

The electron microscopic picture of PPM as shown in figs. 1 and 2 presents the picture of the complex of TCE with the same polymer.

That PPM builds a supermolecular structure of globules or more complicated globule aggregates is quite evident. The complex of the polymer with TCE forms structures not much differing from the structures which are typical of crystalline polymers. A similar tendency of building morphologically perfect structures was observed in the TCE complexes of poly-2-naphthyl methacrylate and polyvinyl carbazole.

It is well known that decrease of flexibility of the macromolecular chain improved its ability to adopt an ordered structure. Presumably, the great difference in the nature of supermolecular structures of PPM and its TCE complex should be ascribed to a change of flexibility of the polymer chain provoked by complex formation.

Further studies will be directed to investigation of the inner arrangement of the polymers and their complexes and to finding out whether the building of such a morphologically perfect structure is to be associated with crystal lattice formation.

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Transparent Sc_2O_3 by Hot-Pressing

The development of transparent ceramic materials has been the object of several investigations in attempting to fabricate material for I-R windows, windows for high temperature application, etc.

Previous studies, primarily concerned with the fabrication of transparent rare earth oxides, have been reported for Y_2O_3 by several investigators [1-4]. A variety of techniques were used in these studies including press forging with and without the use of lithium fluoride as a sintering aid [1, 2], sintering of a 90% Y_2O_3 -10% ThO_2 composition [3], and hot-pressing of Y_2O_3 [4].

In this work, Sc_2O_3 was hot-pressed to determine whether a transparent product could be produced by hot-pressing techniques which had already been used successfully for other ceramic oxides in previous investigations [4, 5].

A commercially available Sc_2O_3 powder* of 99.0 to 99.9% purity was used as a starting material. The powder was analysed by semi-quantitative spectrographic analysis. Trace impurities detected are shown in table I. The "as-

received" powder was dispersed in distilled water and subjected to ultrasonic treatment for 3 to 4 h in a Sonogen Automatic Cleaner†. The dispersion was subsequently filtered through successively finer filter papers to remove the larger agglomerates. The finest filter used was Whatman No. 42 paper.

TABLE I

Element	Amount (ppm)
Ag	10-100
Al	1000
B	10-100
Bi	10-100
Ca	1000
Cd	1000
Cu	100
Fe	100
Mg	100
Mn	10-100
Ni	10
Pb	10-100
Si	100-1000
Sn	10-100
Y	1000

*Semi-Elements, Inc. Saxonburg, Pa.

†Cole-Parmer Instrument & Equipment Co, Chicago, Ill.

The powder which completely filtered through and the powder trapped in the finest filter were combined, dried under an infra-red lamp, and fired at 500°C in air for approximately 1 h. The powder was cold-pressed into a 25.4 mm diameter disc and placed into a graphite die in the vacuum hot-press unit. It was desirable to separate the scandium oxide powder compact from the graphite by using a molybdenum sheet to line the inner die wall and molybdenum sheet discs between the compact and the plungers. After evacuation, the temperature is raised to 600°C and held for approximately 1 h to permit outgassing. Then, the pressure (4.6 Kg/mm²) was applied and the temperature is slowly raised to the hot-pressing temperature (1550 to 1600°C). Pressure was maintained at sintering temperature for 1 to 2 h, then released, and the specimen is allowed to cool slowly in the die to prevent thermal cracking. Although this procedure was used by Dutta and Gazza [4] to produce transparent yttria, the scandium oxide specimens produced were not transparent, only translucent.

A more recently developed procedure [5], which consists of using an incremental heating cycle, was successful in producing transparent specimens. The incremental heating cycle allows full density (transparency) to be achieved by initially permitting diffusion of residual volatile impurities to occur at temperatures where densification and grain growth occur slowly. Also, a more controlled rate of grain growth minimises dissociation of pores from the grain boundary region. A typical cycle consists of raising the specimen temperature to 1400°C, applying the pressure (4.6 Kg/mm²), and holding at this temperature for approximately 60 min. Then, the temperature is raised to 1450°C and held for 60 min, 1500°C for 60 min, 1550°C for 60 min, etc. until the final hot-pressing temperature is reached. At the final hot-pressing temperature a hold of 90 to 120 min is usually employed. In fig. 1, specimens are shown which resulted from final hot-pressing temperatures of 1575 and 1600°C for 90 min. Cracking of the specimen produced at 1600°C is believed to be caused by thermal stresses during cooling. Also, the presence of agglomerates can be observed in the transparent matrix. A darkening in colour of the specimens is attributed to some oxygen deficiency produced by hot pressing in graphite dies.

Microhardness measurements were made on polished transparent specimens with a Knoop

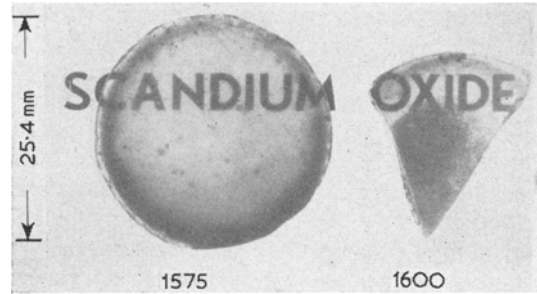


Figure 1 Hot-pressed scandium oxide.

indenter at 100 grams load. The average microhardness value was 910.

Optical transmission was determined, in the infra-red region, with polished specimens. The specimen thickness was 2.0 mm. Fig. 2 shows the infra-red transmission curve for hot pressed Sc₂O₃ with a cutoff wavelength of approximately 6 μm.

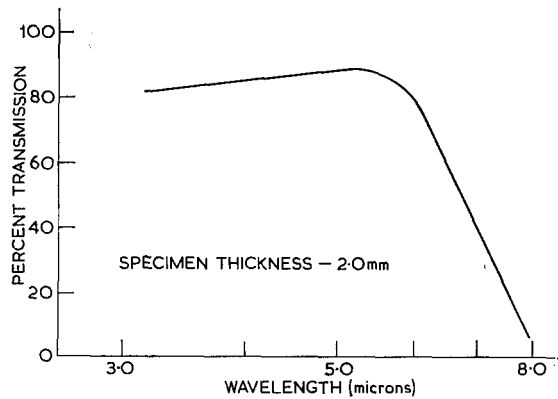


Figure 2 Infra-red transmission curve for hot-pressed Sc₂O₃.*

Grain size and fracture characteristics were determined from electron micrographs taken from fracture surfaces of bend specimens. Fig. 3 shows a typical fractograph from a specimen hot pressed at 1575°C for 90 min. The fracture mode consists of both intergranular and transgranular fracture. Grain size is approximately 1 to 2 μm.

Modulus of rupture data and elastic modulus were determined from bend specimens with dimensions of 25 mm long × 3.125 mm wide × 1.562 mm thick. A four-point bend test fixture was used. Elastic strain measurement required for calculation of elastic modulus were deter-

*19-066-1240/AMC-70 Army Materials and Mechanics Research Center.

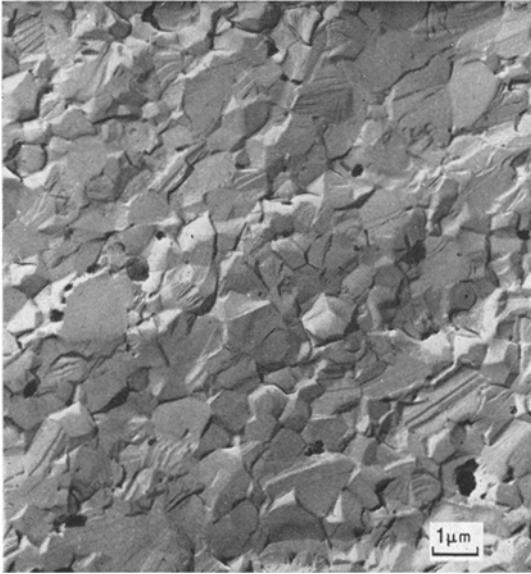


Figure 3 Electron fractograph of hot-pressed Sc_2O_3 .

mined by attaching SR-4 strain gauges (type FAP-06-12) to the tensile surface of the bend bars. From data gathered, thus far, the elastic

modulus for hot pressed Sc_2O_3 was calculated as 24600 Kg/mm^2 and modulus of rupture values were 21.8 Kg/mm^2 .

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The Observation of Helical Dislocations in Sapphire

During the course of investigation of dislocations in sapphire crystals [1], numerous dislocation reactions of the type:

$$[2\bar{1}10] + [\bar{1}2\bar{1}0] + [\bar{1}\bar{1}20] = 0$$

were identified. Each reaction represents a self-pinning point, hence the dislocations involved cannot glide easily. However, a special kind of controlled dislocation climb can take place. If a straight dislocation of predominantly screw character, and pinned on both ends, could climb, it would curve and thereby acquire an edge type component. This type of climb would require a transfer of material and a high activation energy, hence, the implication is that the dislocation will be active only at an elevated temperature. Provided that the climb already described could be accomplished, then prismatic glide [2] parallel to the Burgers vector can occur, as well. In such a case, a dislocation which is pinned on both ends and undergoes limited climb and prismatic glide would curl, and, under certain circumstances, may result in the formation of a helicoidal dislocation. Such helical or helicoidal

dislocations have been observed, using decoration techniques, in ionic crystals such as CaF_2 [3-6] and NaCl [7, 8]. A review of the subject of dislocations in ionic crystals in which the mechanism describing the formation of these helices is given by Amelinckx [9].

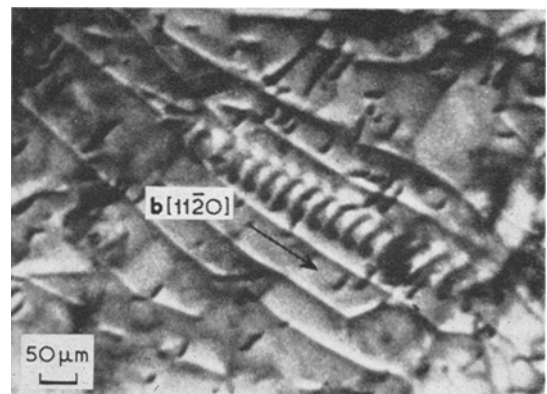


Figure 1 An X-ray transmission topograph of a sapphire plate, cut parallel to the (0001) plane, taken in $\bar{3}030$ reflection. The arrow indicates the direction of the Burgers vector with respect to the helical dislocation. This helical dislocation exhibits total extinction in $\bar{3}300$ reflection.