TEMPERATURE AND PRELIMINARY DEFORMATION INFLUENCE ON MICROMECHANICAL AND ACOUSTIC-EMISSION PROPERTIES OF MgO SINGLE CRYSTALS

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The MgO single crystals were subjected to the annealing at T_{an}=150-600 °C as well as to the plastic deformation over ε=0-6% at 150 and 600 °C. The resting influence on the mechanical properties of these samples was investigated. It was established that with increasing of T_{an} and ε at T_{def}<300 °C the microhardness increases and the acoustic emission decreases. Thus, the conditions for increasing of strength and decreasing of brittleness of MgO crystals were revealed.

1. Introduction

Definite value and type of external influence on the crystalline materials form the corresponding defect structure controlled by activity of the processes of initiation, interaction and annihilation of the introduced defects [1-4].

Changes of external conditions: enhancement or disposal of deformation force, deformation temperature, conditions of the preliminary or subsequent annealing, etc. result in the modification of the material original equilibrium structure. A new non-equilibrium structure arises that excites the relaxation processes trying to form a new more equilibrium microstructure. The formation of a new structure is followed by the modification of internal energy, physical properties of the material, including mechanical ones [5-7]. In this case the coupling of internal material structure - its strength properties is realized [7-8].

The same correlation is, as a rule, realized for both annealed and preliminary deformed materials [9-12]. Nevertheless, influence of actual regularities of thermal treatment (its regime) on the structure and mechanical properties of materials, especially preliminary deformed ones, is studied insufficiently [9, 11, 13]. Peculiarities of influence of defect structure formed at different temperature on modification of the above properties before and after storage are not studied.

Analogous investigations are important because for prediction of property modifications the selection of optimal regimes of plastic deformation, thermal treatment, and ageing is needed. The method of intensive plastic deformation is perspective for structure state and mechanical property modifications [14]. However, mainly strong deformation is realized at room temperature. Deformation at high temperatures results in the specific structure changes of materials that can cause the inadequate modification of their mechanical properties [14]. Search of optimal conditions of reasonable changes of physical-mechanical properties with wider variations of experiment for study of the relation “structure- properties” is needed.

In this connection in the present paper the influence of the preliminary deformation induced at high temperatures as well as the subsequent storage on the changes of regularities and correlation of microhardness and acoustic-emission properties of MgO single crystals was investigated.

2. The experimental procedure

The MgO crystals subjected to annealing and preliminary plastic deformation by uniaxial compression were investigated. The annealing temperature (T_{an}) in the interval 150-600 °C (exposure time at each temperature was equal to 10 min.), and degree of plastic deformation at room temperature (ε) in region 0-6% were varied. The cooling rate of samples was oscillated from 1 to 2 °C/min. (the samples were cooled in a furnace). The annealed samples before and after ageing for 30 days were investigated.

The microhardness (H) was measured by device PMT-3, the Vickers pyramid was used, and indentor load in the interval 20-200g was changed. Registration of acoustic emission signals (AE) under microindentation was made by means of special equipment added to PMT-3. The number of signals during the whole microindentation cycle (N), under loading (indentor penetration) (N_1) and at unloading (indentor removal) (ΔN) was registered. Microhardness measurement and registration of AE impulses at room temperature were performed. Relaxation ratio at unloading is determined by correlation ΔN/N, and relative relaxation at unloading and loading – by $\Delta N/N_1$.

3. Experimental results and discussion

The dependences of microhardness magnitude and AE signal number on the annealing temperature are presented in Fig. 1.

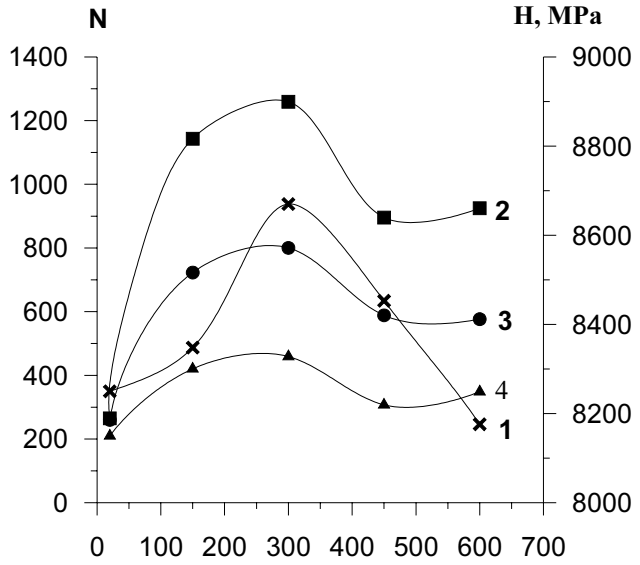

Fig. 1. Dependences of microhardness (1) and number of acoustic emission signals for the whole cycle of microindentation (2), under loading (3) and after unloading (4) on the annealing temperature of MgO single crystals.

It is seen that course (shape) of f = H (T_{an}) and f=N(T_{an}) is two-stage and sufficiently coordinated: the enhancement of microhardness is followed by AE signal number increase on the first stage and on the second one – the H decrease is followed by AE decrease too. Both dependences show the maximum at T_{an}=600 °C. Microhardness after annealing at 600 °C is practically the same as microhardness of initial MgO (Fig. 1, curve 1). The shape of N (T_{an}) dependence shows a character analogous to N_1 (T_{an}) and ΔN (T_{an}). It can be supposed that the difference of MgO crystals structure states arising at thermal treatment (size and distribution

of blocks, their orientation, etc.) is important for determination of behaviour of these dependences.

Depending on annealing temperature the different substructure is forming in MgO crystals, which is to varied degree unstable and accordingly differently influences the material mechanical properties.

It is known that the non-equilibrium defect state is varied for the time due to processes of self-regulating.

For studying the influence of structure state stabilization on mechanical properties of MgO, the samples annealed in the range of 150-600 °C after certain storage were investigated. Data of microhardness measurement and acoustic emission registration obtained after storage of the 30 days duration are presented in Fig. 2. It is seen that microhardness magnitude and AE signal number after storage are increased: curves 2 are placed higher than curves 1. In this case the microhardness enhancement effect increases with the annealing temperature increasing (Fig. 2a).

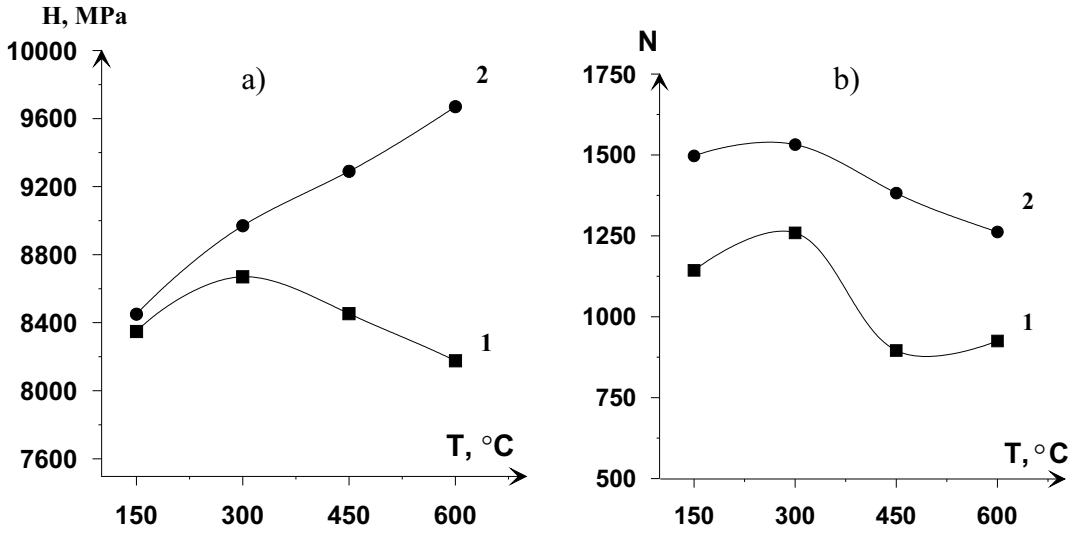

Fig. 2. Dependence of microhardness (a) and acoustic emission (b) on the annealing temperature of MgO crystals. 1 – before storage, 2 – after storage for 30 days.

Thus, the changes of microhardness and acoustic emission as a result of annealing and subsequent storage at room temperature of annealed MgO crystals show a coordinated character: the sample hardening is followed by more intensive emission of elastic waves. However, this correlation is not always realized quantitatively. After storage of the samples annealed at 600 °C the microhardness enhancement is equal to 1500 MPa, for the ones annealed at 150 °C – only 50 MPa, while the AE activity increase in both cases is approximately the same and equal to 400 (Fig. 2). This fact is due to the opposite course of H (T_{an}) and N (T_{an}) dependences, with T_{an} growth the microhardness enhancement increases but AE activity decreases (Fig. 3). The behaviour of the opposite sign before and after storage is shown.

It should be noted that not only the summary account of acoustic emission after storage increases but the acoustic emission under loading as well as under unloading are also changed in this way. The obtained data of the microhardness and acoustic emission changes are probably connected with the stabilization process of internal structure state of MgO annealed crystals during storage.

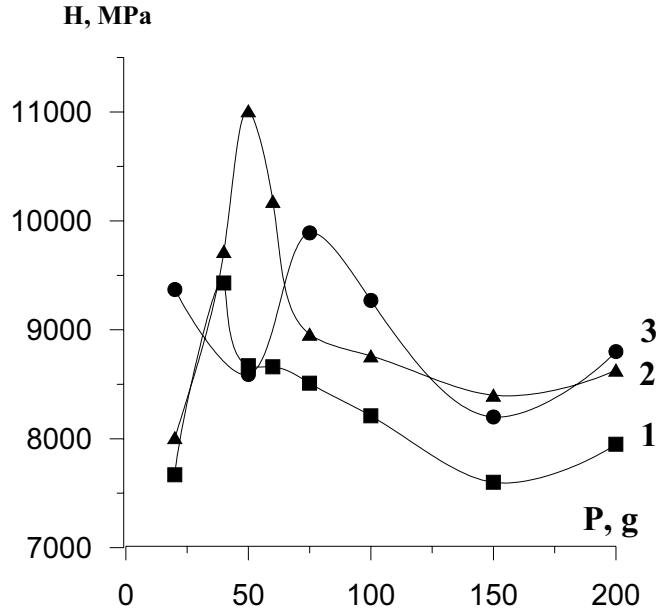

Fig. 3. Dependence of microhardness value of MgO single crystals annealed at 600 °C on indentor load. 1 – directly after annealing, 2 – after storage for 12 hours, 3 – initial samples.

Further more detailed experiments have shown that the effect of thermal treatment and subsequent storage influence is dependent on the load value on indentor (Fig. 3). In the figure the changes of microhardness magnitude on indentor load for MgO crystals annealed at 600 °C before and after storage as well as for initial samples are presented. The non-monotonous character of dependences H(P) for all third series samples is revealed. Clear–cut extremes in the region of relatively small loads (P<50g) are observed. Besides, the microhardness of treated samples directly after cooling for whole P is mainly lower than microhardness of initial MgO: curve 1 is placed lower than curve 3 (Fig. 3).

An interesting effect for thermally treated MgO crystals after their short-term storage (12h) was revealed: the microhardness value increased, extreme on curve 1 was shifted to large P and significantly increased. Microhardness for P=50g increased from 8500 to 11000 MPa, and for large P - the enhancement effect was lower and microhardness value was practically the same as for initial samples (Fig. 3, curve 2). Thus, the annealing at 600 °C resulted in the H decrease practically for the whole P investigated; the subsequent storage of these samples caused the microhardness enhancement and its value recovering to the value of initial samples. We ought to distinguish the load region of 20<P<70g where H (P) dependence extreme modification is revealed.

The dependences of microhardness and acoustic emission number arising under microindentation on P value for MgO crystals annealed at 600 °C and rested in the course of 12 hours are presented in Fig. 4. It is seen that parameter N_{AE} (curve 2) is non-monotonously changed, but on the whole it increases with P increase.♦) Comparison of H (P) and N (P) dependences has shown some discrepancies at 50 and 200g (Fig. 4). In the region of P=50g the maximal microhardness corresponds to AE minimal activity, on the contrary, at P=200g the less values of microhardness correspond to AE activity being twice as much as at P=50g. However, dynamics of the external load growth from 20 to 200g in toto and suitable changes of microhardness and acoustic emission indicate that as a whole microhardness decreased by 21% and AE increased by 80% (Fig. 4).

♦) The N_1 and ΔN parameters the analogous course of dependences are revealed.

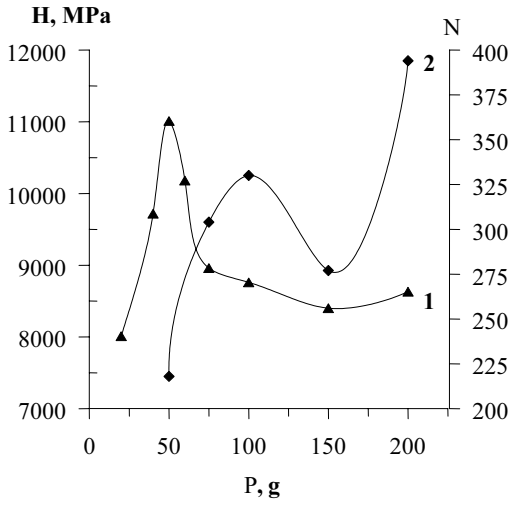

Fig. 4. Dependence of microhardness (1) and summary account of the acoustic emission signals (2) on magnitude of indentor load for the annealed and aged (12h) MgO single crystals.

This means that H and AE modifications are different in sign and quantity. The H and AE changes with P growth are of opposite sign: the H decrease is followed not by AE decrease, but by its enhancement. The number and size of cracks arising within the indentation zone are rather responsible for the observed effect. Investigation of deformation zones around the indentation on the (001) MgO has indicated that the number and size of cracks with P growth increase. At large P in addition to cracks the cleavages are formed, the cleavage capacity and cracks strengthen [4, 15]. It is evident that the crack and cleavage formation is one of the main causes of the microhardness decrease and AE growth, the effect obtained in this work.

The acoustic emission registered under microindentation is mainly determined by the elastic stress relaxation by means of the macro-, micro- and nanocrack formation and their annihilation.

Analysis of the obtained data and the allowance of the AE source nature allow us to conclude that for annealed and rested MgO crystals the microhardness magnitude decreases with load increasing mainly due to elastic stress relaxation by means of cracks formation under microindentation. Nevertheless, such an explanation of the observed phenomena does not always elucidate it.

The data of the relaxation coefficient change ($\Delta N/N$) under unloading are presented in Fig. 5. It is seen that relaxation under unloading is minimal at 50g when microhardness is maximal (Figs. 4 and 5, curves 2). With P increase the relaxation processes under unloading are *ab initio* activated but further they weakly change. This behaviour of relaxation under unloading shows that AE does not always correlate with H changes. The latter is not in agreement with concept of the physical nature of H (P) changes according to that the principal role under unloading is performed by the relaxation processes.

It is evident that the processes of microindentation are also determined by the relaxation under loading.

As it is seen from Fig. 5, $\Delta N/N_1 < 1$ at all P values, that means that $N_1 < \Delta N$, and AE activity during loading is higher than this parameter at unloading. The latter is in agreement with the assumption that microhardness value is determined not only by relaxation at unloading but also by relaxation at loading; besides, the latter one in our case makes a more essen-

tial contribution. However, both $\Delta N/N$ and $\Delta N/N_1$ reach the maximal value at 50g that conformed to the maximal value of microhardness (Figs. 4 and 5). Perhaps, the microstructure formed in this case does not sufficiently promote the relaxation of elastic stresses in the microhardness process, promoting the increased strength of the crystal.

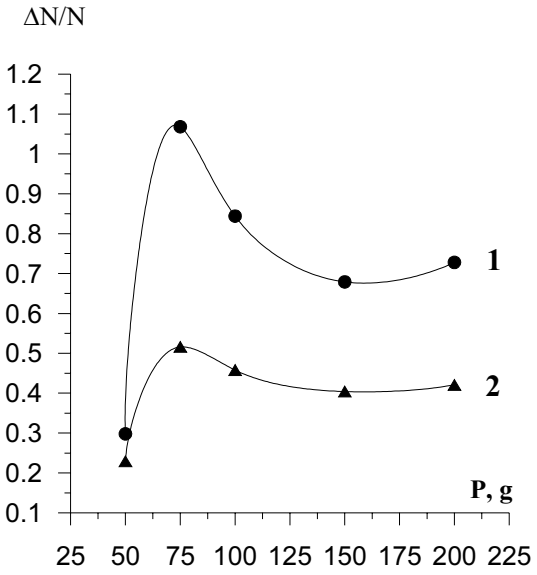

Fig. 5. Dependence of relative emission at loading and unloading $\Delta N/N_1$ (1) and relaxation coefficient $\Delta N/N$ (2) on load value.

Phenomena of the microhardness and acoustic emission changes obtained in the load region 20<P<90g evidenced the occurrence of the undersurface layer specific state of investigated MgO, which resulted in the significant changes of both microhardness and relaxation on the loading and unloading stages at microindentation.For P> 90g modification of the microhardness of annealed and stored MgO is monotonous (Fig. 3, curves 1 and 2).

Thus, it can be concluded that the deeper indentor penetration into surface layers the more stable changes of the microhardness and relaxation degree higher.

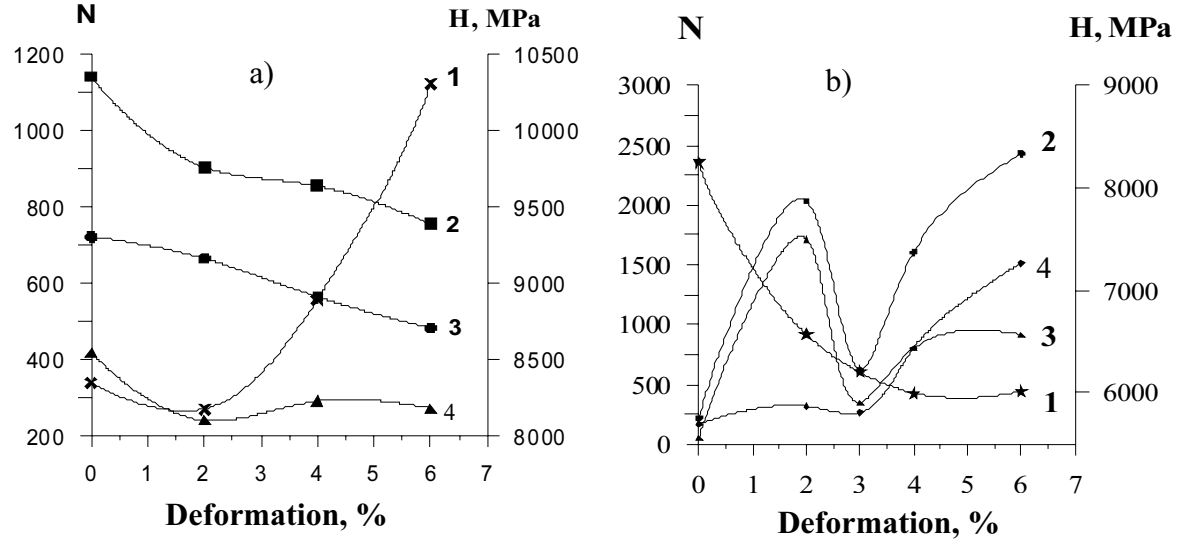

Fig. 6. Dependence of microhardness (1) and acoustic emission on deformation degree at 150 °C (a), 600 °C (b): 2 - for the whole cycle of microindentation, 3 - at loading, and 4 - at unloading.

In addition to the thermal treatment and subsequent annealing the modification of the MgO structure is performed in the following way. Crystals are deformed by uniaxial compression to different strain degree at high temperatures of 150 and 600 °C. The samples preliminary deformed at the above temperatures are kept at each temperature for 10 min and then are cooled with the rate about 1-3 °/min. Thus, these samples are treated in the same way as the annealed but undeformed samples, in this case the crystal structure is changed by both annealing and deformation at annealing temperature (T_{an}). Data of these investigations are presented in Fig. 6.

More detailed investigations were performed. The MgO crystals were deformed by uniaxial compression to different deformation value (ε) at 150 and 600 °C. In this case the material structure changes were different; the structure state was determined by the temperature deformation. The data of microhardness measurement and registration of AE signals are presented in Fig. 6. As it is seen from the figure, the microhardness and acoustic emission dependences are opposite. For the crystals deformed at 150 °C the microhardness magnitude increases with the deformation value increase, in this case the acoustic emission decreases (Fig. 6a).

The microhardness increases by 24% and AE decreases by 34% with deformation rise. The opposite behaviour of these parameters was revealed for the samples deformed at 600 °C; the microhardness decreases with deformation rise while the acoustic emission increases very significantly: about 200% (Fig. 6b). This correlation that the microhardness increase is followed by the decrease of elastic wave emission evidences that material becomes stronger and less brittle.

On the contrary, as usual, the material softening occurs when the material becomes more brittle. This was observed in our case, Fig. 6b. It is seen that MgO crystals become more fragile as evidenced by the significant increase of acoustic emission and the failure picture near the indentation too. In the latter case the number of the cracks around the indentation arises and some of them involve the distorted and branched lines.

The correlation and anticorrelation of H and AE changes obtained in this work are qualitative, some quantitative discrepancies take place. For example, the microhardness increase after annealing at 150 °C is equal to 5 kg/mm^2, and for samples annealed at 600 °C – 130 kg/mm^2. In this case the AE changes are practically in close agreement with each other for two regimes of MgO treatment, Fig. 6. This fact means that there exist other elemental acts, which are responsible for the MgO hardening but do not cause the emission of elastic waves.

The data of AE registration reveal the complex character of the N=f (ε) dependences. This indicates that the evolution of internal stress formation under processes of preliminary deformation of MgO crystals is not systematic but is rather discontinuous.

Analysis of the obtained results allows us to conclude that

– regime of MgO crystal thermal treatment due to the formed internal crystal substructure resulting in the strength increase and brittleness decrease was found. These conditions corresponded to both cases; the temperature of preliminary crystal deformation is lower than 300 °C and after annealing at 600 °C with subsequent storage at room temperature. In both cases the MgO microstructure is probably formed which proves the strength growth and acoustic emission lowering (Fig. 6). These modifications of H and AE show that revealed conditions of MgO treatment result in its hardening and brittleness decrease.
– It was established that modification correlation of microhardness and acoustic emission arising under microindentation is determined by the annealing temperature and two – stage behaviour revealed. In the case of T_{an}<300 °C the microhardness enhancement is fol-

lowed by acoustic emission activation and at $T_{an.}$>300 °C both parameters decrease. The difference of effects for each stage is probably connected with the difference of residual thermoelastic stresses arising in MgO crystals after annealing.

– The conditions of the discrepancies of the microhardness and acoustic emission changes were revealed. The short-term storage (12h) of the samples annealed at 600 °C caused certain mismatches of these parameter changes. These changes are related to the indentor load value, i.e. to the size of deformed zone under microindentation.
– Correlation of H and AE changes on deformation degree at high temperature is determined by the temperature of preliminary deformation: H (ε) increases and N (ε) decreases at T_{def}=150 °C and, on the contrary, H(ε) decreases, N (ε) slightly increases at 600 °C. In both cases the sign of H and AE changes is opposite.
– The opposite changes of H and AE are well correlated with brittleness modification of MgO crystals. Modification of the same sign allows us to suggest the occurrence of some state of defects which at certain conditions differently affected the microhardness and brittleness of MgO crystals. The conditions of the most optimal modifications of the material mechanical properties when the microhardness growth is followed by acoustic emission decrease were found.

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