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Dislocation density in electrolytically-coloured KCl crystals

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Abstract. Dislocation densities have been investigated in potassium chloride crystals, electrolytically coloured in the temperature range of 550–710°C. The results show an increase in the dislocation density with coloration temperature upto 650°C and decrease thereafter. This is attributed to the movement of dislocations and interactions between them during electrolytic coloration of the crystals.

Keywords. Dislocation density; electrolytic coloration; coloration temperature; dislocation annihilation.

1. Introduction

It is well known that dislocations play an important role in the coloration of alkali halide crystals (Seitz 1954; Belyeav *et al* 1968; Koikov *et al* 1967). Dislocation densities in electrolytically-coloured alkali halides have been studied earlier (Deshmukh and Soman 1976; Kolomiitsev *et al* 1978). However, there is no work on the temperature variation of dislocation density in electrolytically-coloured crystals. An attempt is therefore made in this paper to study the effect of variation of coloration temperature on dislocation density in electrolytically-coloured KCl crystals.

2. Experimental procedure

Single crystals of KCl used in the present work were grown from melt by the Czochralski technique. Two crystals (each of size $10 \times 5 \times 5$ mm^3) obtained from the as-grown block were used in the experiments. One of them (to be electrolytically-coloured) was mounted between two platinum electrodes (one pointed electrode and the other flat) and the other was placed beside it in the coloration furnace. The temperature of the crystals was measured with an accuracy of ± 5°C by chromel-alumel thermocouple. The crystals were kept at the required temperature for nearly an hour. The crystal clamped between the electrodes was coloured with a pointed cathode by applying a DC electric field of 120 V. After coloration, both the coloured and uncoloured crystals were quenched by rapidly removing them from the furnace and placing them on the metal plate. In this manner the crystals were coloured at desired temperatures ranging from 550°C to 710°C.

The coloured and uncoloured crystals were cleaved from the central part of the crystals and etched with an etchant consisting of an ethanol (99%) saturated at 50°C with $PbCl_2$. This etchant is found to attack at dislocation sites on (100) faces of KCl crystals. The dislocation density was measured with a NU_2 research microscope on a number of crystal specimens and the average dislocation density was obtained at each temperature.

3. Results and discussion

It is observed that electrolytic coloration proceeds at a faster rate with increase of coloration temperature i.e. the efficiency of formation of colour centres increases with increase of coloration temperature. For example, the coloration over the entire crystal takes 4 min at 550°C and about 30 sec at 710°C.

Figures 1a and 1b respectively show the variation of dislocation density with temperature in KCl crystals for uncoloured and coloured crystals. The dislocation density varies linearly with temperature in uncoloured crystals and nonlinearly in coloured crystals. Dislocation density in coloured crystals reaches a maximum at 650°C and decreases on either side of 650°C. Figures 2a, b and c show the photomicrographs of dislocation patterns in KCl crystals electrolytically-coloured at 550°C, 650°C and 710°C respectively. Figure 2b shows the maximum dislocation density at 650°C.

The observed increase of dislocation density linearly with quenching temperature in uncoloured crystals can be attributed to severe surface stresses developed in the crystals with increasing quenching temperature (Rao and Hari Babu 1976; Chawre *et al* 1978).

For coloured crystals the coloration temperature range can be divided into two parts (i) coloration temperature from 550°C to 650°C in which the dislocation density increases with temperature and (ii) coloration temperature above 650°C in which dislocation density decreases with temperature. In the range 550–650°C, as the dislocation density increases linearly with coloration temperature the behaviour of the coloured crystal is similar to that of the uncoloured one and the increase in dislocation density can again be attributed to surface stresses. In other words, the electrolytic coloration has only a little effect on the increase of dislocation density upto coloration temperature 650°C. Its effects in decreasing the dislocation density in KCl crystals are, however, seen above the coloration temperature of 650°C.

Electrolytic coloration occurs at high temperature with large electric field gradient. Under these conditions, dislocations move inside the crystals. In the range of coloration temperature 550–650°C, the dislocations during their motion may take vacancies (possibly anion vacancies) with them thereby forming positive jogs in them.

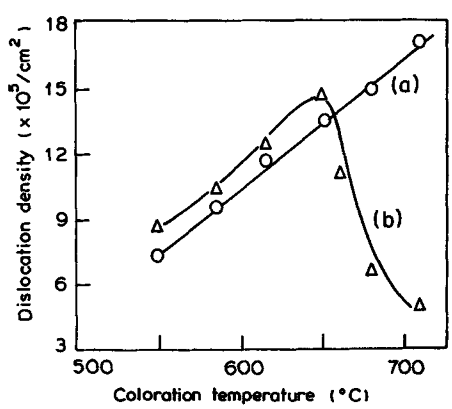

Figure 1. Variation of dislocation density with temperature in KCl crystals (a) uncoloured, (b) electrolytically coloured.

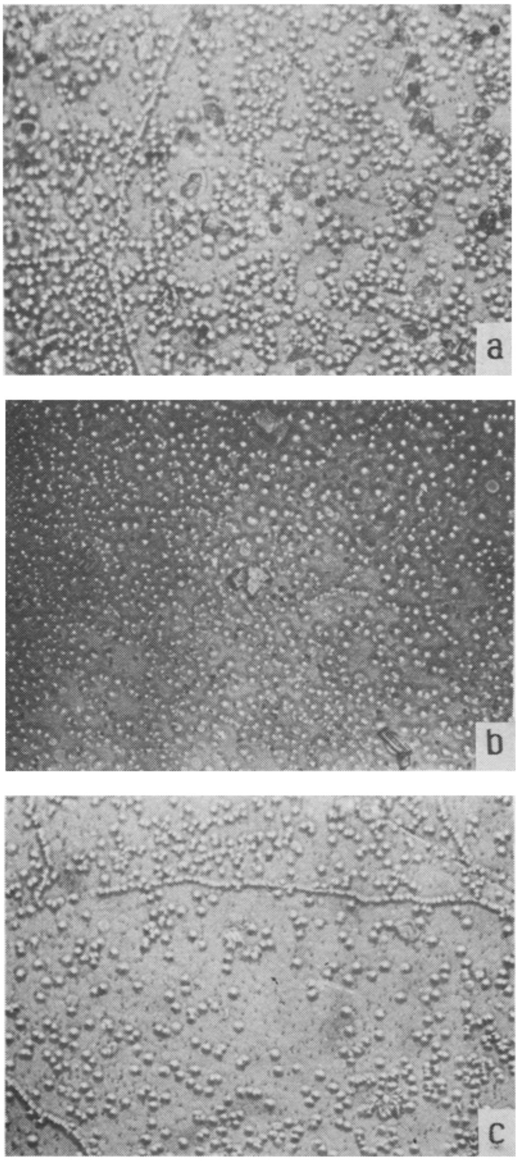

Figure 2. Photomicrographs of electrolytically coloured KCl crystals. **a.** Coloured at 550°C; **b.** Coloured at 650°C; **c.** Coloured at 710°C.

These anion vacancies may be formed due to the dissociation of F-centres by dislocations (Sierra and Cabrera 1975). This presence of jogs in the dislocation impedes its motion. The dislocations will thus move comparatively slowly which will lead to an increase in the dislocation density. The jogs in the dislocation may increase with increase of coloration temperature which will provide greater resistance to the motion of dislocation. As a result, dislocations will move slowly with increasing coloration temperatures thereby giving rise to an increase in dislocation density with increasing coloration temperatures.

In the range of coloration temperature 650–710°C the decrease of dislocation density can be attributed to the mutual annihilation of opposite edge dislocations. This might occur because, in this temperature range the dislocations become sufficiently mobile to cause their annihilation. The annihilation of opposite dislocations may result from the following two processes: (i) when two opposite dislocations glide on the same plane, their mutual interaction results in a perfect lattice; (ii) when two opposite edge dislocations glide on neighbouring planes, their interaction will leave a row of vacancies or interstitials. This row of vacancies or interstitials diffuse away under the conditions of high temperature and high electric field gradient of electrolytic coloration leaving a perfect lattice. This process of diffusion of vacancies or interstitials is energetically costly and thus occurs at high temperatures only. The above two processes contribute to the decrease in dislocation density above 650°C. Deshmukh and Soman (1976) observed such a decrease in dislocation density in alkali halides on electrolytic coloration. The process of annihilation of dislocations increases at increasingly high temperatures which results in decrease in dislocation density at temperatures beyond 650°C. At temperatures upto 650°C the energy supplied is insufficient for the diffusion of vacancies produced during the interaction of opposite dislocations. Hence the process of annihilation of opposite dislocations does not occur before 650°C.

4. Conclusions

Our results show that the dislocation density in the electrolytically coloured KCl crystals increases with coloration temperature upto 650°C and decreases thereafter. The following conclusions can be drawn: (i) The efficiency of electrolytic coloration increases with coloration temperature in KCl crystals, (ii) dislocation density in electrolytically coloured KCl crystals varies nonlinearly with coloration temperature, (iii) dislocations in electrolytically coloured KCl crystals are generated in the temperature range 550–650°C whereas they are annihilated in the temperature range 650–710°C.

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