

# **A New Powder Production Route for Transparent Spinel Windows: Powder Synthesis and Window Properties**

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# Applications of TDA's Surface Functionalized Nanoparticles



## Ceramic Windows



Negative Control  
(No biocides)

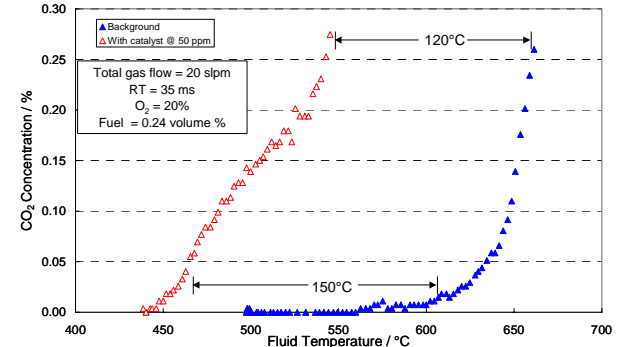
Positive Control  
(0.25% biocides,  
no nanoparticles)

Tethered Biocide  
(0.225% biocide)

Imbedded  
Biocide  
(0.225% IPBC)  
**Zero fungal  
growth**

*Growth on sample  
Surface is less than  
on surrounding agar*

## Nanoparticle Biocides



## Soluble Nanocatalysts

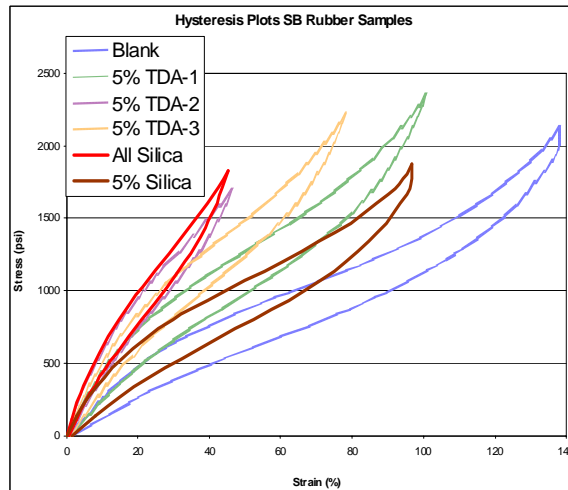


## Nanocolorants



Film edge

## Transparent Nanocomposites



## Hysteresis Improvement in Tires

Alodine 1200/40% Chromate



Alodine 1200/0.5% NanoCl



## Nano Corrosion Inhibitors

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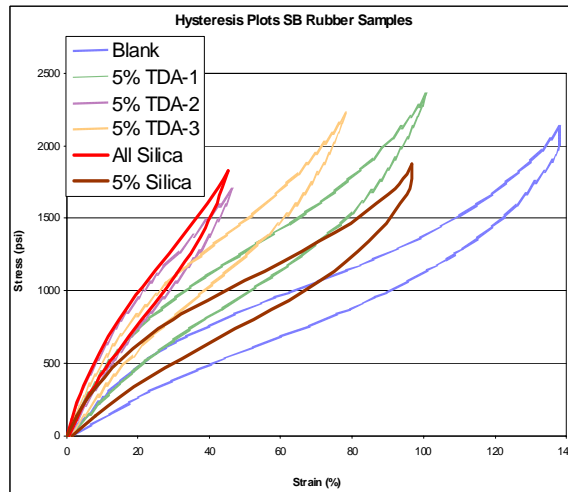


Nanocolorants

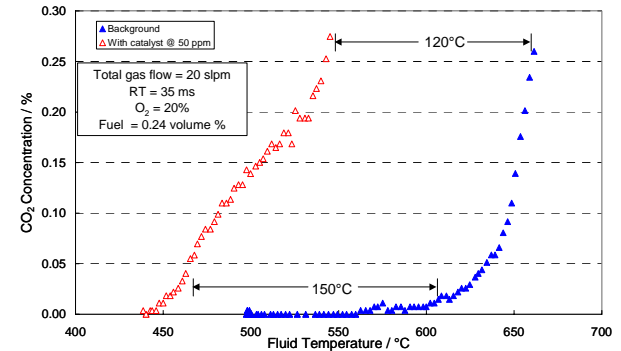


Film edge

Transparent  
Nanocomposites



Hysteresis Improvement  
in Tires



Soluble Nanocatalysts

Alodine 1200/40%Chromate



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Nano Corrosion Inhibitors

# Spinel Powder Production: Needs

- **Impurity Control**
  - With ceramic windows you see your mistakes
    - Both anions and cations degrade optical properties
- **Particle Size Control**
- **Sinterability**
- **Scalability**
- **Cost**

# Methods for Production of Spinel Powders

## ➤ Traditional Methods

### ➤ Ceramics

- Heat Beat and Hope: mixed metal oxide particles mix, heat, grind, repeat.

### ➤ Sol-Gel

- Hydrolyze sol gel: mixed sol-gels or aluminum sol-gel plus Mg complex.

### ➤ Formation of Soluble Complexes

- Various methods including citric acid method

## ➤ Room Temperature Metal Exchange

- Boehmite based

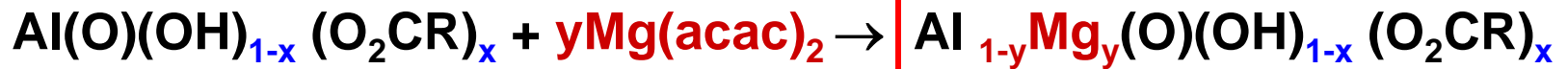
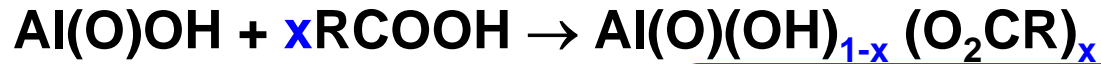
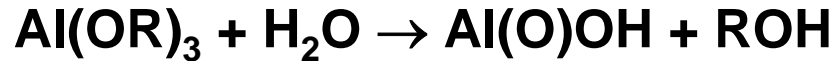
# Boehmite Nanoparticle Based Spinel Powder Production

## ➤ Why Boehmite

- Available in high purity from hydrolysis of aluminum alkoxides
- Hydrolysis conditions have been extensively investigated
  - Particle shape and size are controllable
- Surface modified boehmite undergoes a topotactic metal exchange reaction
- Thermal transformation gives  $\gamma$ -alumina with the spinel structure

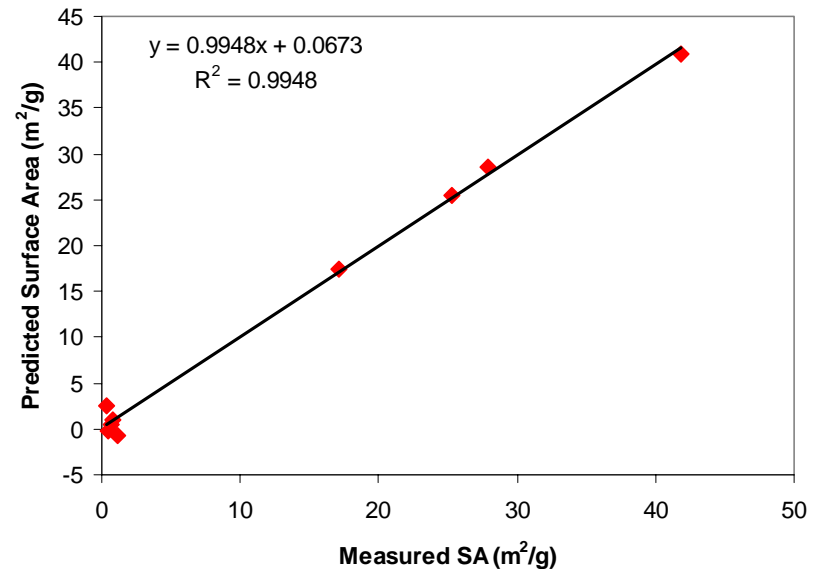


# Powder Processing Flow Chart

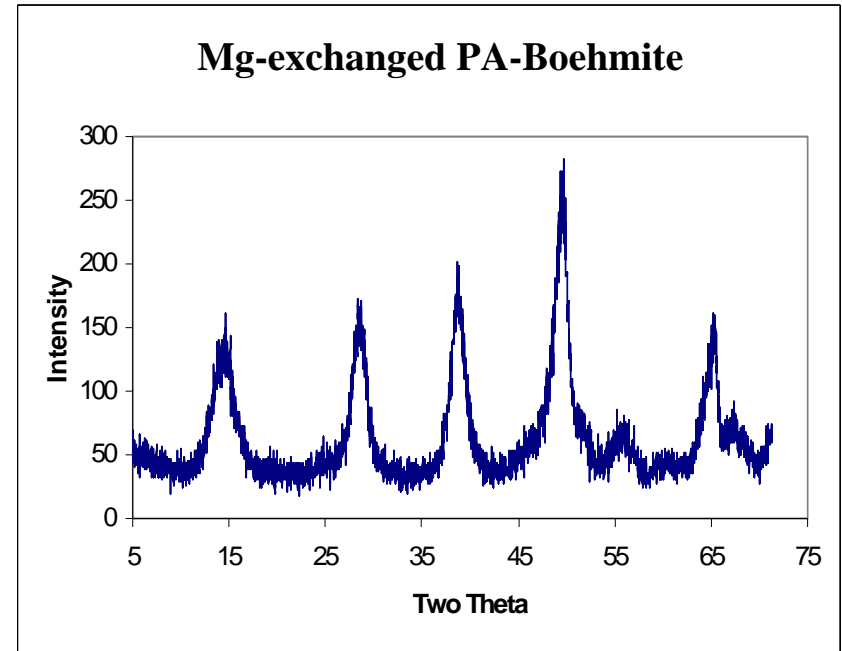
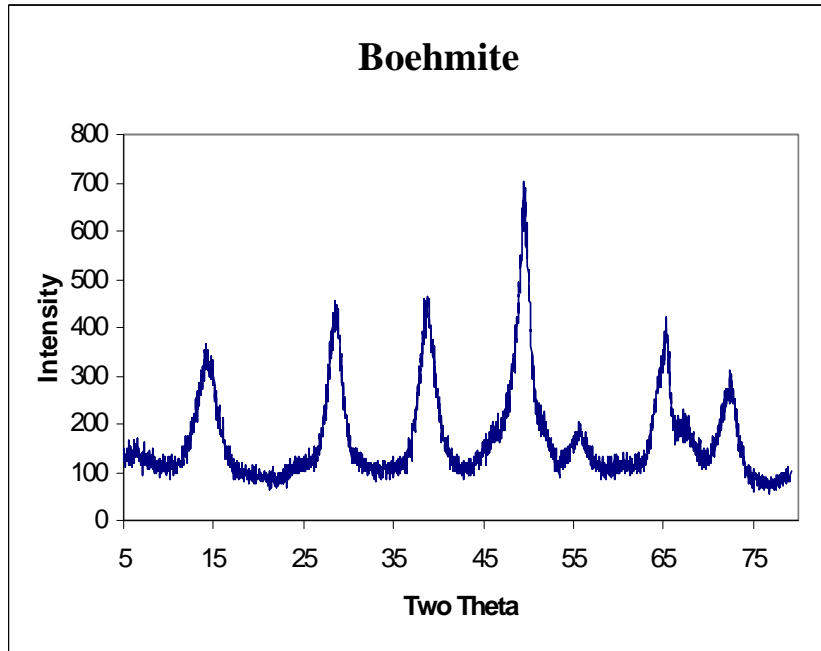


# Controllable Spinel Powder Size

- Synthesis process depends on many co-interacting process variables
- Polynomial neural network used to model synthesis parameters
- Three variables (hydrolysis time, hydrolysis temperature and “ripening time”) control surface area.

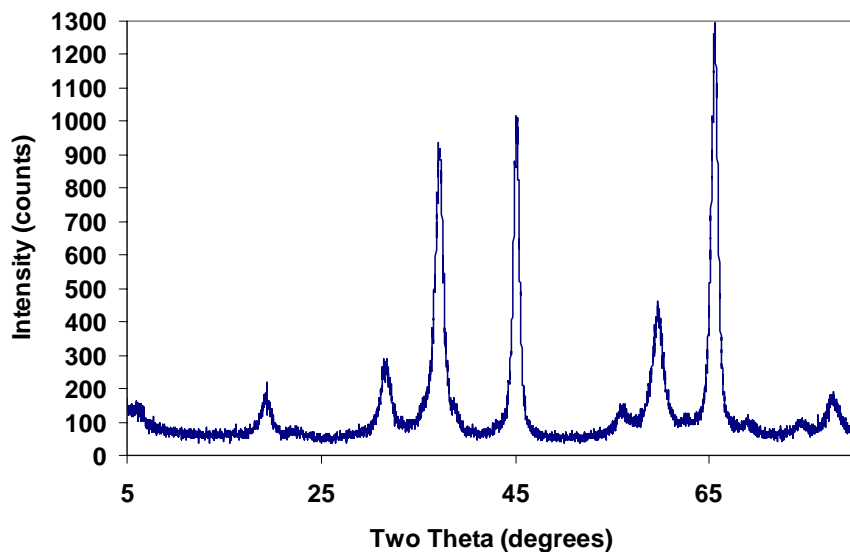


# Topotactic Metal Exchange Process

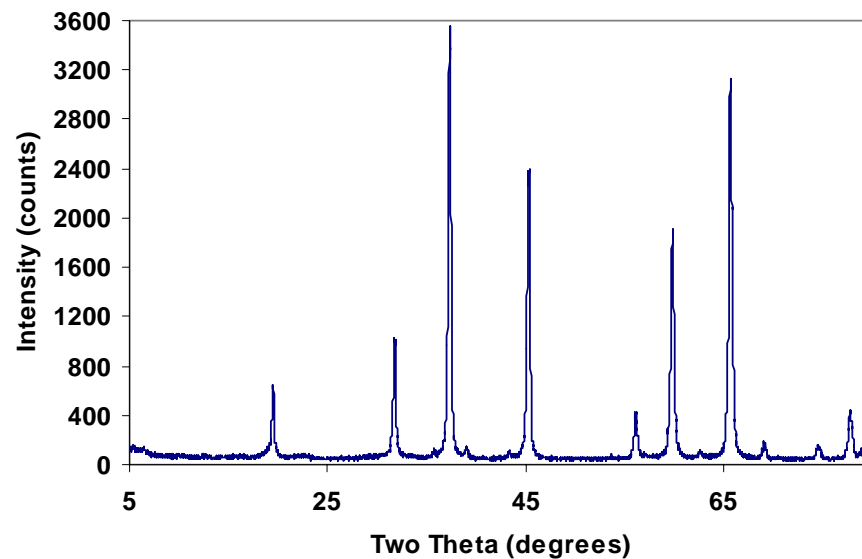


Boehmite crystal structure is unaltered by metal exchange

# Spinel Formed Directly From Metal Exchanged Boehmite



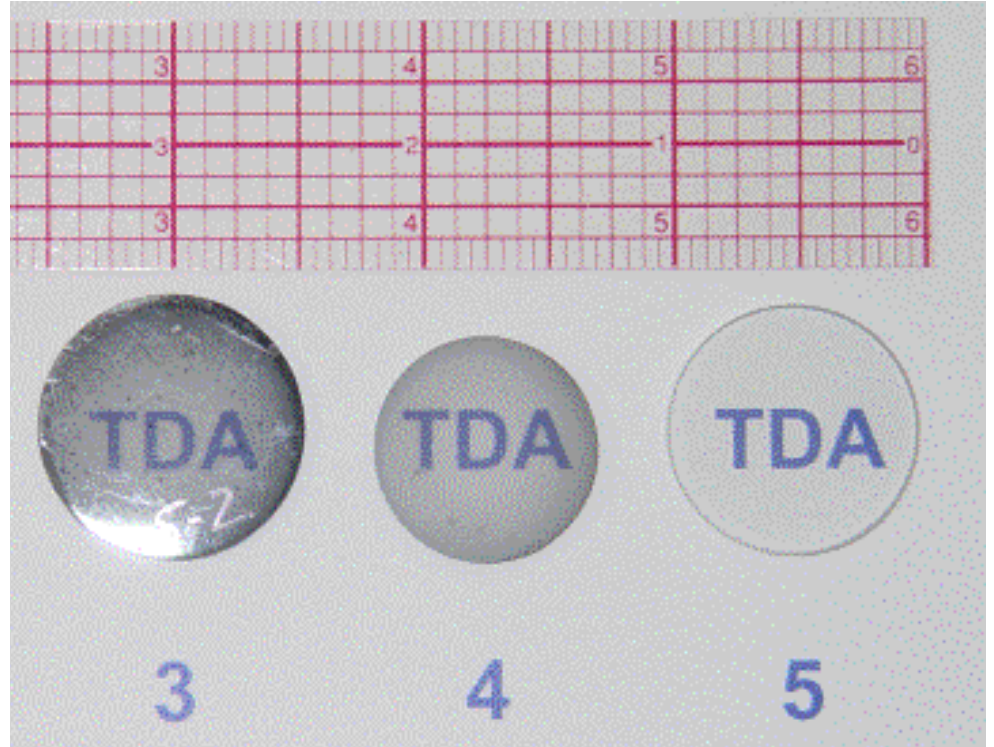
1000°C



1200°C

Single phase magnesium aluminate (spinel) produced from metal exchanged precursor

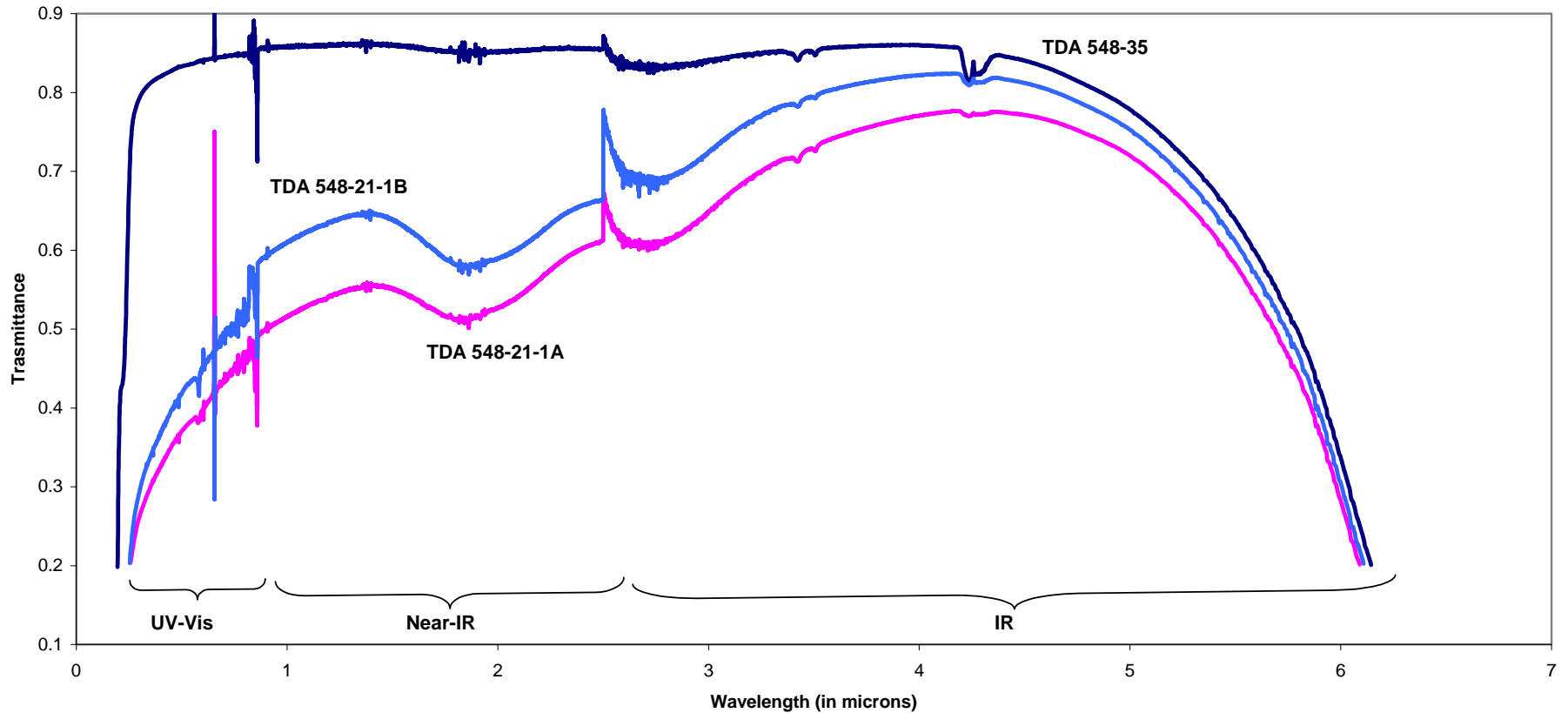
# Hot Pressed Spinel Discs



Spinel with  $\text{ZnAl}_2\text{O}_4$  impurities (3,4)

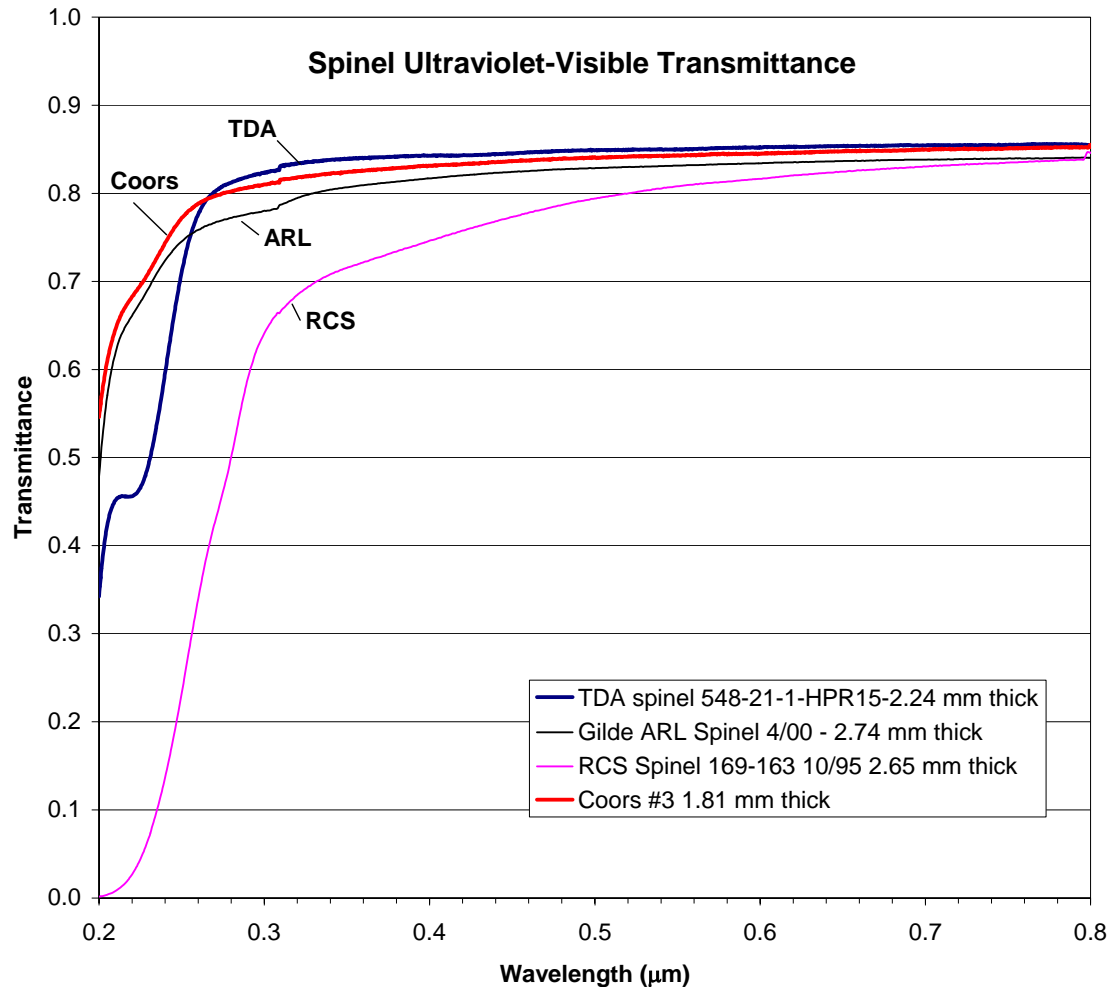
Pure spinel (5)

# Optical Properties: Initial Materials



# Optical Characterization: Best Powders

Hot pressed spinels from TDA powder meet or exceed legacy powders. Tests carried out by Dan Harris (NAWC, China Lake)



# Summary and Acknowledgements

- **Boehmite based spinel powder production offers control over purity, doping particle size**
- **Topotactic metal exchange of boehmite powders offers direct conversion to pure spinel**
- **High quality spinel windows can be produced from metal exchanged boehmite powders**
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