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Indentation studies of alkali halides at elevated temperatures

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Abstract. The hardness of NaCl and KCl crystals has been estimated from the lengths of dislocation rosette formed around indentation at various temperatures up to 400°C. The hardness decreases with increasing temperature. This is due to the softening of the crystals at elevated temperatures which results in the easy movement of dislocations. The results are discussed using a few available relations which connect hardness to temperature. Arms of indentation dislocation rosette are well defined up to 300°C but around 400°C the rosette pattern is spread over a circular region. A possible mechanism is discussed.

Keywords. Mechanical properties; hardness; indentation; alkali halides.

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1. Introduction

Microhardness measurements on alkali halides at room temperature have been reported earlier by several workers (Berzina *et al* 1965; Chin *et al* 1972; Sirdeshmukh and Shah 1965). But there is very little work on the temperature variation of hardness. The variation of hardness of NaCl crystals with temperature up to 400°C was studied by Boyarskaya (1972). These results show that hardness decreases with increase in the temperature.

In view of the meagre data available a systematic study of temperature variation of hardness of some alkali halide crystals was therefore undertaken. The results on NaCl and KCl are reported here.

High temperature hardness measurements are difficult to make on microscope-attached instruments which may get damaged while heating the samples. Here the measurements are made using the instrument and method described in an earlier paper (Kishan Rao and Sirdeshmukh 1984, hereinafter referred to as I).

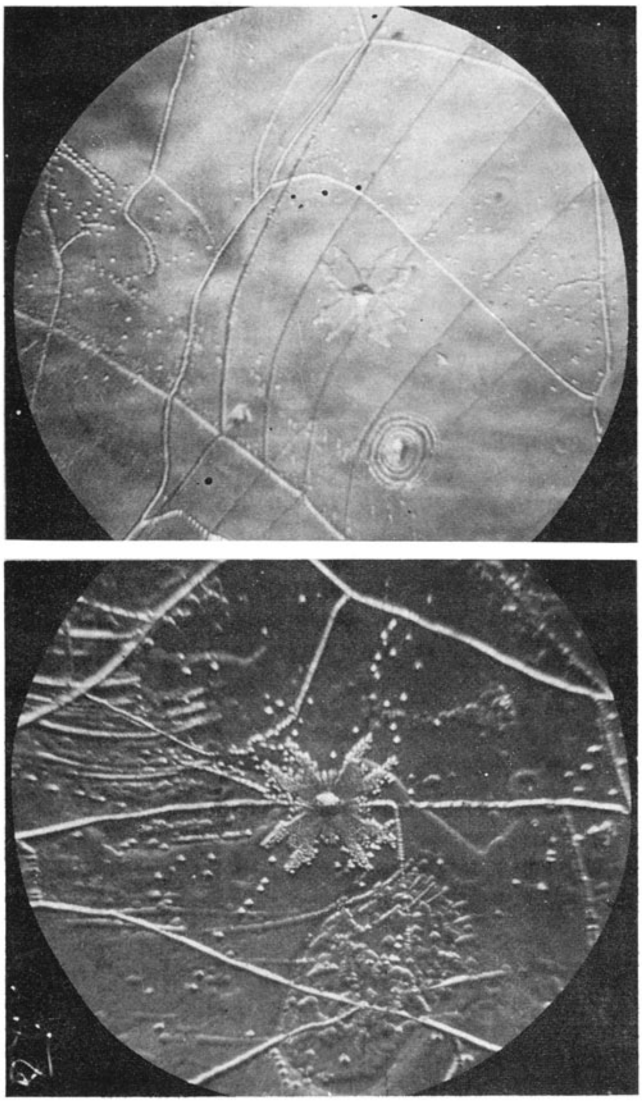
2. Experimental procedure

Measurements have been made using the instrument described in I suitably modified. Freshly cleaved samples of alkali halides are heated to the desired temperatures ranging from room temperature up to 400°C by surrounding the crystal holder by a tubular furnace. The temperature is measured with an accuracy of ± 2°C by a thermocouple attached to the sample holder very near the crystal. The crystals are kept at the required temperatures for about 2 hr. The indentations are then performed at a constant load of 20 g for 15 sec. The crystals are allowed to cool very slowly and are then etched. It is assumed that the dislocation movement caused by indentation at a higher temperature is not altered in the process of cooling because of the slow cooling. The arms of the rosette are measured as described in I and the hardness H_t at any temperature t is calculated in terms of the room temperature hardness H_{RT} from the rosette lengths l_t and l_{RT} using the relation

$$H_t = H_{RT}\, l_{RT}^2 / l_t^2. \tag{1}$$

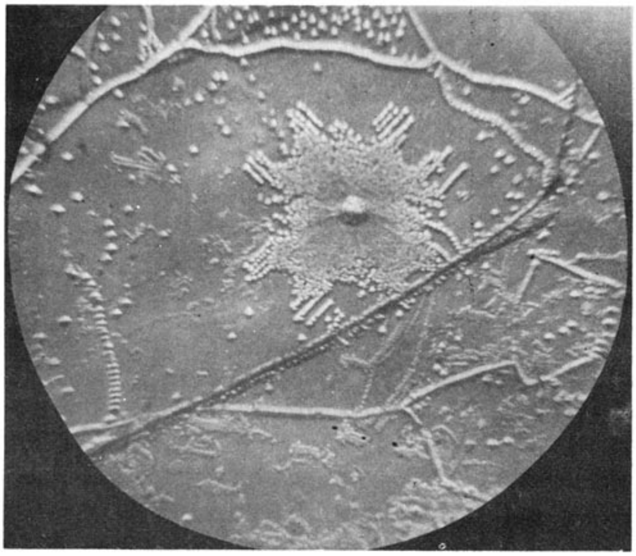
3. Results and discussion

Typical indentation dislocation rosette (IDR) patterns of NaCl at different temperatures are shown in figure 1. From these photographs it can be noticed that the arms of the

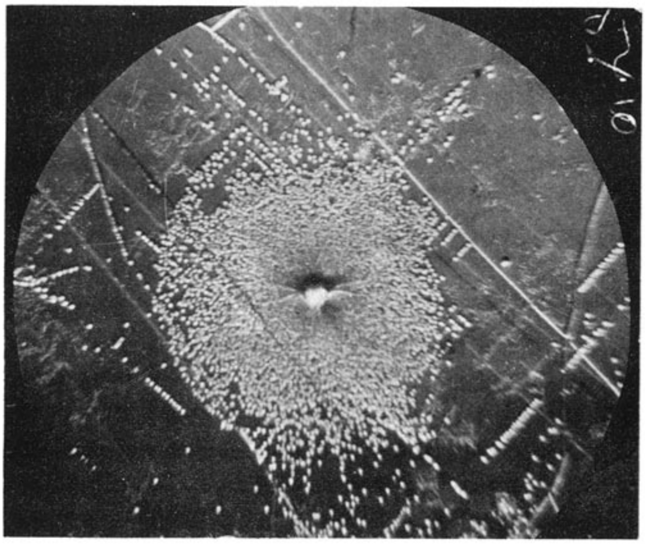


(a)

(b)



(c)



(d)

Figure 1. (× 100) IDR patterns for NaCl at various temperatures **a.** 30°C, **b.** 200°C, **c.** 300°C, **d.** 400°C. The arms of the rosette are in the ⟨110⟩ directions.

rosette increase with increase in the temperature at which indentations are made.

The hardness values at various temperatures are shown in table 1. It can be seen that the hardness decreases with increase in the temperature. The values obtained by Boyarskaya (1972) for NaCl are also given in the table for comparison. The temperature variation of hardness observed in the present work is qualitatively similar to that observed by Boyarskaya (1972). The rate of decrease of hardness with temperature is more in NaCl than in KCl. The decrease in the hardness can be explained as due to softening of the crystal at higher temperatures which results in easy movement of dislocations.

Table 1. Relative hardness of NaCl and KCl at different temperatures.

Temp. (°C)	Hardness (kg/mm^2) NaCl	NaCl*	KCl
30	22·1	19·4(20°C)	13·1
100	20·9	14·1	13·1
200	10·5	10·9	9·8
250			7·9
300	4·6	8·9	6·3
400	3·1	7·7	5·7

* Boyarskaya (1972).

Ito (1923) and Shishokin (1930) proposed an empirical relation

$$H = A\exp(-BT), \tag{2}$$

for the temperature variation of hardness of metals. Merchant *et al* (1973) proposed an Arrhenius equation

$$H = A'\exp(B'/T) \tag{3}$$

Gilman (1975) proposed the equation

$$H/H_0 = 1 - 2K\theta/U_0[\cot h(\theta/T) - 1] \tag{4}$$

for the temperature variation of microhardness of silicon. Here H_0 is the hardness of the crystal at $0°K$, K the Boltzmann constant, θ the characteristic temperature and U_0 the energy barrier for plastic flow at $0°K$. Equation (4) may be written in the linear form

$$H = A'' - B''\coth(\theta/T), \tag{5}$$

with $A'' = H_0 + 2K\theta H_0/U_0$,

and $B'' = 2K\theta H_0/U_0$.

The present authors are not aware of any attempt, theoretical or empirical, to interpret the temperature variation of hardness of the alkali halides. Since experimental data have been obtained in the present work for two alkali halides, an attempt is made to assess the applicability of the relations given above to the alkali halides. It can be seen from (2), (3) and (5) that linear plots should be obtained by plotting (i) $\log H$ vs T, (ii) $\log H$ vs T^{-1} and (iii) H vs $\coth(\theta/T)$. This has been done for NaCl and KCl. For plotting (iii) θ is taken as the Debye temperature and values of 281°K and 230°K have been used for NaCl and KCl respectively (Dekker 1969).

The three plots are shown in figures 2, 3 and 4. The plots are roughly linear with some scattering of data points. By least square fitting the values of slopes and intercepts have been obtained; these are given in table 2 along with their standard errors. It can be seen that for both the crystals the data fit (2) with least errors. Next comes (5). The fit with (3) is clearly the poorest. However, it is not possible to draw conclusions about the relative merits of these relations very forcefully in view of the limited accuracy of the present experimental method. More accurate data at still higher temperatures for larger

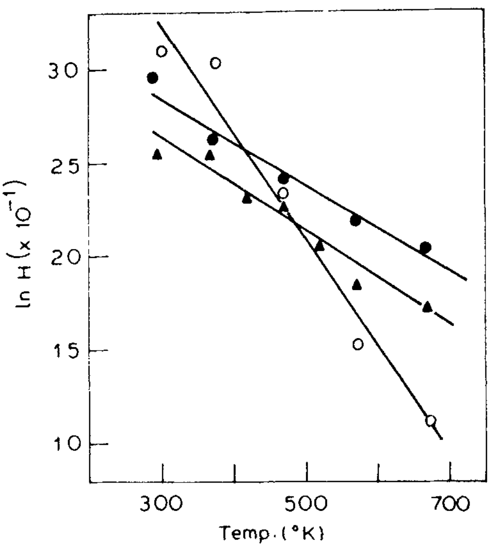


Figure 2. Plot of log H against temperature (T): o—NaCl present data, ●—Boyarskaya (1972), ▲—KCl.

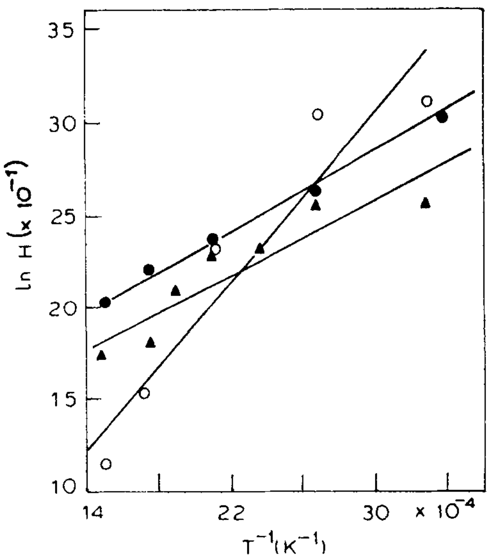


Figure 3. Plot of log H against $(1/T)$ (symbols as in figure 2).

number of alkali halides are desirable to decide between these equations. Equations (2) and (5) can therefore be considered to represent empirically the variation of the hardness with temperature for the alkali halides.

In figures 2–4, the hardness values for NaCl obtained by Boyarskaya (1972) are also included. These values also lie on a straight line although the slope is different from the

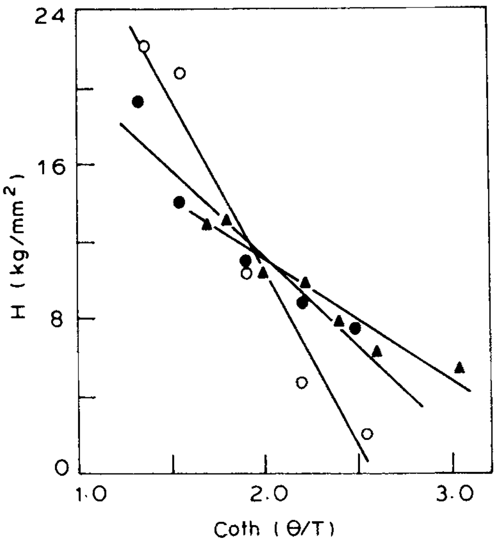


Figure 4. Plot of hardness (H) against cot h (θ/T) (symbols as in figure 2).

Table 2. Slopes and intercepts of the plots from figures 2, 3 and 4.

	log H vs T (figure 2)		log H vs T^{-1} (figure 3)		H vs cot h(θ/T) (figure 4)	
Crystal	Slope	Intercept	Slope	Intercept	Slope	Intercept
NaCl	0·0057 ±0·0004	5·02 ±0·29	1135 ± 235	0·35 ±0·55	42·76 ± 6·67	108·7 ± 13·1
KCl	0·0025 ±0·0002	3·431 ±0·140	1197 ± 92	1·08 ±0·21	14·30 ± 1·79	54·21 ± 4·17

plot obtained with the present values. However, since sufficient details about Boyarskaya's results are not available, the cause of this difference cannot be identified.

Another aspect of IDR pattern is now considered. Figure 1d shows the rosette pattern of pure NaCl when it was indented at 400°C. Apart from the increase in the arm length, the photomicrograph reveals the clustering of the etch pits around the indentation. Unlike the rosette patterns at lower temperatures, it is difficult to distinguish between the rows of the etch pits along ⟨110⟩ and ⟨100⟩ directions in the photomicrographs at higher temperatures. The rosette pattern is spread over almost a circular region. Generally, the rays of the star along ⟨110⟩ direction, which correspond to the edge dislocations, are longer than the rays of the star along ⟨100⟩ direction which are due to screw dislocations. This is due to the fact that the velocity of edge dislocations is higher than the velocity of screw dislocations. Here, the observed clustering of pits at 400°C over a circular area might be due to two reasons. Firstly, the mobility of the edge and screw dislocations seems to become comparable at elevated temperatures. Secondly, some new glide systems appear to be getting activated at elevated temperatures. Shah (1967) studied the dislocation etch pits around an indentation in bismuth crystal. He observed that the density of etch pits is high in the vicinity of the indentation mark, but falls off rapidly away from the indentation. However at any given distance from the indentation mark, the etch pit density is more or less the same all around the indentation even at room temperature. A similar tendency seems to develop in alkali halide crystals also at high temperatures. The clustering of pits will make the present method of hardness measurement more difficult and less reliable at still higher temperatures.

4. Conclusions

The microhardness of NaCl and KCl has been measured as a function of temperature from room temperature up to 400°C. In both crystals the hardness decreases as the temperature increases. The decrease in hardness is represented by the equation $H = A\exp(-B\tau)$. A study of the indentation rosette shows that the mobility of edge and screw dislocations tends to become comparable at high temperatures leading to a uniform clustering of etch pits around the indentation.

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