PREPARED DISCUSSION

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On Electron Microscopy of Sodium and Potassium Chloride Films

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We have previously reported a method of preparing thin single crystal films of non-metallic substances by chemical etching[1]. In the case of sodium and potassium chloride, however, we found it is difficult, although not impossible, to prepare clean films by this method alone. Films of these salts as thinned chemically are very liable to produce "specks" and "tangled strings" under electron irradiation as observed by Tanaka *et al.*, which veil the internal structure of the films. Very recently we found a method to take off the

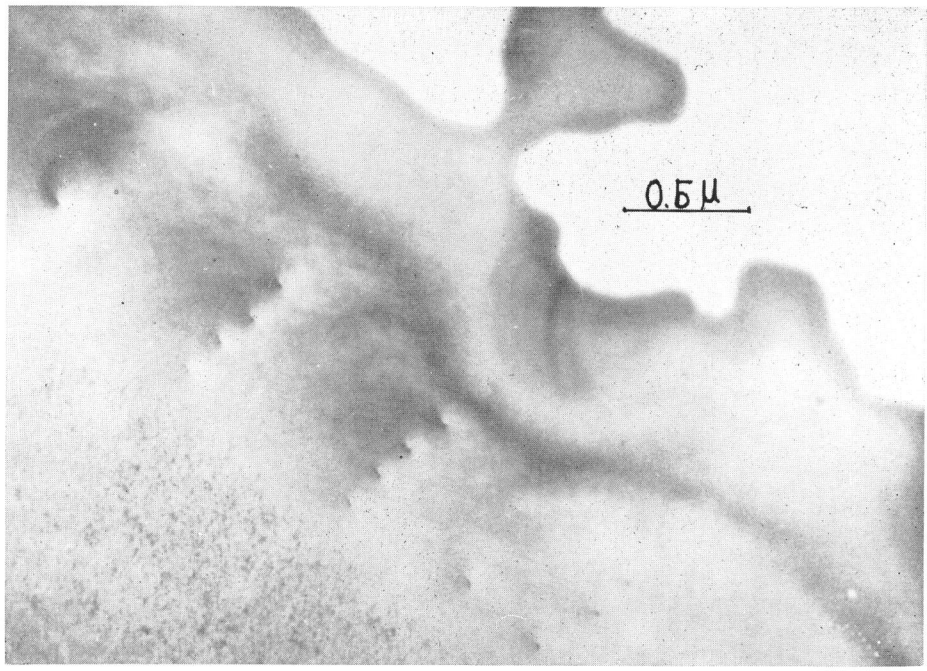

Fig. 1. A transmission electron micrograph of a clean thin sodium chloride single crystal film prepared by the method in the text. Note the arrays of dislocations introduced by bending the crystal before thinning.

veil. The method is to irradiate the films rather strongly by electron beam. Then, parts of surface layer of the films scale off leaving thin areas of several micron squares, or even wider. Thin areas so produced are free from "specks" and "tangled strings" and fairly stable under further electron irradiation. We could observe in such areas the internal structures of the films such as dislocation introduced by bending the crystals using the well-known Joffé effect (Fig. 1).

The fact that "specks" and "tangled strings" vanish when surface layers scale off suggest that they are in the surface layers. It is quite possible that the surface layers of the films prepared by chemical thinning or evaporation of solution in vacuum are more unstable than the interior of the films on account of the effect of solvent in the preparation process. In our opinion, "specks" and "tangled strings" of Tanaka *et al.* and "square voids" of Hibi *et al.* seem to be products in such unstable surface layers and their production under electron irradiation does not seem to be the intrinsic nature of the crystals. The observation using replica technique performed by Hibi *et al.*[2] does not seem to be very conclusive in proving that their square voids were in crystal, since it is a common experience to see that skin of contamination formed on specimen in earlier stage of electron irradiation remains unchanged after the specimen has evaporated out.

Double loop images mentioned in the two reports were observed also in our thin films of potassium chloride. But no such image was so far observed in our films of sodium chloride.

References

1 G. Honjo: *Proc. Int. Conf. Mag. & Cryst. Kyoto, 1961, II*, 277. (J. Phys. Soc. Japan **17** (1962) Suppl. B-II).
2 T. Hibi and K. Yada: *ibid.* 212.

Yada, K.: Prof. Honjo mentioned that the replica method is not useful. However, we think that the replica image has usefulness because some protruding or depressed structures are present on the replica image after intense electron irradiation, while the original surface of the crystal which is not irradiated at all is quite smooth.

Mannami, M.: I don't think that the loop image can be interpreted by the surface effect as Prof. Honjo mentioned. One experimental evidence that the void showing double loop image collapses into dislocations as shown in the movie clearly shows that the loop is due to void inside the crystal. This is also verified by the replica of heavily irradiated KCl.