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Some features of indentation creep of LiF single crystals

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The dependence of the size of the indentation and dislocation rosettes on loading time was investigated on the (001) plane of LiF single crystals. The measurements were performed in temperature range from room temperature to 170° C. The indentation time was varied from 0.2 to 10^3 sec. It was revealed that the change of the indentation size during creep was more significant than the change in dislocation ensemble tracks in the field of the concentrated load. It was shown that the dependence of the length of the dislocation rosette edge arms on loading time, when plotting in double logarithmic scale, was linear. This fact allowed the determination of parameter *m*, characterizing the dependence of the dislocation velocity on stress, using creep experiments. The values of *m* proved to be in good agreement with the results obtained by different methods.

1. Introduction

Two stages in the indentation process of different materials can be distinguished – the formation of the initial indentation (or "zero-time indentation"), and the development of the indentation, depending upon the indentation time (creep). The first process is relatively independent of temperature, while the second is strongly temperature dependent [1]. The investigations performed on alkali-halide crystals with an NaCl lattice, showed a two-stage formation of the dislocation structures which developed around the indentation [2]. Thus, it is shown [2] that almost three-quarters of the dislocation rosette size arises during the first second of the concentrated load action. Microhardness is usually believed to be a less sensitive characteristic than the length of the dislocation rosette arms [3]. This is true in some cases, as in the investigations of the influence of point defects on the mechanical properties [3]. However, an unexpected phenomenon was revealed for NaCl:Ca crystals – the indentation size increased markedly with increase in loading time, but the size of the dislocation rosette was practically constant [4]. Further work is necessary to elucidate the nature of this phenomenon. First of all it is of interest to establish the presence of such phenomena in other crystals. In the present paper, investigations of the dependence of the size of indentations and dislocation structures on loading time were performed for LiF single crystals.

2. Experimental procedure

Investigations were performed on the (001) cleavage plane of LiF single crystals. The equipment used, designed on the basis of microhardness tester PMT-3, allowed the required loading time and the deformation temperature to be carried out. The time of the deformation was varied within the range 0.2 to 1000 sec, the temperature was changed from 16 to 170° C, and the load on the indentor (*P*) was 100 g.

The dislocation rosettes near the indentations were revealed by means of the etch pit technique. The length of the edge and screw dislocation rosette arms (l_e and l_s, respectively) and also the width of the edge arms (*b*) were measured. These investigations were performed only up to ~115° C because at higher temperatures the dislocation rosette involved compact dislocation piling and a network of screw dislocation rows.

3. Experimental results and discussion

Fig. 1 shows the time dependences of the indentation size and parameters characterizing the dislocation rosette at room temperature and at 65° C; similar results were obtained at 115° C. As seen, an increase of all the characteristics under investigation takes place with increase in loading time. However, the indentation size varies monotonically (curves 1 and 2) but there are maxima and minima on the $l_e(t)$ curves, i.e. the increase of the rosette edge arms is non-monotonic. An anologous fact was revealed earlier for the curves characterizing the temperature dependence of the edge dislocation ensemble mobility in the stress field of the concentrated load [5, 6].

It is significant to note that the indentation increase was more intense than that of the dislocation rosettes, i.e. parameter *d* is more sensitive to change in loading time than the parameters *l* and *b* (Table I). Data

TABLE I The relative change in microindentation parameters due to change in loading time*

Deformation temperature (° C)	$\frac{\Delta d}{d}$(%)	$\frac{\Delta l_e}{l_e}$(%)	$\frac{\Delta l_s}{l_s}$(%)	$\frac{\Delta b}{b}$(%)	$\frac{\Delta H}{H}$(%)
16	24	17.1	21.6	17.8	35.5
65	36.4	46.5	16.1	24.2	46
115	27.6	10.5	6.1	23.0	38.8

*$\Delta d = d_{600} - d$, where d_{600} is the size of the indentation diagonal at 600 sec loading time, *d* is the same parameter corresponding to minimum loading time. This is true for other parameters.

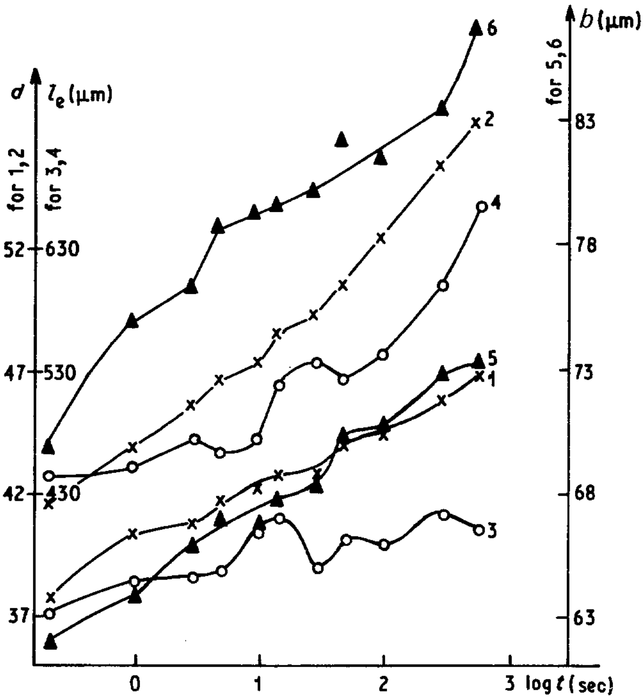

Figure 1 Dependence of the indentation size and parameters characterizing the dislocation rosette sizes on the loading time. Curves 1 and 2: d (indentation diagonal); Curves 3 and 4: l_e; Curves 5 and 6: b. T_D = 16° C for curves 1, 3, 5 and 65° C for curves 2, 4, 6.

presented in Table I show that the relative change of the indentation size (or microhardness H) is more considerable than the relative change of the dislocation rosette size ($\Delta l_e/l_e$ at 65° C is an exception). This result is in good agreement with the phenomenon observed earlier for NaCl crystals — the growth of the indentation diagonal was not practically followed by increase of the dislocation zone size [4]. It will now be of interest to check on the presence of such peculiarities for other crystals, to elucidate factors affecting this phenomenon; this is necessary for the understanding of its nature.

The microphotographs presented in Fig. 2 are a clear illustration of Table I, showing that the well marked increase of indentation size with increasing loading time from 1 to 600 sec is followed only by a relatively small growth of the rosette arm length.

TABLE II Dependence of microhardness and K on the indentation temperature

T (° C)	H_1 (kg mm^{-2})	H_{600} (kg mm^{-2})	K (%)
16	113	84.4	25.2
65	96.3	57.2	40.6
115	85	51.8	39.0
140	67.7	37.3	45.0
170	61.3	33.0	46.2

The dependence of microhardness on temperature for different loading times was plotted using the results obtained in the present study (Fig. 3). As is seen from Fig. 3, the loading time has practically no influence on the shape of the dependence $H(T)$ for LiF single crystals. However, the K ratio, characterizing the sensitivity of microhardness to the loading time ($K = (H_1 - H_{600})/H_1$, where H_1 and H_{600} are the microhardness values, corresponding to the loading times 1 and 600 sec, respectively) is dependent on temperature; it rises with temperature (Table II). It seems entirely natural, as the processes of dislocation slip, climb, multiplication etc., are activated by the increase in temperature. The formula characterizing the dependence of the dislocation rosette arm length from different parameters (these parameters involve the loading time) was obtained by Gridneva et al. [7].

$$l = CP^{m/(2m+1)} t^{1/(2m+1)} \exp\left[-U/kT(2m+1)\right] \quad (1)$$

Here C is constant, P is the load on the indentor, t is the loading time, U is the activation energy for the dislocation motion, m is a parameter characterizing the dependence of the dislocation velocity (V) on the stress (τ):

$$V = \beta(\tau/\tau_0)^m \exp(-U/KT) \quad (2)$$

According to Equation 1 a linear dependence between log l and log t must take place. Fig. 4 shows that such a dependence is roughly true for the edge dislocation ensembles in LiF crystals. The slope of lines presented in Fig. 4 permits m to be estimated. Its value was 18 at room temperature, and 10 at 65° C. These results are in good correlation with the values of m obtained by other methods [7, 8]. Thus, as shown by Gridneva

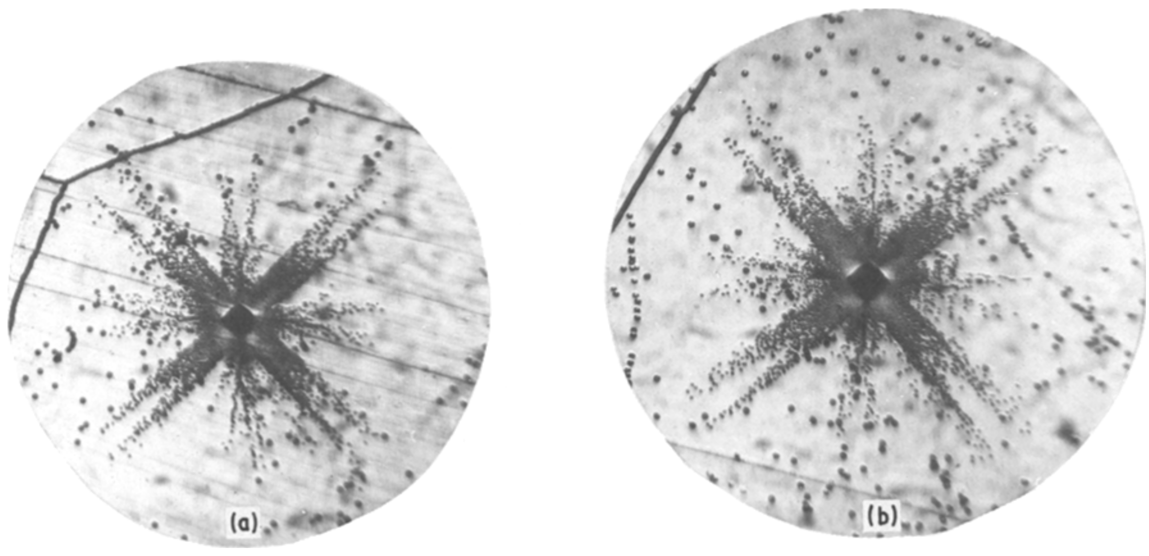

Figure 2 Indentations and dislocation rosettes on the (0 0 1) plane of LiF at the different loading times, t: (a) 1 sec; (b) 600 sec. T_D = 16° C.

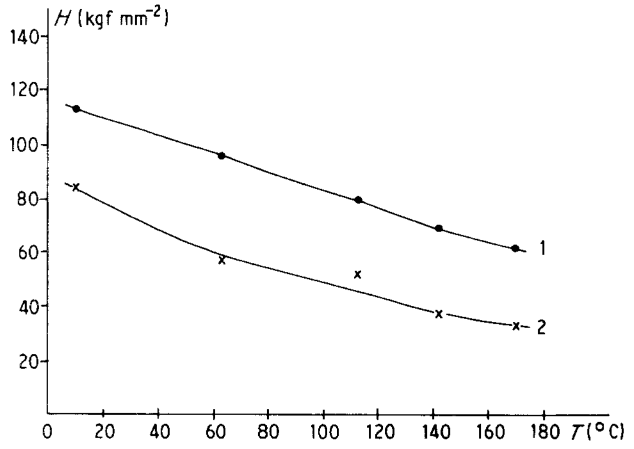

Figure 3 Dependence of microhardness on temperature at different loading times. Curve 1, 1 sec; Curve 2, 600 sec.

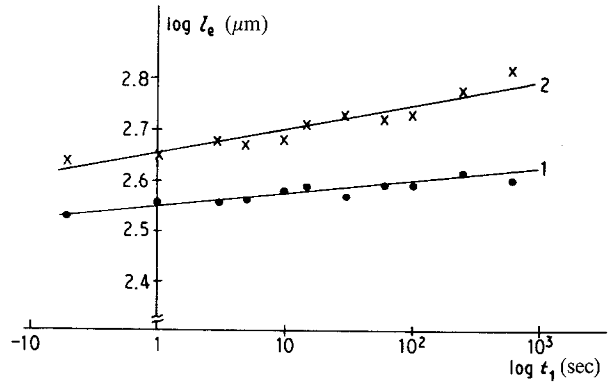

Figure 4 Dependence of the length of the dislocation rosette edge arm on the loading time. Curve 1, $T_D = 16°$ C; Curve 2, $T_D = 65°$ C.

et al. [7] m for ionic crystals was usually found in the interval 20 to 30; that for NaCl single crystals varied in the region 6 to 24, depending upon the doping of the samples and heat-treatment [8].

Thus our results for LiF single crystals confirm the theory of Gridneva et al. [7] concerning the formation of dislocation structures in an indentation; in particular the significant role of thermally activated processes.

4. Conclusions

1. It was revealed that for LiF single crystals the change in indentation size during creep is more significant than the change in dislocation ensemble tracks in the field of a concentrated load.

2. The indentation time does not effect the dependence of the microhardness on temperature. However, the K ratio, characterizing the sensitivity of microhardness to the loading time, is dependent on the temperature rising with its growth.

3. It is shown that the dependence between the length of dislocation rosette edge arms and the loading time, plotted on a double logarithmic scale, is linear.

4. The creep experiments in the indentation of LiF single crystals permitted the parameter m to be estimated (this parameter characterized the dependence of the dislocation velocity on stress). Its values were in good agreement with the data obtained by other methods.

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