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CRYSTALS

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MECHANICAL BEHAVIOR OF ERBIUM OXIDE SINGLE CRYSTALS

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ABSTRACT

Er_2O_3 single crystals were synthesized by optical floating zone single crystal techniques. The average room temperature hardness of these crystals was 7 GPa, while the average room temperature indentation fracture toughness was $0.86 \text{ MPa m}^{1/2}$. Hardness decreased with increasing temperature, while fracture toughness increased at 1200 °C and above. Cleavage planes observed in erbia were {110} and {111}. Macroscopic compressive deformation occurred in erbia at 1640 °C. The observed slip system was {110}<111>.

INTRODUCTION

Erbium oxide (Er_2O_3) is a rare earth oxide with a high melting point of 2430 °C. This material is very thermodynamically stable. It thus has potential applications where high temperature stability and corrosion resistance are required. Only a limited amount of research has been done on the mechanical properties and behavior of Er_2O_3 (1-5).

The crystal structure of Er_2O_3 is body centered cubic, with a lattice constant of 1.055 nm. There are 80 atoms per unit cell, 32 Er and 48 O. Thus, while Er_2O_3 has a relatively simple cubic crystal structure, the unit cell is large and contains many atoms. The coordination on each erbium atom is six-fold, while the coordination on each oxygen atom is four-fold. Erbia can be thought of as a distorted fluorite structure, with one-fourth of the oxygen atoms removed.

MATERIALS AND PROCEDURE

Single crystals of Er_2O_3 were synthesized using a xenon optical floating zone single crystal growth apparatus and the procedures given in Reference 3. It was possible to produce single crystals approximately 5 mm in diameter by 70 mm in length. The growth direction of the single crystals was observed to be <111>.

Because the erbia crystals were grown in a reducing Ar/ H_2 atmosphere, the as-grown crystals were black in color. The heating of black erbia in air at

1600 °C caused the erbia to return to its original pink color. This is due to the fact that erbia becomes substoichiometric to a level of $\text{Er}_2\text{O}_{2.978}$ when melted in vacuum or reducing atmosphere due to a loss of oxygen (6).

Microhardness indentation and indentation fracture toughness measurements were performed on stoichiometric erbia single crystals as a function of temperature using a Nikon QM-2 high temperature microhardness apparatus. Vickers indentations were employed at a load of 1 kg. Indentation fracture toughness was calculated using the approach of Anstis et.al. (7).

Elevated temperature compression tests at a temperature of 1640 °C were performed on the erbia single crystals using an Instron interfaced with a MoSi_2 element split mechanical testing air furnace.

RESULTS AND DISCUSSION

Single Crystal Hardness and Fracture Toughness as a Function of Temperature

Figure 1 shows the hardness of an erbia single crystal as a function of temperature. The indentation plane was {110} and the erbia was stoichiometric.

Figure 1: Er_2O_3 single crystal microhardness as a function of temperature.

The room temperature hardness of erbia was not particularly high, at a level of 7.5 GPa. Thus, erbia is a relatively soft oxide ceramic. The hardness generally decreased with increasing temperature, reaching a value of 2 GPa at 1400 °C.

Figure 2 shows the indentation fracture toughness of an erbia single crystal as a function of temperature. The indentation plane was {110} and the erbia was stoichiometric.

Figure 2: Er₂O₃ single crystal indentation fracture toughness as a function of temperature.

The room temperature fracture toughness of erbia is relatively low at a value of 0.9 MPa m^{1/2}. This indicates that erbia is a relatively brittle oxide ceramic. The fracture toughness was essentially constant up to 1000 °C, but exhibited a substantial increase by approximately a factor of two at 1200 °C and 1400 °C. This increase in fracture toughness is likely due to the initiation of plastic deformation at crack tips in erbia at high temperatures.

Effects of Crystallographic Orientation on Hardness and Fracture Toughness

Figure 3 shows the effects of crystallographic orientation on the room temperature Vickers microhardness of an erbia single crystal. The hardness was observed to be relatively insensitive to the crystallographic plane of the indentation, as would be expected for a cubic material. The average hardness for the various crystallographic planes was 7.0 GPa.

Figure 3: Room temperature Vickers hardness on different indentation crystallographic planes.

Figure 4 shows the room temperature indentation fracture toughness as measured on different crystallographic planes in erbia. As with the hardness, there was no strong dependence of fracture toughness on the crystallographic plane of the indentation, although the toughness appeared somewhat lower on the {112} plane. The average fracture toughness over the crystallographic planes was observed to be $0.86 \text{ MPa m}^{1/2}$ at room temperature.

Figure 4: Room temperature indentation fracture toughness measured on different indentation crystallographic planes.

Figure 5 shows typical indentation fracture patterns observed on the different crystallographic planes of erbium.

Figure 5: Typical indentation fracture patterns on the {100}, {110}, {112}, and {111} planes of erbium.

The best-formed indentation fractures occurred for indentations on the $\{100\}$ plane with indentation diagonals oriented in the $\langle 110 \rangle$ directions. This crystallographic orientation showed a distinct tendency for cleavage fractures on the $\{110\}$ planes of erbia. When indentations on the $\{100\}$ plane were rotated such that the indentation diagonals were along the $\langle 100 \rangle$ directions, fracture traces along $\langle 110 \rangle$ directions still predominated the fracture pattern.

The above suggests that $\{110\}$ planes are cleavage fracture planes in erbia. The fracture toughness on the $\{110\}$ planes was calculated to be $0.65 \text{ MPa m}^{1/2}$. However, it should be noted that the $\{111\}$ planes in erbia were also observed to be cleavage planes of low fracture energy (4). The macroscopic erbia single crystals showed a distinct tendency to fracture on $\{111\}$ planes during handling of the crystals. Thus it is interesting that $\{111\}$ plane fracture is not more prominent in Figure 5. It may be that fractures on other $\{111\}$ and $\{110\}$ planes inclined to the indentation surface obscured a distinct $\{111\}$ indentation fracture pattern on the indentation planes.

Elevated Temperature Compressive Deformation

Elevated temperature compression tests were performed at a strain rate of $3.5 \times 10^{-4} \text{ s}^{-1}$ (4). Attempts to deform single crystals of erbia at temperatures below 1600°C resulted in brittle fracture in compression, with no evidence of plastic deformation. A small amount of plastic deformation was observed in compression at 1640°C . The yield stresses for single crystals oriented with $\langle 112 \rangle$ and $\langle 110 \rangle$ directions along the compression axis were 80 MPa and 90 MPa , respectively.

Dislocations produced by plastic deformation in erbia at 1640°C were observed to have the slip system $\{110\}\langle 111 \rangle$ (4). It is thought that the $1/2\langle 111 \rangle$ Burgers vector dissociates into $1/4\langle 110 \rangle$ and $1/4\langle 112 \rangle$ partial dislocations as shown in Figure 6.

Figure 6: Burgers vector for $\{110\}\langle 111 \rangle$ dislocation slip in Er_2O_3 .

CONCLUSIONS

Er_2O_3 single crystals approximately 5 mm in diameter by 70 mm in length were synthesized by xenon optical floating zone single crystal growth techniques. The average room temperature Vickers microhardness was 7 GPa, and hardness decreased to 2 GPa at 1400 °C. The average room temperature indentation fracture toughness was $0.86 \text{ MPa m}^{1/2}$ and remained constant to 1000 °C, but increased to $1.5 \text{ MPa m}^{1/2}$ at 1200 °C and above. Both hardness and fracture toughness were relatively insensitive to the crystallographic indentation plane. Cleavage planes in erbia are {110} and {111}. Erbia single crystals exhibit plastic deformation at a temperature of 1640 °C. The dislocation Burgers vector in erbia is $1/2\langle 111 \rangle$.

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REFERENCES

1. A. Neuman, M. Platero, R.S. Romero, K.J. McClellan, and J.J. Petrovic, "Fabrication and Properties of Erbium Oxide", *Ceram. Eng. Sci. Proc.*, **18**, 37 (1997).
2. A.D. Neuman, M.J. Blacic, M. Platero, R.S. Romero, K.J. McClellan, and J.J. Petrovic, "Mechanical Properties of Melt-Derived Erbium Oxide", *Ceram. Eng. Sci. Proc.*, **19**, 423 (1998).
3. J.J. Petrovic, R.S. Romero, D. Mendoza, A.M. Kukla, R.C. Hoover, and K.J. McClellan, "Synthesis and Properties of Erbium Oxide Single Crystals", *Ceram. Eng. Sci. Proc.*, **20**, 3-10 (1999).
4. A.A. Sharif, A. Misra, J.J. Petrovic, and T.E. Mitchell, "Dislocation Motion in Erbia Single Crystals", submitted to *Acta Materialia*, 2000.
5. A.A. Sharif, F. Chu, A. Misra, T.E. Mitchell, and J.J. Petrovic, "Elastic Constants of Erbia Single Crystals", submitted to the *Journal of the American Ceramic Society*, 2000.

6. A.E. Miller and A.H. Daane, "Preparation of a New Type of Nonstoichiometric Rare-Earth Oxide", J. Inorg. Nucl. Chem., 27, 1955 (1965).

7. G.R. Anstis, P. Chantikul, B.R. Lawn, and D.B. Marshall, "A Critical Evaluation of Indentation Techniques for Measuring Fracture Toughness: I, Direct Crack Measurements", J. Am. Ceram. Soc., 64, 533 (1981).