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Direct Laser Interference Patterning, 20 years of development From the basics to industrial applications

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ABSTRACT

Starting from a simple concept, transferring the shape of an interference pattern directly to the surface of a material, the method of Direct Laser Interference Patterning (DLIP) has been continuously developed in the last 20 years. From lamp-pumped to high power diode-pumped lasers, DLIP permits today for the achievement of impressive processing speeds even close to 1 m²/min. The objective: to improve the performance of surfaces by the use of periodically ordered micro- and nanostructures. This study describes 20 years of evolution of the DLIP method in Germany. From the structuring of thin metallic films to bulk materials using nano- and picosecond laser systems, going through different optical setups and industrial systems which have been recently developed. Several technological applications are discussed and summarized in this article including: surface micro-metallurgy, tribology, electrical connectors, biological interfaces, thin film organic solar cells and electrodes as well as decorative elements and safety features. In all cases, DLIP has not only shown to provide outstanding surface properties but also outstanding economic advantages compared to traditional methods.

Keywords: Surface functionalization, Direct Laser Interference Patterning, periodic surface pattern formation

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 context, laser based technologies can provide the required technological and economical aspects to reproduce such surfaces.

One example for the formation of 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 photopolymerized and after resist development, a periodic variation of the surface topography results. However, the multistep character of LIL limits the number of possible applications due to the high costs that are associated. Furthermore, only planar surfaces can be treated.

However, if laser systems show sufficient pulse energy, the surfaces of different materials can be directly processed and thus, the method has been called in this case Direct Laser Interference Patterning (DLIP). 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].

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This technique facilitates various metallurgical processes, such as melting, recrystallization, crystallization of amorphous materials (this aspect was intensively studied in the 1990´s 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].

Nowadays, industrial laser systems offer necessary technological characteristics to increase the fabrication speed as well as reducing the power to cost ratio (W/€). For instance, several companies are able to offer ns-pulsed industrial laser systems (e.g. fiber and disc-lasers with an average power between 100 to 600 W) [10, 11]. Furthermore, laser systems with ps pulses (1 – 20 ps) with an average power of several tens of Watts (60 – 400 W) and repetition rates up to the MHz range have been recently developed [12, 13]. On the other hand, the improvement of the average power and repetition rates of pulsed laser systems has increased the demands for laser beam scanning over the current upper limit of beam deflection in case that the maximum average power is used. Furthermore, the resolution of conventional laser writing systems is limited to 5 – 15 μm and thus not allowing for the fabrication of surface topographies with a resolution in the sub-micrometer range which is necessary in many cases for obtaining outstanding surface properties. However, due to the intrinsic characteristics of DLIP (e.g. not processing at the focal position), the advantages of these new developed laser systems can be used without the necessity of high speed beam deflection optics.

This manuscript describes the innovations performed in both basic and applied research, related to the development of the Direct Laser Interference Patterning technology in Germany. Within the last 20 years, the method hast not only permitted to produce surface patterns with feature sizes in the nanometer scale (< 100 nm) (at least one order of magnitude smaller than the resolution achievable using direct laser writing) but also to fabricate those surface patterns at record fabrication speed (~1 m²/min).

In addition to those impressive values, DLIP has shown to be capable to improve the functionality of a large number of materials (polymers, metals and ceramics) in a very wide spectrum of applications, including tribology, healthcare, photovoltaics as well as decoration. Although those achievements have permitted to produce outstanding research in the above mentioned topics (more than 100 publications since 1998), the industrial application of the DLIP technology will only be possible if compact optical-head solutions and laser machines are available. These developments are also shown.

2. THE INTERFERENCE PRINCIPLE

The *direct laser interference patterning* method makes use of interference of two or more laser-beams, like in holographic patterning, but in this case no development of the irradiated sample is needed [14-17]. The most important requirement to produce the periodic structures 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 be of high-power. The microstructuring process is based on mechanisms of photo-thermal, photo-physical or photo-chemical nature, depending on the type of material [18].

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:

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

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) (Fig. 1.1) of the interference-plane, respectively; ψ_i is the initial phase, and k the wave number:

$$k = \frac{2\pi}{\lambda}, \tag{2}$$

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

$$I = \frac{c\varepsilon_0}{2}\left|E\right|^2, \tag{3}$$

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 calculated.

Two-beam interference (n = 2) produces a two-dimensional line-like geometry (Fig. 1a) whereas three laser beams (n = 3) produce different 2D arrays depending on the magnitude of the electric-field of each beam and the geometric configuration. For symmetric configurations, the periodic intensity distribution shown in Fig. 1b is obtained. The complexity of the patterns can be further increased by using additional laser beams.

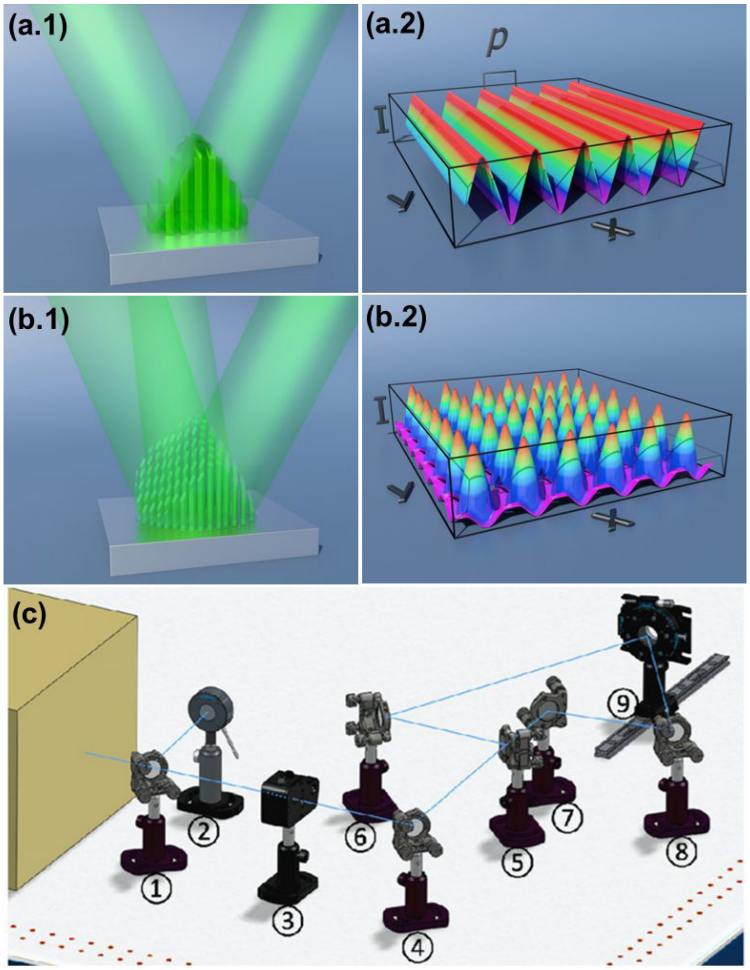

Figure 1. Calculated intensity distribution for (a) two-beam interference and (b) three-beam interference assuming symmetrical configuration (b.1); in (c), an example of a beam-splitter DLIP setup is given: The laser interference patterning setup. (1) A 10% reflection beam splitter for monitoring laser power, (2) a high threshold power meter, (3) a mechanical shutter, (4, 6, 7, 8) are reflection mirrors, (5) a 50/50 beam splitter, and (9) a sample holder.

3. TECHNICAL PROGRESS OF DLIP

Within the last 20 years, DLIP has not only show to be capable of producing functionalized sur-faces but also an impressive technological development to bring this technology to real industrial applications.

At laboratory scale, interference patterns can be obtained by splitting a coherent laser beam into two or more sub-beams which are later overlapped on a workpiece. In case of ns pulse laser systems, this can be realized by using beam splitters and mirrors [4-9].

The first alternative to the laboratory setup, namely *DLIP-High-Speed*, was designed for the fabrication of patterns with a fixed geometry and spatial period (Figure 2). This optical device, developed at IWS, has already achieved impressive fabrication speeds of 0.36 m^2/min on steel and 0.90 m^2/min on polycarbonate (PC) substrates in 2014.

A second alternative, namely *Flex-DLIP*, was developed in 2012. These optical systems are equipped with mobile components to control the intercepting angle between two laser beams fully automatic. The principle of operation of this

DLIP-head can be described as follows. Firstly, all laser beams required to obtain the interference pattern (2, 3 or 4) are focused on the substrate obtaining a circular pixel with a diameter varying from 25 µm to 300 µm, corresponding to resolutions of 1016 and 85 DPI (dots per inch), respectively. Within such a pixel, the interference pattern intensity is transferred in the materials surface. If the spatial pattern periodicity of the pixels is varied, different functionalities can be obtained (e.g. different optical colors under a specific observation angle). Recently, standard galvanometer scanners have been combined with those optics, permitting processing speeds up to 6 cm²/min (Figure 2).

However, the above reported speeds, especially for the *Flex-DLIP* concept, are for several applications still a limitation. Therefore, a DLIP system heads has been recently developed. This system is equipped with three different *Flex-DLIP* modules for utilizing UV, VIS and IR laser wavelegths. The system permits to structure sleeves (or cylinders) up to 600 mm in length and 300 mm in diameter (see figure 2). The structured sleeves are later used in roll-to-roll embossing systems for high speed treatment of polymer foils. Up to now, throughputs of 15 m²/min have been achieved.

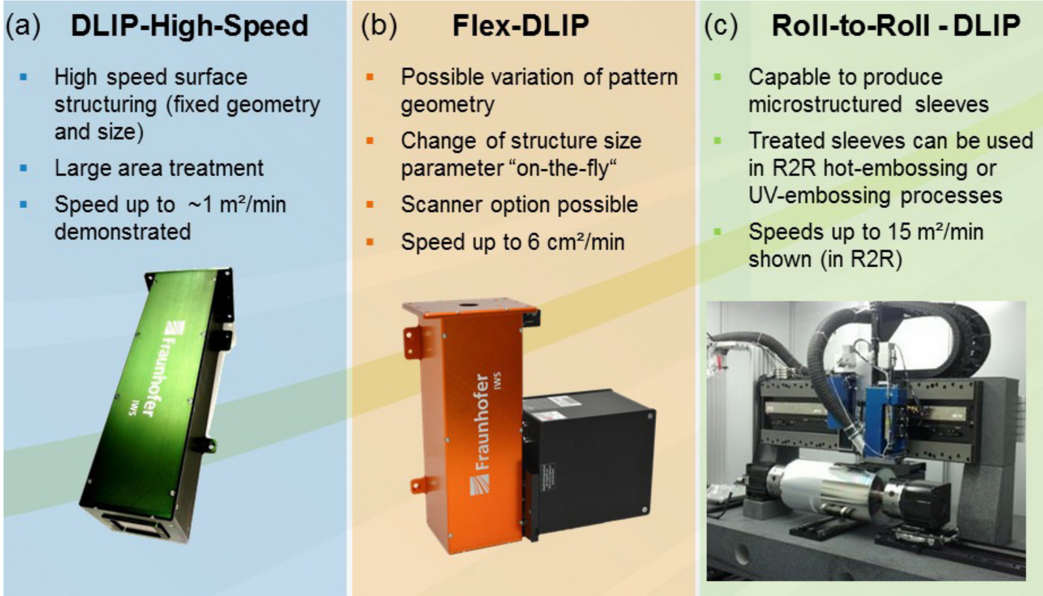

Figure 2. Direct Laser Interference Patterning optical heads and systems: (a) DLIP-High-Speed; (b) Flex-DLIP optic with galvanometer scanner; (c) DLIP-µFAB System developed at Fraunhofer IWS and the Technische Universität Dresden for the treatment of large area sleeves and cylinder for roll-to-roll UV and hot-embossing.

4. ADVANTAGES FOR PRACTICAL APPLICATIONS

4.1 Surface micro metallurgy

Tailoring of microstructures and surface functionalization are key goals in the surface technology of materials. From the technological point of view, we have to provide patterning techniques, which are able to create superior long-range ordered topographies and to tailor the microstructure and surface chemistry of materials with relevant time efficiency and geometrical precision. Advanced periodic interference patterns allow for an accurate topographic design of surface properties such as friction, wetting or optical absorption. Moreover, the localized and well-defined heat input enables us to tune materials microstructures and to directly affect the surface chemistry e.g. oxide film thicknesses or oxide morphology which in turn influence tribo-mechanical properties. In this context, the control of grain size distributions as well as grain orientations in metals e.g. plays an important role in the local modification of for example mechanical properties. It is well known that the hardness and fracture toughness of polycrystalline materials are strongly correlated to each other by the Hall-Petch relationship. Thus, the combination of a periodic distribution of nano- and microcrystalline or ultra-fine grained regions in a composite architecture provides high strength and ductility means high fracture toughness. Surface micro metallurgy by DLIP proved to be successful in creating grain size composites in gold thin films which are applied for example for electrical contacts [19]. Additionally, intermetallic phase composites can be achieved in e.g. TiAl or NiAl multilayer films consisting of hard intermetallic phases which are embedded in a more ductile matrix (Figure 3a) [20]. The resulting mechanical properties are superior to those of unpatterned surfaces.

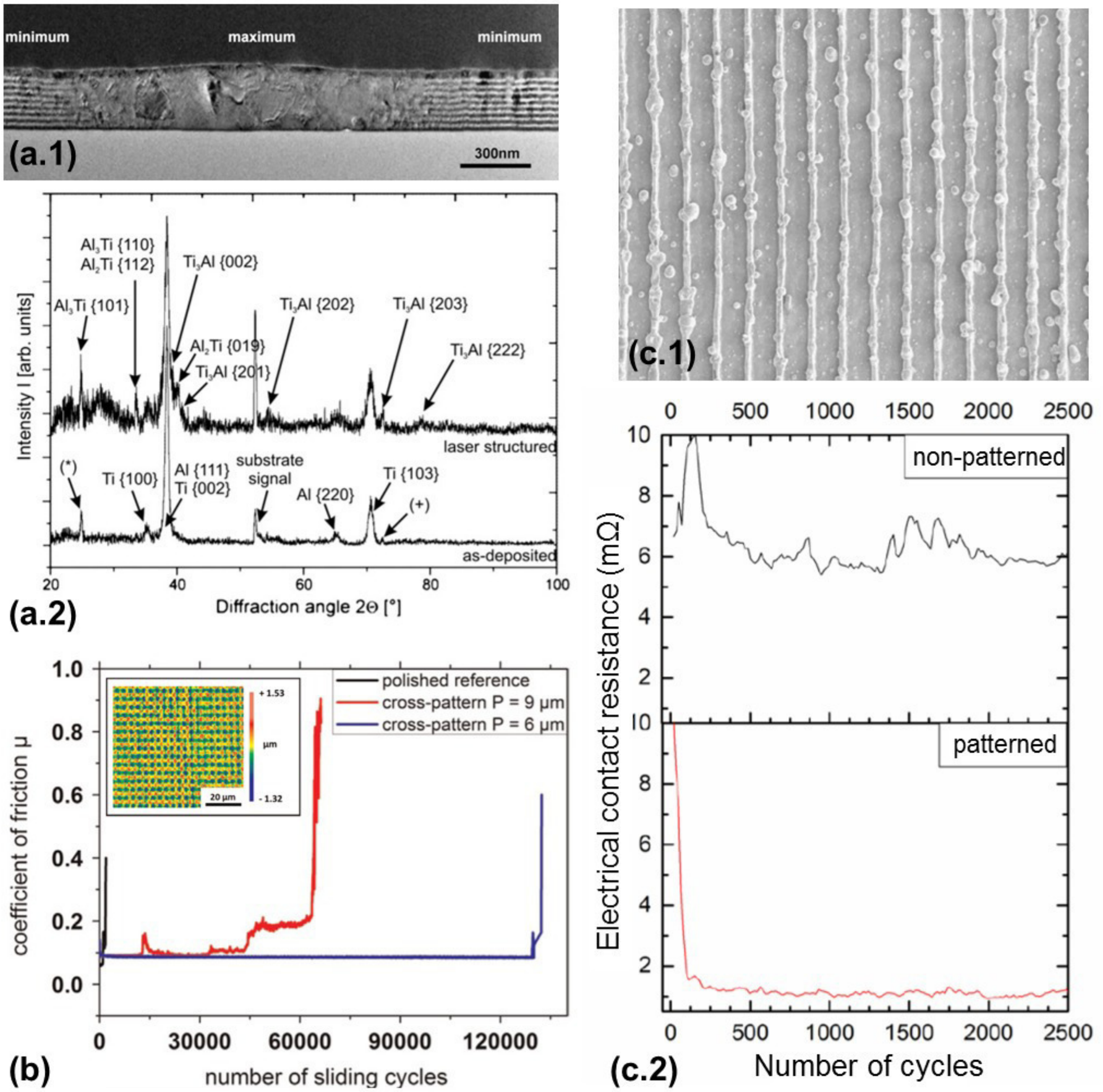

Figure 3. Control of microstructure and tribological and electrical performance of surfaces: (a.1)transmission electron microscope micrograph of a cross-section through the Ti/Al-thin film and (a.2) X-ray diffraction phase analysis measurements of a Ti/Al multilayer film before (lower curve) and after (upper curve) the laser interference treatment. Reflections (*) and (+) denote the presence of intermetallic phases in the as-deposited state [20]; (b) Comparison of the temporal evolution of the coefficient of friction for the polished reference and the cross-like patterns with structural periodicity 6 and 9 µm. The inset shows a 6 µm cross-like pattern [25]; (c.1) DLIP treated Sn-substrate with 9 µm spatial periodicity and (c.2) comparison of the electrical contact resistance as a function of the number of cycles for the treated and non-treated surfaces [34].

4.2 Advanced surfaces for tribological applications by DLIP

Most Friction and wear related damages account for up to 4 % of the GNP of an industrialized nation [21]. An approach for improving the tribological performance of components is laser surface texturing. Between other methods, DLIP has shown to be capable to produce precise topographies which can be used for trapping of wear debris but also to reduce the contact area under dry conditions. As far as lubricated contacts are concerned, the laser-induced features can act as secondary oil source, provide additional hydrodynamic pressure or trap wear particles as well. Therefore, the tribological performance can be significantly improved under different frictional regimes.

Our developed DLIP System allows for both a topographic and microstructural tailoring which is unique in its property design for rubbing surfaces under dry contact conditions. In particular for miniaturized systems, lubrication is very often complicated in small dimensions. It could be shown for thin gold films, that depending on the used periodicity, the surface profile of the rubbing surfaces can be tailored in a way that smaller periodicities result in more plateau-like topographies with an improved load carrying capacity and thus a significantly reduced wear volume compared to the unpatterned sample surface. Moreover, it was possible to directly influence the grain architecture of the materials used by the large heating and cooling rates and so to further affect the tribomechanical properties leading to a better wear resistance of about 65 % in comparison to the unpatterned reference surface [22]. Additionally, dry sliding experiments with stainless steel samples were performed with a line-like interference pattern selecting different periodicities (5, 9 and 18 µm) on substrate and on a 100Cr6 ball as counter body [23]. It could be shown that depending on the relative alignment of the two sliding bodies in contact, the coefficient of friction (COF) could be reduced significantly by a factor of 3.5 compared to the unpatterned reference surface. This is a remarkable frictional reduction for dry contacts and clearly increases the reliability of components in industrial applications.

As far as lubricated contacts are concerned, there is an increasing trend in the use of ultra-low viscous lubricants such as 5W20, 0W16 or even 0W8 for machine components [24]. By doing so, the fuel consumption can be reduced by approximately 0.5 % because the relevant shear forces in the lubricant are lower for those lubricants. But, the lower viscosity leads to thinner lubricant films in the contact zone and thus a reduced separation between the sliding partners. The developed DLIP system offers a great possibility to directly influence the lubricant film thickness in micro- and nanodimensions by initiating a periodic hydrodynamic pressure profile. This facilitates the separation of the contacting bodies and thus reduces wear-related damages.

Furthermore, our research group could show that DLIP results in a strongly increased oil film lifetime for patterned steel surfaces. Here, cross-like laser patterns with a periodicity of 6 µm lead to an enhanced oil film lifetime by a factor of 130 compared with a polished unpatterned steel surface under mixed lubrication (see Fig. 3b) [25]. This directly contributes to the rising demand for resource efficiency.

4.3 Periodic contact deformation arrays

As the electrification of the powertrain continues to make progress in the automotive sector and cars with conventional combustion engines must steadily increase their fuel economy, the demand on modern electrical connectors are rising. High voltages in electric and hybrid cars as well as higher rotational speeds in combustion engines lead to increasing operation temperatures. High rotational speeds also result in strong vibrations during operation causing severe fretting damage to connectors. In addition, the increasing number of electronic systems goes hand in hand with a rising number of connectors and individual pins per connector (400 connectors with 3000 pins in modern luxury vehicles). This results in the need for lower insertion and withdrawal forces, because those connectors require manual installation and maintenance [26-29]. All these partially opposing demands cannot be met by today's connector systems. Tin is the most common contact finish in the automotive sector and cannot be easily replaced by high performing noble metals in the mass market due to cost reasons. As a non-noble metal, tin forms a native oxide layer on its surface, which acts as corrosion protection. However, oxidation makes tin susceptible to fretting wear, which is related to 60 % of the overall electronic problems in vehicles [30].

DLIP opens an innovative way to improve both, the wear resistance and the electrical contact resistance (ECR) of existing tin-based contact finishes [31]. By introducing a periodic array of topographic maxima in the tin surface (periodicity < 10 µm), the local contact pressure is increased thus facilitating the plastic flow of the soft tin layer [32]. As a consequence, the native insulating oxide layer can be removed with a 20 % lesser mean surface pressure, leading to a decrease of the ECR by up to 81 % under stationary conditions. Theoretically, this reduces the contact load of connector springs by more than 95 % [32, 33]. Tests under fretting conditions proved that the produced insulating wear particles can be effectively stored in the topographic minima of the pattern, thus keeping electrically conducting paths open. A decrease of up to 71 % of the ECR combined with an increase of the service life of up to 19 % was achieved (see Fig. 3c) [34].

4.4 Cell scale biological interfaces

Due to the unique range of micro- and nano-pattern periodicities accessible by DLIP, very specific, on-scale interactions of surface structures and biological cells can be induced.

For human lung cells, we showed that not only the overall adhesion, but also the orientation of these cells can be precisely guided by line-and cross-like DLIP structures (15-20 µm) on various polymers [9, 35]. This opened new prospects in tissue regeneration and for biocompatible surface design in general. Furthermore, the DLIP technology has demonstrated to be capable to substitute cost intensive treatment methods sand blasting, cleaning and etching) in the preparation of dental implant surfaces. The technology is actually being transferred to a very well know manufacturer of dental implants in Germany [36].

The enormous potential of DLIP for direct cellular interactions even triggered a new field of re-search: patterning-based antibacterial surface design. Healthcare-related infections cause up to 148.000 deaths per year in Europe [37]. Dangerous bacteria, which survive up to years on touched surfaces, are the main source of infection. Two examples for high infection risk environments (high bacteria resistances and weakened immune system) are remote space stations and intensive care units in hospitals. Amongst 202 research proposals to the European Space Agency (ESA), DLIP anti-bacterial surfaces were awarded with 4th place. "Nano-spacers", which prevent bacterial attachment and dangerous biofilm formation were generated by DLIP and are now readied for actual tests in micro-gravity on the International Space Station (project: NO-BIOFILMS, ILSRA-Call 2014). To combat health-care related infections and multi-resistant germs on earth (e.g. MRSA), frequently touched elements and surfaces in hospitals are increasingly replaced by antimicrobial copper alloys. The bactericidal effect of these materials is significantly enhanced, once bacterial adhesion and surface wetting properties can be controlled. Both were easily tuned by suitable DLIP-structures (P = 2-5 µm) for this purpose [38, 39, 40].

4.5 Improved efficiency for thin film organic solar cells

Organic photovoltaics offer the promise of ultimate scalability and achieved tremendous progress with ever increasing power conversion efficiencies in the last years. To further improve efficiencies, optical optimization approaches as used in other thin-film technologies can prove helpful to achieve maximum concentration in the absorber layer. Surface structures, such as periodic gratings, surface patterns, or rough surfaces can enhance the power conversion efficiency of the solar cell by elongating the optical path of incident light inside the absorber material, creating light trapping geometries or causing Bragg scattering at periodic photonic crystal geometries [41].

DLIP permitted to fabricate large area two dimensional periodic surface patterns on polyethylene terephthalate (PET) substrates, which is use a standard material in organic photovoltaic. After that, the substrates were coated with poly(3,4-ethylenedioxythiophene):poly(styrenesulfonate) (PEDOT:PSS) electrode and a ZnPc:C60 based small-molecule organic solar. All structured cells showed a reasonable electric performance with an open circuit voltage V_{oc} and current-voltage characteristics comparable to a glass or flat PET reference. Both the short circuit current J_{sc} and the power conversion efficiency are strongly affected by the surface structure and the increased light absorption in the active layer. Comparing the power conversion efficiencies to the reference cell on flat PET, a relative increase of 5 % was observed for line-patterns, and a 21 % improvement for the hexagonal pattern with the shorter period (710 nm) (this work was awarded with the German High Tech Champion Prize 2011, BMBF) (see Fig. 4a) [42].

4.6 Pattering of metallic thin film electrodes as potential substitute of ITO

The progressive development of optical thin film electronics, such as photovoltaics or light emitting diodes, requires transparent electrodes with extraordinary efficiencies. This implies a new class of electrical conducting devices which provide both high light transmittance and low sheet resistance. State of the art at present is the usage of ITO coatings, which can be found as front contacts in thin film solar cells, touch screen applications and organic LEDs [43]. However, the major disadvantage of ITO coatings is the high price of the rare earth Indium [44].

A cost-effective way to replace ITO is given with the application of ultrathin metal coatings. Due to the high absorption coefficient of metals, the thickness of the coatings has to be very thin in order to obtain highly transparent electrodes. On the other hand, the electrical conductivity of these very thin films does not allow using these materials as efficient electrodes.

Using DLIP, aluminum and copper thin films deposited on glass substrates were processed using a three-beam configuration. In this way, hole-like structures with spatial periods between 1.7 to 2.7 µm were fabricated. The resulting optical and electrical performance of the produced electrodes showed that the laser treatment method permitted to increase both, the transmission (up to 81 %) and the electrical sheet conductivity of the film, especially for electrodes irradiated with ps-laser pulses. In this frame, electrical sheet resistances between 25 and 50 Ohm/sq. could be obtained, matching the properties of existing ITO electrodes (see Fig. 4b, this project was awarded with the Green Photonics Award 2015 at the SPIE Photonics West in San Francisco, USA) [45].

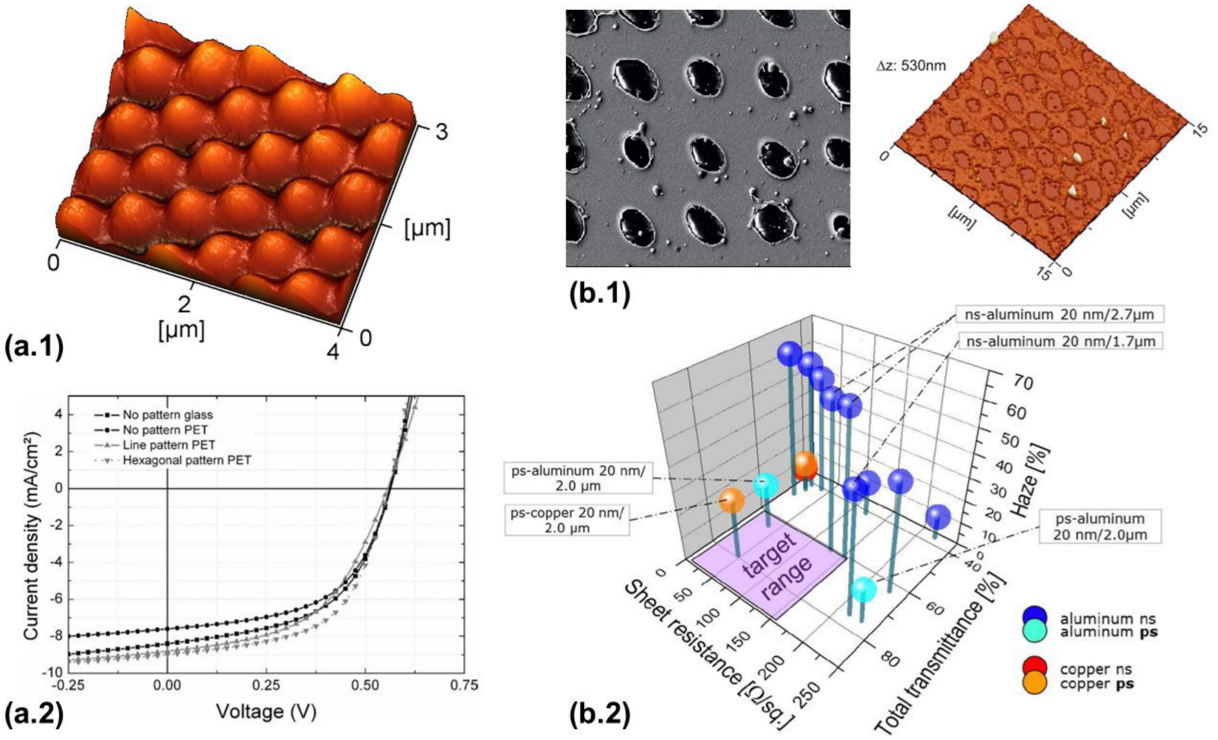

Figure 4. Modulation of optical properties of DLIP treated materials for improving the performance of (a) organic solar cells and (b) transparent conducting electrodes. (a.1) Example of hexagonal arrays with 700 nm period on PET substrate treated at a laser fluence of 100 mJ/cm² (structural depth 140 nm) and (a.2) the characterization of the electrical performance of solar cells with different surface patterns. The hexagonal structure results in a 21% improvement compared to the flat reference [42]; (b.1) Surface topography of ps-structured Cu thin films with a spatial period of 2.0 µm and (b.2) optical and electrical properties of selected Al and Cu films, structured using ps and ns-pulses [46].

4.7 Decorative elements and safety features

Nowadays, embossed holograms and diffractive optically variable structures are well known as visual safety features on different elements such as plastic cards, banknotes and on branded goods and media to protect against counterfeit. This is a response to the explosive growth of computer and reprographics power, particularly in the areas of desktop publishing, scanning, reprographics and color copiers, which has made it possible to get passable reproductions of printed documents.

A common technology being used for the fabrication of safety elements is Electron Beam Lithography (EBL). EBL systems involve the use of an electron beam to locally modify a material which is later developed obtaining the final structure on a substrate. Later, the resist is covered with a metallic layer (Ni) to generate a stamp. The main advantage of EBL is that structure features even in the nm-scale can be fabricated. On the other hand, EBL cannot be directly used for the fabrication of safety features on metals and long processing times (even some hours) are necessary to pattern each individual element.

DLIP has permitted to produce high quality holograms for decorative or counterfeit applications with high throughput and resolution (even in the sub-µm range) as well as high flexibility even directly on the surface of metals (Fig. 5). In this way, the processing cost could be reduced from about 115 €/cm² to 0.5 €/cm² [46].

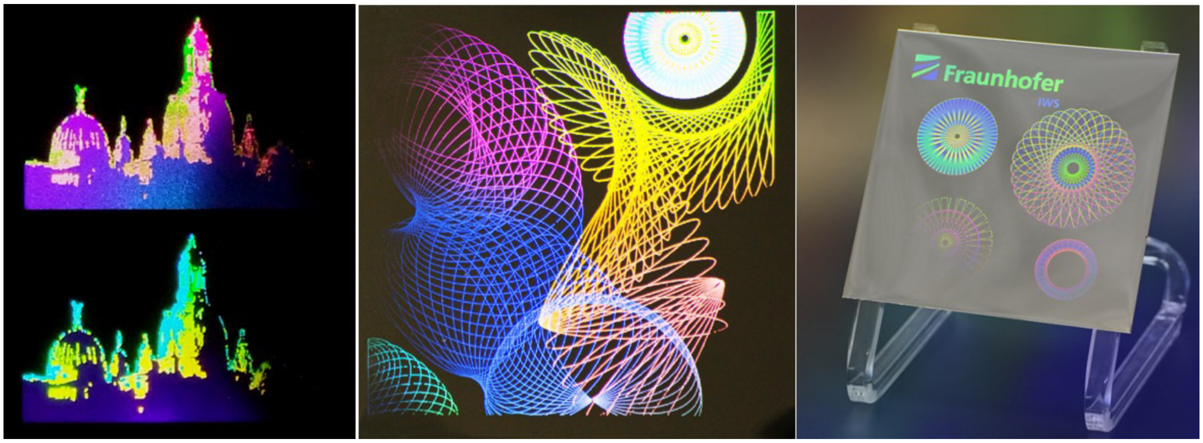

Figure 5. Fabrication of decorative elements using a standard ns-DLIP µFAB system (Fraunhofer IWS, Dresden, Germany).

5. CONCLUSIONS

In summary, we could demonstrate the advantages of the direct laser interference patterning method for the fast and precise tailoring of materials surface microstructures and topographies on industrial relevant scales. Periodic patterns with feature sizes in the range of sub-micrometer up to several micrometers could be created on wide number of material surfaces. From the structuring of thin metallic films to bulk materials using nanosecond and picosecond laser systems, several technological applications could be improved using this method. These applications include the control of the tribological performance of metals (friction reduction and lubricant lifetime improvement), the reduction of electrical resistance in electrical connectors, the improvement of biocompatibility of implants as well as the reduction of pathogen microorganism, the improvement of the efficiency of thin-film organic solar cells as well as transparency of metallic electrodes keeping the electrical conductivity almost unaffected, and the fabrication of decorative elements and counterfeiting features.

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