

Further information on the pressure generation in the tetrahedral apparatus can be derived from Fig. 3, which shows the load/displacement characteristic during the initial stages of compression of a plain pyrophyllite tetrahedron, displacement being measured by means of an inductive transducer mounted between the top anvil and the base plate of the apparatus. This type of curve, with load rising to a maximum, a fall to lower value and a subsequent rise, is typical, as might be expected, of extrusion in the presence of friction<sup>10</sup>. It is reasonable to suppose that the observed maximum in the load at 3.0 tons corresponds to the beginning of the extrusion process which forms the compressible gaskets: at this load the mean pressure on the anvil faces is 1.8 kb. This point is plotted in Fig. 2.

The conventionally obtained bismuth, thallium and barium points are also plotted, and it may be seen that the combined curve obtained by these methods has a fairly complicated shape. It may be divided into three regions: (a) the initial pressure rise, with a slow falling off in slope up to ~ 12 kb; (b) a rapid increase in slope between this pressure and 23–24 kb; (c) a fall off thereafter to a fairly linear relation up to the barium point. While it is possible that metastability of the low-pressure phases could have affected the transition loads, we believe that the straightforward and consistent experimental results obtained make this unlikely. Fig. 2 then indicates that the load/pressure characteristic for the N.B.S. type tetrahedral anvil apparatus is very similar to that obtained by Litster and Benedek<sup>11</sup> for a 'belt' type apparatus, using the pressure variation of the <sup>57</sup>Fe nuclear resonance frequency.

The complicated shape of both curves is undoubtedly related to the precise mechanism of pressure generation.

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JOHN LEES

Ultra-high Pressure Laboratory,  
Standard Telecommunication Laboratories, Ltd.,  
Harlow, Essex.

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## CRYSTALLOGRAPHY

### Two New Binary Chromium Oxides

WELL-DEVELOPED black crystals of two chromium oxides (*A* and *B*) with composition between CrO<sub>3</sub> and CrO<sub>2</sub> have been obtained by heating CrO<sub>3</sub> in a high-pressure autoclave at 200°–300° C. The oxygen pressure was 1.5–3 kilobars.

Single crystal photographs of substance *A* indicate the Laue symmetry *mmm*, and the conditions limiting possible reflexions suggest the space group *Pbcn* (No. 60). The unit cell dimensions of *A* derived from single crystal and powder photographs are:

$$a = 12.04 \text{ \AA}; b = 8.21 \text{ \AA}; c = 8.18 \text{ \AA}; V = 808 \text{ \AA}^3$$

The observed density, 3.68 g cm<sup>-3</sup>, corresponds to a formula weight of 1,790 per unit cell, suggesting a cell content of 4 Cr<sub>2</sub>O<sub>12</sub> (1808).

The Laue symmetry of the substance *B* was also found to be *mmm*, and the missing spectra were those characteristic of the space groups *Cmcm* (No. 63), *C2cm* (No. 40) and *Cmc2*, (No. 36).

The unit cell dimensions of *B* as obtained from single crystal and powder photographs are:

$$a = 8.47 \text{ \AA}; b = 12.88 \text{ \AA}; c = 10.09 \text{ \AA}; V = 1101 \text{ \AA}^3$$

The observed density, 3.34 g cm<sup>-3</sup>, corresponds to a formula weight of 2,210 per unit cell, suggesting a possible cell content of 12 Cr<sub>2</sub>O<sub>8</sub> (2208) or 8 Cr<sub>3</sub>O<sub>8</sub> (2278).

The powder patterns of *A* and *B* are distinctly different from those reported earlier<sup>1</sup> for specimens with the gross compositions ~ Cr<sub>2</sub>O<sub>8</sub> and ~ Cr<sub>2</sub>O<sub>5</sub>.

Crystal structure determinations of these two new chromium oxides as well as further preparative investigations in this field are in progress.

This investigation forms part of a research programme on the crystal chemistry of chromium compounds, which is supported by the Swedish Natural Science Research Council.

KARL-AXEL WILHELM

Institutes of Inorganic and Physical Chemistry,  
University of Stockholm.

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## METALLURGY

### Role of Silica in the Oxidation Resistance of Molybdenum Disilicide

MOLYBDENUM disilicide is of particular importance in the field of high-temperature materials, both as a structural material in its own right and as an oxidation resistant coating for molybdenum alloys. Apart from having a high melting point of 2,000° C (ref. 1) and good oxidation resistance up to 1,700° C, MoSi<sub>2</sub> possesses high-temperature mechanical properties which make it adequate for many applications under static high-temperature oxidizing conditions<sup>2</sup>.

However, a relatively high elastic modulus coupled with low thermal conductivity and high thermal expansion render it prone to crack on thermal cycling, so that a molybdenum test-piece silicided by pack cementation at

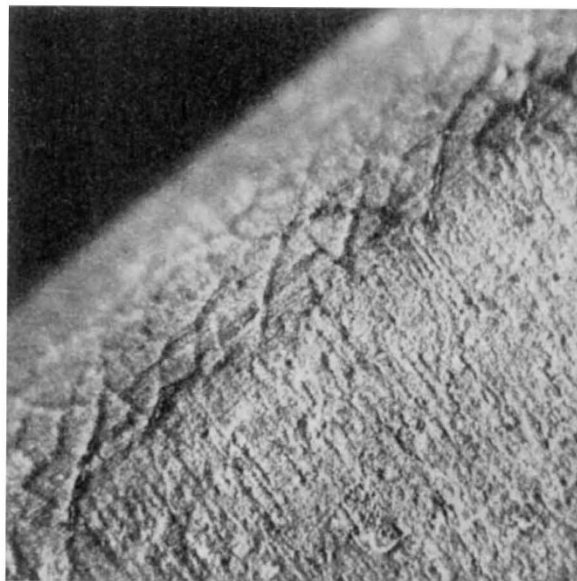


Fig. 1. Edge of silicided molybdenum specimen ( $\times 30$ ) which has craze-cracked as a result of thermal cycling from 1,100° C to room temperature