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2.12 The sputtering of alkali halide surfaces with low energy irradiation

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In alkali halide crystals it is possible to transfer energy from incident electrons or photons to single lattice atoms with extremely high efficiency and produce defects in the material. Several models for the transfer process have been proposed and there is now strong evidence to support those of Pooley and Hersch^{1,2)}. Briefly their models proceed by (i) excitation of a halogen ion in the lattice to a higher state, (ii) movement of the central portion of the ion to an adjacent site with the formation of a molecular ion and (iii) a return to the new ground state. In this last stage the excess energy is imparted as kinetic energy to the molecular ion and is sufficient to dissociate the molecular ion and initiate a replacement collision sequence to separate the vacancy and interstitial. The collision chain is propagated along a line of similar atoms (in this case a $\langle 110 \rangle$ direction) and one expects that if the chain reaches the surface the last atom will be ejected with momentum along the $\langle 110 \rangle$ direction.

Alternative theories of Varley, Klick, Williams³⁾ would also produce ejection from the surface in the $\langle 110 \rangle$ direction but they require more energy for the first stage since they consider this as an ionisation or multiple ionisation of a

halogen ion. Successful sputtering experiments will be reported of KI bombarded with photons up to 5 eV. Electron induced sputtering of KI, NaCl, KBr, KCl and LiF has also been observed with electron energies from 15 to 2 keV.

The threshold energies are too low to support models which involve ionisation, however, they do not exclude the possibility that ionisation mechanisms also exist at higher energies.

One consequence of all these models is that halogen ions or atoms will be preferentially ejected in the $\langle 110 \rangle$ direction. The model do not explain how the alkali metal leaves the crystal. Two possibilities are that this takes place by surface diffusion or simple evaporation. Evidence will be presented which suggests both these mechanisms are operative. The first has been reported from Auger measurements on KCl which was partially coated with silver⁵. During electron irradiation the KCl decomposed and a potassium signal was observed from the top of the silver (i.e. diffusion). In our own experiments measurements of the pattern in the (100) plane show a maximum near the $\langle 110 \rangle$ direction superposed on a broad background. The relative size of the background increases with temperature which is consistent with the two processes; one of the halogen ejection, the other thermal evaporation of the metal. The ejected material is in the form of neutral atoms. There is a thermal activation energy for the total increase in sputtering rate which is of the order of 0.2 eV.

Support for the sputtering taking place in two stages, that is halogen ejection followed by alkali ejection, is also found from Auger studies of the decomposing surface. These show that the halogen is preferentially ejected until one reaches an equilibrium with an alkali rich surface. The rate of sputtering is then limited, one assumes, by the rate at which the alkali ions can be removed by thermal processes.

Because one is studying extremely low energy processes at the surface of the crystal the rate of sputtering is very sensitive to the initial surface condition and one observes a wide range of surface features. In addition the sputtering rate is strongly influenced by the history of the sample in cases where this produced a high dislocation concentration. Some of the features on the surface are common to both air cleaved and U.H.V. cleaved specimens and the topography also resembles that of ion beam irradiated specimens.

Since the Pooley-Hersch mechanism outlined above is relevant to the alkali halides but not to all insulators then any surface coating (e.g. pump oil) inhibits ejection from the crystal until one can remove this coating. This is different from the case of ion beam irradiation where surface contaminants merely give a different sputtering yield and one will eventually remove these and reach the specimen of interest. In both ion beam, electron and photon irradiation partial coating of the alkali halides produces a shadowing effect leading to the growth of large cones or spikes. This was predicted by Stewart and Thompson⁶. One property of such cones is that they always face directly into the beam independent of the relationship of the crystal axes to the beam direction. This result is observed for electron sputtered alkali halides. Such cones may extend to the original surface of the crystal until the shielding material is removed. In extreme cases cones as high as 100 microns have

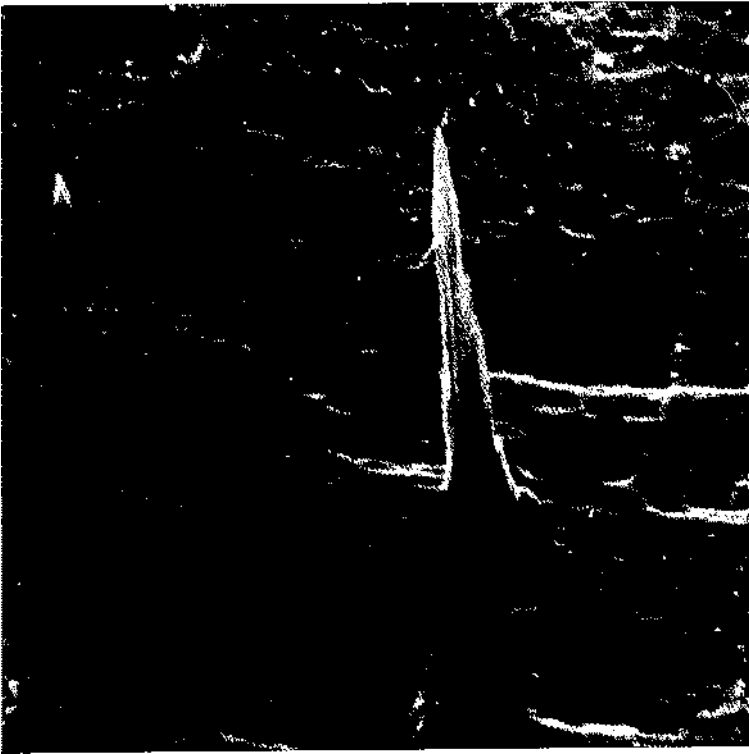


Fig. 1 Examples of cones and (100) edged pits formed during electron irradiation of NaCl.

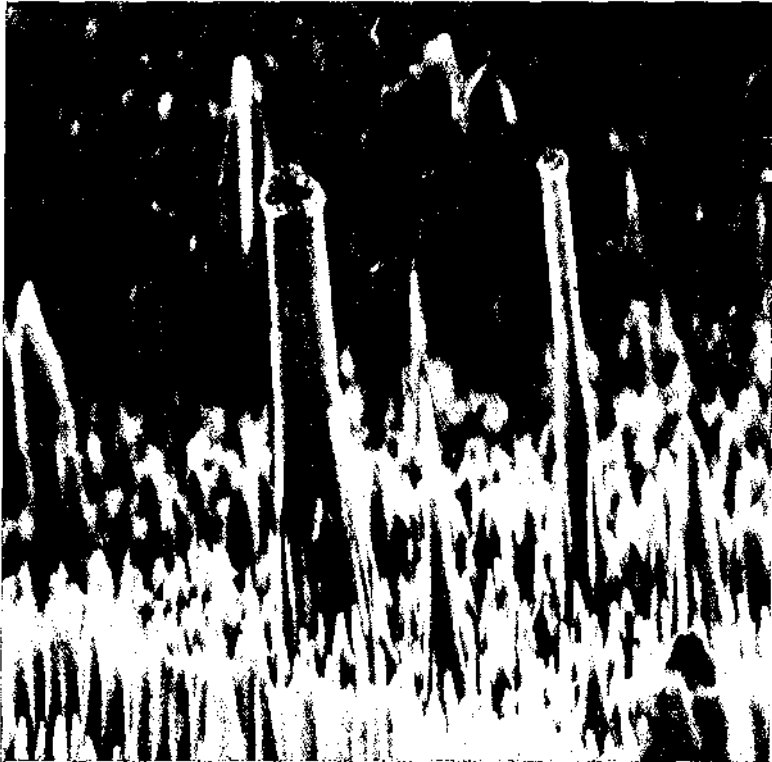


Fig. 2 A sample of KI illuminated with ultra violet-light. The material on the top of the larger conical features is at the level of the original surface.

been observed. The cones, or spikes are quite rough and typically subtend a cone angle of 10 degrees for 1 kV electrons.

In addition to the very large cones which originate from surface contaminants there are also small cones which are typically 10 micron high. These appear even on clean surfaces which are prepared by cleaving the sample in high vacuum. Possibly these represent impurities within the crystal which are exposed by the electron, or photon beam. Alternatively they may demonstrate the formation of specks of colloidal alkali metal which have nucleated on the surface.

The large spikes, once formed, also act as nucleation centres for recrystallization of the material. Such growth takes place predominantly from the tip of the spike. This suggests that it is partially an electrostatic process since this would be the region of maximum electric field if the tip becomes charged through secondary electron emission. Ions from the vapour would thus be attracted to this point. The process is extreme in lithium fluoride and electron induced sputtering is almost entirely inhibited by the growth of redeposited strands of LiF on the surface of the irradiated region.

One further feature associated with the spikes is that some very large spikes are found which are bent, that is, they must have become curved after being formed by the electron beam. These are rare but occur in the midst of normal spikes, so one discounts electron beam „melting“ or accidental physical damage. At present no explanation is offered.

Whereas the cones are unrelated to the crystal axes the other common feature on the surface of electron irradiated samples is crystalline in nature. These are flat bottomed pits with the edges and sides cut along (100) planes. Such pits appear in all sizes and frequently contain other smaller pits. They show the same faces even if the crystal is cut and/or irradiated in a direction other than a $\langle 100 \rangle$ direction. At present the only suggestion to account for this feature is that atoms migrate across the surface during irradiation and produce facets with a minimum free energy and the stable faces are (100) faces. This is obviously inadequate since a high rate of surface diffusion might be expected to destroy the features of the spikes.

Finally, some experiments will be described on 'strained alkali halide crystals. These show a variety of surface features after irradiation, in particular, parallel rows of cones or pits. All the surface features may be interpreted in terms of different sputtering rates for the following three regions. These are a normal rate for pure alkali halide, a reduced rate at impurities (i.e. surface dirt) and a greatly enhanced rate where dislocations intersect the surface. Because impurities tend to nucleate at dislocations this also explains the association of spikes with deep etch pits and the rows of spikes along cleavage steps and grain boundaries.

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2.13 Oxidation patterns due to bombardment under variable incidence of copper monocrystals by oxygen ions

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2.14 Microtopography of metal surfaces eroded by ion bombardment

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2.15 On the origin of surface topography on ion bombarded metal samples

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Our replica and transmission EM studies on ion (Argon) bombarded Cu samples (dose: 10^{16} ions/cm²)¹⁾ and the observations of several other authors²⁾ enables us to propose a model for the formation of the surface structure which appears on the surface of ion-bombarded metal samples.

The fact that our experiments on bombarded bulk material and bombarded thin foils (electropolished before bombardment) give exactly the same results as far as relief structure is concerned, proves firstly that the obtained features are independent of the preparation methods¹⁾ before bombardment of both type of samples, thus excluding electrochemical etching and thermal effects. Secondly that the mechanism must be one compatible with bulk as well as thin material, thus concerns a number of layers not exceeding a thickness of the thin films (≈ 150 atomic layers).

On the other hand, due to the values of the sputtering yield³⁾ that correspond to the crystal orientation of the grains on which this surface structure