MECHANISM OF PLASTIC DEFORMATION OF CRYSTALS UNDER MICROINDENTATION

Yu. S. Boyarskaya

This article was prepared by R. P. Jitaru, N. A. Palistrant, V. A. Rahvalov, O. A. Shikimaka

Institute of Applied Physics of Moldavian Academy of Sciences, 5 Academy str.,
MD-2028 Chisinau, Moldova

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-works.

Abstract

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, have been established. The development (occurrence) of the certain indentation mechanism is depended on the structure lattice and deformation temperature too.

1. Introduction

Many phenomena observed 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.

Low-temperature anomaly of dislocation mobility in the stress field of a concentrated load

Ionic crystals

Unusual behaviour of the dislocation ensembles at microindentation in the beginning was revealed on ionic crystals with a lattice of NaCl type [41]. The investigations were carried out on (001) plane of the KCl, NaCl, NaF, LiF and MgO alkali-halide crystals. Deformation was performed by means microhardness tester PMT-3 at 77 and 293K. The dislocation rosettes around the indentations were revealed by selective etching. The composition of etchants is given in [41]. The parameters \( l \) and \( \gamma = l / d \) as well as \( \alpha = l / P^{1/2} \) were used as dislocation mobility characteristics. Here \( l \) is the maximal removal of dislocations from indentations and \( d \) is the indentation diameter, \( P \) is the indentor load.
The errors of measurements for \( H \) and \( l \) were equal to 3 and 15\%, respectively. As the \( l \) measurements were carried out at room temperature, the influence of heating under change of the temperature from 77 to 293K on this parameter was considered [41]. It was established that the arm length of dislocation rosette practically doesn’t change at a warm-up. This result was confirmed by experiments of crystal etching at low temperature. The results, characterizing the changes of microhardness and edge dislocation mobility by indentation temperature lowering from 293 to 77K, are given in Table 1.

Table 1. The changes of microhardness and edge dislocation mobility at temperature lowering

<table>
<thead>
<tr>
<th>Crystal</th>
<th>( H, \text{kg/mm}^2 )</th>
<th>( H_{77}/H_{293} )</th>
<th>( l, \mu\text{m} )</th>
<th>( l_{77}/l_{293} )</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>293K</td>
<td>77K</td>
<td></td>
<td>293K</td>
</tr>
<tr>
<td>KCl</td>
<td>10</td>
<td>35</td>
<td>3,5</td>
<td>95</td>
</tr>
<tr>
<td>NaCl</td>
<td>20</td>
<td>82</td>
<td>4,1</td>
<td>86</td>
</tr>
<tr>
<td>NaF</td>
<td>67</td>
<td>200</td>
<td>3,1</td>
<td>112</td>
</tr>
<tr>
<td>LiF</td>
<td>120</td>
<td>240</td>
<td>2,0</td>
<td>113</td>
</tr>
<tr>
<td>MgO</td>
<td>720</td>
<td>1200</td>
<td>1,6</td>
<td>64</td>
</tr>
</tbody>
</table>

As it is seen the temperature lowering results in the significant rise of microhardness for all crystals under investigation. The dislocation mobility remains practically unchanged or even increases. Such anomaly behaviour is especially pronounced for NaCl crystals. The rise of the microhardness by 4 times (from 20 to 80 kg/mm\(^2\)) is followed by the increase of dislocation mobility by 1,6 times. In the case of point defect hardening the \( H \) small rise (from 19.3 to 24.4 kg/mm\(^2\)) is accompanied by \( l \) decreasing (from 103 to 43\( \mu \)m).

It follows from Tabl. 1 that the temperature hardening dependences on microhardness of crystals are decreasing with microhardness increasing (KCl crystals is exception). The sharp infringement of these parameters in the wider interval of temperatures (77-423K) have shown the following [43, 44, 45, 66]. The dislocation mobility behaviour in the region of low temperatures (T<293K) differs from the one in the region above 293K. This is especially clear for the screw dislocations (Fig.1), \( \alpha_\text{s} \) is constant in the range of 77-293K and a sharp \( \alpha_\text{s} \) rise exists for T>293K. A critical temperature (\( T_\text{cr} \)) corresponding to the \( \alpha_\text{s} (T) \) curve bend is approximately equal to 0.3 \( T_\text{mel} \) (melting temperature) for all the crystals examined.

\[ \alpha_\text{s} = \sqrt{\frac{\mu \gamma}{kT}} \]

Fig. 1. Temperature dependence of the screw dislocation ensemble mobility for crystals KCl (1); NaCl (2); NaF (3) and LiF (4)
The results presented in Fig. 2, allow to compare temperature dependences of the microhardness and dislocation mobility for NaCl crystals (analogous results were obtained for other crystals). It is seen that at elevated temperatures (T>293K), in contrast to low temperature region, the changes of dislocation mobility and microhardness are in the agreement; the H decreasing corresponds to $\alpha$ increasing. The $\alpha$ changes are more significant than H: the small microhardness changes (by two times) are followed by significant changes of dislocation mobility (by 13 times) in the region of high temperatures. Different situation takes place in the region of low temperatures: the H changes by 4 times, the dislocation mobility is practically constant (Fig.2).

![Fig. 2. Temperature dependence of the microhardness (1) and dislocation mobility (2, 3) [43]](image)

**Doped NaCl crystals**

Investigations were performed for NaCl crystals containing different type of impurities (Ca$^{2+}$, Pb$^{2+}$, Mg$^{2+}$, Mn$^{2+}$, Sr$^{2+}$) [42, 45, 69, 70]. First let us consider the results obtained for two types of impurities: Ca$^{2+}$ - well dissolving, hole-accepting impurity and Pb$^{2+}$ - poorly dissolving, electron-accepting impurity [52, 53]. The Ca$^{2+}$ doping results in a significant hardening of the crystals. The Pb$^{2+}$ doping leads to comparatively small changes of microhardness and yield stress. This is obviously connected with a different state of impurities in the crystals. This supposition was confirmed by the electron-microscopy investigation of decoration pictures of NaCl:Ca and NaCl:Pb crystals containing different impurity concentration [71].

The study of the dislocation mobility showed that the low-temperature anomaly of the dislocation mobility appears in doped NaCl crystals independently of the impurity type (Table 2). Table 2 shows that the significant rise of the microhardness of NaCl:Ca crystals, at lowering the temperature from 293 to 77 K is followed by the increase of the dislocation mobility (not by the decrease of this parameter). This effect is more distinct in NaCl:Pb crystals; a tendency towards the decrease $\gamma_e^{77}/\gamma_e^{293}=k$ appears at increasing of the Ca$^{2+}$ concentration. The conclusion that the type and the state of the impurity have an influence on the anomalous behaviour of the dislocation mobility at low temperatures seems to be true. However, a further investigation of this problem is needed.

**Table 2. Low-temperature anomaly of dislocation mobility in NaCl doped crystals**

<table>
<thead>
<tr>
<th>NaCl:Ca</th>
<th>NaCl:Pb</th>
</tr>
</thead>
<tbody>
<tr>
<td>C, mole %</td>
<td>H$^{77}$/H$^{293}$</td>
</tr>
<tr>
<td>Pure</td>
<td>3.3</td>
</tr>
<tr>
<td>$10^{-3}$</td>
<td>3.2</td>
</tr>
<tr>
<td>5$\times10^{-3}$</td>
<td>3.6</td>
</tr>
<tr>
<td>$9\times10^{-3}$</td>
<td>3.6</td>
</tr>
<tr>
<td>3.6$\times10^{-3}$</td>
<td>3.2</td>
</tr>
<tr>
<td>5.8$\times10^{-2}$</td>
<td>2.8</td>
</tr>
<tr>
<td>1.6$\times10^{-1}$</td>
<td>2.7</td>
</tr>
</tbody>
</table>
The experiments carried out in a much wider temperature region (77-423 K) confirmed that the low-temperature anomaly of the dislocation mobility is more distinct in NaCl:Pb crystals than in NaCl:Ca crystals. Fig. 3 shows the concentration effect on the dislocation mobility obtained at different temperatures. It is seen, that the $\gamma_e (C)$ curve for NaCl:Pb crystals obtained at 77 K, is placed above the curves corresponding to room and elevated temperatures (Fig. 3).

![Fig. 3. Concentration dependence of the edge dislocation ensemble mobility for NaCl:Pb crystals at different temperatures. T, K: 77 (1); 293 (2); 373 (3)](image)

Another result was revealed for NaCl:Ca crystals (Fig. 4): the dislocation mobility in the range of elevated temperatures exceeds the mobility at 77 K for some impurity concentrations. The dislocation mobilities, obtained at different temperatures, are close to each other in the region of the high impurity concentrations.

![Fig. 4. Temperature dependence of the yield stress $\sigma_y$ (1), microhardness (2) and screw dislocation ensemble mobility (3) for NaCl:Ca crystals. $C=9\times10^{-3}$ mole % .](image)

For the region of the elevated temperatures ($T > 293$ K) the rise of the temperature is followed by the dislocation mobility rise, i.e. an anomalous behaviour is absent, however, the $\gamma(T)$ curves have sometimes a non-monotonous character [42]. In Fig. 4 the results are presented for NaCl:Ca crystals containing constant impurity concentration (analogous data were obtained for the other concentrations). As shown, in the region of lower temperatures the rise of the yield stress and microhardness is not accompanied by the decrease of the dislocation mobility, but in the region of higher temperatures there is, in general terms, a good correlation between the changes of these parameters.
Low-temperature anomaly of dislocation mobility for crystals containing other two-valent cation impurities was observed too. For example in Fig.5 the results obtained for slightly hardened NaCl:Mn crystals are presented.

![Image](image1)

![Image](image2)

**Fig. 5. Dependence of microhardness (a) and edge dislocation ensemble mobility (b) on impurity concentration in solution for NaCl:Mn crystals.**

\( T_{def}, K: 77 (1); 180 (2) \) and 293 (3)

The dislocation mobility and microhardness dependence on the impurity concentration in solution are given. From Fig.5 it is seen that the microhardness increases regularly with temperature decrease at different impurity concentrations and is equal to 80 kg/mm\(^2\) at \( T=77K \). In this case the \( \gamma \) parameter characterizing the dislocation mobility increases by 3-4 times (Fig.5b), i.e. low-temperature dislocation mobility is more pronounced for these crystals than for NaCl:Ca and NaCl:Pb ones (Table 2).

The above-mentioned results demonstrate that type and the concentration of impurities in investigated crystals influence the anomalous dislocation behaviour at low temperatures. Therefore it is possible that impurity state affects this phenomenon too. For experimental check the investigation on NaCl:Ca and NaCl:Pb was performed. The impurity state varied by quenching, the quenching temperature (\( T_{q} \)) was changed in the region from 323 to 723K; at each \( T_{q} \) the crystals were kept 3 hours. The indentor load was equal to 10g.

![Image](image3)

![Image](image4)

**Fig. 6. The dependence of microhardness and parameter \( k \) on quenching temperature for (a) NaCl:Pb (2\( \times \)10\(^{-3}\) mol. %) and NaCl:Ca (b) (10\(^{-3}\) mol. %) 1, 2, 3 - \( H_{293}, H_{77}, k \), accordingly**

The obtained results show that the microhardness as well as the dislocation ensemble mobility were affected by the quenching (Fig.6 and Fig.7, accordingly). The pronounced low-temperature anomaly of the dislocation mobility at all \( T_{q} \) was also observed. For example, the results for NaCl:Ca (C=9\( \times \)10\(^{-3}\) mol %) are given in Fig.7b. One can see that the dislocation ensemble mobility at 77K is larger than at 293K for all \( T_{q} \), i.e. for the different impurity state. The anomalous behaviour was more
pronounced for NaCl:Ca. Fig. 7 a, b shows also that there is a good correlation between \( \gamma (T_q) \) dependences at two temperatures: 77 and 293K.

\[ \text{Fig. 7. Dependence of the edge dislocation ensemble mobility on quenching temperature for NaCl:Ca (a, b) and NaCl:Pb (c) crystals. C, mole\%: } 10^{-3} \text{ (a); } 9 \times 10^{-3} \text{ (b); } 2 \times 10^{-3} \text{ (c).} \]

However non-conformities were revealed between the shape of \( H (T_q) \) dependences at 293 and 77K for both impurities types having different concentrations and existing in various states, Fig. 6 (curves 1 and 2). The parameter \( k = \gamma_{77} / \gamma_{293} \) (\( \gamma_{77} \) and \( \gamma_{293} \) determine the dislocation mobility at 77 and 293K respectively) as a characteristic of the low-temperature anomaly of dislocation mobility is introduced. As it is seen from Fig. 6 some correlation between the shape of \( k (T_q) \) and \( H (T_q) \) dependences for NaCl:Ca and NaCl:Pb can be mentioned: \( k_{\min} \) corresponds, as a rule, to \( H_{\max} \).

The occurrence of the correlation between \( k \) parameters characterizing the degree of low-temperature anomaly of dislocation mobility at 77K and the microhardness confirms the correctness of the suggestion proposed earlier about the impulse mechanism of dislocation structure formation under indentation. These results are very important for the understanding of low-temperature dislocation mobility phenomenon.

**Bismuth type semimetals** [6, 72, 73]

The microhardness and dislocation mobility measurements on the (111) complete cleavage plane of these materials in the temperature interval from 77 to 600K were performed. For comparison the investigations on some ionic crystals on the same planes (NaCl, LiF, CaF\(_2\), MgO) were carried out as well. The results obtained in the low-temperature region are listed in Table 3.

One can see that for all investigated monocrystal semimetals the considerable increase of microhardness with temperature lowering from 293 to 77K is not practically followed by dislocation mobility decrease - \( \gamma \) is close to one. That means that for these materials the low-temperature dislocation mobility anomaly takes place in the stress field of concentrated load. However this phenomenon is more pronounced in the ionic crystals (except MgO crystals) (see Table 3).

The dislocation mobility anomalous behaviour in semimetals as well as in ionic crystals occurs only in the low-temperature region.

For example the data for antimony monocrystals (Fig. 8, a) show that at \( T = 293K \) the dislocation mobility starts to rise sharply and it is followed by microhardness decrease. The coordinated changes of these parameters in this range of temperature are observed for fluorite monocrystals as well (Fig. 8b).
Table 3. The microhardness and dislocation mobility of bismuth type semimetals and ionic crystals

<table>
<thead>
<tr>
<th>Crystals</th>
<th>P, g</th>
<th>T = 293K</th>
<th>T = 77K</th>
<th>H_{77}/H_{293}</th>
<th>\gamma_{77}/\gamma_{293}</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sb</td>
<td>20</td>
<td>58.0</td>
<td>130.0</td>
<td>2.2</td>
<td>0.96</td>
</tr>
<tr>
<td>BiSb_{4}</td>
<td>20</td>
<td>28.8</td>
<td>47.1</td>
<td>1.6</td>
<td>0.89</td>
</tr>
<tr>
<td>BiSb_{8}Sn_{0.01}</td>
<td>20</td>
<td>38.6</td>
<td>46.7</td>
<td>1.2</td>
<td>0.93</td>
</tr>
<tr>
<td>BiSb_{8}Sn_{0.05}</td>
<td>20</td>
<td>42.0</td>
<td>54.0</td>
<td>2.3</td>
<td>0.94</td>
</tr>
<tr>
<td>BiSb_{12}Sn_{0.01}</td>
<td>20</td>
<td>39.0</td>
<td>56.6</td>
<td>1.2</td>
<td>1.00</td>
</tr>
<tr>
<td>BiSb_{12}Sn_{0.05}</td>
<td>20</td>
<td>50.0</td>
<td>65.5</td>
<td>1.3</td>
<td>1.00</td>
</tr>
<tr>
<td>BiSb_{7}Te_{0.002}</td>
<td>20</td>
<td>30.5</td>
<td>46.5</td>
<td>1.5</td>
<td>1.15</td>
</tr>
<tr>
<td>NaCl</td>
<td>10</td>
<td>20.3</td>
<td>53.4</td>
<td>2.5</td>
<td>1.14</td>
</tr>
<tr>
<td>LiF</td>
<td>40</td>
<td>104.0</td>
<td>370.0</td>
<td>3.7</td>
<td>1.19</td>
</tr>
<tr>
<td>CaF_{2}</td>
<td>150</td>
<td>160.0</td>
<td>490.0</td>
<td>3.0</td>
<td>1.20</td>
</tr>
<tr>
<td>MgO</td>
<td>200</td>
<td>720.0</td>
<td>910.0</td>
<td>1.3</td>
<td>0.87</td>
</tr>
</tbody>
</table>

Fig. 8. Temperature dependence of the microhardness (1) and dislocation mobility (2) for Sb (a) and CaF_{2} (b) monocrystals. P=20 g (a) and 150 g (b)

The comparison of Fig. 8a and Fig. 8b indicates that H (T) and \gamma (T) dependences in the T interval from 77 to 673K for Sb and CaF_{2} are mainly analogous. In this case the \gamma (T) curves are the same as the \alpha (T) ones for alkali-halide crystals (Fig.2). The start temperature when the dislocation mobility rises sharply, T_{cr}, is equal to 0.32T_{ml} for Sb and 0.35T_{ml} for CaF_{2}. This fact is in a good agreement with the above-mentioned results for alkali-halide crystals and for covalent semiconductors. The MgO crystals are evidently the only from the investigated crystals for which T_{cr} was not achieved (for MgO the T_{ml} = 3070K [74, 75]). Therefore the sharp increase of dislocation mobility with temperature increase was not observed for it (Fig. 9). Fig.9 shows that in the range 400-700K the parameter \gamma even decreases with the decrease of H.
Fig. 9. Temperature dependence of the microhardness (1) and dislocation mobility (2) for (111) MgO crystals. P = 100 g.

The same dependences were mentioned at (001) MgO indentation as well [66, 30]. Thus, in [30] it was found that the dislocation rosette edge arm length with temperature increase in the range 500-850°C was practically constant and microhardness is approximately 5 times less. Thus, in MgO crystals the dislocation mobility anomalous behaviour and the correlation disturbance of this parameter with microhardness takes place in both temperature regions T<293K (table 1, [66, 72]) and T>293K.

In conclusion it can be mentioned that the dislocation mobility anomaly in the stress field of concentrated load is a rather universal phenomenon. This effect is observed in crystals with different structure lattice and various chemical type of bond.

**Dislocation pseudomobility.**

**Impulse mechanism of microindentation process.**

The anomalous behaviour of crystals of NaCl type under low-temperature indentation described in the previous paragraph can be explained as follows. It is known from the literature that the individual dislocation mobility in alkali-halide crystals such as NaCl decreases with the temperature decrease from 293 to 77K [76].

Therefore one can assume that the indentor penetration must be hindered in these crystals at 77 K in comparison with that observed at room temperature. Because of low dislocation mobility at 77 K, the dislocation pileup near the indent may be formed, the development of the dislocation rosette hindered, the dislocation network produced etc. The size of the formed indentation is much smaller than that observed at room temperature.

However, at 77 K the dislocation motion resistance is not so strong as to hinder in general the dislocation pileup as it takes place, for example, in the case of covalent crystals at room temperature [50,55]; therefore the break of these pileups and the formation of the dislocation rosette arms occur at a definite moment. This process is not followed by a marked growth of the indentation and as a result a small indentation (a high microhardness) is observed in the presence of well developed dislocation rosette. The mobility of the dislocations, in this case, may be called pseudomobility, because it is connected with the concentration of high local stresses, while the process of the dislocation rosette formation may be called impulse process in contrast to the usual one.

From the energy consideration one can say that the work of the external load in the case of the impulse mechanism can be spent directly without the increase of the indentation size on the dislocation motion connected with the rosette formation.

The discussing presented above allows to conclude that the microindentation impulse mechanism for the investigated crystal can be observed only in a definite temperature region when the dislocation mobility is small enough to stimulate the appearance of a strong dislocation pileups near the indentation, but at the same time it is sufficiently high to stimulate a burst of these pileups with the formation of the rosette arms. In this case at the certain temperature T* the maximum development of the dislocation pseudomobility can be expected.

From this point of view the data of Table 1 may be explained in the following way. One can see from the table that for the ionic crystals (T = 77K), beginning with NaCl, the regular decrease of
anomalous dislocation mobility effect with hardness increase (except NaCl) is observed. This fact may be concerned with the following: \( T = 77K \) for NaCl is close to \( T^* \), while for harder crystals (LiF and MgO) \( T^* \) is expected to be shifted up and the higher crystal hardness, the larger shift. For the soft KCl crystals, on the contrary, \( T^* \) is probably lower than 77K, so the dislocation mobility low-temperature anomaly is not so pronounced as for NaCl (Table 1). The special experiments carried out on some alkali-halide crystals [65] and data obtained earlier [72] confirm the above explanation. Thus Fig.9 and 10 demonstrate that for all investigated crystals the curves \( l(T) \) or \( \gamma(T) \) have the maxima which are shifted to the higher temperature region with the increase of crystal hardness.

![Fig. 10. Dependence of the dislocation rosette edge arms length on deformation temperature for (001) NaCl (1) and LiF (2).](image)

For LiF crystals the maximum, as it has been assumed, is situated higher than 77K (curve 2, Fig.1), for NaCl - 77K or lower temperature (curve 1, Fig.10) and for MgO (the hardest of the investigated crystals) \(- T^* \approx 340 \div 400K \) [66]. In paper [77] at microindentation of (001) MgO the maximum on the curve \( l(T) \) for the edge dislocation ensembles was found to be situated in the region of higher temperature (~470K). The authors explained this fact with the formation of Cottrel atmosphere, which hinders the dislocation motion.

![Fig. 11. Temperature dependence of the length of the dislocation rosette arms for GaAs (a) and continuous acoustic emission arising in indentation (b) [79].](image)

On the basis of the experimental data, known from literature, one can assume that the phenomenon of the dislocation pseudomobility in indentation occurs not only in crystals such as NaCl. As it has been mentioned previously the dislocation mobility of the covalent semiconductors with a diamond lattice is so small that the dislocation rosettes are not generally developed near the indentation at room temperature [5, 63]. The rise of the indentation temperature (or the annealing of the samples deformed at room temperature) results in an increase of the dislocation mobility and dislocation rosette formation, however, the conditions for the dislocation pseudomobility initiation can be found only in a definite temperature region. Therefore the \( l(T) \) dependence should have the shape of the curve with a maximum. Such a dependence was revealed for germanium [78] and gallium arsenide [79]. Parallelly with the development of the dislocation structures continuous acoustic
emission, connected with the formation of these structures, was measured for GaAs [79]. A sharp increase of this emission was observed at the temperature corresponding to the maximum value of \( t \) (Fig. 11), pointing to the impulse character of the dislocation rosette formation. These results serve, in our opinion, as a direct evidence of the dislocation pseudomobility.

In work [80] the curves obtained under annealing of predeformed by indentor Si crystals the clear-cut maxima within the narrow annealing temperature interval were revealed. The authors noted that it was difficult to explain the nature of these maxima.

The results listed in Table 4 indicate that for semiconductor crystals, as well as for ionic ones, the temperature, adequate to the maximum of arm rosette length, decreases regularly with hardness decrease.

<table>
<thead>
<tr>
<th>Crystal</th>
<th>Plane</th>
<th>H, kg/mm(^2)</th>
<th>T*, °C</th>
</tr>
</thead>
<tbody>
<tr>
<td>Silicium</td>
<td>(111)</td>
<td>900-1060</td>
<td>900-1100 /80/</td>
</tr>
<tr>
<td>Germanium</td>
<td>(111)</td>
<td>710-750</td>
<td>700-750 /78/</td>
</tr>
<tr>
<td>Galium arsenide</td>
<td>A (111)</td>
<td>660</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td>B (111)</td>
<td>630</td>
<td>570</td>
</tr>
</tbody>
</table>

Thus, the regularities observed at microindentation of crystals with different type of chemical bond and different crystal lattice structure (ionic crystals, semiconductors, semimetals) can be explained taking into consideration the hypothesis about the impulse mechanism of microindentation process and the dislocation pseudomobility. On the other hand, it is clear that the results, obtained on different materials which can be arranged in a common scheme confirm the rightness of this scheme.

Now let us consider a number of experimental factors evidenced the impulse character of the plastic deformation under indentation.

a. In accordance with this hypothesis both high microhardness magnitude and dislocation anomalous behavior in the indentor stresses field of alkali-halide crystals at 77K are caused by the same reason - a formation of the powerful dislocation pileups within the deformation zone near the indentor. Therefore the correlation between the changes of both \( H_{77} \) and \( k \) parameter characterizing the low-temperature anomaly of dislocation mobility must be*. In this case it is natural to assume that the microhardness increase will be followed by the pronounced anomaly (under “expulsion” of dislocation ensemble the more powerful dislocation aggregations will be relaxed).

Such case is indeed realised for the NaCl:Pb crystals (C=1.65 \times 10^{-2} \text{ mole }\% , Fig. 12). It is seen from figure that after quenching the correlation between the \( H_{77} \) and \( k \) changes occurs and besides this the first parameter maximum corresponds to maximum of the second one, and minimum – to minimum.

The correlation between \( H_{77} \) and \( k \) can be reflected in another form. The length of the dislocation ensemble ran (the dislocation rosette arm length) is dependent not only on the value of stresses, arising within the deformation zone near the indentation, but also on the resistance force of medium in which the ensemble is moved. If the latter factor is most appreciable then \( H_{77} \) increase can be followed by the dislocation mobility decrease, i.e. the \( H_{77} \) maximum conforms the \( k \) minimum and on the contrary.

* No correlation will be if \( H \) does not relate to the dislocation mobility but is connected with other factors (for example in the case of interstitial microindentation mechanism occurrence) [46-59].
Variant of such correlation for NaCl:Ca and NaCl:Pb is presented in Figure 6 (curves 2 and 3).

**Fig. 12.** Dependence of microhardness and $k = \gamma_{77}/\gamma_{293}$ parameter (for edge dislocations) on quenching temperature for NaCl:Pb crystals. $C = 1.65 \times 10^{-2}$ mole%.

1, 2, 3 – $H_{293}$, $H_{77}$ and $k$, respectively.

b. The presence of the microindentation process different mechanism for the alkaly-halide crystals at the two deformation temperatures 77K and 293K follows from the results obtained for the doped NaCl crystals. The microhardness-concentration dependences of NaCl:Ca crystals treated by storage and quenching are given in Figures 13 and 14. The samples aging storage at room temperature during 12 years is carried out. Then these samples from the temperature in the region from 323 – 723K are quenched.

The keeping time at $T_q$ was equal to 6 hours. Aging effect as shows the comparison of curves 1, 2, Fig. 13, 14, is significantly dependent on a measurement temperature. The samples containing the small impurity concentrations ($C = 2 \times 10^{-4}$ and $8 \times 10^{-4}$ mole %) show the marked hardening at 293K while at 77K – softening. The softening at 293K and practically no H changes at 77K for the samples with the $C = 2 \times 10^{-3}$ mole % were observed.

The H(C) dependences after aging demonstrate the different shape at $T=293$ and 77K as a result of inadequate H changes at different deformation temperatures. In the first case the curve with maximum at $C = 2 \times 10^{-3}$ mole % and in the second one – the curve with minimum at the same concentration are observed.

**Fig. 13.** Impurity concentration dependences of the microhardness of NaCl:Pb monocrystals obtained at room temperature. 1 - before aging [82]; 2 - after aging. A dashed line – H undoped crystals. P = 10g.

Such varied behaviour of NaCl:Pb crystals at 293 and 77K can be explained by the assuming that the different mechanism of microindentation process at these temperatures take place: smooth – at first temperature and impulsive – at second one. Occurrence of such specific and different H(C) curves for the aged crystals (curves 2, Fig. 13; 14) is easy to interpret as follows.
The Pb\(^{2+}\) is poorly soluble into NaCl crystals impurity. Therefore even in the samples with the small impurity concentrations (C=2×10\(^{-4}\); 8×10\(^{-4}\) mole %) under aging the aggregations of cation vacancy – impurity ion dipoles can take place. Formation of such impurity complexes effectively hinders a motion of the dislocations.

Fig. 14. Impurity concentration dependences microhardness of NaCl:Ca crystals at T = 77K.
1 - before aging [82]; 2 - after aging; 3-5 - after quenching at T\(_q\), K: 373 (3), 573 (4) and 673(5) P = 10g.

This is confirmed by the dislocation mobility measurement; the latter after aging became 1.5-2 times as smaller as for samples with impurity concentrations C=2×10\(^{-4}\) and C=8×10\(^{-4}\) mole %. Therefore the microhardness of these samples increases as a result of aging (curves 1, 2, Fig. 13)**.

The higher impurity concentrations (2×10\(^{-3}\); 1.65×10\(^{-2}\) mole %) result in the formation of the large impurity complexes, precipitates which less effectively influence the mechanical properties.

In this case the crystal lattice purification from the hindrances to the dislocation motion perhaps is involved. These processes are probably pronounced for the samples with C=2×10\(^{-3}\) mole %, hardness of which after aging is equal to the hardness of undoped crystals (curve 2, Fig 13). Therefore the H(C) dependence obtained at room temperature for the aged samples represents a curve with minimum.

The electron-microscopic observations of the surface decoration pictures of NaCl:Pb crystals containing the different impurity concentrations confirm that the crystal lattice with C=C×10\(^{-3}\) mole % among the large impurity complexes is the purest [82]. This is also evidenced by the extremum presence on the concentration dependence of potential difference arising under microindentation of NaCl:Pb crystals with C=2×10\(^{-3}\) mole % [83].

The alternative dependences of the microindentation at liquid nitrogen are characteristic. The impulse mechanism of this process results from the generation of the powerful dislocation accumulations bearing responsibility for the intense (strong) microhardness increase of the alkali-halide crystals at 77K. The impurity softening of the aged crystals (curve 2, Fig 14) is perhaps connected with some splitting (parting) of the dislocation by an impurity. The certain optimal concentration of impurity defects C\(_0\) (into lattice between the large complexes, precipitates) which results in the marked softening probably exists. This effect is practically absent for the samples with the purer lattice (C=2×10\(^{-3}\) mole %) and their microhardness at 77K is close to the undoped sample microhardness. So, the H maximum at that concentration is observed. Quenching results in the impurity complexes, precipitates dispersion and as a result, the concentration of effective impurity defects into the formerly softening samples is increased and becomes larger than C\(_0\); the H increase is observed (curve 3-5, Fig 14). Specific behaviour of crystals with C=2×10\(^{-3}\) mole % can be explained as follows. After quenching at T\(_q\)=373K some impurity solution occurs, however the impurity defects

** Under the smooth microindentation process the good correlation between the dislocation mobility and microhardness is observed.
concentration into lattice remains even lower than $C_0$, so the small hardening is observed (curves 2 and 3, Fig. 14). At higher quenching temperatures (573, 673K) this concentration becomes close to $C_0$, the softening takes place (curves 4, 5, Fig. 14). As a result the unusual course of $H(C)$ dependence – the concave curve – is obtained$^\ast$.

Thus, the dependences observed under investigations of impurity state influence on mechanical properties of NaCl:Pb crystals well agree with the idea about the different mechanism of the microindentation process at 293 and 77K temperatures.

c. The results obtained under studying of relaxation phenomena arising at indentor removal also evidence 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 dislocation patterns arising around the indentation were revealed by means of an etch-pit technique. The surface etching near the same indentation was carried out twice – when the indentor is under load and after its unloading (its removal from the sample).

It was established the dislocation structures practically do no change after indentor removal for the softer NaCl and LiF crystals. Another picture is observed for MgO crystals. The dislocation rosette progress and the edge and screw dislocation arms significant elongation after indentor removal occurred. Many new lines of edge dislocations were originated, the latter 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> markedly rose too.

The dislocation displacement, a new glide bands generation under indentor unloading were formerly observed on both alkali halide crystals [85] and semiconductor 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 occurred under unloading.

Both this fact and different behavior of the softer crystals and MgO ones can be explained as follows. It was noted 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 takes 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 stopped. In definite moment these aggregations break out and new arm 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 of 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, that 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].

$^\ast$ As known from literature the analogous concentration dependence of microhardness on the doped NaCl crystals is not observed till now.
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 \[6,8,15\].

The completion effect of square-shaped slip traces in the unloading process was clearly observed for different indentor loads. Fig. 15 shows the result for the indentation orientation

![Fig. 15. The dependence of the size of the slip-line squares \((a_0, a) (1,2)\) and length of cracks along \(<110>\) \((l_0, l) (3,4)\) on load. \(a_0, l_0\) are the parameter values for the indentor under load, and \(a, l\) after unloading.](image)

\(d \parallel <100>\). Analogous data were obtained for the other orientation - \(d \parallel <110>\). It is seen for the case of the indentor under load that the dependence of the side of the square region, \(a_0\), on load is a linear
one. After unloading, \( a(P) \), a straight line is observed (\( a \) is the size of the side of the square region after unloading). It is practically parallel to \( a_0(P) \) but is higher (Fig. 15, lines 1, 2), i.e. the increase in the size of the deformed region near indentations occurs during indenter removal for all the loads in question. However, the relative change \( (a - a_0)/a_0 = \Delta a/a_0 \) decreases with increasing load. The same peculiarities are also observed in measuring the crack length (Fig. 15, straight line 3, 4: the distance between the ends of the cracks propagating along \(<110>\) in the opposite directions was determined. Fig. 15 shows that the \( l(P) \) and \( l_0(P) \) straight lines (\( l_0 \) and \( l \) are the size of the crack length for the case of indenter under load and that after unloading, respectively) are deflected by a specific angle concerning \( a(P) \) and \( a_0(P) \). It is completely regular. The ratios

\[
k_3/k_j = k_2/k_4 = 0.71
\]

must be fulfilled for perfect squares and cracks propagating along their diagonals and ending in their apexes, because \( l = a \times 2^{1/2} \). Here \( k_i \) are the constants from the equations:

\[
a = k_1 P + k_2
\]
\[
l = k_3 P + k_4
\]

The plots presented in Fig. 15 (straight lines 2,4) give us

\[
k_3/k_j = 0.6
\]
\[
k_2/k_4 = 0.64
\]

In spite of the fact that the slip-line squares and cracks along \(<110>\) are observed for the different indenter orientations, the anisotropy of the parameters under investigation is revealed (Figs 16 and 17). The square size and the crack length are dependent on the directions of the indentation diagonals. This anisotropy is small and virtually within the error limits for indenter loads of 100-200 g. However, it increases when the load rises and values \( a_0, a, l_0, l \) parameters are greater for the \( d ||<100>\) orientation than for the \( d \parallel<110>\).

![Fig. 16 The comparison of \( a_0(P) \) (1, 3) and \( a(P) \) (2, 4) dependences for two indenter orientations: \( d \parallel<100>\) (1,2) and \( d \parallel<110>\) (3,4)](image)

The presence of the investigated parameter anisotropy is in agreement with the fact that the microhardness anisotropy is clearly revealed on the \((001)\) plane of \( \text{MgO} \) single crystals. So \( H_{<100>} > \)
H_{<110>}, H_{<10>}, and H_{<110>}, are the microhardness values for the orientations of \(d\|<100>\) and \(d\|<110>\), respectively) for the Vickers indenter [7,11,12,19,20].

**Fig. 17. Comparison of \(l_0 (P) (1,3)\) and \(I(P) (2,4)\) dependences for two indenter orientations: \(d\|<100>\) (1,2) and \(d\|<110>\) (3,4).**

All the results presented here permitted us to conclude that the unexpected phenomenon is observed on the (001) plane of MgO for the different indenter loads and different indenter orientations concerning the crystallographical directions of the deformed plane - plastic deformation having the same sign as in the loading process takes place during indenter removal. The additional material is carried 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: divergent 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 deformed at room temperature, the parameter \(L/d \sim 1.2\) (\(L\) is the spreading distance of hills from the side of the indentation with \(d\|<100>\) 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 indenter 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 structures 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 indenter 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 $\Delta a$ and $\Delta l$ 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.

### About nature of the temperature hardening at the microindentation

The significant crystal hardening at low temperature can be explained by the impulse mechanism of dislocation structure formation during indentation proposed above. The question about temperature hardening can be considered on the model alkali-halide crystals, for example, for which the most detailed, complex investigations were carried out [6, 42, 44, 45, 64, 69, 70, 82, 86].

This type of hardening is different from well-known point defect hardening. So, the $H$ rise by temperature lowering from 293 to 77K can be significantly more than changing of this parameter at doping or irradiation. For example, the $H_{NaCl}$ is enhanced from 20 to 80kg/mm$^2$ with the temperature lowering from 293 to 77K in the first case and from 35 to 40kg/mm$^2$ in the second one [3, 4, 6, 42, 45, 82, 86].

The length of the arm of the dislocation rosettes, arising around the indentation in the case of radiation and impurity hardening is more sensitive parameter than microhardness, the $l$ changes are more significant than $H$. For instance, for NaCl crystals irradiated by proton it was found that $H/H_0=1.2; l/l_0=4.5$ . These ratios are equal to 1.8 and 3.3, respectively, in the case of the Ca$^{2+}$ doped ones [42]. Here $H_0$ and $l_0$ are values of investigated parameters before irradiation (doping) and $H$ and $l$ are after irradiation (doping). The different regularities take place at the temperature hardening: $H$ changes are more significant than $l$ (Table 1).

The correlation between changes of $H$ and $l$ in the case of point defect hardening takes place: the increasing of $H$ is accompanied by decreasing of $l$. A breakdown of this correlation is even observed above (table 1, 2) in the case of the temperature hardening.

The other clear example of the different regularities for two type of hardening is the comparison of yield stress and microhardness behaviour. It is known [6, 42, 70, 82, 86, 93] that under doping the $\sigma_y$ changes more significantly than $H$. For example, the yield stress of NaCl:Sr crystals increases by 4 but $H$ only by 1.2 times [70]. In the [93] was established that in doping the $\sigma_y$ increase by 10 times is accompanied by $H$ increase only by 20-25%. It resulted in the opinion that the yield strength is more sensitive characteristic than microhardness. However the results obtained in our laboratory have shown that other situation is observed in the case of temperature hardening [6, 42, 44, 45, 82, 86]. As an example the results obtained for NaCl:Ca crystals are given in the table 5.

As it follows from Table 5, the $H$ changes at impurity hardening are much less than $\sigma_y$ changes, but the situation is contrary in the case of the temperature hardening. The correlation between $\sigma_y$ and $H$ is also differently expressed for these two types of hardening. For example the $\sigma_y$ ($H$) dependences are given in Figures 18 and 19 for these two types of hardening. It is seen that these dependences are linear for impurity hardening (Fig. 19) and are described by expression:
\[ \sigma_y = \alpha (H-H_0) \quad (1) \]

where \( \alpha \) and \( H_0 \) are the parameters characteristic of given crystals. The parameter \( \alpha \) doesn’t depend on temperature and is equal to 0.24, that is less than \( C \) value \( (C=1/3) \) from known Tabor’s formula \([96, 97]\).

\[ \sigma_y = CH \quad (2) \]

\( H_0 \) depends on temperature and is diminished with growth of the latter. Such relation is characteristic not only of NaCl:Ca, but also of other doped alkali-halide crystals.

<table>
<thead>
<tr>
<th>C, mol/%</th>
<th>Impurity hardening</th>
<th>Temperature hardening</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>( H/H_0 )</td>
<td>( \sigma_y/\sigma_y^0 )</td>
</tr>
<tr>
<td>Pure</td>
<td>1,00</td>
<td>1,00</td>
</tr>
<tr>
<td>( 10^{-3} )</td>
<td>1,06</td>
<td>1,68</td>
</tr>
<tr>
<td>( 5\times10^{-3} )</td>
<td>1,14</td>
<td>3,20</td>
</tr>
<tr>
<td>( 9\times10^{-3} )</td>
<td>1,05</td>
<td>0,83</td>
</tr>
<tr>
<td>( 3,65\times10^{-2} )</td>
<td>1,34</td>
<td>6,40</td>
</tr>
<tr>
<td>( 5,8\times10^{-2} )</td>
<td>1,70</td>
<td>6,10</td>
</tr>
<tr>
<td>( 1,6\times10^{-1} )</td>
<td>1,96</td>
<td>12,70</td>
</tr>
</tbody>
</table>

**Fig. 18. Dependence of the yield stress on microhardness for NaCl:Ca crystals at impurity hardening.**

Absolutely different situation is observed in the case of the temperature hardening. From Figure 18 it follows, that only for small impurity concentration \( \left(10^{-3} \text{ mol.}\%\right) \) the \( \sigma_y(H) \) dependence can be considered linear. The more complicated shape of this dependence at the \( C=10^{-2}-10^{-1} \text{ mol.}\% \) (curves 2, 3, Figure 19) takes place. The constant \( \alpha \) is much less \( (\alpha =0,018) \) in this case than at impurity hardening.

The peculiarities of dislocation structures, arising at indentation, also indicate on the different nature of temperature hardening and hardening by point defects. The arms of dislocation rosettes on (001) NaCl plane are narrow and long at the temperature hardening and short and wide at irradiation hardening. In the first case the view of rosettes allows to assume that the dense dislocation mass as if “sweeping” by the dislocation ensembles. In the second case the formation of the rosette band can be presented as follows. The new ensembles arise in the inhibiting of one dislocation ensemble. These new ensembles spread parallel to the first one and as a result the wide arms are formed.

The view of dislocation rosette arising around the indentation is more different in the
Fig. 19. Dependence of the yield stress on microhardness for NaCl:Ca crystals at temperature hardening (673 – 77K). C, mol. %: 5x10^{-3} (1); 3,65x10^{-2} (2); 1,6x10^{-1} (3).

The case of indentation (111) plane of NaCl crystals. The “star” like rosette appeared in the case of indentation at liquid nitrogen (LN) temperature. The arms of this rosette are directed along the traces of the slip planes on surface. In the case of radiation hardening the deformation area consists of two triangles formed by the etch pit rows. The evolution of appearing and development of these triangles depending on radiation dose is described in [3]. The similar dislocation rosette is observed on (111) plane of doped NaCl crystals [28], too. Thus, we can made conclusion about different regularities of the dislocation structure formation around indenter prints occurred at the temperature and point defect hardening. The representations about smooth and impulse mechanisms of microindentation allow to understand the different behaviour of crystals at two types of hardening. At smooth mechanism of indentation process, which takes place at room and higher temperatures on alkali-halide crystals, the impurity (radiation) hardening is connected with inhibiting of the moving dislocations by point defects and their complexes. The nature of such hardening is well known from the literature [77, 100-104]. The temperature hardening also is connected with decreasing of dislocation mobility at the temperature lowering. However this connection has the specific character. The low dislocation mobilities leads to the formation of the powerfull dislocation pileups within the deformation zone near the indentation, the latter hinders the indenter penetration into crystals. However the resistance to the dislocation motion at 77 is not so strong, therefore the break of these pileups and further development of dislocation rosette arms occur at a definite moment. This process is not followed by a marked growth of the indentation and as a result a small indentation (a high microhardness) in the presence of well developed dislocation rosette is observed.

Such impulse mechanism of deformation is only characteristic of microindentation, the different regularities in the case of yield stress measurements at low temperatures [42, 70, 82, 106] can be observed. The correlation between H and $\sigma_y$ can be different at temperature hardening and hardening by point defects. In latter case the $\sigma_y$ and H increasing is connected, in general, with the inhibiting action of the local obstacles to the moving dislocations [Fig. 18, 19].

Confirmation of the impulse mechanism of microindentation process by the experiments on recording electrical signals

It is known the deformation and fracture of various crystals, including alkali-halide ones are accompanied by occurrence of electrical signals [107-114]. In particular a potential difference between the sample planes under indenter penetration it was revealed [107, 108, 110, 111]. This phenomenon was explained by movement of charged edge dislocations.

If it is true, the regularities of development of the dislocation structures at the microindentation can be explored with the help of electrical signal recording. The indentation was
made by means of a special equipment [83]. It was possible to register simultaneously the indentor penetration depth with a time resolution of 1 ms and the electrical signals (the potential difference) in the frequency region from 10 Hz to 1 MHz [63, 115]. The investigations were performed on a (001) cleavage plane of pure and doped alkali-halide crystals (KCl, NaCl, LiF) and MgO.

Fig. 20. Indentation on (001) plane. Dependence between the dipole moment values, according to saturate stage, and the run length of edge dislocation ensemble.

A row of regularities, confirming the filed potential difference connection with a motion of charged dislocations, was found out. So, it was observed for LiF crystals at room temperature that the time dependences of all investigated plastic deformation parameters (the depth of indentation, the print diagonal, the length of dislocation rosette arms \( l \)) and the potential difference are similar [116-119]. The curves \( h(t) \), \( d(t) \), \( l(t) \) and \( U(t) \) have two stages. The first fast stage is characterized by abrupt rise of the parameters under investigations; its duration was equal to 0.2 s. The rise of the parameters was slower during the second stage and it reached saturation in the course of \( \sim 100s \) for plasticity and \( \sim 300s \) for the dipole moment. There is a linear relationship between the saturation values of the crystal dipole moment \( (P_0) \) and the length of the edge dislocation track \( (l) \) (Fig. 20) [116, 117].

An interesting result was established - the delay of the appearance of the dipole moment with respect to the beginning of the indentation depth registration. The delay time was close to 0.04 s. The calculation of \( P \) value, based on representation, that a charge at the indentation is created under movement of edge dislocations, has allowed to explain this phenomenon [116, 117].

Experiments made on predeformed crystals showed a decrease of the dipole moment due to the plastic deformation. So the dipole moment falls by a factor of 25 after 8% deformation [117].

These facts and literature data [107, 108, 110] indicate that the potential difference, arising at indentation of ionic crystals, is connected with the motion of charged dislocations.

For the understanding of the deformation mechanism it is very important to study the beginning of the deformation process, because of the basic part of the dislocation rosette is formed in this time interval. The most detail experiments were carried out on the LiF crystals [61]. The results obtained for these crystals on (001) are given in Fig. 21. It is visible the indentation obtained depth and potential difference, arising at a deformation, are smoothly changed at room temperature (Fig.21, a). The other situation takes place in the case of the LiF indentation at liquid nitrogen (LN) temperature (Fig.21, b). We can see from Fig.21,b that the increase of potential difference is not smooth now, but step like (Fig. 22, curve 2).

The appearance of "plateau" on \( U(T) \) curves testifies, that in some moments of the identification, the development of the dislocation rosettes near the indentation is practically stopped, strong local stresses appear. Then the stress relaxation takes place, the moving of the dislocation ensembles is observed and accordingly the following increase of the charge occurs. (Fig.21,b, curve 2). So as it was assumed, in the case of the LiF indentation at 77K the creation of the dislocation structures has an impulse character.

The \( h(t) \) dependence at \( T=77K \) starting from some loading depth has wave-like character (Fig.21,b, curve 1). It is better seen at longer observation times (Fig. 22, curve 1). Such character of the indentor motion is connected, apparently, with the peculiarities of the stress relaxation passing in the deformation area near the print. For the studying of this phenomenon it is necessary to carry out
the further experiments. But at moment we can assume that such specific of the indentor motion is one of the factors giving the small print size and high values of the microhardness at 77K.

![Fig. 21. The beginning stage of indentation (001) plane of LiF crystals. 1 - the indentation depth; 2 - the potential difference U arising at indentation. P=260g (a) and 780 (b).](image)

The comparison of the curves U(t) and h(t) (fig. 22) shows that synchronism of the changing of this parameters at the room temperature is broken at 77K, the impulse mechanism takes place.

![Fig.22. (001) plane of LiF. The kinetic of indentor indentation and development of respective potential difference. T=77K, P=920g. 1 - the indentation depth, 2 - the potential difference.](image)

Such regularities of the deformation kinetics took place for some other crystals too. So, for the KCl:Ba crystals (C ~ 10⁻¹ mol. %) the coordination of U(t) and h(t) dependences was broken. For the NaCl crystals the curve h(t) was smooth at the room temperature, but it was wave-like at the liquid nitrogen temperature. On the harder MgO crystals the curve h(t) was wave-like at the room temperature. This result confirms our assuming that on the MgO crystals, unlike to alkali-halide crystals, the impulse mechanism of the deformation must be observed at the 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 another mechanism for the same material in the dependence on deformation temperature can take place in such sequence: in accordance with temperature lowering the smooth mechanism is replaced by the impulse one, then the latter transfers in the 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 of semiconductor
materials at room temperature, and it changes for the smooth mechanism at higher temperatures. There is the following regularity in a series of similar crystals (for example, ionic, covalent) – the higher microhardness of a crystal the 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.

* * *

A brief information after Conclusion

D.Grabco

The behaviour of solids under action of the external load is a key problem in modern material 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 the Laboratory of Mechanical Properties of Crystals in the time period 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 important results of the 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 interval. Thus, physico-mechanical properties (such as, anisotropy coefficient, brittleness, plasticity, dislocation mobility, etc.) typical for soft crystals at low temperature are characteristic of the hard crystals at elevated ones [121].

The existence of the five structural levels of the elastic-plastic deformation at concentrated load action has been noted. There are: (i) superdefect layer having a nano-dimensional thickness, the nearest to the indent; (ii) quasi-destructured (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 full realization of such deformation shape is a function of the many factors: deformation temperature, type of crystal lattice and chemical bond, loading conditions, etc. [121, 122]

The dislocation-disclination-interstitial concept of the deformation mechanism under concentrated load action was formulated and motivated by the experimental data [123]. This concept is a further development of the smooth and impulse mechanism of deformation, forwarded nearly ten years ago by Prof. Yu. Boyarskaya together with her collaborators.

Taking into account the life requests, the range of studied materials and applied methods has been enlarged in the Laboratory of Mechanical Properties of Crystals last year. Many new materials having concrete engineering application along with the crystals studied earlier are under investigation at present. There are: films and nano-dimensional materials on the base of the semiconductors and polymers, multilayer structures, microcrystalline and amorphous materials, metals and alloys, etc.

The nanoindentation method along with microindentation one is used for this material research. The results obtained by the nanoindentation method, on the one hand, allow to learn the mechanical properties of nano-dimensional objects, on the other hand, they further a deeper understanding of physical processes taking place in solids under the concentrated, non-homogenous strain action.

The work for improvement of the device for material brittleness estimation by the registration of acoustic emission signals arising during the indentation and sclerometrical testing is carried out as
well. It will allow to detect the nature of the defects responsible for the crack formation and material destruction.

Many other achievements along with above can be noted. However, even this brief description evidences a high scientific potential of the Laboratory body. No doubt, one can expect that a lot of new important results will be obtained in future in the Laboratory of Mechanical Properties of Crystals, and all of them will be the best memory of the Laboratory founder, Prof. Yulia S. Boyarskaya, the outstanding woman of Moldova, the skilled scientist in domain of the strength and plasticity physics.

References