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Features of the surface profile during laser exposure to copper in an oxygen-containing environment

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Factors of formation of the copper surface profile under pulsed laser radiation in the argon and nitrogen atmospheres and oxygen-containing atmosphere have been considered. In the latter case, the effect of the surface curving direction reversal has been demonstrated for the same sign of the temperature coefficient of the material surface tension. The observed convex profile is associated with partial thermal destruction of the oxide film on the melt surface in the region of maximum heating. The convex surface profile may be a reason for instability of laser technological operations involving impact on copper in an oxygen-containing environment.

Keywords: melt, copper, copper oxide, laser technology.

The process of fusion of copper and other highly reflective metallic materials with focused laser radiation (LR) continues to attract research interest because it is employed in a number of practical applications [1,2]. High characteristic values of reflectivity and thermal conductivity of copper complicate its thermal treatment with laser radiation. To reach the fusion threshold, laser radiation of high intensity ($\geq 1\,\mathrm{MW/cm^2}$) is necessary. At the same time, the process instability and significant variability in the extent of LR impact in an oxygen-containing gaseous environment remain the issues frequently mentioned in literature [3,4]. For instance, the variable delay in the onset of surface fusion by infrared LR in the course of laser spot welding of copper causes variations in the effective pulse-to-pulse duration of welding itself, which, as shown in [4], leads to defect formation.

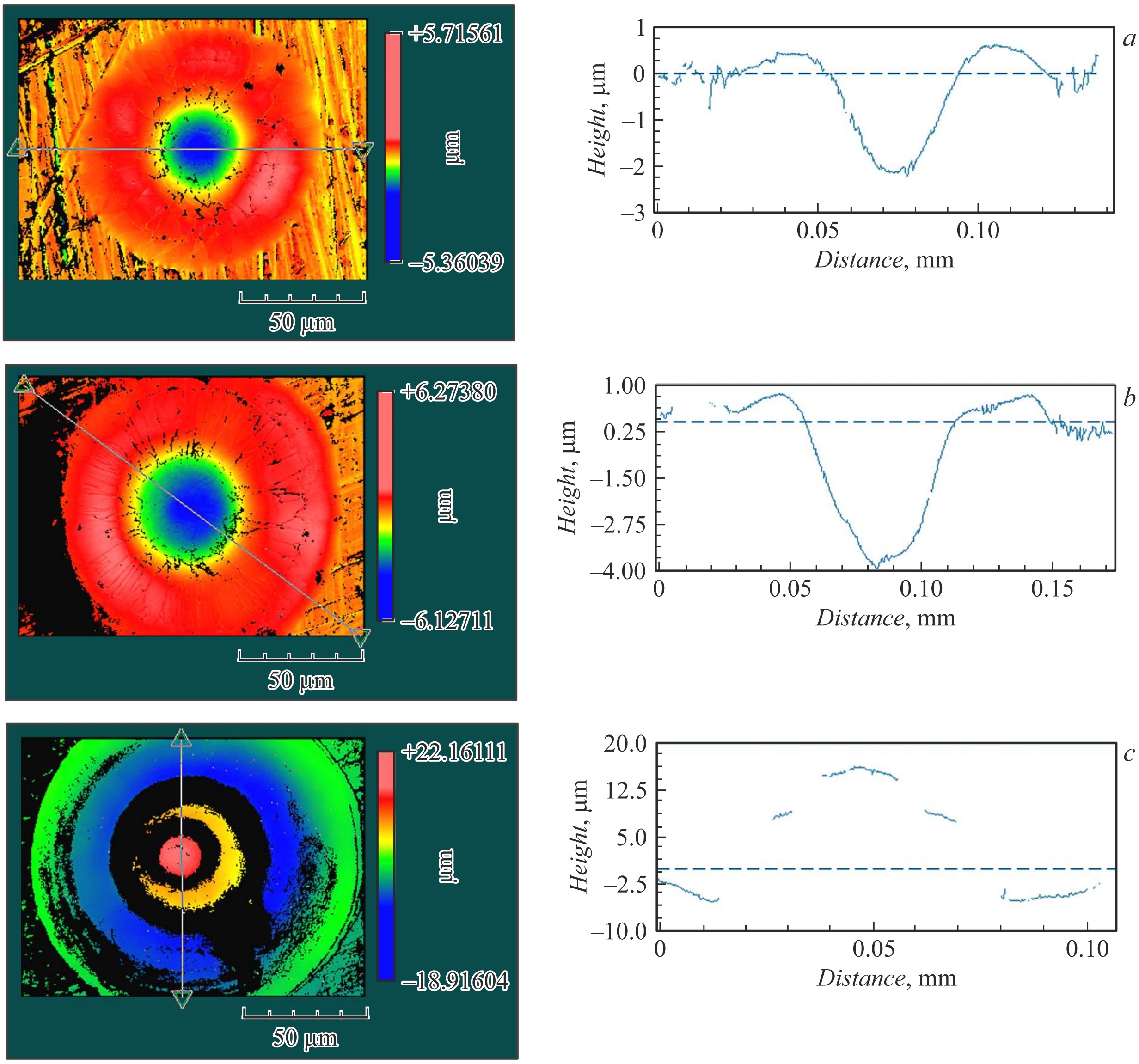
One of the approaches allowing reduction in thermal losses and increase in efficiency of the laser energy utilization in copper processing is power modulation [5]. In particular, the use of nanosecond pulses allows stable laser processing of copper by high-intensity laser radiation with relatively low energy consumption, which is of great importance in nano— and micro-technologies [6]. In [7] it was shown that, when direct current passes through a copper sample, there is observed a decrease to 0.74% in the oxygen content at the bottom of the channel formed in air by a nanosecond LR pulse. The copper heating efficiency increases significantly if the copper surface is blackened or oxidized and also if lasers irradiating in the blue-green or ultraviolet wavelength range are used.

However, the issue of choosing the auxiliary gas remains open. For instance, paper [8] has demonstrated the possibility of increasing relative density of the products and ensuring the absence of cracks in the framework of the additive selective laser fusion technology by deliberately oxidizing the copper powder surface. Authors of [9] have suggested that such results challenge the conventional idea of undesirability of oxidation effects in additive manufacturing. In this work, the surface profiles of copper samples were studied after exposure to laser pulse radiation in order to determine the influence of the auxiliary gas type on the melt pool formation.

In the experiment, we used fiber laser RFL-QCW150 (Raycus) with the wavelength of 1070 nm, peak power of 1500 W, and uniform intensity distribution in the spot. The radiation was focused on the wafer surface with the aid of optical system BW210 (Raytools). Taking into account the measured size of the laser spot ($190\,\mu$m), we estimated the surface radiation intensity at the 100 % power as approximately $5.4\,\mathrm{MW/cm^2}$. As the target, copper wafers M1 (analog of Cu-ETP) $20 \times 36 \times 3$ mm in size were used; the oxygen content was below 0.03%. The area exposed to radiation was blown with gas at the flowrate of 20 l/min. For blowing, argon (with oxygen content below 7 ppm), nitrogen (with oxygen content below 5 ppm) or atmospheric air was used. The resulting surface shape was studied by using interferometric profilometer Zygo NewView 7300 (Zygo Corp.), and also in polarized light by using microscope MMP-2 (BIOMED).

In the experiment, a fixed copper target was exposed to LR in the form of rectangular pulses 0.3 or 0.5 ms long. The radiation power variation range was 1000−1300 W for the 0.3 ms pulses and 1000−1500 W for the 0.5 ms pulses. The power step was 15 W. To assess the results reproducibility, the experiments were repeated many times with a constant set of parameters.

With the pulse length of 0.5 ms, stable formation of the fusion region took place at the power above 1185 W. Below this value, significant instability of the exposure results was observed regardless of the gas type. For example, surface fusion was not observed at the power of 1110 W in 11 cases of 36. On the other hand, when power exceeded 1230 W in air or 1275 W in argon or nitrogen, melt ejection was observed. In the intermediate power range, when the



Surface profile in the region exposed to RL. Left panels: 3D profiles with cross-section lines. Right panels: profile cross-sections. Modes of exposure: *a* — argon blowing, power of 1185 W; *b* — nitrogen blowing, power of 1230 W; *c* — air blowing, power of 1185 W. Gaps in the data are due to limitations in optical measurements on highly reflective surfaces at large angles.

material fusion was stable and free of signs of melt ejection, the resulting surface profile exhibited a dependence on the gas type: in blowing with argon or nitrogen, in the central part of the exposed area a hollow was formed, while in air a bulge emerged (see the figure). Under LR, an axisymmetric profile was formed. In blowing with nitrogen or argon, the observed height difference was $3-4\,\mu$m. In blowing with air, the height difference varied in different prints from 15 to $23\,\mu$m at the same irradiation parameters. When shorter pulses (0.3 ms long) were used, no melt ejection was observed at powers up to 1300 W. At the same time, no surface profile dependence on the gas type was observed: a concave profile was formed, among others, in air.

Polarization microscopy [10] confirmed that in air the Cu_2O oxide film gets formed on the sample surfaces. The oxide film formation in argon and nitrogen was not observed.

When the intensity distribution is Gaussian or uniform, the maximum temperatures are assumed to be in the central part of the pool [11]. The copper melt surface tension is known to decrease with increasing temperature. In such a situation, there typically arises a concave surface profile; this is associated with the melt displacement to the periphery under the action of thermocapillary forces [12], which is observed experimentally in argon or nitrogen blowing. Papers [13,14] present the results of studying the effect of oxygen absorption on the liquid copper surface tension, which demonstrate a decrease in surface tension with increasing oxygen partial pressure. Paper [14] shows that, when the oxygen partial pressure exceeds 1–3 Pa, surface tension of the Cu_2O oxide film decreases with increasing temperature; in this case, the concave profile would be also observed during the melt oxidation in air.

The observed convex surface profile in oxygen-containing environment may be, probably, explained by assuming the occurrence of thermal decomposition of the Cu_2O oxide film. At the temperatures above 2100 K, the Cu_2O compound reduces to copper with releasing oxygen [15]. Thus, in the central region, that is, the region of highest heating, the oxide film is thermally destructed with the copper melt exposure. Since the copper melt surface tension is twice as high as that of Cu_2O [13], after the film destruction a region of surface tension higher than that on the periphery is formed in the pool central part. Such a distribution produces the observed local rise in the melt level by the end of the 0.5 ms pulse. After exposure to the 0.3 ms pulse, no rise in the melt level was observed. Apparently, the process of the convex surface formation needs a longer melt lifetime.

Now let us estimate the possibility of surface deformation due to propagation of capillary waves. Frequency f of capillary waves with wavelength λ on the melt pool surface depends on the liquid depth $h < \lambda$: $f = 2\pi\sqrt{h\sigma/\rho}/\lambda^2$, where ρ is the copper melt density and σ is its surface tension. The propagation time of a wave with length d at $h = 0.01$ mm and $\sigma = 1.3$ N/m may be estimated as $1/f \sim 0.16$ ms which is less than the used pulse lengths. When h increases and λ decreases, the propagation times become shorter. Thus, the surface may get curved during the laser pulse because of the capillary waves propagation over it. However, in this case the resulting surface shape would be determined by the melt pool depth and, because of its variability, would exhibit a scatter regardless of the gas type. At the same time, statistics based on the obtained prints for argon, nitrogen and air reliably correlates the type of the surface profile with the type of gas.

The paper demonstrates the effect of the convex surface formation in the center of the region exposed to LR in air, which is presumably caused by thermal destruction of the melt-surface oxide film in the region of maximum heating. This effect may be a source of the mentioned in literature instability of results of laser technological operations implying the copper exposure to oxygen-containing environment.

Conflict of interests

The authors declare that they have no conflict of interests.

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