MICROSCOPY TECHNIQUES FOR IN-SITU LIPSS DETECTION AND CHARACTERIZATION VIA OPTICAL MEANS

C. MAUCLAIR¹, A. AGUILAR¹, X. SEDAO¹, JEAN-PHILIPPE COLOMBIER¹, RAZVAN STOIAN¹

¹Laboratoire Hubert Curien, UMR 5516 CNRS, Université de Lyon, Université Jean Monnet, 42000, St. Etienne, France

*cyril.mauclair@univ-st-etienne.fr

ABSTRACT

Surpassing the diffraction limit on optical microscopy has lead to important breakthroughs in recent years, especially in the biomedical field, where it is common practice to add a functional group to the biological samples such as fluorophores. However, in applications involving surface micro-nano-structuration and particularly when Laser Induced Periodic Surface Structures are achieved by ultrafast laser irradiation, there is an interest in analyzing these non fluorescent surfaces using non-contact optical techniques. In this report, a wide field super resolution technique, the structured illumination microscopy (SIM), is adapted to observe metallic surfaces. We verify that the technique permits to surpass the diffraction limit by a factor of two. Laser-induced surface micro-structures on stainless steel are characterized with the expected super resolution. The technique enables in-situ characterization of the LIPSS pattern and permits to follow its formation during a multipulse irradiation sequence. We also discuss the potential of another optical technique, namely the Fourier Ptychography Microscopy that permits to overcome the resolution limit of the observing lens by high angle illumination while preserving the large field of view with proof of concept results on a sector resolution target.

1. INTRODUCTION

The generation of Laser Induced Periodic Surface Structures (LIPSS) has attracted a worldwide attention due to the possibility to obtain micro and submicrometric structuring on various substrates with the flexibility of the laser irradiation tool. Many application fields are impacted by LIPSS, mostly related to adding functions to surfaces like hydrophobicity, optical properties and cellular differentiation [1-3]. When using ultrafast laser pulses for surface structuring, the low onset of thermal effects at low repetition rate enlarges the laser process parameters to observe LIPSS formation. Moreover, various kinds of LIPSS are made distinguishable by finely tuning the irradiation conditions of the ultrafast pulses in terms of pulse energy, repetition rate, polarization and so on.

Fine characterization of LIPSS is usually conducted by ex-situ analysis using atomic force microscopy

(AFM) and scanning electron microscopy (SEM). However, these types of post mortem measurements do not allow to easily observe the pulse to pulse evolution of the LIPSS pattern directly within the irradiation set-up. In the following, we discuss the interest of two optical techniques, namely structured illumination microscopy (SIM) and Fourier Ptychograph Microscopy (FPM) for in-situ analysis of micrometric and submicrometric surface structures.

2. SIM FOR IN-SITU LIPSS CHARACTERIZATION

We have recently proposed the use of SIM to characterize the LIPSS formation [4]. By projecting a series of periodic illuminations with a period equal to the objective diffraction limit, a super resolution image with spatial frequencies two times higher than the objectif cutoff frequency can be recontructed [5] (see Fig. 1). In our case, using an UV LED at $\lambda = 405$ nm with an objective of NA = 0.9, yields a Rayleigh criterion of $k_o = 275$ nm. Experimentally, the SIM set-up was to shown to reach a lateral resolution of 155 nm, which is close enough to the theoretical goal of 138nm and sufficient to clearly observe ultrafat-laser induced Low Spatial Frequency LIPSS (LSFL) on metallic glass whose period lies within the 600-700nm range.

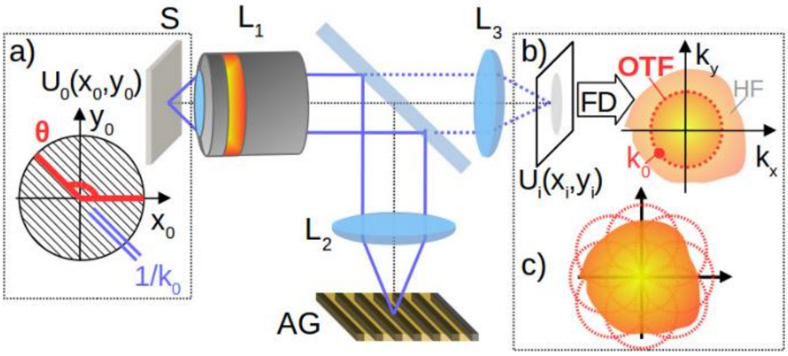

Fig. 1 SIM adapted to LIPSS imaging in reflexion. a) An amplitude grating (AG) is projected on the sample (S) by the 4f system (objective L1 +lens L2). The microscope image field is formed by L3 on the CCD. b) Microscope OTF in the frequency domain (FD) with illustration of the super resolution reconstruction c) for the sample high frequencies (HF).

Figure 2 depicts a comparison of SIM with SEM and regular brightfield optical microscopy (OM). There, it is clear that SIM enables an optical characterization in situ with a sufficient imaging resolution to clearly follow the LIPSS pattern evolution during the multipulse irradiation sequence.

As discussed more in details in [4], several physical mechanisms leading to the formation of the LSFL could be unveiled using the technique such as a phase-locking mechanism that stabilizes and amplifies the ordered corrugation during the multipulse irradiation sequence (not shown here).

If the technique allows for submicrometric in-situ and non-contact analysis of LIPSS, there is not doubt about its interest for ultrafast laser surface structuring applications where this analysis can obviously be utilized as a real-time feedback for process control. However, the sample surface field that can be imaged with SIM is restricted to the objective lens field of view which is typically of a few tens of µm for high NA objectives. This can be a limitation when larger characterization areas are required. Moreover, the working distance of this type of objective is usually very short, below 1 mm. These issues can both be adressed with the technique of Fourier Ptychography Microscopy which is discussed hereafter.

3. FPM FOR IN-SITU LARGE FIELD SURFACE ANALYSIS

Recently, the development of FPM (Fourier Ptychography Microscopy) has drawn a remarkable attention in the field of biomedical imaging [6]. FPM offers the possibility to reconstruct a high resolution image from a series of low resolution acquired through a low NA objective lens with high angle illumination.

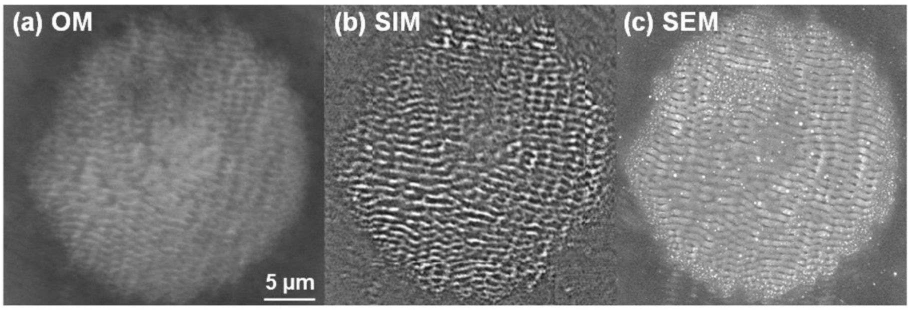

Fig. 2 Visualization of sub-wavelength LIPSS on a metallic glass (Zr-based) surface created after 5 ultrafast laser pulses using a peak fluence of 0.3 J/cm 2 . The same pattern is visualized by a range of imaging techniques. (a) Impact zone observed using a conventional bright field optical microscope (OM) in reflection mode. (b) Impact zone observed in-situ using SIM based technique during structuring. (c) Impact observed ex-situ using a SEM technique.

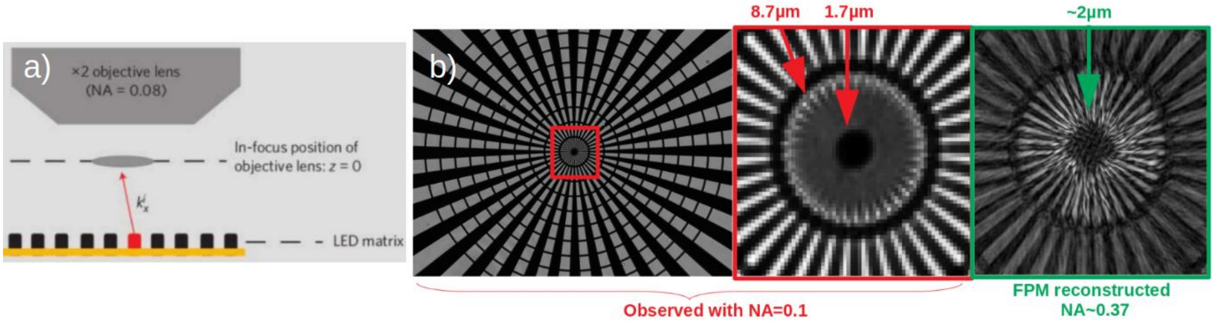

Fig. 3 a) Principle of FPM (adapted from [6]), b) Visualization of a sector target with FPM. Left: original image with low NA objective (0.1) showing a large field of view in the mm range. Center: detail of the original image center showing a 8.7 µm resolution because of the low NA objective. Right: Same area using the FPM reconstruction yielding a better resolution down to 2 µm

This opened the door to large field of view images acquired through an inexpensive low NA objective. The technique was first used for imaging biomedical samples, typically histological cuts. We discuss here the perspective of applying FPM to characterization of laser induced micro-nanostructuring of metallic surfaces.

The principle of FPM relies on a sequential illumination from multiple angle, usually achieved with an array of LEDs (see Fig. 3a)). Each angle of illumination corresponds to a specific zone of the Fourier space, in other words, high angles correspond to high spatial frequency regions in the Fourier domain.

By numerically recombining several angles using an iterative Fourier transform algorithm, a high resolution image can be reconstructed. As a proof of concept, we have performed the FPM technique on a transmission sector target as depicted on Fig. 3b). There, a four times increase in resolution could be achieved using the FPM technique, from 8.7 µm to 2 µm with a field of view in the mm range and a cm working distance. This proof of concept was achieved with a low cost standard LED array. The challenge is now to adapt the technique in reflexion and to push to submicrometric resolution in a reflective scheme.

4. CONCLUSION

Non-contact optical means are ideal tools for in-situ inspection of ultrafast laser induced surface micro and sub micro structures. In particular, we have shown that Structured Illumination Microscopy can offer a sufficient lateral resolution (155 nm) to precisely characterize LIPSS in-situ, enabling to follow the pulse to pulse pattern evolution. In order to expand the concept to larger field of view ond longer working distance, we explore the interest of Fourier Ptychography Microscopy. A proof of concept on a transmission resolution target has shown that 2 µm resolution could be reached experimentally with mm field of view and cm working distance. Further developments of FPM for in situ characterization of LIPSS include adaptation to reflective imaging and increase of the resolution power.

We thank the French ANR Labex Manutech-SISE funding (Sermonis and Ptycholaser).

REFERENCES

[1] P. Bizi-Bandoki, S. Benayoun, S. Valette, B. Beaugiraud, E. Audouard, "Modifications of Roughness and Wettability Properties of Metals Induced by Femtosecond Laser Treatment," Appl. Surf.e Sci. **257**, 5213-5218 (2011)

[2] B. Dusser, Z. Sagan, H. Soder, N. Faure, J. P. Colombier, M. Jourlin, E. Audouard, "Controlled Nanostructrures Formation by Ultra Fast Laser Pulses for Color Marking," Opt. Express **18**, 2913-2923 (2010)

[3] V. Dumas, A. Guignandon, L. Vico, C. Mauclair, X. Zapata, M. T. Linossier, W. Bouleftour, J. Granier, S. Peyroche, J. C. Dumas, H. Zahouani, A. Rattner, "Femtosecond Laser Nano/micro Patterning of Titanium Influences Mesenchymal Stem Cell Adhesion and Commitment," Biomed. Mater. **10**, 5 055002 (2015)

[4] A. Aguilar, C. Mauclair, N. Faure, J. P. Colombier, R. Stoian, "In-situ high-resolution visualization of laser-induced periodic nanostructures driven by optical feedback," Sci. Rep. **7** 16509 (2017)

[5] M. G. L. Gustafsson, "Surpassing the Lateral Resolution Limit by a Factor of Two Using Structured Illumination Microscopy". Journal of Microscopy **198**, nº 2 (1 mai 2000): 82‑ 87 (2000)

[6] G. Zheng, H. Roarke, Y. Changhuei, "Wide-field, high-resolution Fourier ptychographic microscopy," Nature photonics **7**, nº 9, 739‑ 45 (2013)