Application of Inert Radioactive Gases in the Study of Solids. Thorium Oxalate. Comments

Balek recently compared the classical emanation method to Jech's method in a study of the thermal decomposition of some solids [1]. As he pointed out, his results are in fair agreement with emanation and DTA curves established by one of us [2-5].

The DTA curve of fig. 6 in Balek's publication exhibits an exothermic peak, appearing rather large if compared to the preceding exothermic one, which corresponds to about 40 kcal/mole [2]. Balek claims that this exothermic peak is due to a transformation of ThO_2 from the amorphous to the crystalline state. Of course such crystallisation processes are common during thermal decompositions, but the evolved heats are generally not so large [6]. Claudel *et al* paid special attention to the exothermic peak and established a correlation between its area and the oxygen partial pressure in the surrounding atmosphere. This effect has been explained by the catalytic oxidation of CO on the oxide formed, here ThO_2, as it was found later in the decomposition of a number of oxalates [6, 7]. We therefore think that the exothermic peak reported by Balek [1] refers to the combustion of CO in the presence of some unexpected oxygen, in addition to a small contribution from a crystallisation process.

As a final comment we agree with the remark by Balek [1] that the absence of pronounced maximum on the emanation curve during the transition dihydrate → monohydrate suggests a structural similarity of these solids: Claudel *et al* have already given this interpretation and checked its validity by radiocrystallography [4].

References

1. V. BALEK, *J. Mater. Sci.* **4** (1969) 919.
2. B. CLAUDEL, Thèse Lyon (1962) n. 286.
3. B. CLAUDEL, M. PERRIN, and Y. TRAMBOUZE, *C.R. Acad. Sci.* **252** (1961) 107.
4. R. BRESSAT, B. CLAUDEL, and Y. TRAMBOUZE, *J. Chim. Phys.* **60** (1963) 1265.
5. P. BUSSIÈRE, B. CLAUDEL, J. P. RENOUF, Y. TRAMBOUZE, and M. PRETTRE, *J. Chim. Phys.* **58** (1961) 668.
6. R. M. DELL and V. J. WHEELER, "Reactivity of Solids" (Ed. G. M. Schwab, Elsevier Publishing Company (1965)) p. 395.
7. J. W. LINDSAY, H. N. ROBINSON, H. L. BRAMLET, and A. J. JOHNSON, *J. Inorg. Nucl. Chem.* **32** (1970) 1559.

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Operating Characteristics of an Ion-Bombardment Apparatus for Thinning Non-Metals for Transmission Electron Microscopy

The successful application of ion-bombardment for the preparation of thin foils of non-metals for transmission electron microscopy was reported recently by Barber [1]. We have developed this technique independently and the object of this note is to report some of our experiences which may be of interest to others concerned with the defect-structure of non-metallic crystals. Our apparatus is based on the one described by Paulus and Reverschon [2] and is similar to that described by Barber [1], except that the specimen is bombarded from each side by a single ion-beam at normal incidence. This arrangement, instead of multi-beams at glacing incidence, has the advantage of simplicity of construction without any apparent loss of efficiency. The thinning rate varies for different materials but the range is from 1 to 5 μm/h.

Important considerations, not mentioned in detail by Barber [1] are the discharge characteristics of the ion-gun and the energetics of the ion-beam impinging on the specimen surface.

The argon-ion current (called the probe current) is measured by inserting a metal probe in the ion-beam. Fig. 1 shows that the probe current is very sensitive to changes in pressure and we have found that the optimum operating condition (in particular stability) corresponds to the low-pressure side of this peak. The probe current is relatively insensitive to the anode-cathode separation for values from 2 to 5 mm –

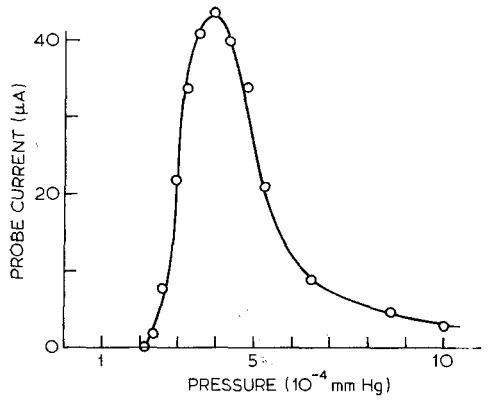

Figure 1 Probe current as a function of pressure in the working-chamber.

as can be seen in fig. 2. The single hole in the cathode erodes during operation of the gun and this modifies the dynamic vacuum conditions since it controls the argon leak-rate. We have found that a cathode-hole diameter of 1.5 mm ensures an operating time of about 80 h, after which the hole has enlarged to about 2.2 mm. Good operating conditions are much more difficult to maintain with a many-hole cathode.

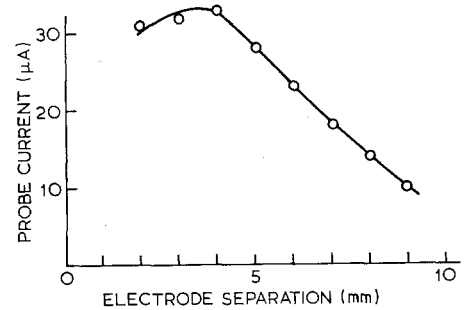

Figure 2 Probe current as a function of electrode separation in the ion gun.

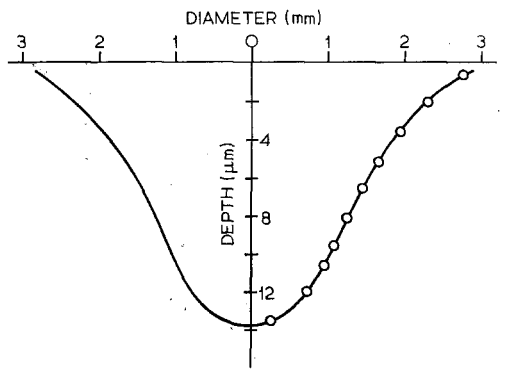

Figure 3 Profile of a hole produced by ion-bombardment of a polished quartz plate.

In considering the effects of radiation damage to the specimen, it is important to know the energy of the ions. This energy has been estimated in our apparatus by applying a retarding potential to the probe and measuring the probe current for a fixed cathode-anode potential. It was found that for a gun-potential of 4 kV, the maximum energy of the ions is considerably less than 4 keV and that most of the argon ions have energies less than 60 eV. Hence, the application of any theory of ion penetration and ion damage to specimens thinned by this technique must use realistic values for the ion energy. Comparison with thin specimens in the form of crushed fracture fragments [3] has shown that the ion beam neither modifies the existing defect structure nor introduces new dislocations or planar defects.

The profile (determined optically from interference fringes) of a hole produced in a polished quartz single crystal by ion-bombardment is shown in fig. 3.

We are at present using this thinning technique principally in the study of dislocations and other defects in naturally and experimentally deformed minerals and rocks [4]. Fig. 4 shows the dislocations in an experimentally deformed single crystal of synthetic quartz. A low-angle boundary consisting of an array of dislocations in a grain of an olivine rock which appears to have undergone creep is shown in fig. 5.

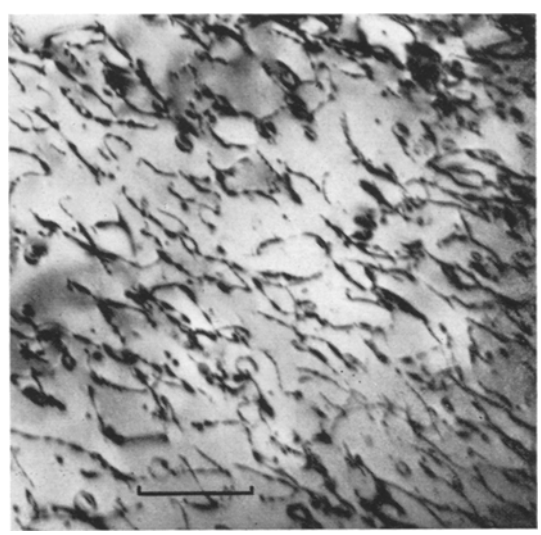

Figure 4 Dislocations in plastically deformed synthetic quartz. Scale line represents 1 μm. Bright-field image.

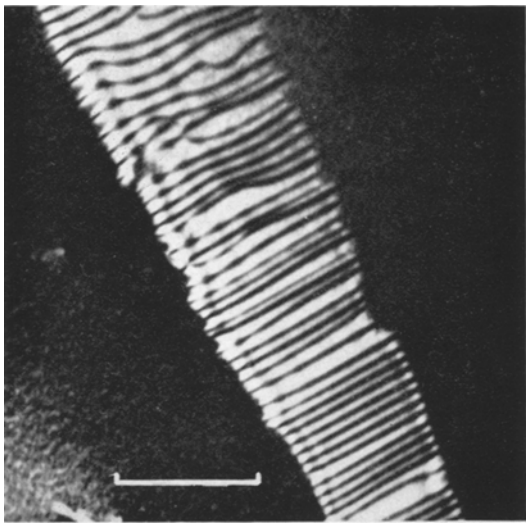

Figure 5 Low-angle boundary in a grain of an olivine rock. Scale line represents 1 μm. Dark-field image.

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References

1. D. J. BARBER, *J. Mater. Sci.* **5** (1970) 1.
2. M. PAULUS and F. REVERSCHON, *J. Phys. Radium* **22** (1961) 103.
3. A. C. MCLAREN and P. P. PHAKEY, *Austr. J. Phys.* **18** (1965) 135.
4. A. C. MCLAREN, B. E. HOBBS, R. G. TURNER, and J. N. BOLAND, *Contributions to Mineralogy and Petrology* (in press).

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Cross-Sectioning Techniques for Scanning Electron Microscopy

The usual methods of examining fibre cross sections with the scanning electron microscope (SEM) are not fully satisfactory. One relatively common cross-sectioning technique involves cutting fibre bundles, gluing them longitudinally to a sample stub, coating with a conductive layer, and tilting the specimen stage to obtain a view of the cut fibre ends. Alternatively, fibre bundles may be pulled through a specially designed stub, the exposed ends cut flush with the stub surface (using a razor blade), and finally, the mounted sample coated with a conductive layer. This technique is an adaptation of the Shirley plate method, commonly used in optical microscopy.

Both methods present imaging problems. Recently an improved technique was published by Anderson and Lipson [1]. These authors described a Hardy microtome method in which the fibres are forced into a slot, sectioned in an epoxy medium, left in the embedding matrix, glued to a sample stub, and coated with a conductive layer.

In our laboratory we have found the most advantageous techniques to be adaptations of the classical paraffin method [2, 3], which is well known to all textile microscopists. Depending on the hardness of the fibre and the cross section thickness desired, one may choose either paraffins of differing melting points and hardnesses, or a mixture of a paraffin and a matrix-hardening additive (i.e. carnauba wax). Our adaptation of the classical paraffin method for preparing SEM cross section samples simply involves embedding the yarns, cutting serial sections, and mounting them onto glass slides with Mayer's albumen. The slides are then washed twice with xylene to remove the embedding matrix. Finally, the slides are washed twice with ethanol (to set the albumen) and allowed to dry. Slides prepared in this manner may then be scanned with a light microscope to select sections for SEM examinations. Those sections to be studied are split from the glass slide with a glass cutter. (While it is not critical, a convenient fragment size is approximately 5×10 mm). The glass segment is mounted onto a sample stub with Duco cement, coated with a conductive layer, and placed in the SEM. This method is handy, since by using multiple embedding fixtures [4], a number of yarns may be embedded, sectioned, and processed simultaneously. This permits the mounting of a number of yarns on the same SEM sample stub. Therefore, direct comparisons between yarn samples are facilitated.

An alternative method we have found to work well, particularly when attempting to match the