metallic surfaces are produced with laser systems having a Gaussian beam shape (TEM00 beam profile). During nanosecond-pulsed laser surface texturing of metals, different thermal effects take place, resulting for instance in recrystallization, melting and/or evaporation of surface material. The ablation behavior of metals typically depends on the processing parameter where large parts of material can be redeposited on the surface due to the recoil pressure of the vaporized phase [2]. This limits the smallest achievable feature sizes and especially the structure quality. Moreover, the lateral resolution of surface features fabricated by DLW is typically limited to about 10–50  $\mu$ m due to the

features in the range of  $25-250 \ \mu m \ [3-6]$ . Thus, the fabrication of structures with spatial resolution smaller than  $25 \ \mu m$  using nanosecond lasers represents a strong challenge. On the other hand, the fabrication of repetitive microstructures with separation distances (also called spatial period) smaller than  $20 \ \mu m$  are of high interest in a wide range of applications since

with separation distances (also called spatial period) smaller than 20  $\mu$ m are of high interest in a wide range of applications since they provide generally a better performance on different surface functions [7,8]. For example, cell orientation, proliferation and differentiation can be stimulated on biomaterials, especially with feature sizes in the micro- and nanometer scale [9–11]. Another application is related to wettability changes, where the performance of structured surfaces with periodic features below ~20  $\mu$ m in conjunction with large aspect ratios can prevent ice accumulation [12] which represents a high value particularly for the aerospace industry.

wavelength-specific diffraction limit. Thereby, large areas can be processed with limited separation between the produced surface

A technique which allows the fabrication of surface periodical structures in the micro and sub-micro range is Direct Laser Interference Patterning (DLIP). This method has been used for a wide range of applications such as antifouling [13], wetting control







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[14–17], tribology [18] electrical conductivity improvement [19] cell adhesion [20] and decorative elements [21]. In addition, DLIP technology has already been used to treat large areas in 2D and 3D parts with periodic micro and nanostructures [22]. The DLIP process is based on the interference process created by overlapping two or more coherent laser beams and thereby producing a periodic variation of the laser intensity. The versatility of the method, in terms of obtainable pattern geometries, has been evaluated by numerical simulations as function of the number of laser beams used, the laser light polarization, intensity and incident angles for each sub-beam [23,24]. Line-like patterns can be created using a two-beam configuration with a controllable spatial period ( $\Lambda$ ), which is a function of the laser wavelength  $\lambda$  and the half angle between the two incident beams  $\theta$ , as described by Eq. (1):

$$\Lambda = \frac{\lambda}{2\sin(\theta)} \tag{1}$$

As it can be concluded from Eq. (1), the minimal achievable spatial period that can be configured is half of the used laser wavelength. Furthermore, Bieda et al. [25] observed that the structure heights that are obtained when using ns-pulsed laser systems and short spatial periods (< 3  $\mu$ m) are generally limited to a few nanometers for different metals (for example heights of ~35 nm for a spatial period of 1  $\mu$ m on titanium). In addition, when nanosecond pulses are irradiated on metallic surfaces, the structuring mechanism is based on Marangoni convection, which means that the molten material at the maxima positions flows towards the minima positions, following a surface tension gradient induced by the surface temperature distribution [26,27].

In this work, we demonstrate the fabrication of homogeneous periodic surface microstructures employing a DLIP-2-beam-setup with a Gaussian beam intensity distribution (TEM00) of a nanosecond laser on Ti6Al4V alloy. The nanosecond IR source was selected due to the lower cost compared with ps (or fs) lasers sources. However, the improvement of the structure homogeneity is not related with the pulse duration of the laser source but with the strategy used during the experiments. Thus, the impact of various DLIP process parameters such as laser fluence, pulse overlap and spatial period on the produced surface microstructures is introduced and the consequences on the surface texture homogeneity are discussed. Furthermore, a method for measuring the pattern homogeneity based on different topographical characteristics is proposed which allows the quantitative analysis of the surface pattern quality. The here presented method is of significant relevance to assure in the future a certain performance over the whole treated area as well as to permit in relevant industrial processes to quantitatively describe the produced topography in terms of homogeneity. This is required for instance for quality management.

# 2. Materials and methods

## 2.1. Materials

Flat samples of Ti6Al4V (VSMPO, Russia) were selected for the DLIP laser experiments due to their high range of applications [28]. This alloy is used for instance in the aerospace industry as well as in bio-applications due to its lightness, non-magnetic and biocompatibility properties [29]. The annealed samples have a thickness of 2 mm and an average surface roughness (*Ra*) of 0.27  $\mu$ m, the root mean square (*Rq*) roughness of 0.29  $\mu$ m and the maximum height of the assessed profile (*Rz*) of 1.34  $\mu$ m. The material's optical reflectivity at 1064 nm is 57% for a normal angle incidence [30].

#### 2.2. Nanosecond Direct Laser Interference Patterning

The laser experiments were conducted on a compact selfdeveloped DLIP system (DLIP-µFAB, Fraunhofer IWS) producing DLIP pixels (irradiated area per pulse which contains the line-like interference pattern) with a diameter of 160 µm in the interference area (ø). The fluence was calculated by means of the laser beam radius  $(1/e^2)$ , using the D-squared method [31]. The system is equipped with a Q-switched Nd:YLF laser (Laser-export Tech-1053 Basic), operating at 1053 nm that provides 12 ns pulses at 1 kHz with pulse energies up to 290 µJ, emitting the fundamental transverse mode (TEM00) with a laser beam quality factor of  $M^2$ < 1.2. In the DLIP optical head, the main beam is split in two beams using a diffractive optical element (DOE), which are later parallelized by a prism and finally overlapped using a lens with a focal distance of 40 mm. The optical configuration allows the fully automatic control of the spatial period between 1.29 um and 7.20 um by varying the angle of incidence of the beams on the sample, further information about the setup was already published elsewhere [32]. To cover larger areas, the substrates were translated in the vertical (y) and horizontal (x) directions, where the pulse to pulse overlap (y direction) was varied from 90% up to 98.5%. The pulse to pulse overlap (OV) can be calculated as a function of the pulse separation distance p and the beam diameter  $\phi$  using Eq. (2):

$$OV = \left(1 - \frac{p}{\varnothing}\right) \cdot 100 \tag{2}$$

A schematic representation of the main components in the DLIP system is depicted in Fig. 1(a). The strategy used for the DLIP process is indicated in Fig. 1(b), following the same methodology already reported in [33]. By setting the interference angles  $\theta$  to 4.23°, 5.24° and 7.09°, spatial periods of 7.20 µm, 5.82 µm and 4.31 µm can be calculated using Eq.(1). For the given angles, the variation of the reflectivity is negligible and thus do not influence the structuring process (compared to the normal incidence condition (0°), the variation on the total reflectivity is smaller than 0.3% calculated for non-polarized laser radiation and an incident angle of 7.09° [34], using *n* (refractive index) and *k* (extinction coefficient) values of 3.57 and 3.50, respectively [35,36]). The laser experiments were carried out in ambient environment without post treatments.

#### 2.3. Surface characterization

The treated surfaces were characterized using White Light Interferometry (WLI) (LeicaSCAN DCM3D) with a 50 X magnification, with a resolution of 340 nm and 4 nm in the x and y direction, respectively. Using this objective, a total area of 351  $\mu$ m  $\times$  264  $\mu$ m could be recorded in each measurement. In order to evaluate surface homogeneity, an extended area was measured considering four adjacent areas, resulting in a total area of 702  $\mu m \times 528 \; \mu m,$ which was significantly larger than the DLIP pixel diameter  $(\sim 160 \ \mu m)$  produced in the interference area. Along the measured area, five individual roughness profiles were taken (perpendicular to the produced line-like patterns) with a length of  $550 \,\mu m$  each. The extracted profiles were added adjacently, resulting in a sampling length of 2.75 mm. The heights of the profile elements were extracted using the software MountainsMap<sup>®</sup> 7.4 utilizing the step height measurements function. The topographical parameter of kurtosis was determined from the roughness profile according to ISO 4287. Further topographical measurements were carried out using a Scanning Electron Microscopy (Zeiss Supra 40VP) at an operating voltage of 5 kV.

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