

# Thermochemical/Thermomechanical Synergies in High Temperature Particle Erosion of CMAS Exposed EBCs

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John A. Setlock<sup>2</sup>, Bryan J. Harder<sup>1</sup>

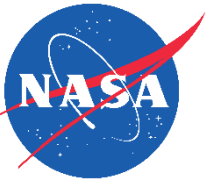
*<sup>1</sup>NASA Glenn Research Center*

*<sup>2</sup>University of Toledo*

## Acknowledgments

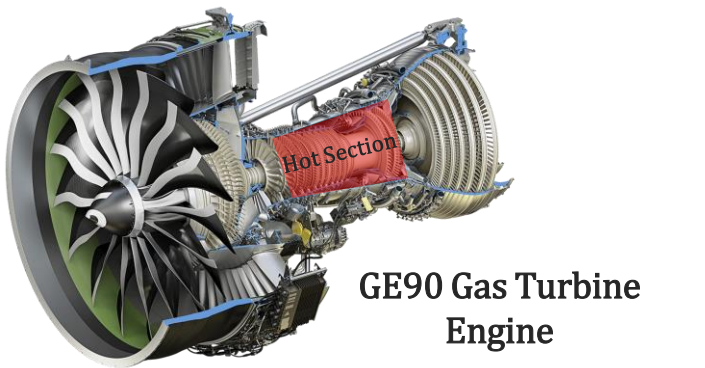
Transformational Tools and Technologies (TTT) Project  
Hybrid Thermally Efficient Core (HyTEC) Project



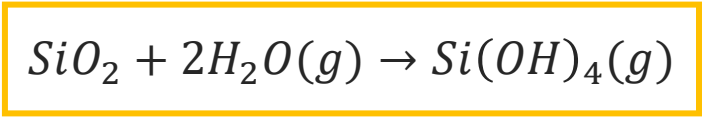
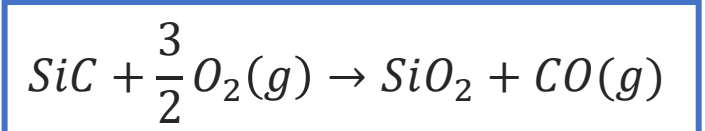


# Introduction and Motivation – Ceramic Matrix Composites (CMCs)

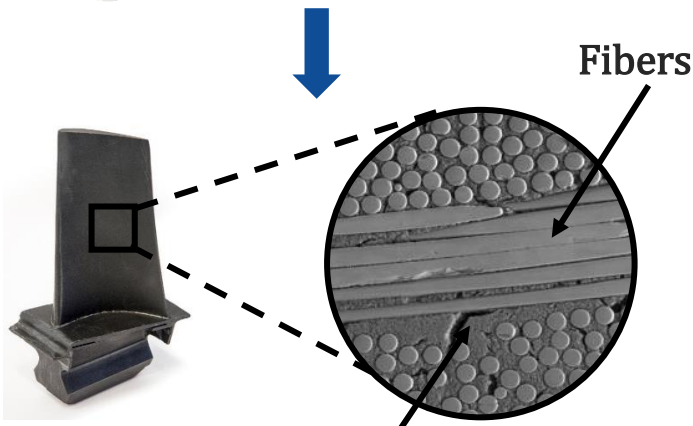
- CMC turbine engine components offer high temperature stability, but recess in high temperature water vapor environments



GE90 Gas Turbine Engine



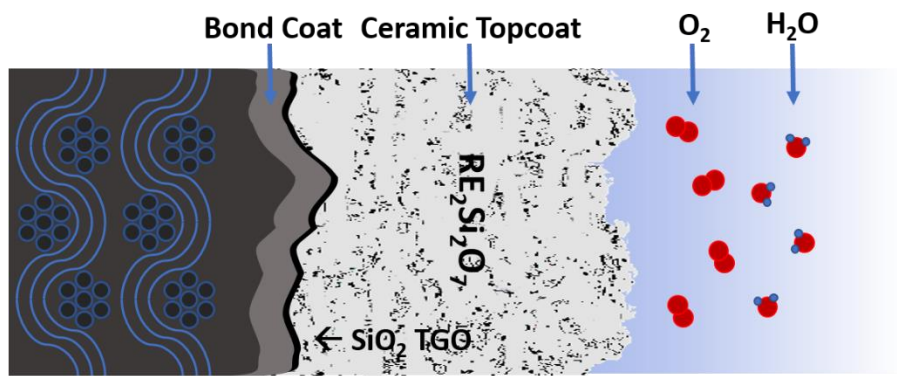
## Environmental Barrier Coatings (EBCs)



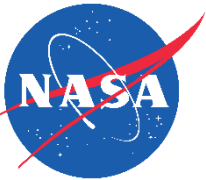
CMC Turbine Blade

Matrix

Fibers

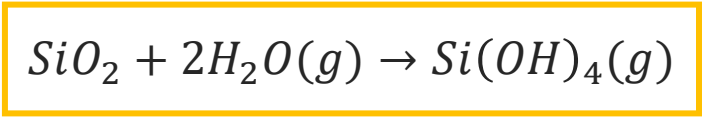
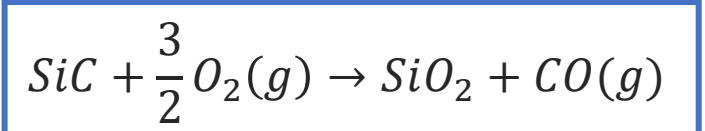


Opila et al. *J. Am. Cer.* **82** (1999)

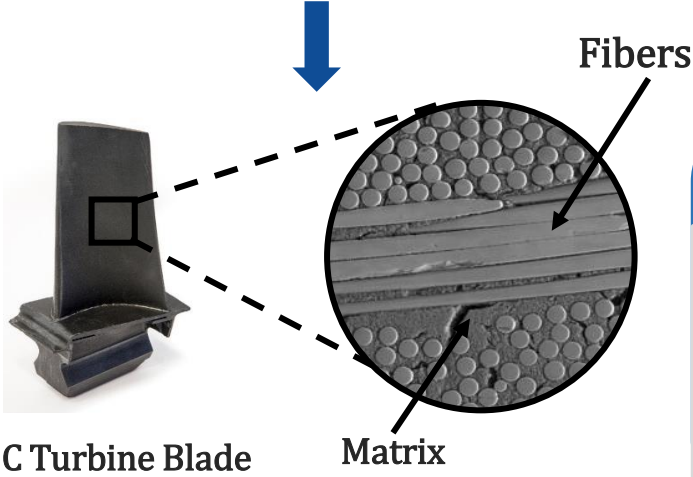


# Introduction and Motivation – Ceramic Matrix Composites (CMCs)

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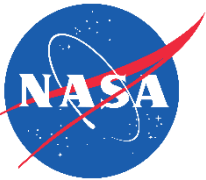
## Environmental Barrier Coatings (EBCs)



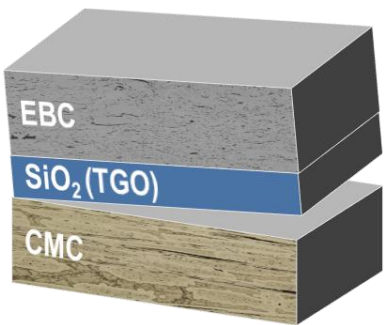
### Intrinsic Material Selection Criteria

- Coefficient of thermal expansion (CTE)
- Sintering resistance
- Low H<sub>2</sub>O and O<sub>2</sub> diffusivity/solubility
- Phase Stability
- Low Modulus
- Limited coating interaction

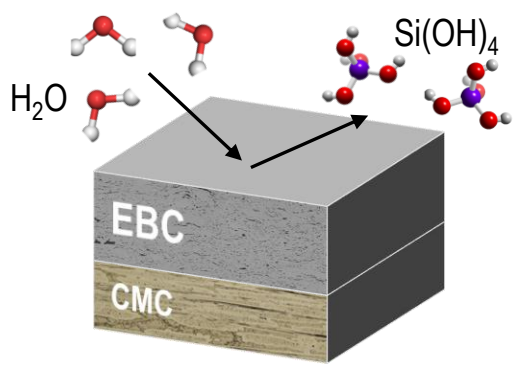




# Introduction and Motivation – EBC Development and Testing



Steam Oxidation

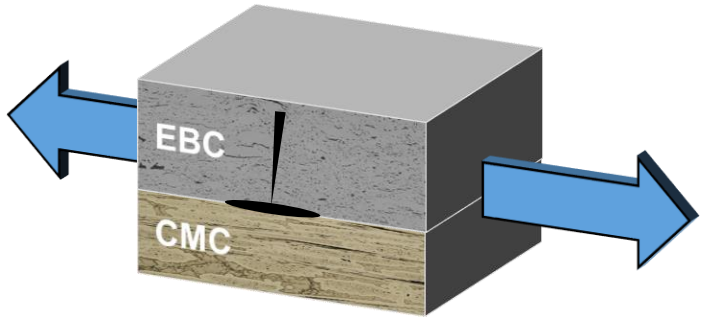


Hydroxide Formation/Recession

**Testing of EBC systems is critical**

**Individual mechanisms must be well understood before evaluating combinatorial effects**

**Synergies between extrinsic failure modes determine EBC lifetime and design requirements**



Thermomechanical Durability

CMAS

EBC

CMC

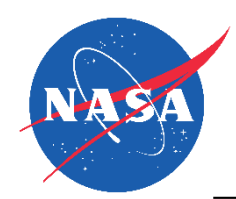
**Molten Silicate Attack and Infiltration**

EBC

CMC

**Erosion and FOD**

Lee, "Environmental Barrier Coatings for CMCs"; in Ceramic Matrix Composites, (2015)



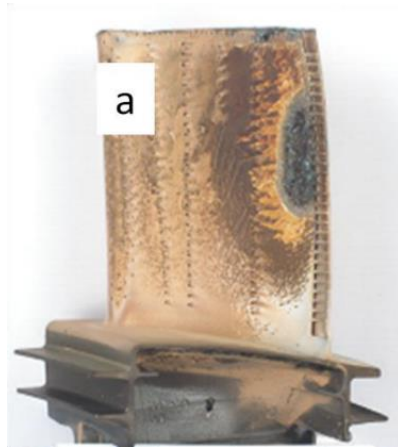
# Introduction and Motivation – EBC Degradation by Particulate Ingestion

## CMAS

- Particulates (i.e. sand, volcanic ash) ingested by engine melt into **Calcium-Magnesium-Alumino-Silicate (CMAS)** deposits above 1200°C
- Molten CMAS degrades EBCs (chemical + mechanical)
  - CMAS infiltration of EBC due to lowered CMAS viscosity at elevated temperatures → CTE mismatch
  - Thermochemical interactions of CMAS with EBC → spallation



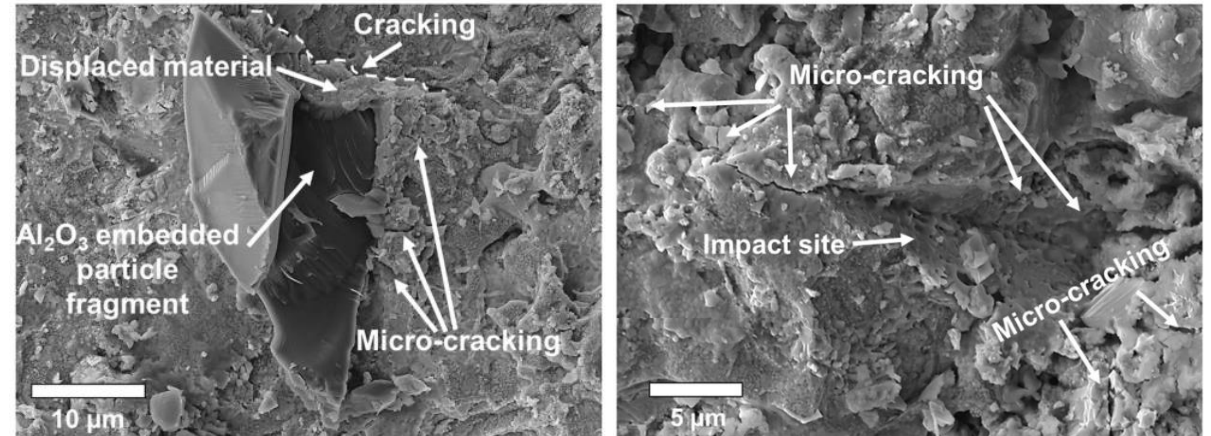
*Eyjafjallajökull volcano eruption in Iceland (2010)*



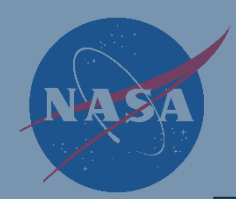
*Damage on a turbine blade caused by CMAS >1200°C*

## Solid Particle Erosion

- Particulates (i.e. sand, volcanic ash) ingested by engine can **mechanically erode** EBCs and CMCs at higher temperatures
- Brittle fracture dominated erosion response of EBCs at high temperature
  - Coating microstructure affects durability



Presby et al., *Ceramics International* **47** (2021)



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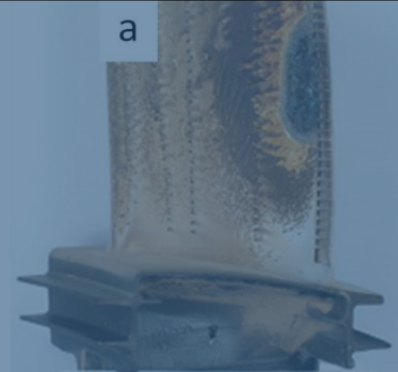
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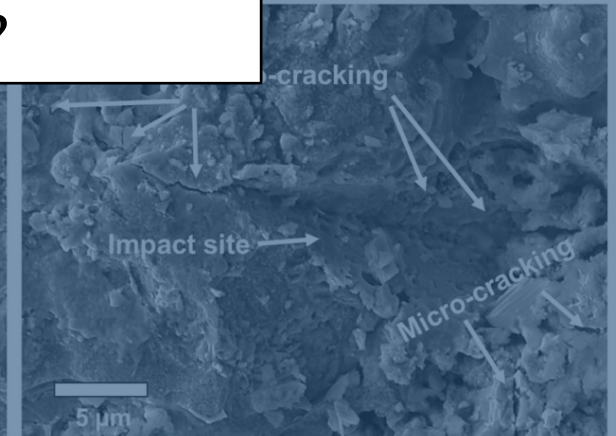
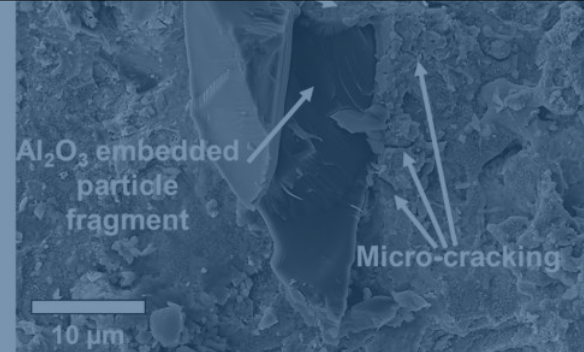
How is erosion durability affected by microstructural and chemical changes caused by CMAS exposure?



Eyjafjallajökull volcano eruption in Iceland (2010)

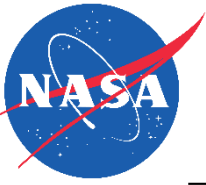


Damage on a turbine blade caused by CMAS >1200°C



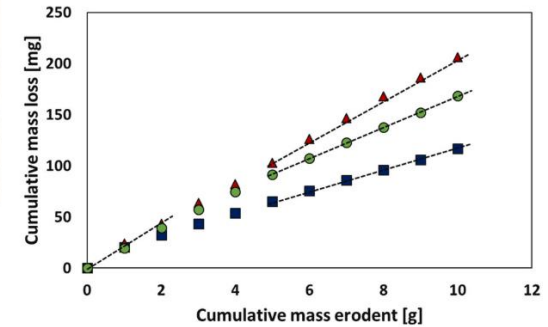
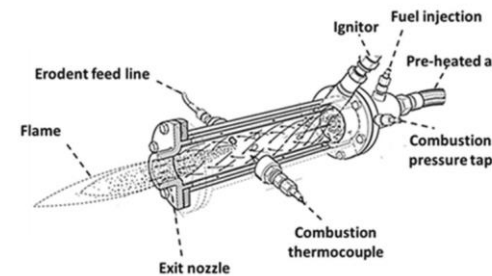
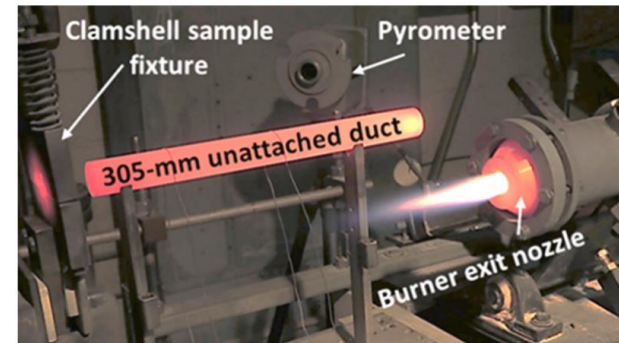
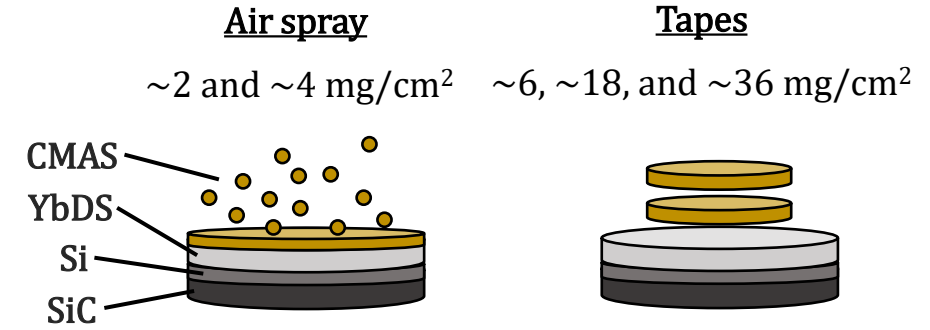
Presby et al., *Ceramics International* 47 (2021)





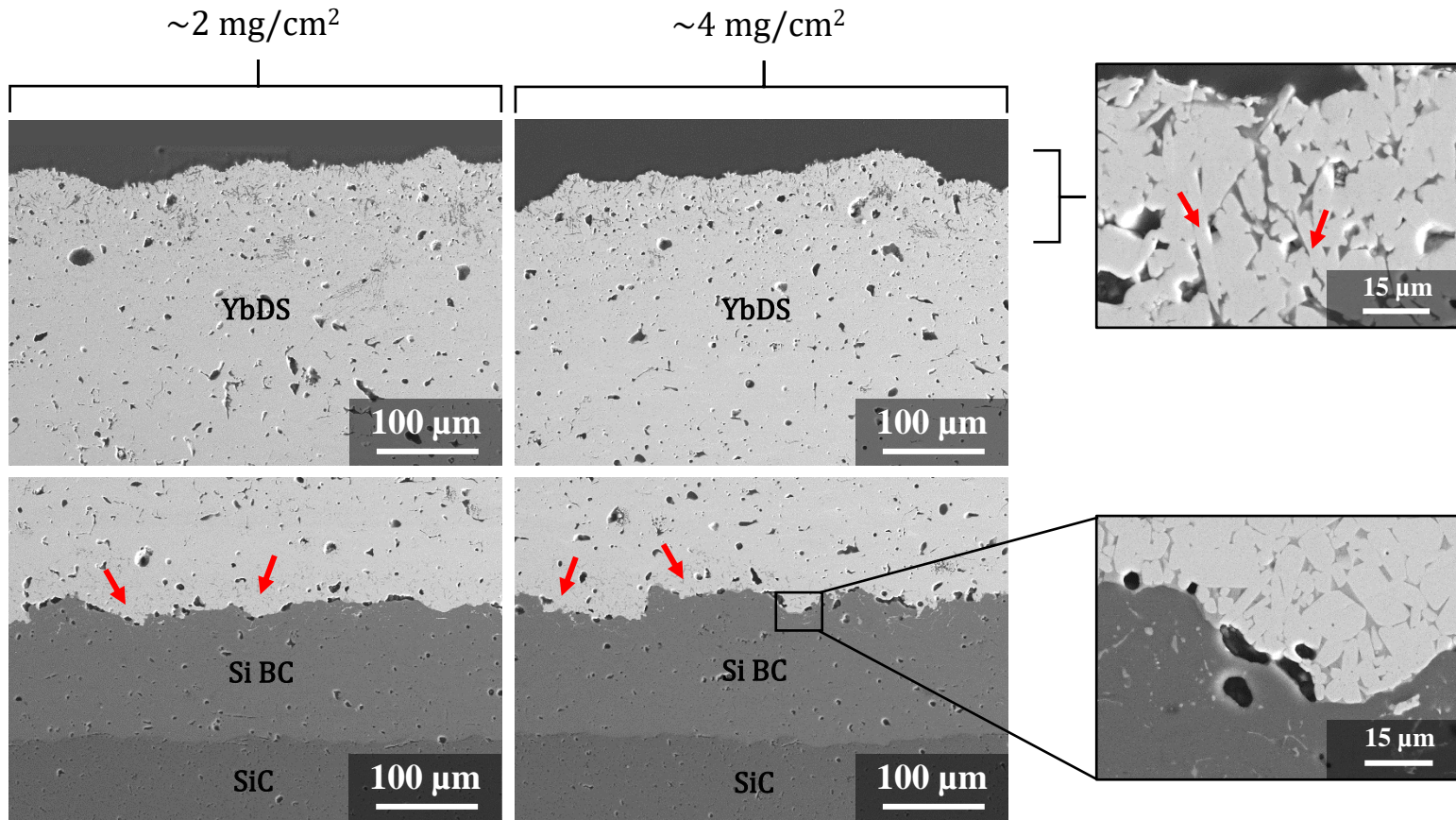
# Experimental Procedures

- Air plasma sprayed modified  $\text{Yb}_2\text{Si}_2\text{O}_7$  (YbDS) coating
  - YAG, mullite, added to improve oxidation performance; Lee, *J. Am. Cer* **102** (2019)
  - $\sim 250 \mu\text{m}$  topcoat with  $\sim 125 \mu\text{m}$  Si bondcoat on SiC SA Hexaloy
- $\sim 2, \sim 4, \sim 6, \sim 18,$  and  $\sim 36 \text{ mg/cm}^2$  loadings
  - $30.67\text{CaO}-8.25\text{MgO}-12.81\text{AlO}_{1.5}-48.27\text{SiO}_2$  (mol.%)
  - Krämer et al. *J. Am. Cer.* **89** (2006)
  - Applied by air spray (REF) and casted tapes (REF)
- All samples furnace heat treated at  $1316^\circ\text{C}$ , 4 hours;
- Reaction products identified using SEM/EDS
- Erosion testing carried out in NASA's Erosion Burner Rig Facility at  $1316^\circ\text{C}$



D.S. Fox et al., NASA/TM- 2011216986 (2011)

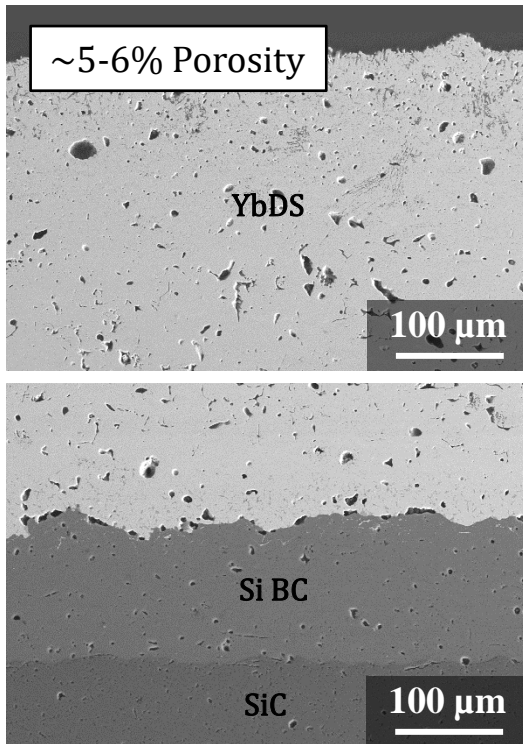
# Results – $\sim 2 \text{ mg/cm}^2$ and $\sim 4 \text{ mg/cm}^2$



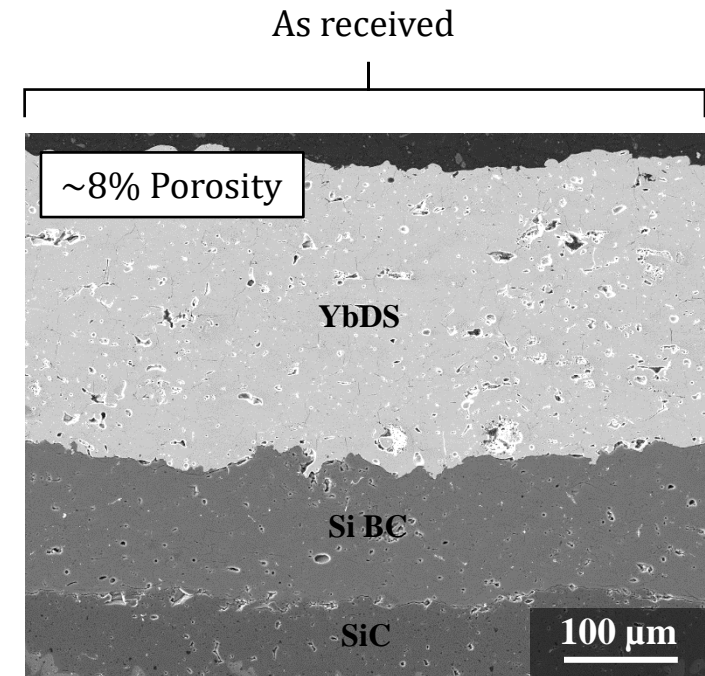
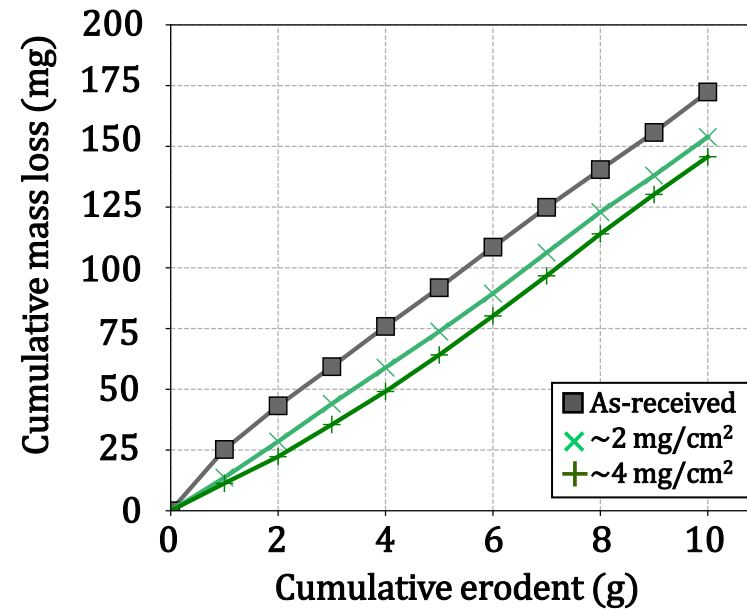
- No residual CMAS was observed
- Pockets of CMAS interspersed with elongated grains having composition consistent with the formation of  $\text{Ca}_2\text{Yb}_8(\text{SiO}_4)_6\text{O}_2$  apatite
- Pockets of CMAS were observed near the bondcoat



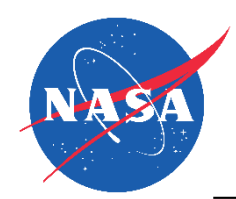
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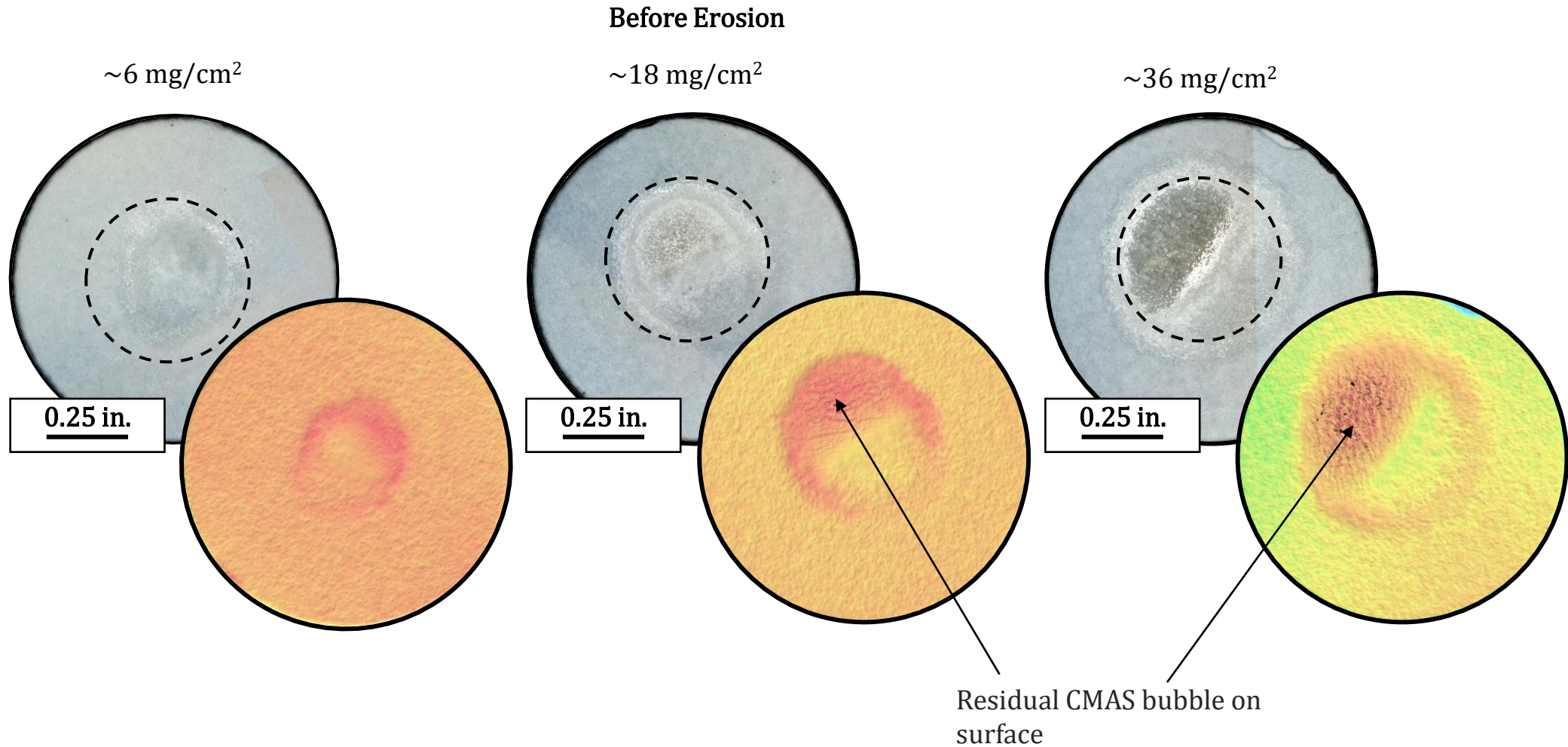
As received  $16.34 \pm 0.32 \text{ mg/g}$   
 $\sim 2 \text{ mg/cm}^2$   $16.08 \pm 0.24 \text{ mg/g}$   
 $\sim 4 \text{ mg/cm}^2$   $16.43 \pm 0.59 \text{ mg/g}$



- Increased loading resulted in slight cumulative mass loss decrease across the entire erosion test.
- Fairly linear behavior throughout the entire test

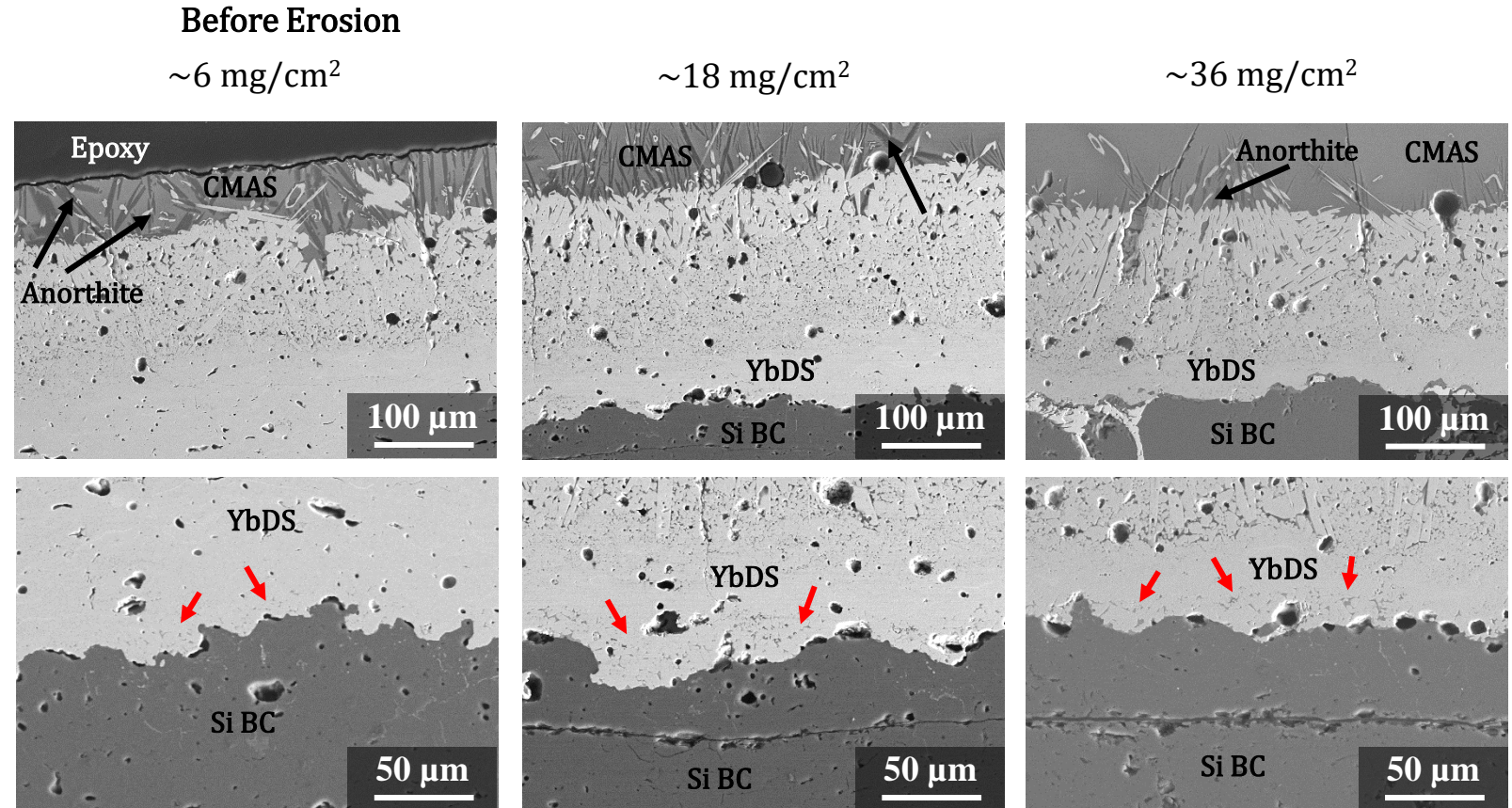


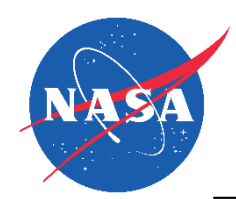
# Results – $\sim 6 \text{ mg/cm}^2$ , $\sim 18 \text{ mg/cm}^2$ , and $\sim 36 \text{ mg/cm}^2$



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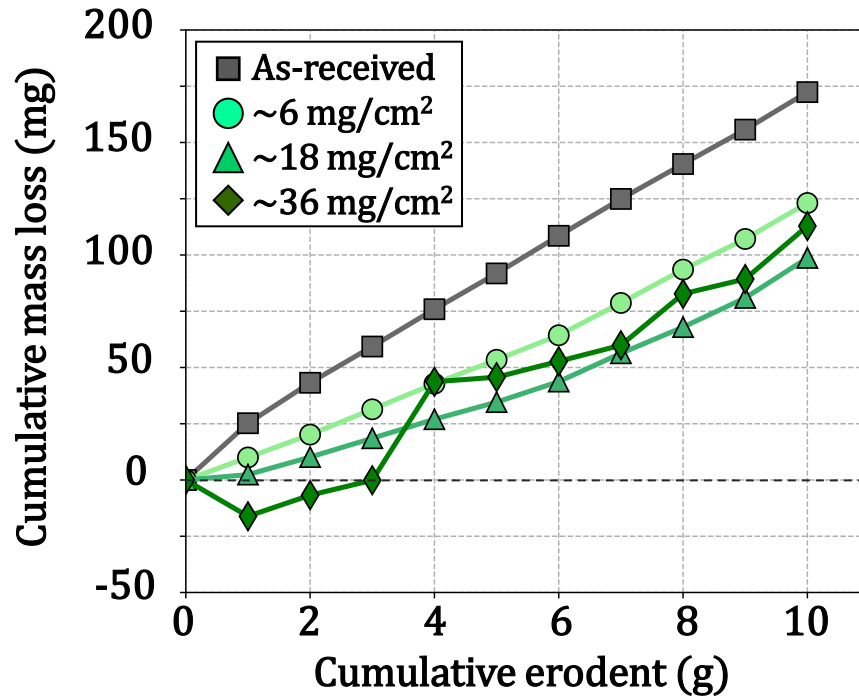
- Thicker layer of apatite formation with increased loading
- Crystallization of residual CMAS to anorthite ( $\text{CaAl}_2\text{Si}_2\text{O}_8$ )
- CMAS pooling between grains at bondcoat consistent with lower loading samples
- Large crack through the bondcoat in  $\sim 18 \text{ mg/cm}^2$  and  $\sim 36 \text{ mg/cm}^2$  samples, extending from the middle of the coating to the edges of CMAS bubble.





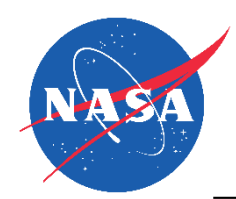
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After Erosion



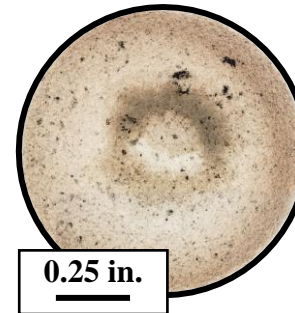
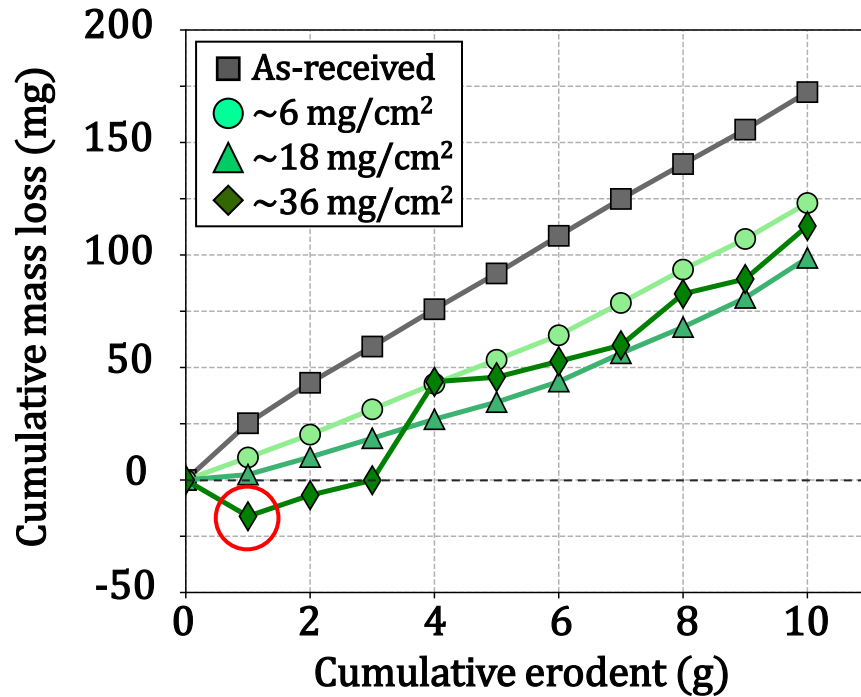
- With increasing loading of CMAS up to  $\sim 18 \text{ mg/cm}^2$ , cumulative mass loss decreased.
- $\sim 6 \text{ mg/cm}^2$  and  $\sim 18 \text{ mg/cm}^2$  exhibited slightly non-linear mass loss behavior
- $\sim 36 \text{ mg/cm}^2$  sample exhibited an initial mass gain up to approximately 3 g of erodent followed by mass loss.



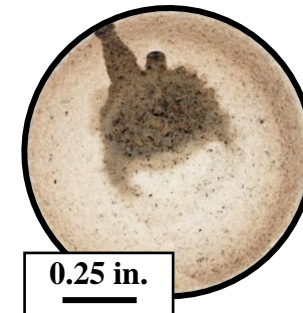


Results –  $\sim 6 \text{ mg/cm}^2$ ,  $\sim 18 \text{ mg/cm}^2$ , and  $\sim 36 \text{ mg/cm}^2$

After Erosion



$\sim 6 \text{ mg/cm}^2$



$\sim 18 \text{ mg/cm}^2$



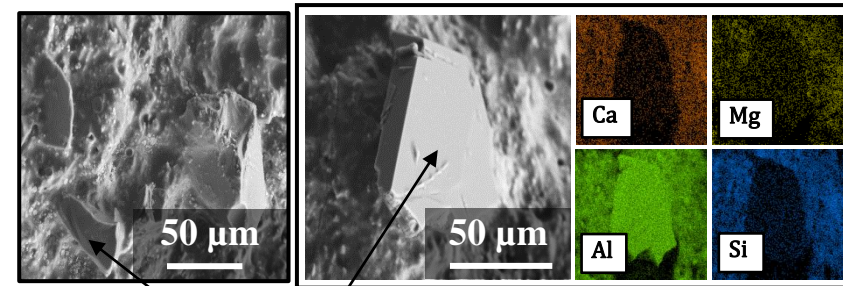
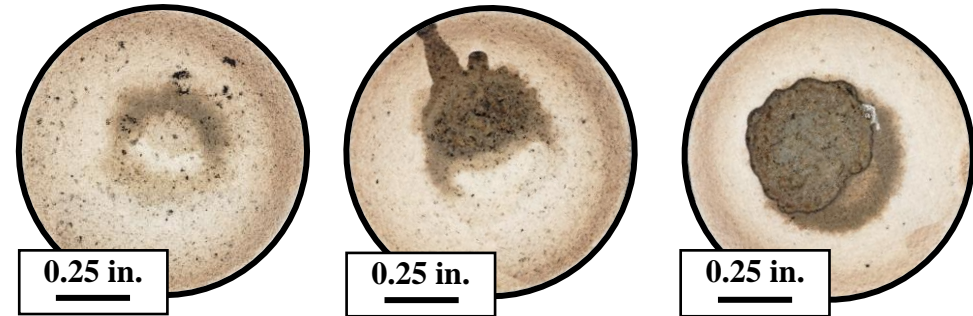
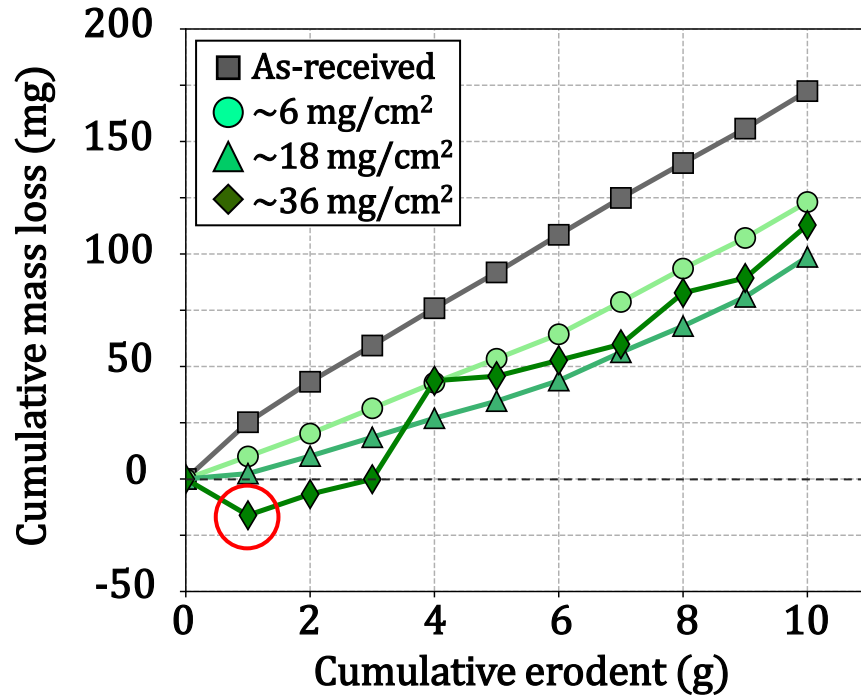
$\sim 36 \text{ mg/cm}^2$

- Sample darker in areas with residual CMAS



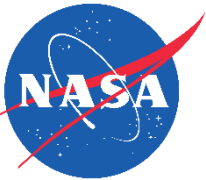
# Results – ~6 mg/cm<sup>2</sup>, ~18 mg/cm<sup>2</sup>, and ~36 mg/cm<sup>2</sup>

After Erosion

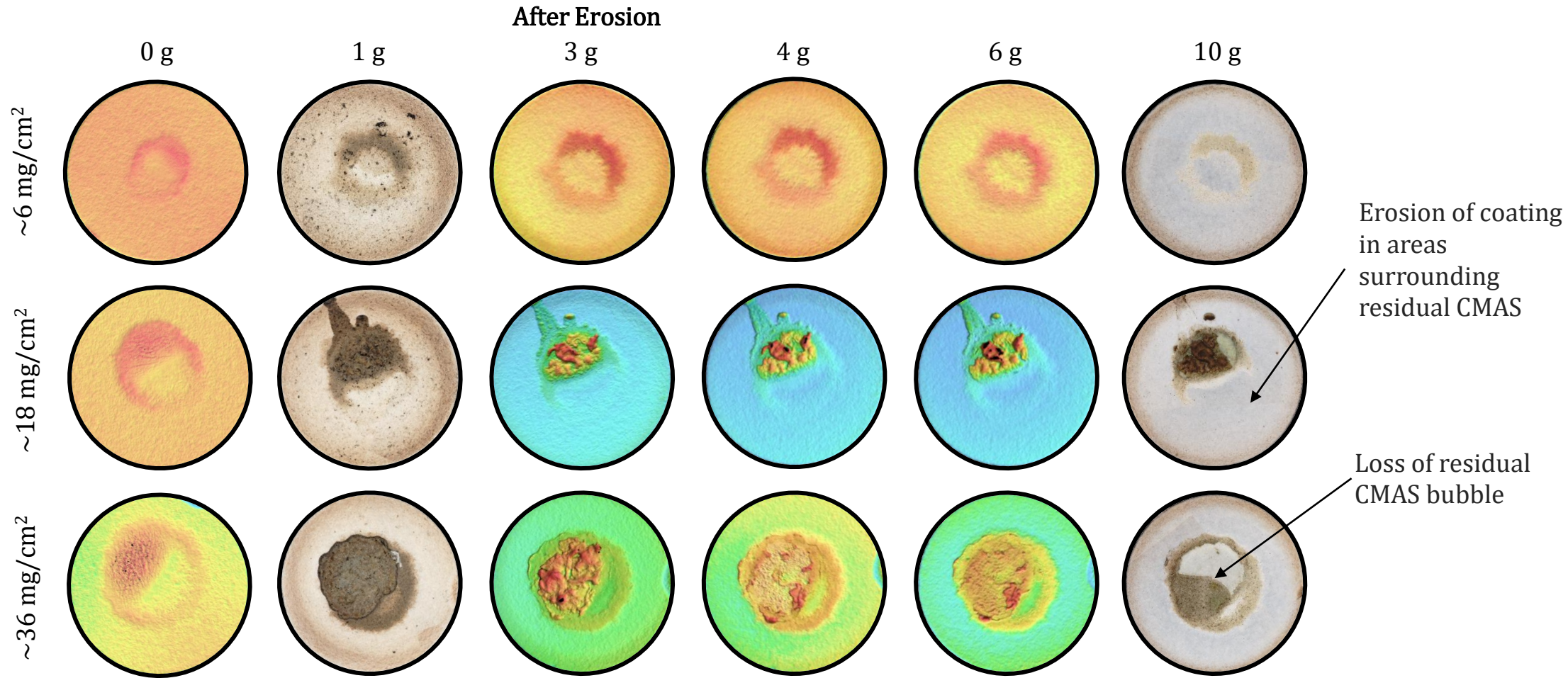


Al<sub>2</sub>O<sub>3</sub> erodent

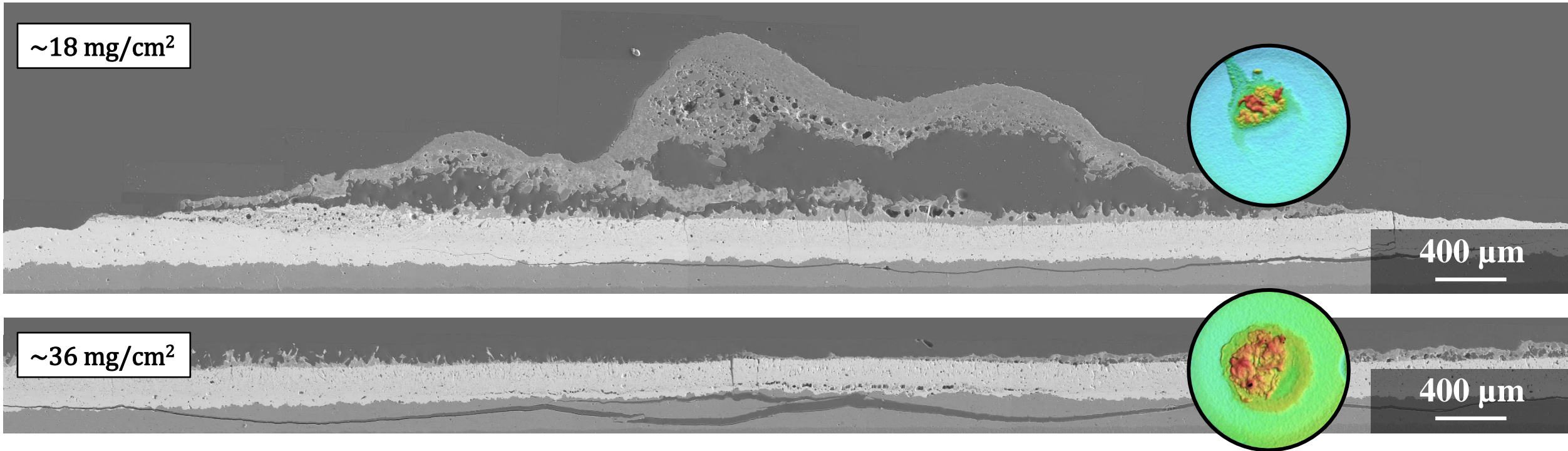




# Results - $\sim 6 \text{ mg/cm}^2$ , $\sim 18 \text{ mg/cm}^2$ , and $\sim 36 \text{ mg/cm}^2$



After Erosion



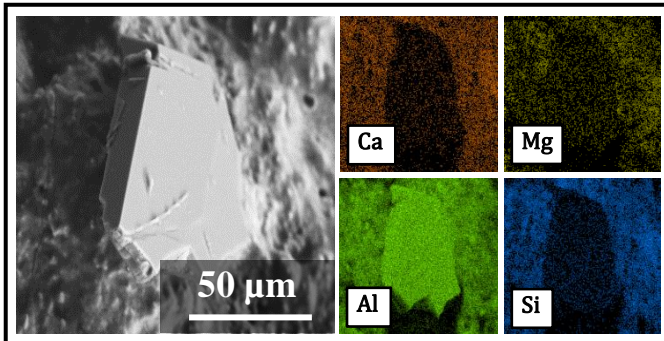
- SEM cross sections show large cracks throughout bond coat; cracks were much wider in  $\sim 36 \text{ mg/cm}^2$  sample after erosion testing.
- Bubbling and rumpling of residual CMAS due to burner rig exposure



# Results – $\sim 6 \text{ mg/cm}^2$ , $\sim 18 \text{ mg/cm}^2$ , and $\sim 36 \text{ mg/cm}^2$

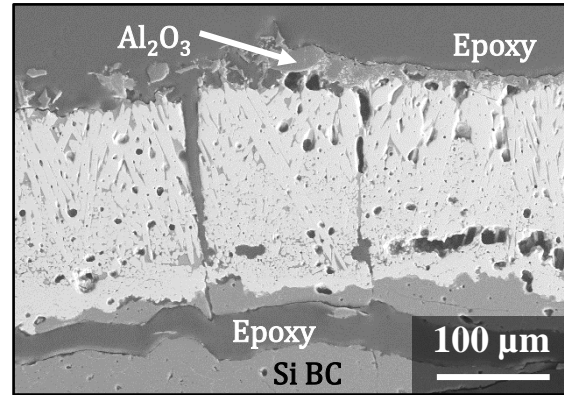
- Additional analyses revealed greater changes in the coating chemistry and morphology, which are not captured in mass loss plots and could be detrimental to the coatings in service.

Erodent accumulation



Are CMAS particles more likely to “splat” and stick to coatings than to remove material due to lower melting temperatures? → greater mass accumulation over time

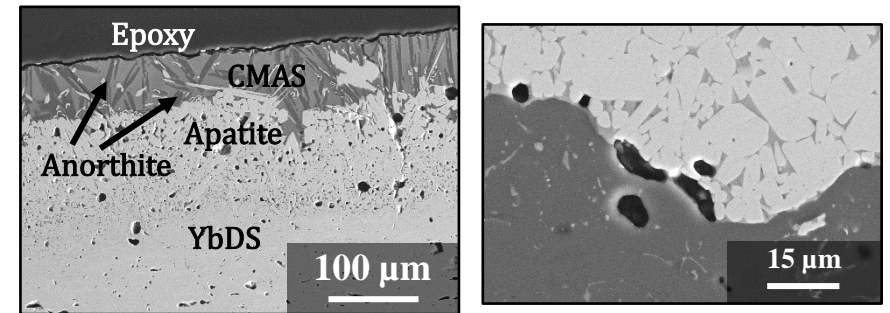
Catastrophic mass loss



Spallation of residual CMAS and coating more likely with increased CMAS loading and heat treatment time?

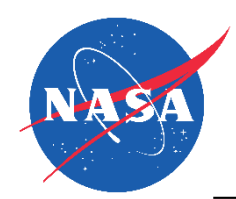
- Thermal shock and thermal expansion mismatch

Morphological changes affecting mechanical durability



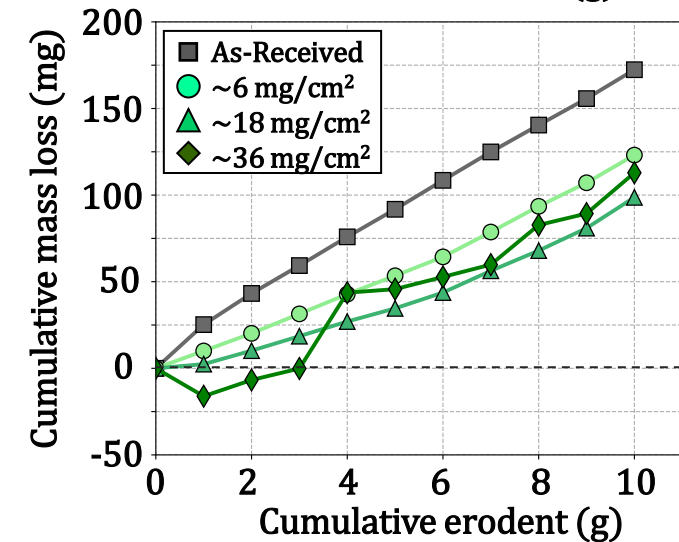
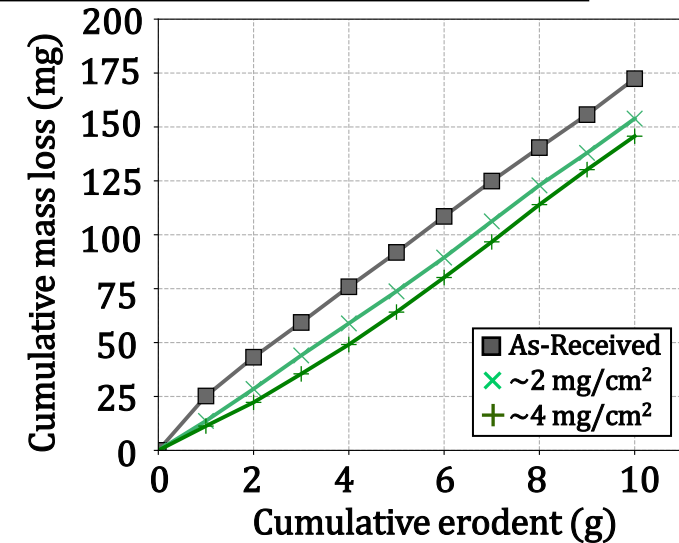
Differences in the mechanical properties (fracture toughness, elastic modulus, hardness) of reaction products will affect durability

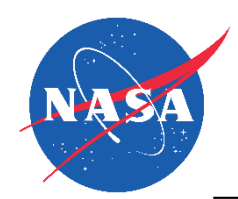
- Grain coarsening observed in the CMAS-reacted samples



# Summary

- Erosion durability of a modified  $\text{Yb}_2\text{Si}_2\text{O}_7$  EBC was evaluated after exposure to low and high CMAS loads.
- CMAS loading amount had large effects on the thermomechanical durability of the coatings.
  - Low CMAS loads resulted in generally no change to erosion durability.
  - Erosion durability at higher loads was more difficult to assess because of the tendency of erodent material to stick to residual CMAS on the coating surfaces.
  - CMAS adhesion, extraneous phase formation as well as the formation of voids and cracks with higher loading meant that detrimental changes in the coatings were occurring that may not necessarily be captured in cumulative mass loss plots.
- Tracking changes in the chemistry and morphology of EBCs will be crucial in understanding the mechanisms of degradation due to high-temperature particle interactions.

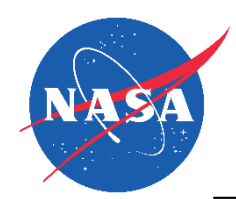




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Thank You!





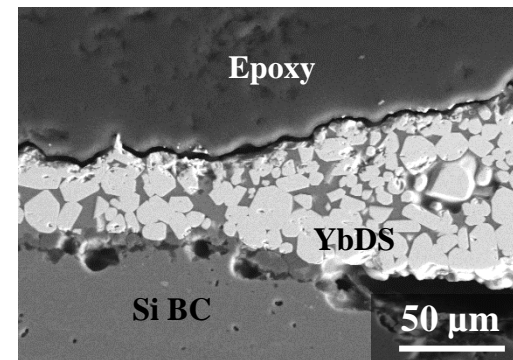
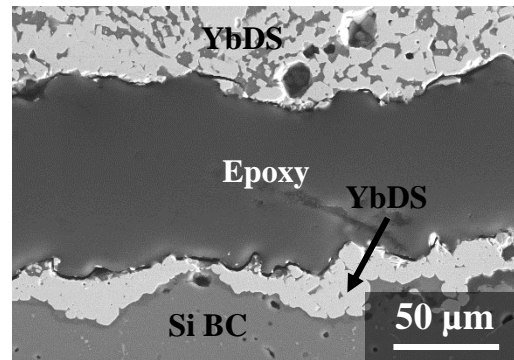
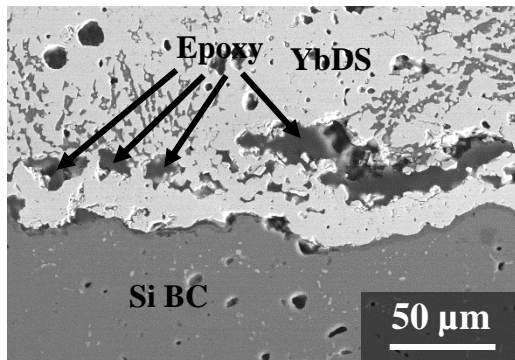
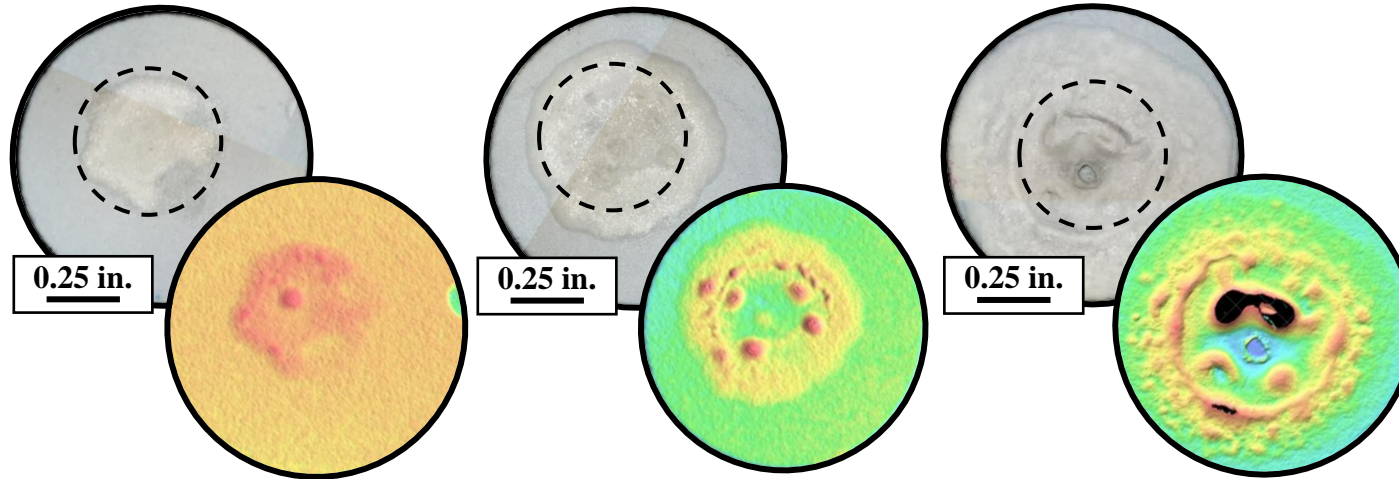
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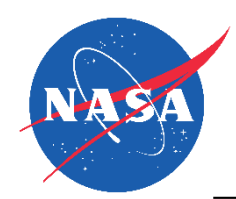
100 hours

$\sim 6 \text{ mg/cm}^2$

$\sim 18 \text{ mg/cm}^2$

$\sim 36 \text{ mg/cm}^2$

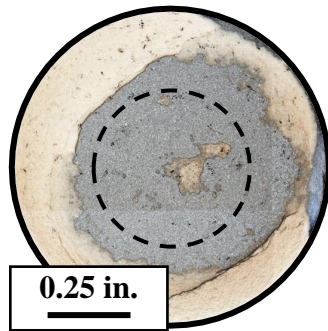
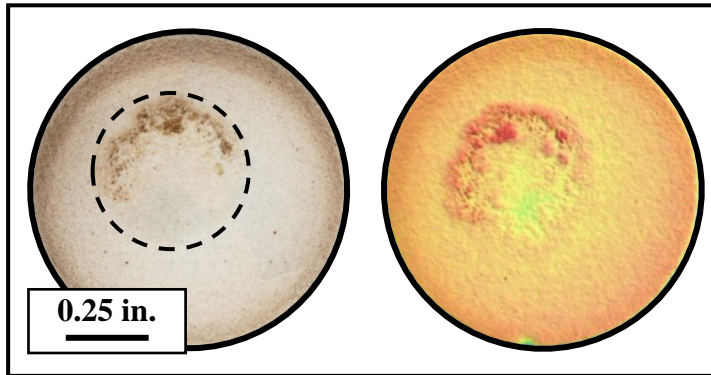




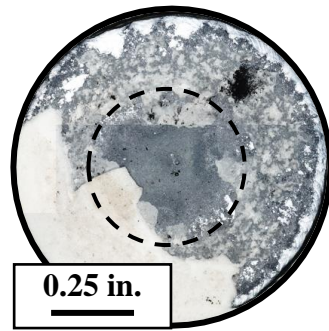
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100 hours

$\sim 6 \text{ mg/cm}^2$



$\sim 18 \text{ mg/cm}^2$



$\sim 36 \text{ mg/cm}^2$

