مجلة جامعة تشرين للبحوث والدراسات العلمية – سلسلة العلوم الأساسية المجلد (34) العدد (3) 2012
Tishreen University Journal for Research and Scientific Studies - Basic Sciences Series Vol. (34) No. (3) 2012

Polarization Effect on Laser Induced Surface Structure on Copper Surface

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(Received 23 / 4 / 2012. Accepted 13 / 9 /2012)

□ ABSTRACT □

Femto-second laser pulses have been used to investigate Laser-induced-periodic-surface-structures (LIPSS) formation. Scientists made great efforts to understand the involved mechanism, but the phenomenon is not yet fully understood. In this work, we used the previous techniques which lead us to control the optical properties of Copper surface and create random Nano-roughness (NR). We focused first a linearly polarized ruby laser on a surface of the Copper material, where we obtained parallel ripples, and then we looked for the macroscopic visual effects related to the type of surface Nano-structure.

We used Fourier space to confirm the periodic Nano-structures on the metal surface. Which in real space presumes a semi-anisotropic distribution of periodicities and explains why the iridescence can occurs in a range of angles, if the grating is rotated along an axis perpendicular to its surface. As a result: Nano-structure formation seems possible on the surface of the Copper material.

Key words: Polarization, LIPPS, Copper surface, Femto-second laser, Nano-particles, Fourier space.

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تأثير الاستقطاب في البنية السطحية الدورية المشكلة ليزرياً على سطح معدن النحاس

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(تاريخ الإيداع 23 / 4 / 2012. قُبِل للنشر في 13 / 9 /2012)

□ ملخّص □

إن تشكيل بنية سطحية دوريةَ ليزرياً (LIPSS) باستخدم اليزرات النبضية (ليزر الفمتو–ثانية) على سطوح المعادن هو أحد الاتجاهات العلمية الطروحة بقوة بسبب تطبيقاتها العلمية والهندسية الممكنة. قام العلماء في هذا الإطار بجهد كبير لفهم تلك الآلية المعقّدة التي لا تزال غير مفهومة بشكل تام. استخدمنا في هذا العمل التقنية السابقة على سطح معدن النحاس لتأكيد إمكانية تشكيل أثلام دوريةَ و جزيئات نانويّة على سطح معدن النحاس عند مسحها ضوئياً باستخدام الليزر. ركزنّا عملنا على تأثير الاستقطاب الخطي لليزر في سطح النحاس. ثم درسنا تأثير خواص الجزيئات النانوية المتشكلة في البنية الجهرية للمعدن. كنتيجة لهذا العمل يمكن التأكيد أن تشكّل الجزيئات النانويّة على سطح معدن النحاس يبدو ممكناً ويؤثر تأثيراً مباشر في الخواص الضوئية الجهرية للمعدن.

الكلمات المفتاحية : الاستقطاب، النحاس، ليزر الفمتو–ثانية، الجزيئات النانومترية الأبعاد، فضاء فوريه

تم إجراء القياسات في معهد الأطياف الفائقة السرعة في المدرسة العليا التقنية (LSU– EPFL) لوزان–سويسرا في عام 2009

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Introduction:

Laser-induced-periodic-surface-structures (LIPSS) were first observed by Birnbaum[1] on several semiconductor surfaces. He focused a linearly polarized ruby laser on the surface of the material and obtained parallel ripples. Since then, such structure were observed on a wider variety of semiconductors [2] [3] [4] [5] as well as on metals [6] [7] [8], dielectrics [9] [10] and, more recently, on polymers [11] [12] [13] [14]. Most of the investigated material were processed with laser at various wavelength [15] [16], operate on pulsed regiem [17] [18] [19] as well as continuous regiem [20].

Over the last decade, scientists have been able to produce increasingly short pulses, thus reaching huge peak power. While nano-second pulse laser were extensively used up to 1980, the development of mode-locked laser allowed ultra-short pulse generation to be achieved at a high repetition rate. A new type of process could take place: Ultra-fast, nonthermal melting. This process could arise from a strong modification of inter-atomic forces owing to laser-induced modification of electron distribution. The atoms begin immediately move and gain sufficient kinetic energy to induce melting in a much lower time-scale than the one needed to convert electronic energy into thermal motion (several picoseconds). Observation of such phenomenon could potentially lead to a better understanding of the processes behind nano-structures formation. Also it can lead to thoughts, that short pulses laser investigate LIPSS formation on the surface of such metals [21] [22] [23].

Since the discovery of LIPSS in 1965, scientists made great effort to understand the involved mechanism. The phenomenon is not yet fully understood. However, there is an explanation commonly admitted today. It is provided by the scattered wave model: The light impulse on the material scatters in all directions along the surface and then interacts with the incident light, which produces interference fringes. Either stated as being a dipole array [24] or plasma [25]. The nature of the field supporting the scattered wave became a subject of controversy. However, the concept of interference seems to be commonly accepted and was the subject of theoretical investigation. Zhou, and co-workers [26] proposed that the surface inhomogeneities, as being a combination of many gratings with different special periods. The incident light could be diffracted by a given grating into a given order that interferes with incident wave and create interference fringes. These coloring fringes, whose period equals to one of the grating emerge from, shape of the energy deposition. Nano-structures are thought to energies from this inhomogeneous energy deposition or, according to the terminology of Slipe et al. [27], from selective energy deposition into specific Fourier component of the initial surface inhomogeneities. This statement is consistent with the influence of perturbing treatment on the periodicity of LIPSS (observed on polymer [28]). Moreover, the selection of one grating among the other is explained in term of feed-back. This mechanism is based on the phase relationship between the initial grating and the related interference fringe. If they are in phase, positive feed-back amplifies the initial grating and rise periodic nano-structures. If they are not, negative feed-back keep the initial grating flooded among the other inhomogeneities.

From the above model, we can get an idea about the number of parameters that could potentially influence LIPSS formation. However, there are few parameters that are found to be at the same time experimentally accessible and determinant for nano-structure formation: Namely, the laser fluence, the number of pulses, the beam localization and the polarization of light. The three former are related to the density of energy on the metal. As we will see, intensity strongly influences the type of nano-structure that will be formed.

Periodic-structures obtained under pulsed laser irradiation are not systematically. Another type of structure could also be observed: the random nano-roughness (NR).

The polarization does determine the orientation of the fringes. Most of the studies were done using linearly polarized light. Under this type of light, the most widely observed ripples are perpendicular to the light polarization (S-type fringe). However, ripples can also be formed parallel to the direction of the latter, giving rise to C-type fringes. Extensively studied by S.E. Clark et al. [15]. Young et al. have related the formation mechanism for each type. They proposed that the structure perpendicular to the polarization does emerge from non-radiative field structure, which they called radiation remnant [29]. The C type fringes induce a localized melting of the surface which shape the grooves perpendicularly to the polarization. On the other hand, the C-type fringe is proposed to emerge under a uniformly melted regime in which surface Plasmon are involved [30]. In this regime, a thin layer of molten material is thought to support ripples wave. The latter"froze" before being damped and thus grow a periodic structure parallel to the polarization. This one is smoother than the previous one and have a larger period. While linear polarization has been extensively investigated, it has to be mentioned that few works [31] [32] also studied LIPSS formation under circular polarization.

Aim and importance of this work

In the present work we focused on the macroscopic visual effect (iridescence, shading and darkening) related to the type of surface nano-structure. As mentioned before, nano-structure formation studied on such metals, and it seems possible on virtually any type of material. Our goal is to extrapolate the results to other types of metals. Thus, using a phenomenological approach, we make an effort on finding quantities that are intrinsic to the material under investigation. For this purpose, we determined the domain of existence of the previously mentioned nano-structure (LIPSS and NR). Another type of structure, the spherical nano-particles, have also been identified and related to optical darkening of the metal surface. The optical properties of these effects on the Cu metal have been recognized.

Methods and Experiment

A mode-locked of Ti:Sapphire laser were used (figure 1). The oscillator yields a nearly square wavelength distribution centered at 800 nm. The pulse width is approximately 20 femto second. Using a beam splitter, we redirected the second order diffraction peak of our beam to an amplifier. This way, we obtained a 50 fs pulse with energy of about 0.5 $mJPulse^{-1}$ at a repetition rate of 1 KHz. For more energy gain, we took the light that is linearly polarized along the horizontal direction from an amplifier. Then, we focused the laser beam with a 500 mm focal length lens on a pure (99.99%) copper surface polished with a 1 micrometer grade abrasive surface. The incident laser beam focused normally on the copper surface. According to both the beam-profiler and the measurement from SEM picture, uses a focused beam of high-energy electrons to generate a variety of signals at the surface of solid specimens. The signals that derive from electron-sample interactions reveal information about the sample including external morphology (texture), chemical composition, and crystalline structure and orientation of materials making up the sample. In most applications, data are collected over a selected area of the surface of the sample, and a 2-dimensional image is generated that displays spatial variations in these properties. The spatial resolution is 50 to 100 nm. The spot diameter, at the working focal length, is equal to 300 μm (FWHM=127 μm). In order to perform the

surface treatment over a large area, the copper samples were mounted on a support moving in a plane by means of two different motors. One moving horizontally, allowing different scanning speeds, and another moving vertically in order to separate the different laser prints from each other. A computer controlled mechanical shutter was synchronized with the vertical motion in order to prevent the laser to shine on the metal while positioning it. We had the possibility to cover a wide range of energy density either by varying the scanning speed or by attenuating the beam with filters.

Once ablated, the copper surface was characterized using Scanning Electron Microscop (SEM). The information contained in the image does not include explicitly the depth of the nano-structures. We then have a mean to characterize qualitatively the amount of damage sustained by a surface exposed to a given amount of energy. This information is however not easy to qualify since the brightness and contrast are different from one picture to another.

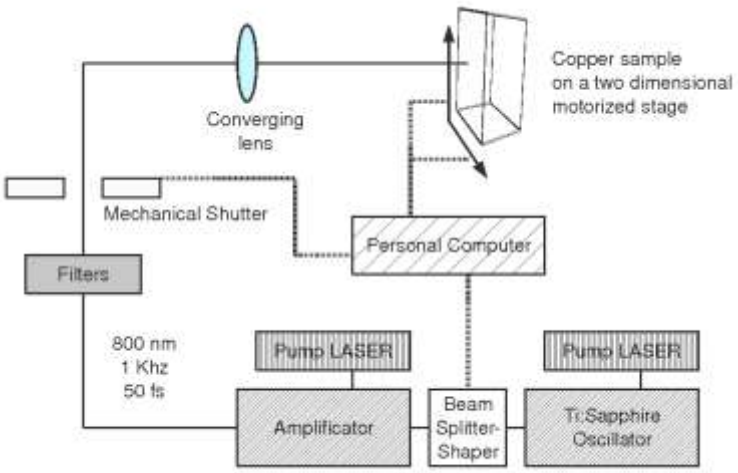


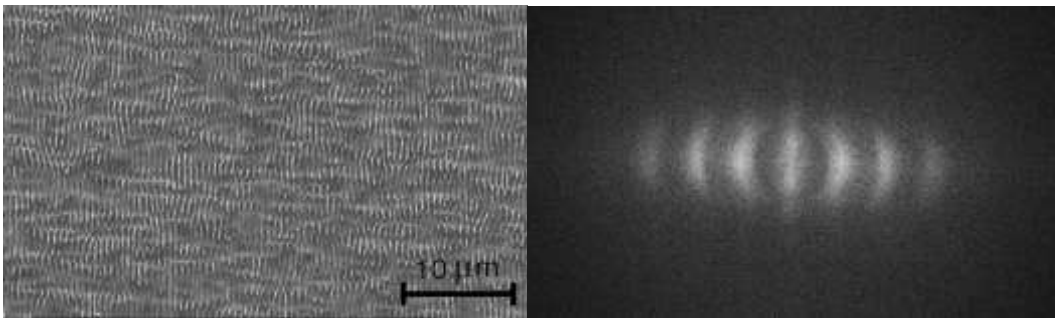
Figure 1: Schematic of the experimental setup.

As we are concerned about periodic structures, we used the Fourier transform of the picture. Fourier analysis gives frequency domain information which is related to the amplitude of the periodic variation: the bigger the periodic variation in real space, the higher the peak in Fourier space relative to noise and to other frequencies. The power spectra have thus been used to qualitatively characterize the damage amplitude.

The most apparent frequency domain information can be seen from two dimensional

Fourier transformation. For instance, figure 2 shows a typical periodic structure both in Real space and in Fourier space. Such two dimensional frequency domain information was obtained by Young et al. [33] by means of the diffraction pattern of a CW Ar^+ laser shone over the periodic surface structure. The colored fringes of their Fraunhofer diffraction pattern occur at inverse of the grating periods. As we were also able to extract this information from our frequency domains, we are confident in the physical meaning of such an approach [34]

One characteristic of the two-dimensional power spectra is the number of fringes that are present. Indeed, at most three pairs of fringes can be seen in our entire sample.



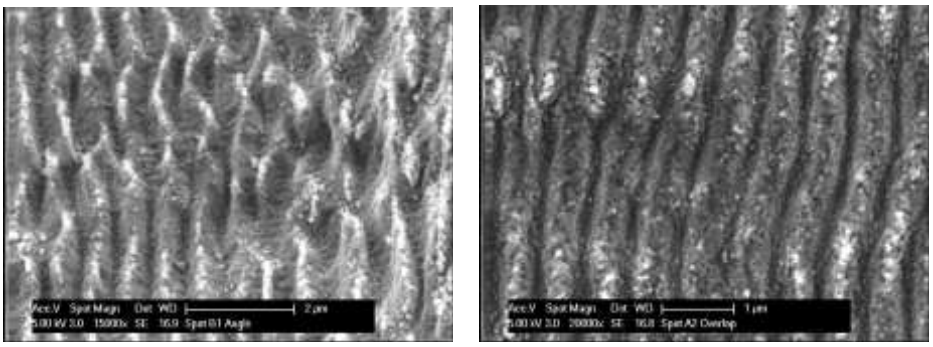
(a) Real space LIPSS **(b) Fourier space components**

Figure 2: SEM picture of a typical LIPPS and its corresponding 2D Fourier Transformation

Each one being separated from the other fringes or from the center by the same distance. We think that this is a consequence of the presence of two types of structure that forms on top of the main relief. Namely, the crest and the valley, give what we called the first and second harmonic respectively. Figure 3 shows a detailed view of these reliefs. The fundamental harmonic emerge from the largest period present in the picture is the distance between two consecutive grooves. In figure 3.a. it crests emerging in between two grooves. This picture was taken under a slight inclination of the sample, and gives an appreciation of the third dimension of the relief. Such structure shows the first harmonic in the power spectrum of the SEM picture. While in figure 3.b, it shows valley in between two grooves. They are responsible of the second harmonic of the fundamental frequency. However, we have to be careful about information from SEM pictures. They can show artifacts in the frequency domain because color change could be uncorrelated with relief variation. A grating will be formed with a period (line spacing) Λ given by relation 1.

$$\Lambda = \lambda/(\eta \pm \sin\theta). \qquad (1)$$

Where $\boldsymbol{\eta}=\mathrm{Re}[\varepsilon/(\varepsilon+1)]^{1/2}$ is the real part of the effective refractive index of the air-metal interface for surface plasmon's, $\boldsymbol{\varepsilon}$ is the dielectric constant of the metal, $\boldsymbol{\lambda}$ is the wavelength of the laser light, and $\boldsymbol{\theta}$ is the incidence angle of laser light.



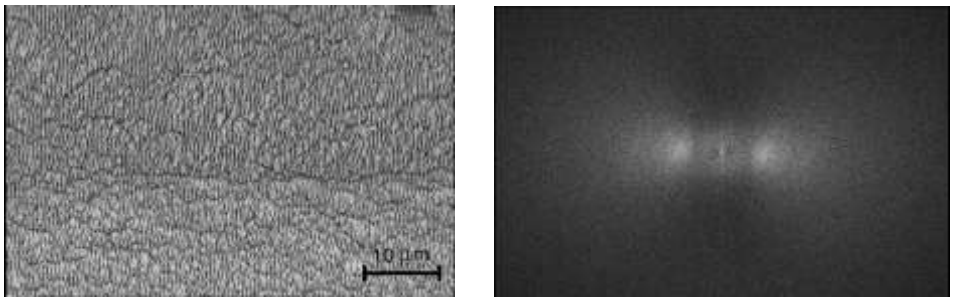
(a) **(b)**

Figure 3: Detail of two types of structure that can be superimposed onto the main periodic pattern.

Since integer-sub-wavelength structures have already been observed on quartz [35] and measured on polymer by AFM [36], we are confident about their existence. However, color based relief information can show artifact without physical meanings. If SEM pictures suggest the mentioned relief, frequency domain spectra are not a proof in themselves of their existence. Further investigation should be done using AFM in order to confirm this observation. For these reasons, harmonic characterisation has to be considered with care since we are dealing with SEM pictures.

It is also important to notice the distribution of intensities in the frequency domain: As shown in figure 4, the first"peak" in the Fourier space is spread over a wide frequency domain. Because the corresponding LIPSS seem "damaged" relatively to the one of figure 2a, the width of the peak can be seen as an information about the consistency of the periodic structure. Their amplitude is an indication on the occurrence of the related period.

In order to compare more quantitatively the Fourier space information, we performed unidimentional Fourier transformation of the SEM pictures along the direction of the polarization (perpendicular to the fringes). The picture have been processed as follow: Each row of pixel has been Fourier transformed independently. We then summed up the logarithm of the amplitude of each transform. This has been done in order to cancel the noise due to inconsistencies along the considered direction. The resulting power spectra were then corrected relatively to its background"noise" and that has been set to 0. Finally, the y-axis has been normalized to one. With these corrections, we obtained spectra which are independent of the brilliance or the contrast of the different picture (see figure 5). This way, we think it is reasonable to compare between different pictures them.



(a) **(b)**

Figure 4: SEM picture of damaged LIPPS (a) and the corresponding 2D Fourier transformation (b)

We performed experiments with fast scans, where each spot is illuminated by a single light pulse, and slower scans, where the consecutive pulses have some spatial overlap. Moreover, experiments were also done with multiple scans, where several fully overlapped horizontal scans were performed before advancing the sample in the vertical direction. Additionally, multiple scans were also performed with partial vertical overlap between the horizontal stripes. It has to be mentioned that, when a surface is excited with an energy density (per pulse) that is at the single pulse damage threshold (Energy density of $2Jmm^{-2}$), the first pulses have little effect, whereas any damage produced strongly increases the interaction with the following pulses. This sensitization effect is a consequence of an increase of the roughness of the surface that decreases its reflexivity. Thus, the energy is absorbed more and more effectively during ablation [24]. This effect, quantified by reflexivity measurement [21][22] is not taken into account in the expression of the integral intensity.

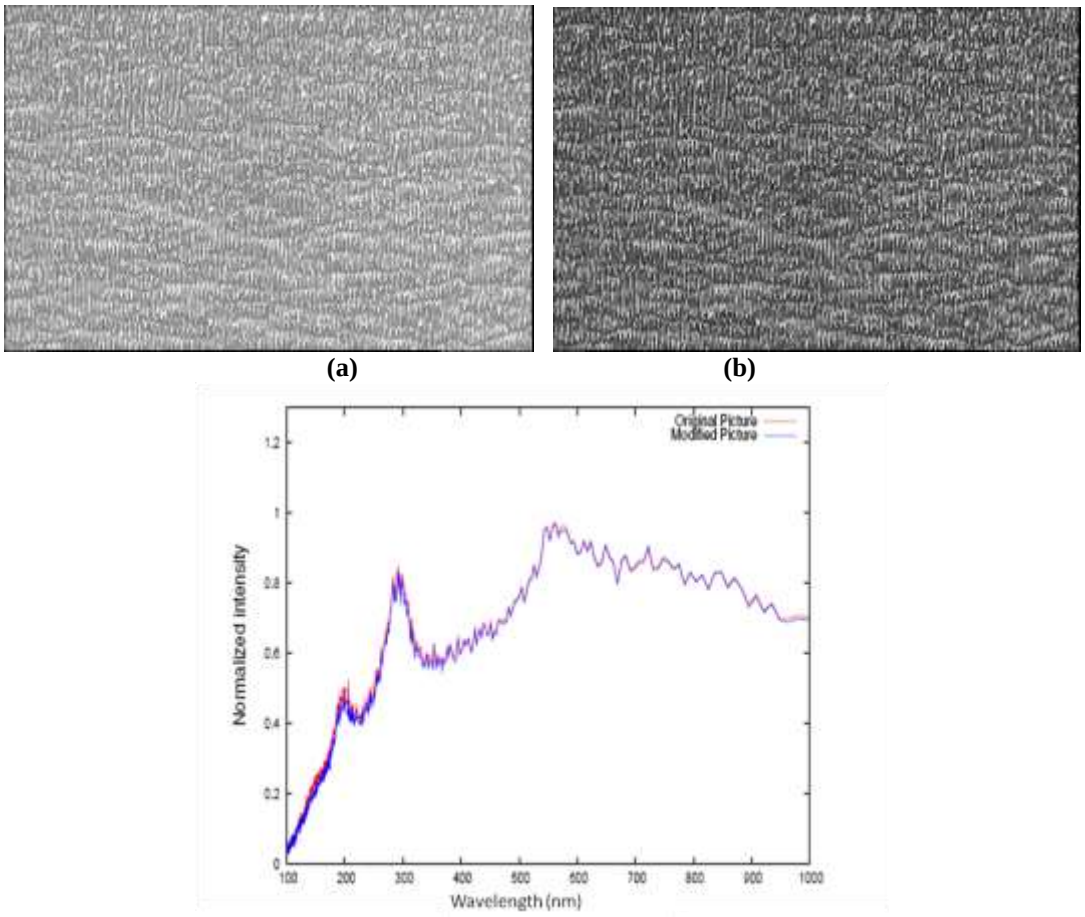


Figure 5: The original SEM picture (5a) and the same picture with contrast and brilliance modification.(5b). In figure 5c, the background correction and the y-axis normalization make the power spectra invariant

Results and discussion

We begin the characterization by comparing surface ablated at different scanning velocities. All the LIPSS that we observed appear after multiple laser pulses above ablation threshold (Energy densities are more than $1 Jmm^{-2}$). We did not perform such an analysis for random nano-roughnes (NR), but we proposed a domain of existence of each observed structure.

Figure 6 compares the periods present in three different spots of sample presented in figure 2. We observe a shift of the wavelength towards higher values (see the values in the inset of the figure) when weaker energy density is used. This has more to do with the depth of the valley which increases with energy density than with sub-wavelength structure [23][36]. We think that higher integral intensities favor the amplification process by virtue of the positive feedback mentioned in the introduction. The sharpening of the relief confirms the groove, the crest and the amplitude of the corresponding period increase. Indeed, this period was already present in smoother grating but has been"confirmed" by following energy impulsion. This effect can also be observed for the integer sub-wavelength structure present in the picture.

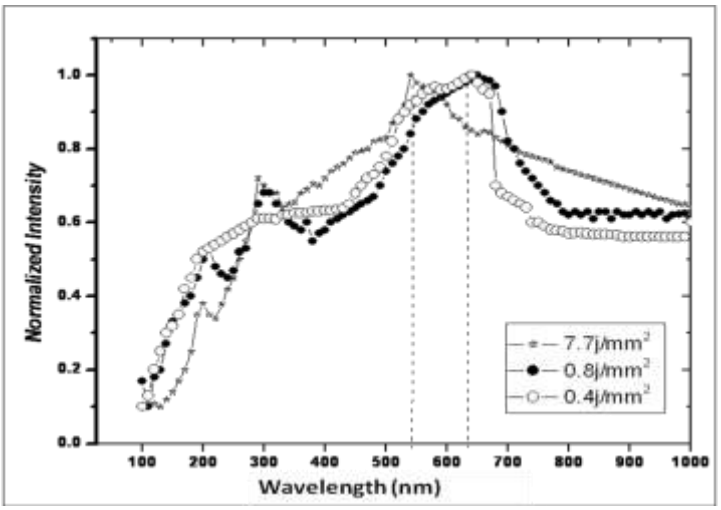


Figure 6: Grating wavelength of three copper surfaces ablated at three different integral energy density for spot A1,B1 and C8 respectively. For the spot A1, the three peaks have their maximum at 197, 296 and 560 nm. Spots B1 and C8 share their common peak at 667 nm.

Another interesting feature of figure 6 is the disappearance of the integer sub wavelength peak with the decrease of integral energy density. The peaks at about 200 and 300 nm vanishes under a wide distribution of frequency, while the main period persists. This suggests that the main period appear first and that it is shaped by following energy impulsion.

As we just see, the amount of energy per unit surface is an important quantity that influences the relief of the LIPSS. This information however, is not sufficient. The rate at which this energy is delivered is also a key parameter. This is highlighted in the three graphs of figure 7. Each one compares the frequency domain of two different integral energy densities in three different copper samples. The graph at the top compares the structure of surface ablated with 9.9 and 7.9 Jmm^{-2}. The difference in integral energy density is about 2 Jmm^{-2} but the frequency domain information is essentially similar considering the position of the peak and their relative intensities. On the other hand, the graphs at the middle and at the bottom of figure 7 show the frequency domain information of spots ablated at integral intensities only slightly different. The difference in frequency domains is obvious and is related to the amount of energy per pulse. It is possible that pulse energy is determinant in the generation of first and second order.

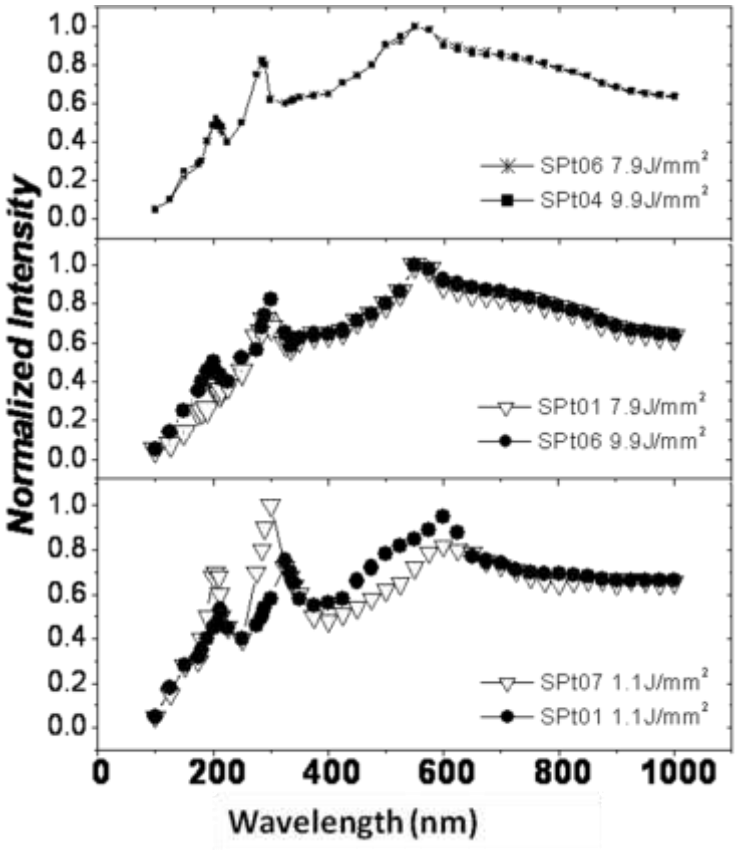


Figure 7: Frequency domain information of spot ablated at different integral energy density. The upper graph is given as a reference to be compared with and show two spots ablated at different intensities but equal pulse energy. The second and third graph show spots ablated at about the same intensity but with difference pulse energies.

Figure 8 shows the periods present in three surfaces where periodic structures begin to disappear. The period of a sample, where typical LIPSS are present, is shown as a reference. As we can observe, the spot A3still contain a period related to the fundamental frequency. However, it is not well defined anymore. At higher intensities, this peak vanishes under a continuous distribution of periods, indicating the predominance of random-NR. In order to define threshold intensity for the formation of LIPSS, we have to consider when does the last peak disappear. However, the definition of a criterion intended to limit the transition energy could be very arbitrary. For this reason, we prefer to talk about a range of intensities within which the transition occurs. From the Fourier domain we presume this range to be between 0.3 and 0.09 Jmm−2.

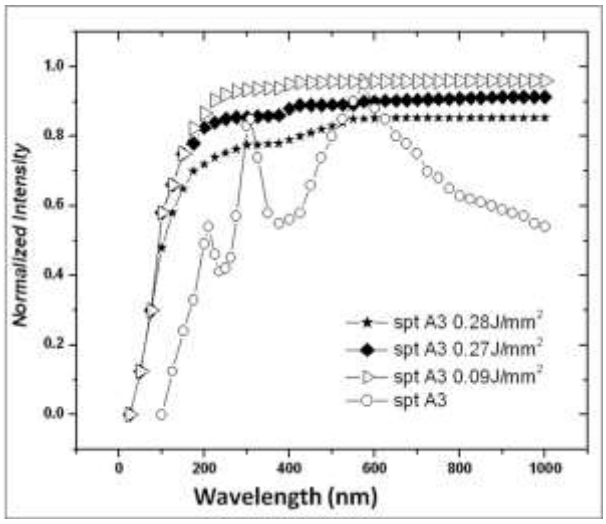


Figure 8: Transition between Laser Induced Periodic Surface Structures, and random nano-roughness. Sample (Spl12) with black line is related to surface with typical LIPSS, and is shown to compare with sample (Spl13) that vanishing fundamental period.

In reality, Fourier analysis will not be useful anymore and further information will be limited to the domain of existence of the so called random nano-roughness. The pulse energy is determinant for the type of structure that will be formed. Thus, the boundaries of the proposed domain are thought to fluctuate with the variation of laser fluence. From several SEM pictures showing surface of samples, it deduce two domains: the domain of existence of a surface homogeneously covered with nano-roughness and the one of a surface where only partial ablation occur. The first domain begins at 0.09 Jmm^{-2} and extends up to 0.045 Jmm^{-2}. Lower intensities do not succeed in covering homogeneously the surface with NR anymore. Table 1 summarizes the range of laser pulse energies and the formations on the samples surface (type of structures).

Table1 Laser pulse energies and type of structures

Energy Range (Jmm−2)	Type of structure
0.00 - 0.045	Partial Nano-Roughness structuring
0.045 - 0.09	Homogeneous Nano-Roughness structuring
0.09 - 0.30	Transition between Nano-Roughness and LIPSS
0.30 - 0.60	Fundamental harmonic (ω) LIPSS
0.60 – 20	Multiple harmonic (2ω, 3ω,..) LIPSS
> 40	Damaged LIPSS

Conclusion:

In this work, we studied the effect of energy density on the formation of nano-structures. We defined the integral energy as the actual amount of energy being shown on the metal surface. Based on this quantity, we established the domain of existence of the different observed laser-induce-surface-structures.

In particular for the LIPSS, we characterized the presence of first and second harmonics (LIPPS as a function of laser pulse energy) as well as the depth of the grooves.

We also defined the domain of existence of random nano-roughness and its related macroscopic visual effect.

We used Fourier transform in order to characterize periodic reliefs. Valuable information's, such as the value of the main period, its consistency along one direction as well as its importance with respect to other structures, can be obtained from such a method. However, we have to be careful since no relief information is directly encoded in SEM pictures. In particularly, the characterization of harmonic is not rigorous and should be followed by AFM measurement in order to confirm their existence.

From this short study, we also realised the importance of the regime at which the energy has to be delivered in order to tune the relief. Pulse energy as well as integral intensity are key parameters that determine the shape of the grating. The integral intensity however, as we defined it, lack precision. Indeed, the sensitisation effect have to be taken into account in order to be able to relate the amount of energy effectively transferred to the metal with quantities such as ionization energy or band gap energy. This way, the relationship between laser fluence and nano-structure could be generalized.

We mentioned how a copper surface can be darkened and which kinds of structure are involved in this process. It is relevant to determine if the dark surface results from copper oxidation (CuO) or is a consequence of spherical nano-particles. Where, the two reasons show same coloring properties. The possibility that oxidation is responsible of this effect should be discarded if the process could be reproduced on gold, where oxidation is not likely to occur. The surface colorized metal resulting from light diffraction seems independent from the angle of incidence light or laser polarization.

Finally, a deeper investigation could be done on how to design grating in order to diffract preferentially a specific wavelength. This could be done by shaping the relief of the grating. The wavelength selected this way is known as the blaze wavelength. As we discussed before, we have at least two means to our disposal in order to shape the periodic structure. Namely, the integral intensity and the pulse energy. Thus, we think it is reasonable to expect a possibility to shape the grating in order to diffract preferentially a specific color.

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