# LASER GENERATED MICRO- AND NANOSTRUCTURES AND THE TRANSFER TO POLYMERS FOR EXPERIMENTAL USE

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### 1. Introduction

Laser surface processing is a powerful tool to design and optimize material functionality for specific applications. A growing number in alternative lasers type offers more opportunities to optimize process parameter for tailoring surface properties efficiently. Improved performance or wear characteristics can be gained and new applications of a given material or technical design is offered by laser surface treatment. Traditionally such manufacturing approach involves selective heat treatment for surface hardening of sensitive metallic parts, laser polishing by rapid melting and cooling of the material surficial area and evaporating and ablating material to form micro structures or cavities; yet by using nanosecond and picosecond lasers new material can be incorporated into an existing surface with less distortions than thermal spraying or welding techniques. Recently the focus is moving towards femtosecond laser ablation in surface processing. The polarization control of such lasers in combination with the so-called cold ablation offers the chance to generate surface textures at nanometer-scale dimensions. In combination with micrometer-scale structures these ripples, more precisely laser induced periodic surface structures(LIPSS), enhance tribological properties of material interfaces and slip-stick problems are reduced. A more recent development is the creation of super hydrophobic selfcleaning surfaces inspired by concepts from nature. Fs-laser surface processing can mimic natural textures and research is targeting with growing interest applications in microfluidics, lab-on-chip devices and cell biology to name just a few. Localized controlled modifications with a large degree of freedom to structure shape and size much smaller than a µm can be made without the need of expensive high vacuum processing with electron beams. The achievable multiscale structures modify material surface chemistry and are of great use in chemical sensors particularly with regard to detect biological cellular response in diagnostics and implants. In the fs-regime it is also possible to generate high spatial frequency LIPPS (HSFL) with spatial periods of only a fraction of the used laser wavelength. HSFL's textured optical surfaces can provide optical features of importance for optical sensors, solar cells, LED lightning, plasmon excitation, resonant structures for quantum dot applications and photonic crystals for research purpose. The size and structure of the obtained surface texture depend both on the laser parameter and on the specific material; and the particular application might call for a specific material, eliminating in this way one degree of freedom in the generation of HSFL. In such a case it is important to have a transfer method to duplicate the laser generated features onto a suitable material interface. We started first experiments with a silicon-based polymer used in the LED industry to produce a surface texture mold from the

original laser-processed surface. As this polymer has a high optical quality, such elastic impression can be used also directly for some optical or photonic experiments. Fs-laser generated microstructures, LIPPS and HSFL's in combination with other micro technological process engineering can provide a cost efficient and effective platform for basic photonic research.

## 2. Experimental Methods

For experiments we used a SPIRIT regenerative amplifier from Spectra Physics, delivering 4W average power at 1040nm and 200kHz repetition rate at 350fs pulse length; the system delivers also 520nm second harmonic (SH), where we get about 1.5W after the 100mm focal length scanner optics. Two older laser system, a HQ femtoREGEN and a HQ picoREGEN delivered respectively 350fs pulses at an average power of 950mWand 100 kHz repetition rate in the fundamental at1040nm and 350-380mW in the SH regime (HQ femtoREGEN) and 10pspulses at 1064nm and 1kHz (HQ picoREGEN). Finally we used a 30W fiber laser at 1064 nm, 80kHz repetition rate and 110ns pulse length. Nearly all surface modifications were made with a 100mm scanner optics at a scan speed varying between 50mm/s and 2000mm/s. Laser parameters, scanning direction and related polarization orientation were selected depending on the specific material and the intended surface structure. In some cases the polarization direction was altered during the laser processing. Post-laser processing were in most experiments simple cleaning routines. In some cases we used sputtering for metallization or dielectric coating before ablation and high temperature oxidation or reactive ion etching (RIE) after the laser treatment. This opened an additional avenue to influence size, shape and distribution of the obtained LIPPS and HSFL structures. By such means we could generate with 520nm fs laser ablation on a silicon nitride coated Si wafer at once HSFL with dimensions of about 50nm to 100nm diameter, LIPPS with period of about 500nm and micro structures in the range of 3to 5µm (Fig. 4A, B, C).

## 3. Results and Discussion

A selection of experimental results is presented in table form to show a short summary of possible surface patterns that can be obtained using lasers operating in different time regimes on various material surfaces. Fig.1A reveals that the surface structure made by the fiber laser at 110ns pulse length applying a 50µm xy-scanning hatch is dominated by melting and solidification of the zirconium oxide ceramic. In Fig.1B a texture, consisting of selforganized structures, is created by parallel scanning in y-direction with a scanning hatch smaller than the structure size using the 10pslaser; the material is aluminum nitride ceramic, no LIPPS on the surface are observed, the dimension of the cones is comparable to the structures in Fig.1A. The same laser produced µm-structures covered with LIPPS on the edges of a bore in carbon steel (Fig. 1C). The laser polarization was tangential to the bore wall and hence rotated during the ablation process. The LIPPS are formed perpendicular to the polarization direction and give the impression of a radial corona at the bore edge. Whether LIPPS are formed or not depends in this case not just from the pulse duration but also from the specific material undergoing laser processing. Below the ablation threshold µmsized self organized structures without significant LIPPS were obtained in silicon carbide with our 350fs femtoREGEN operating in the green (Fig.2A). The same laser on the same material created distinct LIPPS with about 800nm periodic spacing by using the 1040nm fundamental laser beam (Fig.2B). In Fig.2C one can see results of the same laser at identical parameter and scanning speed as Fig.2B on carbon steel. Besides clearly visible LIPPS, a self-organized formation of about 5µm diameter quite evenly spaced pinholes is observed.

For the following experiments (Fig.3, 4, 5) we used our new SPIRIT from Spectra Physics. The system delivers 4 times the maximum power as the older femtoREGEN, and

with the same 100mm focal length scanner optics the focal spot diameter is about half of the one obtained with the femtoREGEN setup, so that the peak fluence is 16 times higher for the new system. Fig.3 and Fig.4 show results on the same basic material which is silicon. However, we used it in three different modifications, plain, coated with a 200nm layer of silicon oxide and coated with a 50nm layer of silicon nitride. Silicon oxide and silicon nitride can serve as a mask material for RIE or oxidation, therefore LIPPS could be a mean to create sub wavelength openings in the mask layer; the differences in the LIPPS obtained in the different mask materials will determine the possibility for subsequent processes.

	Α	В	С
Fig.1			
Fig.2	Market M Market Market Mark		
Fig.3	Actor 10.002 20.002 20.002	13 10.90 10.94 Jan 2010 10.94 10.04	1 1.500 2.507 100 100 100 100 100 100 100 100 100 1
Fig.4			100,00 25.04 ED 201 10.5 Em 11.25
Fig.5	0 SA Imm		

Fig.3A shows the result on the 200nm silicon oxide coated Si specimen. In principle ripples can be created very well in Si but unfortunately the ablation threshold is lower for Si and higher for SiO<sub>2</sub>as can be seen at the right side bottom edges of the ablation area. In this region the coating is intact but the Si below is already ablated. The debris created in this way is kept in place by the SiO<sub>2</sub>layer and in the process melted into the formed ripples before the coating cracks and is removed. The aim of the laser treatment to produce a direct written mask for RIE was not reached. For this purpose the Si<sub>3</sub>N<sub>4</sub>coating is more successful, as Fig.3B shows. Laser processing generated in the nitride HSFL with a diameter of about 100nm which is one fifth of the 520nm laser wave length. The length of the ripples is between 200nm and about 1µm. Occasionally100nm dots are formed. These almost planar structures can be used as a mask for RIE which will transform them in a more 3 dimensional structure. Starting from the same 2D-lasered structures we investigated a different approach to transform them into a more 3-dimensional structure by oxidising the Si. We got a 2,5D structure but unfortunately the HSFL were smoothed out so that the resulting 2,5D oxide formations has no sub wavelength features (Fig.3C). We obtained an unexpected result when the fluence was increased to a level at which the Si<sub>3</sub>N<sub>4</sub> coating was removed entirely by one scan. Practically every single laser shot created a 5µm square-shaped pattern shown in Fig.4A;the squares composing the pattern are located in between adjacent laser spots. Next to the spot where the laser beam hits the target spheres of about 100nm diameter are formed and at some distance from the spot, inside the squares, roughly 500nm spaced LIPPS can be observed. With a single shot the fs laser system created HSFL, LIPPS and microstructures (Fig.4B, C). We got 100nm diameter dots in SiC too when the specimen was rotated and in this way the polarization steadily changed with respect to the material surface; under constant polarization orientation LIPPS are formed in SiC. In plain Si it was possible to generate grating-like structures when the scan direction was about perpendicular to the orientation of the polarization. Fig.5A shows the result obtained with the SPIRIT laser system at the wavelength of 1040 nm. It is possible to produce relatively large areas of these ripple gratings. It appears that ripples develop over a large area by fs-laser ablation in most metals and semiconductors, while LIPPS are not so clearly and distinctly formed on a large-area scale in dielectric materials. However, it was possible to obtain an interesting ripple pattern in simple soda lime glass, as demonstrated in Fig.5B. Coating such structures with a high refractive index material could offer a cost-efficient approach to obtain slot waveguides and there might be applications in solar cells, sensors or as a low cost technology for research purposes. Numerical simulation can help in realizing the potential of these. Not always the required material for photonic use will make it possible to generate the desired structures with laser ablation. In such a case it is important to transfer the laser-induced texture obtained on one substrate (where laser modification achieves the desired geometry) to the material which supports better the application.Fig.5C shows one attempt to generate a casting made by a silicon polymer. At a visual examination the grating looks quite reasonable. However, the quality has to be improved for applications.

#### 4. Conclusion

Our experiments reveal fs-laser texturing can create  $\mu$ m-sized structures, LIPPS and HSFL with practically a single shot. Ps-laser ablation can generate ripples in certain materials but not as distinct as fs- ablation. Additional treatments of material interfaces offer the opportunity to obtain a wider bread thin shape and scale for nanometer structures on a given material. Post-treatment of laser textured-surfaces by sputtering, oxidation, wet etching or RIE will considerably increase application opportunities in photonics and optics. Sensor devices on laboratory scale implementing nano-structures can be generated cost efficient.