

in a hot-stage microscope for an hour at either 550 or 600°C. At either temperature, the acicular structure was transformed. Extremely fine grains were retained at 550°C but the 600°C treatment coarsened the grains to diameters of about 20 μm.

Consolidation of splats to full-density rods was successfully achieved by cold compaction followed by extrusion (reduction of 8:1 or 23:1) at either 510 or 605°C.

Details regarding processing and evaluation are given in the report by Jordan [2]. In case of renewal of interest in rapidly-solidified uranium, further work would apply a more refined version of REP or other means of rapidly quenching metals. Nonetheless, the early work by Kaufmann, Jordan and their colleagues at Nuclear Metals, Inc., is worth recording as a useful point of departure.

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## Room-temperature fracture energy of monoclinic $Gd_2O_3$

Fracture energy data for non-cubic polycrystalline materials is relatively limited. Graphite and  $Al_2O_3$  have been studied extensively, but the bulk of the fracture energy work has been carried out on cubic systems. For the rare-earth oxides in particular, no fracture energy data is available on either the cubic or non-cubic oxides. In this study the fracture energy,  $\gamma_f$ , was measured as a function of porosity for monoclinic  $Gd_2O_3$ .

In order to prepare the  $Gd_2O_3$  specimens, gadolite powder from the Ames Laboratory Rare Earth Separation Group\* was dissolved in nitric acid and precipitated according to a technique developed by Dole *et al.* [1]. The powder was then hot-ground in a mortar and pestle and calcined at 1000°C in an alumina crucible. The powder was dried for 2 h at 150°C and then pressed at 3.45 MPa (500 psi) in a double-action steel die having a rectangular cavity of 7.6 cm × 0.6 cm. Following dry pressing, the resulting bars

were enclosed in rubber containers and isostatically pressed at 206.8 MPa (30 000 psi).

The  $Gd_2O_3$  specimens were sintered in air in a gas-fired furnace at 1300 to 1650°C for 1 to 6 h. In order to limit specimen contamination, the bars were placed on a 2 mm thick bed of  $Gd_2O_3$  powder inside a horizontal muffle tube. Prior to sintering, the specimens were covered with an additional 2 mm layer of  $Gd_2O_3$  powder.

The fired specimens were diamond ground into rectangular prisms having a dimensional tolerance of ±0.005 cm. The resulting bars, which were about 5.0 cm long, 1.0 cm in depth and 0.5 cm thick, were then cut lengthwise producing two bars of nearly identical microstructure, each about 0.2 cm thick after regrinding. The specimens were then annealed in air at 1000°C for at least 8 h.

The room-temperature Young's modulus was measured for each specimen using the sonic resonance technique originated by Forster [2]. The experimental technique has been discussed in detail by Spinner and Teft [3] and a description of the apparatus is available elsewhere [4, 5]. The

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elastic moduli were calculated from the resonant frequency data by the theory developed by Pickett [6] and later modified by Hasselman [7]. The particular computational techniques used for this study are revealed by Manning [4] and Dole [8].

A water immersion technique was used to measure the bulk density of each specimen. X-ray diffraction techniques verified that both the starting powders and the as-fired bars were in the desired monoclinic form.

The fracture surface energy measurements were carried out on  $Gd_2O_3$  using the notched beam test (NBT) [9], performed on a commercial testing machine.\* The specimens were loaded in three-point bend on a 1.75 cm span at a cross-head speed of  $0.05 \text{ cm min}^{-1}$ . Notches equal to approximately one-half the bar depth were cut into the bars using a 0.015 cm thick diamond saw blade. An average of six  $\gamma_f$  determinations were made per specimen.

The fracture surface energy,  $\gamma_f$ , was calculated from an equation derived by Brown and Scrawley [10],

$$\gamma_f = \frac{9F^2 L^2 C}{8YW^2 D^4} \left[ A_0 + A_1 \left( \frac{C}{D} \right) + A_2 \left( \frac{C}{D} \right)^2 + A_3 \left( \frac{C}{D} \right)^3 + A_4 \left( \frac{C}{D} \right)^4 \right]^2, \quad (1)$$

where  $F$  is the load at crack propagation,  $L$  is the span between the knife-edge supports,  $C$  is the notch depth,  $D$  is the specimen depth, the  $A$ 's are parameters depending on  $L/D$  and  $Y$  is the Young's modulus.

Fig. 1 shows the measured fracture surface energy of the  $Gd_2O_3$  specimens as a function of volume-fraction porosity. The average value and the 95% confidence interval for the fracture energy are indicated for each specimen. For a range of volume-fraction porosity between about 0.03 and 0.17, the  $\gamma_f$  values appear in a scatterband, centred about a value of approximately  $50 \text{ J m}^{-2}$ .

Using the same bar and notch geometry, span and loading rate, three sintered  $Al_2O_3$  bars (each having a relative density of about 98%) were tested along with the  $Gd_2O_3$  specimens, in order to provide a check on the  $Gd_2O_3$  data. For three

breaks per bar, the average  $\gamma_f$  values for the three  $Al_2O_3$  specimens were 26.2, 29.9 and  $30.8 \text{ J m}^{-2}$ . These values fall within the 22.6 to  $43.2 \text{ J m}^{-2}$  range of NBT values given by Simpson [11] for sintered  $Al_2O_3$  bars with similar relative densities.

Mecholsky *et al.* [12] have reported that fracture energies of materials are generally proportional to their elastic moduli. For example, in a plot of fracture energy against elastic modulus, cubic polycrystalline ceramics are mostly grouped in a single scatterband, with single-crystal data falling below the band and data for non-cubic polycrystalline materials appearing above the band. A later paper by Wu *et al.* [13] related the relatively high fracture energies of non-cubic polycrystalline ceramics to the branching behaviour of a propagating crack. The crack-branching is in turn thought to be influenced by microcrack formation at the crack tip due to elastic and thermal expansion anisotropy. In short, materials that microcrack due to thermal expansion anisotropy (TEA) (i.e. non-cubics) seem to have higher fracture energy than materials without TEA induced microcracks (i.e. cubics).

Thus, the fracture energy data on monoclinic  $Gd_2O_3$  presented here fits the qualitative trend pointed out by Mecholsky *et al.* [12]. That is, monoclinic  $Gd_2O_3$  does exhibit microcracking due to TEA [14, 15] and the measured fracture energies of about  $50 \text{ J m}^{-2}$  are relatively high when compared to the NBT  $\gamma_f$  values of about 5 to  $10 \text{ J m}^{-2}$  which are typical of cubic polycrystalline ceramics such as  $MgO$  [16] and  $Y_2O_3$  [17].

It is especially interesting to note that Eyring [18] classifies both  $Gd_2O_3$  and  $Y_2O_3$  as rare-earth sesquioxides and that the rare-earth sesquioxides are generally quite similar in their chemical and physical properties. The order-of-magnitude difference in the  $\gamma_f$  values for cubic  $Y_2O_3$  and monoclinic  $Gd_2O_3$  may be a premier example of the empirical link between a material's crystallographic system and its  $\gamma_f$ . If such a link does exist, then one would predict the other monoclinic rare-earth sesquioxides ( $Eu_2O_3$  and  $Sm_2O_3$ ) to have a  $\gamma_f$  in the range of  $50 \text{ J m}^{-2}$ , as  $Gd_2O_3$  does. On the other hand, one would expect the other cubic rare-earth sesquioxides ( $Sc_2O_3$ ,  $La_2O_3$ ,  $Ce_2O_3$ ,  $Pr_2O_3$ ,  $Nd_2O_3$ ,  $Dy_2O_3$ ,  $Er_2O_3$ ,  $Tm_2O_3$ ,  $Yb_2O_3$

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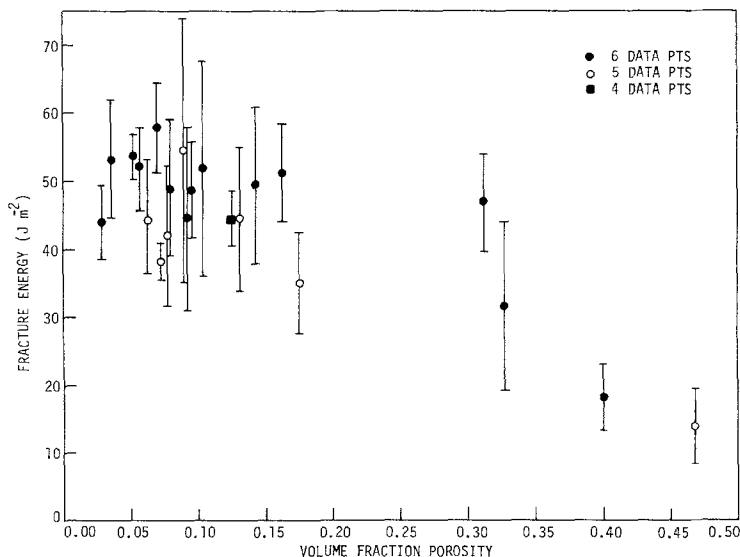


Figure 1 Volume-fraction porosity against fracture energy.

and  $\text{Lu}_2\text{O}_3$ ) to have a  $\gamma_f$  of about  $5 \text{ J m}^{-2}$ , as  $\text{Y}_2\text{O}_3$  does.

In conclusion, at room temperature the NBT value of fracture energy for  $\text{Gd}_2\text{O}_3$  is about  $50 \text{ J m}^{-2}$  over a range of volume-fraction porosities from about 0.03 to 0.17.

Above a volume-fraction porosity of about 0.30, the fracture energy falls off monotonically as porosity increases.

The  $\gamma_f$  value for  $\text{Gd}_2\text{O}_3$  is relatively high compared with  $\gamma_f$  values typical of cubic materials. This agrees with a qualitative trend suggested by Mecholsky, *et al.* [12].

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