Slurry Based
Environmental Barrier Coating (EBC) Concepts

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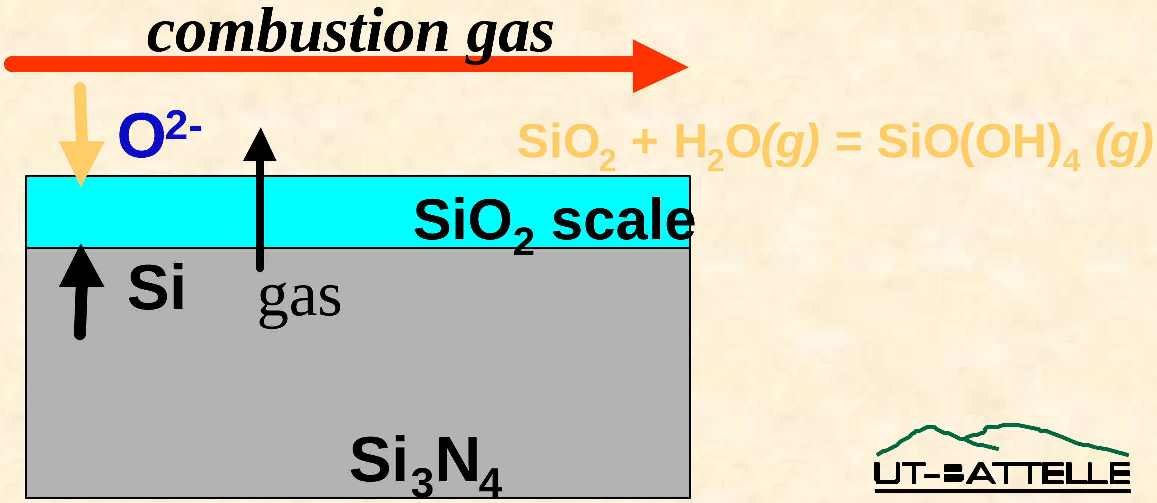
Outline

- **Background: Advanced ceramics for microturbines**
- **Problem: Rapid recession of silica-formers in water vapor**
- **Objective: Develop & fabricate low-cost protective coatings**
- **Slurry processing of protective coating materials**
  - **Approaches**
  - **Results**

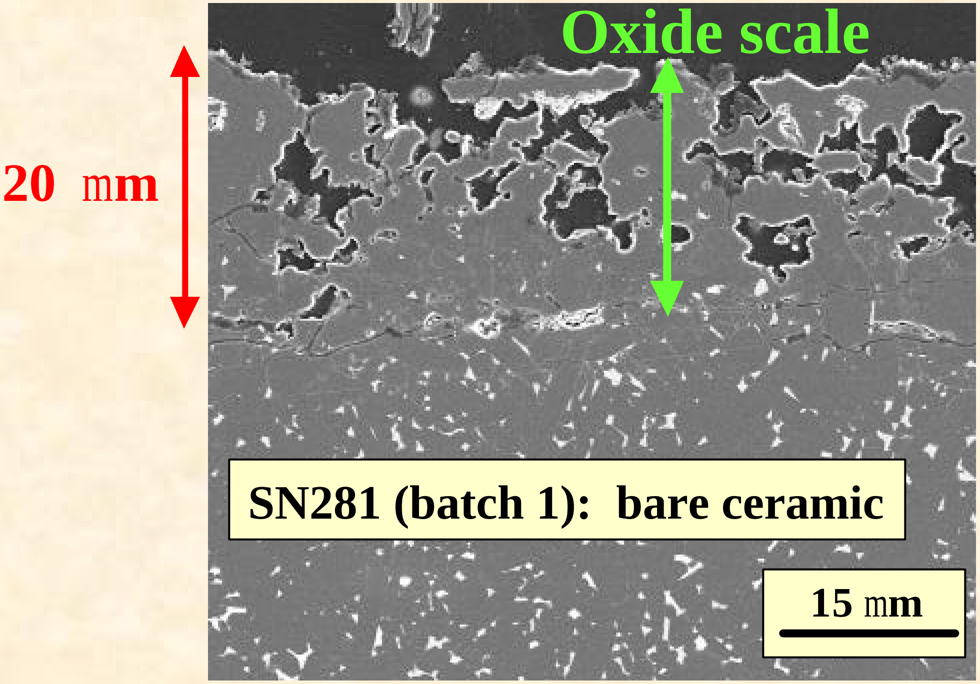
Problem: Degradation of Protective Silica Scales

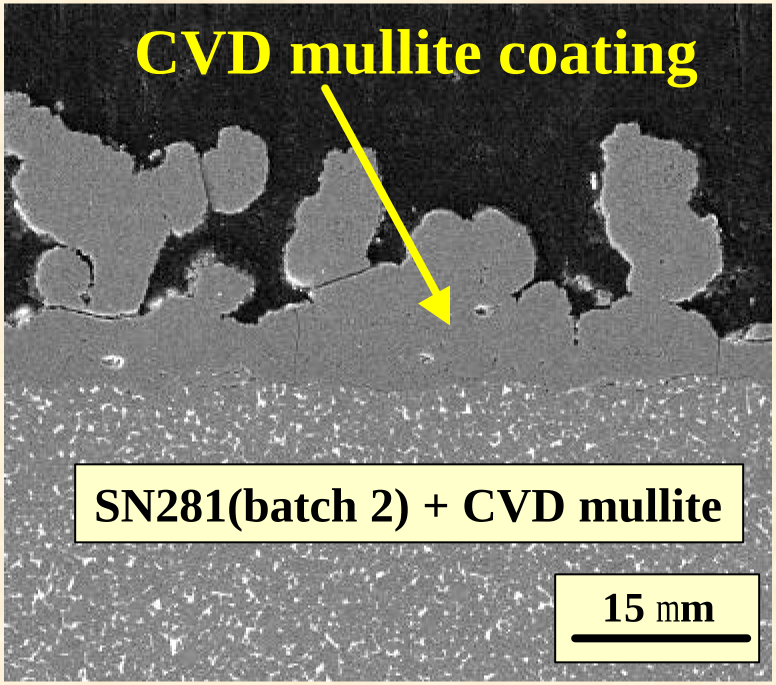
- Si_3N_4 is a candidate high temperature structural material for hot section components within high-efficiency microturbines.
- Protective SiO_2 scales are the basis for the corrosion resistance of Si_3N_4.
- Silica scales can be rapidly degraded in combustion environments.
  - Hot corrosion by molten alkali species (Strangman)
  - H_2O vapor & impurities accelerate SiO_2 growth (Deal, Opila, More et al.)
  - SiO_2 is volatile in H_2O (Venable, Opila)
  - Oxidation/volatilization increase w/ gas pressure & velocity (Robinson)



$SiO_2 + H_2O(g) = SiO(OH)_4\ (g)$

Coatings Are Needed to Enhance Performance





500-h, 1200°C, 10 atm, 15% H_2O

- **Protective coatings will be necessary in order to provide adequate ceramic component lifetimes.**
- **Coatings must be adherent, impermeable, stable and/or protective in high temperature water vapor and/or O_2, thermal expansion matched with Si_3N_4, relatively easy to manufacture, and cost effective.**

Candidate Materials Have Varied Properties

	Mullite	BSAS	$RE_2Si_2O_7$
Est. Silica Activity	1.0	<0.2	<0.2
CTE (ppm/C)	5.3	5.0 to 5.2	5.0 to 8.2*
Est. Temp Limit (°F)	>2400	>2400	>2400
Advantages	Good CTE/match to SiC, well characterized	Good CTE, low silica activity	Potentially good CTE, low silica activity
Issues	High silica activity	Limited experimental and performance history	Limited experimental and performance history

(Eaton, H.E., and Linsey, G.D, “Accelerated Oxidation of SiC CMC’s by Water Vapor and Protection via Environmental Barrier Coating Approach” EuroConference, Seville, Spain, Oct, 2001)

Slurry-Based Coating Processes

Techniques	Advantages	Issues
Vacuum Infiltration	Can coat internal or weave type structures	Viscosity of slurry/depth of slurry penetration
Spin Coating	Thin coating	Difficult to coat 3D structures
Screen Printing*	Controlled coating thicknesses and densities. Scaleable	Difficult to coat 3D structures
Pad Rolling	Controlled coating thicknesses and densities. Scaleable	Difficult to coat 3D structures
Spray Coating*	Inexpensive. Conducive to 3D structures	Thickness/flatness variation. Line of sight.
Dip Coating (glazing)*	Inexpensive. Conducive to 3D structures	Thickness variation as a function of dip direction
Dip Coating (solgel/polymerization)	Conducive to 3D structures	Thickness of coating with one coat. May require multiple passes. Shrinkage may be high.
Dip Coating (precursor/ conversion)	Conducive to 3D structures	Shrinkage may be high. May require multiple passes

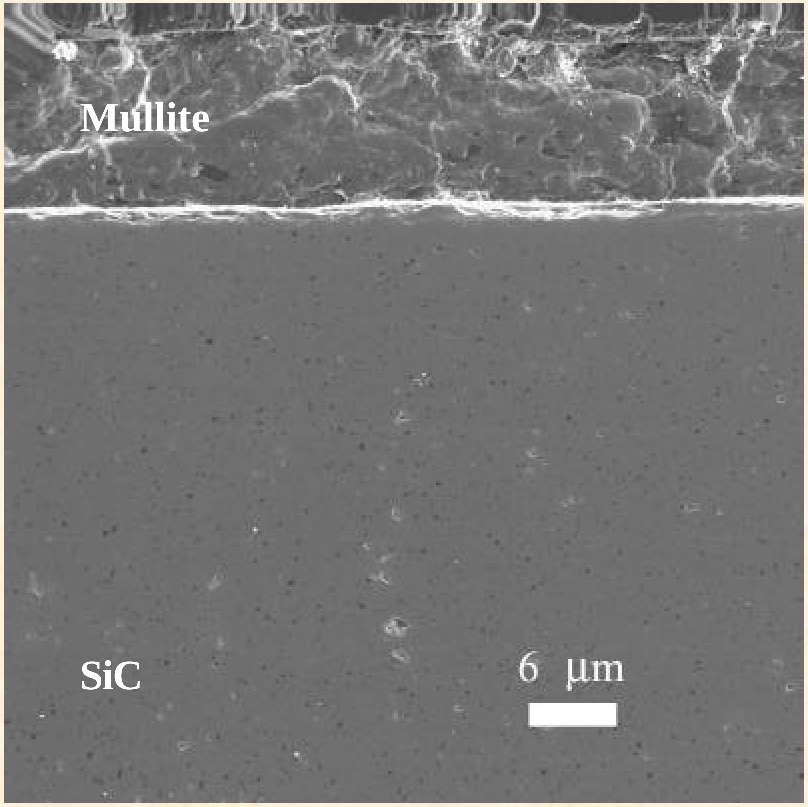
Slurry Processing Issues

- Rheology of the Slurry
  - Particle size (Needs to be submicron to assist densification, but decreasing size leads to increasing viscosity and lower solids loadings)
  - Viscosity (Optimum level needed. Too thin, not enough coverage. Too high, too high surface tension and too thick resulting coat)
  - Shear Thinning (Maintain uniform coating thickness and level across height and width)
  - Stability (Little or No settling or flocculation with time = Charge Balance)

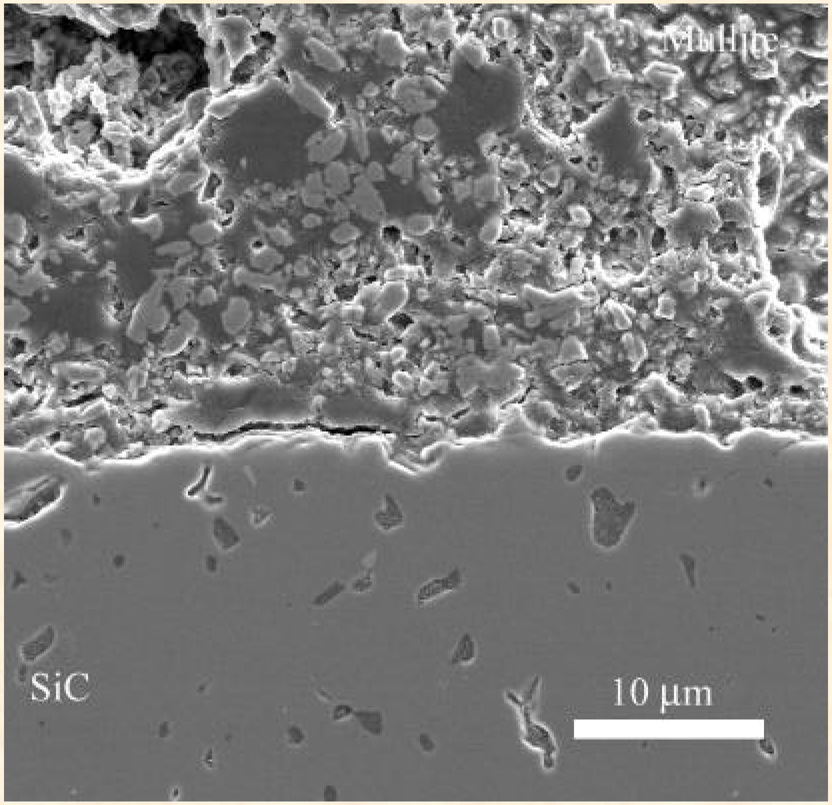
Material Versus Process

- What Affects the Success of the Coating?
  - Density
  - Microstructure
  - Macrostructure (Thickness, Morphology)
  - Chemical Interaction at Interface (Substrate Contributions)
  - Mechanical Interaction at Interface (Surface Roughness)
  - Chemistry of Coating Material
    - Material Properties: CTE, O_2 permeability, oxidation/corrosion…
  - Environment (Is more than one layer necessary?)

Density and Thickness of Coating Resulted from Process Changes



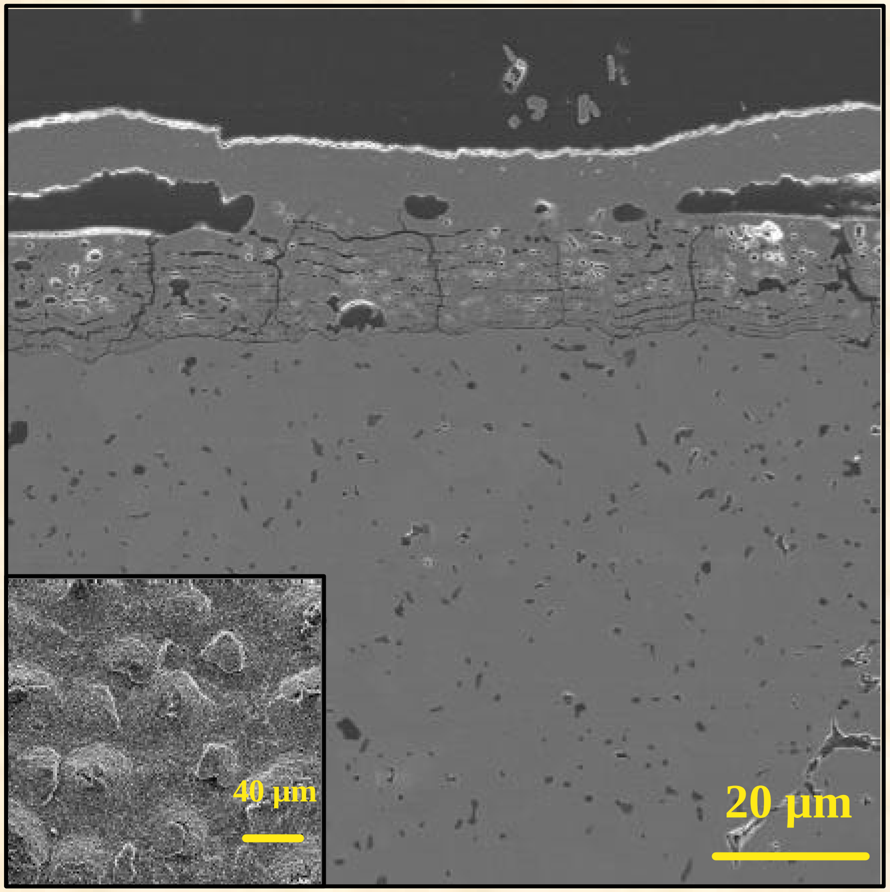
Mullite deposited by screen printing. Sintered at 1600°C in air for 2 hours.

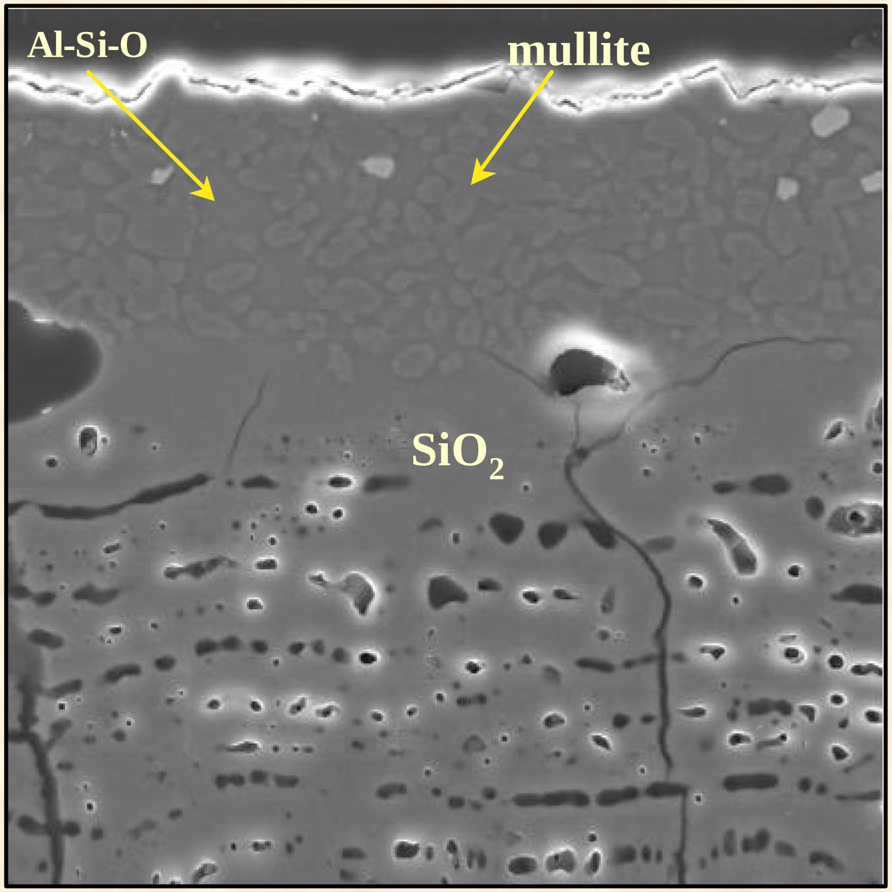


Mullite deposited by spraying. Sintered at 1600°C in air for 2 hours.

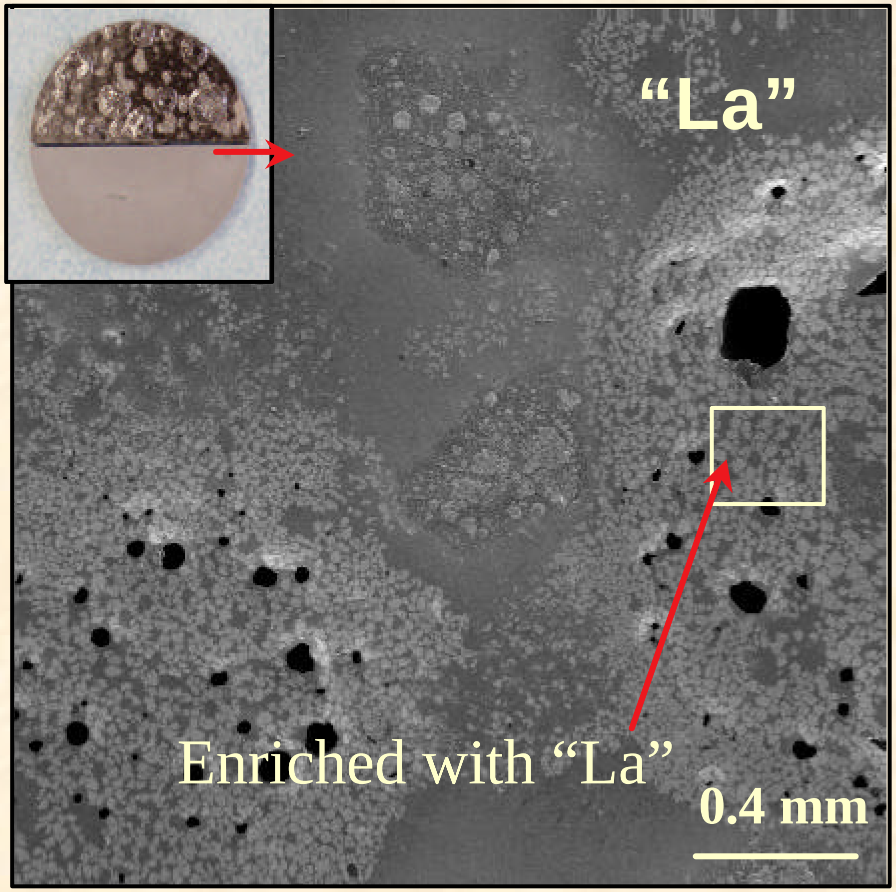
Screen Pattern Visible After Exposure

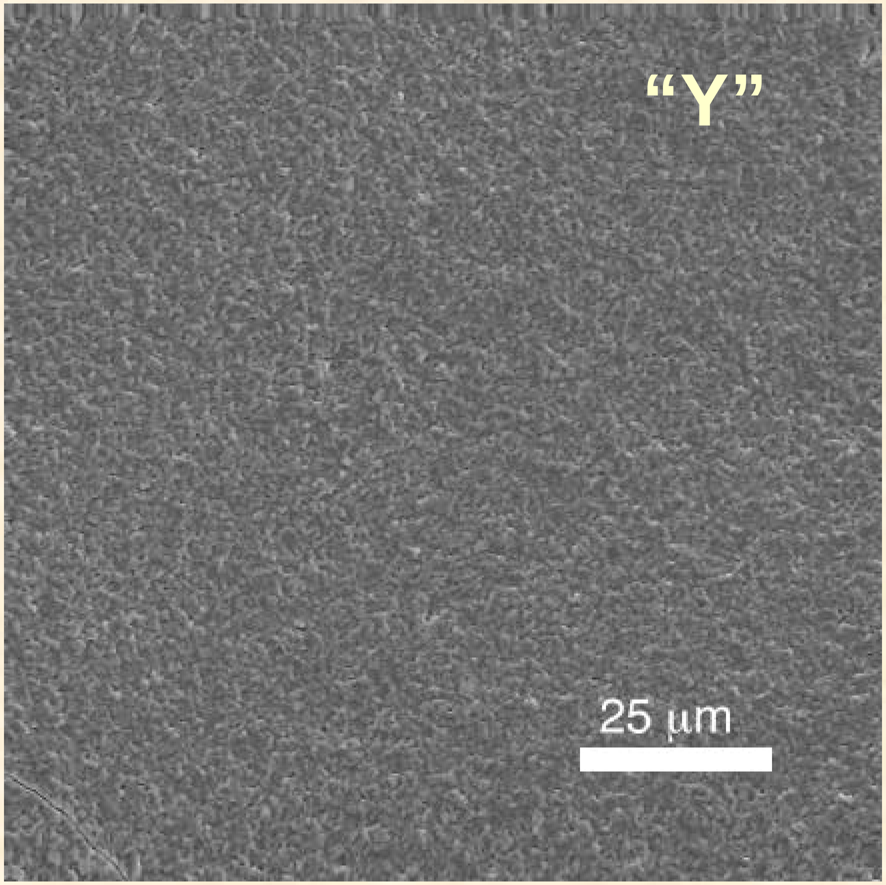
Polished Cross Section of Mullite Coating on SASiC After 500h Exposure at 1204°C, 100% H_2O





Effect of Substrate Interaction
BSAS Coatings on Substrate Additions

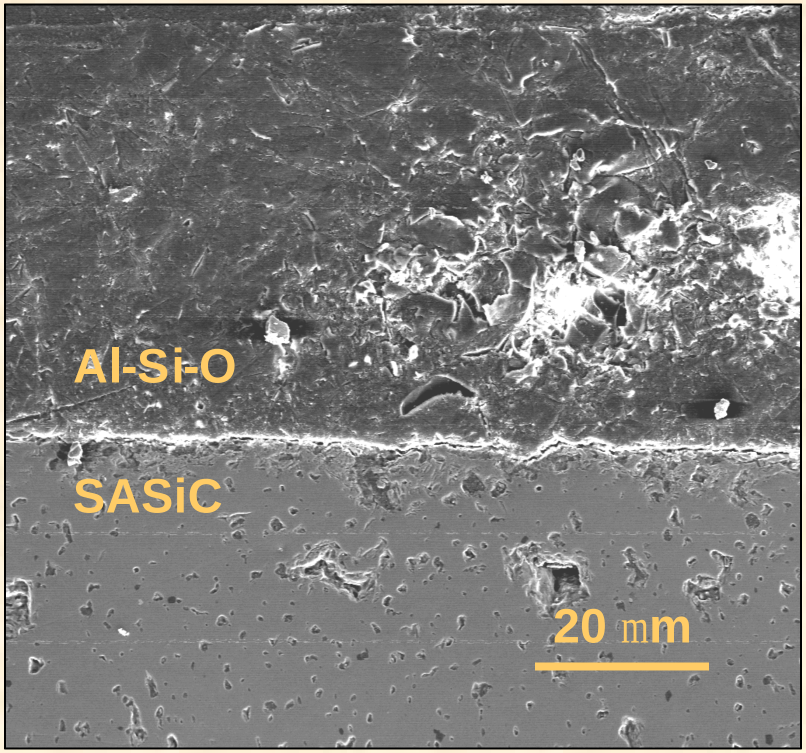




Sintered in argon (1400°C /2 hours) and in air (1000°C/2 hours)

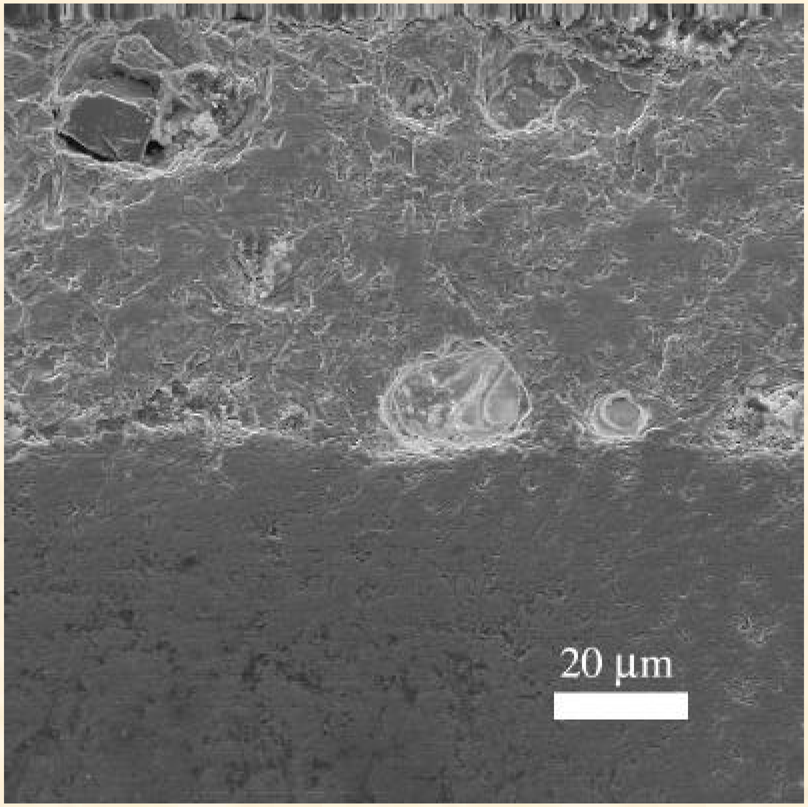
Dip Coated Mullite Layer Adherent on SASiC Substrate after Heat Treatment

- Sintered at 1600°C/2hrs in N_2
- 1.0 wt% binder
- 33 vol% mullite
- Subsequent heat treatment in ambient air at 1300C/0.5 hr resulted in delamination
- High temperature X-ray in process



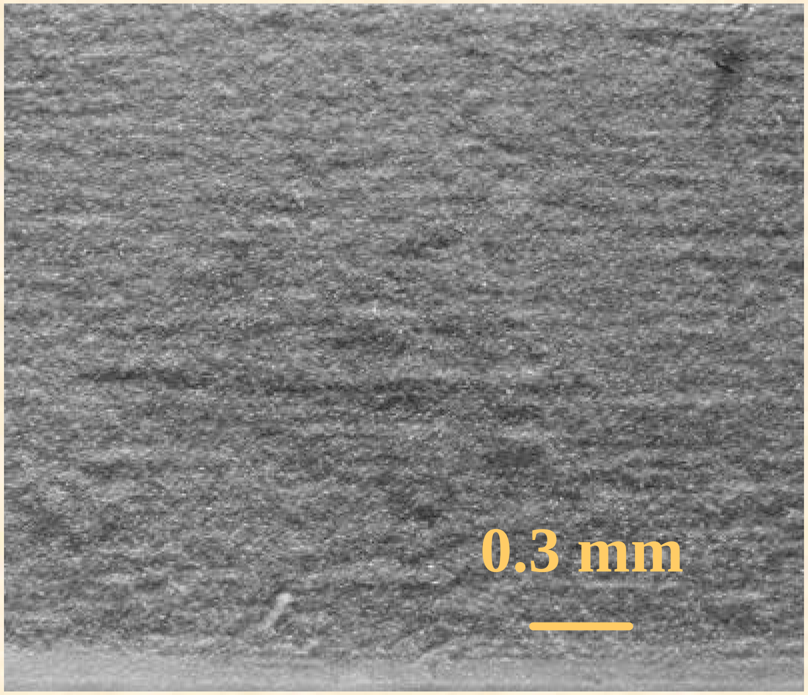
Dip Coated BSAS on NT154

- Sintered at 1400°C/2hrs in N_2
- 1.0 wt% binder
- 26 vol% BSAS
- Subsequent heat treatment in ambient air at 1300C/0.5 hr resulted in delamination
- High temperature X-ray in process



Yttrium Silicate Coatings by Reactive Processing

- 1:1 molar ratio
- Yttrium nitrate/colloidal silica
- Dipped or painted on to AS800 or NT164
- Sintered in N_2 at 1550°C for 6 hours
- Subsequent heat treatment in ambient air planned after high temperature x-ray run completed



Painted and sintered/NT164

Conclusions

- **Processing will affect success of coating material**
- **Preliminary results show dip coating and reactive processing is a viable route to protective coating systems**

Acknowledgements

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  - Paul Becher
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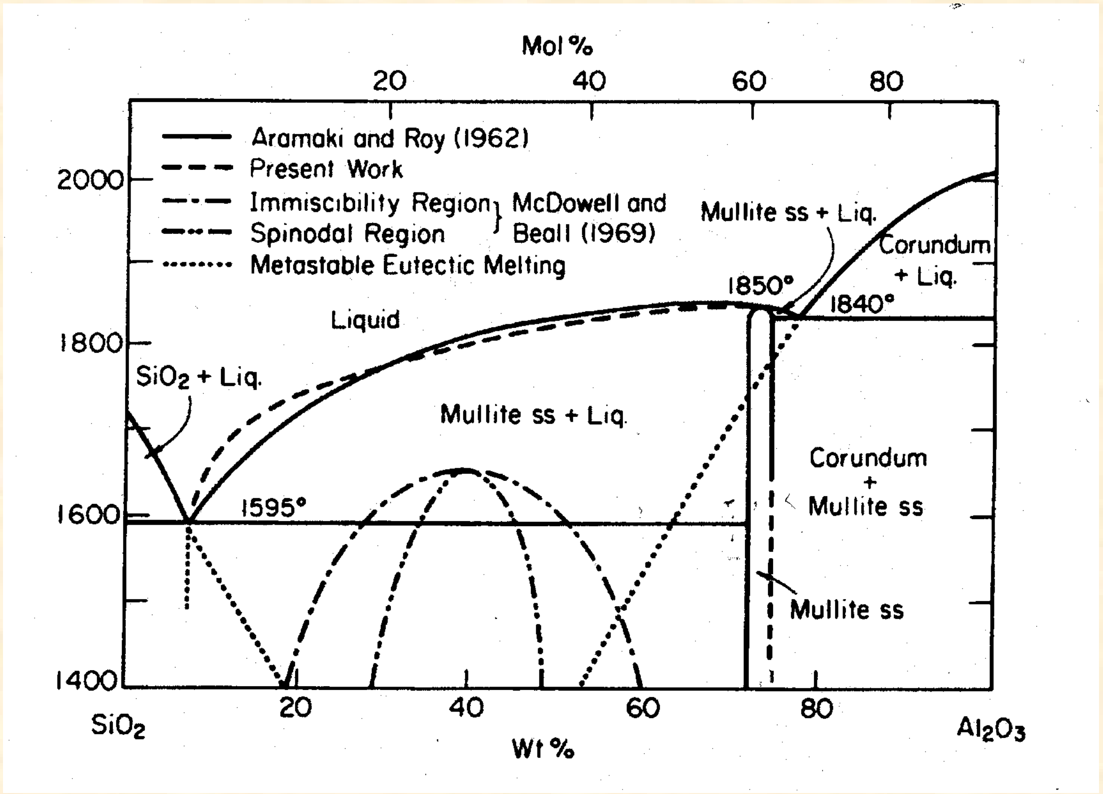
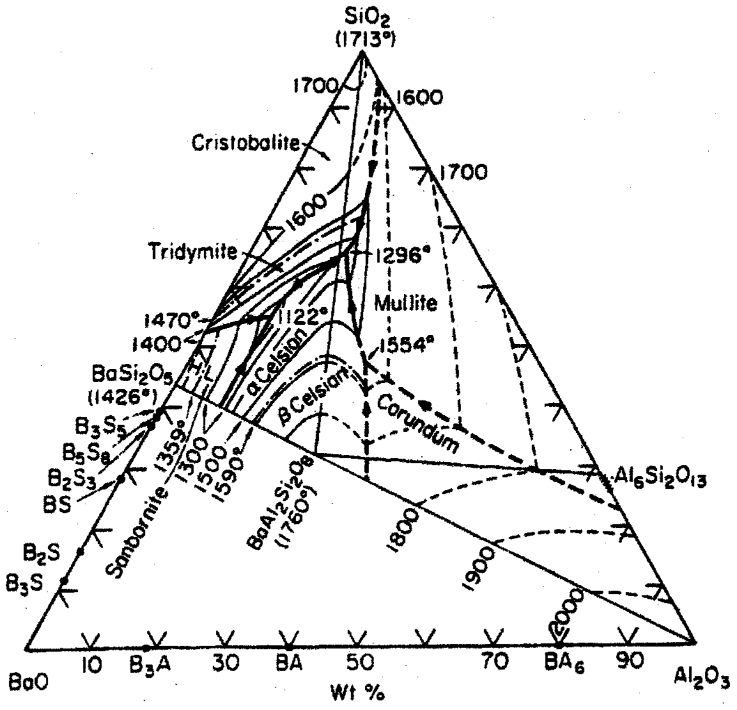
  - Ellen Sun

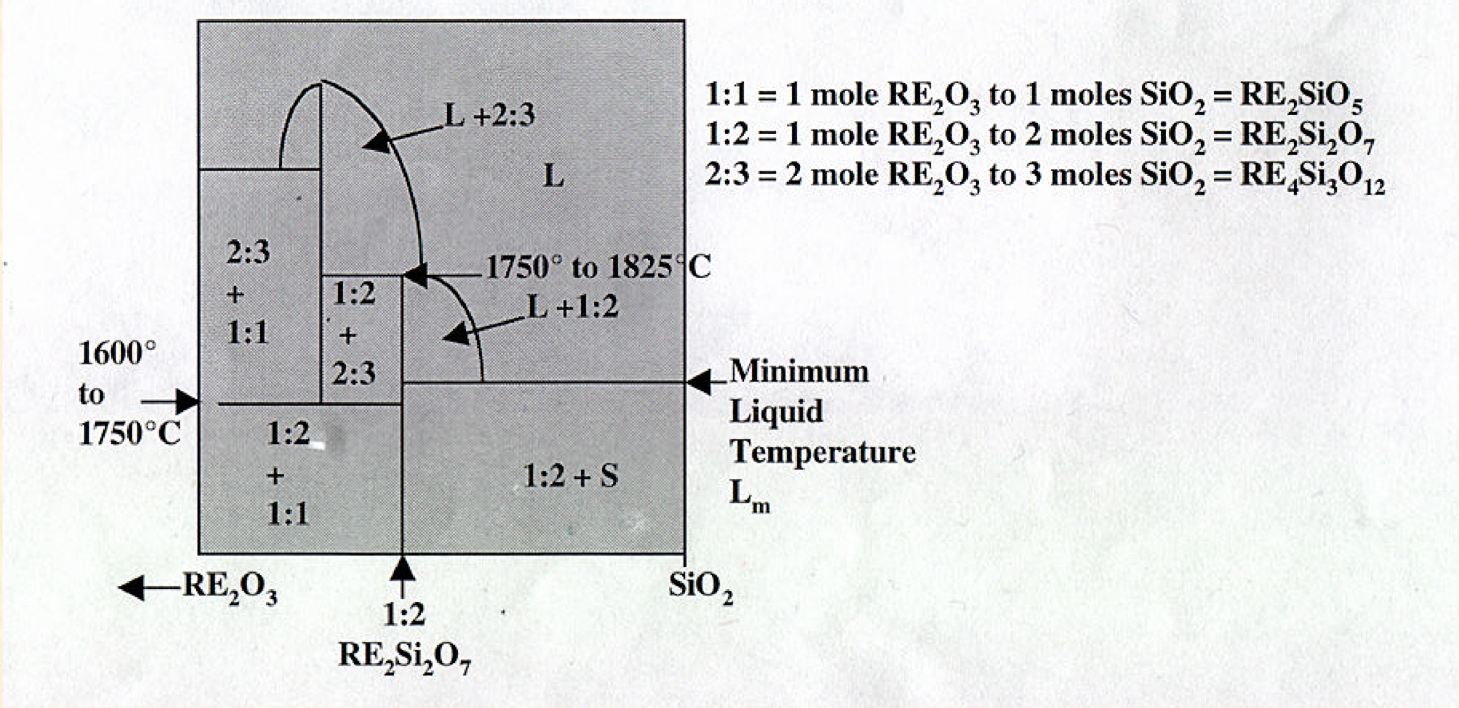
Phase Diagram of Mullite



Phase Diagram of Barium Strontium Alumino-Silicate (BSAS)



Phase Diagram of $RE_2Si_2O_7$



1:1 = 1 mole RE_2O_3 to 1 moles SiO_2 = RE_2SiO_5
1:2 = 1 mole RE_2O_3 to 2 moles SiO_2 = $RE_2Si_2O_7$
2:3 = 2 mole RE_2O_3 to 3 moles SiO_2 = $RE_4Si_3O_{12}$