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# Tailoring birefringence of LIPSS through thin film coatings

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Waveplates modify polarization by generating a phase change. Laser Induced Periodic Surface Structures (LIPSS) have recently started to be studied as waveplates due to the birefringence induced by the nanoripples, easily fabricated in a one-step process by laser, where LIPSS morphology is defined by the characteristics of the laser process parameters and the substrate material. The optical properties of these waveplates are defined by LIPSS parameters such as period, depth or width of the ripples. In this work we have deposited thin film coatings on stainless steel samples containing LIPSS for different coating thickness and composition. Results show that thin film coatings are a good candidate for the tunability of LIPSS birefringence since the coating modifies the induced polarization change and reflectivity of the sample depending on coating thickness and composition, as expected from numerical simulations.

Keywords: LIPSS, femtosecond, nanostructures, polarization, waveplate, thin-film coating

#### 1. Introduction

Polarization control is a key feature in many optical setups and applications. Waveplates, the elements that transform polarization, are based on birefringence, which is present in anisotropic materials such as calcite crystals. These materials exhibit different refractive index for different axes, inducing a shift between the phases of the light in each axis, which changes its polarization.

Transitive waveplates present strong dispersion [1], which is a drawback in applications involving ultrashort lasers. On the other hand, reflective waveplates prevent this issue since light is not required to travel through the element.

Nanostructured surfaces with ripples of periodicity smaller than incident light have been fabricated in recent years as reflective waveplates because the axes parallel and perpendicular to the ripples induce a change in the polarization state, thus acting as artificial birefringent materials. The properties of these waveplates are tunable through control of the parameters of the ripples such as period, depth, shape or orientation [2]. In particular, for light in the visible spectrum or in the near infrared region, ripples should be in the submicron scale. Common fabrication techniques of nanostructures with the mentioned periods include nanoimprint lithography, ultraviolet-molding or electron beam lithography.

Laser-induced periodic surface structures (LIPSS) are directional formations generated in most kinds of materials when irradiated with intense laser radiation. In recent years, LIPSS have gained interest as they present ripples with periods similar to the wavelength of the laser they are fabricated with. LIPSS fabrication is a one-step process and does not require cleanroom conditions. LIPSS have been studied in the last decades, being usually accepted that they

are generated through the interaction of intense incident laser light with an electromagnetic wave scattered at the rough surface, while other models involve surface plasmon polaritons [3]. LIPSS applications include, among others, control of adhesion and migration [4], tribology [5], wetting [6] and light antireflection [7], although their use as reflective waveplates has not been well studied yet.

LIPSS geometry depend on the substrate material and on the laser parameters, such as: number of pulses, fluence, beam polarization or overlap [8]. However, it is difficult to fabricate LIPSS over a wide range of periods and depths with a single laser system, which is a problem for industrial work.

Thin film coatings are layers with thickness ranging from nanometers to micrometers. They are commonly used in optics to fabricate mirrors or as reflective and antireflective coatings, controlling their properties with coating thickness and composition. Although to our knowledge dielectric coatings have never been deposited on LIPSS, they can improve their polarization changing properties and reflectivity by adding new degrees of freedom: coating thickness and composition.

Here we report the simulation and fabrication of birefringent devices consisting of dielectric coatings applied to LIPSS generated on stainless steel. Preliminary results show that induced polarization change and reflectivity can be modified with coating thickness and composition, with improvements over uncoated LIPSS, as expected from numerical simulations, showing that dielectric thin film coatings are a good candidate for the tunability of LIPSS birefringence.

#### 2. Material and methods

Sample preparation was composed of two stages. In the first stage, LIPSS are fabricated on stainless steel. In the second stage, thin film coating is applied on the samples. Additionally, finite-difference time-domain (FDTD) numerical simulations have been performed, studying the influence of coating geometrical configuration on the polarization state of the reflected beam.

# 2.1 Sample preparation

Stainless steel strips of 1 cm width, 20 cm long and 1 mm thickness were cut in a guillotine shear to pieces of 1 cm long. Prior to processing, samples were introduced in an ultrasonic bath with acetone for 5 minutes for surface cleaning purposes.

In order to fabricate LIPSS, a Ti:Sa (titanium-doped sapphire) laser with pulses of 130 fs, central emitting wavelength at 800 nm and repetition rate of 1 kHz was used. Maximum pulse energy available is 3 mJ. An attenuator composed of a half wave plate in combination with a polarizing beam splitter was used to control the energy delivered to the sample, and a neutral density filter further decreased the energy to 1 µJ. The laser beam was directed onto the sample through a 10x microscope objective (Fig. 1). The sample was moved under the laser irradiation describing parallel lines of width 10 µm each until an area of 3x3 mm<sup>2</sup> was filled. The polarization of the laser radiation was controlled with another half wave plate so that the orientation of the LIPSS was perpendicular to the direction of the processed lines. After the processing, the samples were again cleaned for 5 minutes in an ultrasonic bath with acetone.

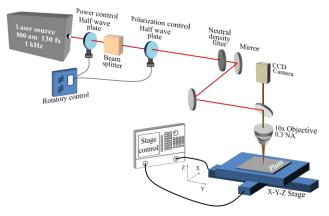


Fig. 1 Setup used in the fabrication of LIPSS.

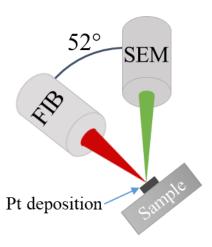
Plasma Enhanced Chemical Vapor Deposition (PE-CVD) was used to create the thin films.  $SiO_2$  was deposited on 4 samples with different thicknesses of 50, 100, 200 and 300 nm, and  $Si_3N_4$  was deposited on another 4 samples with the same 4 thicknesses. An additional sample was left uncoated for comparison.

# 2.2 Characterization

Scanning Electron Microscopy (SEM) was used to obtain images of the surface of the sample. The period of the LIPSS was calculated by means of two-dimensional Fast Fourier transform (2D-FFT), where the relation between

LIPSS period ( $\Lambda$ ) and the distance between FFT peaks (d) is given by the equation  $\Lambda=2/d$ .

Images of the transversal profile of LIPSS and coatings were obtained with the technique Focused Ion Beam (FIB), incorporated in the same microscope along with SEM. FIB uses focused ions which eliminates part of the surface of the sample, so that it is possible to obtain a transversal cut. In order to do this, the sample was tilted 52° (Fig. 2). Before the FIB technique is to be applied a platinum nanometric layer is deposited on the sample, in order to protect the surface, so that the ions only penetrate from the side.



**Fig. 2** Schematic FIB and SEM configuration. The sample is tilted and coated with platinum for FIB operation.

Changes in polarization induced by the samples were determined with a polarimeter (Thorlabs PAX1000IR1). HeNe (633 nm wavelength) and 808 nm diode continuous lasers were incident at 25° with respect to the normal of the sample. Laser polarization was controlled with a half wave plate. To detect birefringence, laser polarization was set to linear at 45° with respect to LIPSS orientation.

Reflectivity was measured using a supercontinuum laser (NKT superk compact), acquiring the reflected light with an integrating sphere connected to a spectrometer.

# 3. Results and discussion

### 3.1 Numerical simulations

In our simulations we have tested three scenarios of sample configuration: LIPSS with no coating (Fig. 3), LIPSS with  $SiO_2$  uniform coating (Fig. 4) and LIPSS with  $SiO_2$  conformal coating (Fig. 5). For all of them, the input was an 850 nm wavelength laser with linear polarization at 45° with respect to the LIPSS orientation, and LIPSS period is 580 nm.

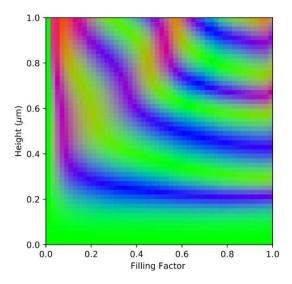


Fig. 3 Q (red) U (green) V (blue) Stokes parameters of the reflected beam from uncoated LIPSS. For the input 45° linearly polarized wave, green means that there is no change, red means change to 0° linear polarization, and blue means change to circular polarization. X-axis indicates the relative width of the ripples.

Results show that polarization can be changed for certain parameters. In the first scenario, where LIPSS are present without coating, there is only change for certain heights, while filling factor is not determining. Also, chances in polarization can only be seen for changes in height of around 200  $\mu m$ , which is difficult to achieve without changing the laser source. In the scenarios with coating, the pattern observed in the simulations is different, with variations also depending on the coating thickness. In addition, varying coating thickness with precision and in a wide range of thicknesses is plausible for most deposition techniques.

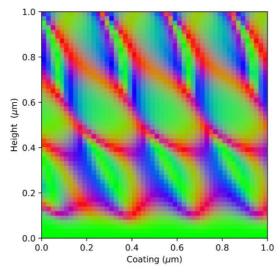


Fig. 4 FDTD simulation for LIPSS with SiO<sub>2</sub> coating, the coating being uniform, this is, having constant height with respect to the horizontal plane. In this scenario, X-axis represents the coating thickness.

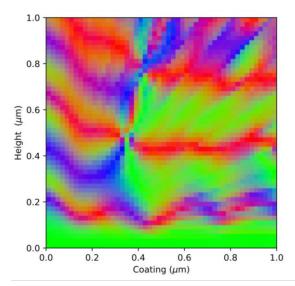


Fig. 5 FDTD simulation for LIPSS with SiO<sub>2</sub> conformal coating. This means that coating thickness is constant along the profile of the LIPSS.

#### 3.2 Sample characterization

SEM images (Fig. 6) show periods around 630 nm. The ripples are not straight along the lines and they have discontinuities, with 2D-FFT showing two frequencies (the second one, around 300 nm has a weaker signal). This could be a source for errors and deviations with respect to simulations.

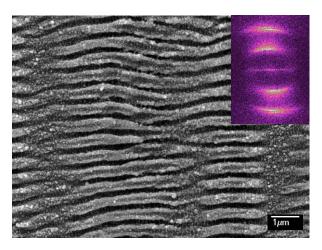


Fig. 6 SEM images of an uncoated sample. LIPSS can be seen along the vertical axis. Inset: 2D-FFT of the image, where it can be noted that LIPSS are starting to show double frequency, which can be related to the darker regions that can be seen in the middle of the ripples.

Images obtained after the FIB procedure (Fig. 7) show profiles where the coatings have a very conformal configuration, which is relevant information for numerical simulations. LIPSS depth is irregular, but a statistical analysis reports an average depth of 340 nm.

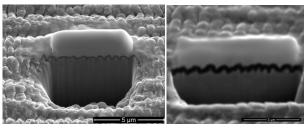
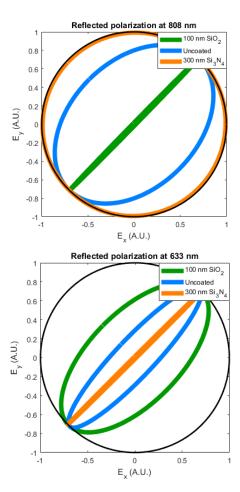


Fig. 7 Images obtained after using the FIB technique. A transverse section of the LIPSS (dark grey) with coating over it (black) can be seen. The amorphous material over the coating (light grey) is the platinum used in the FIB procedure. Left: SiO<sub>2</sub> coating of thickness 50 nm. Right: Si<sub>3</sub>N<sub>4</sub> coating of thickness 300 nm.

#### 3.3 Birefringence

Results obtained in the polarization change show that for certain coating thicknesses and materials, the change in polarization induced differs greatly from one sample to another (Fig. 8), as predicted by numerical simulations. However, they do not show a trivial relation with coating thickness and material. Results are also sensitive to the wavelength of the laser used.



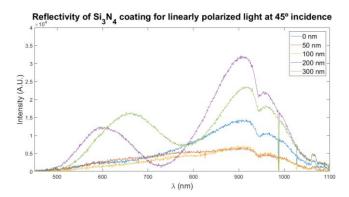
**Fig. 8** Polarization state of the reflected beam for three samples with different coatings. Laser wavelength used is 808 nm (top) and 633 nm (bottom). Incident polarization is 45°. Under certain conditions some samples introduce ellipticity, differing from the response of the uncoated sample.

These results show that it is possible to tune the response of LIPSS without varying their parameters by adding a dielectric coating.

#### 3.4 Reflectivity

Variations of the reflective properties of LIPSS have been observed in the coated samples (Fig. 9). Thin film coatings modify LIPSS reflectivity in certain regions of the spectrum which, depending on composition, thickness, and wavelength, can improve the reflectivity of the uncoated sample.

Since high reflectivity is desirable for applications where low energy loss is expected, this is an optical property to be held into account along with the potential for polarization change.



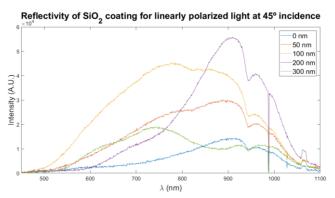


Fig. 9 Reflected spectra obtained for coated and uncoated samples for the two materials used in the deposition: silicon nitride (top) and silicon dioxide (bottom). Higher intensity at certain wavelengths can be seen for some of the samples with coating than that for the uncoated sample (blue line).

#### 4. Conclusions

In the light of these results, we can conclude that dielectric thin film coatings are a good candidate for the tunability of LIPSS birefringence, with reflectivity being an important factor that can also be improved by the coating.

Another important result is that, in the range of studied thicknesses, the coating geometry is conformal. This will be considered in further simulations, where LIPSS irregularity should also be accounted for.

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