

*Micrographs in "The Effect of Micro-structure on the Physical Properties of Glasses in the Sodium Silicate System"* (J. Materials Sci. 3 (1968) 380).

When we submitted an electron micrograph for use on a cover (J. Materials Sci. 3 no. 4) we neglected to properly acknowledge the source. This particular micrograph and the others in our paper were made by Mr Donald Carter, Electron Microscopist at the Owens-Illinois Technical Center. We should like not only to

express our gratitude to Mr Carter for his excellent micrograph but give him recognition for his work. If at all possible, could you please publish in your next issue a short word attributing the July cover micrograph to Mr Donald Carter, Owens-Illinois Technical Center.

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M. B. FIELD  
R. H. REDWINE  
Owens-Illinois Technical Center,  
Toledo, Ohio, USA

*A Note on the Preparation of Th and U Oxychalcogenides*

In various attempts to grow single crystals of ternary Th and U chalcogenides (with Si, Ge, P, As, Sb or Bi as third element) in uncoated silica tubes by a transport reaction, we obtained compounds which turned out to be oxychalcogenides. Since, to our knowledge, these compounds have so far only been synthesised in powder form, we thought it worthwhile to report on our findings. In our experiments the elements in the proper ratio corresponding to a ternary chalcogenide were placed at the hot end of a reaction tube (diameter 25 mm, length 200 mm) which was kept at 900 to 1050° C, while the other end was 50 to 100° cooler. Bromine or iodine in concentrations of 3 to 6 mg/cm<sup>3</sup> was used as transport agent. After one week crystals were found in the cooler part of the tube. In the case of Th/S/X transparent orange-yellow ThOS crystals up to 3 mm formed, most of which had a pseudohexagonal habit. The nearly hexagonal shape of the crystals is a consequence of the axial ratio  $c/a$  which is close to  $\sqrt{3}$ . The largest dimension of these crystals is parallel to an  $a$  axis of the tetragonal PbFCl structure and the two axes perpendicular to it have almost the same ratio as the axes  $a$  and  $b$  of a hexagonal crystal in orthorhombic description. In the case of the corresponding selenium compound ThOSe, crystals of pseudohexagonal habit were the exceptions, although for ThOSe the  $c/a$  value is even closer to  $\sqrt{3}$  (ThOS:  $c/a = 1.70$  [1]; ThOSe:  $c/a = 1.74$  [2]). Instead, truncated square pyramids occurred rather frequently. ThOSe crystals look purplish black, but they are brownish red when powdered. The

crystals of ThOTe, for which  $c/a = 1.83$  [3], clearly revealed the tetragonal symmetry of their structure. ThOTe always formed as thin square platelets of 1 to 3 mm edge length. ThOTe crystals look metallic grey but the powder is black.

By the same method, we also produced pseudohexagonal needles of UOS, which showed a metallic lustre, and square platelets of UOTE, which were very similar to those of ThOTE. Attempts to synthesise USiS, USiSe and USiTe from the elements at 1000 to 1150° C without addition of halogen gas led to microcrystalline UOS, UOSe and UOTE, respectively, and small silicon crystals.

In all these compounds the oxygen stems from the quartz ampoule. If the third element of the initial material is Si, Ge or Sb, then the silica is reduced to elemental silicon. If, however, with thorium (or a rare-earth element as well) the third element is P or As, then these elements combine with Si to form very thin single-crystal flakes of yellow transparent SiP or brownish SiAs, which both crystallise in the GaTe structure.

Doubtless the third element is not essential for the synthesis of the oxychalcogenides and, in fact, ThOS crystals formed also when the starting material corresponded to the composition ThS<sub>2</sub>, provided the silica tube was uncoated.

Since in the PbFCl structure of these oxychalcogenides only cation-anion bonds occur, the cation is tetravalent and the Th compounds, therefore, are weakly para- or diamagnetic while the U compounds, in which the cations carry two excess valence electrons, exhibit a paramagnetism of the Curie-Weiss type. At low temperatures the magnetic moments of