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Reversible Phase Transformation in Y_2O_3 Under High Pressure

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Yttrium oxide (Y_2O_3) is a major component in current optical composite materials owing to its excellent transmittance and mechanical properties. The material exhibits both cubic and monoclinic crystal structures. Due to lack of optical properties of the anisotropic monoclinic phase, only cubic Y_2O_3 is considered to be important. Moreover, a small grain structure (<100nm) also plays an important role in the enhancement of optical properties for mid- and near-infrared materials. However, current processing methods yield large grain structures (> 100μm) [1]. In this work, we report grain size refinement via a pressure-induced reversible phase transformation mechanism. A detailed analysis by analytical electron microscopy techniques will be presented.

In a new technique to refine the grain size of a fully dense sample, bulk coarse-grained Y_2O_3 is subjected to a reversible phase transformation (cubic-monoclinic-cubic) under high pressure. Here, an unusual surface reaction effect that accompanies this transformation is described. It appears to be a consequence of reductive decomposition due to reaction between the Y_2O_3 sample and the graphite heater of the pressure cell.

In a typical experiment, a disc-shaped sample is encased in a resistively-heated graphite heater and exposed to 8 GPa at 1000ºC for 360 min. SEM examination of a fractured surface, Figure 1a, shows predominantly intergranular fracture, apparently along grain boundaries of the original coarse-grained cubic-Y_2O_3, now transformed under pressure into nano-grained monoclinic-Y_2O_3, Figure 1c. Under higher magnification, a faceted columnar-grained structure, Figure 1b and c, covering the entire sample surface to a depth of ~20 μm is observed. Moreover, EDS analysis, Figure 2, shows that that the Y/O ratio for a smooth intergranular fracture surface is depleted in oxygen relative to a rough transgranular fracture surface. Similarly, the columnar-grained structure is depleted in oxygen relative to the transgranular fracture surface. Hence, grain boundaries in the original cubic-Y_2O_3, as well as the surface columnar-grained structure, must have experienced oxygen depletion by reaction with the graphite heater, i.e. by a reductive decomposition mechanism. Progressive reduction of the surface layers of the Y_2O_3 explain the formation of the oxygen-deficient columnar-grained structure, facilitated by relatively fast oxygen diffusion along the directionally-aligned grain boundaries.

Preliminary FTIR optical properties from cubic Y_2O_3 (Figure 3) and mixed phase (cubic and monoclinic) samples show significant differences between the two. Therefore, it is necessary to understand phase transformation mechanism occurring in the sample and to identify the phases. Hence, further tests are underway to increase the thickness of the columnar-grained surface layer by enhancing grain-boundary diffusion at higher temperatures. A thicker layer of the new phase should enable a detailed analysis of its structure and properties.

References:

[1]. Jafar. F. Al-Sharab, Rajendra Sadangi, Vijay Shukla, and Bernard. H. Kear, Synthesis and Characterization of Nanostructured Magnesia-Yttria Based Nanocomposites, Mater. Res. Soc. Symp. Proc. Vol. 1056, 2008 Materials Research Society 1056-HH08-44, Nov.-26-30, Boston, MA.
[2]. This work is support from a grant from DARPA.
[3]. FTIR Optical properties were conducted in Professor Harrington's laboratory with the help of his student, Paul Mark. Discussion with Professor Harrington is gratefully acknowledged.

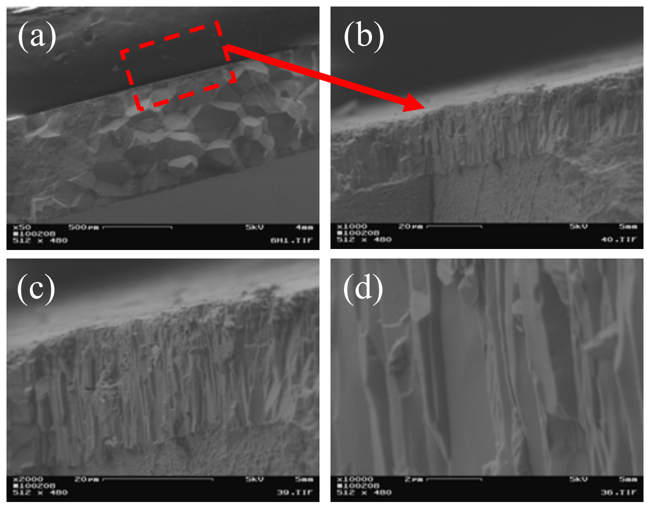

Fig. 1 SEM examination of a typical fractured sample after hot pressing at 8 GPa/1000ºC for 360 minutes, showing predominantly intergranular fracture, apparently along grain boundaries of the *original* coarse-grained (~300 μm) cubic-Y_2O_3, now transformed under pressure into nano-grained (~25 nm) monoclinic-Y_2O_3

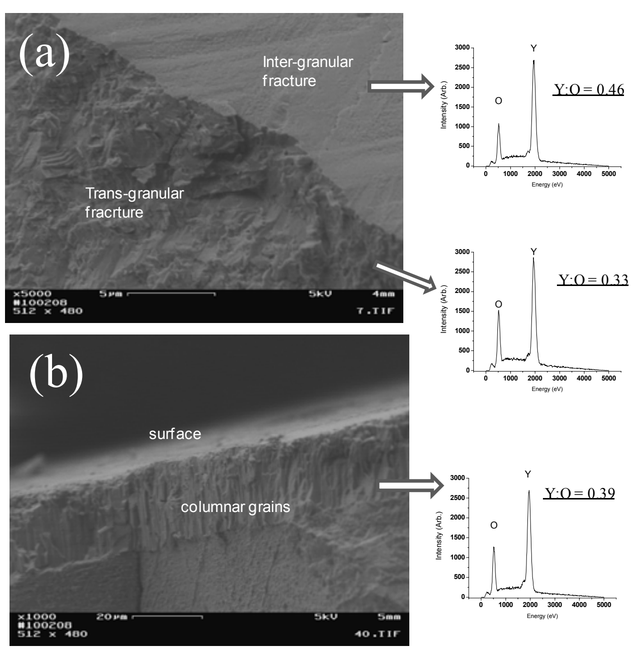

Fig. 2 EDS analysis of a fractured sample, comparing the Y/O ratios for the smooth intergranular fracture, rough transgranular fracture, and faceted columnar-grained

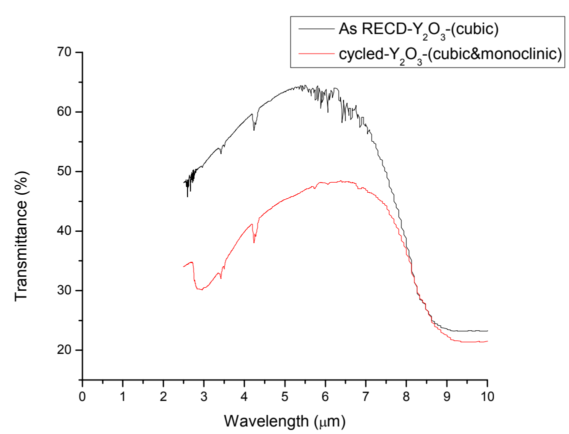

Fig. 3 FTIR Transmittance measurements of cubic-Y_2O_3 (300 μm grain size) and mixed-phase cycled Y_2O_3 (100 nm grain size)

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