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Metal surface coloration by oxide periodic structures formed with nanosecond laser pulses

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ABSTRACT

In this work, we studied a method of laser-induced coloration of metals, where small-scale spatially periodic structures play a key role in the process of color formation. The formation of such structures on a surface of AISI 304 stainless steel was demonstrated for the 1.06 μm fiber laser with nanosecond duration of pulses and random (elliptical) polarization. The color of the surface depends on the period, height and orientation of periodic surface structures. Adjustment of the polarization of the laser radiation or change of laser incidence angle can be used to control the orientation of the structures. The formation of markings that change their color under the different viewing angles becomes possible. The potential application of the method is metal product protection against falsification.

1. Introduction

The modification of the surface properties of metals is a topical issue of studies in various fundamental and experimental scientific fields, among which are color laser marking [1–5], biomedical applications [6–10], enhancement of tribological performance [11,12] or simply decorative uses [13]. In particular, optical properties are important for authentification and traceability of a product. However, in some cases, the existing methods (special holographic labels, infrared and ultraviolet colorants, special barcodes deposited onto the surface via the means of monochromatic laser marking) do not provide reliable protection against falsification. Therefore, it is important to perfect the existing methods of protection as well as to create new ones.

Previously, we have demonstrated the coloration of stainless steel surface during the exposure to the fiber laser emission with nanosecond duration of pulses. It was noted that depending on the view angle, the color can change significantly [14]. A sample color dependence on the light incidence angle (viewing angle) was presented. Reflectance spectra were blue-shifted with an increase of incidence angle on the sample due to a difference in optical path length when passing through the oxide film covering the surface of the generated bead. Such an effect can be used not only for decoration or identification but also as a method of product protection against falsification.

In this paper, an alternative method of product protection against falsification, based on the formation of the laser-induced periodic surface structure (LIPSS, so-called “ripples”) on the metal surface, will be considered. LIPSS were found under the action of femtosecond laser pulses on the semiconductors [15–17], dielectrics [18] and metals [19] and are commonly attributed to interference between the incident laser radiation and scattered or excited surface waves [20,21]. The use of LIPSS as a method of protection against falsification was suggested in [22], where the possibility of LIPSS formation (with a period of 660 nm and a relief height of 200 nm) with ultrashort laser pulses of Ti: Sapphire laser system ($\lambda = 800$ nm) was shown. In femtosecond laser color marking [23] there are several approaches to control LIPSS orientation and consequently surface color, based on the rotation of polarization [1] or tilting sample surface [24].

Although the fact that nanosecond LIPSS are known since the 1960s [25] and obtained on wide range of materials [26,27] as far as authors know, the possibility of using specific periodic surface structure (PSS) formed by “long” nanosecond pulses of a fiber laser with random (elliptic) polarization of emission as a method of protection against falsification was not considered.

Using a fiber laser with its maintenance-free operation and high wall-plug efficiency, the proposed technology of LIPSS formation as a new way of laser marking of metal surfaces for various purposes - decorative, counterfeit protection, etc. - can be considered promising for commercial implementation. The scope of the present work is to show how the obtained nanostructures with laser pulses of nanosecond duration can be precisely controlled and used to obtain needed optical

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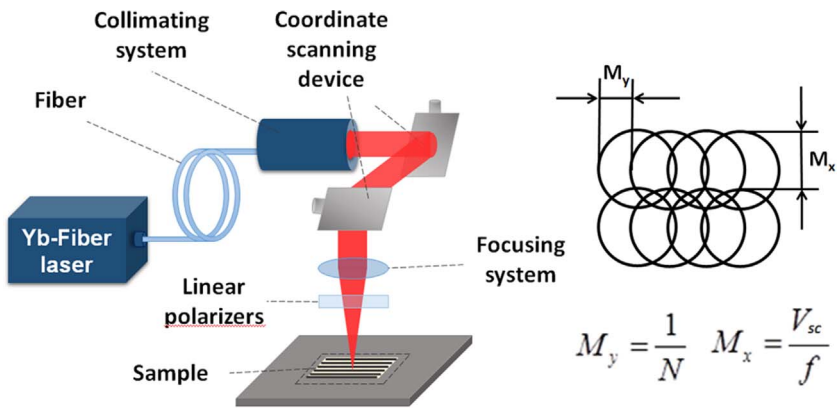

Fig. 1. The experimental setup used for laser treatment (left) and strategy of the laser beam scanning on the metal surface (right).

diffractive effects.

2. Materials and methods

Stainless steel as a common metal for industrial applications was used for experiments. AISI 304 stainless steel 50 mm × 50 mm plates (thickness 0.5 mm, arithmetical mean roughness R_a = 0.02 μm, maximum height R_z = 0.18 μm) was cleaned with ethanol before laser treatment.

The laser exposure was made in air using a commercially available machine on the base of ytterbium pulsed fiber laser with a wavelength of λ = 1.06 μm, which generates pulses of τ = 100 ns duration at repetition rates in the range of f = 20–99 kHz with random (elliptical) polarization in a single pulse (Fig. 1).

The laser spot with a focal diameter of d = 50 μm was moved over sample surface with velocity V_{sc} ranging from 1 to 250 mm/s. Laser intensity I was ranged from $1{\cdot}10^7$ to $3{\cdot}10^7$ W/cm^2. The metal was irradiated using a line-by-line scanning of the surface with a series of laser pulses where a scanning step along the Y-axis: $M_y = 1/N$; along the X-axis: $M_x = V_{sc}/f$, where N – number of lines per mm, V_{sc} – scanning speed, m/s.

Obtained samples were examined by optical microscopy (Zeiss Axio Imager A1M), atomic force microscopy (AFM, NT-MDT Nanoeducator), scanning electron microscopy (SEM, Zeiss Auriga Laser), Raman spectroscopy (Bruker SENTERRA and Horiba T64000). The solid-state laser operated at 532 nm wavelength and provided an output of about 1 mW was used for Raman-scattering spectroscopy. Repeated reproducibility of obtained results confirms that laser exposure at Raman

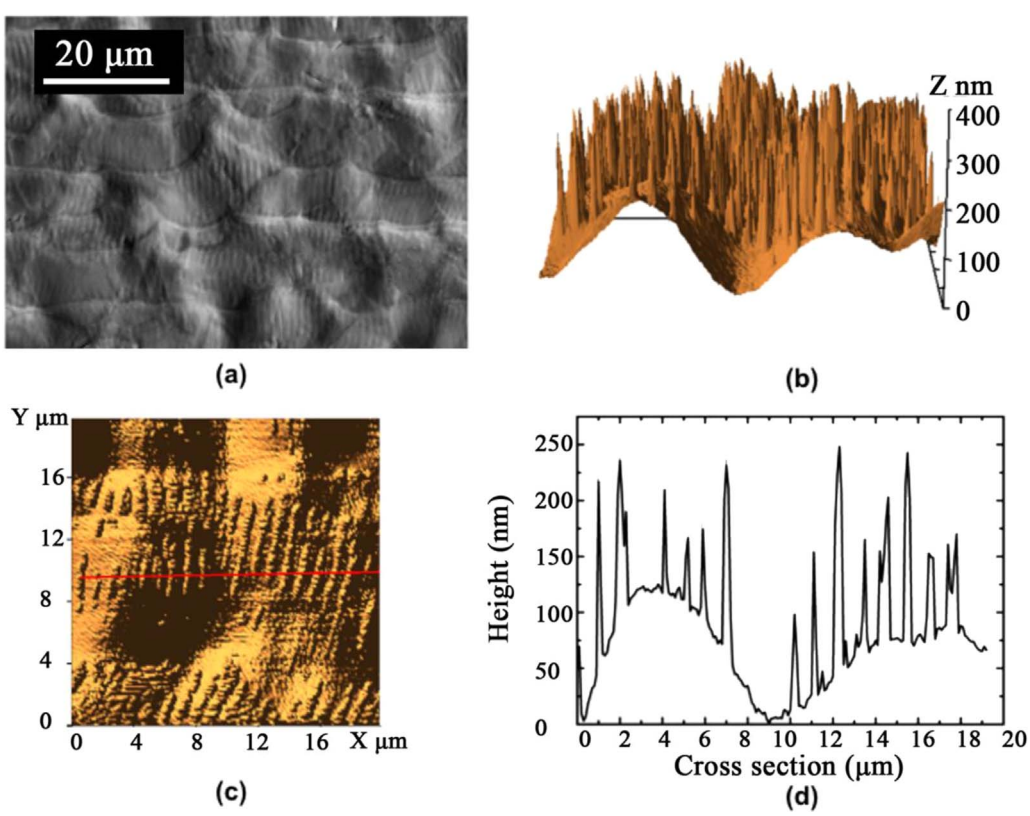

Fig. 2. AISI 304 stainless steel surface after laser exposure (I = $1.73{\cdot}10^7$ W/cm^2; M_x = 0.9 μm, M_y = 12.0 μm): (a) SEM image; (b, c) 3D and 2D AFM images; (d) cross section of the structure obtained from (c).

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