Dedicated to the memory of Prof. Yu.S. Boyarskaya,
the founder and first Head of Laboratory of Mechanical Properties of Materials of IAP ASM,
the founder of this scientific direction in Moldova

DEVELOPMENT OF SCIENTIFIC DIRECTION OF STRENGTH AND PLASTICITY PHYSICS IN MOLDOVA

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Laboratory of Mechanical Properties of Crystals (initially as a Scientific Group, later as a Laboratory) was founded in 1963. Since 2003 Laboratory has a new title: Laboratory of Mechanical Properties of Materials (LMPM). The famous scientist, Professor Yulia S. Boyarskaya (1928-1996) was the founder and the first Head of Laboratory during 33 years. Since 1996 the Laboratory has been headed by Dr. Sci. (Dr. Hab.) D.Z. Grabco. During the whole period (45 years) Laboratory became one of the scientific centers where all-round investigations of the problems of strength and plasticity, of the defect structures and mechanical properties of the crystalline and non-crystalline materials are carried out. The study of a wide range of materials let us to accumulate a persistent experience in the Laboratory. Among the investigated materials, one can mention bulk crystals with ionic, covalent, and mixed types of bond; metals and alloys; crystalline and amorphous semiconductors; thin semimetals and semiconductor wires; composite structures; high-temperature superconductors; construction materials; minerals and rocks, etc.

At present, the fundamental investigation of strength, plasticity, and brittleness of the crystals and other materials, with a view to understand the physical nature of deformation and destruction, is one of the main objectives in the Laboratory activity. The detection of correlation between mechanical and other physical properties, the study of influence of different external factors (irradiation, coating, doping, temperature, pressure) on these properties' modification is another very important direction of the scientific researches. It was shown that microindentation is one of more convenient and promising nondestructive methods that permits defining of a variety of physical characteristics of solids: microhardness, determined by static indentation (H), scratch microhardness (H_s), hardness anisotropy, microbrittleness (γ), microdurability (δ), toughness coefficient (k_{lc}), dislocation mobility (l_e, l_s), etc.

The various methods are used for researches: metallography, transmission and scanning electron-microscopy, atomic force microscopy, microcathodoluminescence, EDX analysis, etch-pit technique combined with optical method for the fine crystalline structure investigation, registration of the acoustic emission signals for the material brittleness estimation, etc.

Investigations carried out in the Laboratory had an appreciable contribution to the solution of many problems of materials science, especially in the domain of physics of strength and plasticity. On the basis of the obtained results 4 monographs, 2 monograph collections, 2 popular science books, 7 preprints, and more than 450 scientific papers have been published. The obtained results have been presented at more than 300 international scientific forums.

Note that all these successes have been achieved due to a great, careful, daily work of the Laboratory staff, enthusiasts and curious researchers, which every day, from year to year worked on a field of the selected scientific direction. Prof. Yu.S. Boyarskaya founded this scientific domain in Moldova in 1955 initially working as a lecturer of the theoretical physics chair at the Kishinev (Chisinau) State University. A new and main stage of development of strength and plasticity physics in our country began simultaneously with the opening of the Academy of Sciences in 1961, when Prof. Yu.S. Boyarskaya moved from the CSU to the ASM. This fact allowed her to study more profoundly the physics of solid deformation taking into account the dislocation evolution and using modern experimental methods, such as optic and electron microscopy, acoustic emission, etch-pit technique, and many others.

At the beginning the scientific group headed by Yu. Boyarskaya was included in the Laboratory for Physical Methods of Condensed Matter Investigation (headed by academician T.I. Malinovskii) of the former Institute of Physics and Mathematics of the ASM. Since 1964, when the Institute of Applied Physics was organized, the Group of Mechanical Properties of Crystals continued to function as a separate unit. Yu.S.Boyarskaya and her first collaborators M.I. Valkovskaya, R.P. Jitaru and D.Z. Grabco applied various methods for the scientific investigations such as polarization optical method, interferometry methods, and selective chemical treatment for the dislocation structures revelation, X-ray method and, in future, the transmission and scanning electron microscopy.

The complexity of studied phenomena is determined by different contribution of the material elasticity, plasticity, and fragility. The main elements of the plastic deformation are the linear defects of the crystalline lattice called dislocations. They can be of edge and screw types and can form the dislocation agglomerations and various structures during loading/unloading process. The point defects are another lattice irregularity, such as vacancies, interstitial atoms, impurities or their crowds, clusters and precipitates. The dislocations can interact with them diminishing the quantity or generate new lattice point defects. In addition to the above mentioned distortions, the kink domains and disclinations, micropores or microcracks can exist in crystals as well. All of them significantly influence the mechanical properties of solids. Therefore, the investigation of the real structure of materials is a very important problem. Along with this, the study of influence of different factors on the mechanical properties of crystals is of great importance for understanding of the physics of indentation process.

The scientific researches of Dr. M.I. Valkovskaya, Dr. Hab. R.P. Jitaru, Dr. Hab. D.Z. Grabco, and Dr. M.S. Kats, side by side with Prof. Yu.S. Boyarskaya and under her guidance, played a vital role in the further development and deeper understanding of many important and urgent problems of strength and plasticity physics. A number of new physical phenomena were revealed and explained. Among them, the elastic recovery of the indentations after unloading (Fig. 1), the polarity of plastic deformation of the material with different type of chemical bond (Fig. 2), the reverse plastic deformation, the mobility of dislocations in the field of the non-homogenous strains, the interstitial mechanism of plastic deformation can be mentioned. The influence of the impurity concentration on the mechanical properties of crystals was studied; modifications of the plastic and frail characteristics of crystals under action of X-ray and γ-irradiation (Fig. 3) were established. As a result of these large and profound investigations, the fundamental monograph "The Deformation of Crystals under Microhardness Testing" was published by Yu.S. Boyarskaya in 1972.

The main regularities of plasticity and brittleness of various ionic, ionic-covalent, covalent crystals, semiconductors, and semimetals were revealed. The models of material plastic flow for these crystals were proposed. The correlation between parameters of plastic deformation under uniaxial loading and concentrated load action was established. Two other monographs with title "Mechanical Properties of Bismuth Type Semimetals" (Authors: D.Z. Grabco, Yu.S. Boyarskaya, and M.P. Dyntu) (1982) and "Plasticity and Brittleness of

Semiconductor Materials under Microhardness Testing” (Authors: M.I. Valkovskaya, B.M. Pushkash, and E.E. Maronchiuk) (1984) were published. In addition to these two scientific collections, seven preprints and two popular brochures were published over the period.

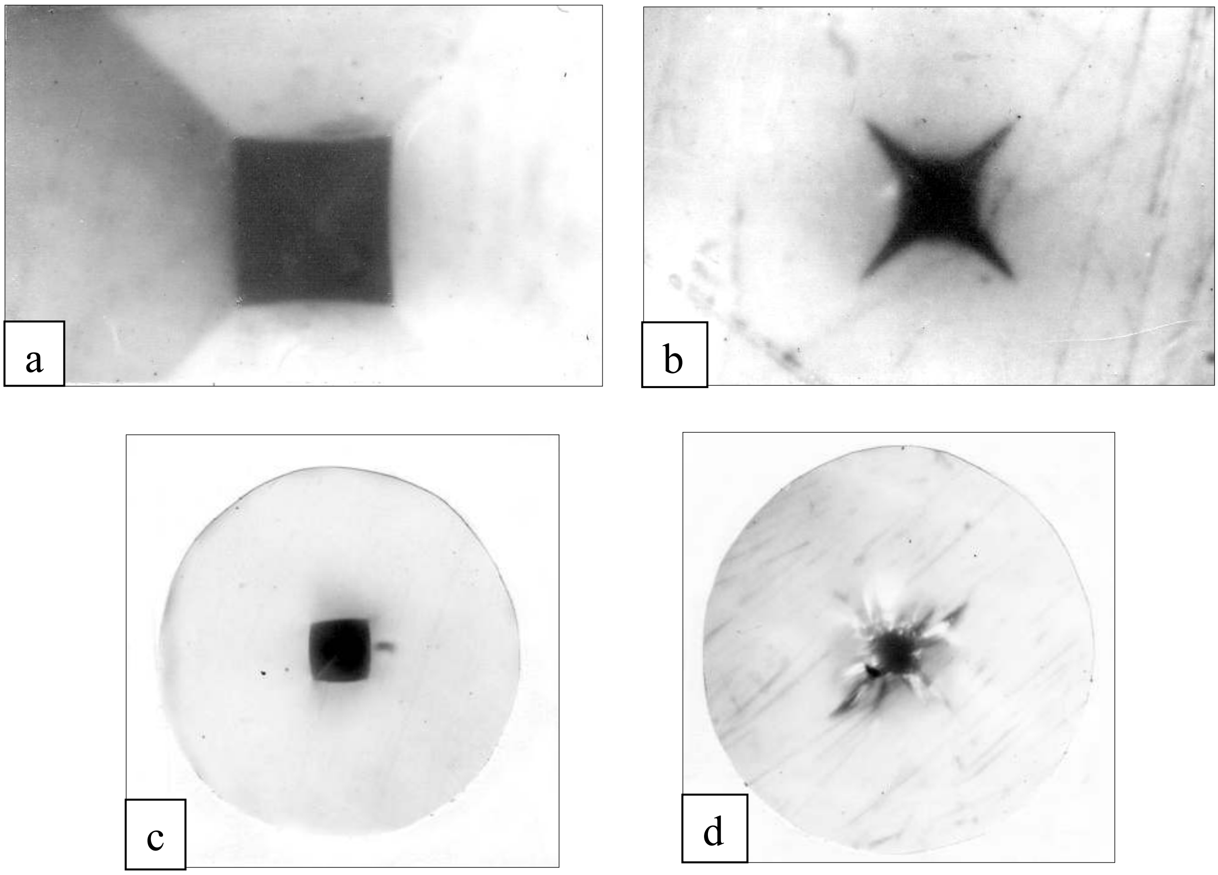
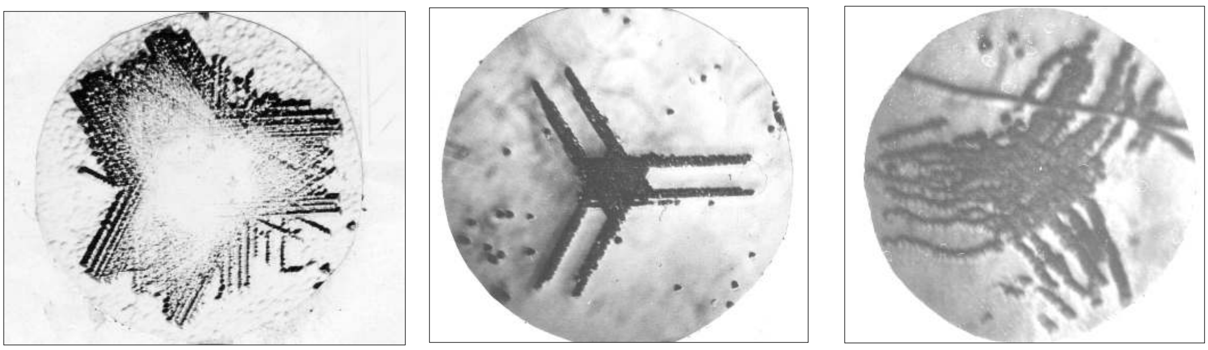


Figure 1. “In situ” examination of the elastic recovery phenomenon during unloading process under microindentation. a, b – plexiglass; c, d – lead glass. a, c – under loading; b, d – after indenter removal.x500 [1, 2].



a b c

Figure 2. NaCl. Polarity of plastic deformation detected in the bulk of crystals under indentations made at different temperatures. T, K: a- 300; b – 77; c – 500. x500 [1, 3].

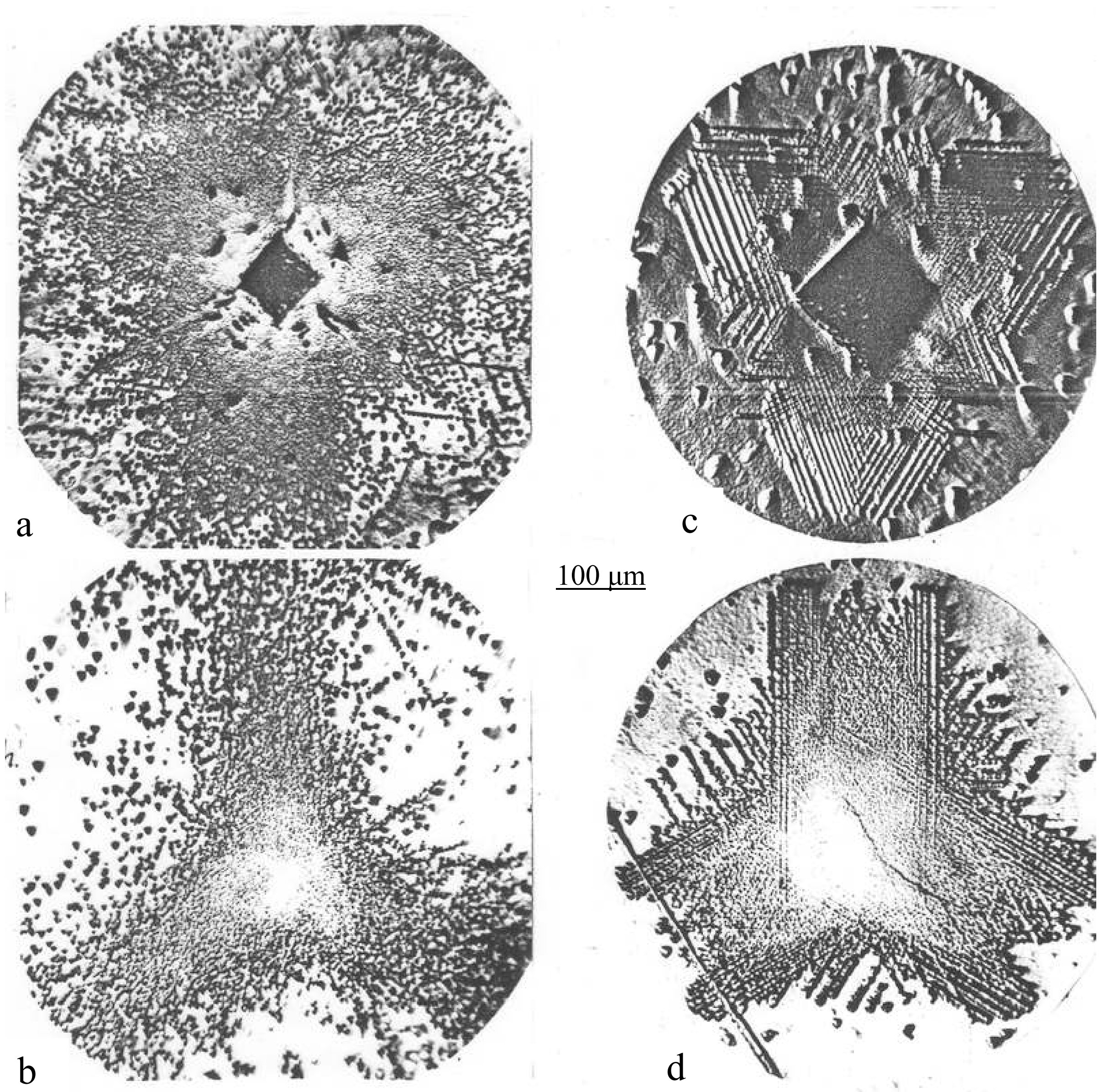


Figure 3. The influence of X-ray irradiation on the modification of the shape of dislocation zones around indentations made on the NaCl crystals. a, b – before irradiation; c, d – after irradiation; $t_{irr.}$=42 h [1, 3].

The efficiency of the microindentation method was enlarged studying the kinetics of the microindentation process. It was established that the deformation temperature of the sample plays an important role in determination of rate of the indenter penetration into material. The dependence revealed experimentally is characteristic of the thermofluctuation processes with the activation energy linearly decreasing with increase of the applied load. The fragility of materials was investigated using the acoustic emission (AE) arising during the microindentation and sclerometric testing, when the process is accompanied by the crack formation and material destruction. A special equipment for the material brittleness estimation using the registration of AE signals was designed and manufactured.

The formation of the Moldavian scientific school in field of the physics of strength and plasticity, which was recognized far outside the country, dates from that period of time. The

Laboratory body increased due to the new collaborators, researchers, and post-graduate students (doctorands). Later M.P. Dyntu, M.A. Linte, E.E. Maronciuk, D.S. Pishkova, S.S. Shutova, and N.A. Palistrant became candidates (doctors) of physics and mathematics, whereas D.Z. Grabco and R.P. Jitaru became Dr. Sci. (Doctor Habilitat) in physics and mathematics. The invaluable contribution to the Laboratory's achievements was made by very fine experiments of engineers E.I. Purich, and B.M. Pushkash, and M.I.Medinskaya.

The close scientific cooperation was established with well-known scientists from Moscow, S.-Petersburg, Kiev, Kharkov, Riga, Halle, etc. They are: Prof. V. Indenbom, Prof. V. Rozhanskii, Prof. A. Urusovskaya, Prof. V. Regel, Prof. B. Smirnov, Prof. O. Kleavin, Prof. R. Garber, Prof. M. Shaskoliskaya, Prof. P. Grau, and many other scientists.

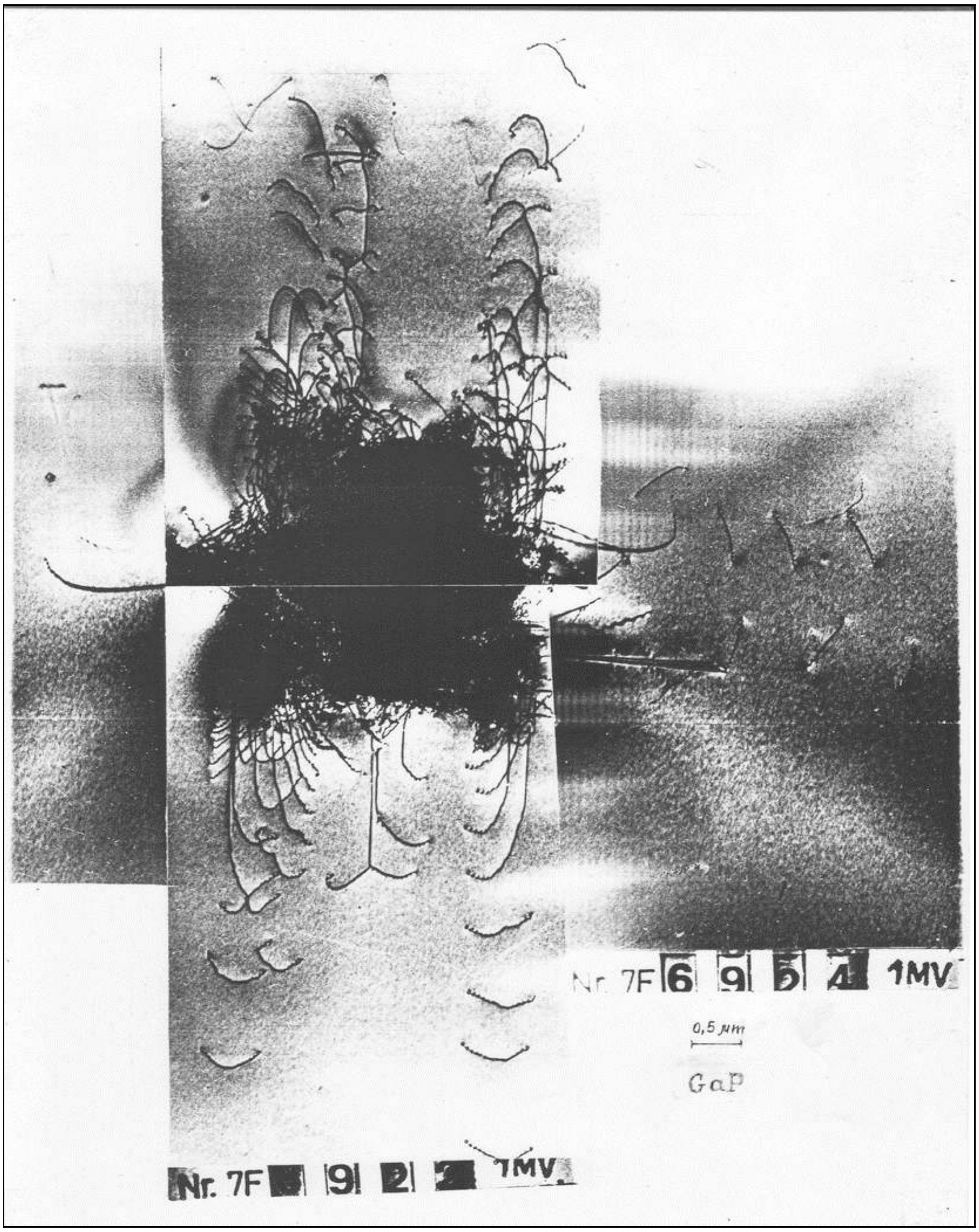


Figure 4. Transmission electron microscopy. The dislocation rosette around indentation made on the (001) plane of GaP single crystals at elevated temperature (T = 600 K) [4].

The knowledge in the theory of elasticity, plasticity and physics of dislocations permitted Yu.S. Boyarskaya to formulate new ideas explaining the numerous experimental data accumulated in the Laboratory. These ideas allowed understanding of the physical nature of the polar anisotropy of the indentations and scratches traced on the octahedral faces of the NaCl, PbS, and CaF_2 single crystals. Another achievement was the concept of the reverse deformation, when the pressure from the indenter is directed inside the sample, whereas the forced out material moves in opposite direction (outside the sample). On this base, models of material plastic flow during the indentation process in ionic and covalent crystals were proposed.

According to these models, the active slip planes can be divided into two groups. So, in the case of the NaCl type crystals, one group consists of the divergent slip planes, forming the tetrahedral pyramids with the apex near the surface and with the base at the depth. The slip planes of the second group form the inverted pyramid. The first plane group is responsible for the material shift to depth of the sample, whereas the latter one accounts for its transport to the surface and for the formation of the pile-ups around indentation.

The selective chemical treatment and polishing of the deformed crystals combined with optical microscopy, scanning and transmission electron microscopy researches (Fig. 4), micro-cathodoluminescence (Fig. 5), and the decoration technique (Fig. 6) allow detecting the real dislocation distribution under indentation in the bulk of crystals. They confirmed the conclusion advanced for various deformation planes and the correctness of the models of plastic flow proposed for different materials: ionic and covalent crystals, crystals with mixed type of bond, semiconductors and semimetals, in the wide range of deformation temperatures (77-800 K).

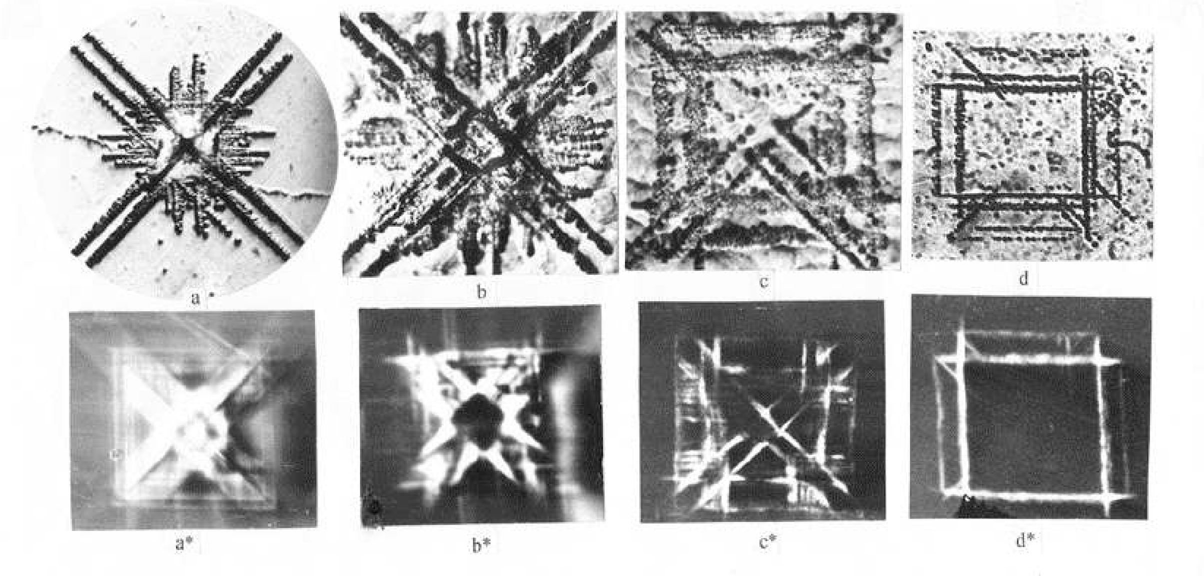


Figure 5. MgO. The change of dislocation rosette (a-d) and cathodoluminescence images (a^*-d^*) near the indentation under consequent polishing; on the initial surface - a, a^*; on the depth, μm: b, b^* - 15; c, c^* -45; d, d^* -60.

An installation comprising the metallography microscope and the loading device, proposed and manufactured earlier in the Laboratory, permitted the observation “in situ” of the indentation and its surrounding deformed region as well as the failure pattern under the load and after unloading. Using this installation, the reverse or recovery elastic/plastic deformation was revealed after the indenter removal. Moreover, it became possible to reveal the occurrence and evolution of the dislocation structures around indentation under the load and after unloading using the chemical methods (Fig. 5). It was proved that the additional mass transport to the surface takes place during unloading process. A new unknown phenomenon was observed and published in the paper by Yu.S. Boyarskaya, D.Z. Grabco, and N.A. Lavrovskaya, *Fiz. Tverd. Tela (Physics of Solids)*, 1992, v. 34, p.2292. Later, this effect was called the prolonged plastic deformation (Yu. S. Boyarskaya, R.P. Jitaru, D.Z. Grabco, and V.A. Rakhvalov. The Prolonged Plastic Deformation Related with the Microindentation of MgO Single Crystals. *Journal of Mat. Sci.*, 1998, v.3, pp.281-285).

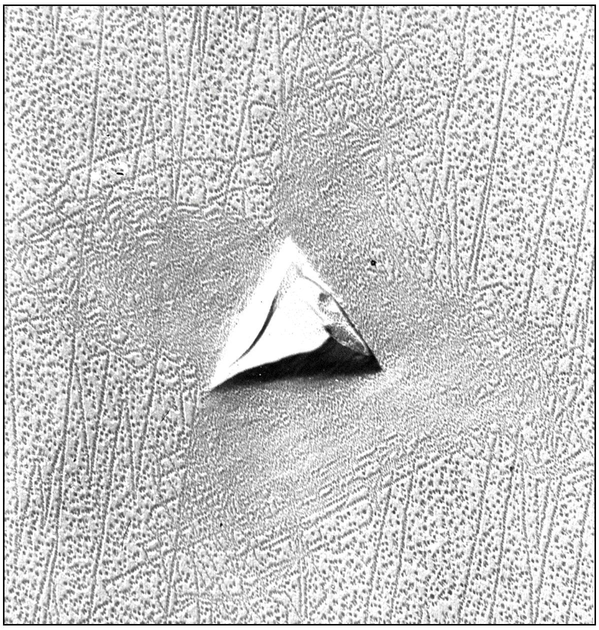


Figure 6. The dislocation rosette detected around the indentation made on the (001) plane of NaCl single crystal using the Au decoration technique [5].

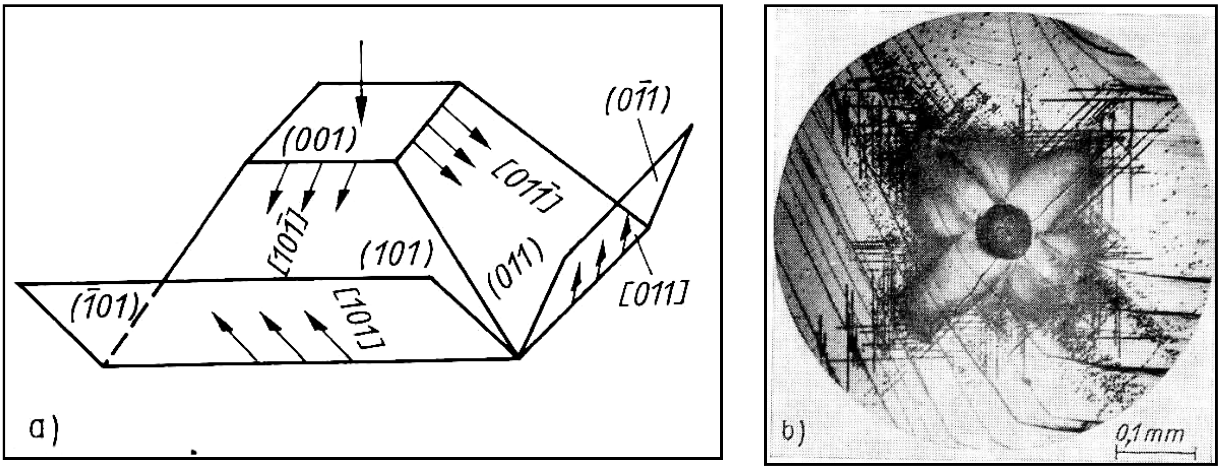


Figure 7. a - Model of plastic flow in indentation on the (001) plane of alkali halide crystals, such as NaCl. B – Dislocation rosette formed by indentation of steel cone on the (001) plane of LiF [1].

The accumulated experimental data allowed putting forward a series of new hypotheses concerning physics of deformation process under action of point loading on solids. They are the *smooth and impulse mechanisms of plastic deformation* and *the pseudo-mobility of dislocations*. These hypotheses were described in some publications, and in the fundamental monograph: Yu.S. Boyarskaya, D.Z. Grabco, and M.S.Kats “Physics of microindentation processes”, *Ed. “Shtiintsa”,* 1986, 283 p. In accordance with smooth mechanism, the formation of both the indentation and dislocation structure (called the dislocation rosette) takes place simultaneously. In the case of impulse mechanism, this synchronism is disturbed when the deformation of hard materials takes place in the range of room temperature or deformation of soft crystals occurs at low temperature (77 K<$T_{def.}$< RT).

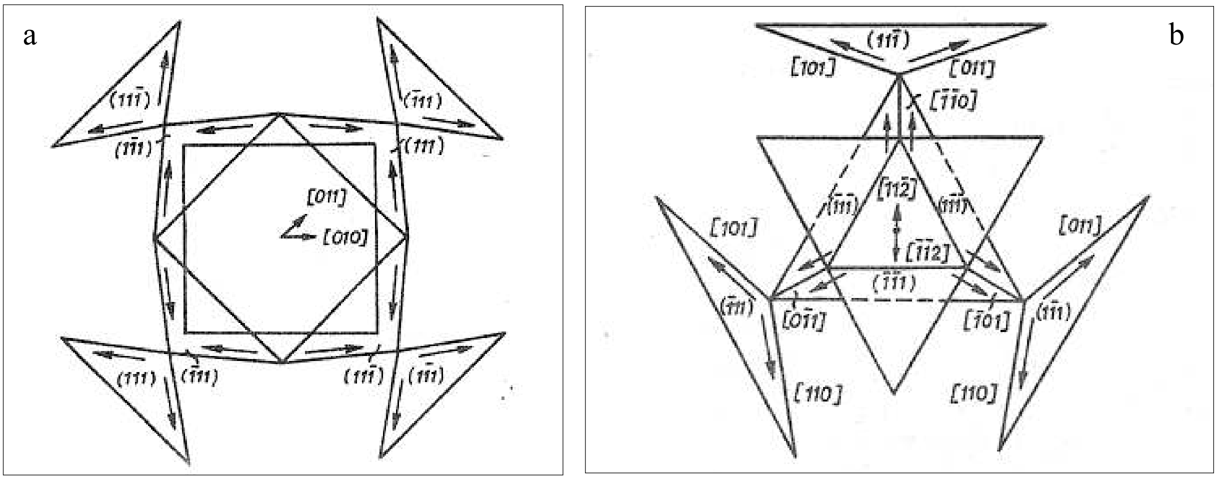
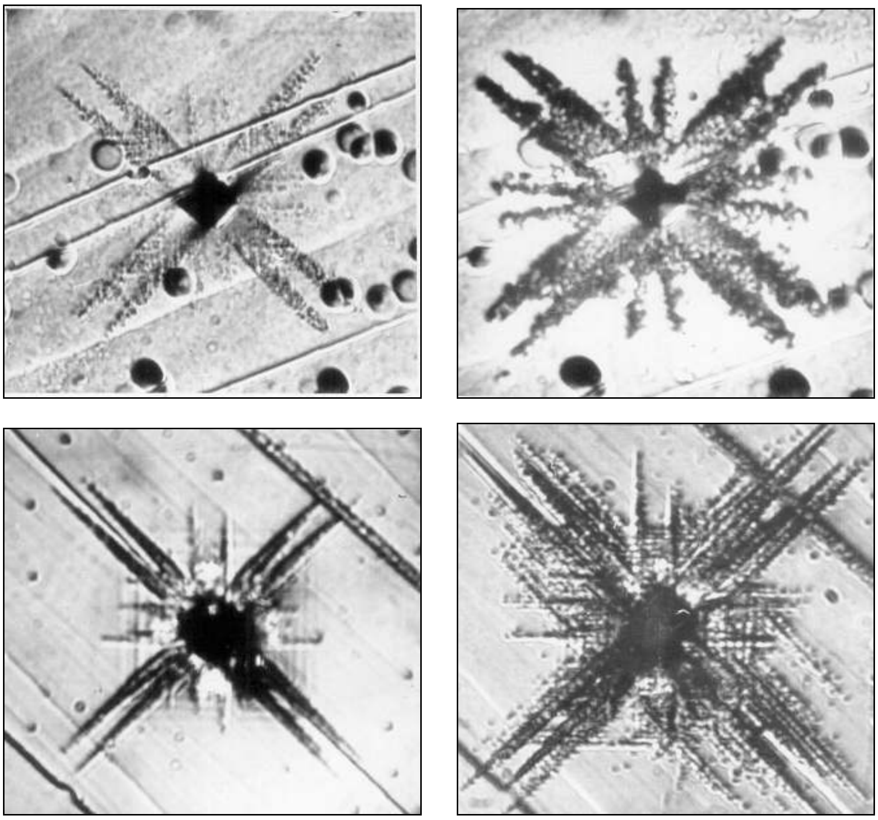


Figure 8. A possible model of plastic flow in crystals with the {111}<110> sliding systems under sclerometric testing when indenter moves along the direction of maximal and minimal hardness. a – plane (100); b – plane (111) [6].

a

b



c

d

Figure 9. Prolonged plastic deformation. Dislocation rosettes near the indentations on the plane (001) of LiF (a, b) and MgO (c, d) crystals made by the indentor: a, c - under loading and b, d - after unloading. P, N: a, b – 1.0; c, d – 3.0 [7, 8].

A number of regularities contributing to solution of many urgent problems of physics of strength and plasticity were established in the ensuing years. These are some of them:

- determination of the diverse character of four types of material hardening: radiation, impurity, lattice and temperature hardening;
- detection of the existence of quasidestructured zone near the indentation; this zone is the basic responsible for the mass transport during the indentation formation (Figs. 10, 11);
- detection of the interconnection of cathodoluminescence and dislocation rosettes appearing around indentation in the middle temperature region and revelation of their modification regularities in the region of low and high temperatures;
- estimation of contribution of the dislocation, disclination and interstitial mechanisms of deformation during microindentation process;
- detection and elucidation of series of the anomalous phenomena taking place due to deformation of the doped and irradiated crystals, etc.

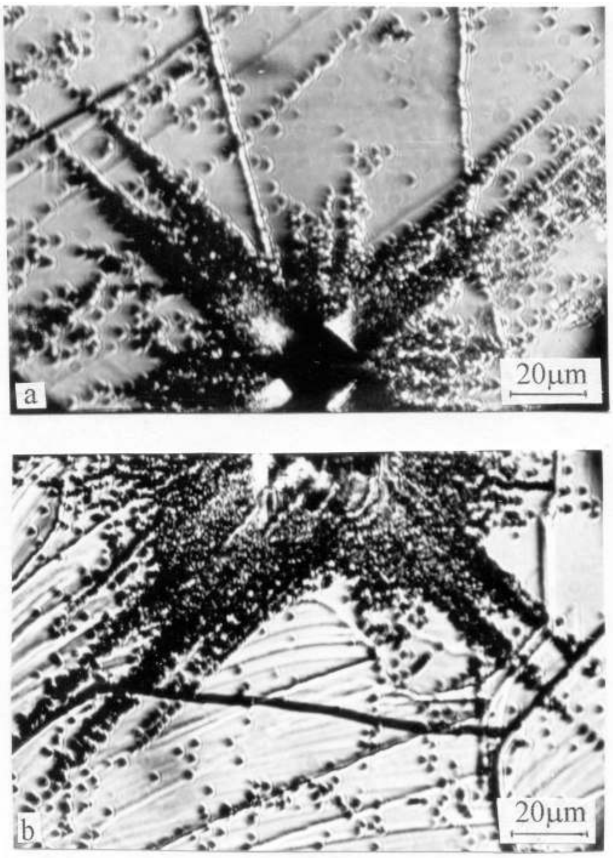


Figure 10. The NaF (001) surface. T_{def}=130K; P=1.0 N .a- the dislocation rosette near the indentation on the deformed surface; b- the deformed zone on the lateral (100) plane under indentation [9, 10].

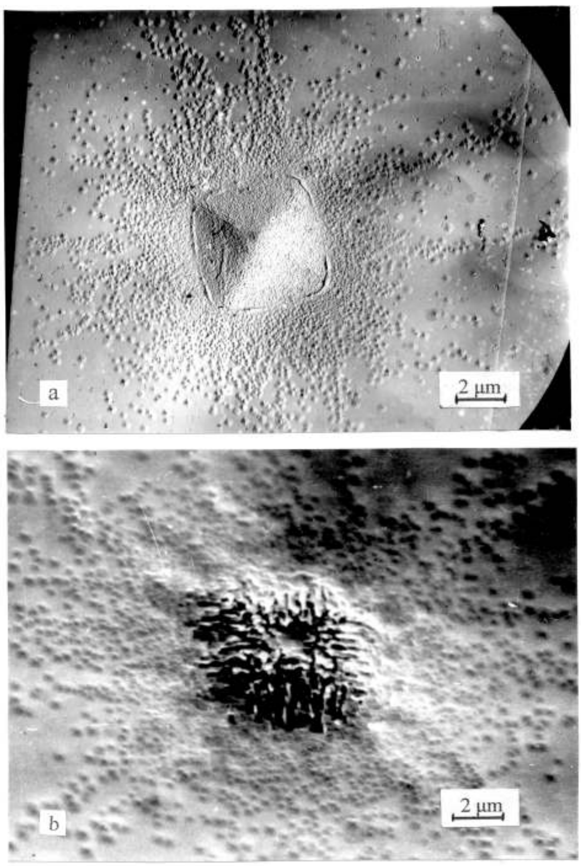


Figure 11. The LiF (001) plane. P=0.05N, T_{def}=293 K; a - the dislocation rosette near the indentation on the deformed surface; b- the quasidestructured zone appearing at certain depth (~5 μm) under indentation after chemical polishing and etching [9, 10].

To improve and extend the tools and methods for the investigation of mechanical properties of materials by microindentation, new devices have been worked out in the laboratory, such as:

– an apparatus for study of material brittleness by registration of the acoustic emission signals;
– a device for the metallographic microscope to study the elastic recovery;
– a device for the microhardness tester to study "in situ" the microdeformation processes;
– a device for microhardness tester to deform crystals and other materials in the large temperature interval (77-900 K), etc.

The behavior of solids under action of the external load is a key problem in modern materials science and technology. Plasticity and brittleness of materials, their ability to accumulate and relax stresses are in many cases the properties, which determine the lifetime cycle of goods, various devices, apparatus, and constructions.

A lot of experimental data enriching our knowledge of the fundamental problems connected with physics of indentation processes has been obtained by the collaborators of Laboratory of Mechanical Properties of Materials after 1996. These results represent a regular continuation of the researches started under the guidance of Prof. Yulia S. Boyarskaya.

We note only some of them. The revelation of the temperature shift of the deformation peculiarities under indentation is one of the important results obtained in recent years. The temperature shift phenomenon consists in the similar plastic, brittle, and other mechanical properties observed in the ionic and covalent crystals in different temperature intervals. Thus, physic-mechanical properties (such as anisotropy coefficient, brittleness, plasticity, dislocation mobility) typical for soft crystals at low temperatures are characteristic of hard crystals at high temperatures (see the article: D. Grabco, B. Pushcash, M. Dyntu, and O. Shikimaka "Thermal Evolution of Deformed Zones around Microindentations in Different Types of Crystals", Phil. Mag. A, 2002, v.82, Nr.10, pp. 2207-2215).

The existence of five structural levels of the elastic-plastic deformation at point load action has been detected. There are: (i) superdefect layer having a nano-dimensional thickness, the nearest to the indent; (ii) quasidestructured (nanostructured) zone where the greatest plastic flow takes place. The most complex dislocation reactions with creation of dislocation walls and disclination structures occur in this zone; (iii) region with great dislocation density; (iv) peripheral dislocation zone; and (v) region of the elastic deformation. The partial or complete realization of such deformation shape is a function of the many factors: deformation temperature, type of crystal lattice and chemical bond, loading conditions, etc. (see the article: Grabco D.Z., Lavrovskaya N.A., and M.I. Medinskaya. "Regularities of Deformation and Destruction under Local Loading of Ionic Crystals", Latvian Journal of Phys. & Techn. Sci., 1991, Nr. 5, pp.72-80 (in Russian)).

The dislocation-disclination-interstitial concept (DDIC) of the deformation mechanism under concentrated load action was formulated and confirmed on base of the numerous experimental data (see the article: D.Grabco. "Dislocation-Disclination Mechanism of Deformation under Microindentation", Mold. Journ. Phys. Sci, 2002, v.1, Nr.3, pp. 94-103). This concept represents a further development of understanding of the smooth and impulse mechanism of deformation, forwarded more than twenty years ago by Prof. Yu. Boyarskaya in the paper: About the Nature of Temperature Hardening of Ionic Crystals with NaCl Lattice (authors Yu.S. Boyarskaya, D.Z. Grabco, and D.S. Pishkova), Cryst. Res. Technol., 1985, 20, 11, K113-K114.

Taking into account requirements of the reality, the range of studied materials and applied methods has been enlarged in the Laboratory of Mechanical Properties of Materials of late years. Many new materials being of concrete engineering application are under investigation at present along with the classical crystals studied earlier. They are films and low-dimensional materials on the base of semiconductors and polymers, composite and multilayer structures, microcrystalline and amorphous materials, metals and alloys, etc.

The nanoindentation method, alongside with microindentation one, is used for research of such materials. The results obtained by the nanoindentation method, on the one hand, allow us to study the mechanical properties of objects dimensional reduced; on the other hand, they contribute to more profound understanding of physical processes which occur in solids under the local inhomogeneous stress action.

Note that the deformation mechanism is the most extensively studied by the microindentation method. However, the specific character of the nanoindentation method has some specific features as compared with the microindentation one: Berkovich indenter, a trihedral diamond pyramid instead of a tetrahedral Vickers one in the microindentation method, significantly smaller depth of nanoindentations in contrast to microindentations, etc. In this connection, there is a question, in what measure the regularities of microindentation processes are valid for nanoindentation one.

To elucidate this question, the examination of the topography of nanoindentations along with the features of the relief around them is carried out using a set of modern methods: atomic force microscopy, optical and scanning electron microscopy, etch-pit techniques, etc. Simultaneously, the comparison with deformation specificity under microindentation is carried out.

Laboratory of Mechanical Properties of Materials continues the collaboration with different scientific Centers of our Institute, studying the microstructure and mechanical properties of crystals and compounds obtained there. The international scientific cooperation takes place with different scientific groups from other countries: UK (Cavendish Laboratory, Cambridge); Germany (Karlsruhe University); Hungary (Research Institute for Technical Physics and Materials Science, Hungarian Academy of Sciences, Budapest), Latvia (Institute of Solid State Physics, University of Latvia, Riga), Russia (Institute of Problems in Mechanics of RAS, Moscow; Ioffe Phys.-Technical Institute of RAS, S.-Petersburg); Ukraine (Institute of Superhard Materials, Kiev); Belarus (Institute of Applied Physics, Minsk, Institute of Technical Acoustics, Vitebsk), etc.

A great attention is paid to both the raising of the level of personnel skill and science educational work with young specialists in the laboratory. The new generation of doctors in physics (dr. N. Palistrant, dr. O. Shikimaka) and post-graduate students work in the Laboratory.

The Laboratory of Mechanical Properties of Materials is appreciated now as before, as a scientific center where the multilateral investigations of durability, plasticity, and brittleness are performed, and where the microstructural defects and mechanical properties of a very large series of solids are studied profoundly. To note, LMPM is unique Laboratory in the country of such profile. The researchers from different scientific groups have access for both the professional consulting and various mechanical parameter testing.

Main directions of activity of LMPM at present are the following:

- Mechanical properties (microhardness, yield stress, microbrittleness, microdurability, hardening, softening, defect structure, etc.) of new and already existing materials; correlation between mechanical properties and other physical ones;
- Influence of different external and internal factors (irradiation, impurities, temperature, load, chemical bond, dislocation structures, etc.) on the mechanical properties;
- Mechanism of the deformation and destruction of crystals and other materials (low-dimensional materials, planar structures, wires, polymers and amorphous materials, metals and alloys, minerals and rocks, etc.) under local load action.
- Application of the microindentation method for studying the different physical parameters and phenomena: hardness anisotropy, fracture anisotropy, mass transport, twinning, dislocation mobility, dislocation-disclination deformation mechanism, etc.

In addition, the "LABMET" Testing Laboratory (TL) accredited for the mechanical testing on the ISO 17-025-2006 level by the Accreditation Centre in the Domain of Evaluation of Production Conformity is acting in frame of the LMPM since 2000.

Many other achievements along with above mentioned could be mentioned. However, even this brief description gives evidence of a high scientific potential of the Laboratory's collective. No doubt, one can expect that in the future a lot of new important scientific results will be obtained in the Laboratory of Mechanical Properties of Materials, which will contribute to further development of one of the major domains of the physical science, namely, physics of strength and plasticity.

Both the former important scientific results accumulated in LMPM and new researches which will be realized in Laboratory of Mechanical Properties of Materials in the future are *the best memory to activity of a famous scientist and a noble person which was Professor Yulia Boyarskaya.*

Some recent publications of the LMPM collaborators:

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