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Surface finishing of alkali halides

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The production of smooth scratch-free surfaces on the alkali halides KCl and NaCl by means of solvent action is described. It is shown by etching studies that very few dislocations are introduced by this process. Surfaces of the type described are of particular value in the study of the optical absorption of very low loss materials.

1. Introduction

In this article attention is focused upon the production of high quality surfaces in the alkali halides NaCl and KCl which are among the best materials for high power laser windows [1]. Since the residual absorption in these materials at the laser frequency of 10.6 μm can be attributed in part to surface preparation, it is obvious that techniques for producing low absorption surfaces are of prime importance. In addition, such surfaces would have importance for optical measurements in other spectral regions as well as for dislocation studies. Conventional methods of surface preparation of alkali halides involving cleavage, grinding, and abrasive polishing leave much to be desired. In addition to the possibility of surface absorption, such procedures leave abrasive particles which can produce hot spots by local absorption of radiation, and scratches which can serve as sites for attack by moisture [2].

The approach involves a several step procedure for processing crystals to avoid the introduction of dislocations as much as possible. Shaping of these water-soluble crystals by water grinding followed by chemical polishing allows gentle treatment without the introduction of abrasives. Some chemical polishing involving weak organic acids has been described for NaCl crystals [3] and Chesters and Zwicker [4] have used flowing water in conjunction with baffles to polish KCl crystals. In this paper, aqueous solutions of HCl are shown to provide a controlled chemical polish for NaCl and KCl crystals. The end result is a scratch free surface showing very few dislocations.

2. Preparation of high quality surfaces on KCl and NaCl crystals

In this section the techniques developed for the preparation of high quality surfaces on NaCl and KCl crystals is described. Here the starting materials were 2.5 cm diameter boules straight from crystal growth. To avoid the introduction of dislocation damage, surface scratches, cleavage steps, internal strains, and particulate matter which might contribute to surface absorption, the following fabrication procedures were used:

(1) Sawing: A water-soaked string saw* has been found suitable to truncate the ends and otherwise section the boules.

(2) Grinding: Grinding to render the faces plane-parallel was performed by hand using a water charged Politex "Supreme" polishing prorometric lap† attached to a glass plate. The lap was treated with a wetting agent aerosol before initial use.

(3) Chemical polishing: This involves immersion of the crystal with the end-faces situated vertically in a hydrochloric acid polish for 30 min. It was then washed with a jet of ethyl alcohol and dried with a jet of dry air.

3. Studies of chemical polishing

3.1. Single crystals

Hydrochloric acid was found to be an excellent rate controllable polish for KCl and NaCl crystals. Since the alkali chlorides are the reaction products of HCl, they are chemically inert to this simple compound which can be readily removed from the surface by washing. To a first approximation this polish regulates their

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rate of dissolution, but it also seems to favour the chemical polishing mechanism of atomic layer removal over other mechanisms of dissolution.

An estimate of the dissolution rates of NaCl and KCl crystals in HCl polishing solutions was obtained by measuring with a micrometer the change in thickness (nominally 15 μm) produced by chemically polishing square cleavage slices (2 cm side) for a known period of time. The specimens were held in the polishing solution with forceps and agitated over a distance of 3 cm at 2 Hz perpendicular to the face. Dissolution rates of 10, 0.05, and 0.0005 $\mu m\ sec^{-1}$ were obtained for the agitated NaCl crystal in HCl polishing solutions consisting of the volume ratios $HCl:H_2O$ = 0:1, 3:1, and 1:0 respectively. The dissolution rates were reduced by the factor 5 when the crystals were not agitated. Agitation of KCl crystals in conc. HCl (volume ratio 1:0) yielded the rate 0.2 $\mu m\ sec^{-1}$. For the controlled chemical polishing of our boules we used the ratio (1:0) for KCl and (3:1) for NaCl with polishing times ranging from 10 to 30 min.

A comparison of abrasive polishing and chemical polishing has been carried out by microscopic examination of the surface and by transverse etching studies to reveal dislocations. In particular, the controlled chemical polish removed the scratches from mechanically polished surfaces as seen in Fig. 1. Mechanical processing techniques such as grinding or lapping with abrasive particles produced a damaged surface region, whereas water grinding produced a relatively damage-free surface as illustrated in Fig. 2. When these specimens were chemically polished, smooth relatively flat surfaces were obtained because the polish preferentially removed the stepped elevated regions.

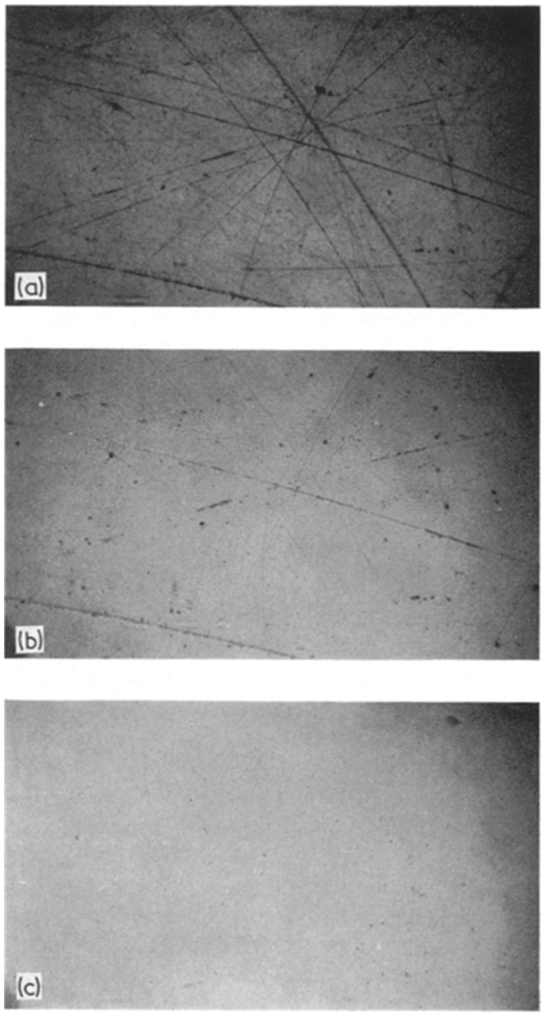

Figure 1 NaCl window (× 36). (a) Mechanically polished, showing surface scratches. (b) Chemically polished 10 sec, showing widened scratches. (c) Chemically polished 5 min, showing a smooth, scratch-free surface.

3.2. Polycrystalline material

Of technological importance is the preparation of laser windows from polycrystalline KCl. One way suitable surfaces might be produced is to use the controlled polish to remove only the scratch-containing layers from a flat mechanically-polished surface. To obtain insight on this problem the effect of the polish for various polishing times on a mechanically polished, press-forged KCl window was examined. The procedure is essentially the same as for single crystals but there is an optimum polishing time as illustrated in Fig. 3. If the time is too short, expanded abrasive markings are formed. If the time is too long, the grain boundaries are revealed. Under optimum polishing conditions, the surface flatness might not be seriously degraded.

4. Conclusions

The results of this work indicate that by proper surface finishing techniques involving controlled chemical polishing, it is possible to obtain surfaces in which little or no mechanical damage has been introduced. Furthermore, the surfaces are largely free of scratches. On the other hand it has been shown that surfaces obtained using

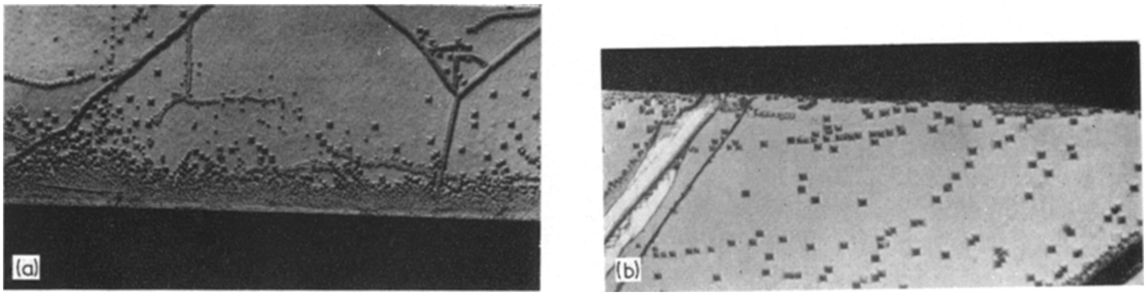

Figure 2 Surface damage in NaCl crystals. (a) Typical dislocation damage introduced by mechanical processing (lapping on glass plate with a 50 g cm^{-2} load using a slurry of no. 7 emery grit and isopropyl alcohol) transverse cleavage face ($\times$ 130). (b) Undamaged surface obtained by water grinding with no abrasive: transverse cleavage face ($\times$ 130).

usual finishing procedures involving abrasives yield surfaces containing scratches, abrasive particles, and dislocations. By employing crystals whose surfaces have been finished in this way, it is possible to reduce the absorption at 10.6 μm associated with the surface.

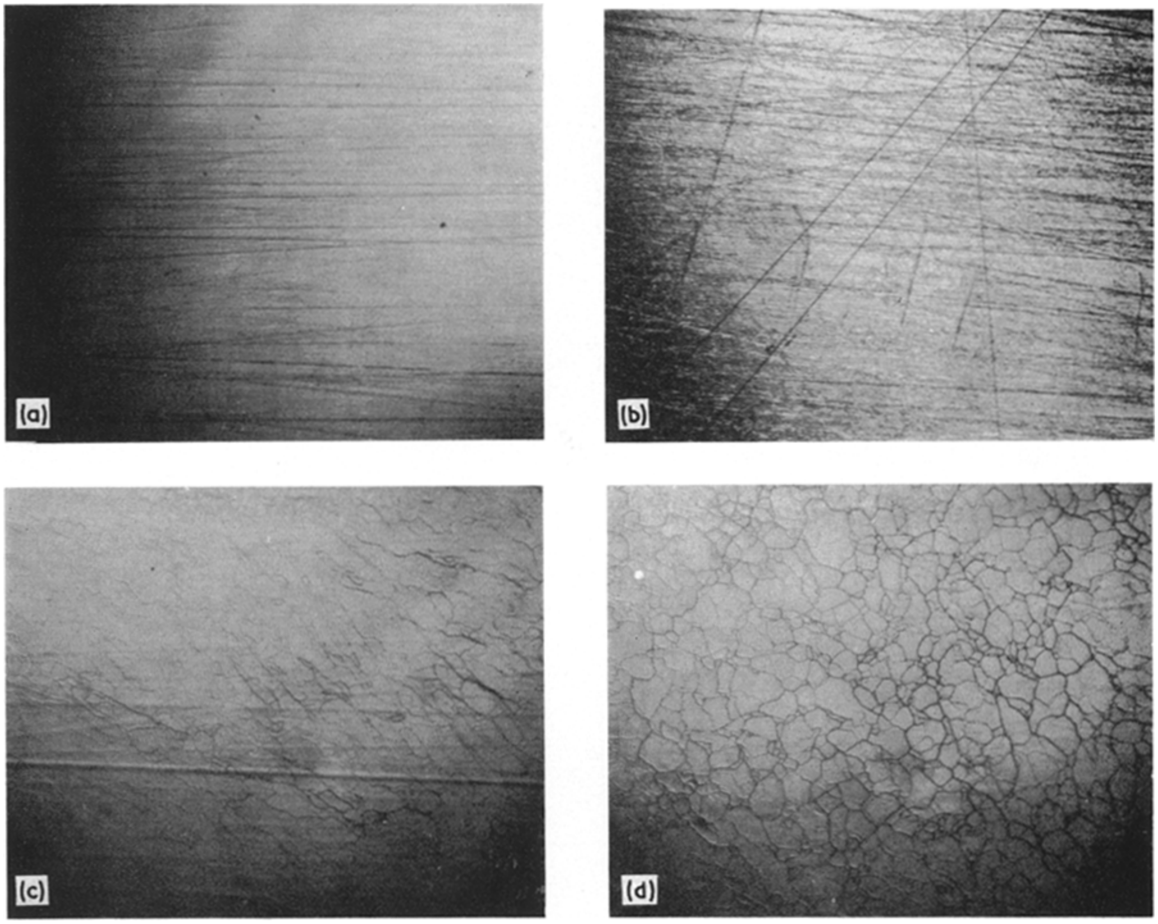

Figure 3 Chemical polishing of press-forged KCl ($\times$ 32). (a) Initial mechanical polish – showing surface scratches. (b) Insufficient chemical polish of forging (10 sec), showing widened scratches. (c) Optimum chemical polish of forging (25 sec). (d) Excessive chemical polish of forging (40 sec), showing grain boundaries.

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