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Large Area micro-/nano Structuring using Direct Laser Interference Patterning

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ABSTRACT

Smart surfaces are a source of innovation in the 21st Century. Potential applications can be found in a wide range of fields where improved optical, mechanical or biological properties can enhance the functions of products. In the last years, a method called Direct Laser Interference Patterning (DLIP) has demonstrated to be capable of fabricating a wide range of periodic surface patterns even with resolution at the nanometer and sub-micrometer scales. This article describes recent advances of the DLIP method to process 2D and 3D parts. Firstly, the possibility to fabricate periodic arrays on metallic substrates with sub-micrometer resolution is shown. After that, different concepts to process three dimensional parts are shown, including the use of Cartesian translational stages as well as an industrial robot arm. Finally, some application examples are described.

Keywords: Surface functionalization, Direct Laser Interference Patterning, high speed fabrication

1. INTRODUCTION

Surfaces with controlled topographic characteristics have shown in the past to provide enhanced surface properties in comparison to surfaces with a “random” roughness [1]. Several examples of surfaces with an ordered topography (e.g. periodic surface structure) can be found on the surfaces of different plants and animals, resulting from several thousand years of evolution. In this way, nature has shown to be the best technologist to overcome any survival challenge. In this frame, laser based technologies can provide the required technological and economical aspects to reproduce such surfaces.

One technology capable to produce periodic surface structures is Laser Interference Lithography (LIL) [2]. In LIL, the standing wave pattern existing at the intersection of two or more laser beams is used to expose a photosensitive layer such as a resist. In the case of a negative resist, the positions corresponding to the interference maxima positions are photo-polymerized and after resist development, a periodic variation of the surface topography is obtained. However, the multi-step character of LIL only allows low fabrication speeds and the processing of planar surfaces. Furthermore, this process is very expensive.

An innovative solution for high speed surface patterning of any periodic structure in one processing step is Direct Laser Interference Patterning (DLIP). Here, DLIP enables the formation of periodic patterns having different features with a defined long-range order on the scale of typical microstructures given by the interference periodicity [3].

This technique facilitates various metallurgical processes, such as melting, recrystallization, crystallization of amorphous materials (this aspect was intensively studied in the nineties for Silicon by Stutzmann, Aichmayr or Kelly et al. for example [4-6]) or the formation of intermetallic phases and grain size architectures in microstructures parallel to the surface, but also precise topographies in metals, ceramics or polymers [7-9].

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DLIP makes use of interference of two or more laser-beams, like in interference lithography (LIL), but in this case no development of the irradiated sample is needed [10-13]. The most important requirement to produce the periodic structure with this method is that the material to be processed must absorb the energy of the laser at the selected wavelength and the laser must provide sufficient pulse energy (from tens of μJ to some J) to ablate or modify the material directly. The microstructuring process is based on mechanisms of photo-thermal, photo-physical or photo-chemical nature, depending on the material's type [14].

Under the assumption of plane waves, the total field (E) of the interference pattern can be obtained by the superposition of each individual “ j ” beam [3]:

$$E = \sum_{j=1}^N E_j = \sum_{j=1}^N E_{j0} e^{-ik \sin \alpha_j (x \cos \beta_j - y \sin \beta_j) + \psi_j} \quad (1)$$

where E_j are the amplitudes of electric field of each j -beam; α_j and β_j which are the angles of the beams with respect to the vertical (polar angle) and the horizontal axis (azimuthal angle) of the interference-plane, respectively; ψ_j is the initial phase, and k the wave number:

$$k = \frac{2\pi}{\lambda}, \quad (2)$$

with λ denoting wavelength. Then, the total intensity of the interference pattern can be calculated as:

$$I = \frac{c\epsilon_0}{2} |E|^2, \quad (3)$$

where c is the speed of light and ϵ_0 the permittivity of free space. Using Eq. 3, the interference pattern of n -beams can be easily calculated.

Two-beam interference ($n = 2$) produces a two-dimensional line-like geometry (Figure 1a) whereas three laser beams produce different 2D arrays depending on the magnitude of the electric-field of each beam and the geometric configuration.

Figure 1: Calculated intensity distribution for (a) two-beam interference (d), (b) three-beam interference assuming symmetrical configuration (e), and (c) four-beam interference under the conditions shown in (f) [3].

For symmetric configurations (Figure 1e), the periodic intensity distribution shown in Figure 1b is obtained. The complexity of the patterns can be further increased by using additional laser beams, such as the four-beam interference pattern shown in Figure 1c. For this symmetric configuration, the periodic intensity distribution shown in Figure 1f is obtained. The complexity of the patterns can be further increased by using additional laser beams.

Within the last 10 years, DLIP has not only shown to be capable of producing functionalized surfaces but also an impressive technological development has been observed, permitting to bring this technology to real industrial applications. The recent progresses of this technology are summarized in this article, presenting different application examples as well as the processing of 2D and 3D surfaces.

2. RESULTS AND DISCUSSION

2.1 Direct Laser Interference patterning of 2D and 3D parts

2.1.1 Fabrication of periodic patterns on metals with sub-micrometer resolution

The interference of two or more coherent laser beams can be applied to produce periodic patterns with spatial periods in the micrometer and sub-micrometer range if ps-laser systems are used. In the case of metals, DLIP is based on a photo-thermal process that involves local melting and/or selective ablation at the interference maxima positions. For nanosecond laser pulses, the primary material removal mechanism is ablation but substantial melting occurs. The minimum pitch is therefore limited by the thermal diffusion length, which is roughly 1 μm for stainless steel and titanium and 2 μm for copper [15, 16]. When the laser pulse width reduces to the order of picosecond or femtosecond, little thermal damage is observed and pitches below 1 μm are feasible.

In our experiments, a Yb:YAG laser system operating at 515 nm wavelength with a 35 ps pulse width was used. It is evident that for this laser wavelength the minimum possible pitch is 258 nm (for $\theta = 180^\circ$). The structuring of the metallic samples was conducted either using one or two laser pulses. The repetition frequency of the laser was 1 Hz.

Figure 2 shows fabricated periodic structures on Cu, stainless steel and Ti. The images reveal that well defined periodic patterns could be fabricated on all materials, especially when using a single laser pulse. The measured spatial period was approximately 700 nm corresponding to an intercepting angle of $\sim 43^\circ$. For the periodic patterns produced using two or more laser pulses, a distortion of the pattern was observed, especially for Cu. This can be explained by the lower melting point of Cu compared to Ti and stainless steel substrates. However, in all cases it is evident that the materials have been molten even when using the short laser pulses (35 ps).

Figure 2. Periodic line-like structure upon DLIP on a) Cu, single pulse; b) stainless steel, single pulse; c) Ti, Single pulse; d) Cu, two pulses; e) stainless steel, two pulses; f) Ti, two pulses.

2.1.2 Direct Laser Interference Patterning of three dimensional parts

In addition to two-dimensional parts, DLIP can be also utilized for the treatment of three-dimensional elements. Three different strategies are possible.

The first strategy consists on translating the part or the DLIP optical head vertically and adjusting the z position accordingly to the geometry of the part (Figure 3a). In this case, the curvature of the surface described by the inclination angle γ has a significant effect on the spatial period. If the laser beams used to obtain the interference pattern are collimated (no variation of laser beam diameter with position) and the periodic lines are perpendicular to the curvature direction, the spatial period can be described using Eq. 4:

$$\Lambda(\gamma) = \frac{\Lambda(at \gamma = 0)}{\cos \gamma}, \quad (4)$$

where $\Lambda(\gamma=0)$ denotes the spatial period when the part is perpendicular to the interference pattern volume and can be calculated for a two-beam interference pattern using Eq. 5:

$$\Lambda = \frac{\lambda}{2 \sin \alpha}. \quad (5)$$

Later, the percentage of variation of the spatial period $\Delta\Lambda(\%)$ can be calculated as function of γ using Eq. 6:

$$\Delta\Lambda(\%) = \left(1 - \frac{1}{\cos \gamma}\right) \cdot 100\%. \quad (6)$$

This variation is shown in Figure 3b. As indicated by the previous equation, the spatial period variation does not depend on the initial spatial period ($\Lambda(\gamma=0)$) and is only a function of the curvature angle. This behavior was experimentally confirmed using the experimental setup shown in Figure 3a for 2, 4 and 8 μm spatial periods using a DLIP- μFAB System (Fraunhofer IWS). In this case, the interference pattern was projected perpendicularly to the symmetry axis of a PET cylinder. Then, a laser pulse was irradiated to produce the periodic surface pattern at different positions and the spatial period was measured as function of the angle γ .

Figure 3. (a) Experimental setup utilized for the treatment of a cylindrical PET part using a DLIP optical head (Fraunhofer IWS). (b) Calculated and measured deviation in the spatial period as function of the curvature angle γ for 2, 5 and 8 μm spatial periods.

The results indicate that the percentage of deviation is below 6.4 % for angles of inclination smaller than 20° . This means that for 3D parts with slightly curvatures ($0\text{--}10^\circ$) can be treated using this strategy. On the other

hand, and especially for parts to be treated with large interfering angles (e.g. for fabricate periodic patterns with a spatial period smaller than $1\text{ }\mu\text{m}$), the vertical position of the part (or the DLIP optical head over the 3D part) has to be perfectly controlled to assure the overlap of the individual laser beams for each z-coordinate.

If three dimensional parts with a higher angle of curvature have to be treated, an industrial robot can be utilized, providing the required flexibility. This strategy is of great advantage when treating very complex geometries. In our experiments, a Kuka KR 60 HA robot arm was utilized. The main advantages of this system are the large working space ($2033\text{ mm} \times 2230\text{ mm} \times 2429\text{ mm}$) and a maximal linear speed of 2000 mm/s . On the other hand, drawbacks of the robot are low path and position repeatability ($\pm 160\text{ }\mu\text{m}$ and $\pm 50\text{ }\mu\text{m}$, respectively). However, the main objective in this study was to evaluate the proof of principle of the method.

The schematic setup for this strategy is depicted in Figure 4a. The DLIP optical head (developed at Fraunhofer IWS) was fixed to the robot arm together with the laser system (ns-pulsed Nd:YLF laser Tech-263 from Laser-Export Ltd) as well as the power unit supply. Because of that, there are not limitations of the position of the optical head as well as the angles which can be used to treat the parts.

Figure 4. (a) Used experimental setup of DLIP using a robot arm. The DLIP optical head (Fraunhofer IWS) is fixed to the robot arm (Kuka KR 60 HA) together with the laser system (Nd:YLF laser Tech-263, Laser-Export Ltd) as well as the power unit supply. (b) Optical micrograph of the cylindrical PET treated part and the used DLIP optical head over the cylinder. (c) Treated PET cylinder at different positions corresponding to different linear speeds. The inset show the structures produced at 400 mm/s with an positioning error of $\sim 90\text{ }\mu\text{m}$.

For demonstration of this strategy also a cylindrical PET part was used (Figure 4b). Different trajectories were programmed in order to cover areas of approximately $1\text{ mm} \times 31.4\text{ mm}$ corresponding to half of the cylinder's perimeter. Three different processing linear speeds were chosen: 200 mm/s , 400 mm/s and 600 mm/s . The size of the structured area per laser pulse was $\sim 2043\text{ }\mu\text{m}^2$ corresponding to a spot diameter of $51\text{ }\mu\text{m}$ (Figure

4c). The results show deviations of 60 μm , 90 μm and 131 μm accordingly to the above mentioned linear speeds. Clearly, for the lowest linear speed produced the smallest positioning error ($\sim 60 \mu\text{m}$). This error can be attributed to the accuracy limitations of the used system but can be improved in the future by using more accurate robots (e.g. IRB120 from ABB with a repeat accuracy of 10 μm). Because in this case the DLIP optical head was placed perpendicular to the position to be treated, no variations in the spatial period were observed ($< 0.5\%$).

Finally, a third strategy designed especially to treat symmetrical parts was developed. Two different examples are shown in this section. Similarly to the previous case, using the DLIP- μFAB system, a PET sample was processed (Figure 5). Because of the high absorption of this material in the UV-range, also the 263 nm laser system was used. The DLIP- μFAB system was equipped with a rotation stage where the PET sample (a typical 500 ml PET bottle) was fixed. Accordingly, the PET bottle was translated with a linear stage and rotated to produce three decorative elements (LMO, Fraunhofer IWS and DLIP- μFAB logos) directly on its surface. Due to the high accuracy of the used translational stages and rotation axis, the accuracy between the treated area (holographic pixel) per laser pulse was better than 1 μm ($\sim 0.5 \mu\text{m}$).

Figure 5. (a-b) Treated PET bottle using a DLIP- μFAB system (Fraunhofer IWS). The accuracy of the system is below 1 μm . The system is equipped with 4 axis including 200 mm x 200 mm x,y translational states and a rotational axis (360°). The DLIP-optical head is mounted over a z-translational stage with travel length of 100 mm.

2.1.3 Direct Laser Interference Patterning of sleeves for roll-to-roll hot embossing

For the case that larger parts must be treated, a special DLIP system (developed at the TU-Dresden) can be used as shown in Figure 6. This system permits to treat cylindrical parts up to 600 mm in length and 300 mm in diameter. The system is equipped with both ns and ps- laser systems. Similarly like in the DLIP- μFAB system (Figure 5c), different optical heads can be utilized. Preliminary results of a treated Ni-sleeve (300 mm length and 300 mm in diameter) are shown in Figure 6b. Four different treated regions can be observed corresponding to patterns produced using different laser parameters. In Figure 6c, also the possibility to produce dot-like geometries on the Ni-sleeve using three laser beams is also shown. The spatial period in this case was approximately 5 μm with a structure depth (ΔZ) of 1.2 μm . The structured sleeve was later utilized for printing a PET polymer foil using a Roll-to-roll hot embossing system. The temperature was fixed to 85°C . The obtained structure on the PET foil can be observed in Figure 6d. The negative shape of the initial pattern can be observed and the structure height was in this case 0.6 μm .

Figure 6. (a) Large area DLIP system for the processing of 2D and 3D parts. (b) 300 mm Ni-sleeve (300 mm in diameter) processed with ns-DLIP optical head. (c) Fabricated dot-like geometry on Ni-sleeve using three-beam interference and (d) its imprint on a PET-foil using a Roll to Roll hot embossing system.

2.2 Application examples

2.2.1 Controlling wetting properties of DLIP treated Polyurethane

It is well established that micron sized topographies greatly affect cell-surface interactions [17] such cell adhesion, morphology, orientation, migration and differentiation [18, 19]. In addition, polyurethanes (PU) are one of the most popular groups of materials used in medical devices. They offer wide versatility in terms of tailoring their physical properties, blood and tissue compatibility and, more recently, their biodegradation character [20].

A two-beam Direct Laser Interference Patterning setup was used to fabricate line-like patterns on PU substrates using 266 nm laser radiation. Typical images of the line-like patterns produced with different spatial periods are shown in Figure 7. As it can be seen, by using moderate energy densities (ranging from 0.15 to 0.59 J/cm²) well-defined and homogeneous patterns could be fabricated. The total structured area patterned per pulse was approximately 16 mm² resulting from the overlap of two square like shaped beams with a size of ~4 mm x 4 mm (see Figure 7a).

During the short-pulse UV laser treatment process, the sinusoidal energy distribution of the interference pattern is directly transferred into the PU as consequence of material ablation at the positions corresponding to the interference maxima (Figure 7b, e). For relatively high laser fluences and especially for small spatial periods, the quality of the periodic structure reduces by showing a significant number of particles (probably resulting from the stronger ablation process) as well as non-straight lines (see Figure 7d). The maximal structure depths obtained using one laser pulse varied between 0.88 up to 1.25 μm for spatial periods between 2.0 and 5 μm and up to 270 nm for spatial periods between 500 nm and 1.0 μm [21].

For characterizing the wetting behavior of the patterned and non-patterned polyurethane samples, sessile drop measurements were conducted. Thereby, the water drops were seated on the substrates and their static contact angle across and along the line features was analyzed. Figure 8a shows the contact angle values measured across the line-like features as a function of the filling factor (FF) for spatial periods between 2 μm and 5 μm . The filling factor depicts the

ratio between ridge width (FWHM) and period, in which $FF = 1$ corresponds to the smooth surface. Non-patterned PU exhibits a slightly hydrophilic surface showing a contact angle of $81 \pm 2^\circ$. As it can be seen, the measured contact angle was increased by the different periodic structures (spatial periods from 2 to 5 μm) and shows a linear correlation with FF. The maximal observed contact angle was of $102 \pm 2^\circ$ for the 3 μm periodic structure with a FF of 0.41 [21].

Such behavior can be explained by heterogeneous wetting on microstructured surfaces, where the liquid water drop builds up a composite interface with the solid tops of the ridges and the air in between these features (contact angle: 180°) [22]. This leads to a reduction of the surface energy and a larger averaged contact angle between the liquid and its counterparts which can exceed 90° . Thus, an originally hydrophilic material may become liquid repellent due to micro structuring of its surface [23]. In addition, the contact angle is scaled up with similar slopes for different periods by decreasing the filling factor, which is related to the reduction of droplet seating area on the surface (liquid/solid interface) and increase of liquid-gas interface.

Figure 7. DLIP ablation experiments on castor oil based polyurethane films. In (a), both structured and non-structured areas are shown. The total irradiated area per laser pulse was $\sim 16 \text{ mm}^2$ (b-d) SEM images of patterned PU surfaces with a spatial period of (b) 5 μm and (e, f) 500 nm. The following average laser fluences were used: (a) 0.70 J/cm^2 , (b) 0.58 J/cm^2 , (c) 0.15 J/cm^2 , (d) 0.37 J/cm^2 [21]

However, from Figure 8a it can also be observed that periodic structures with similar FF but different spatial periods present differences in the wetting behavior. Periodic patterns with 2.0 and 3.0 μm spatial periods show higher contact angles than surfaces with 4.0 and 5.0 μm . This might be explained by a discrepancy between the filling factor based on the measurement of FWHM and the actual drop seating area (see inserts in Figure 8a). The periodic patterns produced in PU with 2 and 3 μm periods exhibit a sinusoidal structure shape which means that the actual drop seating area is smaller than the area calculated at FWHM. At larger periodic distances (4 and 5 μm periods) this difference is smaller because the structure morphology is less tapered (sigmoidal shape).

A different behavior was observed for the 500 nm and 1.0 μm periodic surface patterns (see Figure 8b). Although the wetting angle is partially increased on 500 nm spatial period, it does not exceed 90° indicating a still hydrophilic PU surface. For the 1.0 μm patterns, the contact angle was even reduced compared to the reference value of $81 \pm 2^\circ$. This droplet behavior on sub-micron structures can be explained by the Wenzel theory [24]. In this case, the original hydrophilic PU becomes more hydrophilic due to the produced surface roughness because the structure is not capable to

retain air between the topographic features, leading to a homogenous contact between the water droplet and the PU surface. The reason could be related to the significantly smaller structure depth (generally smaller than $0.2\ \mu\text{m}$) compared to those obtained for larger periods (from 0.26 to $1.25\ \mu\text{m}$).

Figure 8. Contact angle of water depending on the filling factor of the structures. (a) $\Lambda = 2.0\text{--}5.0\ \mu\text{m}$. (b) $\Lambda = 0.5\text{--}1.0\ \mu\text{m}$. The contact angle was measured across the lines. The dashed lines in (a) are only to guide the eyes [21].

2.3 Controlling tribological performance

The efficiency, reliability, and durability of machine components depend on friction occurring at the sliding contact interface [25]. Inefficient lubrication may result in high friction and wear losses. This can adversely affect the fuel consumption of engine systems and powertrain components [26–28].

Figure 9. Friction coefficients obtained from ball-on-disk experiments for unstructured (reference) and structured samples (lines, crossed-lines, and dots). The samples were lubricated with oil (10W-60) and the sliding speed was $1.8\ \text{cm/s}$. The samples were patterned with three laser pulses at $1.2\ \text{J/cm}^2$.

DLIP was used to fabricate periodic line-, cross-, and dot-like structures on polished 100Cr6 steel samples. In these experiments, the utilized range for pulse energy density was from 0.8 to $1.6\ \text{J/cm}^2$ and the pulse number was varied

between 1 and 3. Both energy density and the number of laser pulses were varied in order to obtain a sufficient structure quality, and to generate surface topographies with a significant difference in the structure height.

The friction tests were carried out at three different radii at constant rotation frequency (1.75×10^5 cycles) for different values of sliding speeds ($v_1=0.6$, $v_2=1.8$, and $v_3=3.0$ cm/s). Figure 9 shows the recorded data of the friction coefficient for both a non-patterned (reference) and patterned samples (lines, crossed lines, and dots) at $v_2=1.8$ cm/s (recorded distance ~ 1594 m). The patterns were fabricated using three laser pulses at 1.2 J/cm² to obtain aspect ratios (structure depth over spatial period) of 0.14 (lines), 0.06 (crossed lines), and 0.08 (dots) [29].

In Figure 4 can be observed that the friction coefficient of the non-patterned 100Cr6-surface increases before reaching its maximum at approximately 35 000 cycles, followed by a steady decline. The fluctuations of the curve can be attributed to the presence of wear particles between the frictional partners. Differently, the coefficient of friction (COF) evolution of the patterned surfaces is more stable, showing only minor fluctuations within the investigated amount of cycles. The patterned samples appear to reach the steady state immediately, which indicates a lower surface roughness of the patterned areas after local laser melting. It is evident that the COF of the patterned samples is significantly lower than for the non-patterned. Ultra low friction with a friction coefficient of $\mu=0.015$ was obtained for crossed lines, and dot patterns, whereas it was 0.02 for the line-like pattern at the end of the measurement. The smaller friction coefficient of the structured 100Cr6 surfaces can be explained as a result of diminished adhesive interaction due to a reduced contact area at the ball/surface interface [29].

3. CONCLUSIONS

The results here reported demonstrate the capabilities of the Direct Laser Interference Patterning method to produce periodic surface patterns on 2D and 3D parts. The use of pulsed ps-laser systems with especially designed optical heads permitted to produce surface patterns on metallic substrates with sub-micrometer resolution (spatial period ~ 700 nm). The strategies developed to treat three-dimensional parts included the use of Cartesian translational stages with and without a rotation axis as well as an industrial robot. Furthermore, the possibility to process large sleeves (up to 600 mm length and 300 mm in diameter) was shown as well as its use for imprinting polymer foils using a roll-to-roll hot embossing system. Finally, two different application examples were introduced, including the modification of the wetting properties of Polyurethane substrates as well as reduction of friction coefficient on 100Cr6 steel. The obtained results show the flexibility of this technology for processing several materials as well as improving different surface functions.

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