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Change of oxide ceramics microhardness under action of high current pulsed beam of low energy electrons

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The main point of the process of impact of a high-current pulsed beam for low-energy electrons (HP-BLE) is that a kinetic energy of accelerated particles turns into a thermal energy. A dynamic of thermal fields and mechanical stress waves, driven in the target, determinate by cathode beam parameters, is capable to determinate appropriate structure-phase transformations in surface layers of materials and, finally, a character of their physico-mechanical properties changes.

This article describes the first attempt to study effects of HPBLE impact on oxide ceramic structures.

1 Experimental technique

A subject of inquiry was lithium-titanium ferrite ceramics of the chemical composition; $\text{Li}_{0.649}$ Fe_{1.598} Ti_{0.5} Zn_{0.2} Mn_{0.051}O₄. Beaming was carried out in vacuum ($p=10^2$ Pa) by single beam impulses of low-energy electrons with the following parameters: E=15 keV, current density in the impulse (12-25) A/sm², impulse duration-(50) mcs, repletion rate of impulses is 1 Hz. Energy density in the impulse $W_{\mu} = (9, 5-19)$ J/sm² conformed to the indicated parameters. number of cathode beam impulses was N=1. 10. Surface morphology was traced with the help of optical microscopy method. Micro-hardness measuring was carried out with the use of the micro-hardness measuring device Π MT-3. A roentgen-phase analysis was implemented with the use of the device Π POH 4-07. It is determined that electron processing did not involve noticeable changes in a phase composition of the ceramics, that kept its spinel structure.

2 Experimental results

2.1 Impact of the indentor loading amount of P on the micro-hardness (H_v) of the ferrite ceramics before beaming has been studied (Table 1). A fairly strong dependence of values H_v on value P has been determined. Scientific literature explains the described effect by a phenomenon of dent elastic restitution ^[1,2]. To determine real influence of elastic restitution on the material micro-hardness measuring results one may use the following correlation between indentor loading and a material resistance at its implantation^[2-1]:

$$P=C_1 d + C_2 d^2, \qquad (1)$$

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where coefficients C_1 and C_2 characterize a dent elastic restitution and a resistance of deformable material, d is a length of the dent diagonal.

Table 1 Micro-hardness of non-beamed ceramics at various indentor loadings

ſ	Р, Н	0.3	0.5	1.0	1.5	2.0
	HV, GPa	9.7	8.7	7.6	6.7	6.4

Calculated values of coefficients C_1 and C_2 of the non-beamed ceramics are illustrated in the Table 2 (column 1). According to the table data parameter C_1 is non-zero for a non-beamed sample. Thus, the micro-hardness testing results of the ceramic beamed samples must be analyzed taking into consideration a scale effect.

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W_i , J/sm^2	0	9.5	9.5	9,5	14	19
	1	2	3	4	5	6
number of impulses N	0	1	1	10	10	10
h, mem	0	15	25	10	10	10
C _I , H/mm	18.5	7.0	12.2	23.2	27.0	35, 4
$C_2 \times 10^{-3}$, H/mm^2	2, 3	3.1	2, 9	2.8	3.4	2.3

Table 2 Calculated values of coefficients C_1 and C_2 for ferrite ceramics before and after its processing by HPBLE with a various density of the energy in the impulse

2.2 Beaming with a single impulse of the cathode beam $W_i = 9.5 \text{ J/sm}^2$ of ferrites involved formation of a crack network on the surface and determination of the ceramics microstructure. Micro-hardness value, measured at the loading P=0.3 H was $H_v = 5.7$ GPa. Measuring H_v at big P was not possible because of chips and detachments of the micro-assemblies adjoining to the dints' borders.

Thus, this mode of electron processing resulted in a considerable decline of the calculated value H_v . Under those conditions of measuring for a non-beamed sample $H_v = 9.7$ GPa (Table 1).

Grinding with the beamed surface of layers with thickness of 15 and 25 μ m is accompanied with increase in H_v up to values 7.2 and 7.8 GPa, respectively. A full recovery of values H_v took place at a length h of 60 mcm from the beamed surface. After removal of the surface layers of the thickness over 10 mcm, it was possible to carry out some experiments on measuring micro-hardness under various indentor loadings that helped determine values of coefficients C_1 and C_2 (Table 2, columns 2 and 3).

Results, indicated in Table 2, testify that after beaming the effect of dent elastic restitution influences micro-hardness measuring insignificantly. This is clearly indicated by the values of coefficient C_1 of the processed ceramics. While the distance from the surface is increasing coefficient C_1 tends to the values typical for a non-beamed ceramics.

2.3 Impact of 10 impulses of the cathode beam resulted in a strong erosion of the whole surface of the samples. When the electron bunch hits a ceramic button, deepening all over the beaming area was observed. Intensive thermal heating by the cathode beam was obviously accompanied by melting of a thin surface layer and active substance evaporation.

Because of the strong erosion of the surface at this mode of the ceramics electron processing microhardness measuring could not be done. That is why the second polishing of the surface was required involving removal of the layer with the thickness of (5-10) mcm in the beamed zone. Its thickness exceeded the run of electrons with the energy of 15keV that was 2 mcm in this material. That is why experimentally studied effects of the cathode beam behavior refer to the medium which is outside of the beamed layer.

It was determined that a multiple impulse electron processing involves increase in calculated values of micro-hardness in deep layers of ceramics up to 30%-50%. This effect is growing with the rise of energy density in the beam. At the same time, while indentor loading and indentor implantation are increasing, changes in micro-hardness are less noticeable.

The calculated values of coefficients C_1 and C_2 , for these conditions of the experiment are indicated in Table 2 (columns 4-6). It can be marked that electron processing influences the most coefficient C_1 , the value of which increases with the increase W_* almost twice. At the same time a tendency of coefficient C_2 increase is appeared much weakly. Consequently, one of the reasons for calculated values increase of microhardness of the beamed samples is a more effective process of the dint reconstruction after the loading is off in comparison with the non-beamed ceramics.

We can assume that ceramic sample beaming with 10 impulses resulted in a compression stress in the surface layers. Micro-indenting without the loading intensifies the relaxation processes, causing the indentor-dent downsizing, providing reduction of local internal stresses in its area. Micro-hardness calculation by the reconstructed dint resulted in obtaining overestimated values.

A single impulse of the cathode beam causes the situation when scale effect impact on a resultant value of micro-hardness is reducing. The result is an apparent decrease in micro-hardness of the beamed ferrite ceramics.

From the data, illustrated in Table 2, we can see that both a single-impulse beaming and poly-impulse beaming there is a clear tendency towards coefficient C_2 rise. Consequently the both modes of electron processing may cause ceramics hardening. This effect, in "pure form" is stipulated by structural changes of the material. The latter is most likely caused by a spasmodic change in the temperature of the surface and the bordering medium taking place under the HPBLE. Thermal hit causes generation of high-power waves of thermal stresses sufficient for plastic deformation processes and increase in dislocation density and ceramics hardness.

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