

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

Jamesa L. Stokes¹, Michael J. Presby¹, Rebekah I. Webster¹,
John A. Setlock², Bryan J. Harder¹

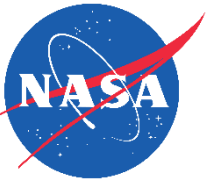
¹NASA Glenn Research Center

²University of Toledo

Acknowledgments

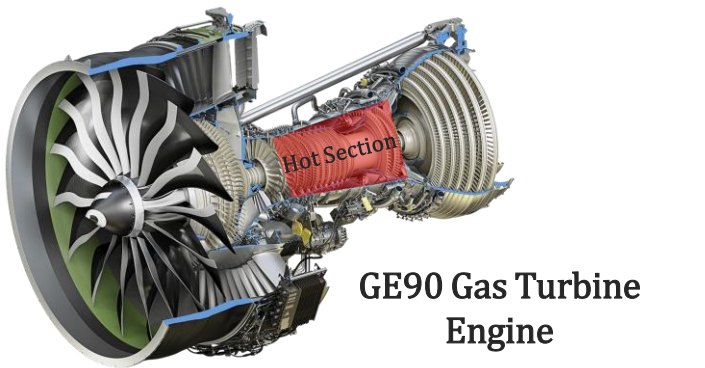
Transformational Tools and Technologies (TTT) Project

Hybrid Thermally Efficient Core (HyTEC) Project

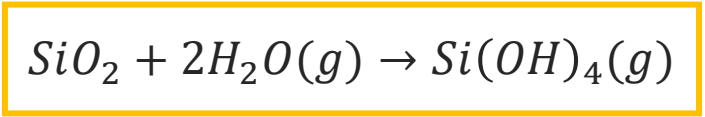
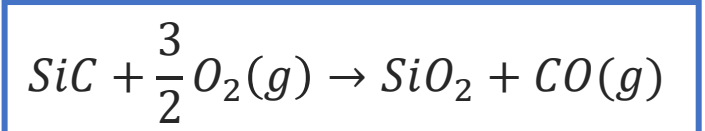


Introduction and Motivation – Ceramic Matrix Composites (CMCs)

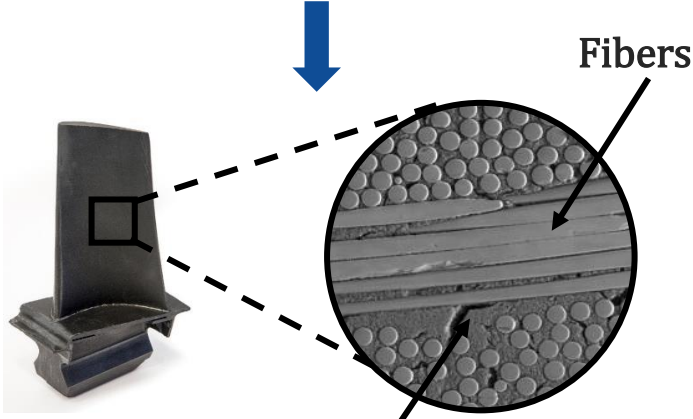
- CMC turbine engine components offer high temperature stability, but recess in high temperature water vapor environments



GE90 Gas Turbine Engine



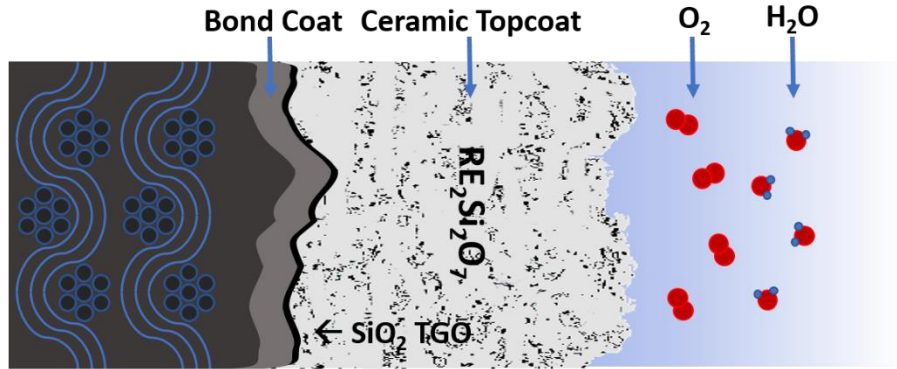
Environmental Barrier Coatings (EBCs)



CMC Turbine Blade

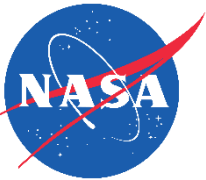
Matrix

Fibers

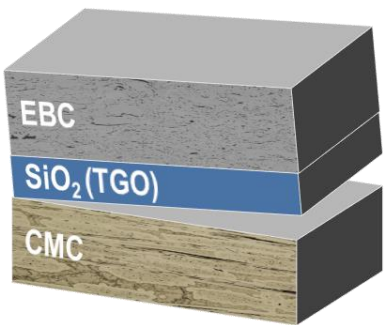


Opila et al. *J. Am. Cer.* 82 (1999)

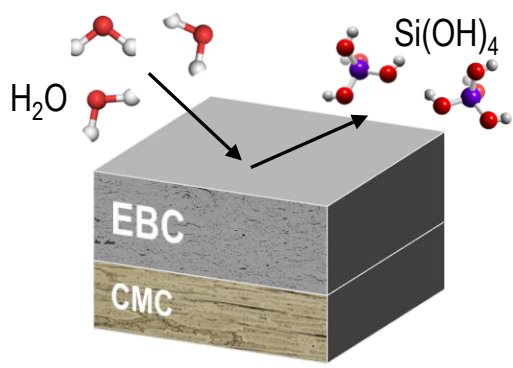




Introduction and Motivation – EBC Development and Testing



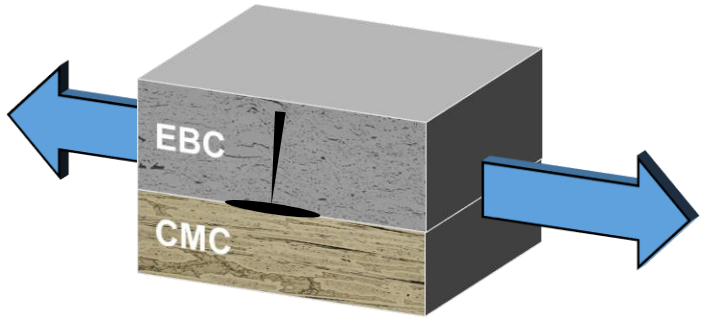
Steam Oxidation



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

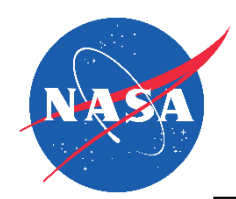


Thermomechanical Durability

Molten Silicate Attack and Infiltration

Erosion and FOD

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



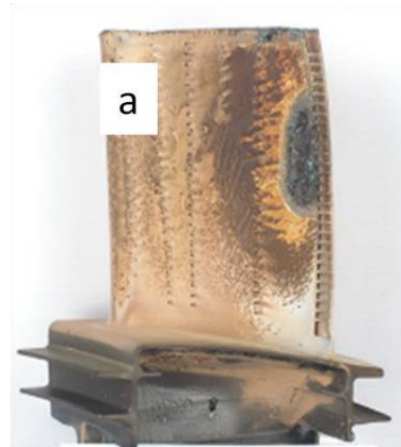
Introduction and Motivation – EBC Degradation by Particulate Ingestion

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



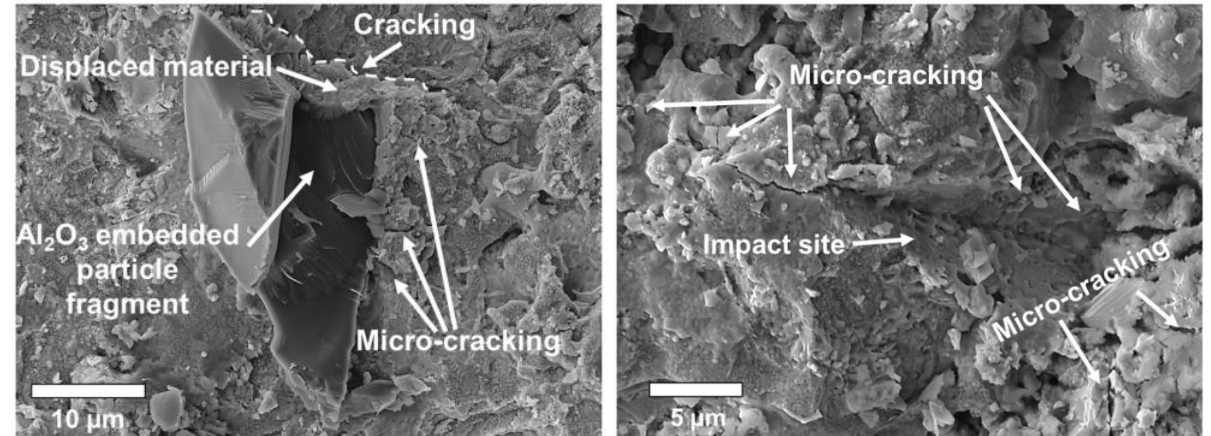
Eyjafjallajökull volcano eruption in Iceland (2010)



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

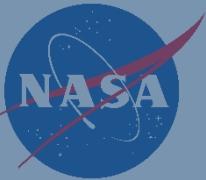
Solid Particle Erosion

- 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)

Presby et al., *Coatings* **13** (2023)



Introduction and Motivation – EBC Degradation by Particulate Ingestion

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 - Thermochemical

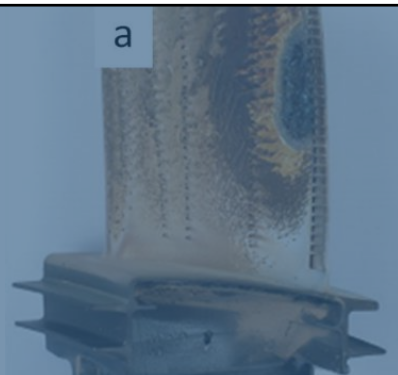
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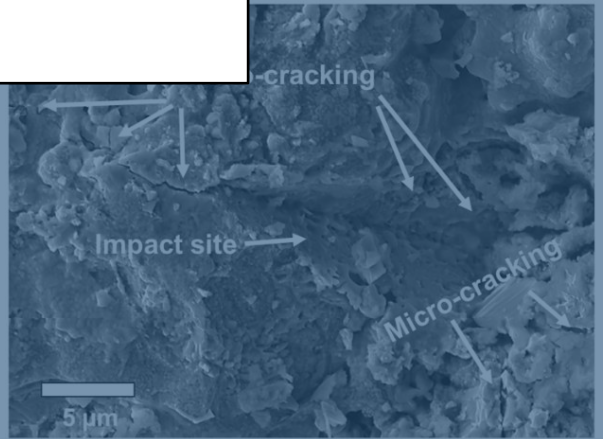
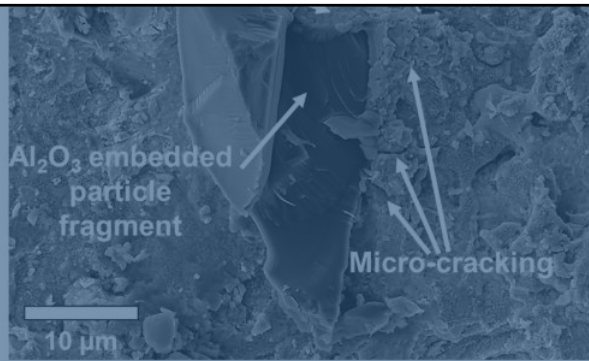
How is erosion durability affected by microstructural and chemical changes caused by CMAS exposure?



Eyjafjallajökull volcano eruption in Iceland (2010)



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

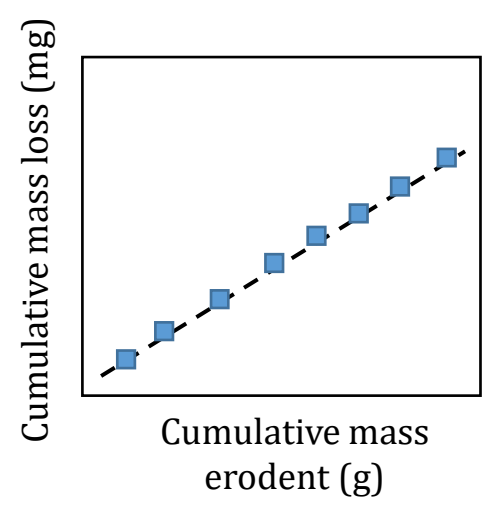
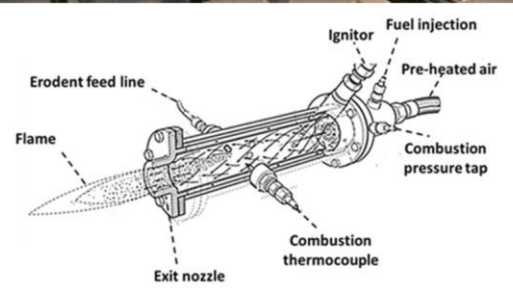
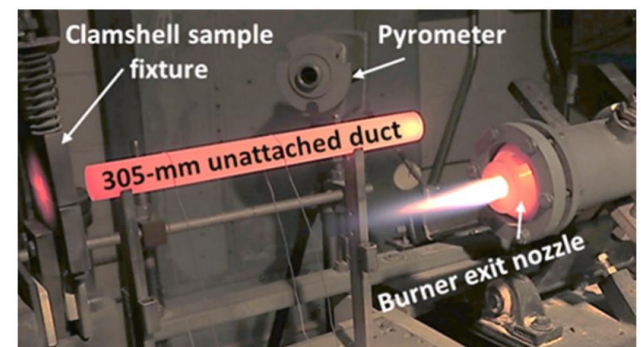
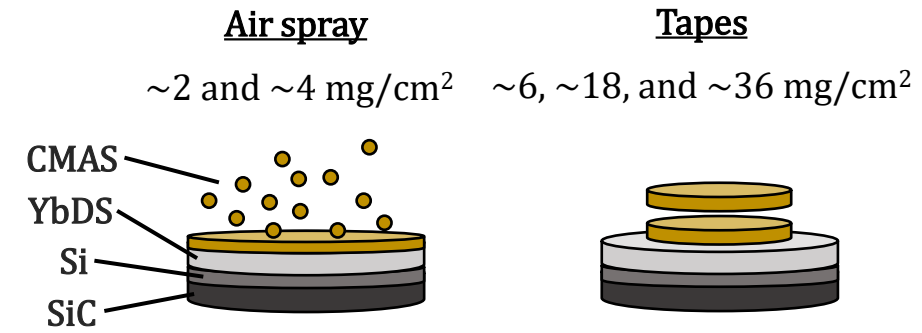


Presby et al., *Ceramics International* 47 (2021)
Presby et al., *Coatings* 13 (2023)



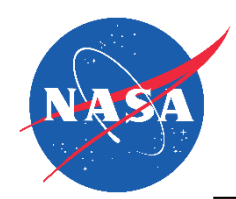
Experimental Procedures

- 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 Hexoloy
- $\sim 2, \sim 4, \sim 6, \sim 18,$ 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 (Harder et al. *In Preparation*) and casted tapes (Kowalski et al. *J. Am. Cer* **106** (2023))
- All samples furnace heat treated at 1316°C , 4 hours;
- Reaction products identified using SEM/EDS
- Erosion testing carried out in NASA's Erosion Burner Rig Facility at 1316°C
- $\sim 60 \mu\text{m}$ Al_2O_3 erodent

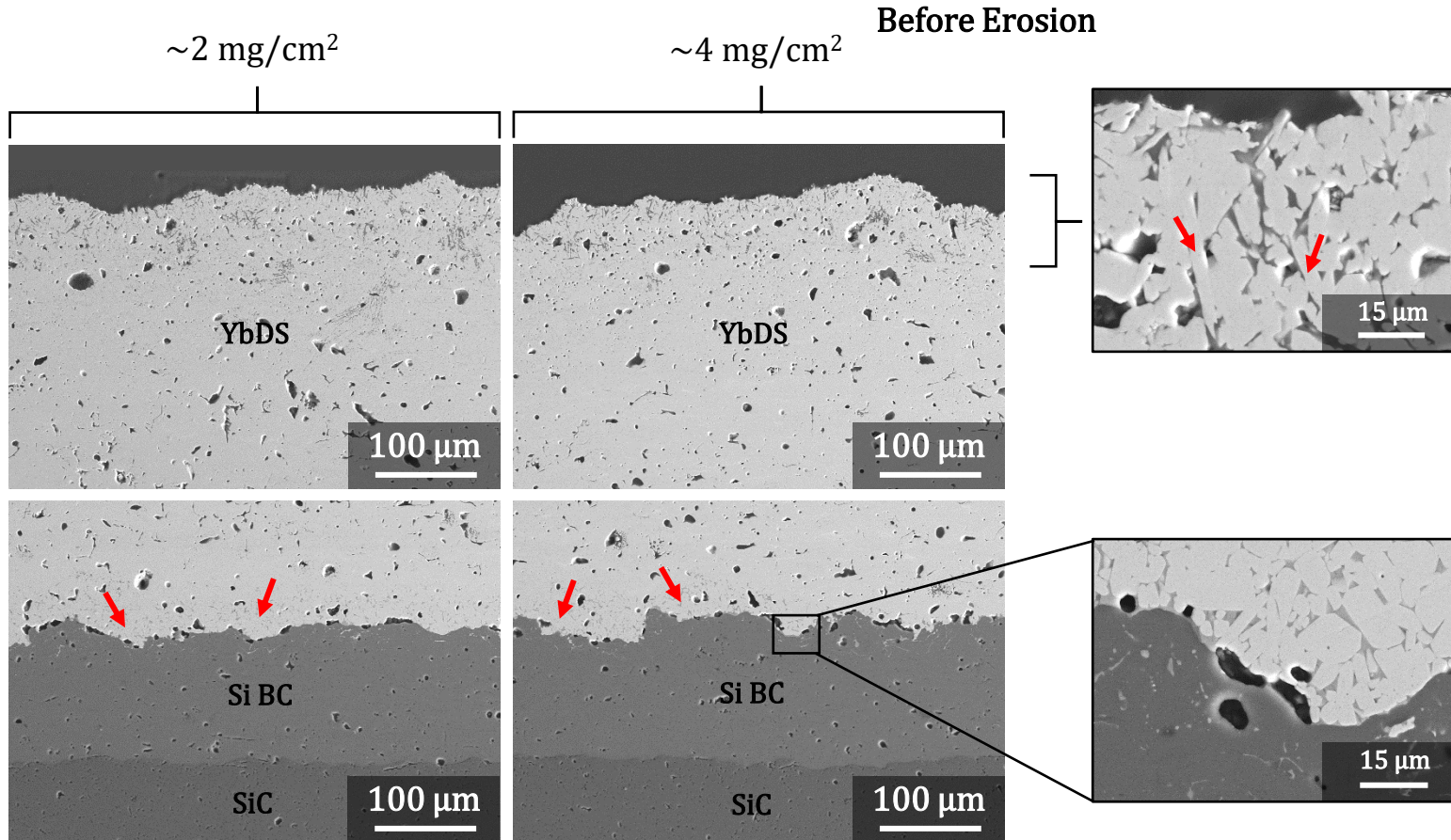


D.S. Fox et al., NASA/TM- 2011216986 (2011)

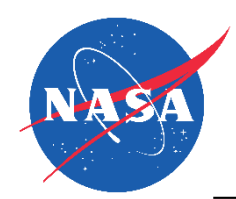




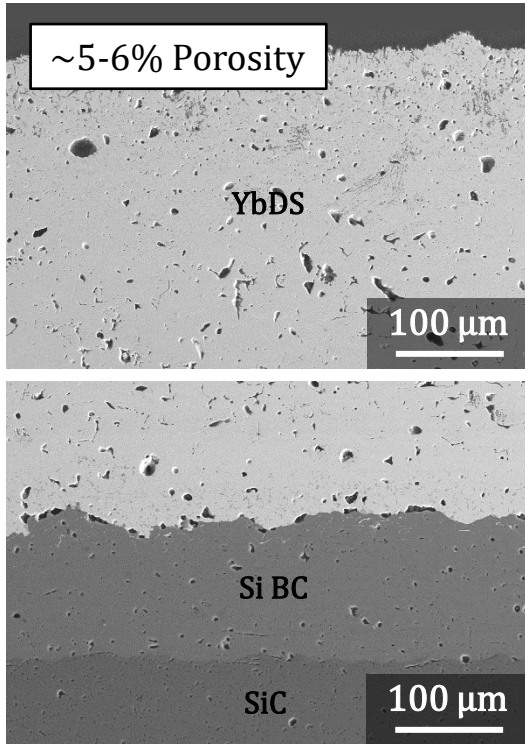
Results - $\sim 2 \text{ mg/cm}^2$ and $\sim 4 \text{ mg/cm}^2$



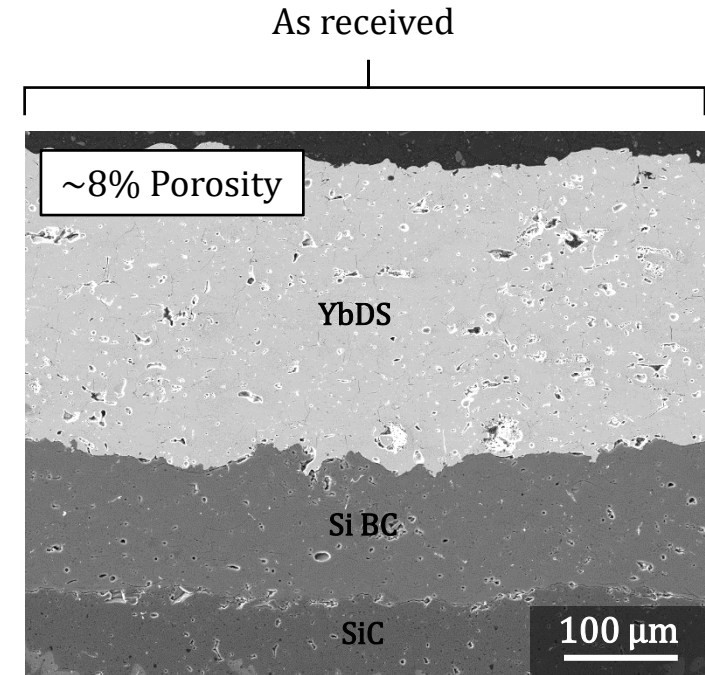
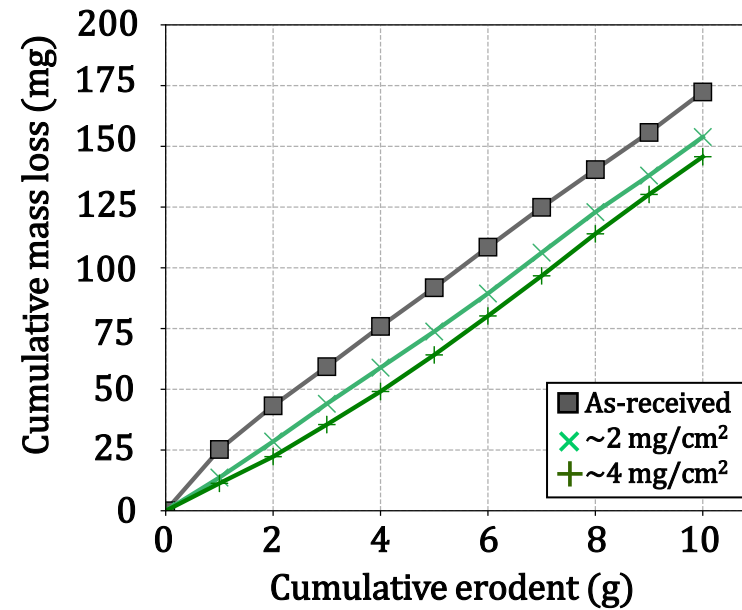
- No residual CMAS was observed
- Pockets of CMAS interspersed with elongated grains having composition consistent with the formation of $\text{Ca}_2\text{Yb}_8(\text{SiO}_4)_6\text{O}_2$ apatite
- Pockets of CMAS were observed near the bondcoat



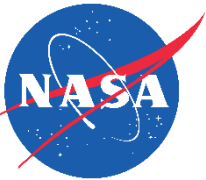
Results – $\sim 2 \text{ mg/cm}^2$ and $\sim 4 \text{ mg/cm}^2$



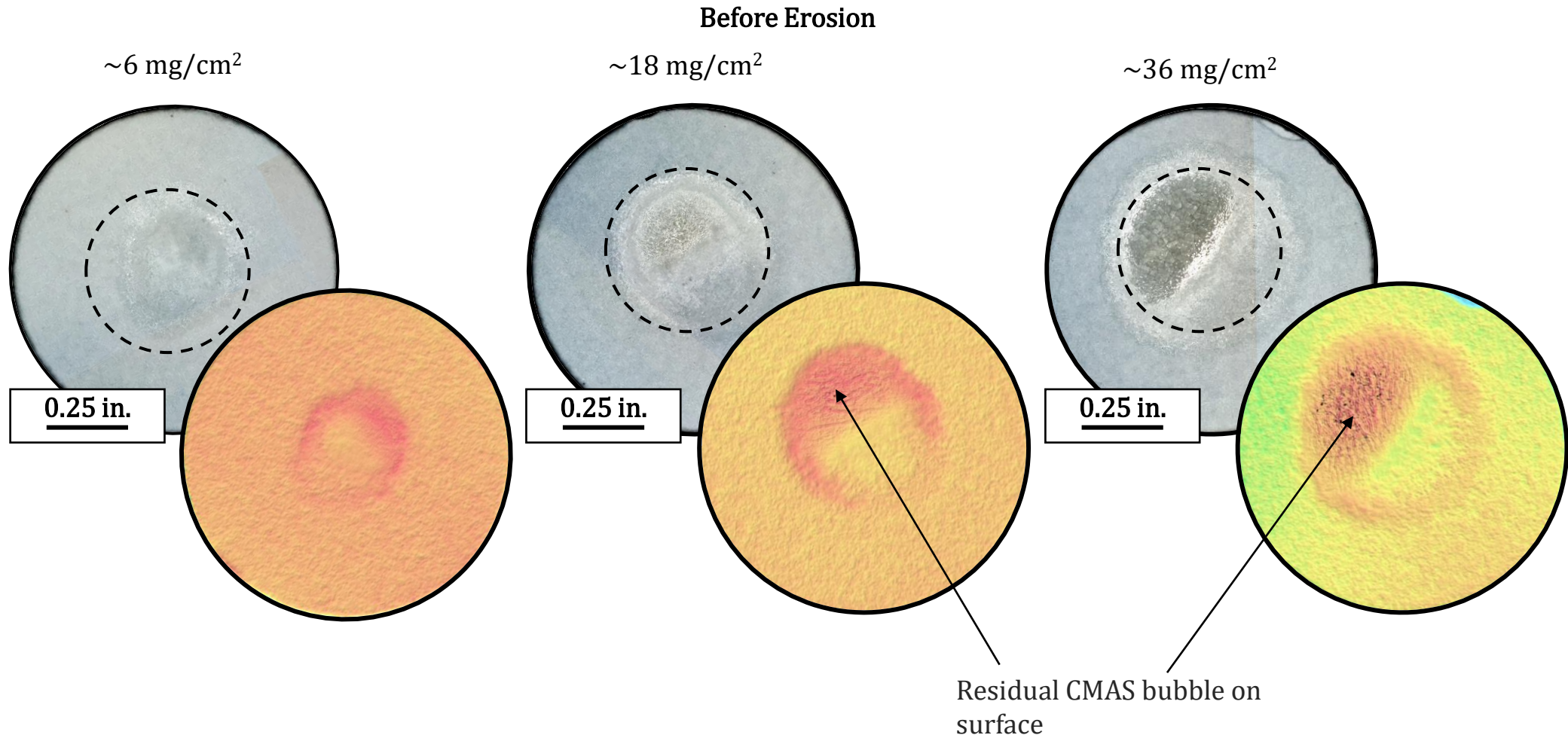
As received 16.34 mg/g
 $\sim 2 \text{ mg/cm}^2$ 16.08 mg/g
 $\sim 4 \text{ mg/cm}^2$ 16.43 mg/g

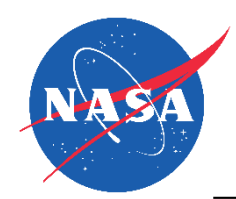


- Increased loading resulted in slight cumulative mass loss decrease across the entire erosion test.
- Fairly linear behavior throughout the entire test



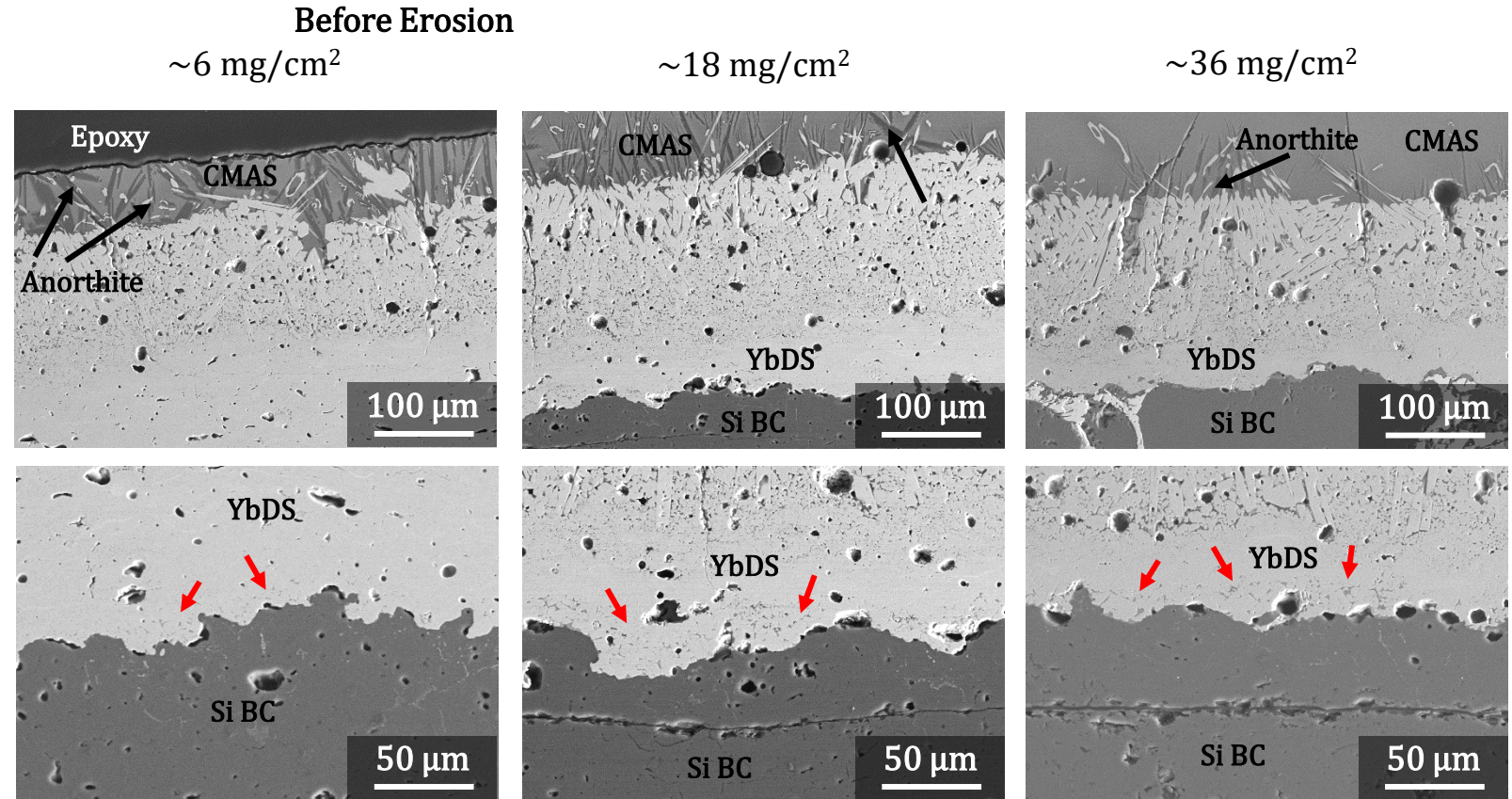
Results - $\sim 6 \text{ mg/cm}^2$, $\sim 18 \text{ mg/cm}^2$, and $\sim 36 \text{ mg/cm}^2$

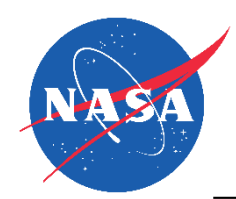




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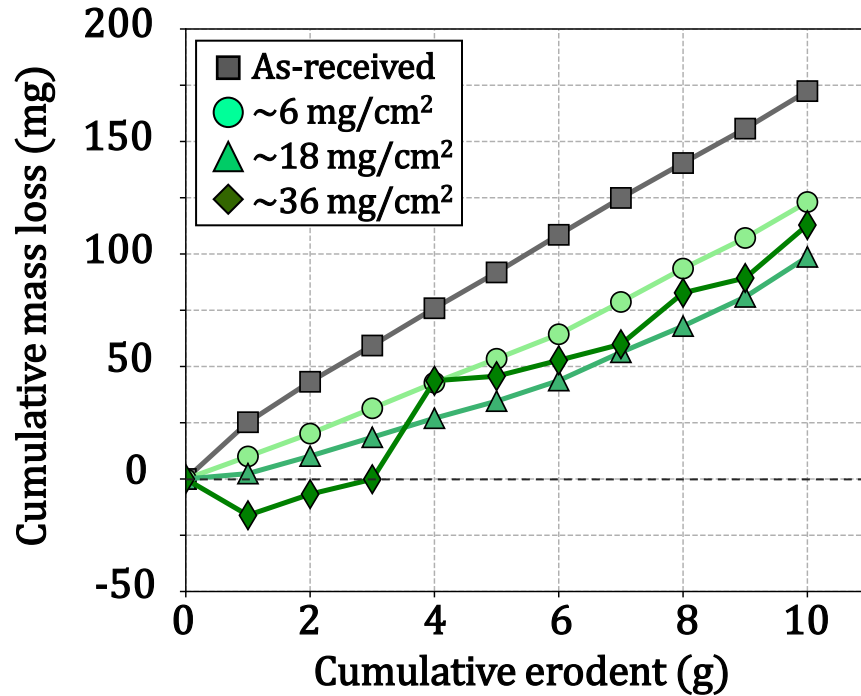
- Thicker layer of apatite formation with increased loading
- Crystallization of residual CMAS to anorthite ($\text{CaAl}_2\text{Si}_2\text{O}_8$)
- CMAS pooling between grains at bondcoat consistent with lower loading samples
- Large crack through the bondcoat in $\sim 18 \text{ mg/cm}^2$ and $\sim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



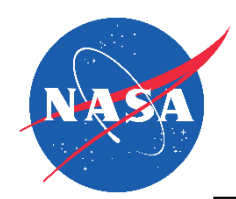


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After Erosion

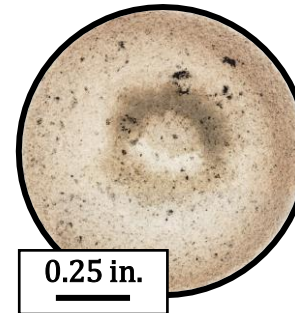
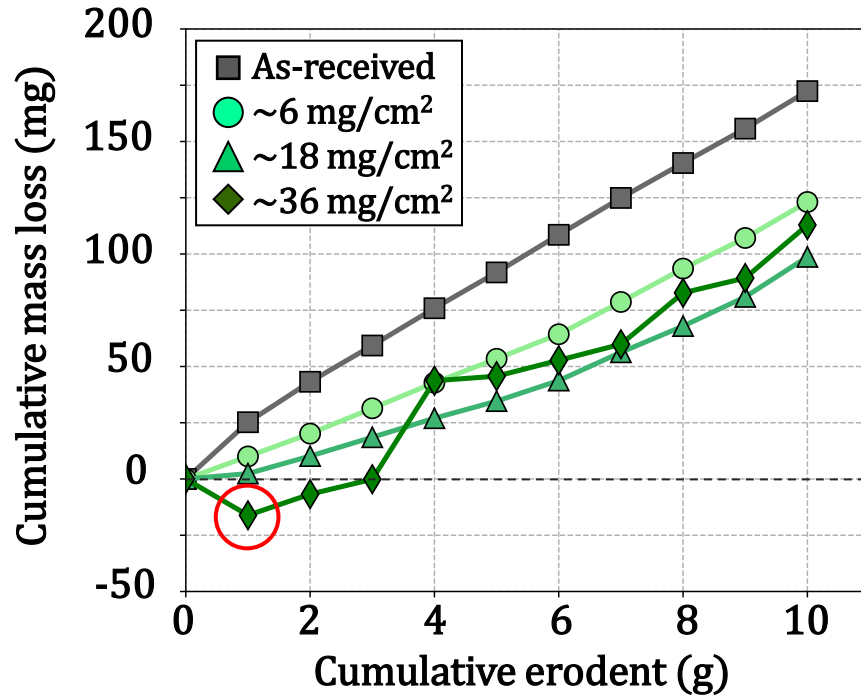


- With increasing loading of CMAS up to $\sim 18 \text{ mg/cm}^2$, cumulative mass loss decreased.
- $\sim 6 \text{ mg/cm}^2$ and $\sim 18 \text{ mg/cm}^2$ exhibited slightly non-linear mass loss behavior
- $\sim 36 \text{ mg/cm}^2$ sample exhibited an initial mass gain up to approximately 3 g of erodent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

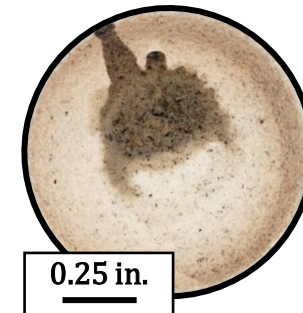


Results - $\sim 6 \text{ mg/cm}^2$, $\sim 18 \text{ mg/cm}^2$, and $\sim 36 \text{ mg/cm}^2$

After Erosion



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

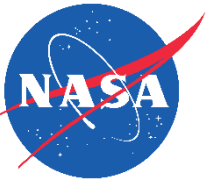


$\sim 18 \text{ mg/cm}^2$

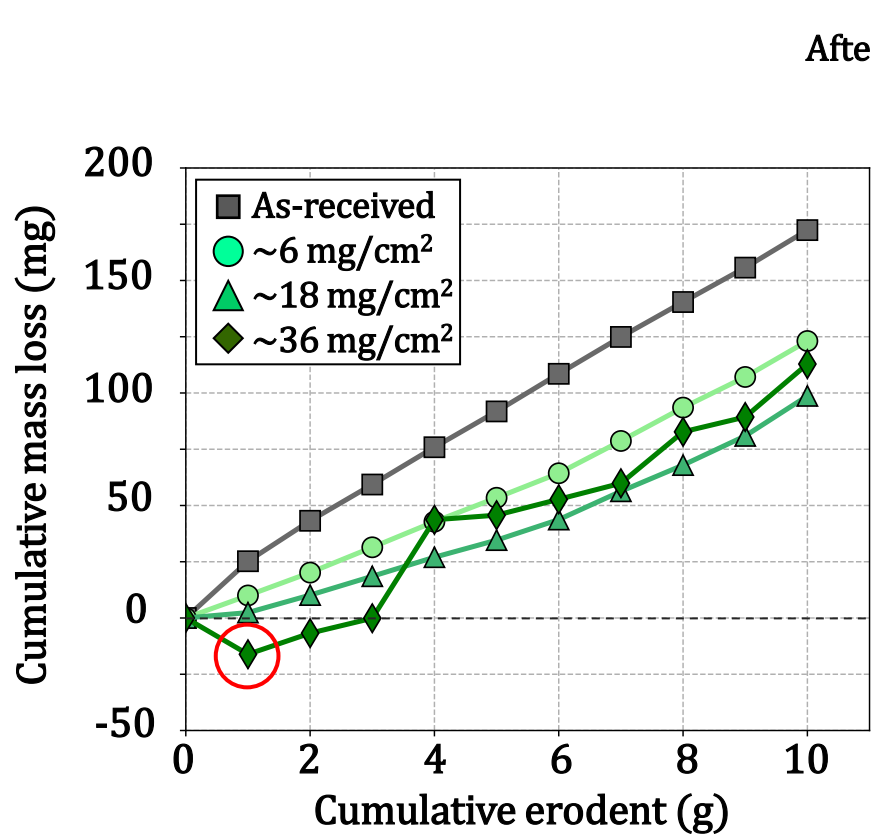


$\sim 36 \text{ mg/cm}^2$

- Sample darker in areas with residual CMAS

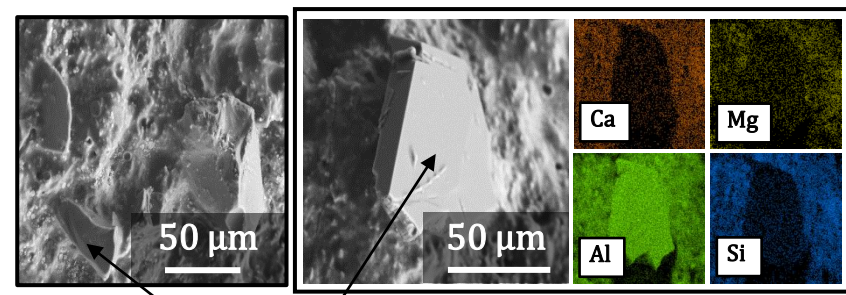
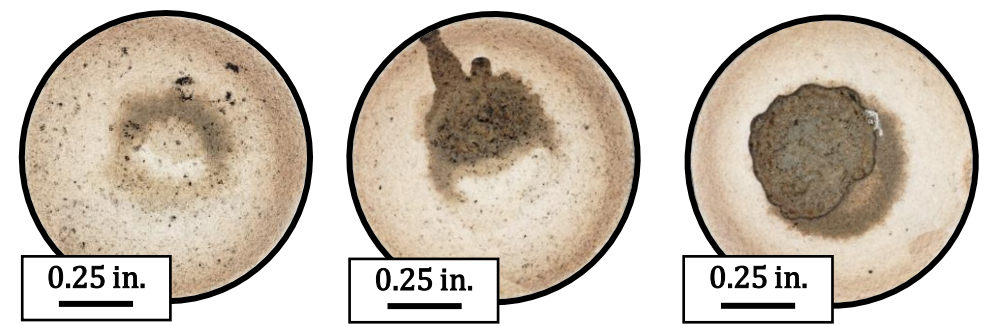


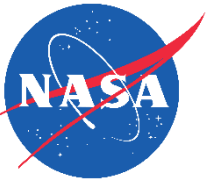
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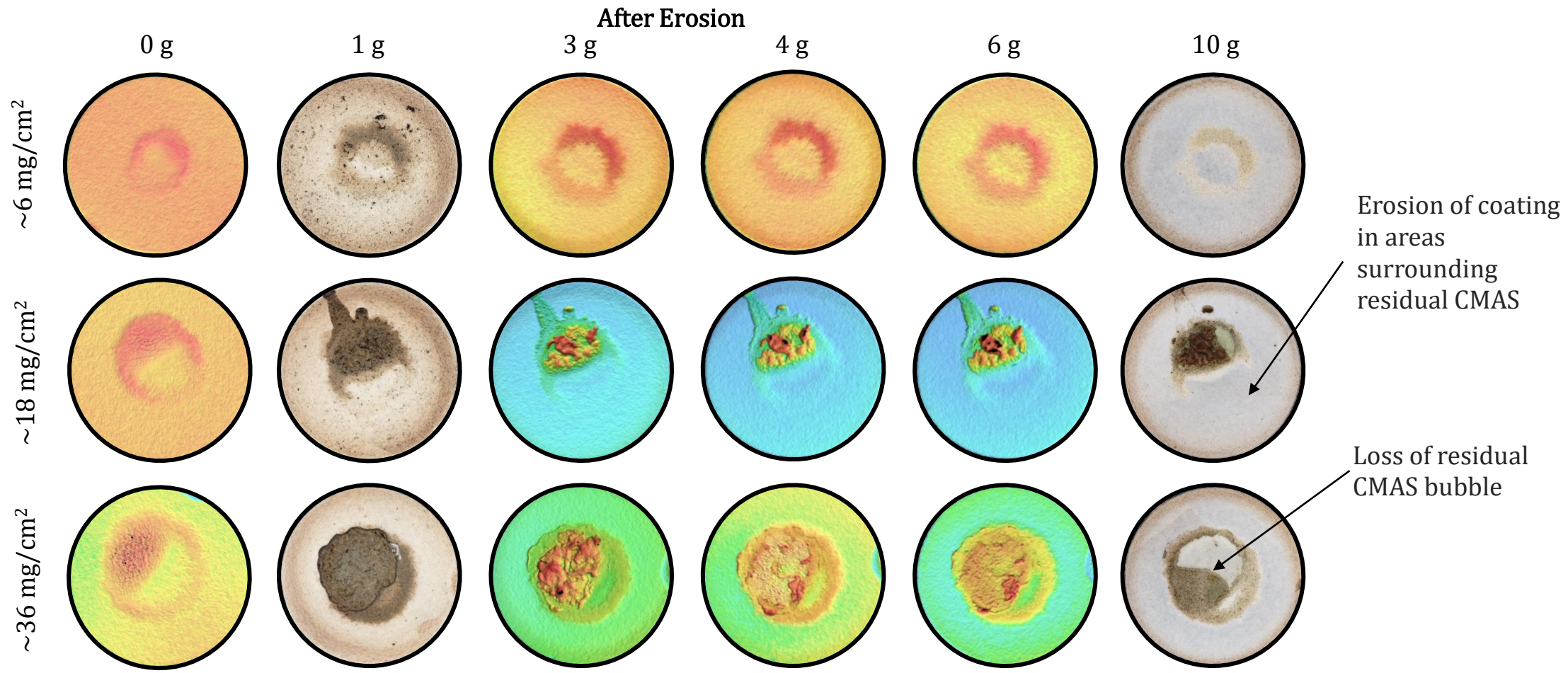
After Erosion

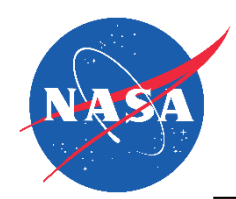
As received 16.34 mg/g
 $\sim 6 \text{ mg/cm}^2$ 14.05 mg/g
 $\sim 18 \text{ mg/cm}^2$ 12.68 mg/g
 $\sim 36 \text{ mg/cm}^2$ 13.38 mg/g





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





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After Erosion

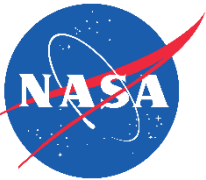
$\sim 18 \text{ mg/cm}^2$

400 μm

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

400 μm

- 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



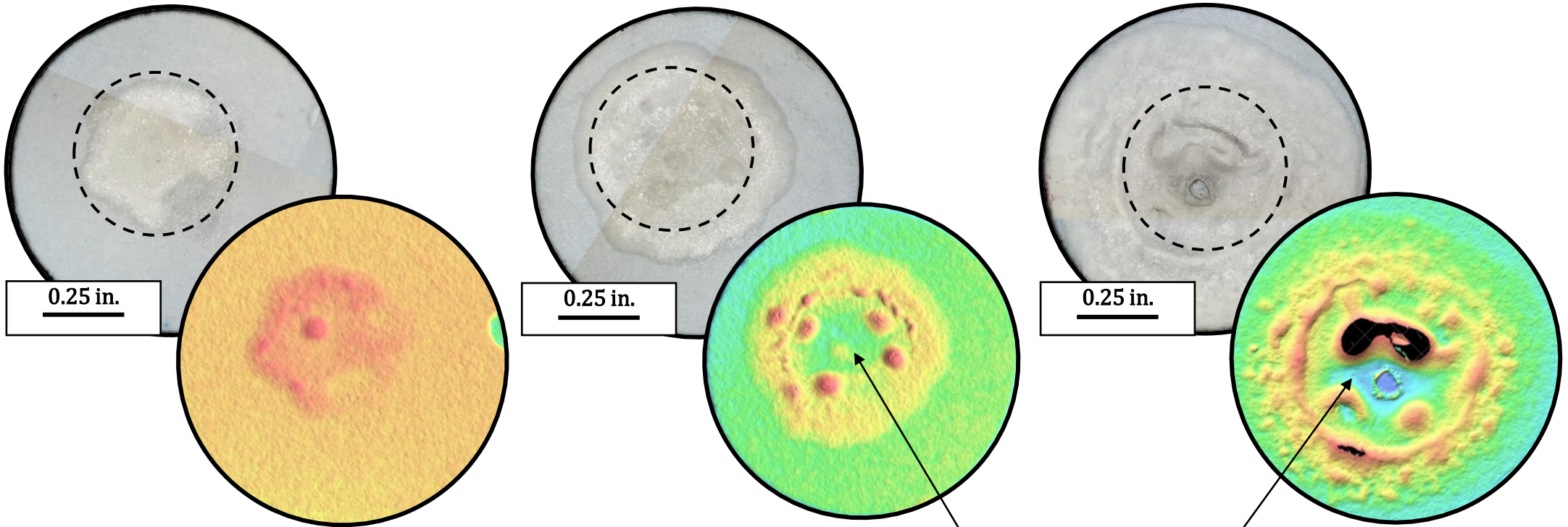
Results - $\sim 6 \text{ mg/cm}^2$, $\sim 18 \text{ mg/cm}^2$, and $\sim 36 \text{ mg/cm}^2$

100 hours

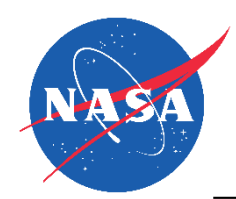
$\sim 6 \text{ mg/cm}^2$

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

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

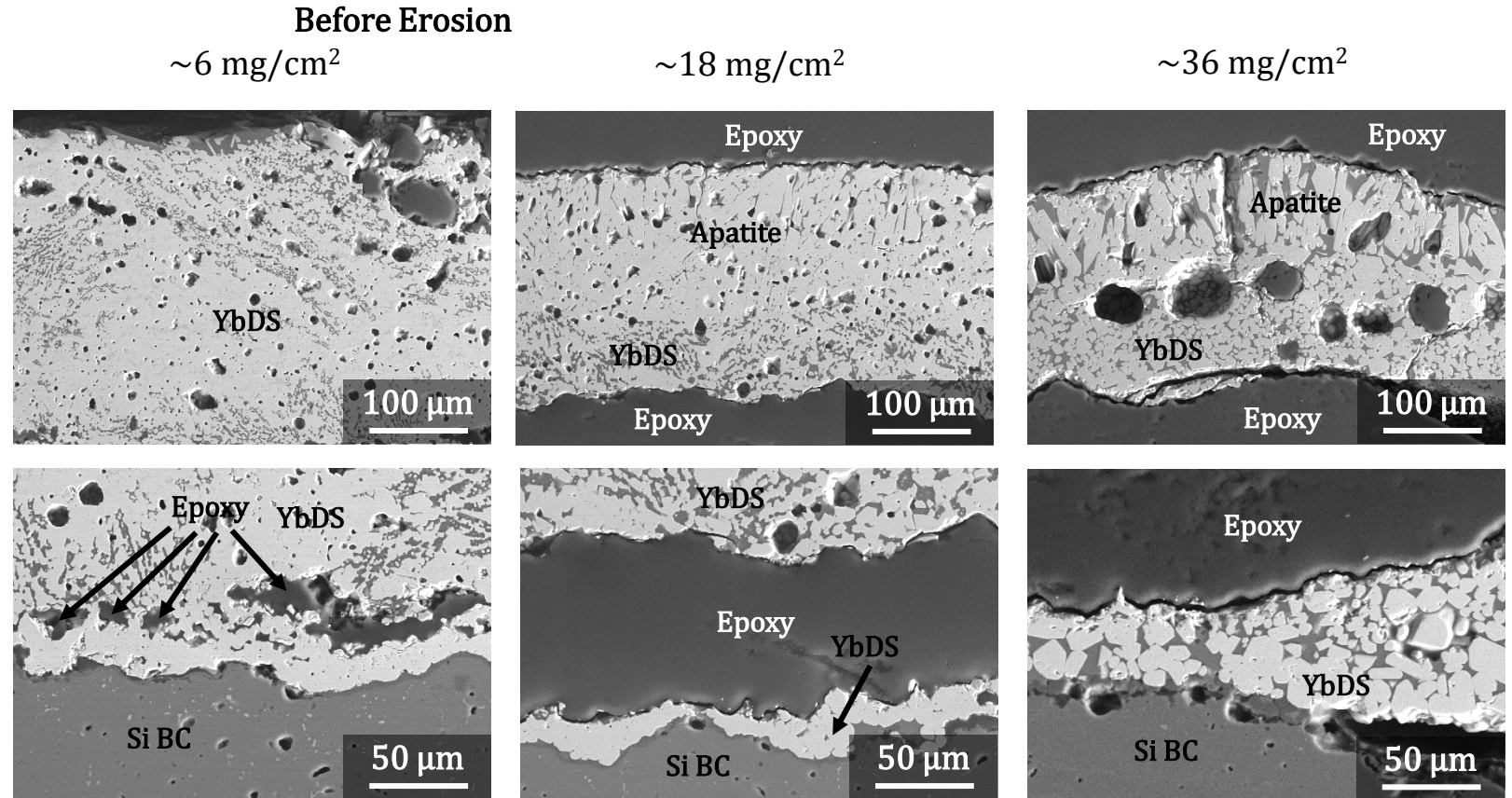


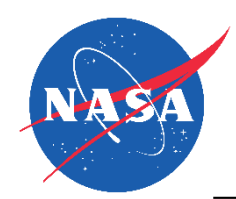
Radial spreading of affected area after heat treatment



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

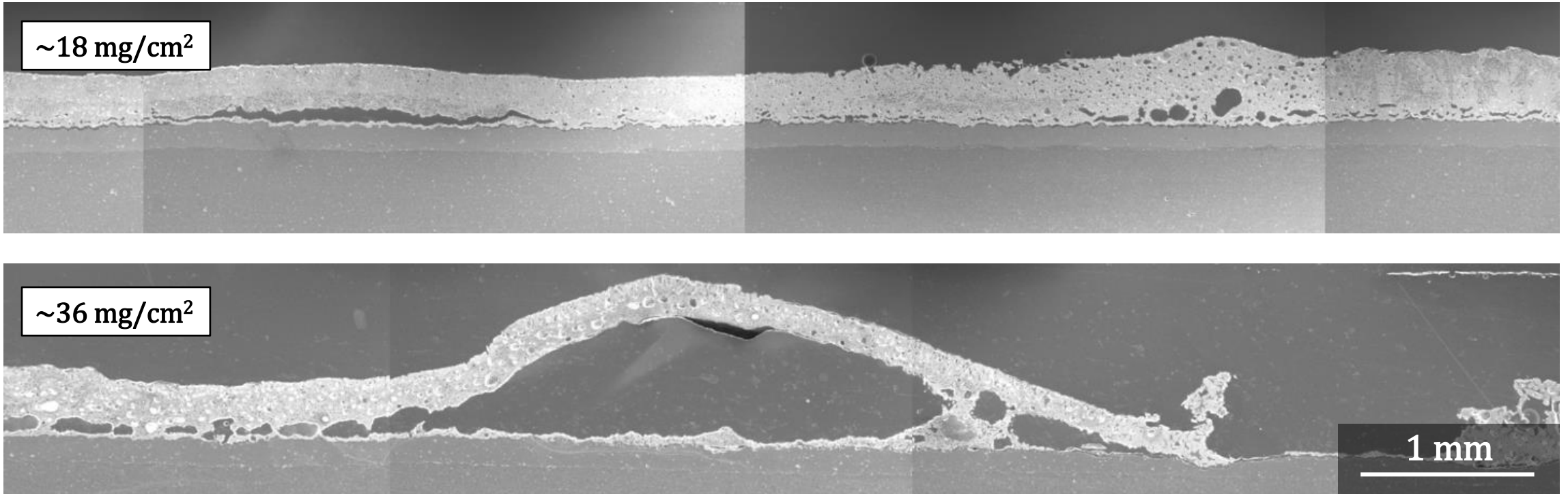
- No residual CMAS on surfaces of coatings
- Apatite and YbDS grains coarsened after 100 hours
- Less apatite grains visible in $\sim 6 \text{ mg/cm}^2$ sample after 100 hours compared to shorter exposure
- Large void formation at bond coat interface and delamination of coating
- Pockets of CMAS between grains at bondcoat interface have grown in size



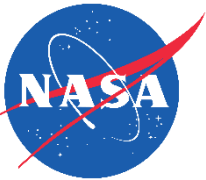


Results – $\sim 6 \text{ mg/cm}^2$, $\sim 18 \text{ mg/cm}^2$, and $\sim 36 \text{ mg/cm}^2$

Before Erosion



- SEM cross sections show bubbling and rumpling of residual CMAS due to longer furnace heat treatment time



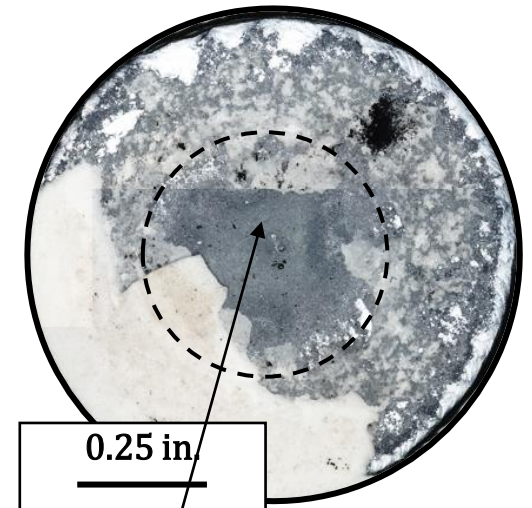
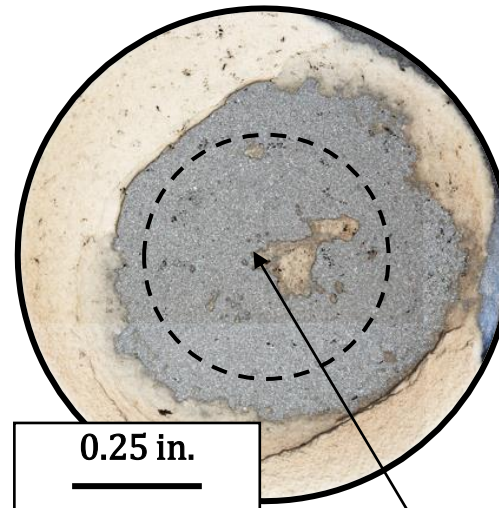
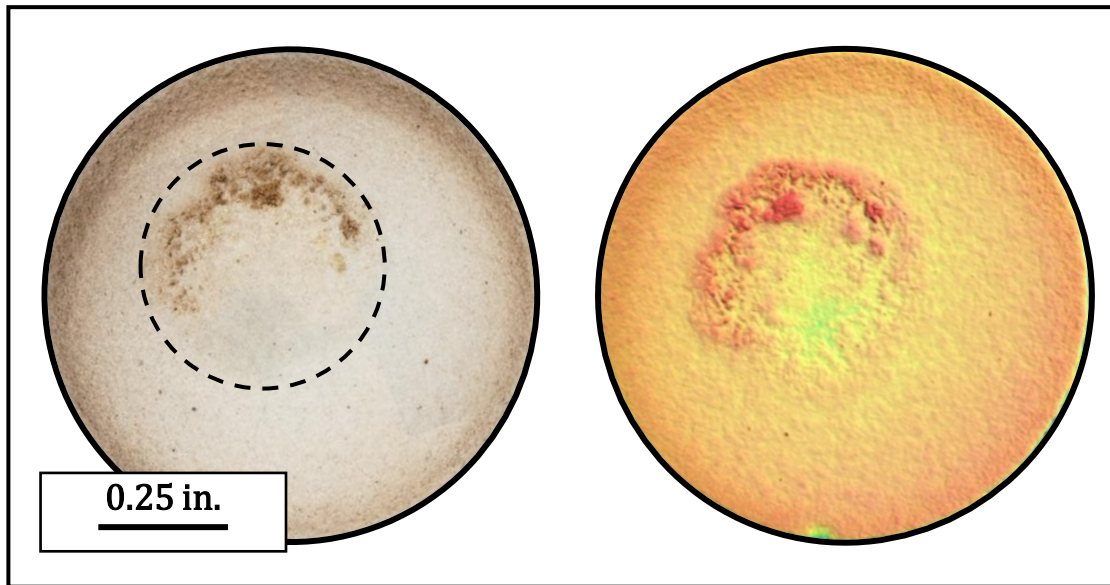
Results - $\sim 6 \text{ mg/cm}^2$, $\sim 18 \text{ mg/cm}^2$, and $\sim 36 \text{ mg/cm}^2$

After Erosion

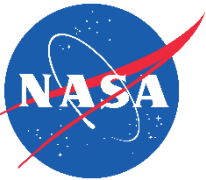
$\sim 6 \text{ mg/cm}^2$

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

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



Catastrophic failure upon heating in burner rig



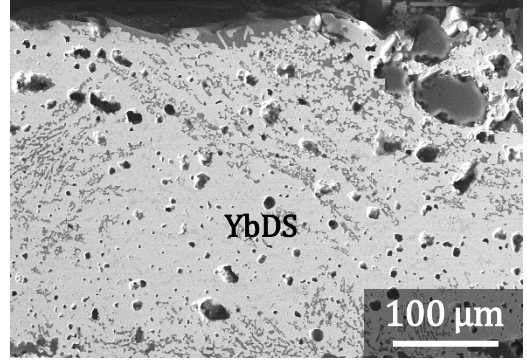
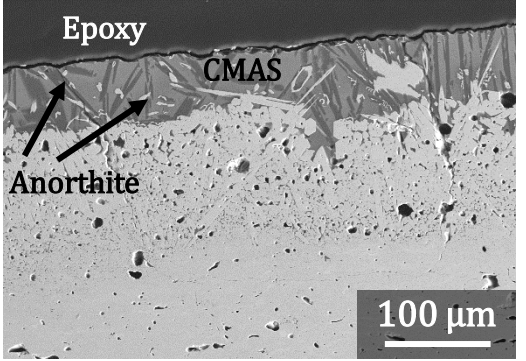
Results - $\sim 6 \text{ mg/cm}^2$, $\sim 18 \text{ mg/cm}^2$, and $\sim 36 \text{ mg/cm}^2$

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

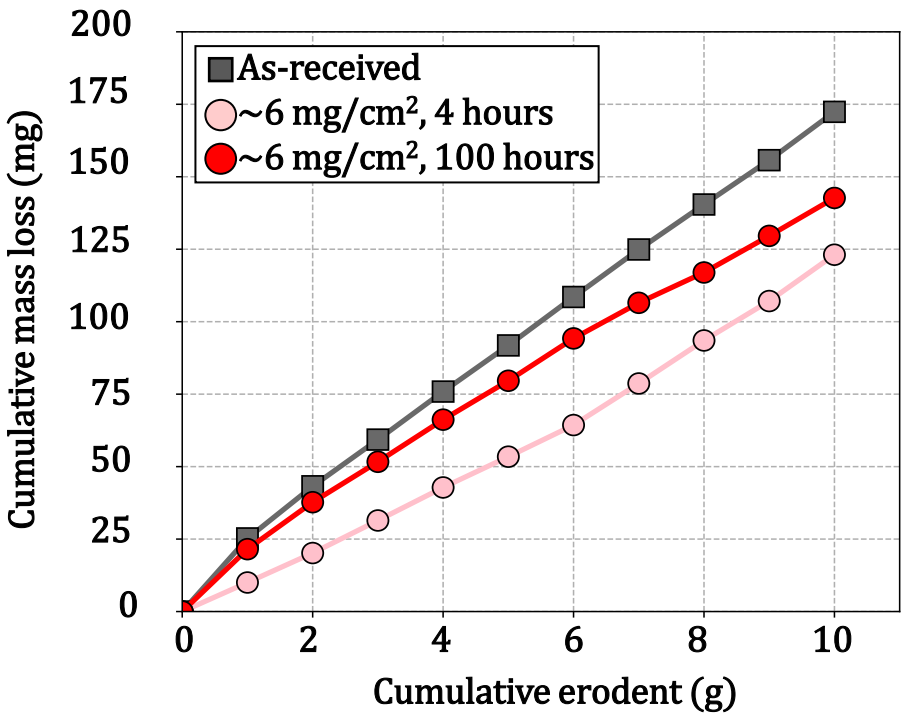
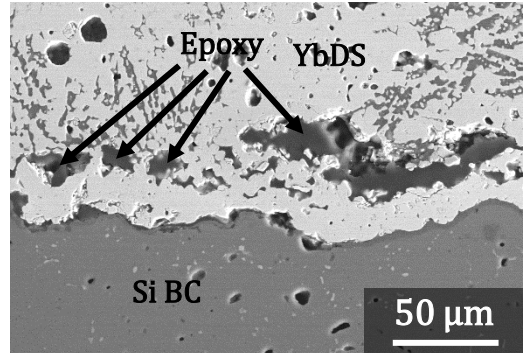
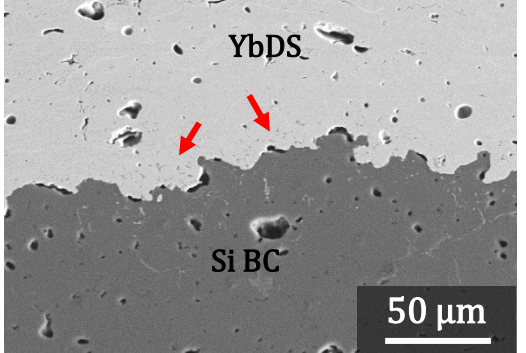
4 hours

100 hours

EBC



Bondcoat Interface

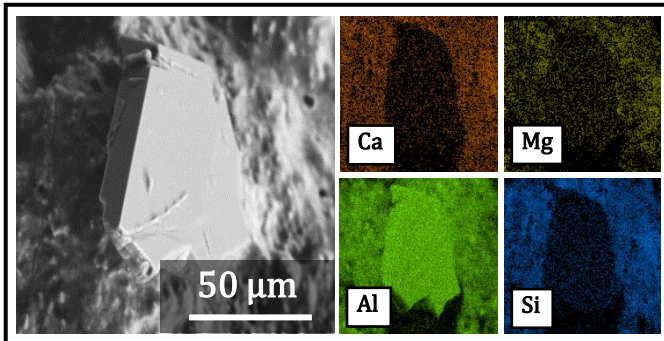


- With increasing exposure time at $\sim 6 \text{ mg/cm}^2$, there was cumulative mass loss increase.

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

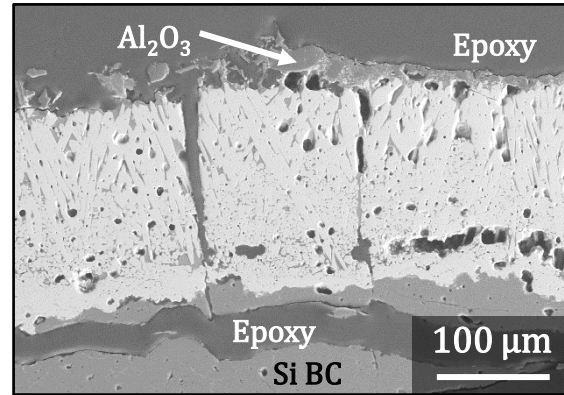
- Additional analyses revealed greater changes in the coating chemistry and morphology, which are not captured in mass loss plots and could be detrimental to the coatings in service.

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

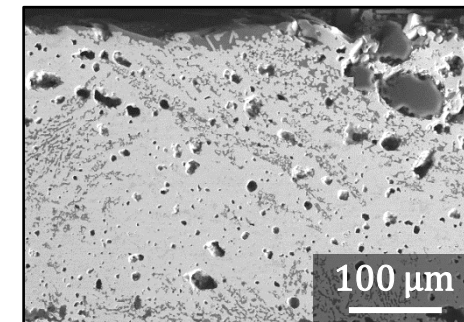
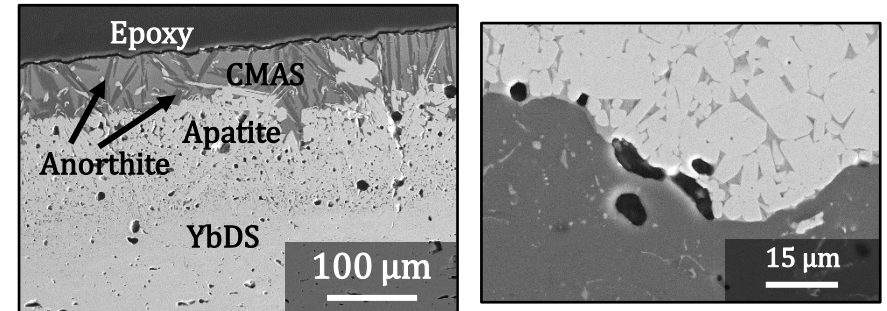
Catastrophic mass loss/coating failure



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

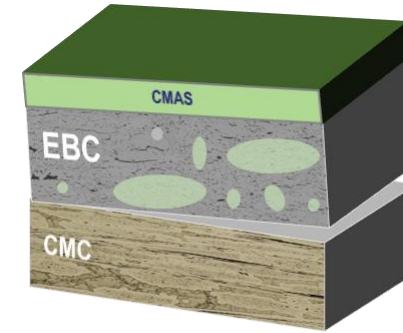
- Thermal shock and thermal expansion mismatch

Morphological changes affecting mechanical durability

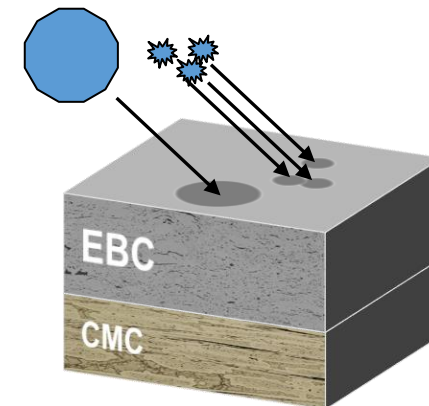


- Void and porosity formation from CMAS interactions
- Differences in the mechanical properties (fracture toughness, elastic modulus, hardness) of reaction products will affect durability

- 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.
 - 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.
 - Higher loading at longer heat treatment time led to catastrophic failure of the coatings upon heating in the burner rigs
 - Tests with lower loadings would be more representative in investigating long term degradation synergies between these two damage mechanisms
- Tracking changes in the chemistry and morphology of EBCs will be crucial in understanding the mechanisms of degradation



Molten Silicate Attack and Infiltration



Erosion and FOD