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Acoustic Detection of Resonance Plasticizing of LiF Crystals Under the Influence of Crossed Magnetic Fields in the EPR Scheme

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Abstract: For the first time, the possibility of acoustic detection of resonance plasticizing of non-magnetic crystals when exposed to magnetic fields in EPR scheme was demonstrated. It is experimentally observed the sharp leap of dislocation internal friction in LiF crystals in crossed magnetic fields: constant field B_0 = 340 μT and HF field $B_{\sim}$ = 10 μT at the frequency of 9.525 MHz, corresponding to the paramagnetic resonance condition $h\nu = g\mu_B B_0$ for g = 2 (h is the Planck's constant, g is the Lande factor, and μ_B is the Bohr magneton).

Keywords: Magnetoplastic Effect, Internal Friction, Dislocation, Resonance Plasticizing, EPR

1. Introduction

For three decades after the discovery of magnetoplastic effect (MPE) [1], extensive studies were carried out by some researchers [2, 3], including us [4, 5]. They showed that the basis of magnetically plasticizing (or hardening) of nonmagnetic crystals is rearrangement of the structure of impurity centers, namely pinning centers limiting the mobility of dislocations, as a result of the spin-dependent electron transitions in a dislocation–impurity system in a magnetic field.

In [6], it was first time predicted theoretically, and then shown experimentally [7] that the combined effect of the crossed constant B_0 and high-frequency $B_{\sim}$ magnetic fields can lead to resonance plasticizing of nonmagnetic crystals, if the frequency ν of the alternating field satisfies the electron paramagnetic resonance (EPR) condition:

$$h\nu = g\mu_B B_0,$$

where h is Planck's constant, μ_B is the Bohr magneton, and g is the Lande factor.

In this case, the change of the spin states is not due to evolution in a constant magnetic field, but by means of resonant transitions between Zeeman levels.

Almost all MPE experiments were performed by standard methods that are associated with the movement of dislocations over macroscopic distances and irreversible plastic deformation. Only a very small part of research (and under the influence only of a constant magnetic field) was conducted by acoustic methods [8 – 10], i.e. the internal friction (IF) method, when the dislocation deformation is reversible and dislocation displacement is not more than 0.01 μm, whereas in the above mentioned methods this value is around 10 – 100 μm.

This work presents the results of the detection of acoustic resonance plasticizing of LiF crystals under the influence of crossed magnetic fields in the EPR scheme.

2. Experimental Technique

The LiF samples of required size were cut from the block along the {100} cleavage planes, then annealed at 800°C for 6 h and slowly cooled in the furnace.

Magnetosensitive samples were selected for acoustic experiments from crystals of different origins and impurity composition. The analysis of these crystals showed that they contained divalent impurities: Mg (11 ppm) and Pb (58 ppm).

The indentation was carried out by a diamond Vickers pyramid. Then the sample's surface was chemically etched to identify dislocation patterns occurring during the indentation. Thereafter, we measured the dislocation rosette ray length l and the diameter d of the indentation. The microhardness H was calculated from the formula

$$H \approx 1.854\, P / d^2,$$

where P is the load on the indenter.

After the initial microhardness measurements, the samples were incubated for 20 min in the crossed magnetic fields and then etched; with the constant, B_0 = 340 μT, and high-frequency (HF), $B_{\sim}$ = 10 μT, magnetic fields. The frequency varied in the range of 9.2 – 9.8 MHz.

At these frequencies, we observed intense shifting of etch pits corresponding to the only edge components of dislocation loops c (Figure 1), which are perpendicular both to directions of B_0- and $B_{\sim}$-fields. Edge dislocation a, which is parallel to B_0-field and dislocation b, which is parallel to $B_{\sim}$-field, do not move at all.

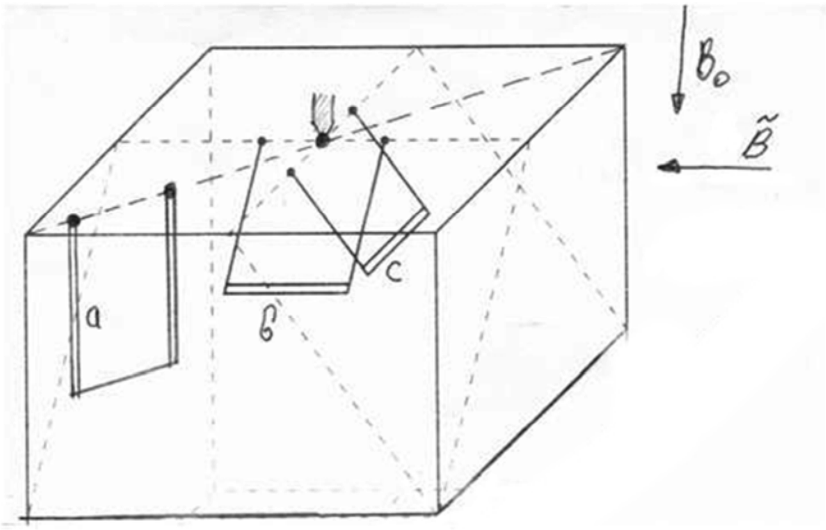

Figure 1. *The scheme of dislocation loops formed in the slip planes of LiF crystal at indentation. A double line indicates the edge portions of dislocation loops. The arrows indicate the direction of the constant B_0 and the alternating $B_{\sim}$ magnetic fields.*

As previously observed in [11], the effect depends on the orientation of the line dislocation relative to B_0- and $B_{\sim}$-vectors. Thus, preliminary experiments indicate that LiF crystals examined in crossed magnetic fields (in the EPR scheme) have a resonant movement of dislocations.

To quantify the observed effect, microhardness measurements were used. Figure 2 shows that the microhardness is least at a frequency of 9.525 MHz, which corresponds to the above condition of the paramagnetic resonance for $g = 2$.

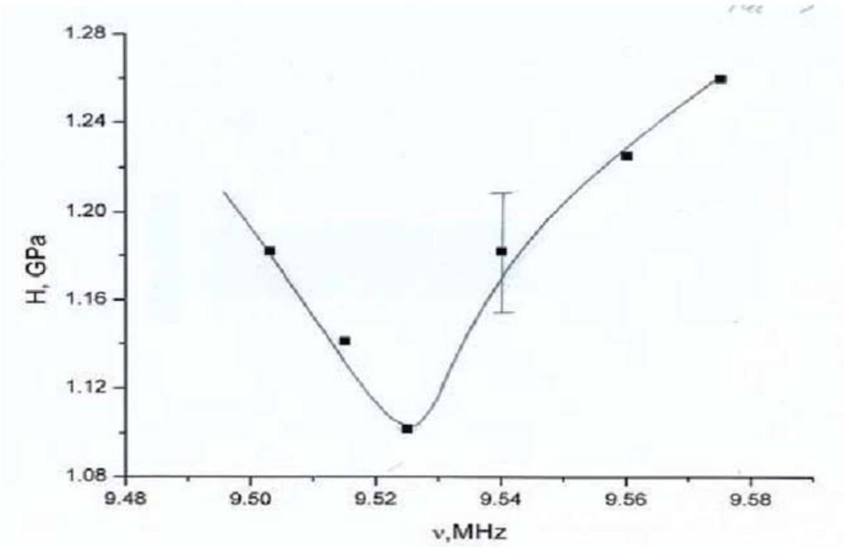

Figure 2. *The dependence of the microhardness of LiF on frequency of alternating magnetic field $B_{\sim}$ crossed with the constant field B_0 in the EPR scheme.*

After selecting magnetosensitive crystals, we started acoustic experiments. Figure 3 shows the experimental scheme.

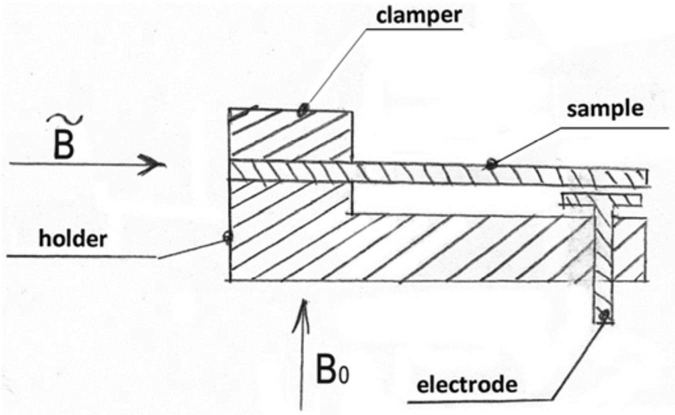

Figure 3. *Scheme for the measurement of internal friction in the crossed magnetic fields, B_0 and $B_\sim$.*

The samples were fixed in a holder, which is placed in a vacuum chamber. The constant magnetic field B_0 is created by the Helmholtz coils located outside the vacuum chamber, and is directed perpendicular to the sample large planes (001). The coil generates a HF field $B_\sim$ with frequency of $\nu = 0.5 - 10$ MHz, inside the vacuum chamber. The $B_\sim$-field is directed along the plane (001) and perpendicular to the B_0-field.

For the measuring of the internal friction, we have developed an acoustic spectrometer [12] based on the resonance method of the vibrating reed with electrostatic excitation of vibrations. The device allows measurements in continuous mode when the amplitude of the oscillations of the sample is stable at the predetermined level.

The most important problem of acoustic measurements is the increase in the mechanical Q factor of a vibration system. The main channels of parasitic dissipation of the mechanical vibration energy are the losses on the external friction between the sample and its clamp that can be $1/Q^{-1} \approx 10^{-3}$. These losses limit the method's sensitivity and are unacceptable in the cases when the lower background dissipation is necessary.

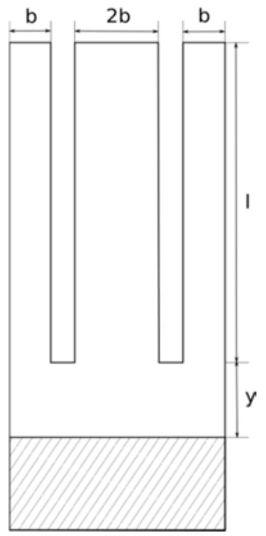

Figure 4. *Three-reed tuning fork.*

To decrease the instrument dissipation background and the dependence of the resonator vibration parameters on the holder properties, we used a three-reed tuning fork as a sample under study (Figure 4). The new-type tuning fork [13] is a plane-parallel plate with three tongues of the same lengths. In this case, the middle reed has a doubled width as compared to the width of the edge tongues.

As bending vibrations of any one of the reeds are excited, other reeds also start to vibrate in the direction perpendicular to the plate plane; the edge reeds vibrate in phase to one another and out of phase to the middle reed. Each of the tongues is a quarter-wave vibrator on thebending vibrations at the first-harmonic eigenfrequency:

$$f_0 \approx (0.162\, a / l^2)\, (E / \rho)^{1/2}$$

(E and ρ, respectively, are Young's modulus and density of the material).

The tuning forks were pricked out (cut out) along planes (100) from the 35 mm × 15 mm plates with thickness of 1.5 mm; the middle reed width was $2b = 4$ mm, the width of the edge reeds was $b = 2$ mm, and the reed length was $l \approx 20$ mm. The slot width was determined by the diamond disc used for cutting and was 1 mm. At these sizes, the LiF crystal has the first-harmonic frequency $f_0 \approx 3$ kHz. Varying the reed thickness and length, we were able to vary the eigen frequency of the sample vibrations within the acoustic diapason.

The tuning fork was clamped on a cantilever for its base between two claiming blocks (Figure 3). We had found empirically that the claim line must be at the distance $y \geq 2b$ from the reed bases (as was mentioned $2b$ is the middle reed width). Since the tuning fork base vibration amplitude was almost zero, the friction losses in the claims were minimal, and the mechanical Q factor of the vibration system was determined by the properties of the sample itself. As the tuning fork was clamped, the deformation caused by the clamping blocks was localized in its base (the dashed region in the Figure 4) and did not reach the sample (reed) itself. As a result, the same sample can be claimed as many times as is wished without risk to be damaged; in this case, the minimum background damping is provided reproducibly.

The vibrations are excited and recorded using a plane electrode with the diameter equal to the middle reed width disposed (Figure 3) at distance $d \approx 0.1$ mm from the middle reed surface (near to its edge).

The vibrations are excited by simultaneous applying to the electrode of direct current (DC) polarizing voltage V_P and exciting voltage $V_0 \sin 2\pi\nu t$ at a frequency equal to the eigenfrequency of the sample vibrations.

3. Results and Discussion

Before the acoustic measurements, the indentations were made with the indentation load 2N (~ 100 times) to create fresh dislocations.

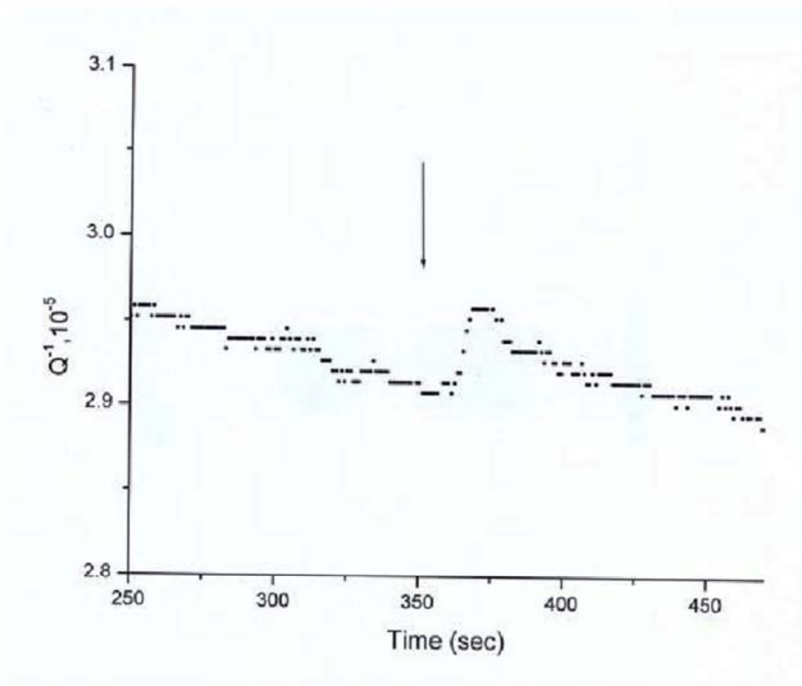

Figure 5. *The dependence of the internal friction on the time after deformation LiF crystal. The arrow marks the time of inclusion of crossed magnetic fields in the EPR scheme.*

Vibrations of the crystal were excited at small strain amplitudes. Then constant field B_0 = 340 μT was applied to the crystal; the effect of this field was not observed. When switched on (crossed with B_0) HF field $B_{\sim}$ = 10 μT at a frequency f = 9.525 MHz corresponding to the paramagnetic resonance condition, there was a jump of IF, as shown in Figure 5.

The effect was irreversible: crystals did not respond to the disconnection and reconnection of the fields. To regain the effect it was necessary to re-introduce fresh dislocation.

To explain the experimental fact, we turn to the mechanisms of dislocation IF. According to the Granato–Lücke model [14], at small strain amplitudes (in the amplitude-independent region of the IF), the main contribution to IF brings dynamic losses during vibration dislocation segments between the points of fixing (pinning centers). When (for whatever reason) depinning dislocation happens, dislocation segments oscillation amplitude increases and therefore dramatically increases the dissipation (amplitude-dependent region of the IF).

It is natural to assume that the observed increase in the dissipation occurs due to magnetically induced detachment of dislocations from pinning centers. The physical cause of the observed effects is the transformation of the structure of impurity centers. The mechanism of this transformation in alkali-halide crystals looks like this [11, 15].

In the crystal, bivalent metal atoms were in the form of magnetically inactive Mg^{2+} ions. At the approach of the dislocation, these ions are activated by grabbing electrons from anions that are on the edge of the extra plane. Since Mg^{+} ion and F^{0} atom contain unpaired electrons, a radical spin pair is formed (spin nanoreactor):

$$Mg^{2+} + F^{-} = Mg^{+} + F^{0}.$$

In crossed fields (in the scheme EPR) microwave pumping at Zeeman transitions singlet pair translates to the triplet state; thus dramatically increasing the lifetime of the spin nanoreactor without Coulomb interaction. As a result, at this frequency occurs depinning dislocations, resulting in increase in the IF. The small dissipation jump, $\Delta = 0.05 \cdot 10^{-5}$, can be explained by two factors:

1) The density of dislocations involved in the MPE is insufficient (evaluation of the full amount of dislocations formed by local deformation, gives a value of about10^{5} cm^{-2}); and

2) The impurities of divalent metal, existing in the crystal, may cause magnetoplastic effect of different signs [5]. Since Mg causes plasticizing of the crystal and the Pb strengthens it. As a result of these competing processes, the cumulative effect is small.

4. Conclusions

Thus, in the present study we demonstrated for the first time the possibility of acoustic detecting of resonance plasticizing of non-magnetic crystals under the action of the crossed magnetic fields in EPR scheme.

Acknowledgements

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