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Transformation of the mechanical properties of materials by the geomagnetic resonance

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ABSTRACT

The strong geomagnetic influences on mechanical properties of crystals due to their exposure to ultralow crossed magnetic fields, the Earth's magnetic field (~50 μT) and the AC magnetic field (~3 μT) in the electron paramagnetic resonance scheme, are discussed. Resonance relaxation displacements of dislocations in *NaCl* crystals are found both for a harmonic pump field and for a pulse AC field of resonance duration ~0.5 μs. Resonant changes have been also detected in the microhardness of *ZnO*, triglycine sulfate, and potassium acid phthalate crystals after their exposure in the same EPR scheme. The both effects manifest new strongly anisotropic properties. In particular, the frequency of the resonance is very sensitive to the mutual orientation of the sample and the Earth's magnetic field. Physical mechanisms and practical significance of the phenomenon are discussed.

1. INTRODUCTION

The magnetoplastic effect (MPE) was discovered as the movement of dislocations in *NaCl* crystals held in a DC magnetic field $B \approx 0.5$ T in the absence of a mechanical load (Alshits 1987). Numerous subsequent independent studies (Alshits 2003; Golovin 2004; Morgunov 2004; Alshits 2008) showed that this phenomenon is due to a magnetically induced change in the structure of impurity stoppers on dislocations, which results in the reduction of the force of pinning of dislocations and their relaxation displacements in the field of internal stresses. The role of the magnetic field is reduced to changing the spin states of the impurity centers with the removal of the quantum exclusion of a certain electronic transitions, which leads to their transformations.

The phenomenon manifests itself also in the macroplasticity of crystals: under active loading (Golovin 1995) when the stress load is increasing with the constant rate: $\dot{\sigma} =$

const, active deformation $\dot{\varepsilon}=\mathrm{const}$ (Urusovskaya 2003), creep $\sigma=\mathrm{const}$ (Smirnov 2001) and internal friction (Tyapunina 1999).

The magnetoplastic effect is observed in crystals of various types and not only in a static magnetic field but also in crossed static and alternating magnetic fields in the electron paramagnetic resonance (EPR) regime at the resonance frequency

$$\nu_r = g\mu_B B / h\,, \tag{1}$$

where the g-factor is usually close to 2, μ_B is the Bohr magneton, and h is Planck's constant. Such resonances in *NaCl* and silicon crystals were found by the three independent groups (Golovin 1998, 2000, 2002; Osip'yan 2004; Badylevich 2005) at the frequency of 9.5 GHz and the related magnetic field ~0.3 T.

More recently, a similar resonance in *NaCl* crystals was detected and studied in ultralow magnetic fields: the Earth's magnetic field ~50 μT and the pump magnetic field with an amplitude of 3 μT and a frequency of ~1 MHz (Alshits 2016). This particular low-frequency resonance is the subject of this paper. As it will be seen, it has a number of new very informative properties, primarily the strong anisotropy of the effect with respect to the mutual orientation of the crystal, dislocations, and magnetic fields. In particular, the resonance frequency becomes very sensitive to the angle θ made by the sample with the Earth's magnetic field (giant anisotropy of the effective g-factor: $g_{\mathrm{eff}} \approx g_0\cos\theta$). In addition, as it will be shown, this resonance also manifests itself in changing the microhardness of the zinc oxide (*ZnO*), triglycine sulfate (TGS), and potassium acid phthalate (KAP) crystals after their exposure in the same EPR scheme in the Earth's magnetic field together with the very strong anisotropy of the effect including even the giant anisotropy of the g-factor.

2. EXPERIMENT

To create the EPR conditions, the samples were placed in the crossed static and alternating magnetic fields. The Earth's magnetic field was used as the static magnetic field in most experiments. Its direction and magnitude were measured at the place of a sample. The vector $\mathbf{B}_{\mathrm{Earth}}$ made an angle of 29.5° with the vertical direction and had a length of 49.97 μT. The alternating pump field $\tilde{\mathbf{B}}$ was produced in a screened coaxial chamber around rectilinear conductor *1* through which a sinusoidal current flowed (Fig. 1a). Its frequency ν varied in the range of 10 kHz to 1.5 MHz. The amplitude of the harmonic pump field was $1\div6$ μT and the time of exposure was from 15 s to 2 h depending on the type of tests.

In situ effects were studied when the response of a crystal to the magnetic action occurs in the process of exposure and directly involves moving dislocations. "Memory" effects were also studied when the response occurs in a certain time after exposure. They were found by measuring the microhardness of *ZnO*, TGS, and KAP crystals.

In the former case, histograms of paths of fresh dislocations in the *NaCl* crystals introduced in the samples just before the exposure were measured. Using these histograms, the mean free paths *l* for various physical parameters and conditions was calculated. Their positions before and after exposure were determined by the selective etching method. In addition to the absolute mean path *l*, the normalized dimensionless path $l\sqrt{\rho}$, which is the ratio of the l to the average distance $1/\sqrt{\rho}$ between dislocations, where $\rho \sim 10^4$ cm^{-2} is the total density of dislocations, was used.

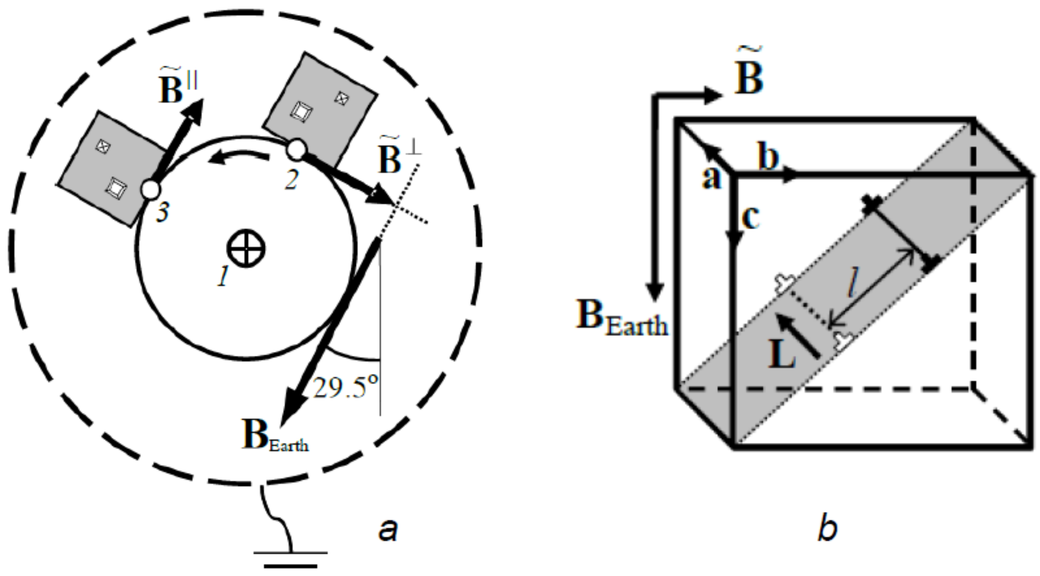

Fig. 1 (a) Scheme of the experiment in the crossed magnetic fields and (b) the geometry configuration of dislocation motion in the *NaCl* crystal.

3. RESONANCE OF DISLOCATION DISPLACEMENTS

3.1 Basic features of the harmonic resonance

<u>Effect of the mutual orientation of dislocations and magnetic fields</u>

The *NaCl* samples were chipped out along the {100} cleavage planes and had approximate dimensions of 3×3×5 mm. Fresh dislocations were introduced in a sample by a weak impact. In this case, most dislocations were rectilinear being directed along the directions **L** || **a** = [100], **b** = [010], or **c** = [001]. Their slip planes belong to the {110} system. Here the experiments with the sample oriented so that the edge **c** was parallel to the Earth's magnetic field and the **a** edge was orthogonal to the plane of the magnetic fields $\{\mathbf{B}_{\text{Earth}}, \widetilde{\mathbf{B}}\}$ (Fig. 1b) are described. In this geometry of crossed magnetic fields, **a**-dislocations (**L** || **a**) are the most mobile whereas **b**- and **c**-dislocations had noticeably smaller paths under the same conditions. The orientation **L** || **a** $\perp \{\mathbf{B}_{\text{Earth}}, \widetilde{\mathbf{B}}\}$ at the chosen position of the sample with respect to the magnetic fields remains optimal for all studied *NaCl* crystals.

The intensity of the discussed resonance depends also on the mutual orientation of the fields $\mathbf{B}_{\text{Earth}}$ and $\widetilde{\mathbf{B}}$. However, this type of anisotropy is not universal and is manifested in different degrees in crystals with different impurity compositions. Figure 1a shows two positions *2* and *3* of the sample that correspond to the mutually orthogonal and parallel fields: $\widetilde{\mathbf{B}} = \widetilde{\mathbf{B}}^{\perp} \perp \mathbf{B}_{\text{Earth}}$ and $\widetilde{\mathbf{B}} = \widetilde{\mathbf{B}}^{\parallel} \parallel \mathbf{B}_{\text{Earth}}$. Normally, for the usual EPR with high-frequency pump field (~10 GHz) the first of these orientations is much more preferable. In our case of the low-frequency EPR (~1 MHz) in *NaCl* crystals with different impurities both traditional situation and the cases of comparable dislocation responses are met. Figure 2a demonstrates the example of a traditional case, which is realized in the *NaCl* crystals with *Ni* impurities where the pump field $\widetilde{\mathbf{B}}^{\parallel}$ causes the EPR of very suppressed amplitude.

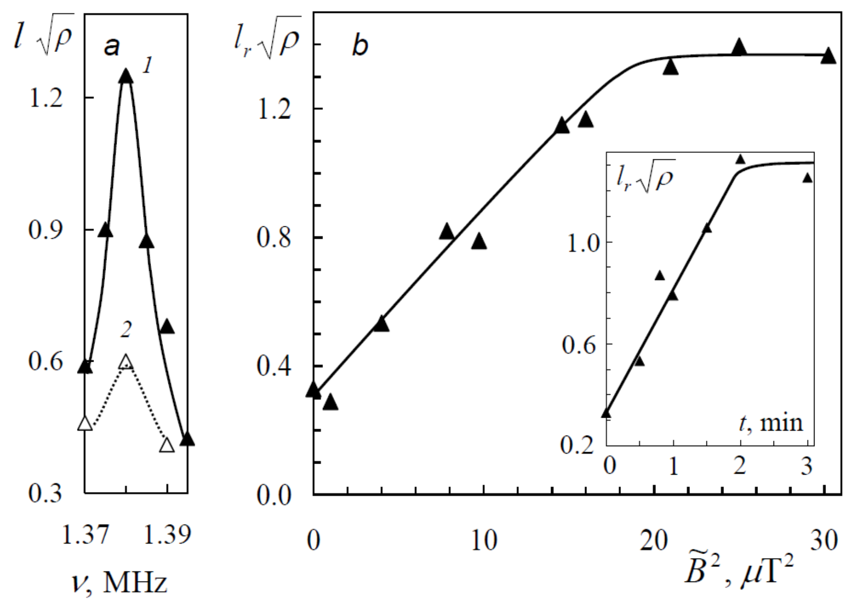

Fig. 2 (a) Peaks of mean dislocation paths *l* in the $NaCl_{Ni}$ samples exposed for t = 3 min to the fields $\widetilde{\mathbf{B}}^{\perp} \perp \mathbf{B}_{\text{Earth}}$ (*1*) and $\widetilde{\mathbf{B}}^{\parallel} \parallel \mathbf{B}_{\text{Earth}}$ (*2*) at $\widetilde{\mathbf{B}}$=3.12μ T in the vicinity of the resonance frequency 1.38 MHz of the pump field and (b) the maximum paths l_r versus the square of pump field amplitude (at t = 1 min) and the exposure time t for $\widetilde{\mathbf{B}}$=3.12μ T.

Dependence of the resonance mean path on the key parameters

The dependences of the resonance mean paths l_r on the amplitude of the pump field $\widetilde{\mathbf{B}}$, time t of the exposure of samples, and the concentration C of the impurity are discussed here. Dislocations and crossed fields are chosen mutually orthogonal in order to ensure the maximum effect.

Figure 2b shows quasi-linear dependences of the mean path amplitude l_r on $\widetilde{B}^2$ and t with saturation at large paths, which corresponds to the depletion of relaxation of the dislocation structure. The presented results are obtained for the same $NaCl_{Ni}$ crystal as data in Fig. 2a. The linear part of these dependences can be represented by the empirical formula $l_r = l_0 + k\tilde{B}^2 t$. Here, l_0 is the background path because of the etching of near-surface stoppers and manipulations with the samples. The linear dependence of the path $\Delta l = l_r - l_0$ on the time t means that the process of displacement of dislocations is quasi-stationary, and the proportionality $\Delta l \propto \tilde{B}^2$ likely corresponds to the quadratic dependence of the effect on the amplitude of the pump field usual for EPR.

The effect of the concentration of impurity on the height of the peak of dislocation paths was studied for *Ca* impurity in a series of Hungarian $NaCl_{Ca}$ crystals for four *C* values (Alshits 2013a). The dependences $l_r(C)$ were measured for **a**-dislocations at two orientations of the magnetic fields $\tilde{\mathbf{B}} \perp \mathbf{B}_{\text{Earth}}$ and $\tilde{\mathbf{B}} \parallel \mathbf{B}_{\text{Earth}}$. The approximate proportionality $\Delta l = l_r - l_0 \propto 1/\sqrt{C}$ was observed within the experimental spread in both cases, and the effect in parallel fields is suppressed by a factor of 1.5÷2. This dependence appears to be the same as in the case of the magnetoplastic effect in a static magnetic field (Alshits 2003 and 2008) and is due to the same kinematic reason: after depinning from the complete series of impurities on dislocation, this dislocation moves to the next series of stoppers spaced from the first series at a distance of $\sim 1/\sqrt{C}$.

3.2 Anisotropy of the resonance frequency

The resonance frequency ν_r = 1.38 MHz of the pump field in Fig. 2a is given by Eq.(1) at **B** = $\mathbf{B}_{\text{Earth}}$ and g = 1.97 and it does not change under the rotation of the pump field. However, the resonance frequency appeared strongly dependent on the orientation of the sample in the Earth's field. In particular, at the rotation of the sample about its edge **a** by the angle θ from the $\mathbf{B}_{\text{Earth}}$ direction, the peak of dislocation paths shown in Fig. 2a is shifted toward lower frequencies proportionally to $\cos\theta$ (Fig. 3):

$$\nu_r \approx \nu_0 \cos\theta, \quad \nu_0 = g\mu_B B_{\text{Earth}} / h, \tag{2}$$

where ν_0=1.38MHz.

Each peak specified by Eq. (2) at a fixed value is accompanied by an additional peak at the resonance frequency:

$$\nu_r' \approx \nu_0 \cos(90^\circ - \theta) = \nu_0 \sin\theta. \tag{3}$$

Such a pair of peaks in Fig. 3a corresponds to angles of 30° and 60°, and these peaks at 45° are joined into one.

Thus, by Eq. (2), our low-frequency EPR resonance is characterized by the giant anisotropy of the effective g-factor: $g_{\text{eff}} \approx g\cos\theta$. Interpretation of this property will be given in Sec. 4.

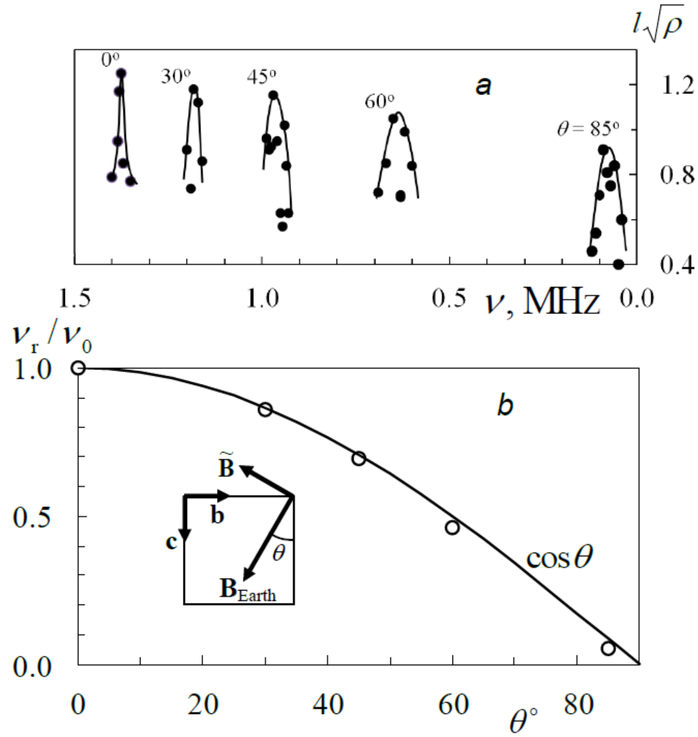

Fig. 3 (a) Peaks of resonance mean paths of **a**-dislocations for various angles θ. (b) Angular dependence of the relative resonance frequency ν_r / ν_0 (ν_0=1.38 MHz). The inset shows the scheme of the mutual orientation of the sample and magnetic fields.

3.3 The resonance with a pulse pump field

There is the other variant of dislocation EPR when a pulsed one replaces a harmonic pumping field. In this case, the resonance frequency ν_r in (1) is replaced by the resonance pulse duration τ_r : $\nu_r \rightarrow \tau^{-1}_r$. This effect under the Earth magnetic field was studied in collaboration with the group of V.A. Morozov at the St. Petersburg State University (Alshits 2013b). The Earth's field at the place of the sample in the setup was obviously different from that in the Moscow experiments. This field was specially measured and was 66 μT at an angle to the vertical of 7.7°. Certainly, this field contained some admixture of the lab background field. The pulsed magnetic field was produced in a solenoid loaded by a rectangular pulse with the controlled duration and amplitude from the generator. Single pulses with the amplitudes $\tilde{B}_m$ = 4.0÷17.6 μT and durations τ = 0.50÷0.57 μs were used in the experiments described below.

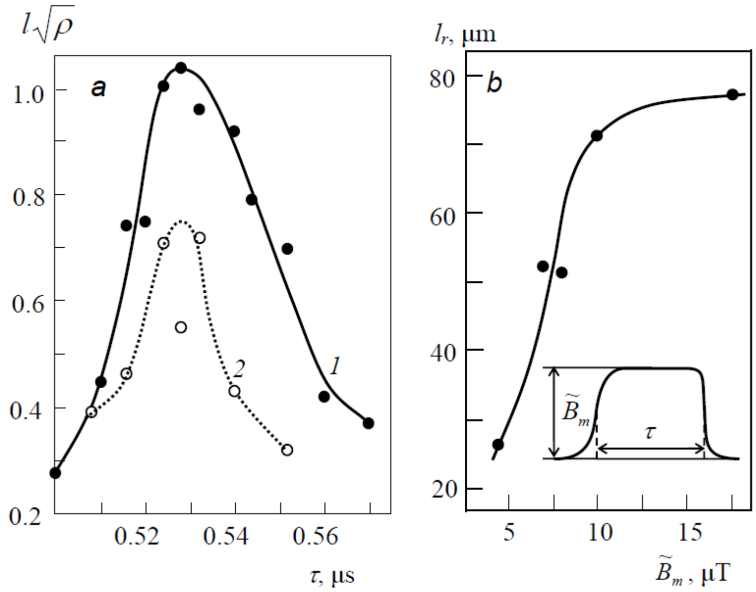

Fig. 4 (a) Normalized mean dislocation path versus the duration of the pulse and (b) the resonance maximum path versus the amplitude of the pulse for *NaCl*Ca crystals.

In the first series of experiments (Fig. 4a), the dependence of the mean paths l of dislocations on the duration τ of the pulses of the pump field with a constant amplitude of $\tilde{B}_m$ = 17.6 μT for two orientations of this field, $\tilde{\mathbf{B}} \perp \mathbf{B}_{\text{Earth}}$ (*1*) and $\tilde{\mathbf{B}} \parallel \mathbf{B}_{\text{Earth}}$ (*2*) and at the orientation of the sample like shown in Fig. 1b were studied. As in the above cases, **a**-dislocations are the most mobile. Their paths at both orientations of the pulsed pump field form pronounced resonance peaks, reaching a maximum at the same duration $\tau_r \approx 0.53$ μs, which corresponds to the *g*-factor of EPR

$$g = h / \tau_r \mu_B B_{\text{Earth}} \approx 2 \,. \tag{4}$$

In the parallel fields $\tilde{\mathbf{B}} \parallel \mathbf{B}_{\text{Earth}}$, the amplitude of the effect from which background paths are subtracted, is almost half of that as in the orthogonal fields, which approximately corresponds to the relation for the same crystals under harmonic pumping.

At the same time, the relative path $l_r\sqrt{\rho}$ observed under the optimal conditions of orthogonal fields is larger than unity (the absolute value is $l_r \approx 80$ μm). This indicates a high degree of relaxation of the dislocation structure, particularly taking into account that the relative density of mobile dislocations approaches 100%. This result seems striking because it is reached in half of a microsecond. We recall that the same degree of relaxation under harmonic pumping is reached in ~5 min.

It was assumed (Alshits 2013b) that the observed “explosive” relaxation is a collective process of self-organization of dislocations at their very fast almost simultaneous magnetically induced depinning when the ensemble becomes unstable. A certain threshold of the effect usually characterizes such processes. In this case, it is a threshold in the amplitude of the pulsed field. Indeed, such a threshold of the effect was experimentally revealed. As is seen in Fig. 4b, the resonance path l_r at a decrease in the pulse amplitude begins to decrease abruptly below 10 μT, reaching background values at ~5 μT.

4. RESONANCE MODIFICATIONS OF MICROHARDNESS

Resonance memory effects (Alshits 2012 and 2018) were studied in *ZnO* crystals grown by the hydrothermal synthesis method, as well as in triglycine sulfate and potassium acid phthalate crystals grown from aqueous solutions by the temperature reduction method.

Microhardness was measured by the Vickers method on a Neophot-21 optical microscope. Each value of microhardness was determined from the averaged size of diagonals of $2 \div 5$ dents of the indenter. The error of these measurements was 1.5÷3%. Measurements were performed before magnetic exposure, immediately after it, each hour during the first 4÷6 h, and then 1÷3 times over the next several days.

Preliminary exposure of the crystals in crossed magnetic fields for an appropriate resonance frequency of the pump field resulted in an increase in the microhardness in the *ZnO* crystal and in its decrease in TGS and KAP crystals. The maximum change in the microhardness (10÷15%) was reached 1÷3 h after the magnetic treatment; then, the microhardness gradually returned to its initial value on the first day. After a sufficient pause, the effect was completely reproduced under the same conditions. The delay of the response of the crystal to magnetic exposure occurs apparently because of diffusion processes after the spin transformation of impurity centres.

Figure 5a shows the dependences of the relative change in the microhardness on the frequency of the pump field for all the crystals measured in definite optimal delay times after the exposure. It is seen that these dependences are clearly resonant. Magnetic memory has strong anisotropy. The effect was completely or partially suppressed when a certain direction of a crystal coincided with the Earth's magnetic field $\mathbf{B}_{\text{Earth}}$; this direction for each crystal was found. In the *ZnO* and TGS crystals, these are axes of symmetry 6 and 2, respectively. This direction in the KAP crystal is the <100> direction, which lies in the cleavage plane and is orthogonal to axis 2.

The most important anisotropic property of this effect is again the strong dependence of the resonance frequency on the sample orientation in the Earth's magnetic field. As it is seen from Fig. 5a, the three groups of peaks related to different angles of the sample inclination from the vector $\mathbf{B}_{\text{Earth}}$ are characterized by different resonance frequencies. The simple checking shows that the corresponding relative frequencies ν_r / ν_0 are proportional to $\cos\theta$ (Fig. 5b).

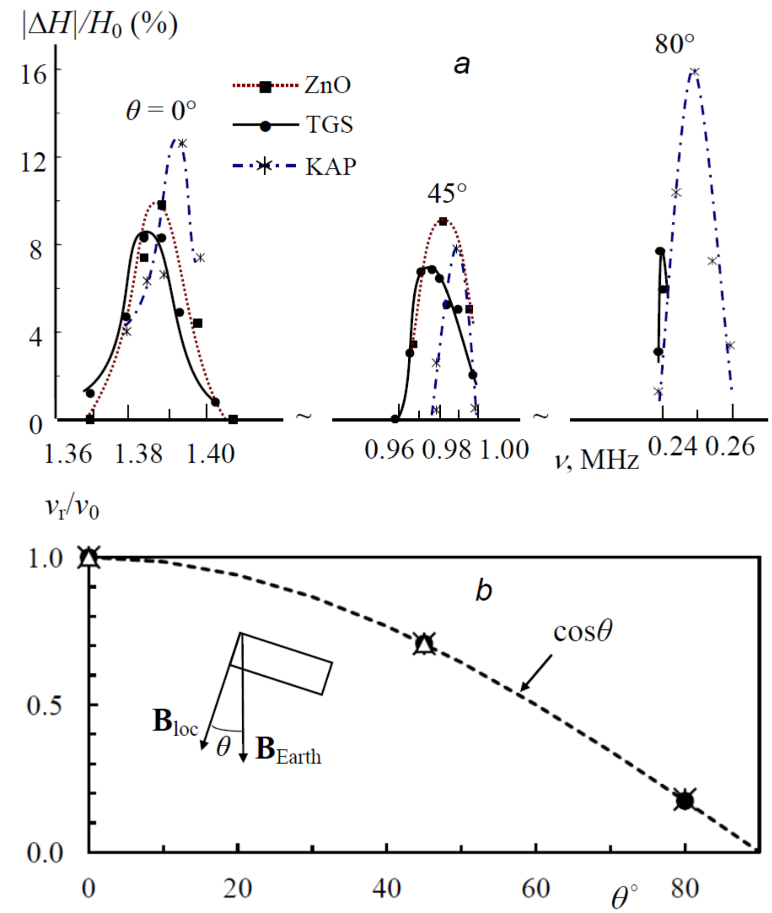

Fig. 5 (a) Resonance peaks of the microhardness changes $|\Delta H|/H_0$ for ZnO, TGS and KAP at three angles θ of the sample rotations by 0, 45° and 80° about the $\mathbf{B}_{\mathrm{Earth}}$ direction. (b) The relative resonance frequency ν_r / ν_0 versus the angle θ of sample rotation.

Thus, in complete analogy with the dislocation effect in *NaCl* (Fig. 3) a giant anisotropy of the effective *g*-factor: $g_{\mathrm{eff}} \approx g\cos\theta$ is met again. This means that the observed anomaly is a general property of the studied low-frequency EPR.

As it was shown (Alshits 2018), the discussed effect probably arises due to local magnetic fields $\mathbf{B}_{\mathrm{loc}}$ in crystals, which have definite orientation, and rotates together with the sample. Ordinarily, the field $\mathbf{B}_{\mathrm{loc}}$ is by 2÷3 orders less than the static field $\mathbf{B}$ in standard EPR, so it produces the ordinary small anisotropy of the *g*-factor. However, in our case $\mathbf{B}_{\mathrm{loc}} >> \mathbf{B}_{\mathrm{Earth}}$ the situation is radically different. According to (Alshits 2018) the Zeeman's splitting of the magnetic energy level is determined by

$$h\nu_r = g\mu_B(|\mathbf{B}_{\mathrm{Earth}} + \mathbf{B}_{\mathrm{loc}}| - B_{\mathrm{loc}}), \qquad (5)$$

where for $B_{\mathrm{loc}} >> B_{\mathrm{Earth}}$ one has

$$|\mathbf{B}_{\mathrm{Earth}} + \mathbf{B}_{\mathrm{loc}}| - B_{\mathrm{loc}} \approx \sqrt{B_{\mathrm{loc}}^2 + 2\mathbf{B}_{\mathrm{Earth}} \cdot \mathbf{B}_{\mathrm{loc}}} - B_{\mathrm{loc}} \approx B_{\mathrm{Earth}}\cos\theta. \qquad (6)$$

Thus, we come to the observed dependence (2), which may be described by the effective *g*-factor $g_{\text{eff}} \approx g\cos\theta$.

5. CONCLUSIONS

The features of the resonance magnetoplasticity in ultralow magnetic fields have been studied primarily for *NaCl* crystals, which have been used for a long time as a convenient model objects for the extensive studies of the various properties of the magnetoplastic effect. This work concerns a new direction of studies where the exposure of crystals occurs in the EPR scheme with the use of the Earth's magnetic field and radiofrequency pumping.

It has been found that the observed resonance of dislocation mobility has new, very specific, and strongly anisotropic properties. The main such feature is the sensitivity of the mean path and the resonance frequency on the orientation of the sample and the directions of dislocations with respect to the crossed magnetic fields. Such sensitivity was not previously observed under the usual EPR conditions corresponding to the pump frequencies of 10 GHz and 150 MHz.

Pulse EPR has also been detected in the *NaCl* crystals in the Earth's magnetic field. The resonance peak of the paths appears in a narrow range of durations of a pump field pulse near 0.53 μs (Fig. 4a). At fairly large amplitudes of the pump pulse beginning with a threshold level of ~10 μT (Fig. 4b), an explosive coherent relaxation of the dislocation structure occurs: almost all fresh dislocations move in 0.5 μs to the same distances of ~100 μm as under harmonic pumping in a time of exposure of ~5 min.

The resonance change in the microhardness of the *ZnO*, TGS, and KAP crystals after their exposure in the same EPR scheme in the Earth's magnetic field indicates that this phenomenon and its specific anisotropic features are fairly general. It occurs in very different materials and is responsible for both in situ and in magnetic memory effects. In essence, this concerns a very wide range of relaxation processes that have magnetic resonance nature.

An important consequence of the existence of magnetic resonance transformations of defects is the recognition of the new dangers of modern technical civilization. The mentioned processes can occur uncontrollably in critical elements of constructions and devices, leading to their degradation, when weak background radiofrequency fields are imposed on the Earth's magnetic field. These are not abstract possibilities but are the real processes, e.g., the accidental observation in 1985 of mass displacements of dislocations in a *NaCl* crystal under the action of magnetic pickups because of click of a switch as described in (Alshits 1999). This observation initiated the study of the magnetoplasticity effect.

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