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Structure changes in metals after their laser treating in different conditions

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Abstract. A change in the crystal structure of a number of metals in the zone of exposure to laser radiation with a flux density of 10^4–10^5 W/cm^2 was detected by X-ray diffractometry. It is shown under the action of laser radiation on the surface of metal samples, their crystal structure changes in the irradiated zone from cubic face-centered to distorted.
Keywords: laser radiation, x-ray diffractometry, changes in the crystal structure, correlation function.

1. Introduction

Today, laser processing technology is receiving increased attention, since it is an effective, and most importantly, productive method. The scope of its application is very wide: surface hardening of metals, microprocessing of materials, modification of properties and surfaces, laser cutting and welding, etc. In this regard, there is a need to study the processes occurring during laser processing of a solid. The aim of this work is to study the changes and relaxation of the structure of a number of metals exposed to laser radiation (LR) with a flux density of 10^4–10^5 W/cm^2, as well as explanation of the detected structural changes.

2. Experimental equipment and experimental results

The scheme of the experimental setup used in the study is presented in Fig.1. The number *1* indicates the source of laser radiation (LR) – the GOR-100 M ruby laser (with a wavelength of λ = 0.694 μm) operating in the free-running mode (pulse duration τ ~ 1.2 ms). The number *2* denotes the focusing system, passing through which the LR was sent to the sample *3*. Both single-lens and two-lens systems were used as the focusing ones, which made it possible to construct the image of the aperture *4* on the surface of the sample *3* as a spot with sharp edges (the diameter was varied in during experiments from 1 to 2 mm), which, in turn, ensured a change in the radiation flux density *q* from 10^4 to 10^6 W/cm^2.

Part (~ 4%) of the laser radiation was directed by the front face of the glass wedge *5* to the IMO-2N *6* energy meter, the entrance pupil of which was located in the focal plane of the lens *7*. The energy of the E_0 laser pulses varied from 5 to 60 J. The radiation reflected from the back of the wedge sent to the FEK-14 *8* coaxial photo-detector, which signal was fed to the input of the S8-13 oscilloscope, and was used to record the temporal shape of the laser pulse.

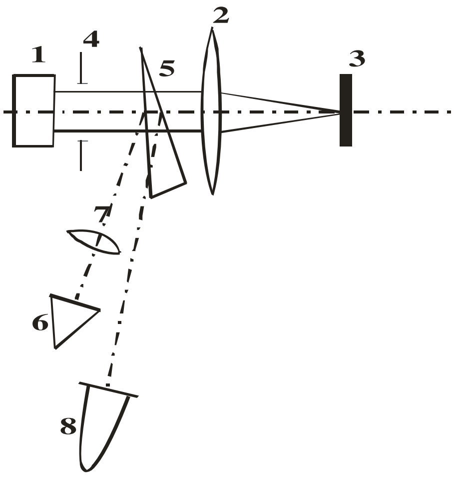


Fig.1. Scheme of the experimental setup.

TheDRON-2.0 X-ray diffractometer was used to determine the structural changes in metal samples (solid polycrystalline), in equilibrium with a cubic face-centered crystal lattice, before and after exposure to laser radiation. The Kα line from a tube with a copper anticathode filtered by a nickel standard filter at a wavelength of 154.050 pm was used in the diffractometer.

Calculation of temperature changes after action on the target of each pick of free oscillation pulse was also fulfilled.

Consider the effect of LR on copper (Cu). Since copper belongs to cubic syngony and its cell has no phases other than that described by the Fm3m group, we can say that the first two mechanisms of changes in this case are not able to lead to changes that could be detected by X-ray diffractometry. In addition, in our case, the laser radiation energy is not enough to ionize the core, and, therefore, neither the internuclear distances nor the Fermi energy change. Therefore, it is necessary to find out whether irradiation affects the imperfection of the structure of the copper crystal.

Table 1 shows the theoretical and experimental values of interatomic distances (a), coordination number (K) and half widths of maxima (Δ) on the experimental curves of correlation functions for a copper sample.

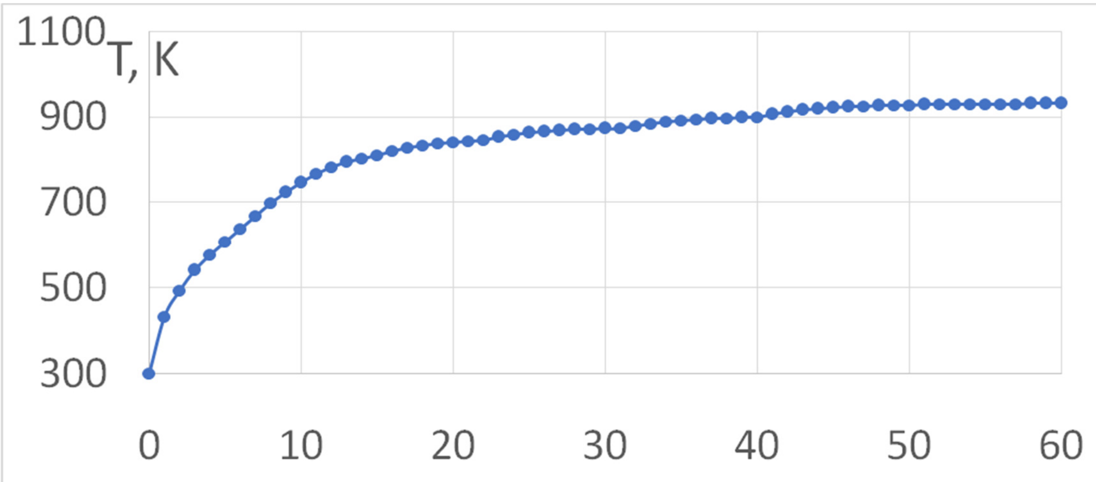


Fig. 2. Calculated temporal dependence of temperature of titanium surface irradiated by laser pulse with a flux density of 10^5 W/cm^2.

The experimental values from the table 1 show that laser processing leads to a change in the intensity of reflexes, their position, and profiles. Moreover, these changes significantly exceed the experimental errors. In addition, it was recorded that, during local short-term heating and rapid cooling, mechanical stress arises in the sample, which leads to a change in the crystal structure of the metal in the irradiated region from cubic face-centered to tetragonal, which explains a significant (up to 30%) increase in the microhardness of the metal surface.

Table 1. Parameters of the correlation functions of a copper sample before and after laser irradiation with $q \sim 5{\cdot}10^5$W/cm^2 [1]

№	Theoretical values		Unirradiated sample			Irradiated sample		
	***a*, Å**	***K***	***a*, Å**	**Δ, Å**	***K***	***a*, Å**	**Δ, Å**	***K***
1	2.550	12	2.58	0.7	13	2.48 2.60	1.2	6 6
2	4.416	24	4.50	0.5	26	4.50	1.0	24
3	5.100	12	–	–	–	–	–	–
4	5.702	24	5.75	0.6	36	5.82	0.3	36
5	6.246	8	6.40	0.3	5	–	–	–
6	6.746	48	6.80	0.3	45	6.50	0.8	52

For irradiated copper samples at $q \sim 5 \cdot 10^5$ W/cm^2, not only smearing, but also splitting of the first distribution maximum is observed. This indicates that, after laser treatment, the crystalline structure really transforms, as mentioned above. It is also worth noting that at LR flux densities sufficient for melting the metal in the treatment zone ($q > 10^6$ W/cm^2), a change in the diffraction patterns was not observed, and the microhardness decreased slightly.

3. Discussion

When the height of the first maximum of correlation functions before and after irradiation were estimate, it was found that 83% of the cells are subject to transformation. These data are in good agreement with the calculated ones.

The proportion of cells that changed their state was estimated using the following procedure.

At the first stage, it is necessary to find out how many cells are in the irradiation zone. For this, it is necessary to know the sample volume and the volume of one cell. Since the copper crystal be longs to the space group Fm3m and has a cubic face-centered (FCC) unit cell, is described by one parameter $a = b = c$ ($\alpha = \beta = \gamma = 90°$), then the cell volume before processing can be considered the equal

$$V_{cell} = a^3 = (3.597 \cdot 10^{-10} m)^3 = 4.65 \cdot 10^{-29} m^3 .$$

To calculate the sample volume, the following facts must be taken into account. First, laser radiation cannot penetrate deeper into the sample to a depth exceeding the wavelength. Secondly, along the width, the LR cannot go beyond the boundaries of the focusing spot.

Therefore, as a test sample, we will consider a cylinder with a height equal to the wavelength of a ruby laser ($h = \lambda = 0.694$ μm) and a diameter equal to the diameter of the focusing spot ($d = 2$ mm). In this case, the sample volume can be found as follows:

$$V_{Cu} = \frac{\pi d^2}{4}\lambda = 2.17 \cdot 10^{-12}\ \mathrm{m}^3.$$

Thus, we can determine the number of cells per volume under study:

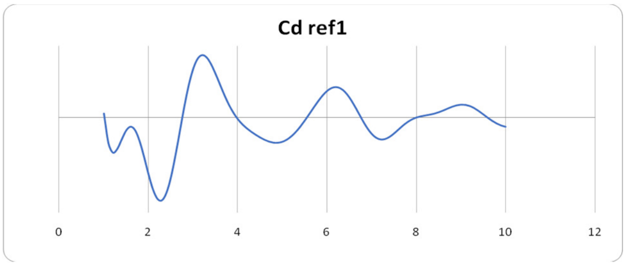
$$N_{cell} = \frac{V_{Cu}}{V_{cell}} = 4.65 \cdot 10^{16}\ \text{cells}.$$

At the second stage, it is necessary to determine how many cells could be transformed into parallelepipeds if all the energy of the acting beam was spent on the destruction of the cell.

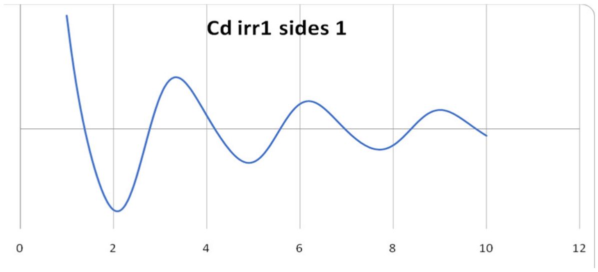
First, we determine the energy of the crystal lattice in the ground state. The quantum calculation of the binding energy of metal particles is one of the most difficult problems in solid state theory. This problem can be attributed to the problem of many bodies. At present, a rigorous solution to this problem is unknown in either classical or quantum mechanics, even in the case of three bodies. However, there are approximate calculation methods (the Haber method, the Winger and Seitz method, etc.), and the energy calculations are in good agreement with each other [2–6]. We use the ionic model described in [2, 3].

Given that a metallic type of chemical bond is realized in copper crystals, we can say that a copper crystal consists of a lattice of positive ions that are immersed in an electron liquid (gas). In this case, the adhesion energy can be calculated with methods used in calculating the adhesion of ionic crystals.

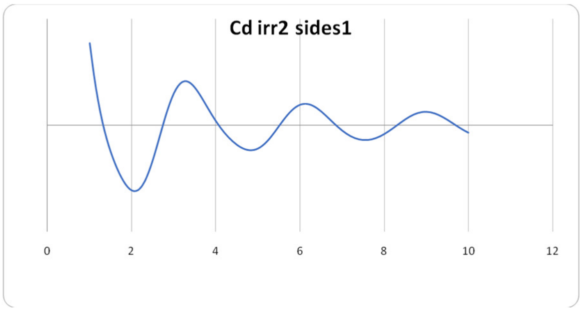
Let's now consider the effects of laser radiation on cadmium samples. Fig.3 represents correlation functions $P(u)$ for cadmium samples a – before irradiation, b – after laser irradiation of one side of a sample, c – after laser irradiation of one both sides of a sample.



a
Degree of crystalline is 63.49%



b
Degree of crystalline is 84.35%



c
Degree of crystalline is 86.84%

Fig.3. Correlation functions $P(u)$ for cadmium samples: a – before irradiation, b – after laser irradiation of one side of a sample, c – after laser irradiation of one both sides of a sample.

As is known, in equilibrium, the attractive forces must be balanced by the repulsive forces. Since repulsive forces in metals are related to the movement of electrons creating pressure inside the metal, and pressure is related to their kinetic energy, it can be argued that attraction balances the kinetic energy of electrons. As you know, the energy of the crystal lattice characterizes the amount of energy that must be spent to break the crystal lattice into its components and to remove them from each other at an infinitely large distance, that is, to overcome the forces of attraction. Thus, we get:

$$E_{cell} = \frac{3}{5} E_F = \frac{3}{5} \frac{\hbar^2}{2m_e} \left(\frac{3\pi^2 \rho N_A}{\mu} \right)^{\frac{2}{3}} = 4.14 \text{ eV},$$

where E_F is the Fermi energy, h – is the Planck constant, m_e is the electron mass, ρ is the copper density, N_A is the Avagadro constant, and μ is the molar mass of copper.

Define the number of cells capable of change the crystal structure in the irradiated region from cubic face-centered to tetragonal.

$$N_{trans} = \frac{E_{abs}}{E_{cell}} = \frac{3.12 \cdot 10^{19}}{4.14} = 7.53 \cdot 10^{18} \text{ cells},$$

where E_{abs} is the absorption energy (5 J).

However, it should be noted that not all laser beam energy is absorbed. A part is reflected, refracted, and scattered, and only the remaining energy (~ 0.5%) penetrates the sample, is absorbed, and passes into heat. So, the real number of cells capable of about $3.77 \cdot 10^{16}$ cells will change the crystal structure, which is 81% of the total number of cells.

Number of cells changed the crystal structure after laser irradiation of one side of a sample is 64%, after laser irradiation of one both sides of a sample – 67%.

Table 2. Parameters of the correlation functions of a cadmium sample before and after laser irradiation with $q \sim 10^5 W/cm^2$

		Ev	**min**	**max**	**Δ**	**σ**
Cd ref	a	2.967	2.951	2.974	0.024	0.007381
	c	5.586	5.472	5.627	0.155	0.044975
Cd irr 1 sides	a	2.959	2.946	2.968	0.021	0.009531
	c	5.578	5.526	5.625	0.099	0.03164
Cd irr2 sides	a	2.920	2.803	3.078	0.275	0.101995615
	c	5.459	5.154	5.638	0.484	0.197518

Let's now consider the effects of laser radiation on titanium samples. Typically, titanium can crystallize in two crystalline structures: α – Ti and β – Ti. When it crystallizes at low temperatures (room temperature), an α – Ti hexagonal close packed (HCP) structure is formed. While if it crystallizes at high temperatures, a body-centered cubic (BCC) β – Ti structure is formed. Complete transformation of the crystal structure from one to another occurs at a temperature of 882±2 °C.

Consider the effect of laser radiation on an α – Ti sample. Fig.4 shows the X-ray diffraction patterns of the samples before and after its processing, from which it can be seen that after laser processing there is a change in the intensity of the reflexes, their position and profiles. For example, reflex 103 practically disappeared.

Each intensity maximum in the X-ray diffraction pattern is an n-order reflection from a series of planes (hkl) with an interplanar distance dhkl corresponding to the slip angles. According to the Hell chart, for determining the X-ray patterns of crystals with a hexagonal compact structure, we determine hkl.

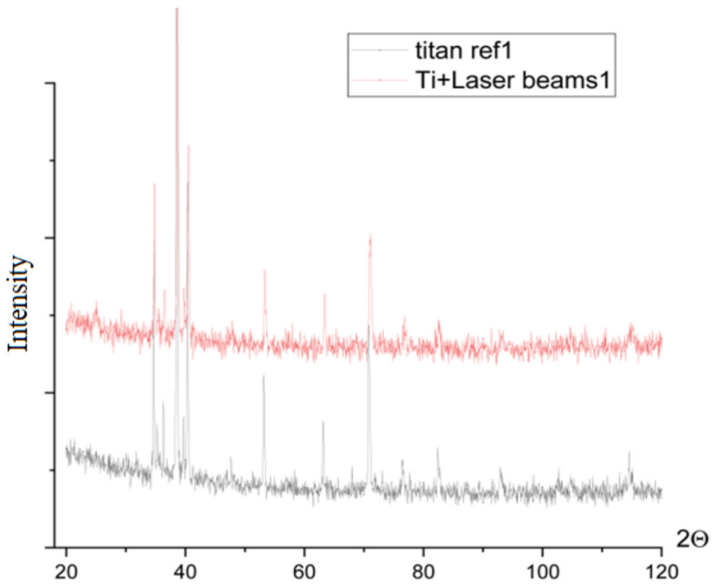


Fig.4. X-ray patterns of α – Ti before (lower) and after (upper) laser treatment. Degree of crystalline is in reference sample 36.13%, after laser irradiation of one side of a sample 38.91%, after laser irradiation of one both sides of a sample 47.63%.

To clarify the indication of reflexes, it is necessary to compare the experimental (hkl) exp and theoretical (hkl) theoretical indices of the plane.

The next step is to determine the experimental parameters of the cell.

From Table 3 it follows that the distribution of the parameters of the unit cells before and after laser irradiation is quite different from each other.

For the titanium samples treated by laser radiation with the flux density of radiation $q \sim 5\cdot10^4$ W/cm^{-2} splitting of correlation function maxima was not observed. However, the width of X-ray difractograms was essentially changed. This effect testifies the considerable growth of crystalline grid defects concentration in the irradiated zone.

Micro-hardness of titanium samples in the irradiated zones also considerably increased. The calculations show that after treating of titanium sample by laser radiation with the flux density $q \sim 5\cdot10^5$ W/cm^{-2} number of crystalline elementary cells were subjected to the transformation from the cubic side-centered to the distorted (having a form of parallelepiped, different from the cub) can reach 50%.

Table 3. Comparison of X-ray diffraction patterns of α – Ti before (1) and after (2) irradiation: average, smallest (min) and largest (max), theoretical values of the cell parameters which determined by all reflexes (in Å), σ_{11} - standard deviation of the parameters (in Å)

		The average	min	max	σ_{11}	Theory
a	1	2.708	2.546	2.992	0.18	2.937
	2	2.518	2.364	2.577	0.09	
c	1	4.747	4.660	4.956	0.12	4.582
	2	4.660	4.421	4.923	0.21	

4. Conclusions

The investigations showed that under the action of laser radiation on the surface of metal samples, their crystal structure changes in the irradiated zone from cubic face-centered to distorted (having a parallelepiped shape different from the cube).

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