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.

Received 15 July and accepted 18 September 1970.

P. BUSSIÈRE
Institut de Recherches sur la Catalyse CNRS, 39 Bd du Onze-Novembre 1918, 69. Villeurbanne, France

B. CLAUDEL
Present address:
Department of Chemical Engineering, University of Missouri, Columbia, Mo 65201, USA

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 –