Influence of Elemental Doping to Microstructures and Mechanical Properties in Polycrystalline Spinel

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Abstract

The chemical process of introducing certain elements into magnesium aluminate spinel, known as doping, is one of very popular approaches to improve mechanical properties. In this study, samples were fabricated by hot pressing $MgAl_2O_4$ spinel nanopowder with 500 ppm calcium, or uniaxially pressing with 500 ppm yttrium. Samples were annealed at 1400 °C for 24 and 48 hours, and then prepared for characterization. Microhardness testing was conducted based on ASTM standard C1327-15 and the residual indents were observed by scanning electron microscopy to measure crack propagation induced via indentation. For Calcium (Ca)-doped hot-pressed spinel, it was shown that the longer sintering results in further sample densification and consequently increased hardness values. In uniaxially pressed Yttrium (Y)-doped spinel, longer annealing may result in pore growth and hence decreased hardness.

Introduction

- Spinel is a large group of minerals that includes magnesium aluminate ($MgAl_2O_4$).
- $MgAl_2O_4$ takes on the structure known as a cubic crystal system. As seen in Figure 1.
- $MgAl_2O_4$ is known for its beneficial properties of high strength and ability to become transparent. As seen in Figure 2.

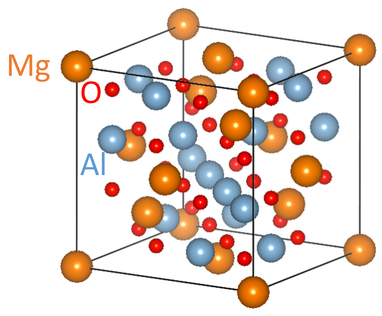

Figure 1: Cubic crystal system

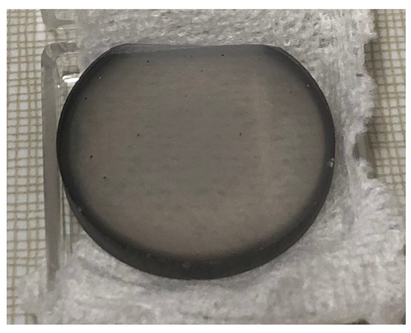

Figure 2: Transparent $MgAl_2O_4$ Spinel

- The purpose of this experiment was to find an element that would increase the strength of $MgAl_2O_4$ spinel

Sample Fabrication

For Calcium-doped spinel:

1. A 500 ppm Ca Nitrate doped nanopowder was prepared
2. Using ~18 g a 10 x 25.4 mm^2 cylindrical sample was hot-pressed
3. Sample was cut into 8 cubes with dimensions of ~ 5 x 5 mm^2
4. Two samples were put in the furnace for 24 hrs and two put in for 48 hrs at 1400 ℃
5. Heat treated samples were mounted in epoxy for grinding and polishing
6. Half the samples were used for hardness testing and other half used for microscopy characterization

For Yttrium-doped spinel:

Similar procedure was used, however due to unavailability of equipment, samples were uniaxially pressed instead of being hot pressed.

Results and Discussion

For Calcium-doped spinel:

Table 1: Hardness values for Ca-doped spinel

Hardness Values (HV)	Pure Spinel	24 hr Spinel	48 hr Spinel
Average	1509.56	1735.04	1790.96
Standard Deviation	17.50	72.25	94.27

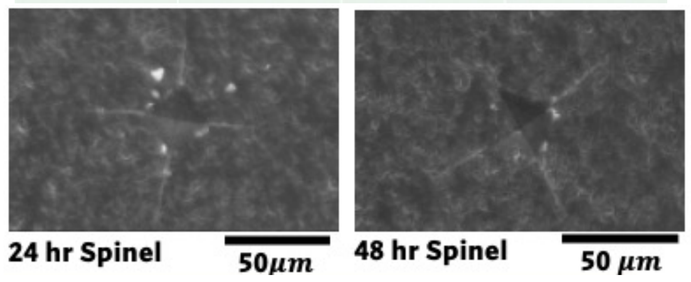

Figure 3: 24 hr spinel indentation (left); 48 hr spinel indentation (right)

Table 2: Fracture toughness values for Ca-doped spinel

Fracture Toughness ($MPa.m^{½}$)	Pure Spinel	24 hr Spinel	48 hr Spinel
Average	1.5	2.31	2.64
Standard Deviation	N/A	.39	.22

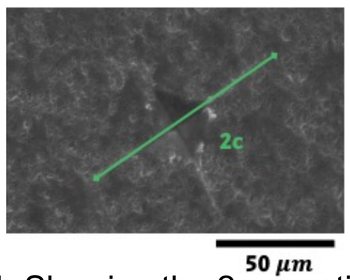

Figure 4: Showing the 2c equation value

For Yttrium-doped spinel:

Table 3: Hardness and crack length values for Y-doped spinel

	Hot Pressed	Uniaxially Pressed	
	Pure Spinel	24 hr Spinel	48 hr Spinel
Hardness (HV)	1508.7 ± 16.7	604.7 ± 28.0	324.2 ± 13.0
Crack Length (μm)	**36.9** ± 4.2	**24.1** ± 5.5	**21.9** ± 16.2

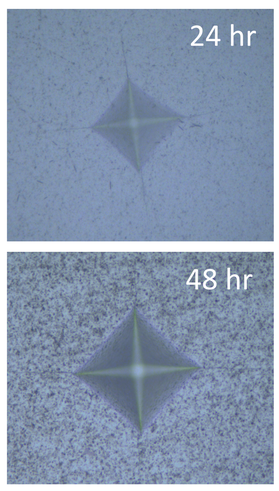

Figure 5: Differences in porosity in Y-Spinel

Conclusions

For Calcium-doped spinel:

1. By doping with Ca, Spinel strength increases
2. Sintering for 48 hrs increases hardness values and fracture toughness compared with sintering for 24 hrs

For Yttrium-doped spinel:

1. Hardness is lower for uniaxially pressed samples compared to hot pressed ones.
2. Pore coarsening with longer sintering time corresponds to decreased hardness

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References

1. Cui, F. Y. (2018). *Micro-scale Fracture Toughness Testing and Finite Element Analysis of Transparent Ceramics*(Doctoral dissertation, Lehigh University). Lehigh Preserve Institutional Repository.

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