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INTERNAL FRICTION OF DEFORMED NaCl

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Résumé. - Le frottement interieur des monocristaux de NaCl déformés dans la direction <100> a été mesuré en fonction de la contrainte d'écoulement, de l'amplitude de vibration, de la fréquence, de la température et du dopage bivalent. Après la déformation de NaCl pur, on observe un maximum de frottement interieur PI aux basses températures. Avec un dopage croissant de Sr++, un second pic P2, moins prononcé, apparaît aux hautes températures. Les variations de PI dues aux paramètres utilisés sont typiques d'une relaxation de Bordoni. La tension de Peierls estimée pour le système de glissement (110)<110> se compare bien avec celle obtenue pour la limite élastique. P2 est dû à l'interaction dislocation-impureté.

Abstract. - Internal friction of deformed <100> oriented NaCl single crystals has been measured as a function of flow stress, strain amplitude, frequency, temperature and divalent doping. In pure NaCl after deformation a maximum of internal friction PI is observed at low temperatures. With increasing Sr++-doping a second but much weaker peak P2 appears at higher temperatures. The dependencies of PI on the parameters used are typical for a Bordoni-relaxation. The Peierls stress estimated for slip on the (110)<110> system compares well with that obtained from yield stress measurements. P2 is due to impurity-dislocation interaction.

1. Introduction. - The measurement of internal friction of deformed crystals is one of the most sensitive methods of studying dislocation dynamics. Among the internal friction phenomena related to dislocation motion much attention has been focussed on the interaction between dislocations and the lattice (Peierls barrier) [1] and between dislocations and obstacles like solute atoms etc. [2]. Compared with metals only a few papers have been devoted to the study of internal friction in ionic crystals [3 to 10]. In these materials below room temperature two peaks have been observed which have been discussed in terms of the Bordoni relaxation and dislocation - impurity interaction, respectively. However, because of undefined purity of the crystals used, the classification of the peaks to one or the other mechanism is rather controversial. It is the main purpose of the present work to study the effect of divalent doping on the relaxation peaks in NaCl in order to clarify this question.

2. Experimental. - "Pure" (Merck, pro analyti) and Sr2+-doped NaCl single crystals have been grown by the Kyropoulos-method. From these crystals <100> oriented specimens (5 x 5 x max. 50 mm3) have been obtained by cleavage. To get a defined starting condition all specimens have been annealed on a platinum foil in air at 920 K for 24 h and afterwards furnace cooled. The deformation of the samples was performed at room temperature in dynamic compression using an Instron-machine. The average strain rate was about $\dot{\varepsilon} \approx 10^{-4}$ sec$^{-1}$. The internal friction of undeformed and deformed crystals has been measured by a composite resonator technique using a piezoelectric quartz glued onto the specimen with "Devcon" two component resin. The apparatus used has been described in detail by Alber [11]. The internal friction has been measured in the strain amplitude independent regime at temperatures between 95 and 300 K. Special care had to be taken with the cooling rate (<1 K/min) and the use of small strain amplitudes ($\varepsilon \approx 3 \times 10^{-3}$) and thin layers of glue ($\approx 50 \mu$m) in order to avoid crack formation in the specimens, especially in doped crystals. The dopant concentration has been estimated from the yield stress-concentration relation measured by Skrotzki [12]. All concentrations are given in mole ppm.

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3. Results. - The internal friction has been measured in \(<100>\) oriented NaCl single crystals as a function of plastic deformation, strain amplitude, frequency, temperature and \(\text{Sr}^{2+}\)-concentration. For "pure" (undoped) NaCl after plastic deformation a damping peak \(P_1\) is found at low temperatures. This peak has a high-temperature flank reaching up to room temperature (Fig. 1). With increasing doping a second peak \(P_2\) appears at significantly higher temperatures superimposed onto the high-temperature flank of \(P_1\) (Fig. 2). The characteristics of both peaks will be described below.

**Fig. 1.** - Internal friction curves of "pure" NaCl crystals for different flow stresses.

**Fig. 2.** - Evolution with flow stress of the internal friction spectrum in 67 ppm \(\text{Sr}^{2+}\) doped NaCl. Measurement c had to be stopped because of crack formation.
PI:

- The peak height of P1 obtained by subtraction of the background damping increases with flow stress (Fig. 3).
- For a given flow stress $Q^{-1}_{\text{max}}$ decreases with dopant concentration (Fig. 3).
- In "pure", weakly deformed specimens the peak height has been found to increase with increasing oscillation amplitude.
- The peak temperature $T_{p1}$ is independent of flow stress.
- $T_{p1}$ increases with increasing dopant concentration (Fig. 4).
- The peak temperature increases with frequency according to an Arrhenius-law with an activation energy $E = 0.09$ eV for "pure" and 0.11 eV for 60 ppm Sr$^{2+}$ doped NaCl; the attack frequency $f_0$ is about $2 \times 10^9$ sec$^{-1}$ (Fig. 5).

P2:

- The peak height of P2 obtained by subtraction of the high-temperature flank of P1 increases with increasing flow stress (Fig. 6).
- For a given flow stress with increasing dopant concentration $Q^{-1}_{\text{max}}$ runs through a maximum.
- The peak temperature $T_{p2}$ decreases with increasing flow stress (Fig. 7).
- For a given flow stress within the large scatter of the data $T_{p2}$ shows a slight increase with dopant concentration.
- $T_{p2}$ increases with frequency.
- Because of the small height of P2 decent results could be obtained only by cumulative straining of the same specimen. However, easy crack formation in the doped samples prevented measuring more than three data points. Nevertheless the scatter in the data of P2 is much larger than for P1.

![Graph](image_url)

Fig. 3. - Variation of the peak height of P1 as a function of flow stress for different dopant concentrations.
Fig. 4. - Increase of the peak temperature of PI as a function of dopant concentration.

Fig. 5. - Frequency dependence of the peak temperature presented in an Arrhenius plot. Open data point is from Taylor [3].
Fig. 6. - Peak height of P2 as a function of flow stress for different dopant concentrations.

Fig. 7. - Peak temperature of P2 as a function of flow stress for different dopant concentrations.
4. Analysis and discussion. - In broad outline the features of the peak P1 resemble those observed for the Bordoni peak in metals [1]. Therefore the thermally activated relaxation process leading to P1 may be interpreted in terms of the Peierls mechanism, i.e. double kink formation during dislocation motion. This process can give rise to an internal friction phenomenon under the action of a stress alternating at a frequency \( f \). The internal friction will be a maximum if the frequency \( f \) is of the order of magnitude of the frequency \( \nu \) of the formation of double kinks:

\[
\nu = \nu_0 \exp \left( - \frac{H}{kT} \right)
\]

with an activation enthalpy \( H \) given by

\[
H \approx b^2(\sigma_P E_L)^{1/2}
\]

(\( b \) = magnitude of the Burgers vector, \( \sigma_P = \) Peierls stress and \( E_L = 1/2 \mu b^2 = \) line tension, \( \mu = \) shear modulus) [13]. Taking \( H = E = 0.09 - 0.11 \) eV eq. (2) yields \( \sigma_P = 5 - 7 \) MPa for slip on \((110)<110>\). This value agrees well with that extrapolated to \( T = 0 \) from yield stress measurements: 10 - 11 MPa [14]. However, because of the small frequency range covered (50 - 170 kHz) this result should not be overstressed. For LiF and MgO the frequency dependence of \( T_{\text{max}} \) has been measured over 5 orders of magnitude [3 to 10]. However, for different frequencies limited by the experimental techniques used, crystals have been used from the same companies (LiF: Harshaw, MgO: Norton) but most probably of different purity as is indicated by the scatter of the data at the same frequency. An estimate of \( \sigma_P \) from these results gives 9 MPa for LiF and 19 MPa for MgO to be compared with 17-20 MPa and <60 MPa, respectively, obtained from yield stress measurements [15]. Because of the effect of doping on \( T_{\text{max}} \) these values only give the order of magnitude. To facilitate the interpretation of further studies on the internal friction of ionic crystals more attention has to be put on the purity of the materials used.

The characteristics of P2 suggest this peak to be caused by thermally activated overcoming of solute ions by dislocations. However, insufficient data as well as the large scatter do not permit a quantitative analysis of P2. To get detailed information about solid solution hardening in ionic crystals from internal friction measurements either other methods or other materials have to be used in order to avoid crack formation.

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