Environmental Barrier Coatings for SiC Ceramic Matrix Composite Gas Turbine Blades

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Rolls-Royce has been developing silicon carbide (SiC) Ceramic Matrix Composites (CMCs) to be used in place of single crystal super alloys for gas turbine blades due to their lower weights and higher operating temperature limits. However, SiC degrades in a high-temperature combustion environment, creating a need for an environmental barrier coating (EBC) to protect the SiC. It has been hypothesized that an EBC containing mullite and barium strontium aluminosilicate (BSAS) can prevent the glassy phase from forming, while also displaying limited cracking when thermally cycled. During experimentation, this hypothesis was not confirmed, as adding BSAS to mullite caused the coating to bubble during thermal cycling.

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Project Background

- CMCs have the potential to increase operating temperatures inside gas turbine aircraft engines and thereby increase fuel efficiency.
- SiC/SiC CMCs are desirable due to their low weight, low coefficient of thermal expansion (CTE), and high melting temperature.
- SiC is problematic in high temperature environments due to interaction with water vapor, which causes oxidation and the formation of a low-T_m glass phase[1].
- An EBC is needed to protect SiC from the water vapor present in a combustion environment.
- BSAS has a CTE that is similar to SiC, but is easily penetrated by water.
- Mullite prevents water penetration, but cracks when thermally cycled, due to a large difference between its CTE and the CTE of SiC.
- A relatively crack free, impenetrable EBC with a porosity level of 20%, and a thickness of 25-75 µm, is required to meet Rolls-Royce's need.
- A coating containing both mullite and BSAS has been hypothesized to resist both cracking and water penetration.

[1] Lee, K. (2014). Environmental Barrier Coatings for SiC_f/SiC. *Materials, Modeling and Technology Ceramic Matrix Composites,* 430-451. Retrieved November 23, 2015, from Wiley Online Library.

Experimental Procedure

Slurry Production

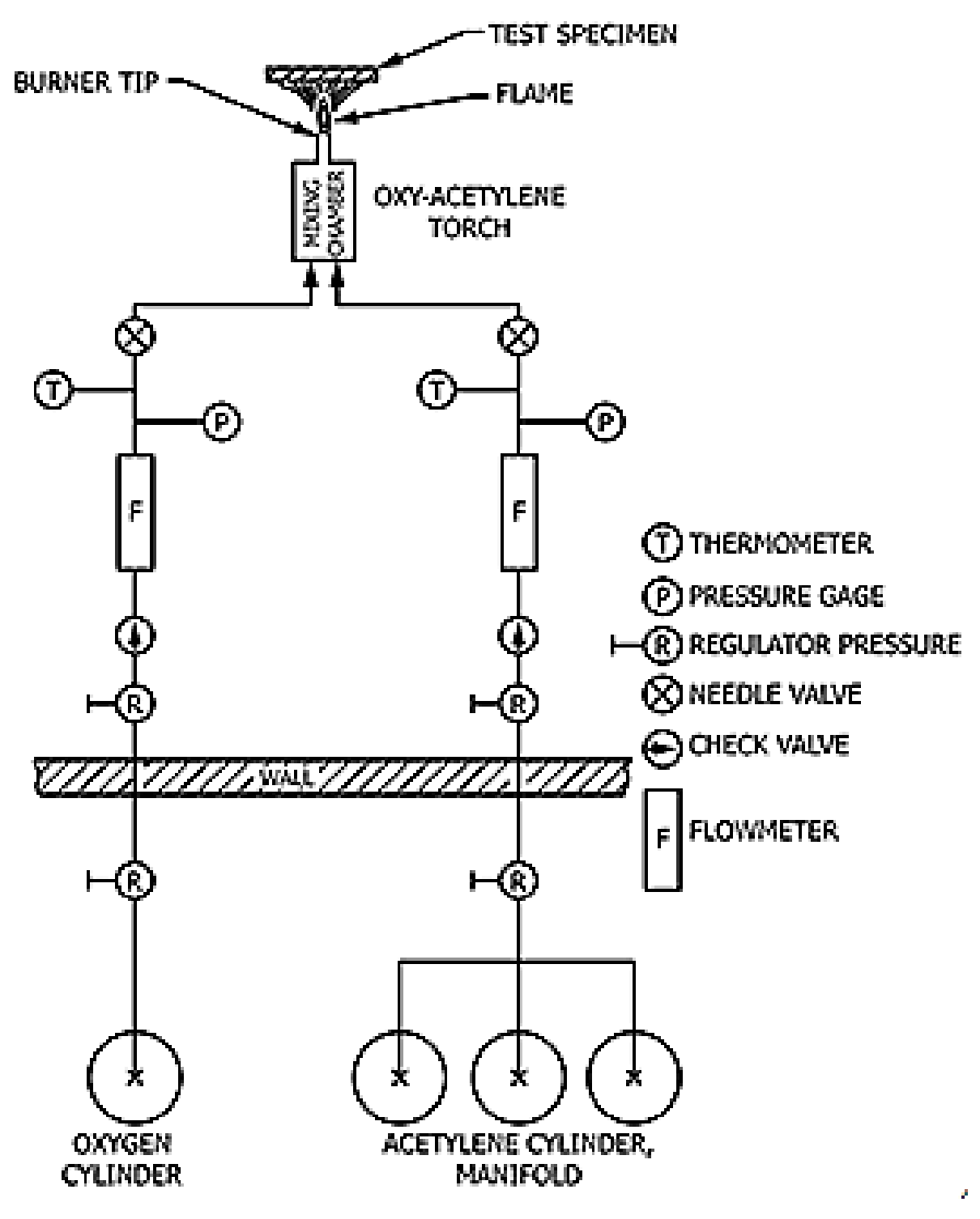
1) Attrition milling was used to reduce particle size of mullite and BSAS powders.
2) Compositions of varying weight percentages of mullite, BSAS, polyvinyl butyral (PVB), and sintering aid ($LiCO_3$ or MgO) were added to a Darvan-C, ethanol mixture.
3) The slurry was mixed on a ball mill for 24 hours to uniformly mix ingredients for dip coating.

Dip Coating

1) In preparation for dipping, air was removed from the slurries using an ultrasonic bath.
2) 1.27cm x 2.54cm (0.5” x 1”) SiC coupons were dipped in the slurry and spun at 600 RPM to remove excess slurry.
3) Coatings were air-dried for 24 hrs before being sintered at 1300°C for 5 hrs.

Thermal Cycle Testing

Samples were thermally cycled by ablation testing[2]. Samples were ablated 3 times for 1 minute at 1400°C, with 1 minute of rest between cycles.



[2] ASTM Standard E285-08, 2015, “Standard Testing Method of Oxyacetylene Ablation Testing of Thermal Insulation Materials,” ASTM International, West Conshohocken, PA, 2015.

Results

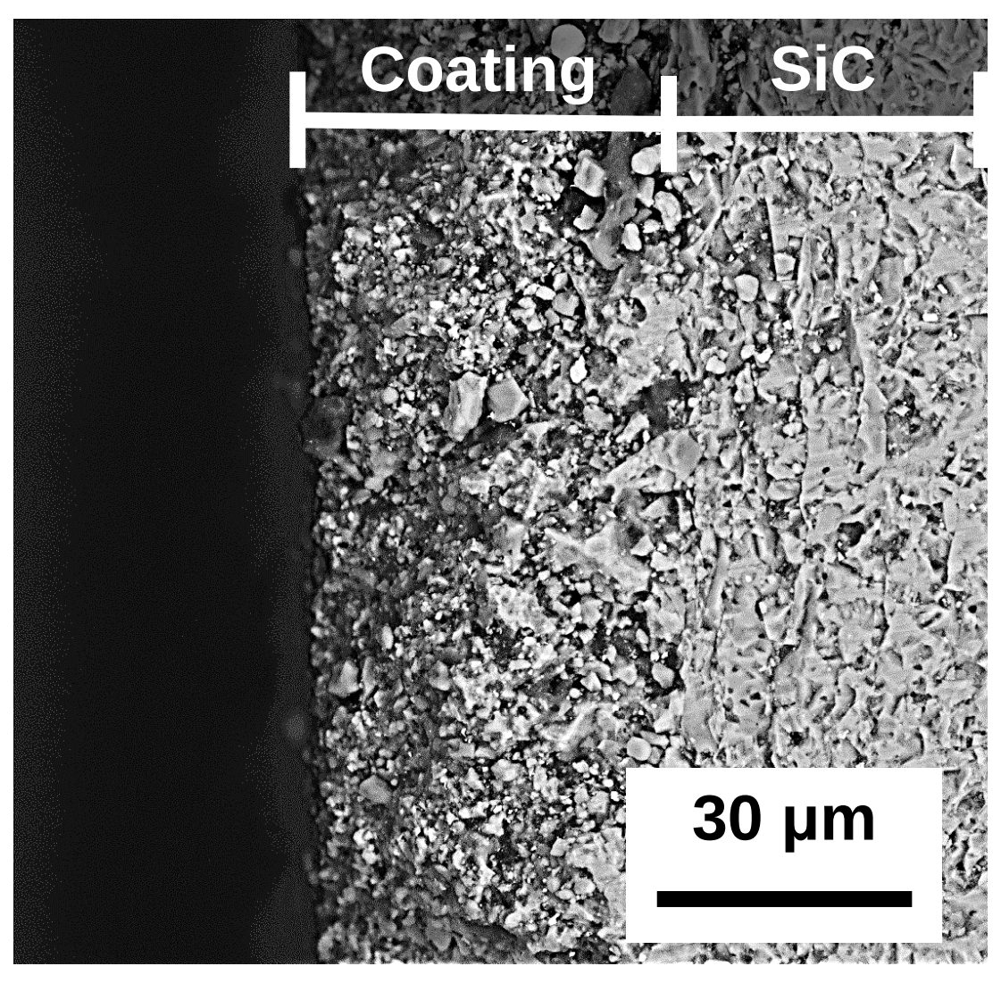
Coating Process

Effective slurries contain

- 1 wt. % Darvan-C
- 4 wt. % PVB
- 1:1 wt. % Ethanol:Powder

Observations

- Coatings adhered to SiC substrate after sintering.
- When $LiCO_3$ was used as a sintering aid, coating uniformity was inadequate for continued testing.



SEM cross-sectional micrograph of mullite/BSAS coating

Constant Parameter	w/ 2.5 wt. % MgO					4:1 wt. % Mullite:BSAS			
Wt. % Mullite:BSAS	9:1	4:1	7:3		Sintering Aid	0 wt. %	1.25 wt. %	2.5 wt. %	5 wt. %
Sintered (Y/N)	Y	Y	Y		Sintered (Y/N)	N	N	Y	N

Surface Characterization

- Mud cracking appears in both single and double dipped coatings.
- Double-dipping reduced number of through-thickness microcracks.
- Double-dipping increased number of large through-thickness cracks.

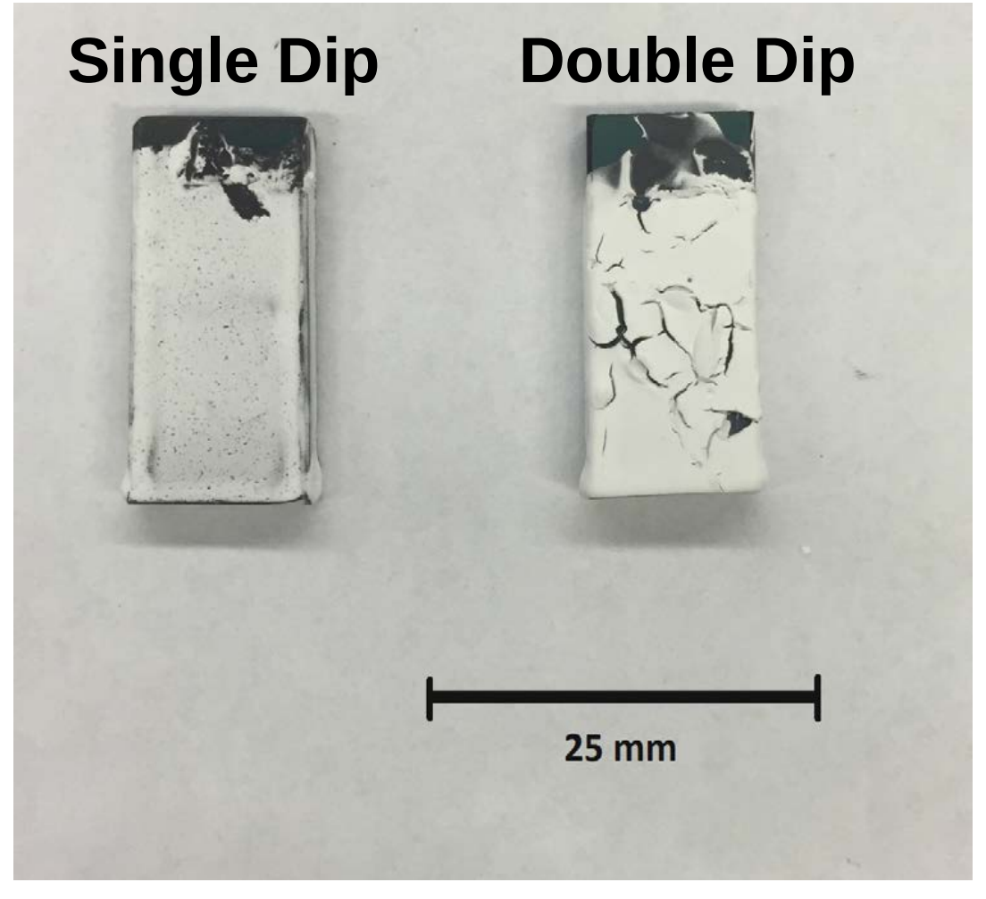
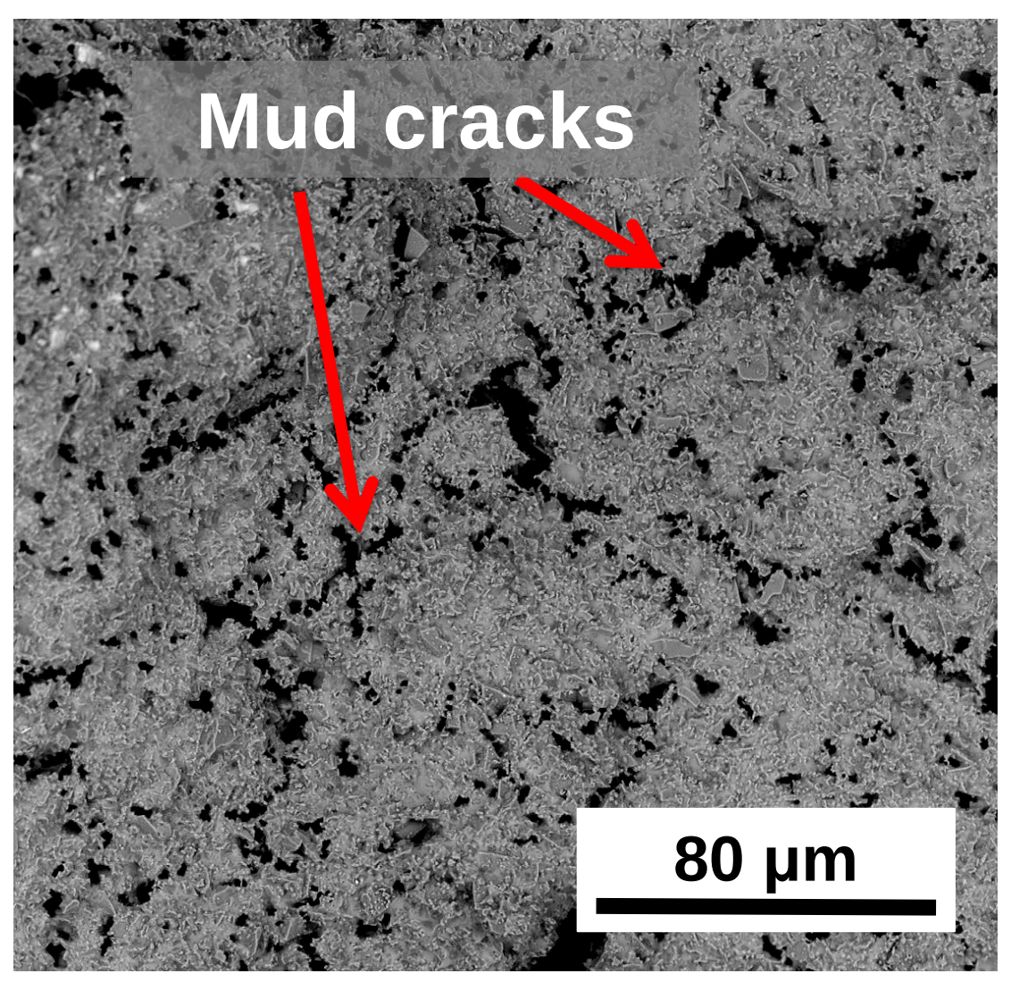
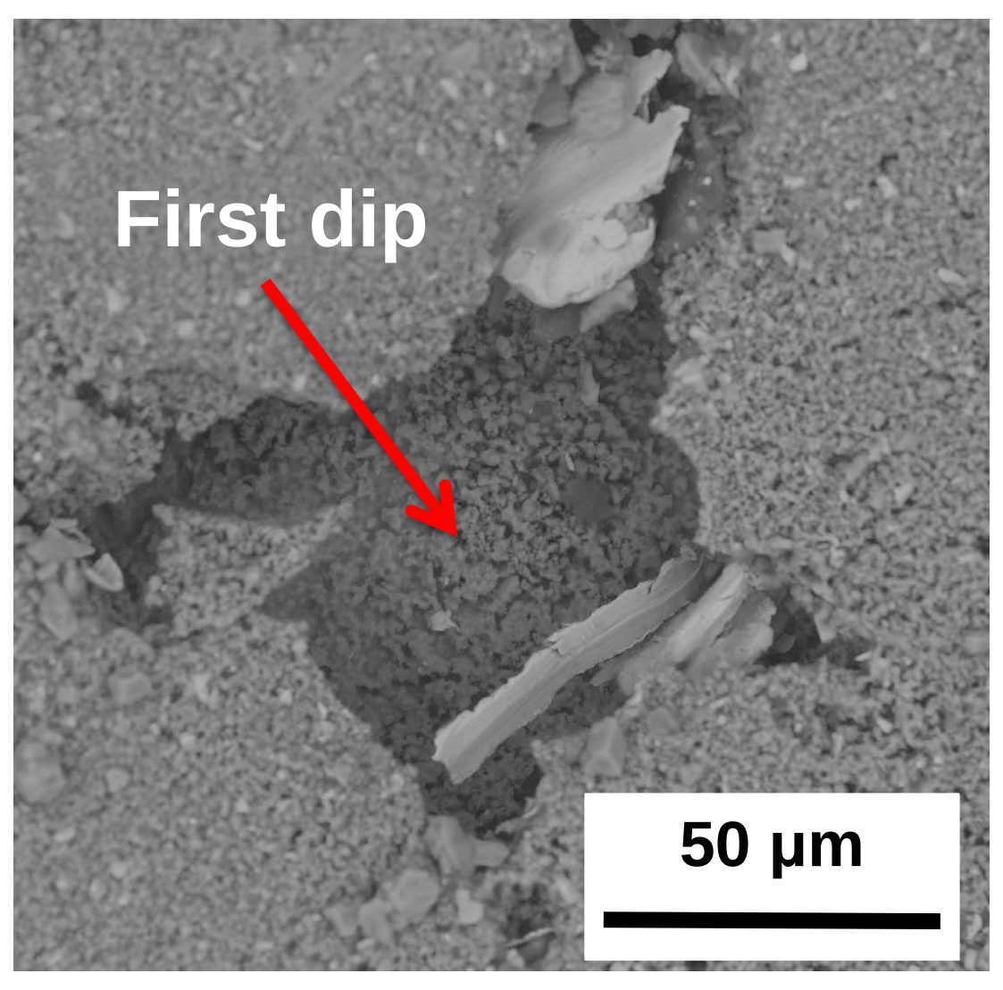


Image of single dipped (left) and double dipped (right) coating

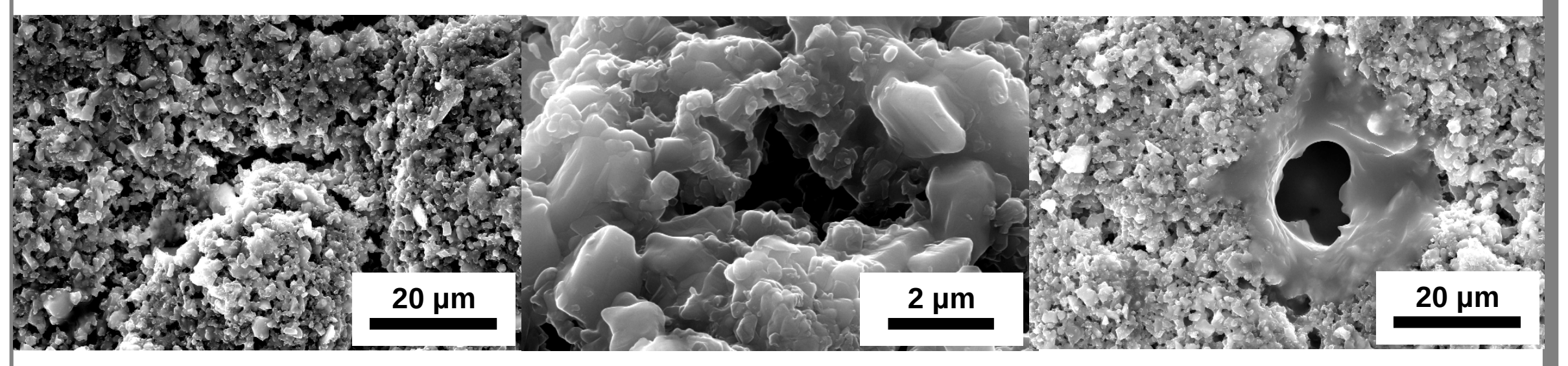


SEM micrograph of single dipped coating with arrows pointing to through-thickness mud cracks



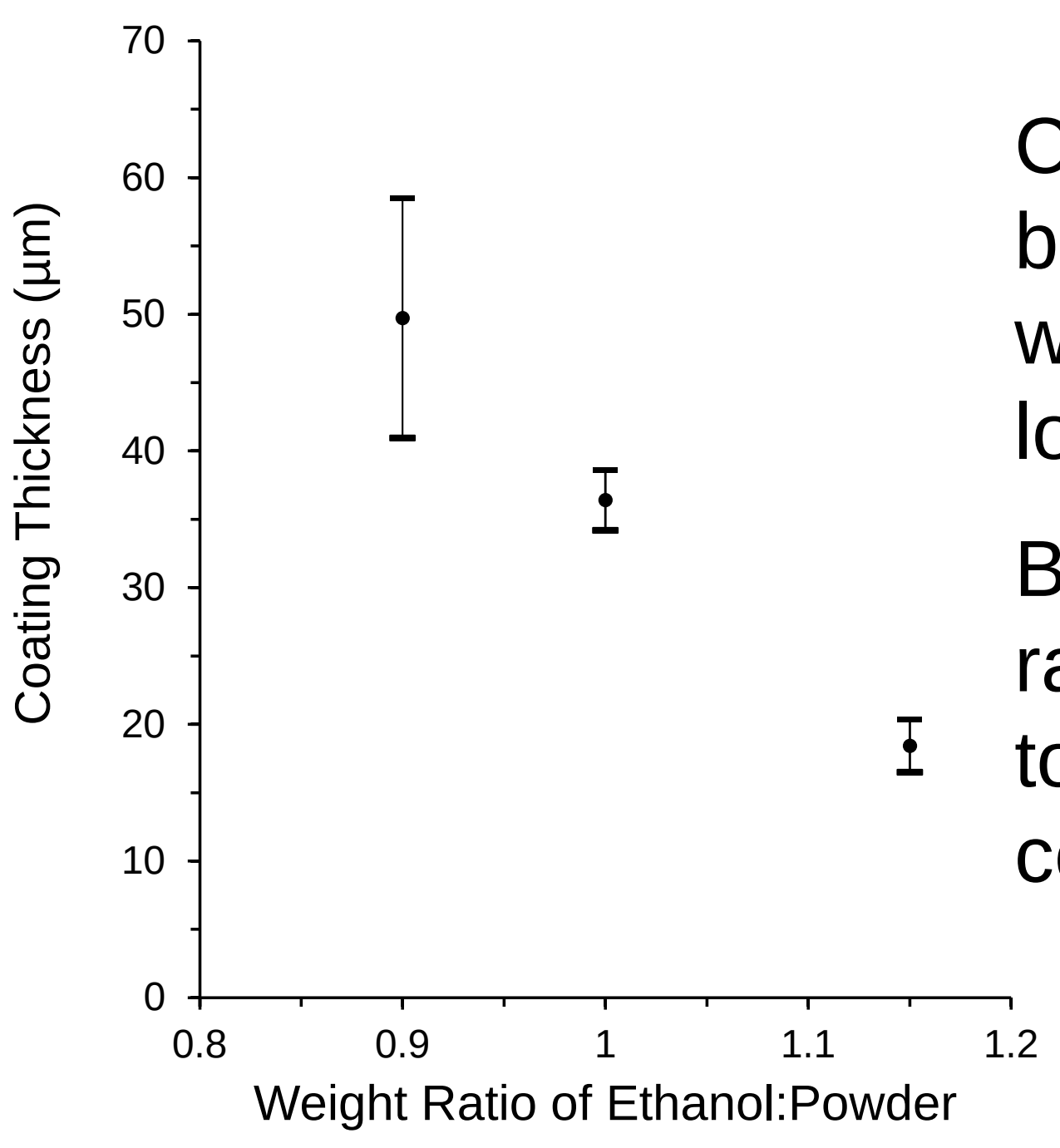
SEM micrograph of double dipped coating with arrow showing stemming of through-thickness cracks

Pinholes nucleated in regions that experienced increased densification during sintering.



(a) (b) (c)

SEM micrographs of coating containing 7:3 wt.% mullite:BSAS, with 2.5 wt% MgO. Sintering shown between particles in images (a) & (b). Increased densification shown around hole in image (c).

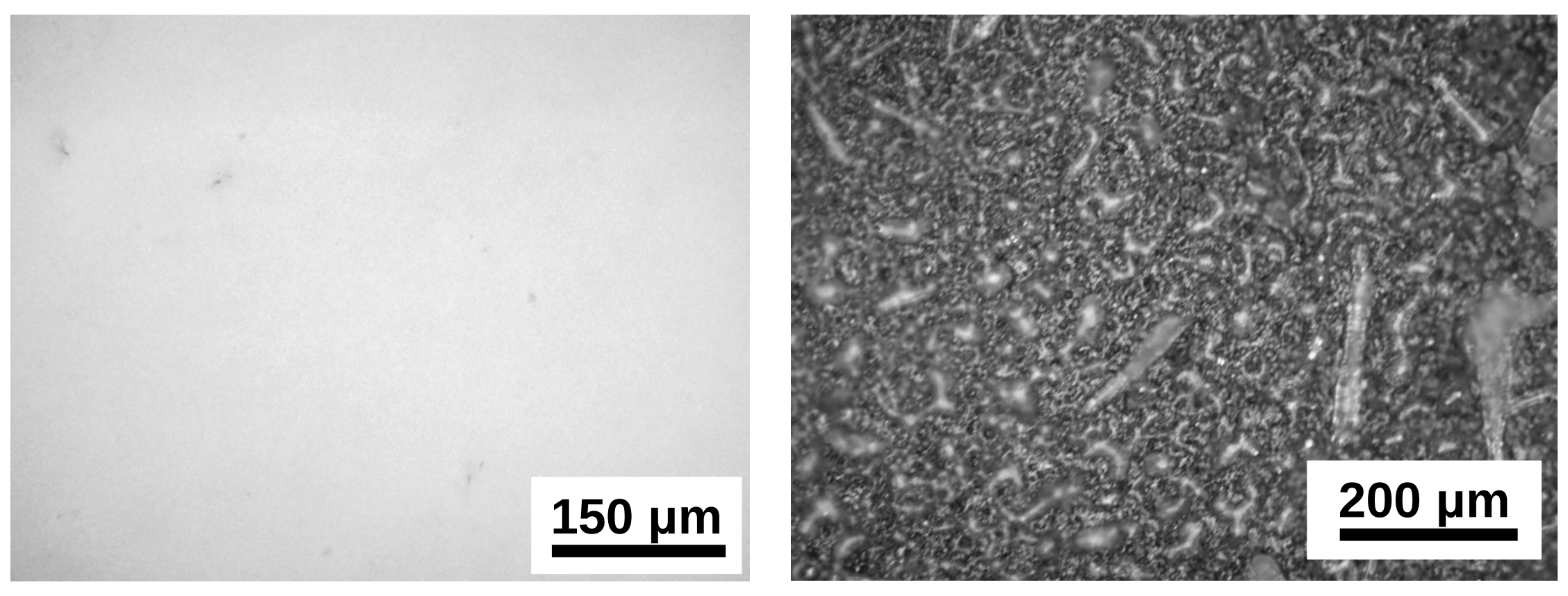


Coating thickness became more variable with increased powder loading.

Below 1.15 EtOH:Powder ratio, coating becomes too thin to meet project constraints.

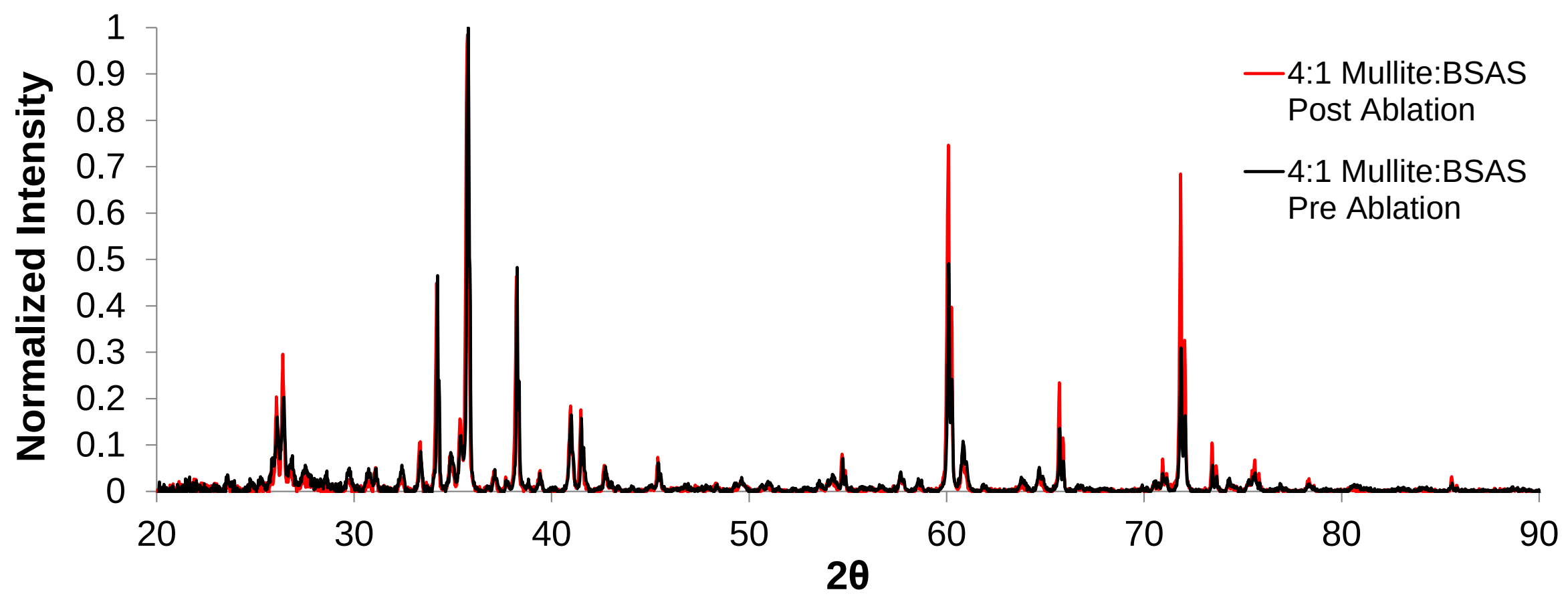
Results Continued

Thermal Cycle Testing



Images of a 4:1 BSAS to mullite coating before thermal cycling (left) and after thermal cycling (right)

Most of the mullite/BSAS coating ablated off of the SiC substrate.



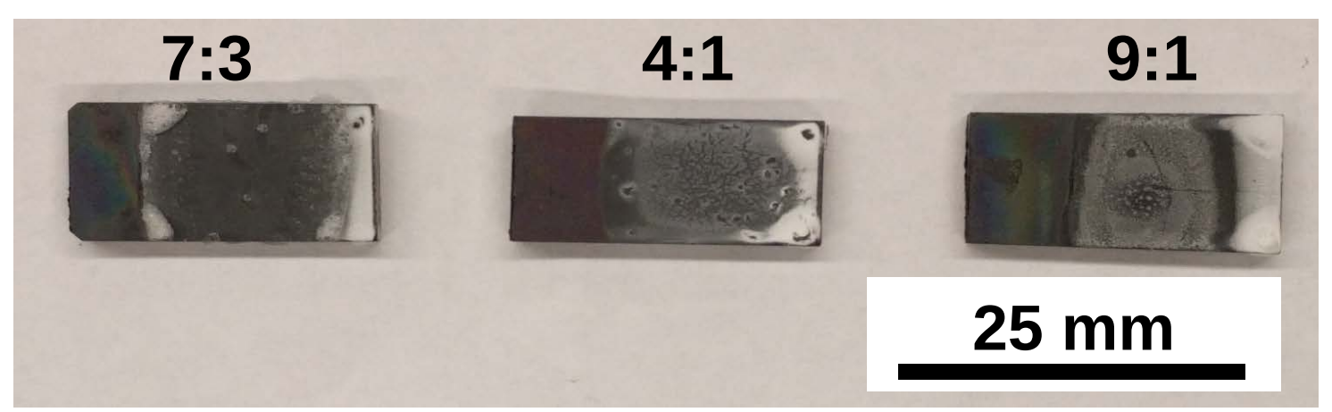
Discussion

Investigation of binder

- A polymerization reaction between PVB and water causes a skin to form on the surface of the slurry and reduces coating uniformity during dipping.
- Binders that do not react in air would improve the repeatability of the dipping process.

Thermal cycle testing

- Sample with lowest mullite:BSAS ratio displayed bubbling in the coating, which suggests water penetration.



Possible causes of coating failure

- Based on XRD analysis, there is no evidence to suggest that a new phase formed in the coating when samples were thermally cycled.
- Evidence therefore suggests that pressure from the ablation nozzle, the possible penetration of water, or differences in CTE between the coating and the substrate caused spallation to occur.

Possible causes of regions with increased densification

Formation of a silicate glass due to reaction occurring with mullite or BSAS during sintering.

Conclusion

- Single dipped coatings are more desirable than double dipped coatings due to uniformity of surface finishing.
- A coating that mixed mullite and BSAS was insufficient to prevent the delamination that BSAS causes.
- Greater ethanol:powder ratios are ideal for thinner coatings.

Future Works

- Use dilatometer to quantitatively assess the changes in coating sintering temperature based on levels of sintering aid.
- Investigate solvents that evaporate more slowly, in order to reduce mud cracking before sintering.

MSE 430-440: Materials Processing and Design