FEATURES OF CHANGES IN THE NANOSTRUCTURE AND COLORIZING OF COPPER DURING SCANNING WITH A FEMTOSECOND LASER BEAM

G. Liedl [1], R. Pospichal [1], S.P. Murzin [2]
[1] Vienna University of Technology, Vienna, Austria,
[2] Samara National Research University, Samara, Russia

Abstract

We have studied the nanostructuring and colorizing of the copper surface by scanning with a femtosecond laser beam with a near-Gaussian beam profile. The experimental studies were conducted using a femtosecond laser comprising a Ti:Sapphire oscillator and a multi-pass amplifier with the maximum pulse energy of 0.7 mJ, pulse frequency of 1 kHz, and pulse duration <30 fs. It is shown that the use of a short-pulsed femtosecond laser leads to the formation of wavelength scale periodic surface structures and eventually increases the brightness of the color of the copper surface. It is revealed that via reciprocally scanning the copper surface by multiple ultrashort laser pulses with a weakly asymmetric spatial energy density distribution and an energy density below the material ablation threshold, it is possible to create a combined nanostructure composed of low-spatial-frequency laser-induced periodic surface structures coated with nanoscale roughness. It is shown that relatively minor changes in the nanostructures obtained by scanning the copper surface by multiple ultrashort laser pulses can lead to a significant change in the color during surface colorizing.

Keywords: femtosecond laser beam, copper colorizing, nanostructure, forward and reverse scanning, energy density.

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Introduction

Diffraction gratings, being dispersive optical elements, are widely used for spectral instruments and in other fields of science and technology [1]. Most spectral instruments, except for a few produced by diamond cutting techniques [2], use holographic diffraction gratings, which are produced by using the method of recording an interference pattern from a laser source [3]. A photosensitive material applied to a substrate is used for such a registration. After a chemical treatment of the material, a relief-like structure of lines with a quasi-sinusoidal profile shape is formed on the surface of the substrate. An additional promising method for manufacturing of reflective diffraction gratings is the irradiation of metallic samples by ultrafast laser pulses. Such an irradiation can cause the formation of periodic structures on the surface of samples. The laser-induced periodic surface structure acts actually as a diffraction grating for light which illuminates the surface with a wavelength close to the periodicity of the structures. Such ultrafast laser generated periodic structures are able to decompose incoming light into its spectral components, generating a bright colorful appearance of the sample surface.

Ultrafast lasers, which is a generic term for picosecond and femtosecond lasers, have created a new path to laser processing of materials in terms of the capabilities in ultrahigh precision micro- and nanofabrication of not only opaque but also transparent materials and threedimensional (3D) and volume processing [4 – 6]. The pulse width of ultrafast lasers is defined as several tens of femtoseconds to tens of picoseconds, where a pulse width shorter than picoseconds is typically used for fundamental research, while longer pulses are used for commercial and industrial applications because of the high output power and high reliability. Laser nanostructuring using an ultrafast pulse laser source has been used to induce surface micro/nanostructures on metals and thus to obtain surfaces with unusual optical properties or wettability [7 – 10]. For a number of technologies of computer optics, the problem of adhesion is extremely topical [11 – 15].

For the first time, the effect of colorizing of a metal surface by means of ultrashort pulses was described in [16]. Such a method of generating surface periodic structures by means of ultrashort pulses allows the colour marking of surfaces of almost any solid structure, which is especially important for materials that are weakly oxidized or have an opaque oxide (for example, copper). Ref. [17] shows a possibility of achieving material modifications using ultra short pulses, via polarization dependent structures generation, that can generate specific colour patterns. These oriented nanostructures created on the metal surface, called ripples, show a periodicity typically smaller than the laser wavelength and in the range of the visible spectrum.

Ripples with a subwavelength period were induced on the surface of a stainless steel (301 L) foil by femtosecond laser pulses [18]. By optimizing the irradiation fluence of the laser pulses and the scanning speed of the laser beam, ripples with large amplitude (~150 nm) and uniform period could be obtained, rendering vivid structural colours when illuminating the surface with white light. Ref. [19] confirms that the colorizing phenomenon mainly ascribes to the grating diffraction effect of the laser-induced periodic surface ripples, which would help to enable the flexible control of the colorizing effect induced by laser processing on pure copper. Ref. [20] reports the modification of optical properties of 304 stainless steel surfaces by femtosecond laser direct writing. Regularly arranged ripples with a spatial period of ~700 nm were obtained, rendering vivid structural colours when the surface was illuminated with white light. This study adds a new parameter, the scanning pitch, to the list of parameters in the production of controllable colorized metal.

In [21] functional copper surfaces combined with vivid structural colours and superhydrophobicity were fabricated by picosecond laser processing. Laser-induced periodic surface structures (LIPSS), i.e. ripples, were fabricated by picosecond laser nanostructuring to induce rainbow-like structural colours which are uniquely caused by the grating – type structure. The effects of laser processing parameters on the formation of ripples were investigated. The increased amount of nanoscale structures decreased the adhesive force to water and increased the contact angle simultaneously. Ref. [22] clarified the mechanism underlying the transition of picosecond laser microstructured aluminium surfaces from a superhydrophilic nature to a superhydrophobic one under ambient conditions. The aluminium surface studied exhibited superhydrophilicity immediately after being irradiated by a picosecond laser. Periodic microstructures with different topographies were fabricated on copper surface via femtosecond laser irradiation [23]. The topography of these microstructures can be controlled by simply changing the scanning speed of the laser beam. After surface chemical modification, these as-prepared surfaces showed superhydrophobicity combined with a changed adhesion to water. Surfaces with deep microstructures showed self-cleaning properties with extremely low water adhesion, and the water adhesion increased when the surface microstructures became flat.

Ref. [24] presents a method for manipulating the nanoscale surface topology, as well as the chemical composition of titanium surfaces by scanning a femtosecond laser beam with an asymmetrical spatial fluence distribution over the surface. For the experiments, an asymmetric beam, which has had initially a Gaussian energy distribution, has been deformed by using a diaphragm where only half of the beam could pass. However, no information was provided as to whether there can be significant differences in the formed relief during forward and reverse scanning by a weakly asymmetric beam of a femtosecond laser with a nearly Gaussian energy density distribution without any beam masking. It is possible to assume that phenomena found for titanium could be observed for other materials, too. But it is plausible that individual features will be present. Additionally, it is advisable to study the possibilities of the formation of various combined nanoreliefs during a forward and reverse scanning of a material surface with a beam of ultrashort pulses with an energy density below the material ablation threshold. Besides, as such a change of topological properties influences copper colorizing and is an opportunity to receive bright colours. The purpose of this research is to study of the features of change of topological properties and colorizing of copper during forward and reverse scanning with a femtosecond laser beam with a nearly Gaussian energy density distribution in the range below the material ablation threshold.

Results of experimental studies

For the performance of experimental studies a femtosecond laser was used: Ti: Sapphire oscillator and multi-pass amplifier with max. 0.7 mJ pulse energy, 1 kHz pulse frequency and pulse duration < 30 fs. Beam mode TEM_{00}, pulse contrast $> 10^9 : 1$. Central wavelength was 800 nm, bandwidth approx. 100 nm. The laser beam has been focused on the surface of pure copper plates by a metal mirror with a focal length $f = 101.6$ mm; for sample processing a 2D displacement system was used. Spot size was adjusted to get the energy density of 1.49 J/cm². Spot sizes were 0.3 mm height and 0.23 mm width, spot area 0.0538 mm².

The sample was produced by scanning with the focused laser beam over a surface of a copper plate. The processed area size was 20×20 mm². The plate has been moved 20 mm in the positive *x*-direction during laser exposure. At the end of the line, the laser was turned off and the plate was displaced by 0.3 mm in the positive *y*-direction. Following that, the laser was switched on again and the sample was moved in the negative *x*-direction. The whole processing cycle was repeated until an area of 20×20 mm² has been structured by ultrashort laser pulses.

Thus, the copper surface has been treated with femtosecond laser pulses. Samples have been moved during processing in *x*-direction first, and then, after a small displacement in *y*-direction the moving direction was reversed. So, a line-by-line processing, first in +*x*-direction, followed by processing in *x*-direction could be achieved. Speed of motion was 135 mm / min, distance between lines 0.3 mm, overlap ~ 0 %. From experiments were able to observe that visible colours (with a more blue or more red appearance) depend only on the direction of movement. Fig. 1 shows an image of the treatment zone obtained using a metallographic optical microscope. The arrow indicates the direction of movement of the sample during laser processing.

A similar pattern was observed when spot size changes to 0.26 mm height and 0.27 mm width, spot area 0.07 mm². In this case the energy density was 1.05 J/cm², speed of motion 180 mm/min, and the distance between lines 0.28 mm. Fig. 2 shows the zone of a single pulse action on the sample surface. Results of our experiments indicate that a forward and reverse scanning of a copper surface by a beam of ultrashort pulses with a weakly asymmetric spatial distribution in a multipulse mode and an energy density below the material ablation threshold can lead to significant colour differences between different scanning directions.

It is known that during normal incidence of femtosecond, linearly polarized laser pulses, low-spatial-frequency laser-induced periodic surface structures (LSFL) are formed on the copper surface, which demonstrate the effect of diffraction staining [25, 26]. That is, the appearance of a surface relief demonstrating properties of a diffraction grating in the visible range is observed. However, generated at normal incidence, LSFL do not allow to obtain bright colours in the long-wavelength part of the visible electromagnetic radiation [27, 28]. By using a femtosecond laser with a pulse duration of 30 fs the formation of near-wavelength periodic surface structures makes it possible to increase the brightness of colorized copper surfaces.

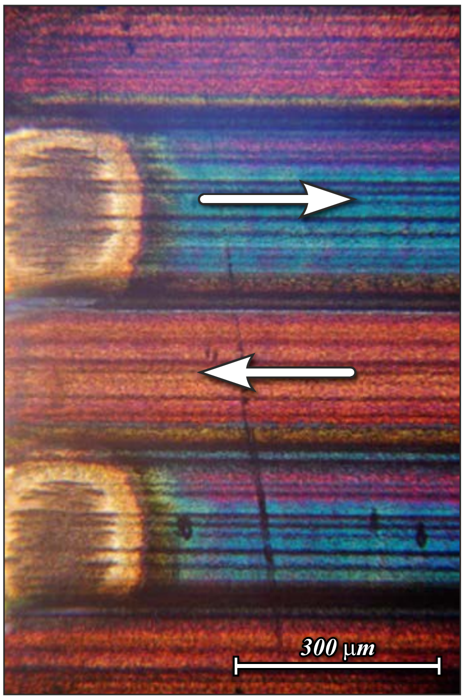

Fig. 1. Image of the treatment zone obtained using a metallographic optical microscope. Visible colours (more blue or more red) depend only on the direction of movement

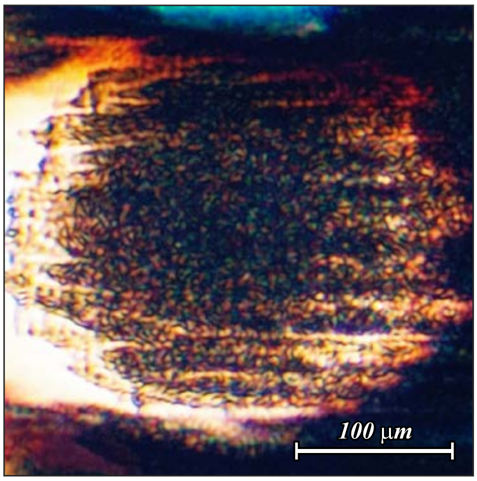

Fig. 2. Zone of a single pulse action on the sample surface

Accordingly to the many times experimentally confirmed interference model [29, 30]; the process of the formation of periodic structures can be schematically represented as follows: the process begins with the appearance of a periodically modulated interference light field in a space near the surface. The reason for its appearance is the interference of an incident light wave with a wave scattered by a certain surface roughness. The interference of an incident wave with resonant components of the diffracted field is most effective. A spatially inhomogeneous heating of the surface occurs in a periodically modulated intensity field. In this case, the temperature distribution along the surface obviously correlates with the distribution of the intensity of the interference light field. If the intensity of the laser radiation is sufficiently large, inhomogeneous heating of the surface can cause inhomogeneous melting, and then evaporation and removal of matter: the interference relief is "remembered". The above considerations can only be considered in general. For a more rigorous description it is necessary to consider the problem of an inhomogeneous imbedding of the electromagnetic field energy into the irradiated rough surface. The total electromagnetic field on the surface has the character of a periodic structure only if the scattered wave has a different tangential component of the wave vector than the incident wave. This is the situation when light is reflected from an even slightly rough surface: in the reflected light field there are not only mirrored components of the reflected wave, but also components that have experienced diffraction on various Fourier components of the roughness spectrum. Any real rough surface can be represented as a set of sinusoidal gratings with random orientations of the strokes, random periods and relief amplitudes. Then the scattering of the incident light wave on the surface roughness can be regarded as diffraction on various Fourier components of the roughness spectrum. Because of the addition of the fields of the incident and surface electromagnetic waves in the skin layer, the formation of interference maxima occurs, and consequently an inhomogeneous or periodic heating of the surface.

The action of a femtosecond laser in a multipulse mode leads to the appearance of tracks on the material, which were studied by scanning electron microscopy. For the examination of laser processed tracks, an analytical scanning electron microscope VEGA \\ SB, Tescan was used, whose accelerating voltage range is between 0.2 – 30 kV; the electron source is a tungsten cathode with thermionic emission. Fig. 3 shows an image of tracks on a sample surface formed as a result of scanning the surface with multipulses from a femtosecond laser.

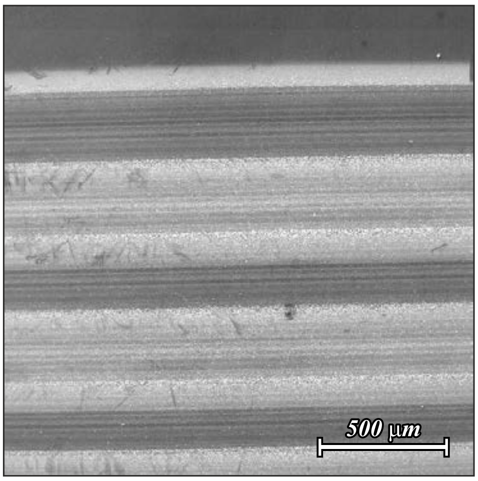

Fig. 3. Image of tracks on a copper sample formed as a result of multipulse scanning with a femtosecond laser

Nanostructures formed on the surface at an energy density below the one-pulse ablation threshold for the normal incidence of ultrashort laser pulses in the multipulse mode show a ridgelike, one-dimensional lattice structure. These ridges are orientated perpendicular to the polarization vector of the ultrashort laser pulse with an average periodicity of 0.68 μm, which is slightly shorter than the wavelength of the laser radiation. Fig. 4 shows

SEM images of such nanostructures created on the surface of copper corresponding to red and blue areas. In the scanning mode, all types of periodic surface structures coexist, which occurs as a result of superimposing different zones of a laser beam (different in energy density of the exposure regimes) onto one surface region, and the formation of various types of surface structures corresponding to them. Therefore, the evolution of the surface structure on a fixed region of the sample which is irradiated by multiple laser pulses during scanning consists of three stages: the front wing (related to the moving direction) of a laser beam with a relatively low energy density forms nanoroughness. When the maximum intensity of the laser beam approaches that spot LSFL replace nanoroughness and finally at the rear wing of the beam, the nanoroughness covers the remaining LSFL.

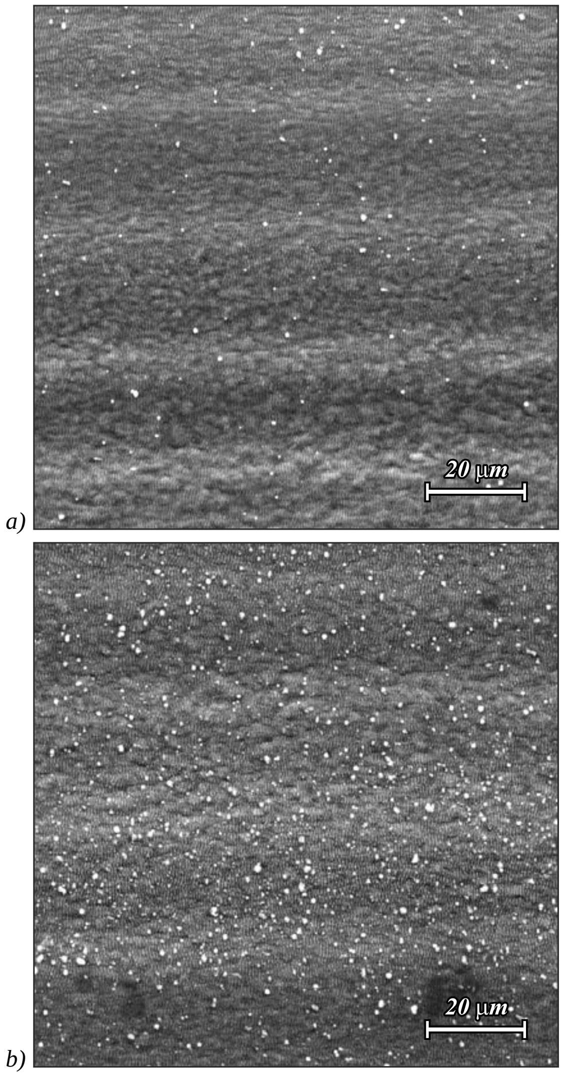

Fig. 4. SEM images of nanostructures recorded on the surface of copper corresponding to red (a) and blue areas (b). Field of view is 108.3 µm

For obtaining information on the structure of a surface, images in secondary and in backscattered electrons were investigated, presented in Fig. 5. It is known that the contrast in secondary electrons most strongly depends on the surface contour, and backscattered electrons contain, except an information on the morphology of a surface, additional information on the composition of the sample. Since the sample has a rather smooth surface, the output of the reflected electrons remains almost unchanged, independent of the position of the beam. The intensity of the reflected electrons is practically independent of the topography of the sample surface, and the resulting images characterize the chemical elemental composition. The brighter areas correspond to greater oxygen content, which indicates copper oxidation with the formation of copper oxide during processing. The elemental chemical analysis of the surface of microvolumes of the sample showed that the oxygen content in various microvolumes of the treatment zone varies within the limits of 1.5 – 12.5 by weight, and the copper content is within 98.5 – 87.5 % wt %.

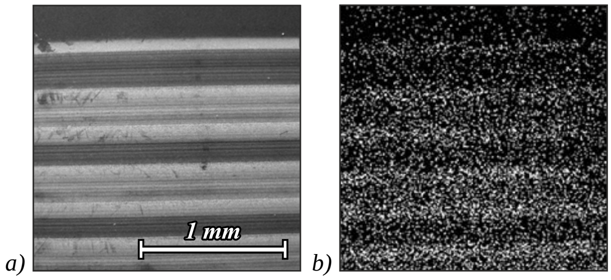

Fig. 5. Images in secondary (a) and in backscattered electrons (b): the brighter areas correspond to greater oxygen content and lower content of copper

It has been determined that on the laser treated surface the resulting nanostructure is not completely regular; various oxidation levels at different zones could be identified. The morphology of the surface in the regions of integral red and blue colours differs, which is due to the presence of oxide inclusions. On the surface of the integral red zone there are significantly more sub-micron oxide deposits in a form close to the globular (Fig. 6). That is, it has been experimentally confirmed that relatively small changes of the nanostructure obtained by scanning the copper surface with a beam of ultrashort pulses in a multipulse mode can lead to a significant colour change during diffraction staining of the surface.

Conclusions

Features of change in the nanostructure and colorizing of copper has been studied during scanning a femtosecond laser beam with a nearly Gaussian energy density distribution. It is shown that the use of a femtosecond laser with a pulse duration of 30 femtoseconds leads to the formation of near-wavelength periodic surface structures. By structuring the surface, it was possible to increase the brightness of colours achieved during colorizing the copper surface. It has been determined that nanostructures formed on the surface at an energy density below the one-pulse ablation threshold for the normal incidence of ultrashort laser pulses in the multipulse mode have the form of a one-dimensional lattice of ridges oriented perpendicular to the polarization vector of the ultrashort laser pulse with an average periodicity of 0.68 µm, which is slightly shorter than the wavelength of the laser radiation. This can be the basis for further improvement of the manufacture method of reflecting diffraction gratings using ultrashort laser pulses, which can take a worthy place among other methods of computer optics on the basis of interaction of laser radiation with a metallic surface [31 – 35].

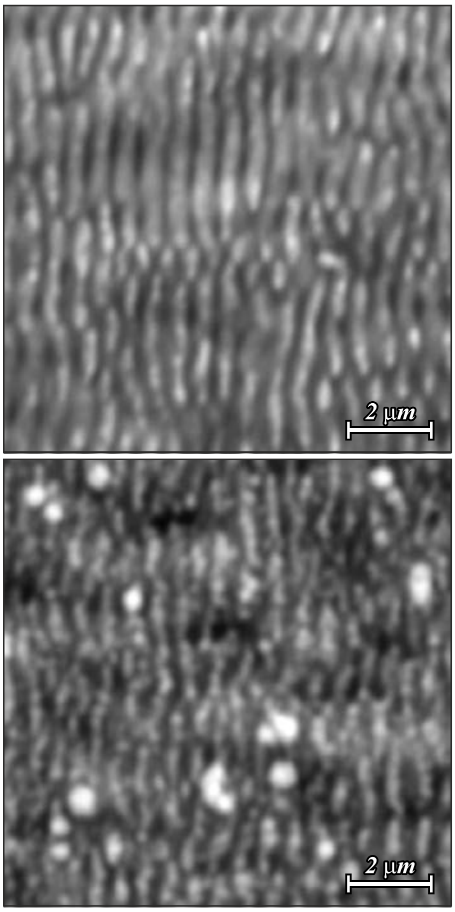

Fig. 6. SEM images of nanostructures produced on the surface of copper corresponding to red (a) and blue areas (b). Field of view is 10,83 μm

It was revealed that during forward and reverse scanning of the copper surface by a beam of ultrashort pulses with a weakly asymmetric spatial distribution in a multipulse mode and an energy density below the material ablation threshold it was possible to create a combined nanorelief consisting of low-spatial-frequency laser-induced periodic surface structures (LSFL) coated with nanoroughness. The obtained experimental results indicate that different overlapping regimes during the scanning the surface play an important role on the development of resulting structures. An overlapping of various regions of the laser beam during the scanning process leads to the formation of a combined topology - nanostructures and LSFL.

On the treated surface, the nanorelief is not completely regular; various oxidation levels of different zones take place. The morphology of the surface in the regions of integral red and blue colours differs, which is due to the presence of oxide inclusions. On the surface of the integral red zone there are significantly more sub-micron oxide deposits in a form close to the globular. Thus, it is revealed that relatively minor changes in the nanostructure obtained by scanning the surface of copper by a beam of ultrashort pulses in a multipulse mode can lead to a significant change in the resulting colour during surface colorizing.

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Authors' information

Gerhard Liedl (b. 1960) graduated (1991) from the Institute of Applied Physics at the Vienna University of Technology. He received his Doctor of Technology (2000) degree from the Vienna University of Technology on laser micromachining. Actually, he is professor at the Vienna University of Technology at the Institute for Production Engineering and Laser Technology, where he heads the laser application group. He is mainly focused on applications of laser material processing with emphasis on ultra short laser pulses. E-mail: *gerhard.liedl@tuwien.ac.at* .

Robert Pospichal (b. 1983) student of Technical University Vienna, research assistant at Institute of Production Engineering and Laser Technology. He is recently working on femtolaser-applications. E-mail: *robert.pospichal@tuwien.ac.at* .

Serguei Petrovich Murzin (b. 1963) graduated (1986) from S.P. Korolyov Kuibyshev Aviation Institute (KuAI, presently, Samara National Research University, short – Samara University) majoring in Aircraft Engines. He received his Doctor in Technics (2005) degree from Samara National Research University. He is holding the position of a professor in Samara University at the department of Automatic Systems of Power Plants. He is the head of the Research & Education Center of Laser Systems and Technologies. He is a specialist in laser technology and nanotechnology, laser physics and optics. He is a co-author of over 90 scientific papers, two monographs, and 18 inventions and patents: *murzin@ssau.ru* .

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