Semi - annual Report Spring 2012

Densification and Characterization of Transparent Al_2MgO_4 Spinel Doped with Al_2O_3 and Al_2O_3

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CCMC RU UNM PSU Ceramic and Composi Materials Center

Long-range Goal

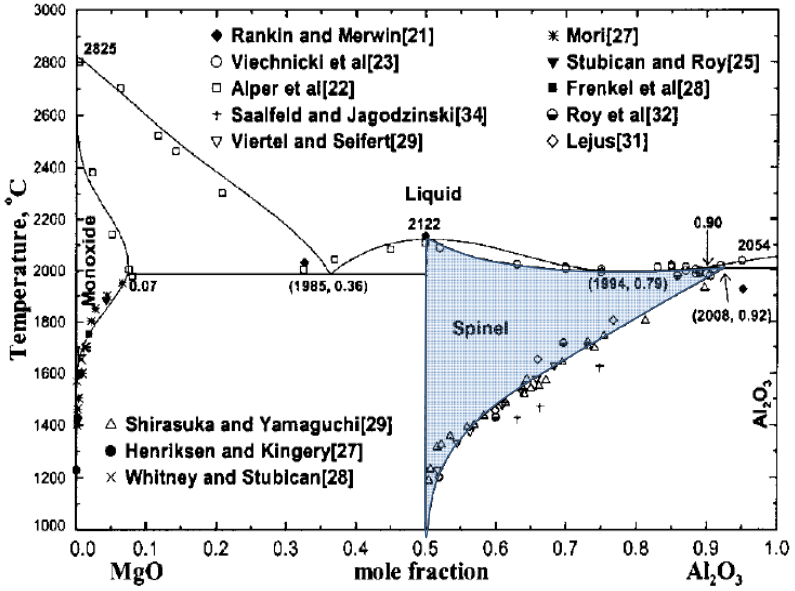
- **Improve the mechanical performance of transparent Al_2MgO_4 spinel.**

- **Characterize the microstructural, optical and mechanical properties of Al_2O_3 and Y_2O_3 – doped spinel**

Typical properties of glass and ceramic armor materials

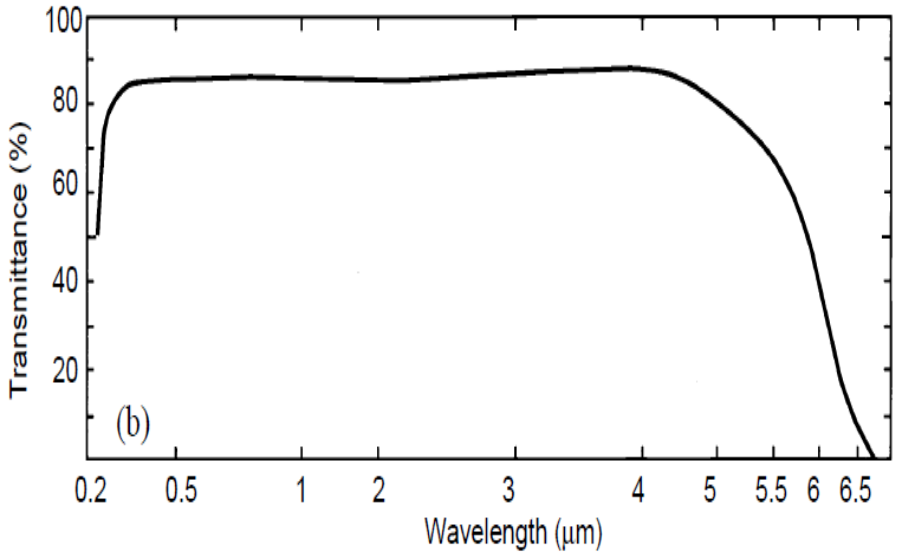
Property	Units	AlON	Fused silica	Sapphire	Spinel
Density	kg/m^3	3.69×10^3	2.21×10^3	3.97×10^3	3.59×10^3
Areal density (at 1″ thickness)	kg/m^2	93.89	55.85	100.97	90.86
Elastic modulus	Pa	334×10^9	70×10^9	344×10^9	260×10^9
Mean flexure strength	Pa	380×10^6	48×10^6	742×10^6	184×10^6
Fracture toughness	Pa $m^{1/2}$	2.4×10^6	0.78×10^6	3.0×10^6	1.7×10^6
Knoop hardness	Pa	17.7×10^9	4.5×10^9	19.6×10^9	14.9×10^9
Transmission in visual spectrum	%	82–85	91–92	75–82	–
Maturity of technology		Relatively new technology (becoming commercially viable)	Well established technology	Well established technology	Established, continued advancements
Cost		3–5 times that of glass	Lowest material and processing costs	Higher than ALON™	Lower than ALON™
Manufacturing costs		High due to high processing temperature, proprietary powder, and surface finish requirements	Relatively low due to lower melting temps	High due to high temperature processing and surface finish requirements	Moderate due to surface finish requirements
Bottleneck		Cost and limited dimensions	Limited ballistic protection enhancement	Cost and limited dimensions	Limited dimensions
Commercial availability		Sumert Corp., limited availability	Widely available	Widely available in smaller sizes	In the process of becoming more commercially available
Environmental resistance		Low chemical reactivity and highly scratch resistant		Low chemical reactivity and highly scratch resistant	

M. Grujicic et al. Materials and Design 34 (2012) 808–819



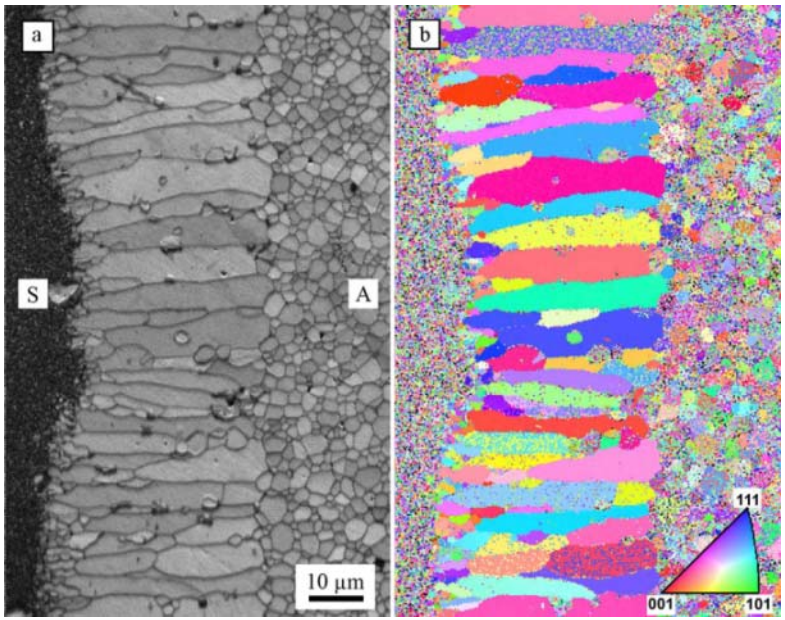
Phase diagram of the MgO – Al_2O_3 system

Jung et. al. JPEDAV (2004) 25:329-345



Transmission of 1.73-mm-thick Coors spinel

Harris, Proc. of SPIE Vol. 5786

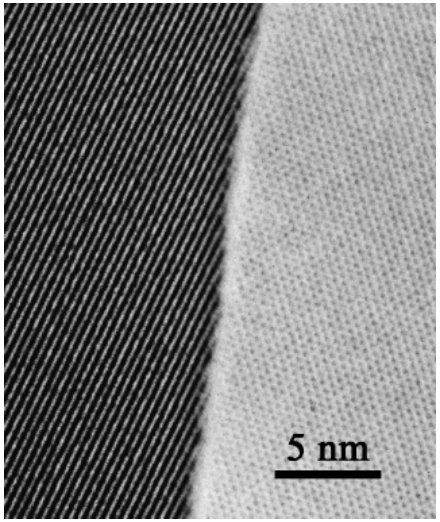


The formation of the interlayer in alumina-spinel co-sintered at 1550°C for 16 h

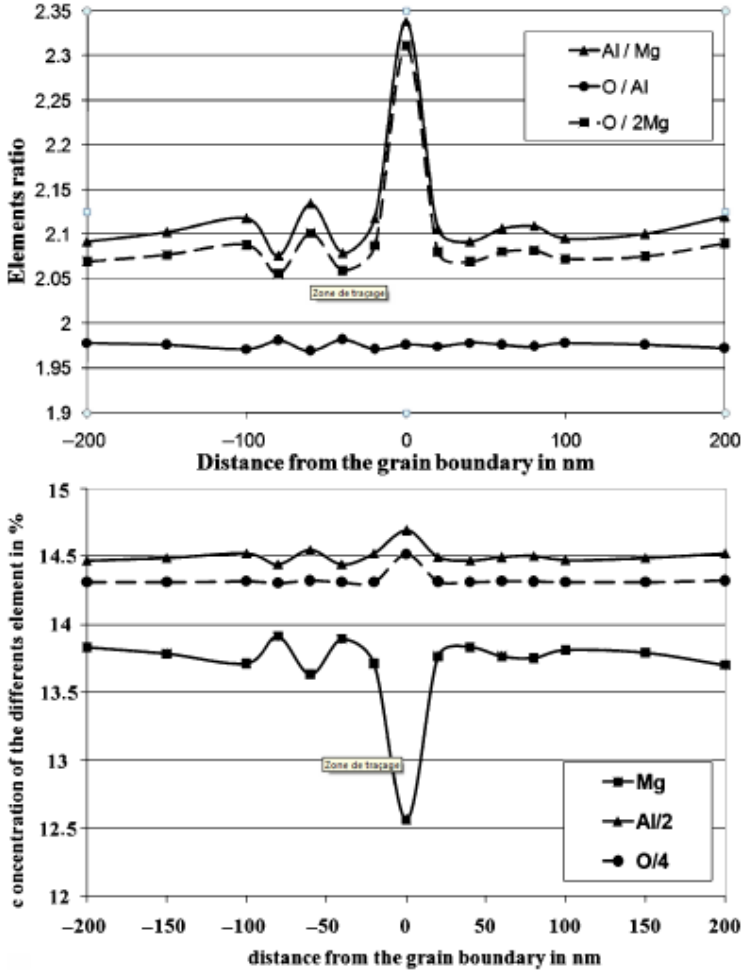
Yalamac et.al. J. Euro. Ceram. Soc (2011) 31: 1649 -1659

Van Capellen and Doukhan's method:

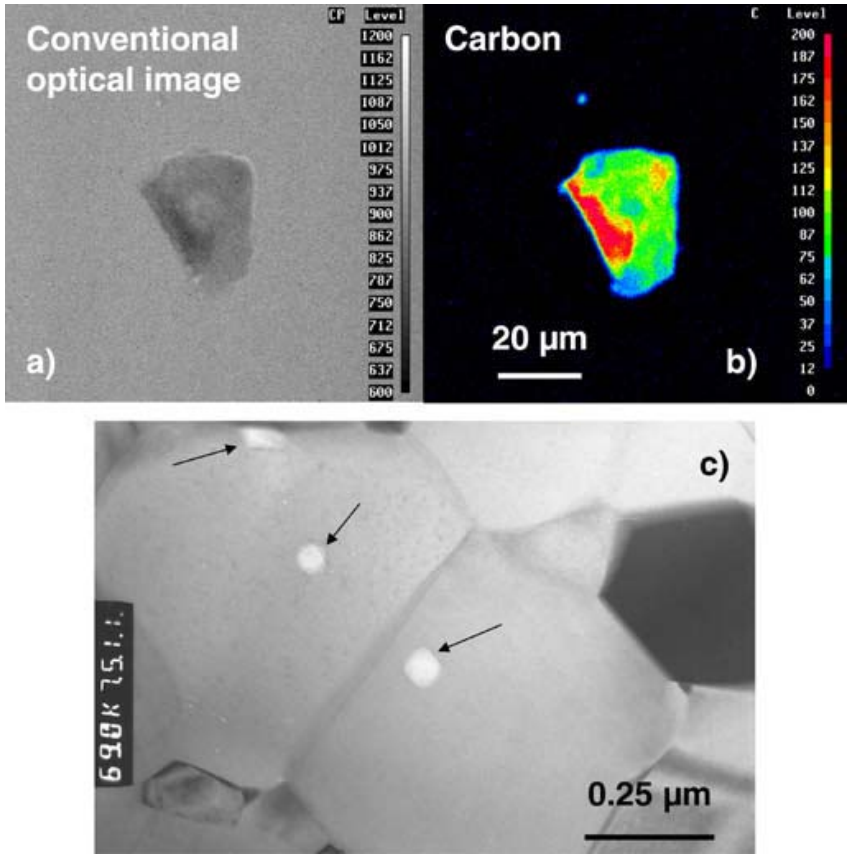
$$\frac{C_A}{C_B} = \frac{I_A}{I_B} K_{A/B} \exp(-\mu t)$$



HRTEM of a grain boundary of MgO 1.05Al_2O_3 HIPed at 1380°C under 190MPa

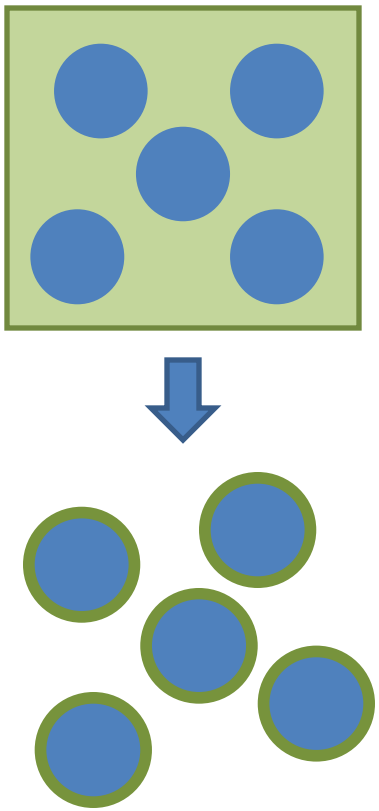


The concentration gradients of Al, Mg and O at sub-grain boundary of nonstoichiometric spinel

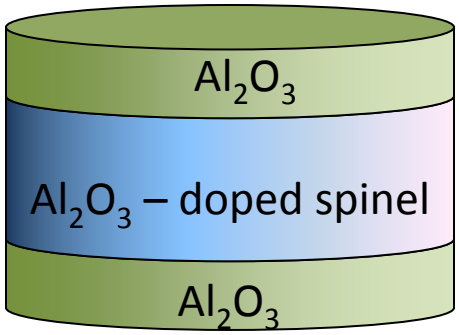


Second-phase imaging of carbon contamination and residual pores in SPSed spinel
(a)optical aspect (b) carbon (c) residual pores

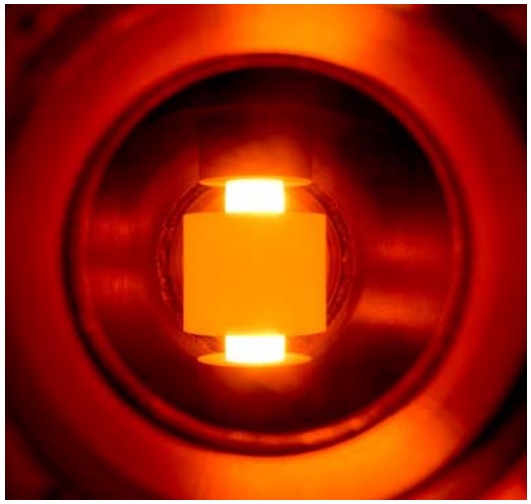
Bernard-Granger et.al. Scripta Materialia
60 (2009) 164–167



Colloidal processing

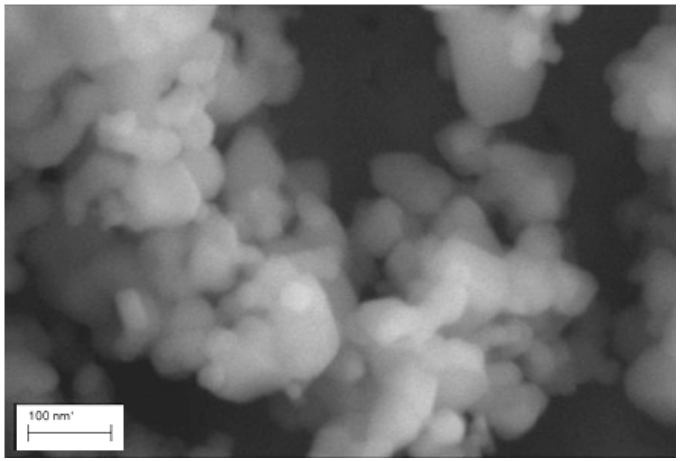


Alumina – spinel co-sintering

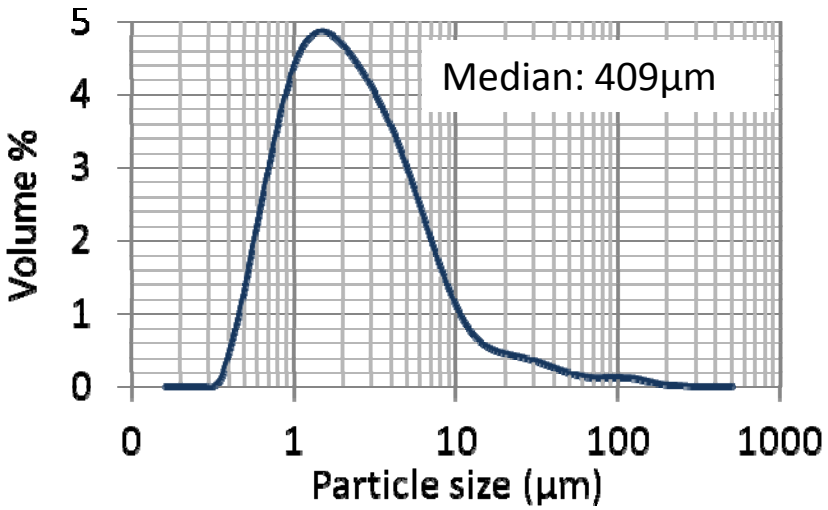


Spark plasma sintering

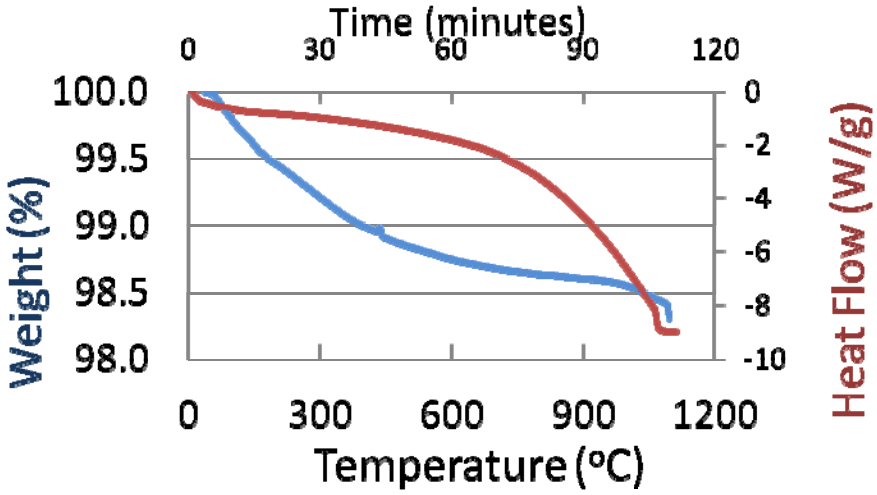
Baikowski spinel S30CR



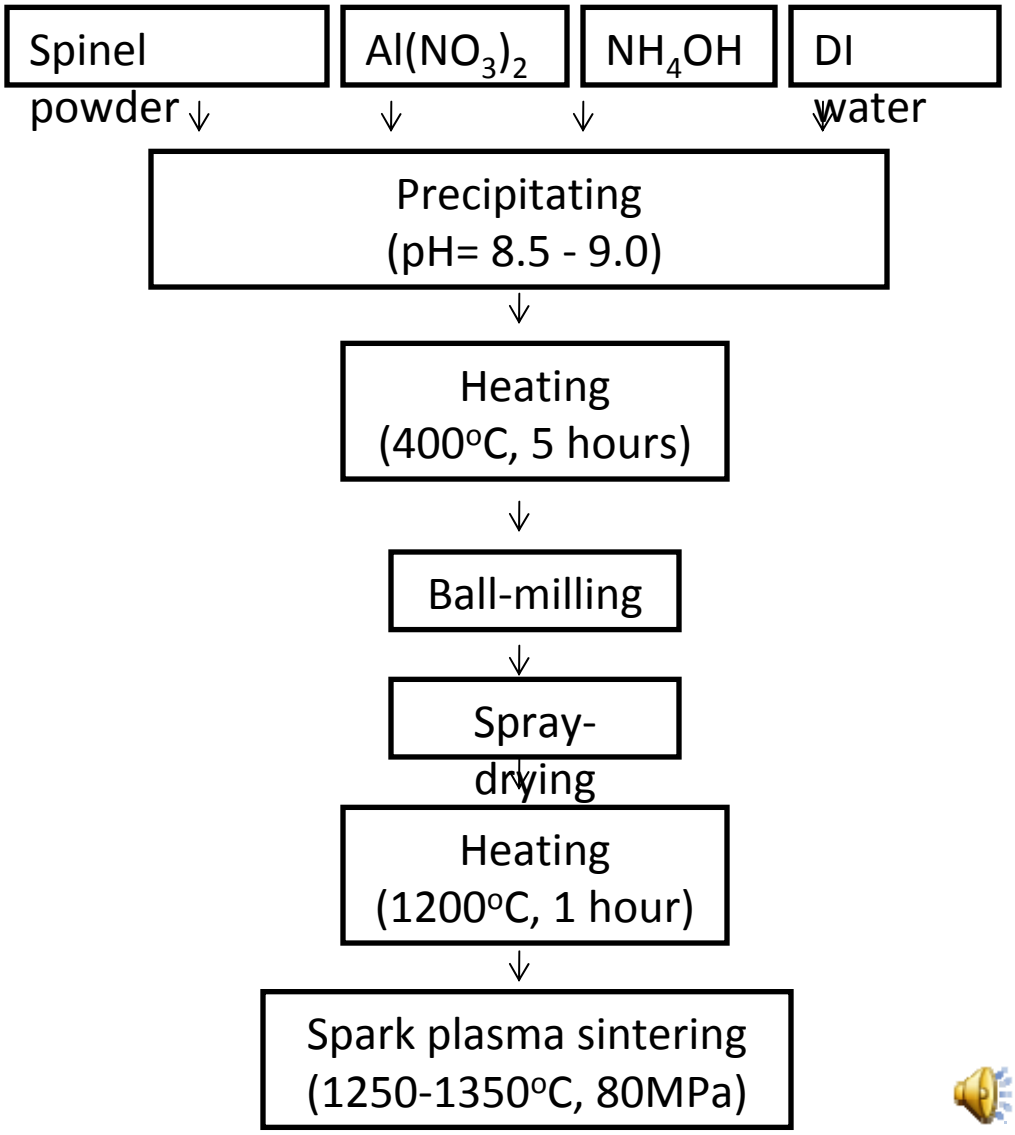
Particle size distribution

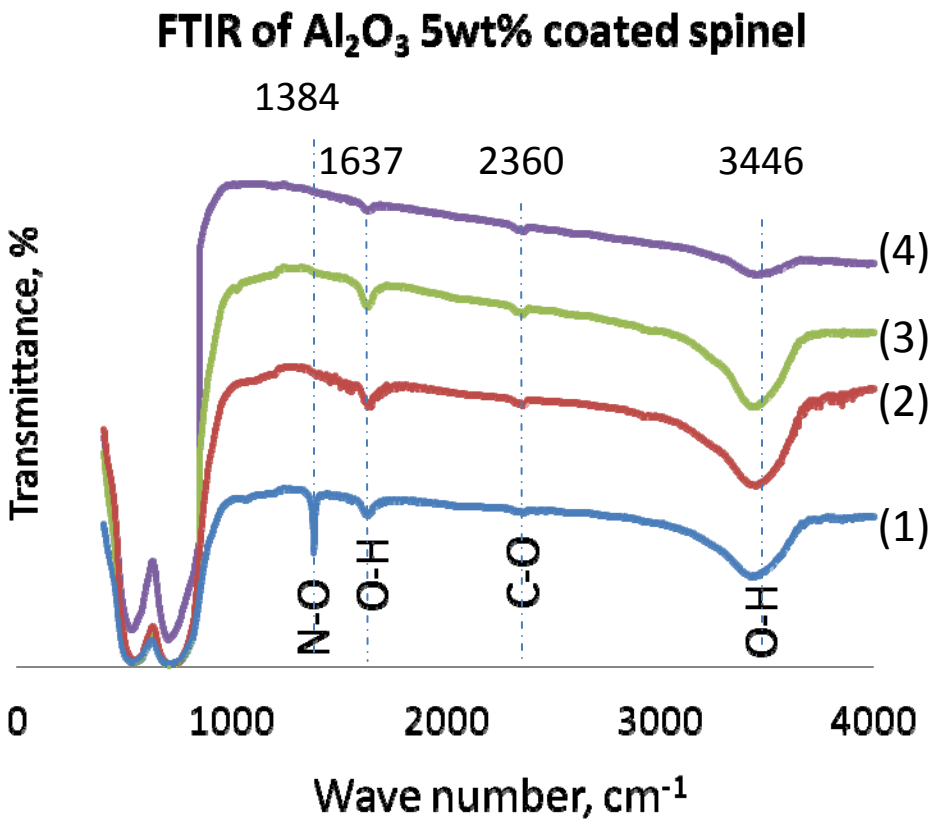


DSC – TGA of the as-received spinel



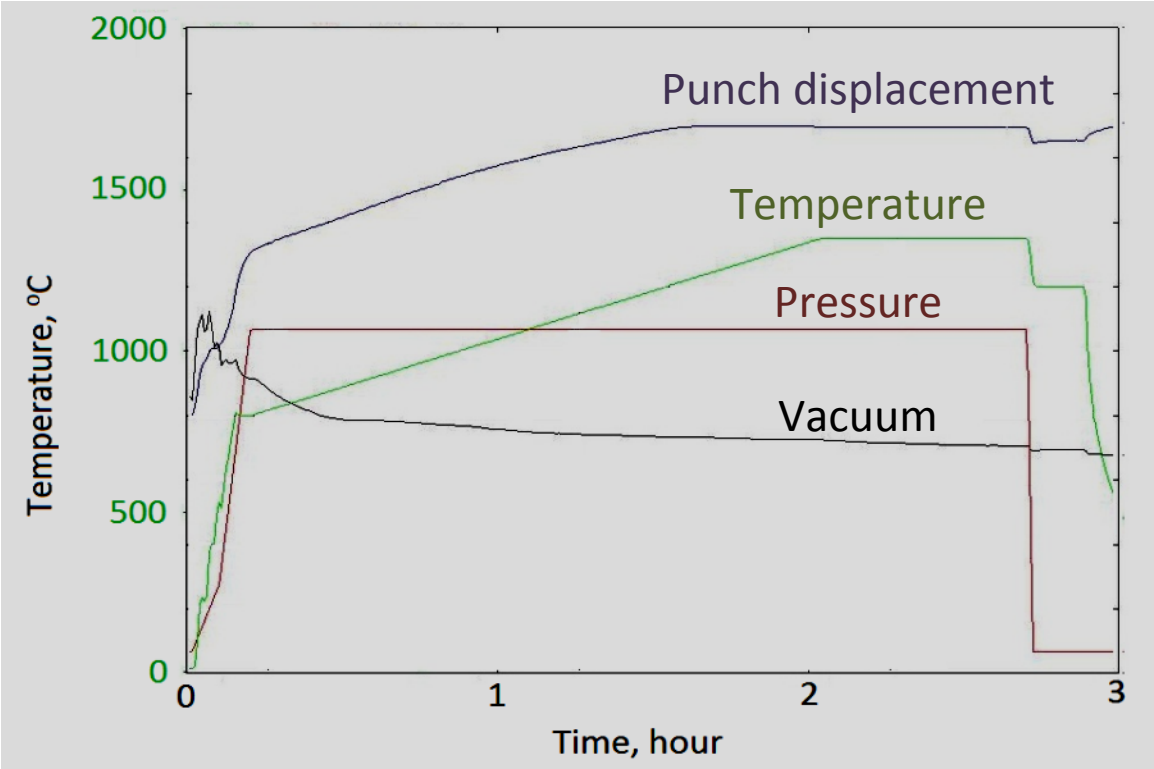
Label	Doping wt%	T_{SPS} °C	T_a °C
S0A	0	1250, 1300, 1350	1100
S1A	1	1250, 1300, 1350	1100
S3A	3	1250, 1300, 1350	1150
S5A	5	1250, 1300, 1350	1200

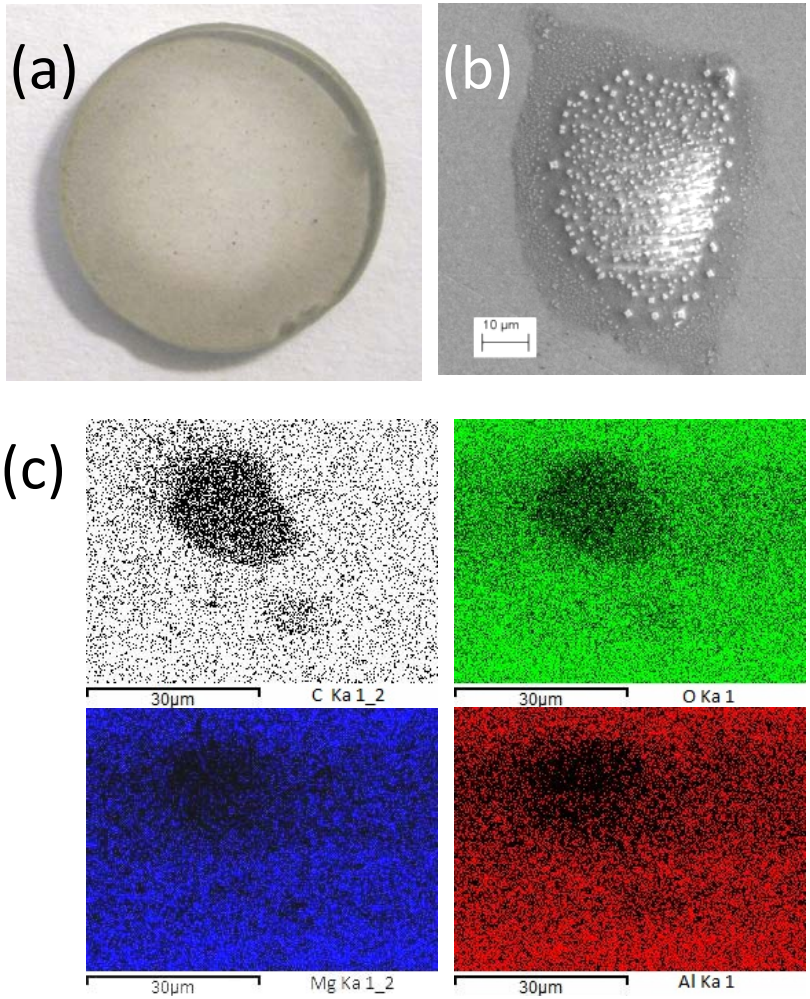




(1)- S5A after heated at 400°C, 10 min.
(2)- S5A after heated at 600°C , 10 min.
(3)- S5A after heated at 800°C , 10 min.
(4)- S5A after heated at 1000°C , 10 min.

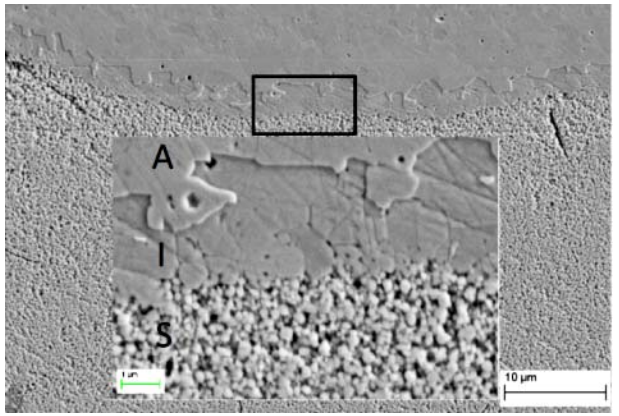
Punch displacement, temperature, pressure, and vacuum profiles during a SPS cycle

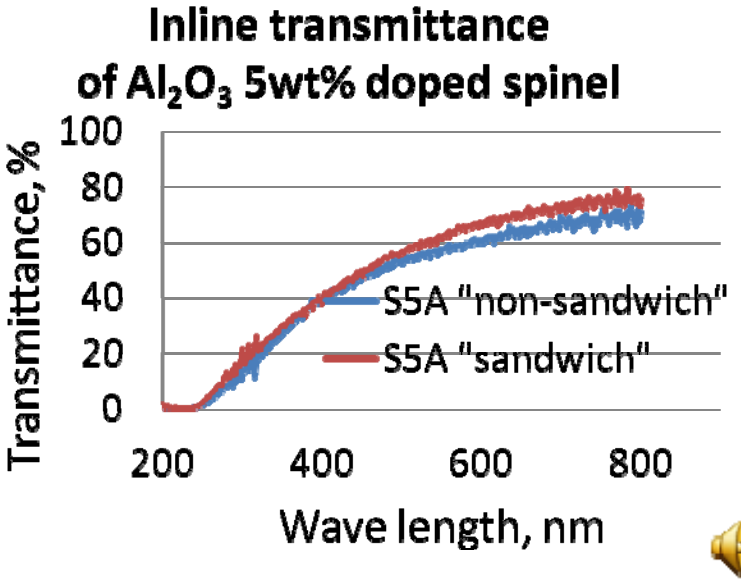




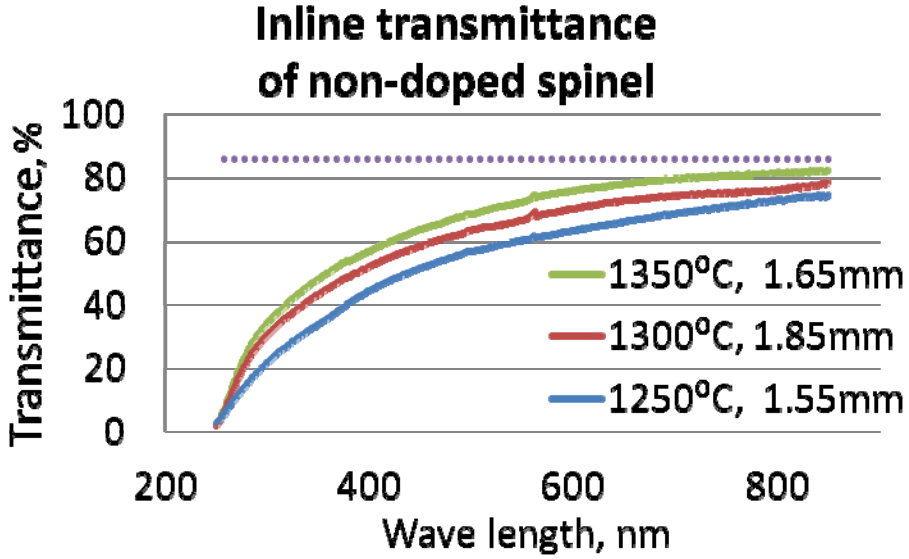
(a) optical, (b)SEM, and (c) EDS imaging of carbon- contaminated region

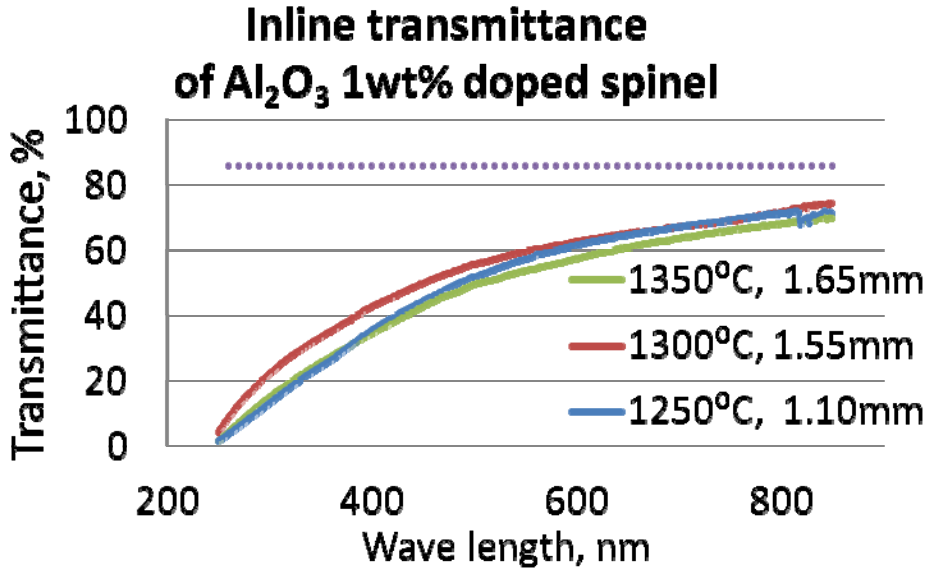
Co-SPSed alumina - Spinel

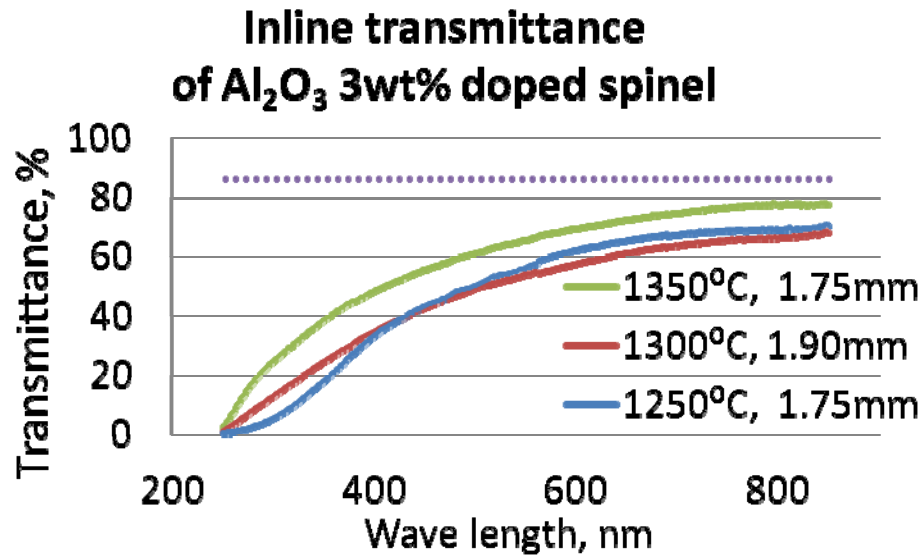


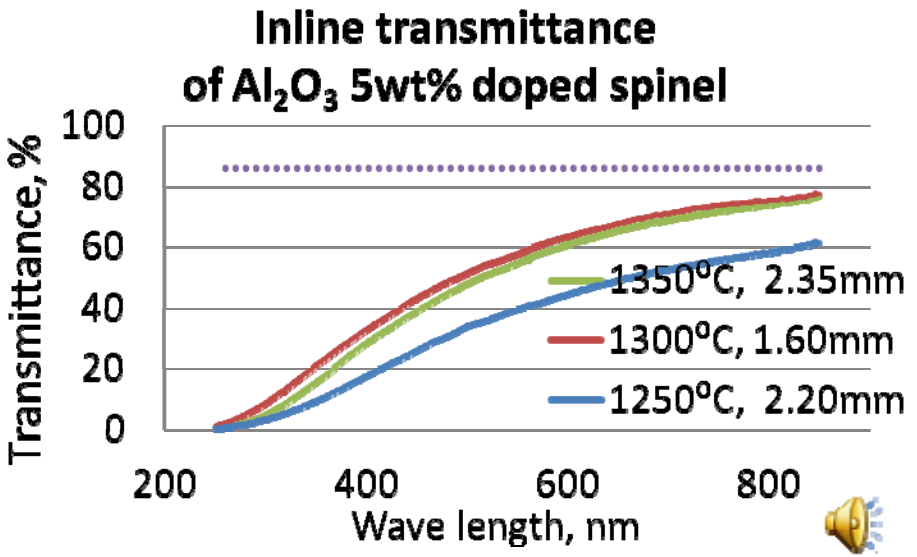



S0A_1250
S0A_1300
S0A_1350
S1A_1250
S1A_1300
S1A_1350
S3A_1250
S3A_1300
S3A_1350
S5A_1250
S5A_1300
S5A_1350

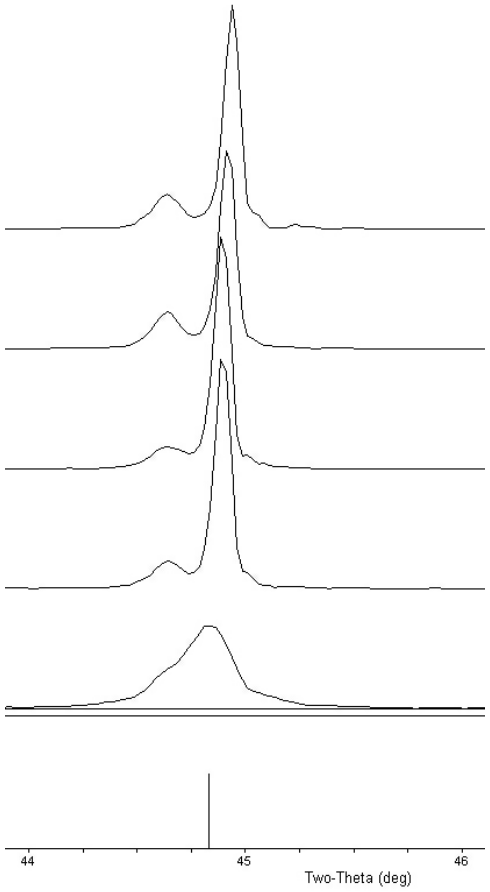

Inline transmittance of non-doped spinel
Transmittance, %
100
80
60
40
20
0
200
400
600
800
Wave length, nm
1350°C, 1.65mm
1300°C, 1.85mm
1250°C, 1.55mm

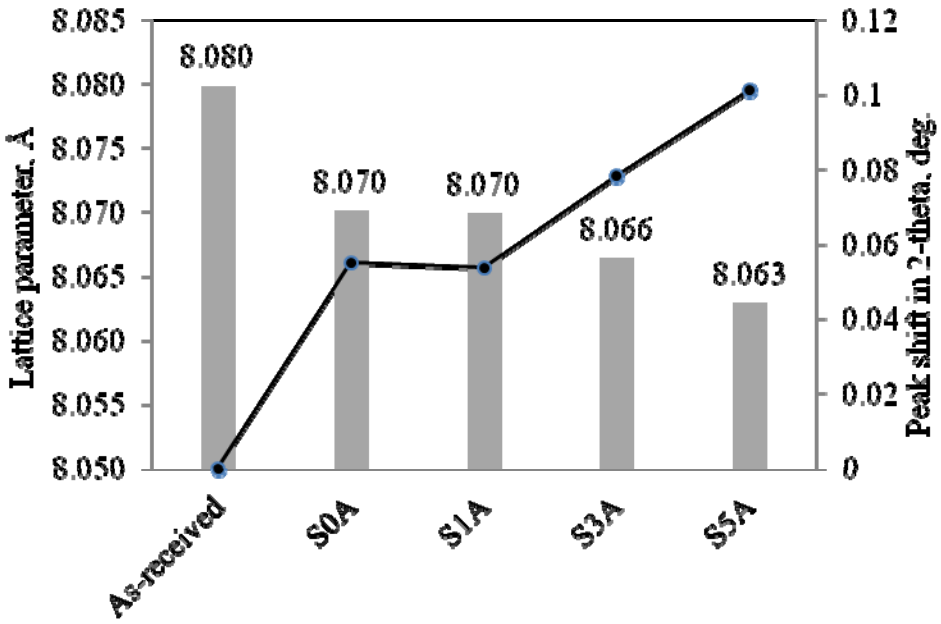

Inline transmittance of Al2O3 1wt% doped spinel
Transmittance, %
100
80
60
40
20
0
200
400
600
800
Wave length, nm
1350°C, 1.65mm
1300°C, 1.55mm
1250°C, 1.10mm


Inline transmittance of Al2O3 3wt% doped spinel
Transmittance, %
100
80
60
40
20
0
200
400
600
800
Wave length, nm
1350°C, 1.75mm
1300°C, 1.90mm
1250°C, 1.75mm

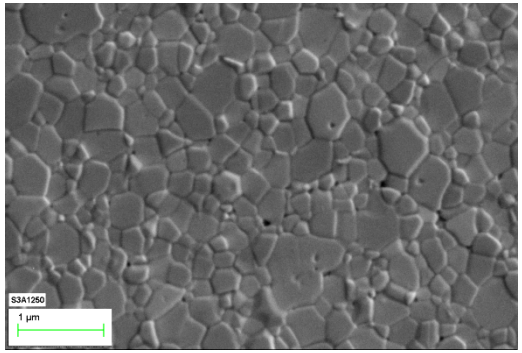

Inline transmittance of Al2O3 5wt% doped spinel
Transmittance, %
100
80
60
40
20
0
200
400
600
800
Wave length, nm
1350°C, 2.35mm
1300°C, 1.60mm
1250°C, 2.20mm

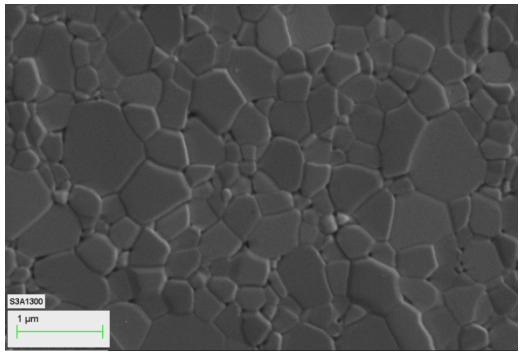
Al ions take place in the spinel lattice and change the lattice parameter

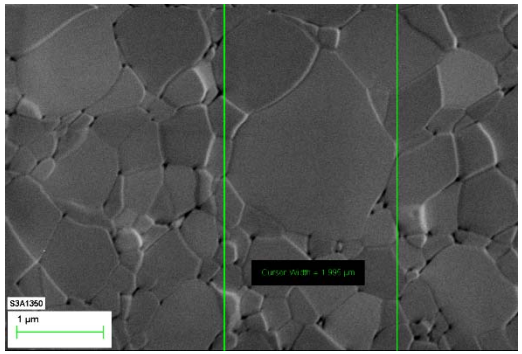


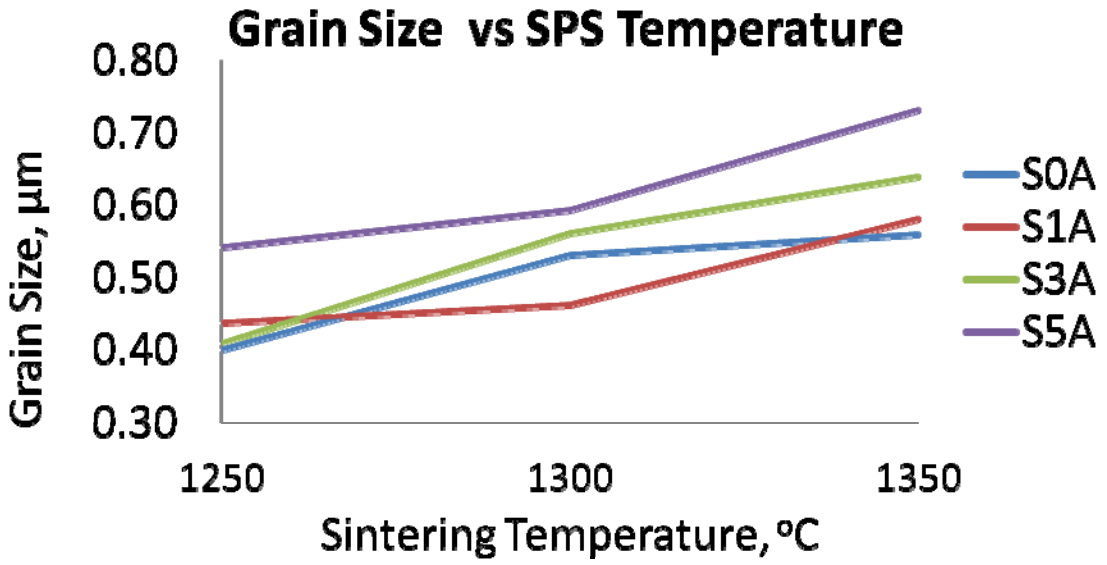


Lattice parameters and peak shift of spinel samples SPSed at 1300ºC



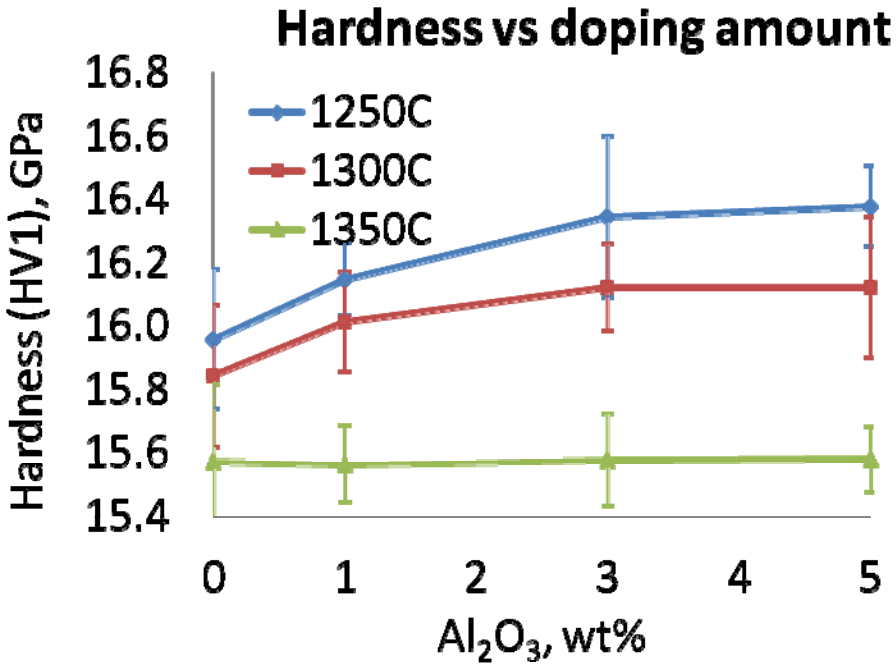




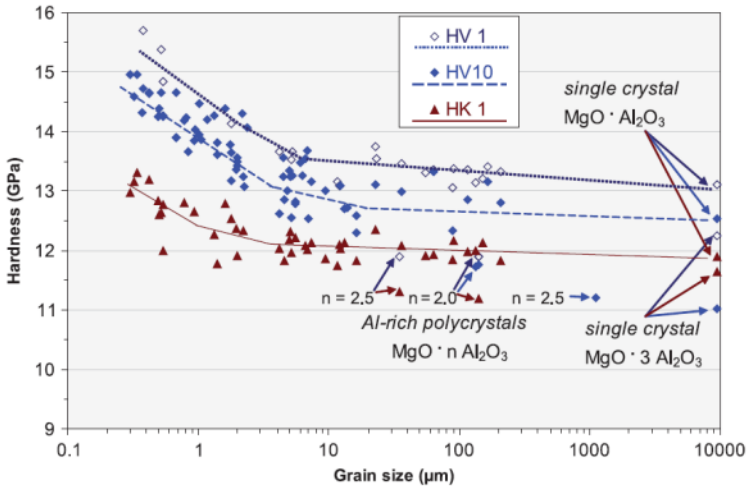


Sample	Ave. grain size, nm, at		
	1250°C	1300°C	1350°C
S0A	474	655	671
S1A	437	463	581
S3A	409	562	639
S5A	542	593	731

Sample	Density, g/cm^3, at		
	1250°C	1300°C	1350°C
S0A	3.575	3.576	3.578
S1A	3.577	3.575	3.575
S3A	3.578	3.577	3.575
S5A	3.581	3.579	3.579



Sample	Hardness (HV1), x10² GPa		
	1250°C	1300°C	1350°C
S0A	1596	1584	1557
S1A	1615	1601	1556
S3A	1634	1612	1558
S5A	1638	1612	1558



Krell and Bales
Int. J. Appl. Ceram. Technol. (2011)
8 [5] 1108–1114

- "Sandwich" SPS helped to increase the optical transmittance by forming a carbon diffusion barrier.
- Although the sintering temperature was not high enough, Al ions did take place in the spinel lattice and changed the lattice parameter
- The doping of Al_2O_3 has improved the hardness at SPSed temperature < 1350°C
- Samples SPSed at 1250°C performed the highest hardness values, but their transmittance was the worst
- Samples SPSed at 1350°C did not show an improvement in hardness at all doping levels
- Spinel doped with 3wt% Al_2O_3 or 5wt% Al_2O_3 and SPSed at 1300°C might be the best choice regarding strength and transmittance
- The densification should be further improved

Future work

- Study the effects of Y_2O_3 and Y_2O_3 + Al_2O_3 additives on the microstructure, strength and transmittance of spinel

- Characterize the microstructure of spinel using HRTEM

Thank you for your attention!