MECHANISM OF PLASTIC DEFORMATION OF CRYSTALS UNDER MICROINDENTATION

Dr. hab. **Raisa P. JITARU**
Dr. **Natalia A. PALISTRANT**
Veaceslav A. RAHVALOV
Dr. **Olga A. SHIKIMAKA**
Institute of Applied Physics

This article is held in memory of Prof. Boyarskaya Yulia Stanislavovna the outstanding scientist in the field of the strength and plasticity physics. The main part of this scientific review was wrote by Yu. S. Boyarskaya. The colleagues of Laboratory of mechanical properties of crystals, founded by Boyarskaya, had expanded and prepared the final paper variant. Thus this article reflects in brief the most important results in the phenomena under microindentation published by Yu. S. Boyarskaya with co-workers. The article was published in Moldavian Journal of Physical Sciences, vol. 2, page 17 (2003), and is rewritten with some shortenings.

Summary. The profound, total analysis of the deformation peculiarities of the crystals with different chemical bond type, with different lattice structure and hardness has been performed. Three mechanisms of the microindentation process: smooth, impulse and rosetteless, has been established. The development (occurrence) of the certain indentation mechanism is depended on the structure lattice and deformation temperature too.
Keywords: microindentation, microhardness, plasticity, lattice, dislocations, rosette.

Rezumat. Este efectuată o analiză profundă și completă a particularităților cristalelor cu diferite tipuri de legătură chimică, cu diferită structură a rețelei și cu diferite durități. Au fost evidențiate trei mecanisme ale procesului de microindentare: lent, impulsiv și fără rozete. Prezența și dezvoltarea unui mecanism anumit de indentare depinde de structura rețelei și de temperatura ei.
Cuvinte-cheie: microindentare, microduritate, plasticitate, rețea, dislocație, rozetă.

Dr. hab., prof. univ. Iulia BOIARSKAIA
(29.03.1928–15.01.1996)

INTRODUCTION

Many phenomena observing under microindentation of the different type crystals can be explained in term of the dislocation plasticity. For example, the microhardness anisotropy [1-23]; the influence of the temperature [3, 4, 6, 7, 23, 24], irradiation [3, 4, 6, 25] and doping [26-29]on the change of this anisotropy character; the regularities of material failure near [4, 6, 30-36] and inside [37] indentor print, and others belong to such phenomena. There is, as a rule, a good correlation between the changes of the yield stress, microhardness and dislocation mobility in the stress field of concentrated load [6, 38-40].

However in alkali-halide crystals such as NaCl [6, 41-45] as well as in the other materials [6] the unusual phenomenon was established: a significant rise of the microhardness by lowering deformation temperature from 293 to 77K was followed by the increase (not by the decrease) of the dislocation mobility [41].

Can we in virtue of this fact suggest that in our case the interstitial [45-59] (but not dislocation) mechanism takes place? The analysis of the data obtained under investigation of the low-temperature anomaly of dislocation mobility resulted in the hypothesis of their pseudomobility and impulse character of the microindentation process [6, 44, 45, 60]. Further, this hypothesis was confirmed by direct [61] and indirect [62-65] evidences. Thus, it was shown that the anomalous phenomenon under microindentation can be explained in framework of dislocation plasticity.

DISLOCATION PSEUDOMOBILITY. IMPULSE MECHANISM OF MICROINDENTATION PROCESS

The results obtained under studying of relaxation phenomena arising at indentor removal also evidence about the occurrence of smooth and impulse microindentation processes in ionic crystals [62]. The experiments on (001) NaCl, LiF, MgO crystals were performed. The change of the dislocation structure arising under microindentation, change after sample unloading was studied.

The dislocations patterns arising around the indentation were revealed by means of an etch-pit technique. The surface etching near the same indentation was carried out two times – indentor under load and after its unloading (its removal from sample).

It was established the dislocation structures practically no change after indentor removal for the more soft NaCl and LiF crystals. Another picture is observed for MgO crystals. The dislocations rosettes progress and the edge and screw dislocations arms significant elongation after indentor removal were occurred. Many new (rows) lines of edge dislocations were originated, the last result in the marked broadening of edge arms. The number of the screw dislocation lines was essentially increased too. As a result the network of glide bands oriented parallel to <100> is markedly rose too.

The dislocation displacement, a new glide bands generation under indentor unloading it was formerly observed on the both alkali halide crystals [85] and semiconductors materials [8]. However quite unexpected is fact that after indentor removal the „prolonged" plastic deformation not the recovery one opposite to the deformation under indentor loading took place. That means the further development of the dislocation structure arising during indentor penetration is occurred under unloading.

Both this fact and different behavior of the more soft crystals and MgO ones can explain as follows. It was note above that at room temperature for the NaCl and LiF crystals the smooth microindentation mechanism is characteristic, but for MgO - impulsive one. In the first case the synchronous development of the both indentation and dislocation structure under loading take place. Therefore these structures are formed before unloading and after indentor removal do not practically change. In the second case this synchronism is disturbed.

Within the deformed zone the powerful dislocation pileups are formed; the dislocation structure progress is stoped. In definite moment these aggregations break out and new arms formation and the elongation of the old ones are occurred. These relaxation processes are not perhaps completed to the unloading moment. Therefore after indentor removal the further building of the dislocation rosettes arising at the loading will be observed. And such picture is characteristic for the MgO crystals.

It was mentioned above about an unexpected fact which was revealed for MgO crystals [62].

The further development of the dislocation structure arising during indentor penetration took place after unloading. In that work the investigations were performed using the etch-pit technique. However, there is an opinion in the literature that this method is not very reliable, because change in the dislocation structure can occur under the action of etchant [36]. Therefore, the necessity arises to prove the presence of prolonged plastic deformation, but not the reverse, i.e. a deformation having the same sign during indentor penetration and whilst standing in the sample.

MgO crystals are a suitable object for such a study. The system of slip lines arises near the indentation in the case of deformation at room temperature, i.e. one can observe the deformed zone without the use of the etch-pit technique [36]. The crystals are transparent, which permits us, using the methods developed elsewhere [3, 6], to observe the deformed region related to the indentor under load and after unloading. Therefore, the purpose of the continued work was to study the peculiarities of plastic deformation observed during the process of indentor removal using MgO crystals as an example [120].

The plates 1-1.5 mm thick were cleaved from the MgO single crystals by the {001} cleavage planes. An installation comprising a metallography microscope and a loading device was used for the deformation [3, 6]. This installation permitted observation of the indentation and its surrounding deformed region, and the failure patterns made by the indentor under load and after unloading. The Vickers diamond pyramid was used as the indentor. The indentor loads, P, were varied within the limits 100- 500 g. The experiments were performed for two indentor orientations: indentation diagonals, *d*, parallel to the directions <100> and <110>. The surface relief near the indentations was studied using an interference microscope.

A system of slip lines in the form of squares one inside the other (square sides parallel to the <100> directions) arises around the indentations on the (001) face of MgO. This distribution of slip lines was earlier observed in several papers [6, 36]. It was explained using a model of material plastic flow related to the indentation [6, 31, 36, 37]: the squares of the slip lines coincided with the emergence of slip planes, responsible for the upward movement of material, on the plane under investigation [6, 31, 36, 46]. The main slip system of MgO is {110} <110>. The {110} planes can be divided, taking into consideration their arrangement relative to the (001) plane into two types: $\{110\}_{90}$ which are perpendicular to the {001} plane, and $\{110\}_{45}$ which form an angle of 45° with this plane.

Observation of the deformation patterns for the indentor under load and after unloading enabled us to establish that the slip-line squares (a) arose during the loading process and their completion took place during load removal. Accordingly, the length of cracks (*l*) directed along the square diagonals, i.e. along < 110>, increases during the process of indentor unloading. These specific cracks have been observed by a number of authors for MgO single crystals and for the other ionic crystals with the NaCl lattice [4, 6, 31, 36, 37]. They end up, as a rule, in the apexes of slip-line squares [6, 36]. Their formation seems to be related to the occurrence of pile-ups of sessile dislocations which arise by the interaction of dislocations moving in the $\{110)_{45}$ slip planes [6, 8, 15].

All the results presented here permitted us to conclude that the unexpected phenomenon is observed on the (001) plane of MgO for the different indentor loads and different indentor orientations concerning the crystallographical directions of the deformed plane - plastic deformation having the same sign as in the loading process takes place during indentor removal. The additional material is carried out to the surface by the $\{110\}_{45}$ convergent planes which are responsible for the formation of the hills of pressed-out materials near the indentations. According to the material plastic-flow scheme for the (001) plane indentation of ionic crystals such as NaCl, the {110} active slip planes can be divided into two types: diver-gent ones which form tetrahedral pyramids with the apex near the surface and base at a depth, and convergent ones which form the inverted pyramid. The former planes are responsible for the material shift to a depth of the sample, and the latter ones for its transport to the surface [3, 4, 6, 36, 37]. The latter is evinced by the following facts. The bulging of the sample surface in the region of the slip-line square is observed for both MgO [31, 36] and other ionic crystals such as NaCl [3, 4, 6]. Our observation, performed by use of the interference microscope, confirmed in this fact. The spreading distances of hills along <110> directions near the indentations on the (001) plane were measured for a series of ionic crystals, including MgO. For MgO crystals de-formed at room temperature, the parameter $L/d \sim 1.2$ (L is the spreading distance of hills from the side of the indentation with *d*÷II<100> orientation). Measurements of L' from the indentation side to the apex of the slip-line square showed that $L/d = 1.25$, i.e. the hill along <110> ends at this apex.

The occurrence of prolonged plastic deformation during the process of indentor removal can be explained as follows. An opinion was expressed concerning the presence of smooth and impulse mechanisms of the microindentation process of crystals [6, 44]. This opinion was confirmed by a series of experimental data [61, 62, 65, 120]. The synchronous development of the indentation and deformed zone around it takes place for the smooth mechanism. This synchronism is disturbed for the impulse mechanism. Rather strong dislocation pile-ups arise in the deformed region near the indentation, and the broadening of this region is stopped. At a certain time, the bursting of the dislocation pile-ups occurs and the intensive formation of a deformed region takes place. That is, in this case, the process of development of dislocation struc-

Un moment de respiro după conferinţa ştiinţifică de la Vadul lui Vodă, anii 1980.

tures near the indentation has a relaxation character. The appearance of new slip bands, dislocation rosette arm formation, etc., is connected to the action of stress sources. Some of these sources can be non-active during indentor penetration, but they can act during the indentor resting in the sample and during unloading. The latter can operate as a trigger, resulting in the action of stress sources arranged in the deformed region. Therefore, the deformation during unloading can have the same sign as in loading and whilst the indentor rests in the crystal.

Such considerations are quite applicable to MgO crystals. The opinion has been expressed earlier that an impulse mechanism of microindentation must occur in these crystals, in contrast to the alkali halide ones, at room temperature [6, 44]. Therefore, one can expect the following. If the time an indentor stands in the sample is sufficient, the stress sources can be partly brought into action under load. Then D*a* and D*l* changes during the unloading must be less in comparison with those for the usual loading time (~ 15 s).

Thus, an unusual phenomenon was revealed by the use of MgO single crystals as an example: further development of the plastic deformation region near an indentation occurs after indentor removal from the sample. The enhancement of slip-line squares formed near the indentations on the (001) plane of MgO takes place during unloading. These slip lines are the traces of the $\{110\}_{45}$ planes which are responsible for the material transport from under the indentor to the surface, and for the formation of hills of pressed-out material. Thus the additional material transport to the surface occurs during the unloading process. The phenomenon of prolonged plastic deformation can be explained by considering the fact that the impulse mechanism of the microindentation process of MgO takes place at room temperature.

CONCLUSION

The analysis of the deformation regularities of the crystals with different chemical bond type, with different lattice structure and hardness (ionic and covalent crystals, semimetals) shows the existence of three mechanisms of the microindentation process: smooth (the development of print and dislocation rosettes around it occur synchronously), impulse (this synchronism is broken), and rosetteless (the formation of a print is not accompanied by formation of dislocation structures). One or other mechanism for the same material in the dependence of deformation temperature can take place in such sequence: in accordance with temperature lowering of the smooth mechanism is replaced by impulse, then last transfers in rosetteless one. For example for alkali-halide crystals the smooth mechanism is displayed at room temperature, but at liquid nitrogen temperature the impulse mechanism takes place. The rosetteless mechanism is characteristic for semiconductor materials at room temperature, and it changes on smooth mechanism at higher temperatures. There is a following regularity in a series of the similar crystals (for example, ionic, covalent) – than the microhardness of a crystal is higher the area of temperatures, in which the impulse mechanism of a deformation takes place at the indentation, is also higher. The presence of the impulse mechanism of deformation was confirmed by special experiments on ionic crystals.

REFERENCES

1. Mott B.V. Hardness Tests by Microindentation, Metallurgizdat, Moscow, 1960.
2. Lebedeva S.I. Definition of the Mineral Microhardness, Moscow, 1963.
3. Boyarskaya Yu.S. The Deformation of the Crystals at the Indentation Tests, Shtiinta, Chishinau, 1972.
4. Boyarskaya Yu.S., Shutova S.S., Jitaru R.P. et al. In: Deformation of the Crystals at Action of a Concentrated Load, Shtiinta, Chishinau, pp. 3-67, 1978.
5. Valikovskaya M.I., Pushkashu B.M., Maronchuk E.E. Plasticity and Brittleness of Semiconductor Materials at Microhardness Tests, Stiinta, Chishinau, 1984.
6. Boyarskaya Yu.S., Grabco D.Z., Kats M.S. Physics of the Microindentation Processes, Shtiinta, Chishinau, 1986.
7. Boyarskaya Yu.S., Grabco D.Z., Medinskii M.I., Pishkova D.S. Microhardness Anisotropy of Ionic Crystals with the NaCl lattice, Preprint, Chishinau, 1988.
8. Hirsch P.B., Pirouz P., Roberts S.G. and Warren P.D., Phil. Mag., B, vol. 52, 3, 759 (1985).
9. Aerts E., Amelinckx S. and Dekeyser W. Acta Met., vol.7, 1, 29 (1959).
10. Rickerby D.G., Amer.Ceram.Soc., vol. 62, 34, 229 (1979).
11. Geberteau F., Dominigues_Rodriguez A., Marquez R., Castaing J., Revue Phys.Appl., vol. 17, 777 (1982).
12. Daniels F.W., Dunn C.G. Trans. Amer. Soc. Metals, vol. 41, 419 (1949).
13. Arnell R.D., Appl. Phys., vol. 7, 9, 1225 (1974).
14. Brookes C.A., O'Neill J.B., Redfern B.A., Proc. Roy. Soc.Lond., vol. 322, 1548, 73 (1971).
15. Sawyer G.R., sergent P.M., Page T.F., J. Mater. Sci., vol. 15, 4, 1001 (1980).
16. Papirov I.I., Kapcherin A.C., FMM, vol. 51, 3, 625 (1981).
17. Button T.W., McColm I.J., Wilson S.J., J. Mater. Sci., vol. 14, 1, 159 (1979).
18. Sosnina E.I., Balan V.Z., Metal Physics, vol. 4, 1, 87 (1982).
19. Brookes C.A., Hooper R.M., Lambert W.A., Phil. Mag. A, 1983, vol. 47, 5, L9 (1983).
20. Hannink R.H.J., Kohlstedt D.L., Murray M.J. Proc. Roy.Soc. Lond. A, vol. 326, 409 (1972).