

Bringing the Direct Laser Interference Patterning Method to Industry: a One Tool-Complete Solution for Surface Functionalization

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Direct Laser Interference Patterning is an emerging technology capable to produce micro- and nanostructured surfaces on a wide variety of materials. Although, this method has shown to be capable to produce functionalized surfaces in a one-step process, it will have impact on real applications only if compact systems and solutions are developed. This study shows recent developments of interference patterning optical heads capable to produce periodic patterns at high fabrication speeds (0.1 – 5 m²/min) as well as to solutions to be integrated in roll-to-roll processes. A concept of an interference patterning system (DLIP- μ Fab) is also described. Application examples showing control of tribological properties of 100Cr6 steel, cell orientation on patterned polyimide surface as well as the fabrication of high resolution decorative elements are discussed.

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1. Introduction

Surface functionalization is a key discipline in order to bring new properties to common materials. In several cases, the ability to produce surface structures at very small (sub-micrometer) length scales as well as controlling the chemical and/or physical properties of surfaces at this level is crucial.

A possibility to fabricate these surfaces is provided for example by laser processing techniques. Laser based fabrication methods offer several advantages due to their remote and thus contactless operation, their flexibility during materials processing as well as their precise energy deposition. At the micro scale, direct laser writing (DLW) has been utilized to pattern and process several materials with features generally between 1 and 100 μ m [1, 2]. In DLW, nanosecond (ns) pulsed laser systems are often used. Recently, also laser systems providing picosecond (ps) and femtosecond (fs) pulses have gained interest at the industry scale due to the development of fiber based systems. During the structuring process, the component surface is scanned with the laser beam using galvanometric mirrors. Achieving resolutions lower than 5 μ m is normally associated with an increased technical complexity and long processing times (see Figure 1). With a laser pulse frequency of 1 MHz for example, 10⁶ structures per second can be written sequentially. With a structure size of 1 μ m, this is equivalent to a processing speed of only 6 · 10⁻⁵ m²/min.

Other techniques such as conventional micromilling processes can achieve higher surface structuring speeds (~10² m²/min) but with a limited resolution, generally between 15 and 100 μ m.

Electron and Ion-Beam lithographic methods [3] have been also used to fabricate high resolution surface patterns with feature sizes even smaller than 100 nm. On the other side, several hours up to several days are required for the processing of 1 m² areas (see Figure 1). In addition, almost all lithographic methods are limited to planar surfaces.

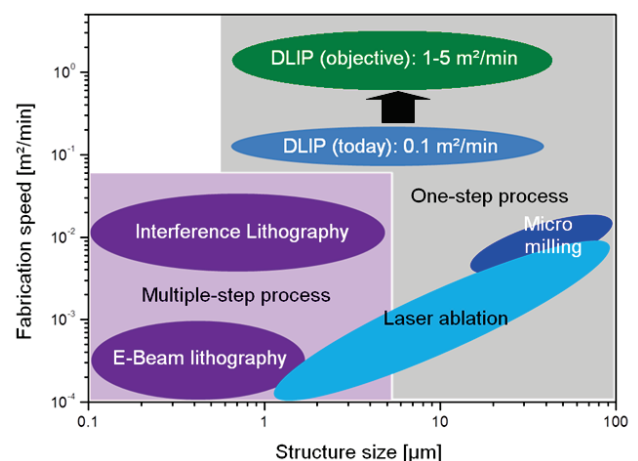


Fig. 1 Achievable technology dependent surface fabrication speed as a function of structure size.

In order to solve this problem, techniques involving large-area surface processing for rapid fabrication of two and three dimensional (2D and 3D) structures are required.

One example is Laser Interference Lithography (LIL) [4]. In LIL, the standing wave pattern existing at the intersection of two or more laser beams is used to expose a pho-

tosensitive layer such as a resist. In the case of a negative resist, the positions corresponding to the interference maxima positions are photopolymerized and after resist development, a periodic variation of the surface topography results. Although this method has been already applied for the treatment of large areas, the multi-step character of LIL only allow low fabrication speeds. Furthermore, the method only allows for the processing of planar surfaces.

An innovative solution for high speed surface patterning of periodic structures in a one step process is Direct Laser Interference Patterning (DLIP). Here, periodic structures can be produced in different materials including metals, ceramics, polymers and coatings in a single step process [5-7].

To obtain an interference pattern, the primary beam of a laser system is split into two or more beams to interfere with each other on the sample surface (Figure 2). By the combination of a polarizer and a half-wave plate it is also possible to adjust the intensity impinging on the sample. The intensity can also be adjusted by selecting beam splitters with a specific reflection/transmission ratio. Also a mechanical shutter can be used to control the number of pulses reaching the sample. By a two-lens telescope it is possible to change the diameter of the laser beam and therefore the energy density received on the sample surface. To reduce influences from the optical elements, high flatness of the splitters and mirrors can be utilized.

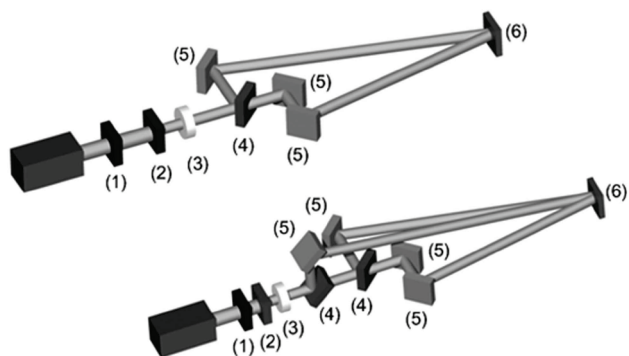


Fig. 2 Conventional two and three laser beam interference setup for ns-pulsed laser systems. The intercepting angle between the beams and the laser wavelength determine the spatial period of the interference pattern. (1-2) Beam expander; (3) mechanical shutter; (4) Beam-splitters; (5) Mirrors; (6) Substrate.

Up to now, processing speeds of about 0.1 m²/min with sub- μ m resolution have been achieved. However, the DLIP technology can be scalable at least one order of magnitude to achieve record processing speeds between 1 and 5 m²/min (Figure 1). However, the first step to improve this technology consists on developing compact DLIP systems that can be operated at normal atmospheric conditions.

In this study, the fabrication of spatially ordered structures with micrometer and submicrometer length scales on different materials at high surface processing fabrication speed is demonstrated. Firstly, geometrical constructs of DLIP optical heads developed at our group are described. In addition, a compact Direct Laser Interference System (DLIP- μ FAB) recently developed at Fraunhofer IWS is introduced for the first time. After that, different application examples of structured surfaces are described. The

applications include the development of advanced surface patterns for tribological applications, the fabrication of structured polymer films for cell orientation, as well as the direct fabrication of decorative elements on technological steels.

2. Direct Laser Interference Patterning optical heads and Systems

In the last years, different interference patterning optical heads have been developed [8]. These optical heads offer the possibility not only to process planar surfaces but also complex three dimensional mechanical components including mechanical seals, piston rings, and bearings [9].

Three different options are available. The first alternative is designed for the fabrication of a fixed structure geometry type (e.g. dot or line-like pattern) as well as a fixed structure period. An example of such a system to produce 1.5 μ m line-like periodic arrays in substrates up to 300 x 300 mm² is shown in Figure 3a. The DLIP optical head is located in this case 500 mm above the working plane with a typical head size of 150 x 200 x 300 mm³. This alternative is preferable for those applications requiring high speed surface patterning.

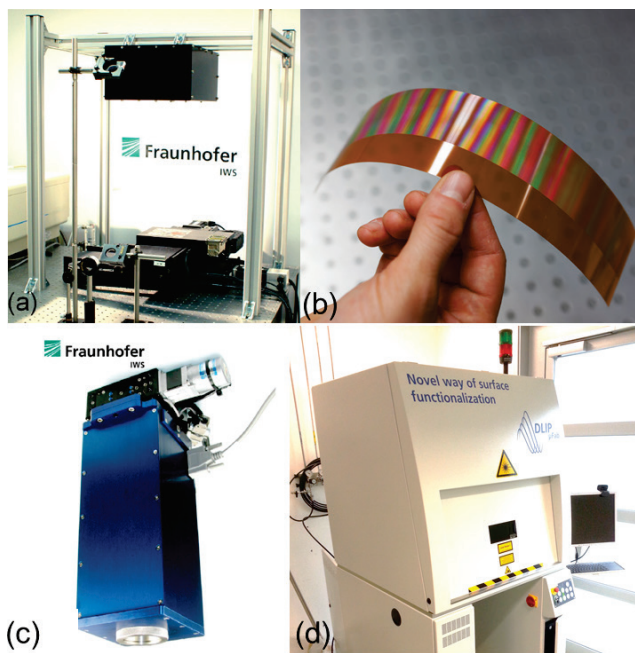


Fig. 3 Developed optical heads and direct laser interference systems at Fraunhofer IWS. (a) DLIP system for the processing of 300 x 300 mm² substrates with a fixed structure geometry and period (in this case, line-like array with 1.5 μ m spatial resolution). (b) Polyimide foil processed with Roll to Roll DLIP optical head. (c) Newly developed DLIP-head that permits variation of spatial period and pattern orientation. (d) DLIP- μ FAB System equipped with IR (1053 nm) ns-laser system.

The second alternative was developed for the large area structuring of foils on roll to roll processes. Similarly to the first case, the DLIP optical heads are designed for the fabrication of a fixed geometry and spatial period. Up to now, foils with widths up to 200 mm have been processed. However, it can be scalable to larger sizes if enough laser power is provided to process a certain material. An example of a

treated polyimide foil using this configuration is shown in Figure 3b.

The third configuration includes the use of newly developed DLIP-heads that permit to vary the structure period and pattern orientation during the structuring process. Therefore, a large variety of surface structures with different functionalities can be produced with only one system. The developed optical head is shown in Figure 3c. The shown optical system permits to vary the spatial period from 0.40 to 3.75 μm at a working distance of 35 mm (wavelength: 532 nm). The size of the DLIP head is 125 x 115 x 210 mm^3 .

A complete solution including the DLIP optical head, translational stages and automatized software for multi-geometry surface periodic arrays is shown in Figure 3d. The shown system (DLIP- μFAB , Fraunhofer IWS) is equipped with a 5 ns pulsed laser system operating in KHz range at 1053 nm of wavelength, and is suitable for the direct processing of metallic substrates (e.g. steels, nickel) [10]. Also laser systems with wavelengths in the green and ultra violet as well as picosecond pulsed laser systems can be integrated.

3. Application examples

3.1 Control of tribological properties on 100Cr6 Steel

The efficiency, reliability, and durability of machine components depend on friction occurring at the sliding contact interface [11]. Inefficient lubrication may result in high friction and wear losses. This can adversely affect the fuel consumption of engine systems and powertrain components [12, 13]. Due to more strict regulations to reduce carbon dioxide (CO_2) emission from cars, innovative solutions must therefore be developed to improve the tribological performance of mechanical components. Surface structuring has been found beneficial for improving the tribological properties of many mechanical components. Due to the intrinsic characteristics of the DLIP technology (high processing speed and resolution), primordial investigation concerning the fabrication of textured surfaces for tribological applications were conducted.

The DLIP experiments were performed with two and three beam configurations as shown in Figure 1, obtaining periodic line- and dot-like structures on polished 100Cr6 steel samples. The laser wavelength was set to 355 nm and intercepting angle between the laser beams was set to 2.0° and 2.3° for the two and three beam configurations, respectively, obtaining 5.0 μm spatial periods. The pulse energy density was varied from 0.8 to 1.6 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. After measuring the structure height, the aspect ratio defined as the quotient between the structure height and the spatial period, was determined for all processing conditions. Examples of line and dot-like surface patterns are depicted in Figure 4. The white arrows depicted in the figure indicate the direction of height measurement to determine the aspect ratio.

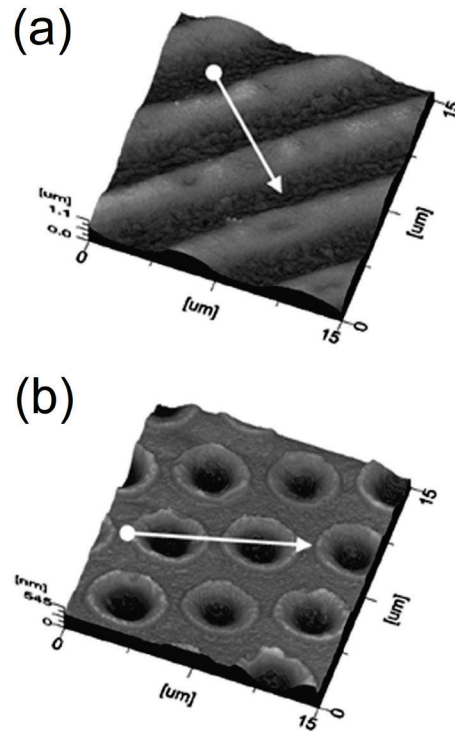


Fig. 4 Atomic Force Microscope images of 100Cr6 structures using (a) two and (b) three laser beam interference setup. The arrows indicate the direction of height measurement to determine the aspect ratio [15].

The tribological performance of the patterned surfaces as function of the structure aspect ratio (AR) is shown in Figure 5 (lubricated conditions, ball-on-disk configuration (100Cr6 vs. 100Cr6) in rotating mode at room temperature, SAE10W-60 oil). v_1 , v_2 and v_3 denote the linear speed during the tribological measurement. It can be seen that the coefficient of friction (COF) has a minimum value if the AR is between 0.07 and 0.11 for line- and dot-like patterns. Thus, the best performance (i.e., the smallest COF) was achieved for an optimal value (AR_{Opt}). If the AR is smaller or bigger than AR_{Opt} , the friction coefficient increases. In the case of $\text{AR} < \text{AR}_{\text{Opt}}$, the AR approaches the limit, which is determined by the reference surface (non-patterned sample). Conversely, if $\text{AR}_{\text{Opt}} < \text{AR}$, the slope of the microstructures and the COF increases. This behavior can be explained by calculating the hydrodynamic pressure for an arbitrary surface roughness using the Reynolds equation. For a given AR, the contact area between the friction partners decreases with increasing hydrodynamic pressure, which consequently results in a minimum amount of friction (see reference [14] for further details).

3.2 Cell orientation on periodic structured polymer substrates

According to the most recent studies, cells can actively adapt to the surface on which they adhere, activating specific intracellular signals that can influence cell survival and behavior [7]. Particularly, several reports have shown that geometrical parameters (depth and spatial period) of periodic surface topographies determine the cellular response to the patterned surfaces.

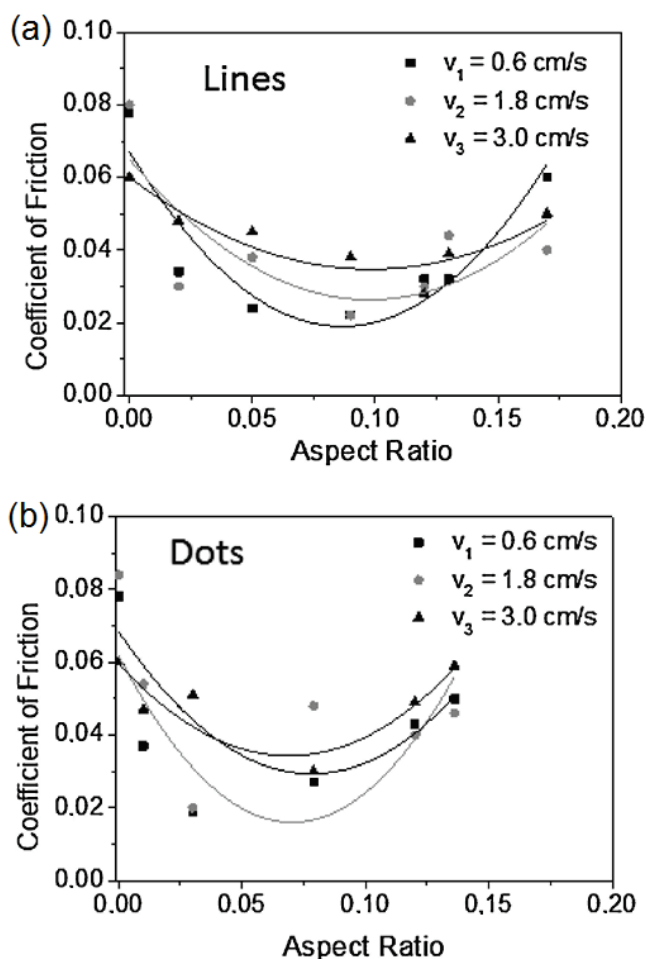


Fig. 5 Friction coefficients versus aspect ratios for (a) line- and (b) dot-like patterns. v_1 , v_2 and v_3 denote the linear speed during the tribological measurement. The fitting curves are only shown to guide the eye [15].

To prove the capability of the DLIP method for biological functionalization of surfaces, interference patterning was used to fabricate line-like arrays on polyimide (PI) substrates, to serve as templates for cell growth experiments with mouse mammary adenocarcinoma cells (LM2). By controlling the angle between the laser beams, the spatial period was varied between 500 nm and 10 μm . The laser fluence (F) was selected with regard to the optimal structure quality (e.g. avoiding collapse of the fabricated array) for each spatial period. For our experiments, the range of laser fluence was between 0.2 to 1.0 J/cm^2 . It was observed that for small spatial periods, laser fluences of 0.3, 0.4, and 0.7 J/cm^2 preserved the morphology of the patterns for 0.5, 1.0, and 2.0 μm arrays, respectively.

After that, the PI substrates were tested for their ability to guide mouse mammary adenocarcinoma cells (LM2). For comparison, the cells were also grown on nontreated PI samples. The spatial disposition of the LM2 cells on non-patterned and patterned PI surfaces and their morphology after incubation of 96 h is shown in Figure 6. The images correspond to the superposition of the optical microscopy picture and fluorescence microscopy with the nucleus of the cells stained with the DNA-specific fluorescent dye (Hoechst 33258). The arrows at the upper right corner of the images represent the orientation of the line-like structure. As can be seen in Figure 6(a), the mostly multipolar

cells show no specific direction but are rather randomly orientated and frequently overlapping on the non-patterned surface. In contrast, the cells on the patterned surface are clearly orientated to the microstructure (Figure 6, b–f). Particularly for smaller periods (500 nm to 2 μm), the cells are highly aligned to the direction of the structure (Figure 6, (b–d)), while also displaying a smaller size and an elongated morphology. On wider spatial periods of 5 and 10 μm (see Figure 6, e,f), it is possible to observe non-aligned cells. Further information about this study can be found in [15].

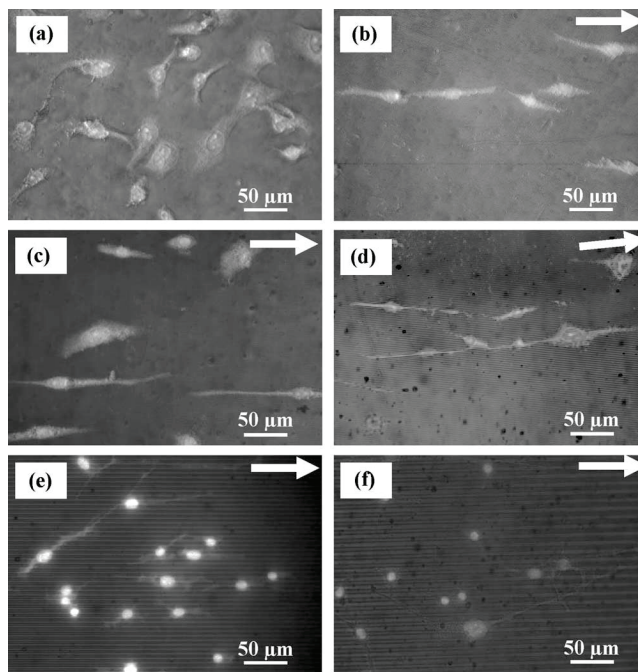


Fig. 6 Fluorescence microscopy images of cells grown on (a) non-patterned and patterned PI surfaces with spatial periods of (b) 500 nm, (c) 1 μm , (d) 2 μm , (e) 5 μm , and (f) 10 μm . The arrows indicate the direction of the line-like pattern [16].

3.3 Fabrication of high resolution decorative elements

Through the newly developed DLIP- μFAB System [10], freely selectable visual motifs with structural diffraction colors can be directly fabricated on metallic surfaces. The visualized colors are the result of diffraction of white light at the periodic patterned surface obtaining the characteristic colored spectra located at both sides of a central reflected white band. In this manner, a change in the inclination angle of the substrate with respect to the incident light produces also a change in the local observed color. A similar effect can be achieved by controlling the structure period: smaller periods produce a higher dispersion of the colored spectra and thus different colors can be observed at a same observation angle for substrates locally structured with different spatial periods.

To fabricate the decorative element, firstly the colors of an original image have to be converted to discrete colors using an image processing software tool. Then, the different colors of the motif are converted into raster images which are further processed and transformed to machine code for the surface structuring process. Each color of the processed image is assigned to a defined spatial period.

As example, an image of the Church of Our Lady fabricated on a stainless steel surface is shown in Figure 7. This

concept has been also extended for the processing of larger areas as shown in Figure 8. In this case, a 200 x 300 mm² Ni-foil was patterned with 6 different spatial periods ranging from 2.2 to 3.1 μm .



Fig. 7 Processed image of the Church of our Lady (Dresden, Germany) on stainless steel (50 x 75 mm²) using DLIP- μ FAB system.

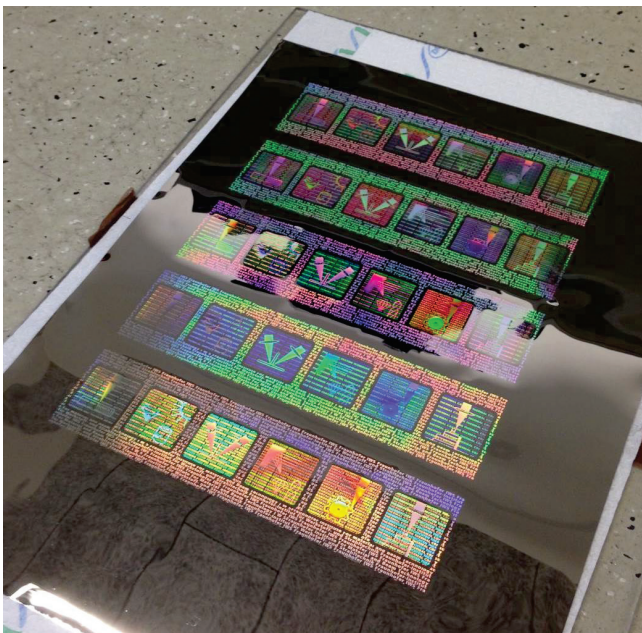


Fig. 8 Processed Ni-foil using DLIP- μ FAB system with 6 different spatial periods ranging from 2.2 to 3.1 μm .

Conclusions

Direct Laser Interference Patterning has been used to fabricate periodic surface patterns up to in the sub-micrometer level on different materials. It has also been shown that new surface properties such as low friction coefficient, cell guiding effects as well as decoration (structural colors) can be realized in a one step process.

The development of different optical heads as well as its integration into laser processing systems demonstrated the actual maturity of the method, where integration at industrial scale is already possible. On the other hand, the possibility to achieve fabrication speeds higher than 1

m²/min will depend on the available laser power as well as on the design of proper structuring strategies. Therefore, new laser systems providing long coherence length as well as high power have to be developed. This issue is actually being investigated in cooperation systems with a laser manufacturer as well as laser processing systems with the objective to achieve a record fabrication speed of 5 m²/min by 2016.

Acknowledgments and Appendixes

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