Quantitative Analysis of Crack Propagation in Doped MgAl₂O₄ Spinel Metri Zughbi, Alexánder Campos-Quirós, Animesh Kundu, Masashi Watanabe Department of Materials Science and Engineering, Lehigh University, Bethlehem, PA

Introduction

- Magnesium Aluminate Spinel (MgAl₂O₄) is a ceramic with high strength, high transparency and low density. Potential uses are in lens, extreme-condition windows, and transparent armor. Traditional processing methods of spinel uses LiF as a sintering aid, which causes grain-boundary embrittlement. Ionic Ca and Y are being investigated as alternative sintering aids to increase grain-boundary strength, while maintaining optical properties/transparency.
- Ca and Y were chosen due to their abundance (inexpensive) and ionic size relative to Mg and Al in crystal lattice.

Hardness Testing

- Samples were fabricated by hot pressing high purity magnesium aluminate powder with 500 ppm of the doping element. Samples were annealed at 1400° C. Then hardness was measured by microhardness testing and microstructure observation and grain orientation analysis were performed by a scanning electron microscope (SEM).
- Cracks were formed during hardness testing. The crack path relates to the strength of grains and their grain-boundaries. Gatan Digital Micrograph software was used to analyze the crack propagation behavior through orientation maps

taken using electron

backscatter diffraction

(EBSD) analysis on SEM.

- 25 µm
- Results show the fracture toughness was not compromised with the addition of dopants. (True before and after annealing.)





- \triangleright boundaries are assumed to be. limitations of slicing code.



Ca segregation to grain boundaries $\overline{\mathbf{r}}'$ were confirmed by X-ray energy Dispersive Spectroscopy (EDS) in the aberration-corrected scanning transmission electron microscope (STEM). Therefore, the Ca dopant can be primarily responsible for the change in crack propagation path.

Grain boundary strength is preserved after doping with Ca in comparison to pure magnesium aluminate spinel. The presence of dopants in magnesium aluminate spinel yields a quantifiable difference in the transgranular fracture.



Quantitative Analysis of Crack Propagation

The crack propagates through either the grains (transgranular) or between grains (intergranular). The ratio between these two fracture paths was counted along the crack using horizontal slices through the crack. The more transgranular fracture occurs, the stronger the grain

Analysis was semi-automated but required detailed refinements due to

- There is a quantifiable difference in transgranular fracture with the addition of Ca as a dopant.
- Preliminary results show a statistically significant difference in Y-doped samples.

1000 Ca-doped Spinel: **900** 500 ppm, 1400 C, 48 h -OKα 800 ►AIKα EDS Off 700 ► MgKα **600** Boundary 500 400 300 200 have marked and and have marked and the second **100**| X-ray Energy (keV)

Conclusion











Acknowledgements

David and Lorraine Freed Undergraduate Research Symposium, Lehigh University Funding from NSF REU CMMI-2016279

