MECHANISMS OF MICRO-VOIDS FORMATION CAUSED BY OPTICAL BREAKDOWN IN KCl SINGLE CRYSTALS UNDER LASER EMISSION

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The phenomenon of optical breakdown has been studied experimentally for KCl single crystals exposed to laser emission focused on the neodymium glass with modulated quality-factor, pulse duration $5 \cdot 10^{-8}$ s, wavelength $\lambda = 1054$ nm, and pulse energy of the order 1 J in the regime of local intrinsic absorption of the laser emission by the single crystal. Evaluations of local heat flash energetic constituents and characteristic durations for both local area heating and relaxation processes and following comparison with experimental results have shown that the relaxation process takes place in two stages: the first is fast phase followed by crowdion mass transfer with shock wave participation, and the second is slow phase with participation also dislocation mass transfer.

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INTRODUCTION

Under radiation influence by elementary particles or electromagnetic quanta onto solids, there appear structure defects. As a rule the primary defects are Frenkel pairs: vacancy–interstitial atom. The interstitial atoms may be positioned simply in interstitial spaces or can form dumbbell or crowdion configurations which can form more complex defect configurations during interactions. Various radiation damages of the crystalline lattice strongly affect strength and plastic properties of materials, so, are the subject of permanent attention [1-4].

Optical range laser emission transmitting transparent bodies under definite conditions – high density of the emission flux, existing absorbing inhomogeneities in the material structure – is locally absorbed that results in substantial local heating, plasma formation under high pressure, and local breakdown of the crystal with formation of micro-voids (optical breakdown) [5]; this degrades exploitation properties of functional materials [6]. The problem is complex. For its solution it is necessary: 1) revealing the mechanisms of the local absorption and heating of the crystal; 2) studying the mechanisms and kinetics of the substance transfer from the heated area resulting in void formation.

From very general considerations, the mechanisms of laser emission local absorption for high-pure materials may be as following: impact ionization of lattice atoms by electrons originating from photoionization of impurity atoms by laser emission; multi-photon ionization of the lattice atoms [5]; "metallization" of a dielectric with narrow enough band-gap under laser high density emission [7]; or generation of primary electrons due to cascade Auger-transitions in the valence band of alkali-haloid crystals [8]. At high enough laser emission density achieved, specifically, by the emission focusing, the laser emission intrinsic absorption by the substance may occur enough, possibly at crystalline structure defects where local properties differ from average over bulk causing lowered barriers to optical quanta absorption [5]. In real solids, the absorbing extrinsic inclusions, as a rule, are the sources of the optical breakdown [9, 10].

Taking into account the observed strongly dislocated crystal structure near pores, the mass transfer was considered to be realized by the dislocation mechanism. However, consecutive studying the dislocation structures near appeared small (about micron size) pores has shown that only about 5% of the pore volume is caused by the dislocation mechanism of mass transfer [11]. The hypothesis has been made that the main carriers are the crowdions in spite of the existing conception that for different type interstitial atoms, the formation energy is rather high (of the order of several electron-volts) [12]. The crowdion energy in copper, for instance, is of the order of 5.5 eV [13].

In this connection, analysis of the mass transfer process in a locally heated area of a crystal is thought actual taking into account the dynamics of heating and substance movement under conditions of high pulse intensity of the process from the point of view of continuum mechanics.

The aim of the work is studying the mechanisms of mass-transfer in a locally heated area during a micro-void formation as a result of optical breakdown in the KCl single crystal under laser emission.

EXPERIMENT AND RESULTS

In the experiments carried out at room temperature, KCl single crystals grown by Kyropulos method from raw material of ХЧ (chemically pure) grade were used. For irradiation, the pulse laser with modulated quality on the neodymium glass with pulse duration $\tau \approx 5 \cdot 10^{-8}$ s, wavelength $\lambda = 1054$ nm, and energy per a pulse near 1 J was used. The laser with infra-red emission and short pulse duration was chosen in order to lower the losses for absorption and scattering, including that at the crystal non-polished surfaces, and to obtain high energy flux density enough for optical breakdown due to the crystal intrinsic absorption in the lens focal plane. The laser emission was focused by a lens with focus distance $F = 10$ cm and working aperture diameter $2b \approx 1.5$ cm. The emission flux density in the working zone of the focal plane was determined by the pulse energy and the focal zone size. According to [14], the focal zone in the lens focus has elongated, to a first approximation, cylindrical shape,

with $l_{||}$ length and $l_{\perp}$ radius which are determined by the relation [14]:

$$l_{||} = \lambda (F/b)^2; \; l_{\perp} = \lambda (F/b), \quad (1)$$

where F is lens focus distance, λ is the wavelength of incident light.

Taking into account the values of the parameters included, for our case we obtain: $l_{||} \approx 130 \cdot 10^{-4}$ m, $l_{\perp} \approx 10 \cdot 10^{-6}$ m. The focal area volume is $V \approx 4 \cdot 10^{-14}$ m^3.

In order to provide precise positioning the crystal relative to the light beam under laser exposure, in the previous experiments we found precise spatial position of the laser beam axis and focus under microscope using the glasses with deposited thin metallic films. The crystal was positioned in the point of the light beam geometric focus and irradiation was carried out. The irradiation conditions were chosen in order to obtain pores of micron size.

The main result of these experiments was that the formed voids were always positioned practically on the beam axis close to the center of the focal area (geometric focus). Most often only one pore formed, rarely – two pores, more rarely – more than two. In Fig. 1 the case is shown where two pores positioned practically on the axis of the focal area have appeared.

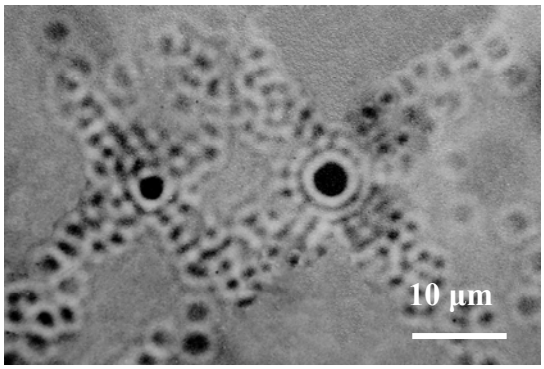

Fig. 1. Dislocation structure near the pore formed

Formation of a pore chain, most likely, begins from the distant (along the beam) pore. New voids develop towards the beam, because every time the emission weakens and defocuses behind the void. Thus, in our experiments every time the optical breakdown takes place near the lens focal area, on its axis. This is not occasional fact as the detailed analysis of the light field intensity distribution in the lens focus shows that the field in the focus vicinity is complex [15]. In the meridian plane along the optical axis, the intensity is characterized by a function $[(1/x)\cdot\sin x]^2$. In the focus there is a principal maximum and a number of additional maxima of lower intensity are along the lens axis, their intensity lowers as the distance from the focus increases (symmetrically – before and behind the focus) [15]. In our case, the distance to lateral maxima is about ± 150 μm. The second pore appeared at the distance only 19 μm (see Fig. 1), i.e. in the vicinity of the principal maximum.

The fact that voids are formed preferentially in the area of light maximum intensity allows assuming the role of intrinsic absorption (including non-linear one connected with emission flux high density and multi-photon absorption [10]) being predominant. The following supports such conclusion. Using the light-scattering method as in [16] we determined the concentration N of inhomogeneity particles in the crystals under study and also their size d: $N \leq 10^{14}$ m^{-3}, $d \leq 10^{-6}$ m. As the focal area volume is $V \approx 4 \cdot 10^{-14}$ m^3, probably several (≈ 4 ones) foreign particles can be within it. Evidently, the probability of occasional being of even one particle in the center of the focal area is significantly less than a unity; at the same time, the optical breakdown was always observed just near the center of the focal area. It is worth to be taken into account as well that among the inhomogeneities revealed by light scattering method in pure enough alkali-haloid single crystals, the micro-voids non-absorbing the light are predominant [17, 18].

DYNAMICS OF LOCAL HEATING AND PLASMA FORMATION DISCUSSION

For constructing the model best of all describing the dynamics of destruction in a single crystal it is necessary to estimate its characteristic durations and energetic constituents. Let us evaluate the duration of heating the crystal area where plasma with temperature $T_{pl} \approx 5.5 \cdot 10^3$ K. The plasma temperature was taken from [6] and supported by many other works where it was determined by spectroscopic investigations.

The average value of laser emission flux density J per a pulse in the lens focal plane is determined by the relation:

$$J = E_{pulse} / \tau \cdot \pi (l_{\perp})^2, \quad (2)$$

where E_{pulse} is the pulse energy ($E_{pulse} \approx 1$ J); τ is the pulse duration ($\tau \approx 5 \cdot 10^{-8}$ s), $l_{\perp}$ is an average value of the focal zone radius (defined by (1)). After substitution of numerical values we obtain $J \approx 6.37 \cdot 10^{16}$ W/m^2.

To concretize further estimations it is necessary to take the shape and sizes of some initial area of absorption. In [19] the filming of destruction process was made, and in the first shots of the film, the optical contrast zone with size about 70…100 nm was observed which was identified as a void occupied by plasma. Taking into consideration the identity of our experimental conditions, we take for estimations the initial absorption area (plasma nucleus) as a cube with 100 nm edge. The energy flux I_{abs},which is absorbed by such nucleus is:

$$I_{abs} = J \cdot S \cdot k, \quad (3)$$

where S is the irradiated square; k is absorption coefficient which is unknown precisely, but it is known that infra-red radiation is practically completely absorbed by a plasma layer of micron size [19]. In [20] it was shown that luminous substance is non-transparent, so in following we take $k = 1$. Assuming for simplicity that the radiation enter into the cubic nucleus through one of the cube face with square $S = 10^{-14}$ m^2 we obtain $I_{abs} = 6.37 \cdot 10^2$ W.

Let us evaluate the energy losses caused by heat radiation and thermal conductivity in the crystal. The heat radiation flux density J_{rad} is defined as

$$J_{rad} = \sigma T^4, \quad (4)$$

where $\sigma = 5.669 \cdot 10^{-8}$ W/(m$^2 \cdot$K^4) is Stephan-Boltzmann constant; T is plasma temperature. Substituting σ and $T = 5500$ K in (4) we obtain $J_{rad} \approx 5.2 \cdot 10^7$ W/m^2. The flux of energy losses for heat radiation I_{rad} is defined as

$$I_{rad} = J_{rad} \cdot 6S. \quad (5)$$

Substituting the numerical values we obtain $I_{rad} \approx 3 \cdot 10^{-6}$ W.

The flux density of losses for thermal conductivity J_{therm} into the surrounding crystal we estimate writing the equation for the flux density as follows:

$$J_{therm} = -\lambda \, grad \, T = -a^2 \rho c_V \, grad \, T, \quad (6)$$

where λ is thermal conductivity coefficient; a^2 is thermal diffusivity coefficient; ρ is KCl crystal density; c_V is specific heat of KCl crystal.

The temperature gradient at the plasma-crystal boundary we evaluate in assumption that the maximum temperature $T_{max} = 5.5 \cdot 10^3$ K falls to room value at the distance about the taken size of plasma cloud $l_{pl} \approx 10^{-7}$m, therefore

$$grad \, T \approx T_{max} / l_{pl} . \quad (7)$$

Taking into account $a^2 = 7.3 \cdot 10^{-6}$ m^2/s, $\rho = 2 \cdot 10^3$ kg/m^3, $c_V \approx 580$ J/(kg·K), we obtain $J_{therm} \approx 4.7 \cdot 10^{11}$ W/m^2. The heat energy losses flux from the plasma cloud into the crystal is $I_{therm} = J_{therm} \cdot 6S \approx 2.8 \cdot 10^{-2}$ W. Thus, the energy losses for heat radiation and thermal conductivity are found to be by orders of value less than the absorption energy flux I_{abs} that provides fast local heating and plasma formation.

To evaluate the time period of plasma formation at temperature $5.5 \cdot 10^3$ K we estimate the energy value necessary for its formation E_{heat} neglecting the losses. To a zero approximation, neglecting dependences of specific heat on aggregate state and temperature of the heating volume we can write:

$$E_{heat} = m \, [c_V (T_{pl} - T_m) + (\Delta H_m + \Delta H_{ev})]. \quad (8)$$

Taking the temperatures of melting and evaporation as $T_m = 1045$ K and $T_{ev} = 1686$ K, respectively, the plasma temperature $T_{pl} = 5.5 \cdot 10^3$ K, the heats of melting and evaporation $\Delta H_m = 3.43 \cdot 10^5$ J/kg, $\Delta H_{ev} = 21.7 \cdot 10^5$ J/kg, respectively, for the plasma cloud of $\approx 10^{-7}$ m size we obtain estimation $E_{heat} = 10^{-11}$ J.

Taking into consideration the absorption flux value ($I_{abs} = 6.37 \cdot 10^2$ W) and the obtained energy E_{heat}, we evaluate the heating time τ_{heat}, which essentially defines the dynamics of whole relaxation process: $\tau_{heat} = E_{heat}/I_{abs} \approx 1.57 \cdot 10^{-14}$ s. This evaluation can be found noticeably understated on account of inexactly known both absorption coefficient (k) and growing with time absorbing area size.

Finite size of the plasma cloud is found to be of the same order [11, 19, 20], as the size of the void formed; therefore, if the cloud shape is taken also cubic but with edge of the order of 10^{-6} m, the size of heating area increases by three orders of value, and the absorption flux increases only by two orders, the fact in turn results in increasing the heating time by an order of value: $\tau`_{heat} \approx 1.57 \cdot 10^{-13}$ s.

Thus, the time of plasma formation and heating is by orders of value less than the laser pulse duration $5 \cdot 10^{-8}$ s; the plasma cloud size variation does not practically change the relation between the absorption flux and the energy loss one, and consequently does not practically influence on the plasma heating time.

The pressure in the energy release area at $T = T_{pl}$ can be estimated by the expression $P_{max} \approx K (\delta V/V)$ (assuming the heating area remaining as a compact formation), where K is the modulus of volume compression, $\delta V/V = 3\alpha\Delta T$ is the volume jump connected with heating. Taking into account $K = 1.74 \cdot 10^{10}$ N/m^2, $\alpha = 48 \cdot 10^{-6}$ K^{-1}, and $\Delta T \approx 5200$ K we obtain $P_{max} \approx 1.3 \cdot 10^{10}$ N/m^2. From the viewpoint of the mechanics of continua the process under study where the pressure achieves value exceeding the theoretical strength limit for the time less 10^{-6} s, should be considered as explosion-like [21] or shock process [22].

From the experiment (see Fig. 1) it is follows that the substance from the "explosion area" jumps out at a distance about 10 sizes of the pore formed, i.e. in our case, at the distance of the order of 10^{-5} m in the directions of easy sliding. The process of the mass transfer follows the heating process overlapping to some degree, therefore the duration of the main mass transfer correlates with the heating time period $\tau_{heat} \approx 10^{-13}$ s, while the former remaining always more but not exceeding the laser pulse duration $5 \cdot 10^{-8}$ s. Indeed, the pulse duration variation from $5 \cdot 10^{-8}$ s to 10^{-8} s in no way influences the destruction picture [11]. It was shown in [19] that the void becomes practically formed for tens nano-seconds.

The minimum time of plastic relaxation τ_{rel} due to deformation by dislocation mechanism (as the most fast) can be obtained in assumption of dislocation movement with extremely high velocity close to acoustic speed. In aluminium, for example, the dislocation speeds of the order of 0.8 υ_{ac} were observed under extremely high loads [23]. In our case, the destruction area size is about 10^{-5} m, therefore the relaxation time is $\tau_{rel} \approx 2 \cdot 10^{-9}$ s, i. e. the dislocation mechanism is, in principle, could be realized, but experimentally it was not observed in the beginning stage. In Fig. 1 we observe dislocation loops with diameter larger than the void diameter. That indicates the dislocations have appeared more later, seemingly, before crystallization of melt remained in void walls [11], and the total contribution of dislocations into pore formation, as mentioned, is very small.

Taking into account the observed explosion-like heating dynamics we can assume that in such process an shock wave appears which can move with supersonic velocity. It was shown in [24] that under certain conditions crowdions can move with velocity exceeding the acoustic value. In [25] using computational modeling for Ni 2D-model the crowdion movement speed range was established as $(12.3 \ldots 14) \cdot 10^3$ m/s. These two factors: existing the shock wave and high velocity of crowdions can provide the necessary high speed of mass transfer in the beginning stage of the process during the shock wave passes the relaxation area.

The general scheme of plastic deformation arising from abovementioned estimations and observations is seemed as follows. In the beginning, under action of the shock wave the crowdions are generated which carry the substance from the high pressure area and move along close-packed atomic rows (‹110› type directions in KCl crystals); the void is formed almost completely during the shock wave passing the relaxation zone cross-section. This time is of the order of τ_{rel}, i. e. $10^{-9} \ldots 10^{-8}$ s. After falling temperature and pressure and vapor condensation into liquid, at the end of relaxation

process, the void boundaries expand already under liquid melt pressure, and the mass transfer dislocation mechanism comes into action providing additionally some enlarging the void volume. This process continues also after stopping the laser emission, during the crystal cooling down to the melt crystallization in the void and formation of a pore with size observed.

In [19] the data are presented on the micro-filming a void growth during irradiating the crystal by laser short pulses with duration 80 ns. The obtained in [19] data we showed at the plot (Fig. 2) in the linear scale with axes d/d_0 and t (d is diameter of optical contrast picture which is considered in [19] as the void boundary, d_0 is the finite diameter of the void formed). In such presentation of results [19], concerning to the stage of the void growth, indeed as it was supposed, two characteristic parts are observed. If the beginning part of the void fast growth (about from 80 to 200 ns) and the second part – slow growth (from 300 ns to 10 μs) are extrapolated by direct lines, one can see that the void growth velocity in the second stage decreases by about four orders of value in comparison to the first stage (from 10^2 to 10^{-2} m/s). At that in the first stage, the void quickly reaches practically its finite size as it is observed at the end of the experiment (see Fig. 1).

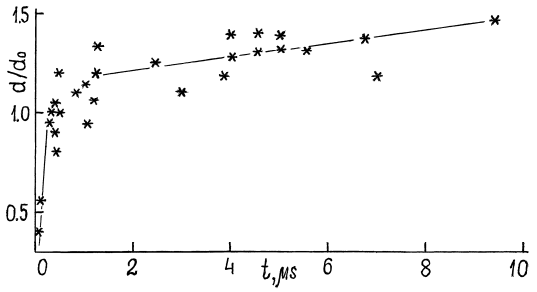

Fig. 2. Growth kinetics of a void transverse size in the stage of radiation exposure to 10 μs (laser pulse duration 80 ns, d is pore size in t time moment, d_0 is the pore finite size)

It can be assumed that the following drastic slowing the void growth speed is connected with increasing the void volume and decreasing the pressure in it, as well as stopping the plasma heating by laser pulse. It is in this stage the dislocation mechanism comes into action. This follows from the fact that observed in Fig. 1 dislocation loops have size larger than the observed finite size of the void d_0. Their size corresponds to the void size in the second (slow) part of its growth (see Fig. 2).

The observations and conclusions described above are in logical accordance both with estimations of the process dynamics and the developed scheme for action of void formation mechanisms under optical breakdown.

Of interest is the evaluation of energy expended to void formation in the stage of crowdion mechanism action. This can be evaluated by the order of value as the energy for generating crowdions in necessary quantity (taking into account crowdion high mobility), i. e. $E_{cr} = (V_{void}/\omega)\cdot\varepsilon_f$, where V_{void} is void volume; ω is atomic volume; ε_f is formation energy of a single crowdion. Substituting the numerical values we obtain: $E_{cr} \approx 5\cdot10^{-8}$ J. If consider the total energy of a void formation $E_{total} = P_{max}\cdot V \approx 8\cdot10^{-8}$ J, the obtained E_{cr} seems reasonable and logically agrees with E_{total}; therefore, the developed conception is thought to be right and experimentally supported.

CONCLUSIONS

The experiments have been carried out where the conditions for effective realization of optical breakdown were provided in KCl single crystal due to local intrinsic absorption. Evaluations of energetic constituents and characteristic durations of the process in different stages have been fulfilled. Based on the evaluations and the picture of the breakdown it has been shown that the single real mechanism of substance carryout from the energy release zone in the beginning stage may be the mechanism of crowdion generation and moving with high, even supersonic velocity, possibly, with shock wave participation.

The dislocation mechanism enters practically at the end of the process and contributes a little. A void grows under melt pressure and emits prismatic dislocation loops with size larger than the size of the void remaining after melt crystallization.

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МЕХАНИЗМЫ ОБРАЗОВАНИЯ МИКРОПОЛОСТЕЙ ПРИ ОПТИЧЕСКОМ ПРОБОЕ МОНОКРИСТАЛЛОВ KCl В УСЛОВИЯХ ЛАЗЕРНОГО ОБЛУЧЕНИЯ

М.А. Волосюк

Экспериментально исследовано явление оптического пробоя монокристаллов KCl сфокусированным излучением лазера на неодимовом стекле с модулированной добротностью, длительностью импульса $5 \cdot 10^{-8}$ с, длиной волны $\lambda = 1054$ нм, энергией импульса порядка 1 Дж в режиме локального собственного поглощения излучения лазера монокристаллом. Оценки энергетических составляющих процесса локальной тепловой вспышки и характерных времен продолжительности разогрева локальной области и длительности релаксационного процесса и сравнение их с экспериментальными данными показали, что процесс релаксации в таких условиях протекает в две стадии: первая – быстрая, сопровождаемая краудионным массопереносом с участием ударной волны, и вторая – медленная, с участием и дислокационного массопереноса.

МЕХАНІЗМИ УТВОРЕННЯ МІКРОПОРОЖНИН ПРИ ОПТИЧНОМУ ПРОБОЇ МОНОКРИСТАЛІВ KCl В УМОВАХ ЛАЗЕРНОГО ОПРОМІНЮВАННЯ

М.А. Волосюк

Експериментально досліджено явище оптичного пробою монокристалів KCl сфокусованим випромінюванням лазера на неодимовому склі з модульованою добротністю, тривалістю імпульсу $5 \cdot 10^{-8}$ с, довжиною хвилі $\lambda = 1054$ нм, енергією імпульсу порядка 1 Дж у режимі локального власного поглинання випромінювання лазера монокристалом. Оцінки енергетичних складових процесу локального теплового спалаху і характерного часу тривалості розігрівання локальної області і тривалості релаксаційного процесу та порівняння їх з експериментальними даними показали, що процес релаксації в таких умовах протікає в дві стадії: перша – швидка, супроводжувана краудіонним масопереносом за участю ударної хвилі, і друга – повільна, за участю і дислокаційного масопереносу.