
**STRENGTH
AND PLASTICITY**

Peculiarities of the Behavior of Point Defects under the Optoplastic Effect in Copper

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Abstract—The authors previously discovered a new optoplastic effect and observed it under the action of a nanosecond UV laser pulse irradiation of subcritical intensity. In this paper it is shown that under this effect no micropores arise in the subsurface layer of metal. This proves the statement that swelling of metal under laser impact of moderate (subcritical) intensity occurs due to interstitial atoms migrating to the surface and not due to melting with formation of bubbles. At a abrupt cooling (for $\sim 20 \mu\text{s}$) interstitial atoms migrate to the surface by the Schottky mechanism due to abnormal mass transfer and the less mobile vacancies have no time to coagulate with formation of micropores in the time of the process.

Keywords: optoplastic effect, optical durability, polished surface, metal swelling, plastic deformation, point defects, Schottky mechanism, scanning electron microscopy

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INTRODUCTION

The efficiency of laser technologies considerably depends on the radiation wavelength [1]. The absorptivity of a larger share of materials increases as the wavelength decreases. For instance, in the IR range copper reflects up to 98% of the radiation, while in the UV range (with a wavelength of $\lambda = 355 \text{ nm}$) it reflects 10% [2]. Therefore, despite a considerably lower performance of UV lasers compared to widely used CO_2 lasers ($\lambda = 10.6 \mu\text{m}$), UV lasers are often applied for certain types of operations [3, 4].

Works [5, 6] presented the preliminary analysis of publications about detecting the traces of high-temperature plastic deformation in copper and its alloys after irradiation by a nanosecond pulse ultraviolet (UV) laser [2, 7–10]. These results are interesting in that they are obtained at an energy density from 0.1 to 1.0 J/cm^2 , which is below the threshold of metal melting, which is $\sim 1.0 \text{ J/cm}^2$ in this case. It was shown that the results have a significant similarity to the phenomenon of radiation swelling of metals [11, 12], as well as to the well-known acoustoplastic [13], electroplastic [14, 15], and magnetoplastic [16, 17] effects. An inverse effect, the photoplastic effect, these effects [18]: under illumination of a transparent semiconductor by light, nonequilibrium electric charges arise in it that lead to a reduction in the velocity of dislocation drift and crystal compaction. However, our results have noticeable differences, which allowed calling the detected effect optoplastic.

At the same time, it is well known that action by a femtosecond laser leads to swelling of a metal surface due to the appearance of micropores in the subsurface layer [19]. In this case the surface of the exposed metal melted and the vapor bubbles froze because of extremely rapid solidification of the melt. In [20], molecular dynamics simulation of the structural changes of the surface layer of metal under short-time high-energy external impact was performed and it was shown that under laser ablation of metals this process occurs with formation of pores.

In the current work we experimentally show that under action of the radiation of a nanosecond UV laser [5–9] on a metal surface (in the mode of the optoplastic effect; at an energy density of $0.1\text{--}1.0 \text{ J/cm}^2$), no micropores are observed (at least those larger than 30 nm).

EXPERIMENTAL

We studied specimens of oxygen-less copper [21] with the initial roughness of the laser spot region of 20 nm. They were polished using standard optical technologies [22]. The relief of the surface before and after the impact was studied using a Zygo NewView 7300 optical profilometer.

Further, we studied the surface layer of irradiated specimens using an Auriga Crossbeam system (Carl Zeiss, Germany) with an electron beam and a focused ion beam. The system is equipped with an ion column

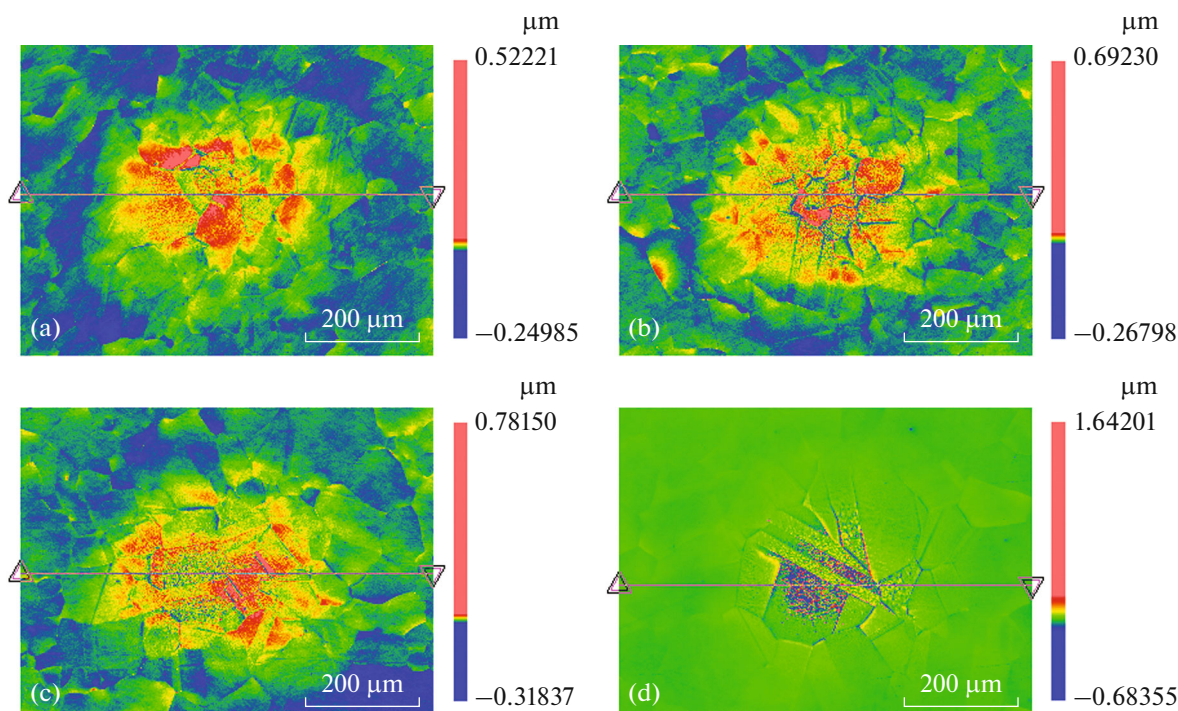


Fig. 1. 3D profile diagrams of copper specimens exposed to different numbers of laser pulses with an energy density of 0.82 J/cm^2 : (a) 1 pulse, (b) 3, (c) 5, and (d) 30 pulses.

based on liquid gallium. The Intralens detector and the Everhart–Thornley detector were used to obtain the images of the specimen surfaces in the mode of secondary electrons. The SmartSEM software package (CarlZeiss, Germany), which is intended to control the Auriga Crossbeam system, was used to obtain the electron microscopy images of the surface and control the ion beam in etching the specimen, as well as to record and export the data for subsequent processing and analysis in other software packages.

Using a pulse-periodic Nd:YAG laser Optolette HR 2731 (OPOTEC Inc., United States), radiating at the third harmonic ($\lambda = 355 \text{ nm}$, pulse energy up to 8 mJ , duration of 10 ns , and pulse repetition rate of 10 Hz), we irradiated the surface of specimens with a package of 30 pulses of the UV laser at an energy density of 0.82 J/cm^2 .

We focused the laser beam on the surface of the specimen in a spot $100\text{--}200 \text{ }\mu\text{m}$ in diameter with a fused silica lens with a focal distance of 250 mm . We registered the radiation energy using a Nova II instrument (Ophir Optronics Solutions Ltd., Israel) with a pyroelectric sensor. The electromechanical shutter controlled the number of pulses incident on the specimen. To average the results, we divided the readings of the Nova II by the number of pulses.

The laser impact was carried out at a temperature of approximately 20°C . In the laser mode we used, the condensed state of the irradiated metal was preserved

without noticeable melting and evaporation. The temperature of the specimen bulk material was almost constant, because the average power of the laser was below 1 W .

RESULTS

Figure 1 presents the 3D profile diagrams of Cu specimens exposed to a different number of laser pulses with an energy density of 0.82 J/cm^2 . The lines show the cross section in which we obtained the 2D profiles given in Fig. 2.

It is clearly seen that the damage of the subsurface layer increased as the number of acting pulses increased. The roughness of the initially polished surface in the zone of laser radiation action sharply increased. A significant number of peak formations occurred. The approximate height difference in the field of view of the profilometer was $0.75 \text{ }\mu\text{m}$ after one and three laser pulses, $1.1 \text{ }\mu\text{m}$ after five pulses, and $2.3 \text{ }\mu\text{m}$ after 30 pulses. These estimates were approximate because of the insufficient spatial resolution of sharp peaks by means of optical profilometer.

In Fig. 3 we show in detail the results of the study of the specimen exposed to 30 laser pulses with an energy density of 0.82 J/cm^2 . Figure 3a presents the micrograph of the surface in the action zone obtained using the optical profilometer. Twinned formations were well visualized in the copper specimen.

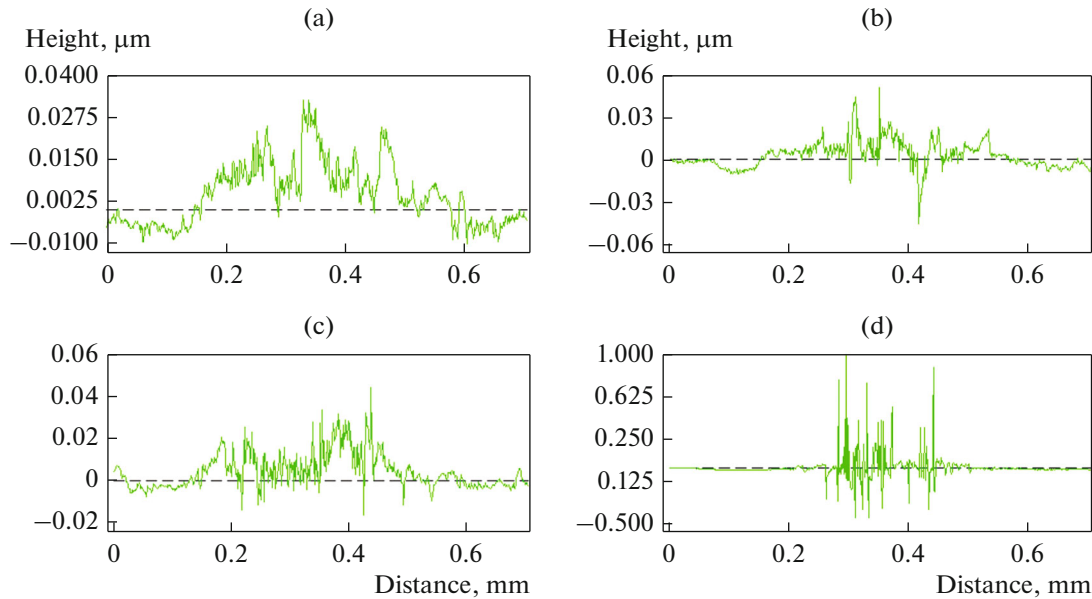


Fig. 2. The 2D profile of the laser spot on copper specimen in the region of cross section shown in Fig. 1; the number of applied pulses: (a) 1, (b) 3, (c) 5, and (d) 30.

In Fig. 3b we show a microscopic image of the surface section of the same copper specimen obtained by a scanning electron microscope. In the zone of action we etched the surface in the central part of the irradiated zone by a focused ion beam. For etching we used a current of the ion beam of 1 nA at an accelerating voltage of 30 kV. The surface was visualized by an electron beam with an accelerating voltage of 5 kV. Using the accelerating voltage in this range allowed us to obtain the maximal signal-to-noise ratio and decrease the damage of the specimen by the electron beam. The current of the electron probe and the objective aperture were adjusted according to the chosen magnification. The obtained images of the specimen surfaces have a resolution of not less than 1024×768 pixels. On the surface we clearly see the traces of the crystallographic sliding inside the grains. We vividly see the cut in the metal with a width of $\sim 10 \mu\text{m}$ created by the focused ion beam. On the surface of the cut in the subsurface volume of the laser-irradiated section of the copper specimen, we clearly see that there are no micropores in the metal (at least those within the resolution of the used electron microscope).

In Figs. 3c and 3d we show the 3D and 2D profile diagrams of the section of this spot that were obtained at an objective magnification of $200\times$. We clearly see the traces of the crystallographic sliding inside the copper grains. Their geometry indicates that they are in the planes of dominating motion of $\{111\}$ dislocations. It is easy to see that propagation of the sliding bands is decelerated by the grain boundaries. The typical distance between the sliding bands is $\sim 2 \mu\text{m}$. The height of the bands is approximately $0.01 \mu\text{m}$. However, in different grains the width of the bands and

their density were markedly different. Under the action of a series of laser pulses, the surface of the sharp edges of the formed sliding bands was slightly melted.

DISCUSSION

As shown in [2, 5–10], the optical breakdown of copper and its alloys with generation of a crater on the specimen surface was observed at an energy density above 1 J/cm^2 . We investigated the processes at the subcritical energy density ($E = 0.82 \text{ J/cm}^2$). The estimates performed in [2] show that the temperature of the subsurface layer of metal in this mode of action reached a value close to the melting temperature. The volume of the specimen remained at a temperature close to room temperature, and the entire specimen was in the condensed state. Thus, the described intense heat treatment by pulse laser radiation led to noticeable changes in the structure of subsurface layers of the specimen.

These results unambiguously testify to the fact that action by nanosecond pulses of the UV laser with a subcritical intensity on the polished surface of oxygenless copper leads to high-temperature plastic deformation in the exposed zone. We clearly observe the classical crystallographic sliding along the $\{111\}$ planes inside the grains and slip along their boundaries. On the surface of polished specimens, in the zone after the action, we observed neither grain boundaries nor blocks. These defects appeared only as a result of laser impact.

We cannot guarantee that the observed twins (Fig. 3a) unambiguously arose due to laser radiation.

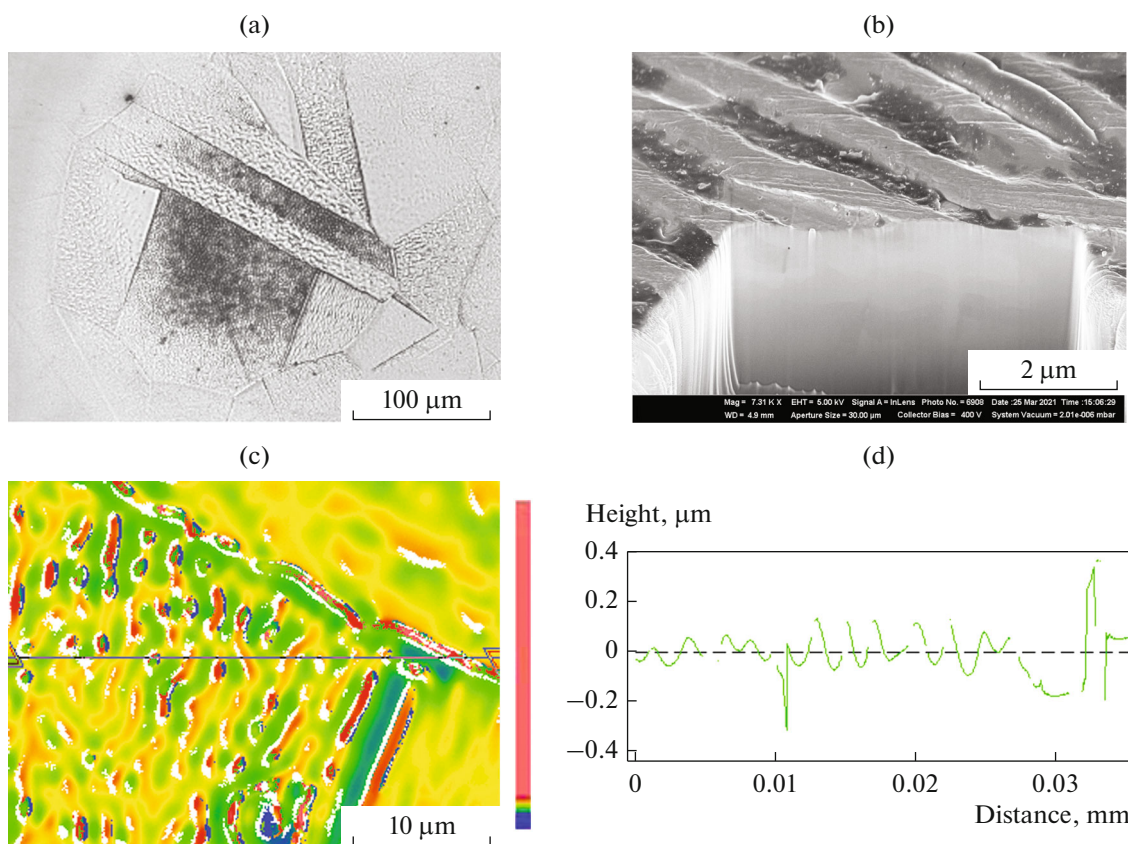


Fig. 3. The surface of oxygen-less copper specimen after 30 laser pulses. 3D and 2D profile diagrams of this specimen are given in Figs. 1d and 2d; (a) entire microscopic image of the laser spot; (b) SEM image in the region of cut; and (c) 3D and (d) 2D profile diagrams.

The time of cooling of the surface layer of copper after the pulse radiation with the used parameters was estimated in [2, 5–10]. It was shown that complete cooling occurs for 20 ns, which is considerably shorter than the interval between the pulses of 100 ms. Thus, each pulse of the series acted upon the specimen surface at the initial temperature of approximately 20°C.

It is well known that metals absorb light due to transfer of the photon energy to the electron component of the skin layer with a thickness of ~15 nm [23]. Such a high pulse loading considerably overheats the electron subsystem. This process occurs over times on the order of 2 ps. During this time the electrons transfer their excess energy to the phonon subsystem. Consequently, heating of the subsurface layer of metal occurs during the action of the laser pulse, 10 ns, and its complete cooling lasts ~20 ns. Thus, during approximately 30 ns the complete cycle of heating and cooling of the exposed section of the specimen surface occurs. During this time, under heating and rapid cooling with ~1000°C the size of the spot expanded and, respectively, decreased by ~0.5 μm.

Such a significant high-gradient thermomechanical action led to a sharp increase in the concentration of point defects arising upon knocking-out a certain

part of the ions of the metal to the interstitial site by the electron flow; these ions had no time to return because of the rapid cooling. Thus, the so-called Frenkel pair arises (vacancy + interstitial atom). In radiation physics there is a well known similar phenomenon called radiation swelling of metals (see, e.g., [11, 12]). We assume that local swelling of the metal surface after exposure by laser pulses can have a similar character, particularly as point defects, as is well known, have a great impact on the processes of copper structure formation [24–27]. However, noticeable differences exist between the radiation swelling and local swelling of the metal surface under the action of a laser.

In radiation physics, metals are usually exposed to long irradiation by flows of high-energy particles. Interstitial atoms, being the most mobile defects, are absorbed by dislocations, grain boundaries, etc., and come to the surface (the Schottky mechanism), while the vacancies coagulate by the diffusion interaction with formation of micropores. In our case micropores (at least those with a size larger than 30 nm), as seen in Fig. 3b, have no time to generate, because the duration of the process appeared to be insufficient for coagulation of vacancies that were mainly attached in the metal near the site of their origin. At the same time,

the duration of the process appeared to be sufficient for local swelling of the metal surface in the zone of impact. The volume of this zone, measured with a profilometer, in its order of magnitude corresponds to the number of interstitial atoms that occur as a result of high-temperature processes in the subsurface layer of the specimen. These interstitial atoms apparently diffused to the surface due to the mechanism of abnormal mass transfer under pulse actions on metals [28, 29]. At abnormal mass transfer the diffusivity can increase by 6–10 orders of magnitude and exceed the value of the diffusivity in the liquid phase. In Fig. 1 we clearly see that as the number of acting pulses increase the height of the swelled formation grows, that is, the effect of accumulation is observed.

Here, we revealed the difference between the local swelling of the surface we obtained and that in [19], in which the effect of metal surface swelling was obtained as a result of the use of high-power femtosecond radiation. In [19] swelling is explained by formation of micropores in the subsurface layer due to melting of the subsurface layer of metal and freezing of the vapor bubbles upon extremely rapid solidification of the melt.

We can note that the optoplastic effect in copper is similar in many aspects to the effects observed in high-speed deformation [30, 31].

CONCLUSIONS

The above results, as well as the data of [2, 5–10], confirm that as we act on copper with a nanosecond pulse UV laser radiation of subcritical intensity we reveal a new optoplastic effect. This consists in swelling of the metal surface as a result of laser impact, similar to radiation swelling, and in subsequent high-temperature plastic deformation by sliding along the grain boundaries and crystallographic slip in them. The optoplastic effect is an inherent part of the groups of processes of plastic deformation in metal under the actions of various pulse fields such as the acoustoplastic [13], electroplastic [14, 15], and magnetoplastic [16, 17] effects.

One of the main causes of the optoplastic effect is the process of abrupt growth in the concentration of point defects in the heated subsurface layer. Excessive concentration of interstitial atoms relaxes upon their arrival at the surface by the Schottky mechanism due to the phenomenon of abnormal mass transfer, while the less mobile vacancies have no time during the process to coagulate with the formation of noticeable micropores (larger than 30 nm in size).

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CONFLICT OF INTEREST

The authors declare that they have no conflicts of interest.

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