

Optical and Mechanical Properties of Nano-Composite Optical Ceramics

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Submicron and Nanostructured Ceramics

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Introduction

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- **Background**
 - Current MWIR transparent materials
 - Nano-composite oxides
- **Processing/Microstructures**
- **Optical Properties**
- **Mechanical Properties**
- **Summary**



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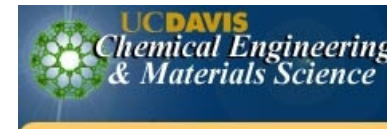
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Maximizing the Optical and Mechanical Performance

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The goal is to achieve all of the following simultaneously:

DARPA Goals:

- High Strength - equivalent to Sapphire
- Scaleable Method - able to produce 3” domes
- MWIR Transparent - equivalent to Spinel

Raytheon “Stretch Goal”

- MWIR Transparency - equivalent to Yttria

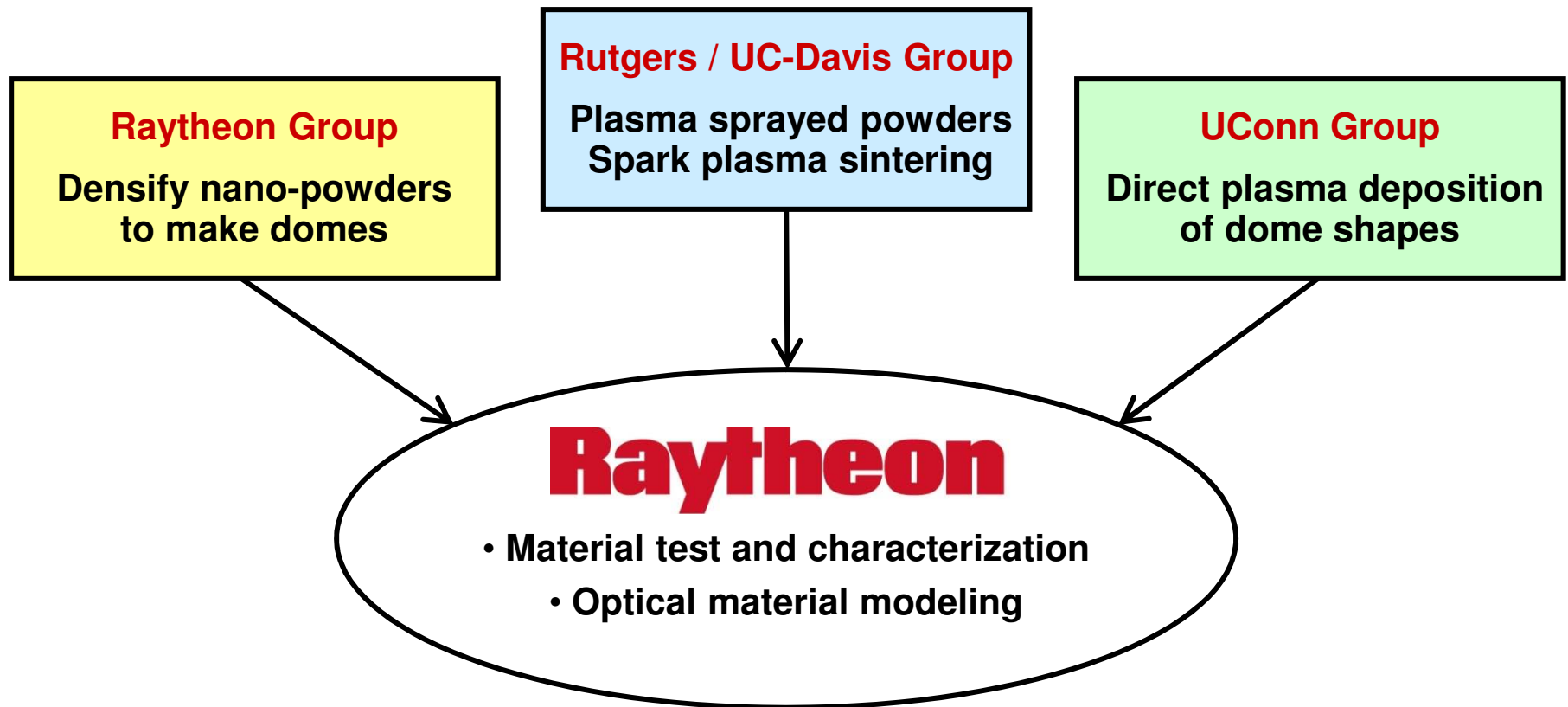
To achieve these goals:

- No pore phase (large $\Delta n \sim 0.8$ = scatter)
- Minimize grain size / grain growth ($G.S. \leq \lambda/20$ for transparency)
- Uniform 2-phase microstructure (small $\Delta n < 0.2$)
- Avoid MWIR absorptions due to Si-O and Al-O bonds



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Project Approaches



Background

Sapphire (single crystal Al_2O_3) is the current MWIR dome material of choice.

- High strength
- Excellent erosion durability (rain/sand)
- High thermal shock resistance
- Low optical scatter
- Intrinsic birefringence
- Lacks full 3-5 μm transparency (absorption at 5 μm)
- Significant MWIR emission at elevated/operating temperatures
- High temperature mechanical properties degradation
- High cost due to single crystal growth and optical finishing



Objective:

Make much stronger dome materials and retain full MWIR transmittance

	Optical Properties			Mechanical Properties			
	Absorption	Scatter	Optical Isotropy	Mech. Strength	Impact Resist.	Thermal Shock Resist.	Machinability
Sapphire							
Spinel							
ALON							
Y ₂ O ₃							
MgO							
ZrO ₂							
YAG							

 Excellent
 Marginal
 Poor

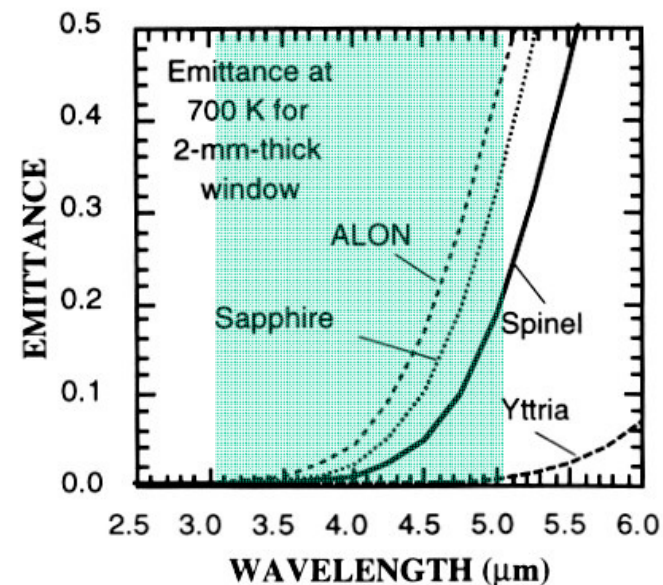
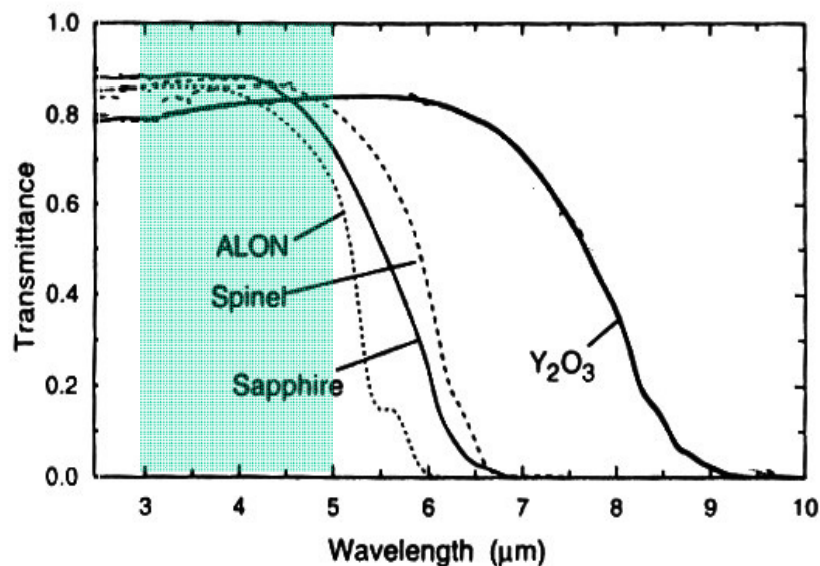
	Absorption	Scatter	Optical Isotropy	Mech. Strength	Impact Resist.	Thermal Shock Resist.	Machinability
Oxide Nano-Composites							



Maximizing the Optical Performance

Problem: Most durable MWIR dome materials contain Al-O bonds. However, Al-O bonds absorb at $\lambda > 4$ microns

Solution: Select nanocomposite systems without Al-O bonds (Y_2O_3 , MgO , ZrO_2)



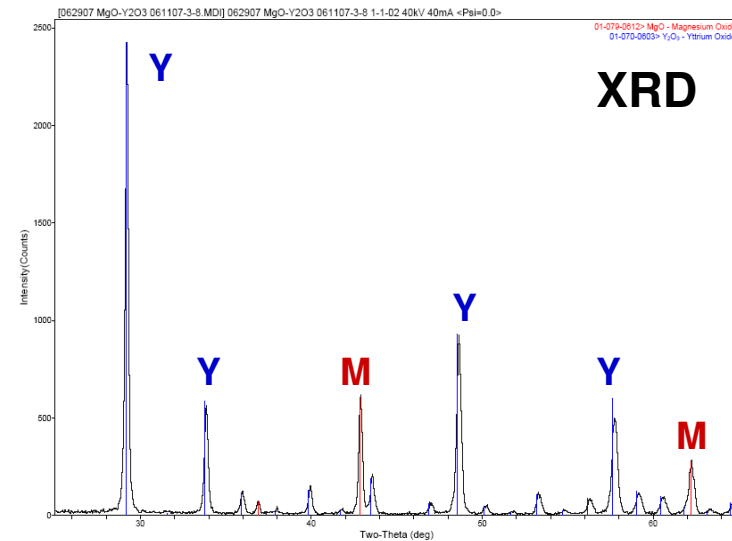
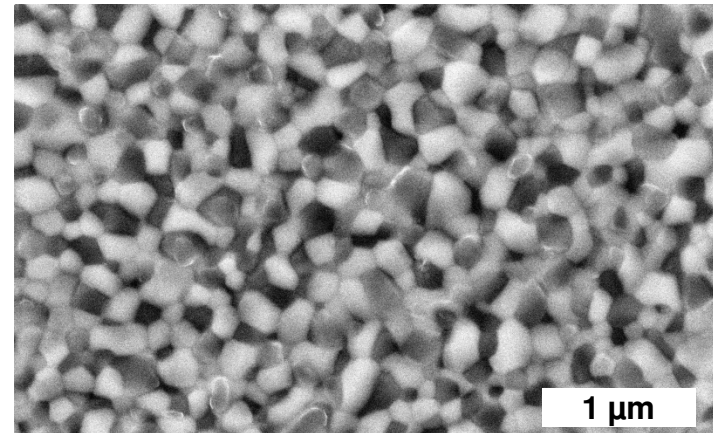
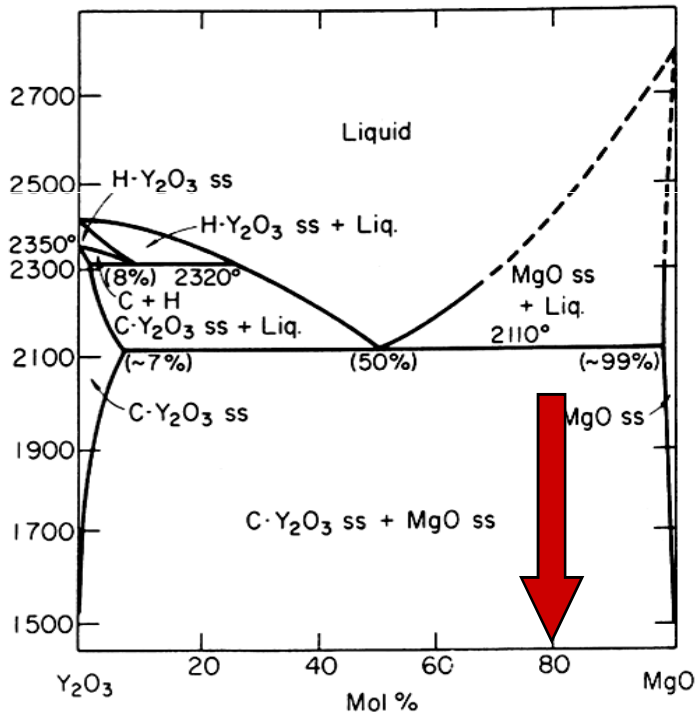
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Nanocomposite Composition

Baseline Material System: Yttria : Magnesia

$\text{Y}_2\text{O}_3:\text{MgO}$
20:80 Mol% $\rightarrow$ $\text{Y}_2\text{O}_3:\text{MgO}$
50:50 Vol%



Nanocomposite Optical Ceramics:

A new class of MWIR dome materials

Approach: *Reduce grain size* of transparent polycrystalline ceramics to *increase strength*:

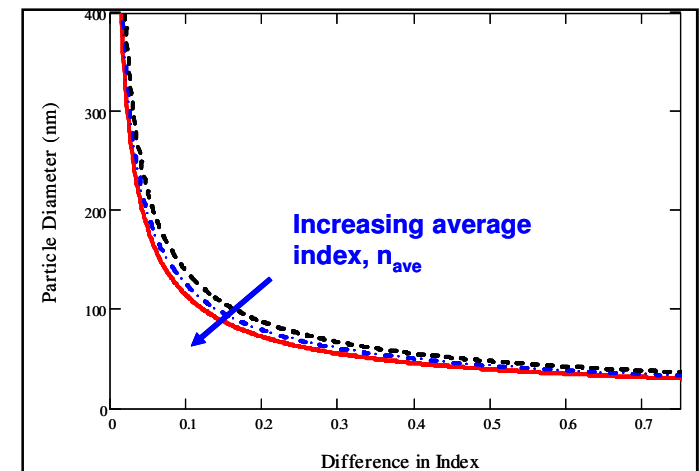
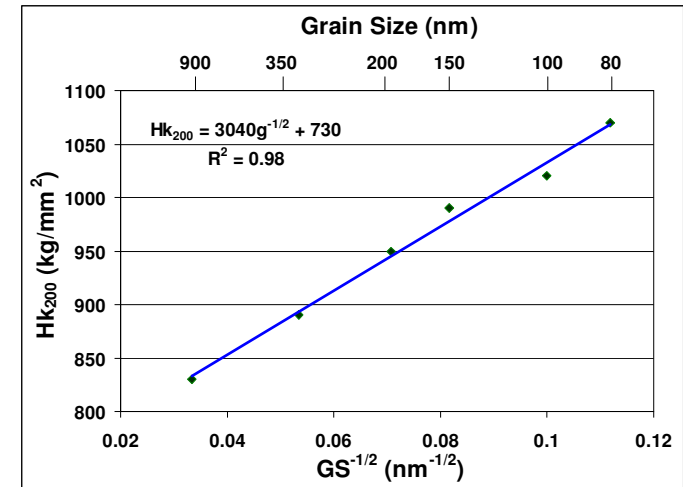
Hall Petch relation: $\sigma \propto (\text{g.s.})^{-1/2}$

Problem: Processing conditions (high T & P) required to densify to optical clarity promote grain growth

Solution: Use significant volume fractions of two or more mutually insoluble transparent ceramics (e.g. $\text{MgO} + \text{Y}_2\text{O}_3$)

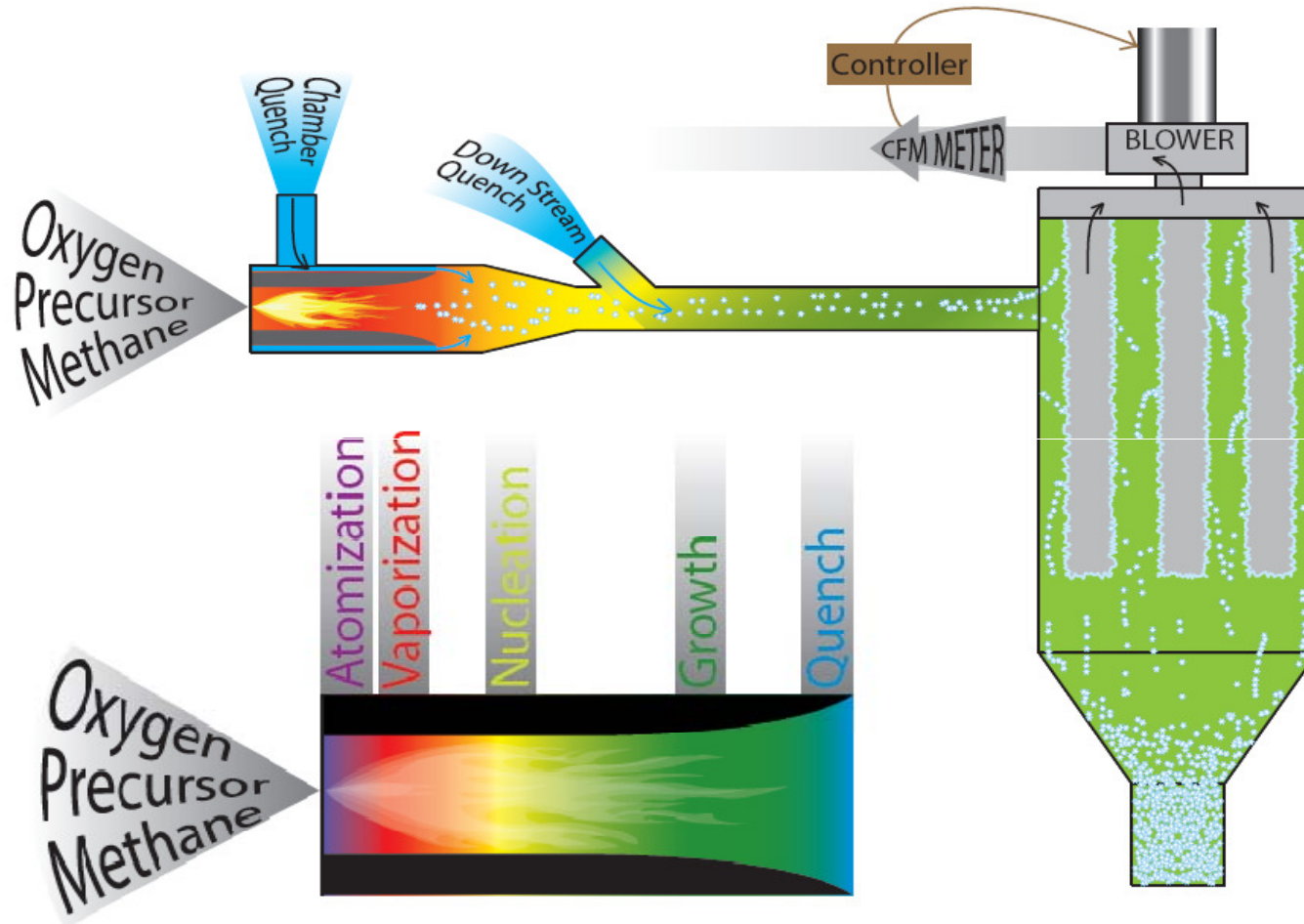
Problem: Refractive index differences between phases cause scattering by the grains

Solution: Reducing the grain size to $< \lambda/20$ eliminates scatter and transparency is restored:
 $4\mu\text{m}/20 = 200 \text{ nanometers !}$



Nanopowder Production via Liquid Flame Spray Pyrolysis

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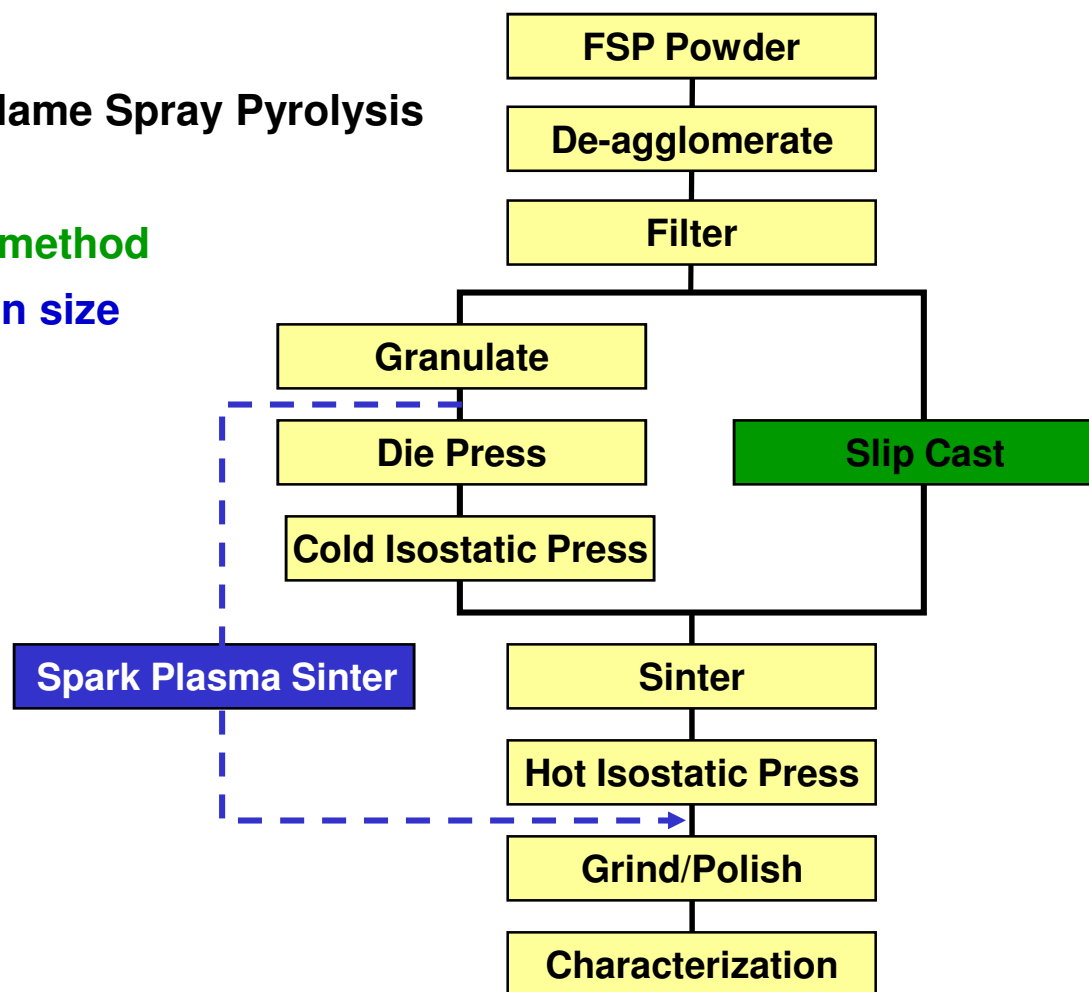


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Process Overview

Powder Process

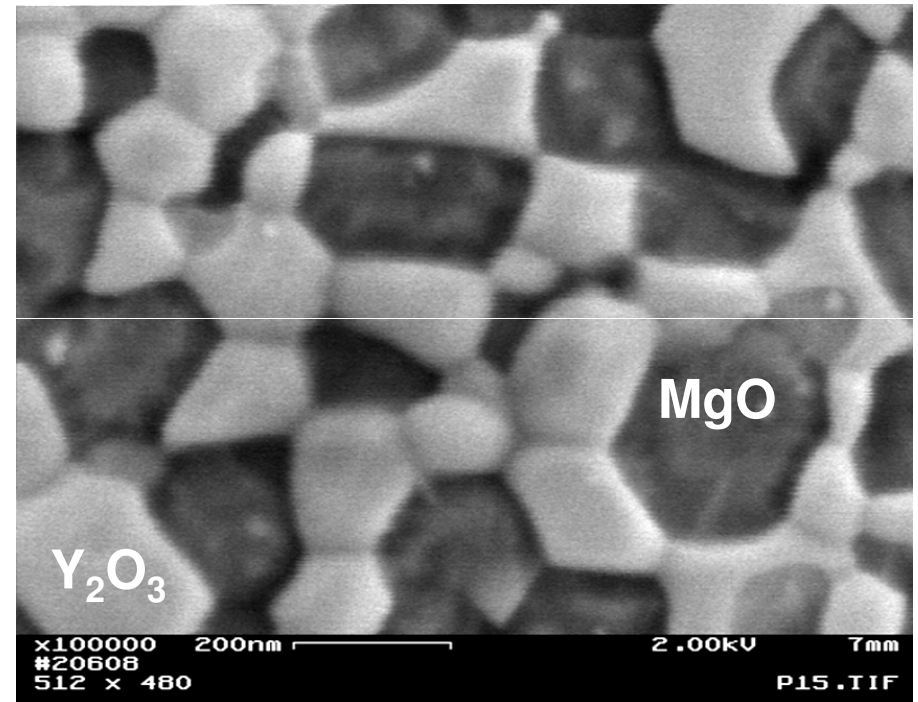
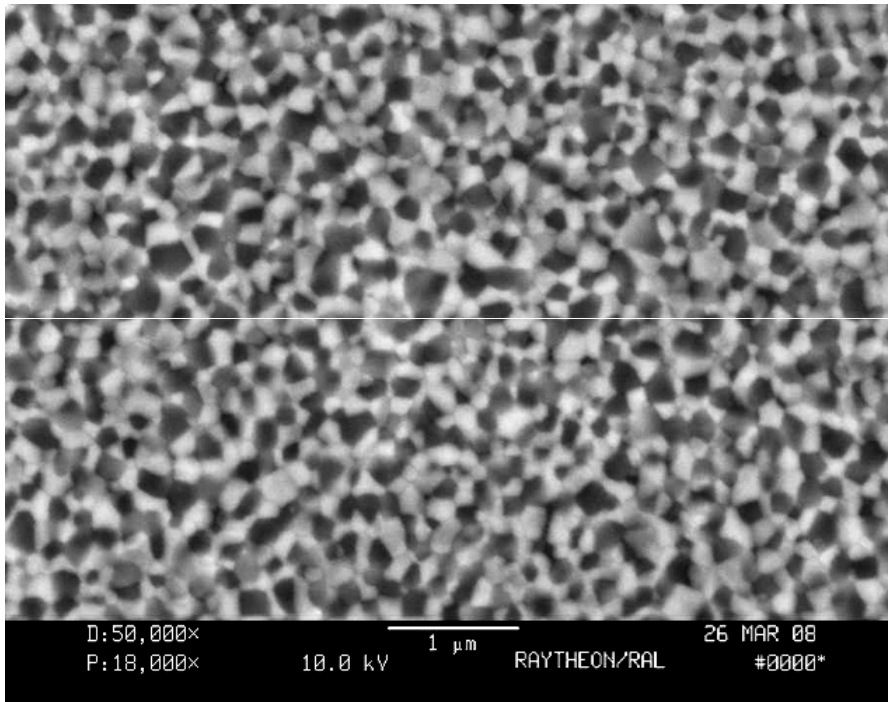
- Starting powders produced by Flame Spray Pyrolysis
- Densified by Sinter + HIP
- Slip Casting – alternate forming method
- SPS densification – smaller grain size



Nanocomposite Microstructure

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Backscattered electron images.
50:50 Vol% Yttria:Magnesia



Uniform microstructure with ~150nm grain size.



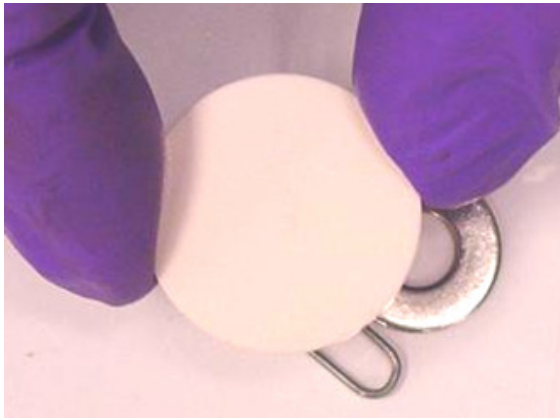
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Optical Properties

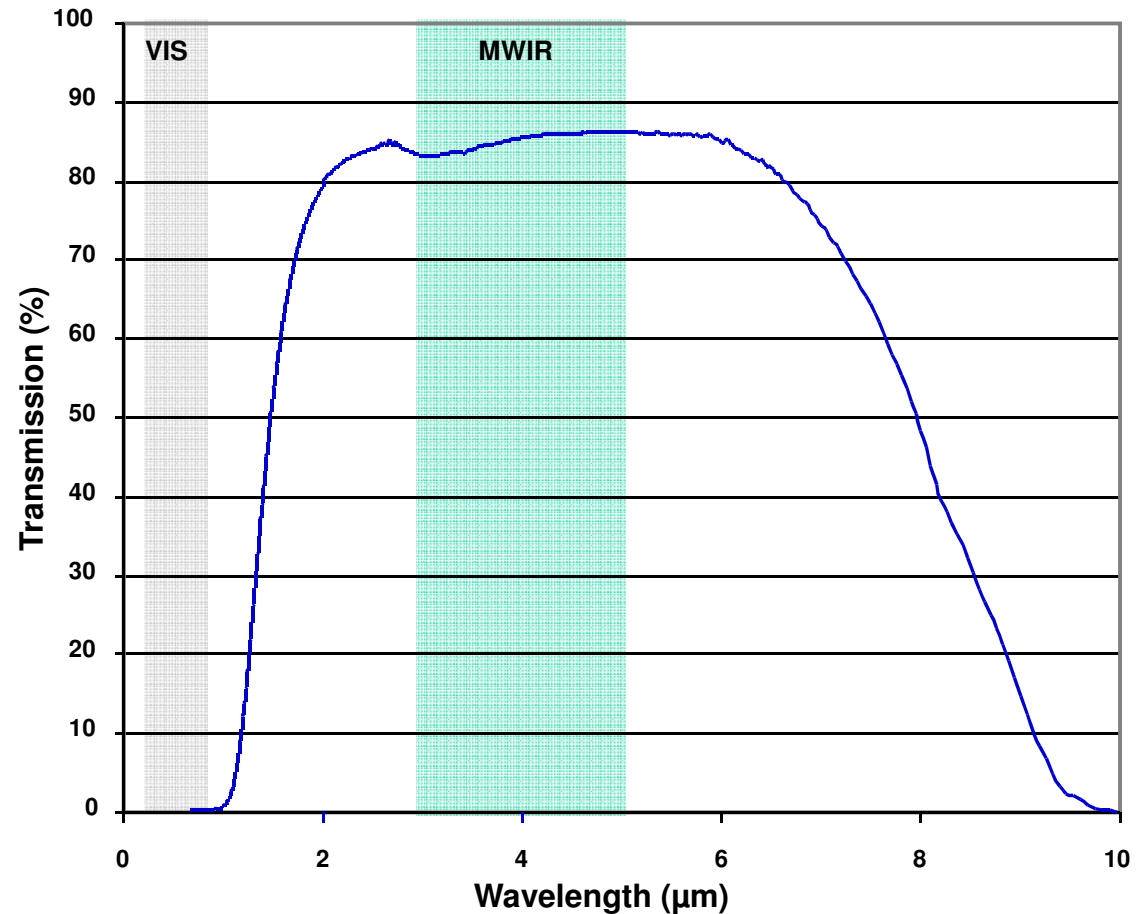
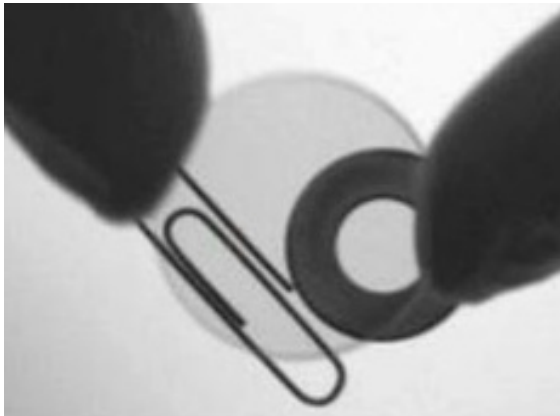
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MgO:Y₂O₃ Nanocomposites

In the visible band



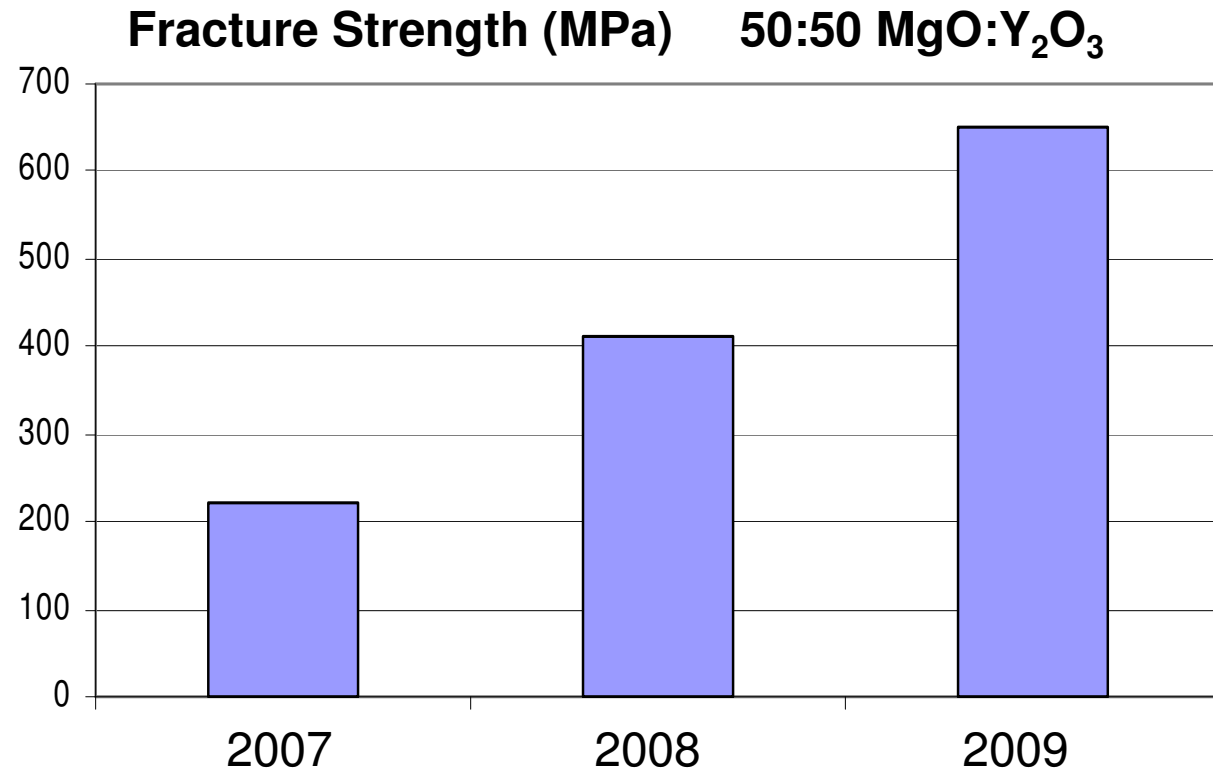
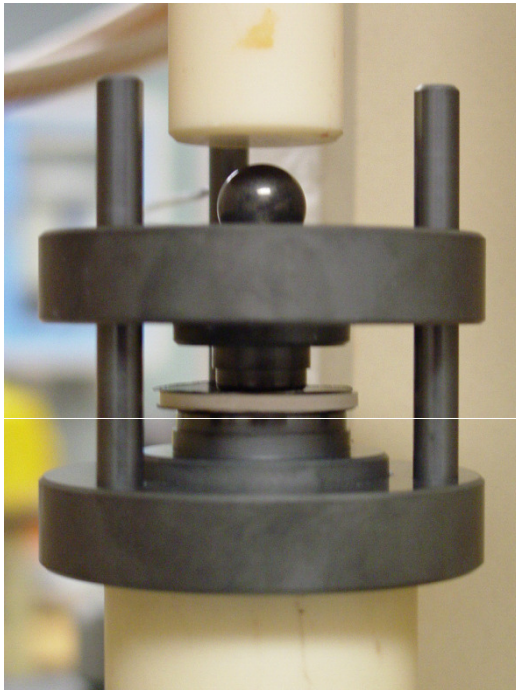
In the MWIR band



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Powder Process Optimization

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- Fracture strength improved with optimized powders and processing.
- New material systems and/or more energetic processing needed for 1200 MPa!



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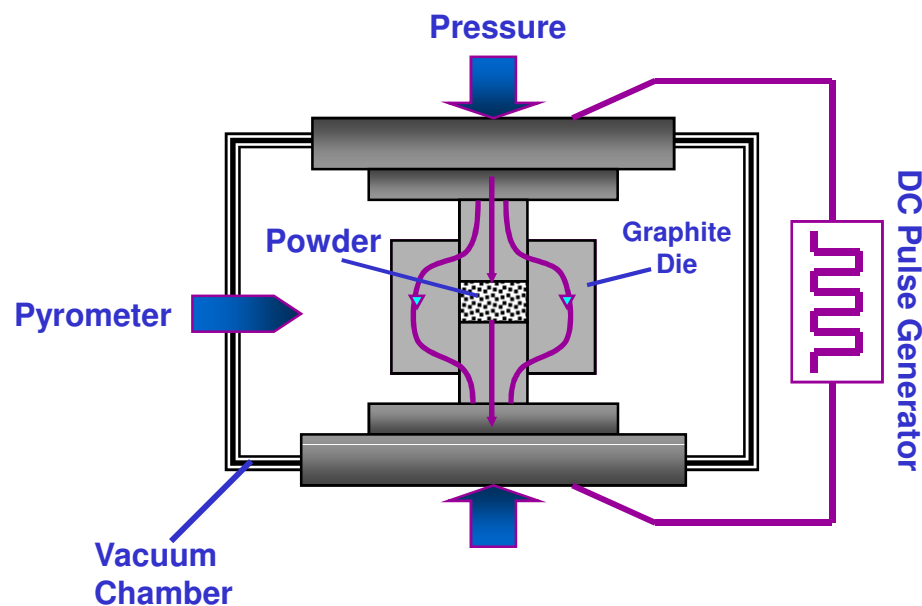
Material Property Goals

Material Property Metrics for 3-5 micron Nano-Composite Optical Ceramics

Material Property	Units	Phase I Metrics	Achieved	Phase II Metrics
Absorption Coefficient (ave, 3-5 μ m)	cm ⁻¹	≤ 0.1	0.05	≤ 0.1
Optical Scatter (Fwd TIS @ 3.39 μ m)	%	≤ 2.0	0.6	≤ 0.5
Fracture Strength at 600°C (average of 10 biaxial disks)	MPa	≥ 600	650	≥ 1200
Hardness (μ -indent: 50g load)	kg/mm ²	≥ 2200	2350	≥ 2200
Thermal Shock Resistance (requires thermal conductivity measurement)	calculated FoM: R'	- - -	1.3 X Sapphire	$\geq 2X$ Sapphire
Sand Erosion Resistance (blowing sand – conditions TBD)	grams/std test	- - -		$\geq 2X$ Sapphire
Water Drop Threshold Velocity (Marshall SFC – 3mm drop)	m/s	- - -		$\geq 2X$ Sapphire

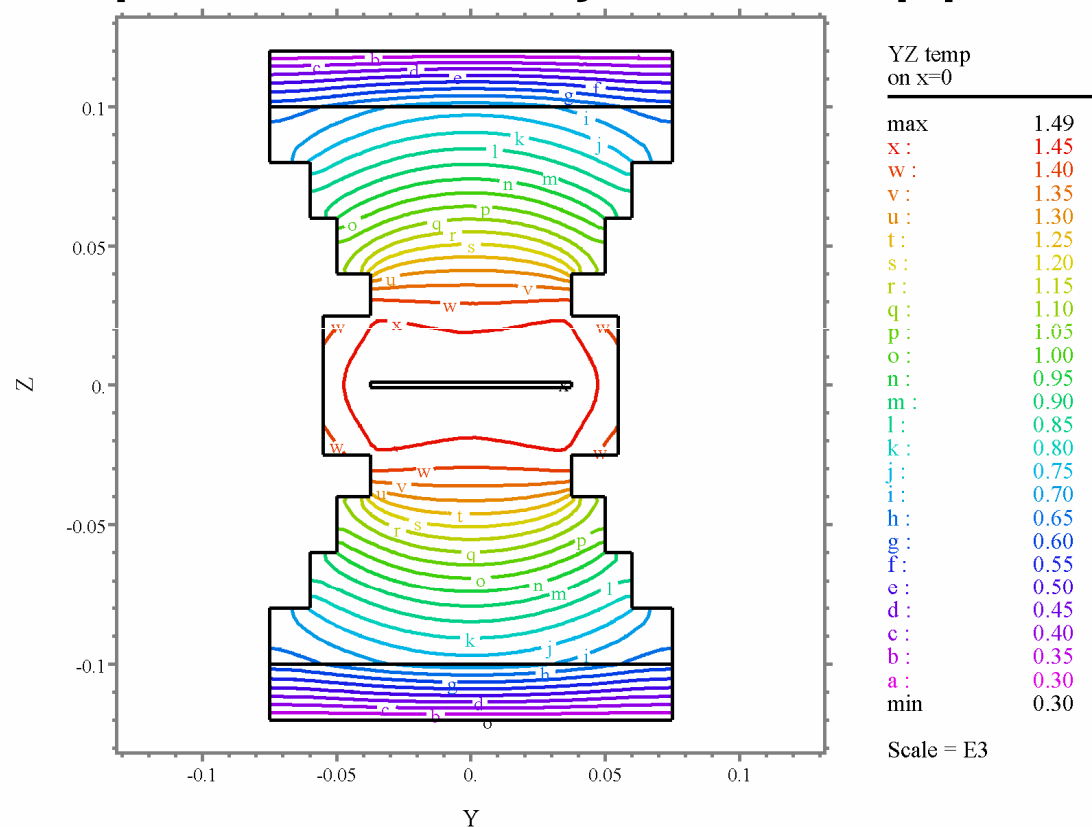


Spark Plasma Sintering

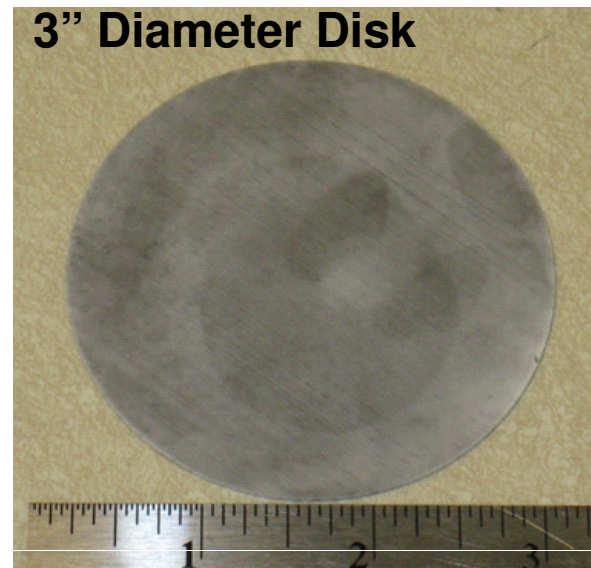


Spark Plasma Sintering

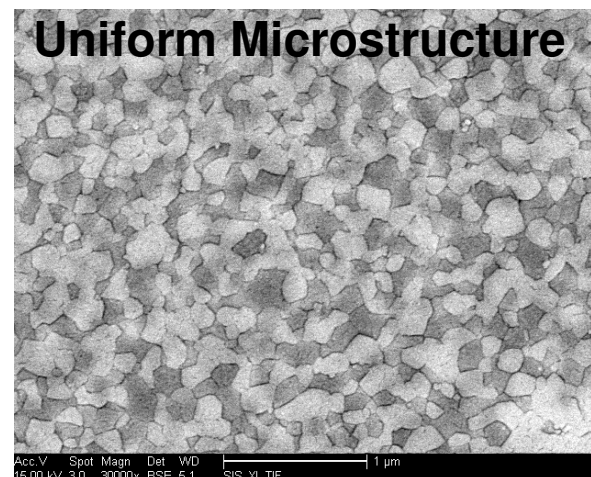
Modeling die geometries to improve temperature uniformity in scaled-up process



3" Diameter Disk



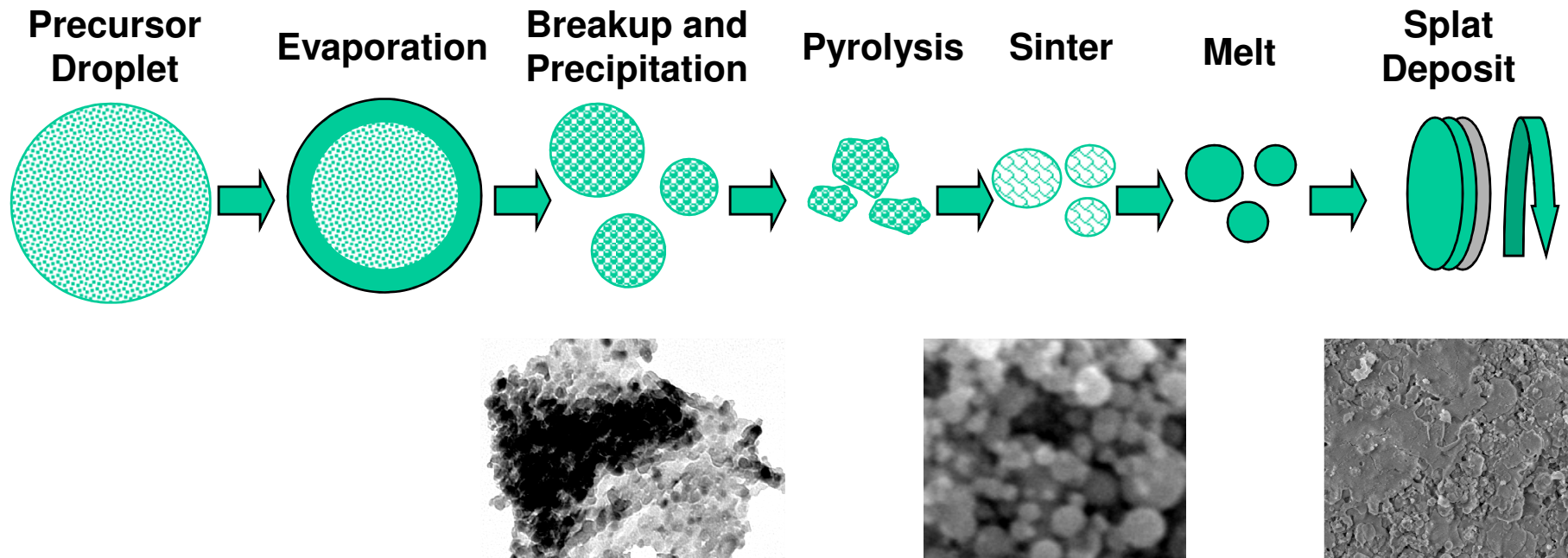
Uniform Microstructure





Direct Deposition

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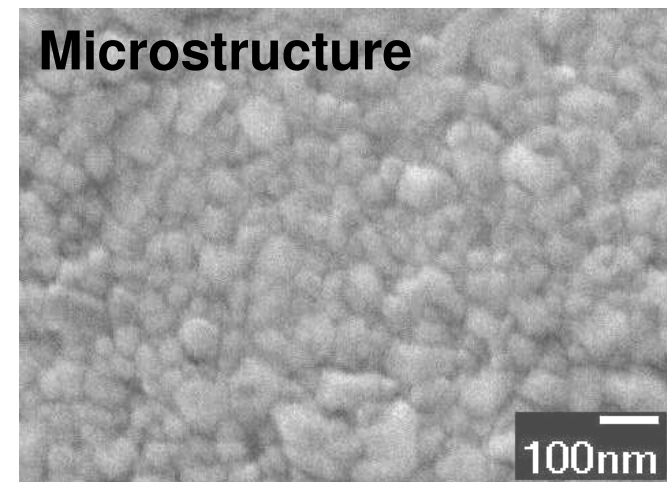
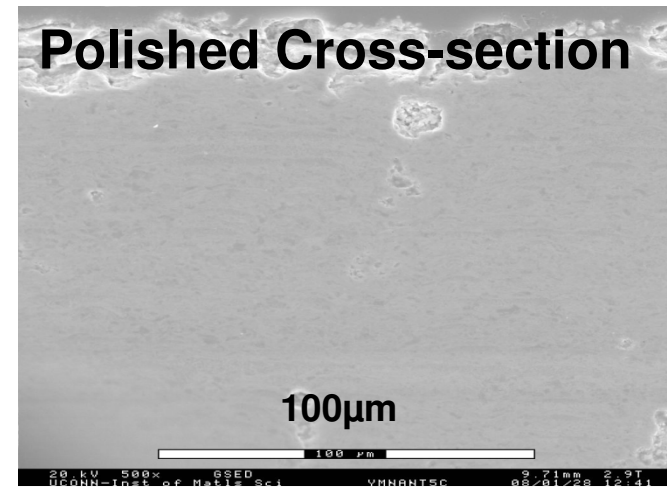
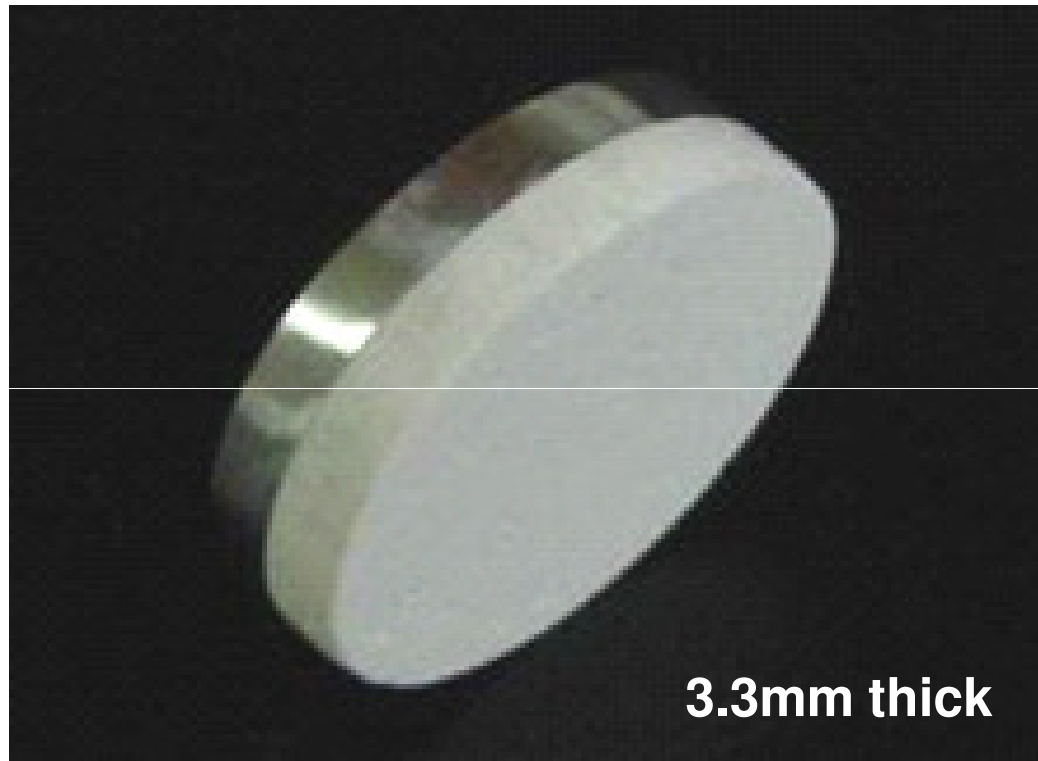
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Direct Deposition

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50:50 MgO:Y₂O₃



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Summary

- **MgO:Y₂O₃ based nanocomposite ceramics have been developed using traditional ceramic processing routes and demonstrated:**
 - **Sapphire equivalent mechanical durability**
 - **Yttria equivalent MWIR optical transparency**
- **New nanocomposite material systems show potential for greater mechanical durability with inherently more durable crystallographic phases.**
- **More energetic fabrication techniques are showing promise for refined microstructures and improved mechanical properties.**

