Calcium-Magnesium Aluminosilicate (CMAS) Interactions with Advanced Environmental Barrier Coating Material

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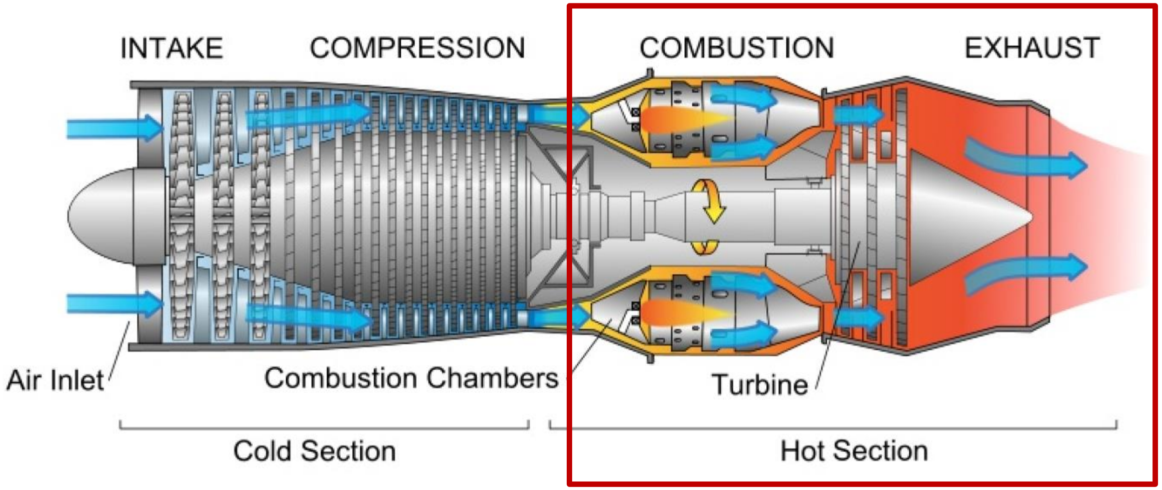
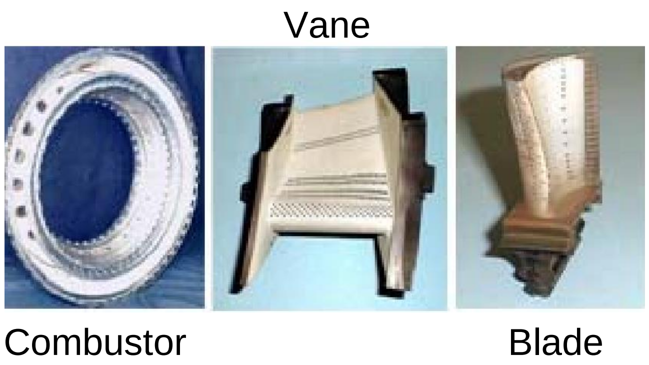
Environmental Barrier Coatings for Ceramic Matrix Composites

- Improve air-breathing turbine efficiency by replacing metal-based components with ceramic matrix composites (CMCs)
- Environmental barrier coatings (EBCs) protect CMC components from oxidation and corrosion in hot section of gas turbine engines
  - Rare-earth silicates



Target: 1482ºC



D. Zhu et al., "EBCs for Turbine Engines," *NTRS* (2009).

Molten CMAS Damage to Protective Coatings

- Particulates (i.e. sand, volcanic ash) ingested by engine melt into **Calcium-Magnesium-AluminoSilicate (CMAS)** glass above 1200ºC
- Molten CMAS degrade EBCs

Eyjafjallajökull volcano eruption in Iceland (2010)

Dust storm in Phoenix, Arizona (2014)

➢ **Need EBC materials resistant to CMAS glass attack above >1200ºC**

USA Today, "Massive dust storm sweeps through Phoenix" (2014)

High-Temperature Interactions between EBC Material and CMAS Glass

Objective:

- Evaluate thermo-chemical interactions between yttrium disilicate ($Y_2Si_2O_7$) EBC material and a desert sand glass at temperatures 1200ºC-1500ºC

- **Yttrium Disilicate ($Y_2Si_2O_7$)**
  - Comparable coefficient of thermal expansion to silicon-based CMCs
  - Water vapor resistance

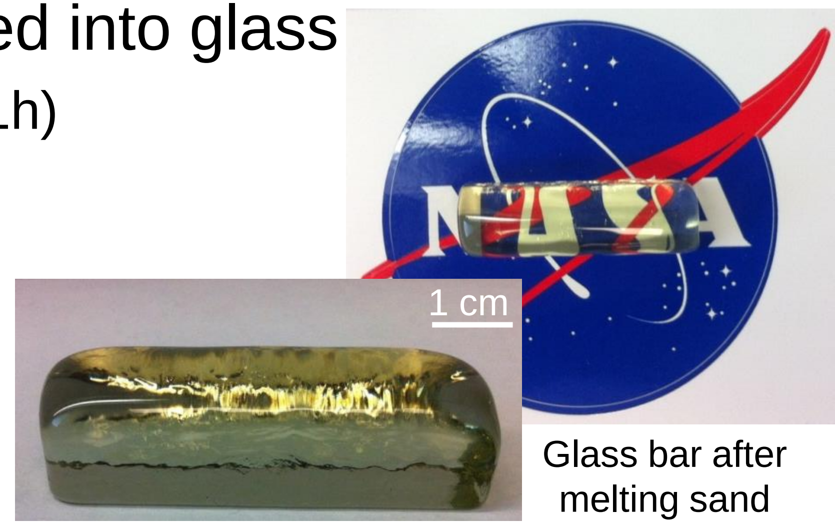
- **Desert Sand (CMAS) Glass**
  - Actual sand sample
  - Relevant CMAS composition to aviation

Aircraft engine ingests sand upon take-off

[1] N.S. Jacobson, Journal of the American Ceramic Society, **97**[6] 1959-1965 (2014).
[2] N.P. Bansal, S.R. Choi, Ceramics International, **41**[3, Part A] 3901-3909 (2014).

Preparation of Desert Sand Glass

- As-received desert sand melted into glass
  - Heated at 10ºC/min to 1550ºC (1h)
  - Quenched melt in water
  - Grind glass frit in planetary mill zirconia milling media
  - Pass through sieve (<297 µm)



Glass bar after melting sand

- Chemical analysis of glass by inductively coupled plasma atomic emission spectrometry (ICP-AES)

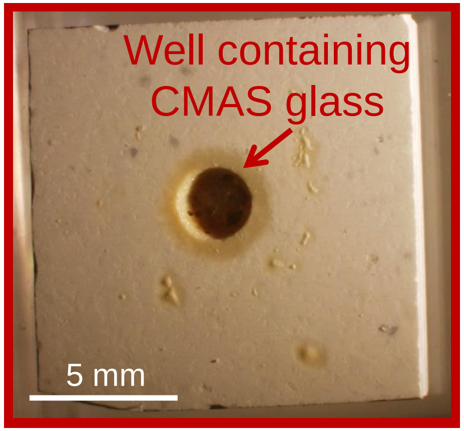
Composition (mol.%)	CaO	MgO	Al_2O_3	SiO_2	K_2O	Fe_2O_3
Desert sand glass	**27.8**	**4**	**5**	**61.6**	**1**	**0.6**
Common CMAS glass[1,2]	33	9	13	45	-	-

- Desert sand glass comprised of CMAS and trace oxides

[1] S. Krämer, J. Yang, C.G. Levi, C.A. Johnson, Journal of the American Ceramic Society, 89 (2006) 3167-3175.
[2] B.J. Harder, J. Ramìrez-Rico, J.D. Almer, K.N. Lee, K.T. Faber, Journal of the American Ceramic Society, 94 (2011) s178-s185.

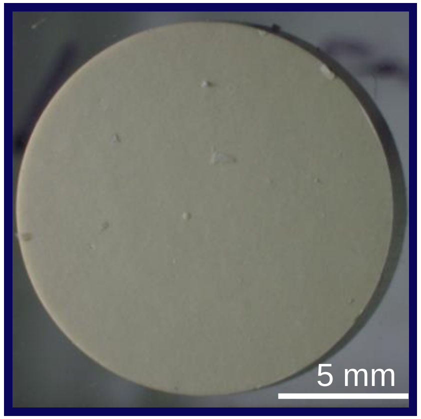
Evaluate sand (CMAS) glass interactions with $Y_2Si_2O_7$ material

- **CMAS glass on hot-pressed $Y_2Si_2O_7$ substrate**
  - Load substrate with CMAS glass ~35 mg/cm^2
  - 20h heat treatments at 1200ºC, 1300ºC, 1400ºC and 1500ºC in air
  - Evaluate microstructure and composition of $Y_2Si_2O_7$/CMAS glass interface with SEM/EDS and EPMA



$Y_2Si_2O_7$ substrate loaded with CMAS glass after heat treatment

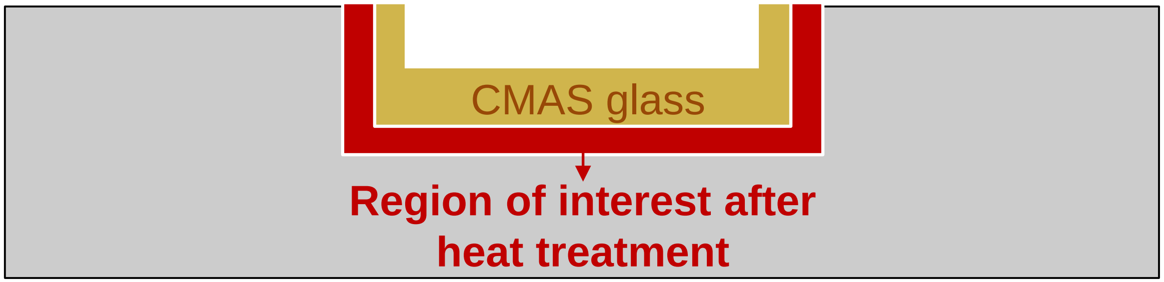
- **Cold-pressed pellet of $Y_2Si_2O_7$ and CMAS glass**
  - 80 wt.% $Y_2Si_2O_7$, 20 wt.% CMAS glass
  - 20h heat treatments at 1200ºC, 1300ºC, 1400ºC and 1500ºC in air
  - Analyze resulting phases using XRD



Cold-pressed $Y_2Si_2O_7$/CMAS glass pellet

CMAS Glass on $Y_2Si_2O_7$ Substrate

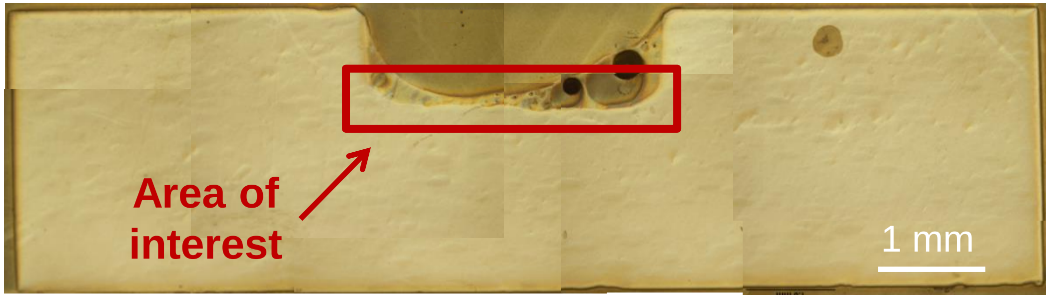
Heat >1200ºC, CMAS glass melts and penetrates/reacts with $Y_2Si_2O_7$ substrate

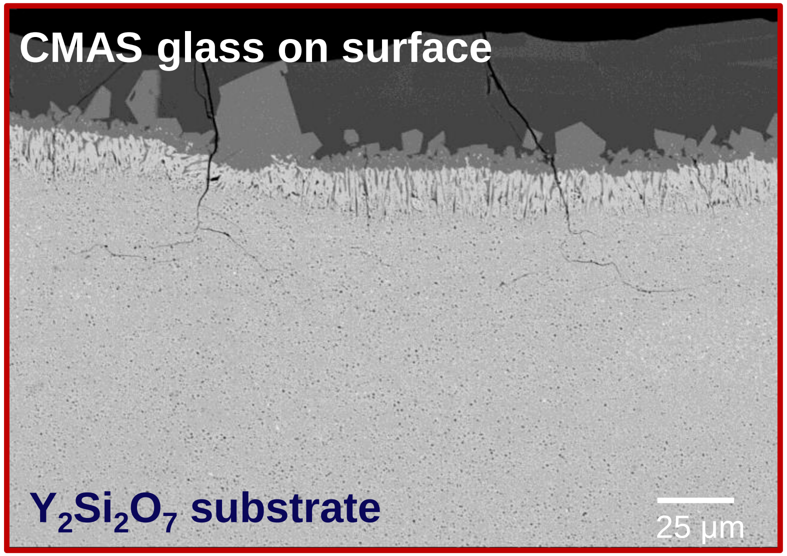


1. CMAS glass infiltration into $Y_2Si_2O_7$ substrate
2. Thermo-chemical interactions of $Y_2Si_2O_7$/CMAS glass

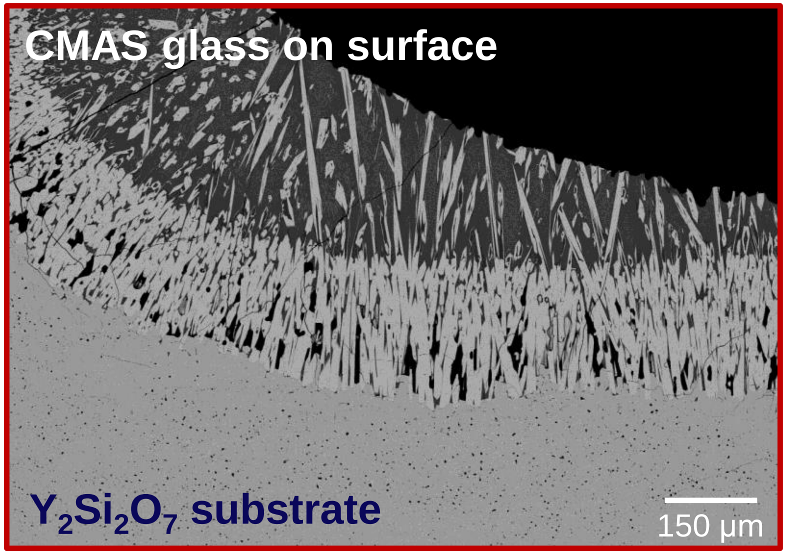
SEM Cross-Section of CMAS/$Y_2Si_2O_7$ substrate

- Scanning electron microscopy (SEM) to evaluate cross-sections of heat treated CMAS glass/$Y_2Si_2O_7$ substrates





1200ºC



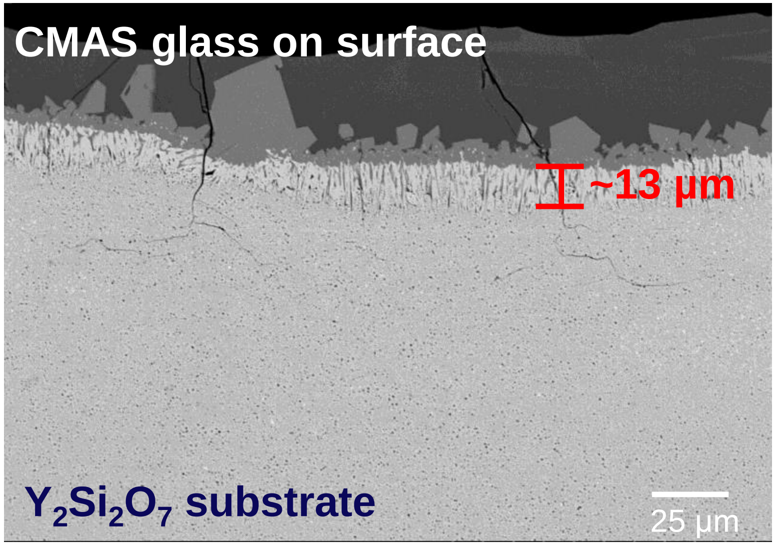
1500ºC

Interface between $Y_2Si_2O_7$ substrate/CMAS glass after heat treatment

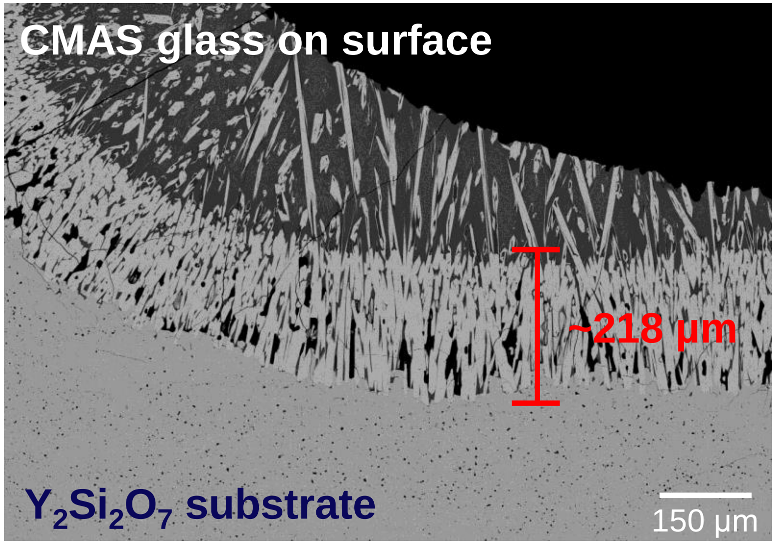
SEM Cross-Section of CMAS/$Y_2Si_2O_7$ substrate

- CMAS glass penetration into $Y_2Si_2O_7$ substrates
  - Infiltration depth increases with temperature

Heat Treatment	Depth of CMAS Infiltration
1200ºC for 20h	12.7 ± 2.5 μm
1300ºC for 20h	80.9 ± 14.2 μm
1400ºC for 20h	215.8 ± 17.6 μm
1500ºC for 20h	217.6 ± 19.6 μm



1200ºC



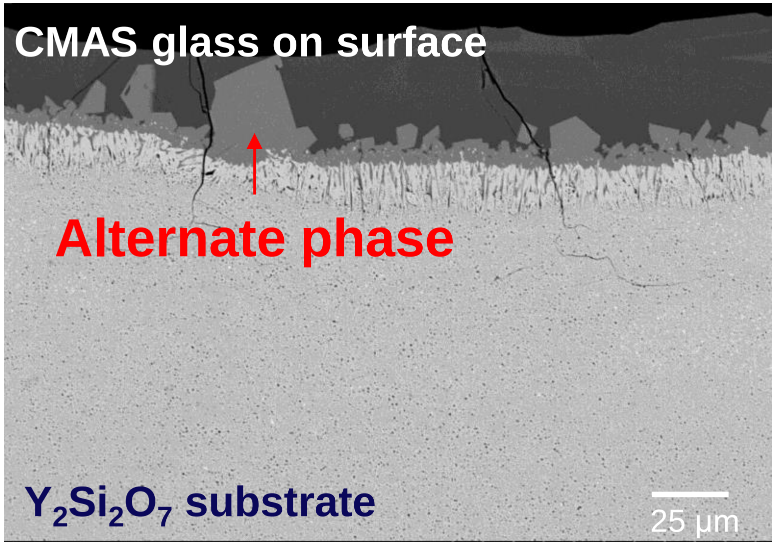
1500ºC

Interface between $Y_2Si_2O_7$ substrate/CMAS glass after heat treatment

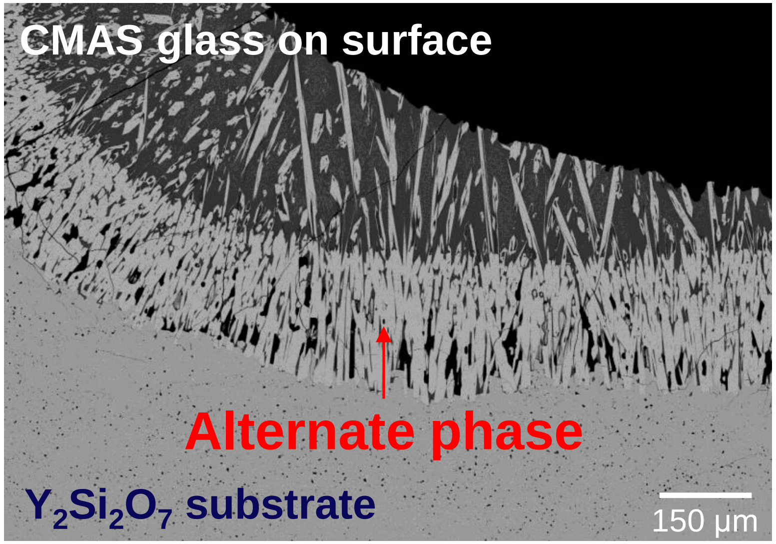
SEM Cross-Section of CMAS/$Y_2Si_2O_7$ substrate

- CMAS glass penetration into $Y_2Si_2O_7$ substrates
  - Infiltration depth increases with temperature

- Thermo-chemical interactions
  - Precipitation of alternate phase in CMAS glass and infiltrated region



1200ºC

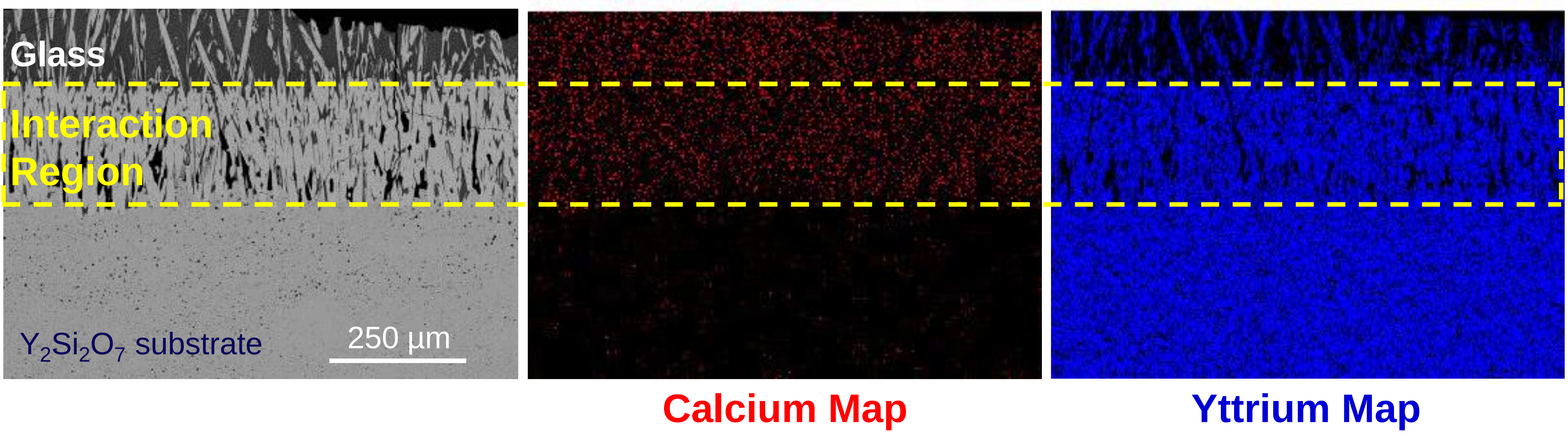


1500ºC

Interface between $Y_2Si_2O_7$ substrate/CMAS glass after heat treatment

EDS Mapping of Interaction Region

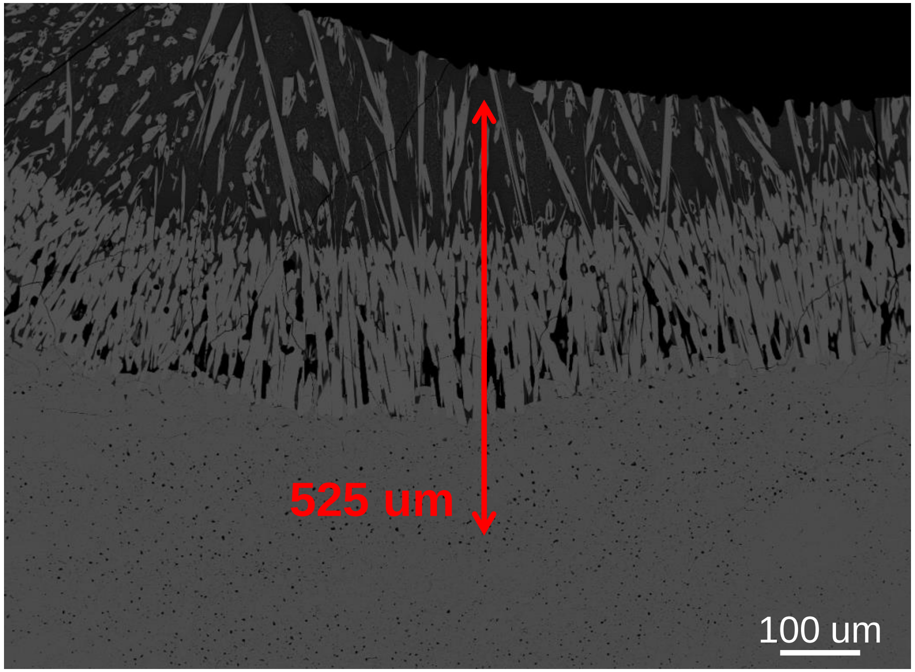
- Yttrium incorporated into CMAS glass
  - Yttrium signal detected above substrate surface in glass
  - $Ca_2Y_8(SiO_4)_6O_2$ oxyapatite silicate phase expected
- Calcium infiltrated $Y_2Si_2O_7$ substrate
  - Depth of calcium infiltration corresponds to microstructural deformation in interaction region



Interface between $Y_2Si_2O_7$ substrate and CMAS glass after 20h heat treatment at 1500ºC

Quantification of Composition by EPMA

- Electron probe micro-analysis (EPMA)
  - Evaluate composition along line normal to substrate surface



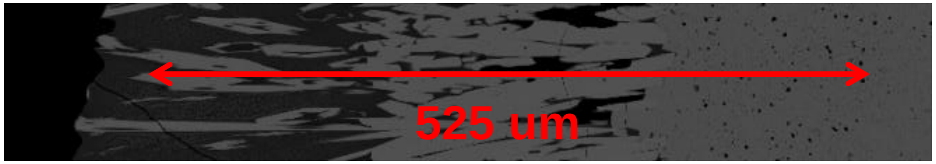
BSE image of $Y_2Si_2O_7$ substrate and CMAS glass after 20h heat treatment at 1500ºC

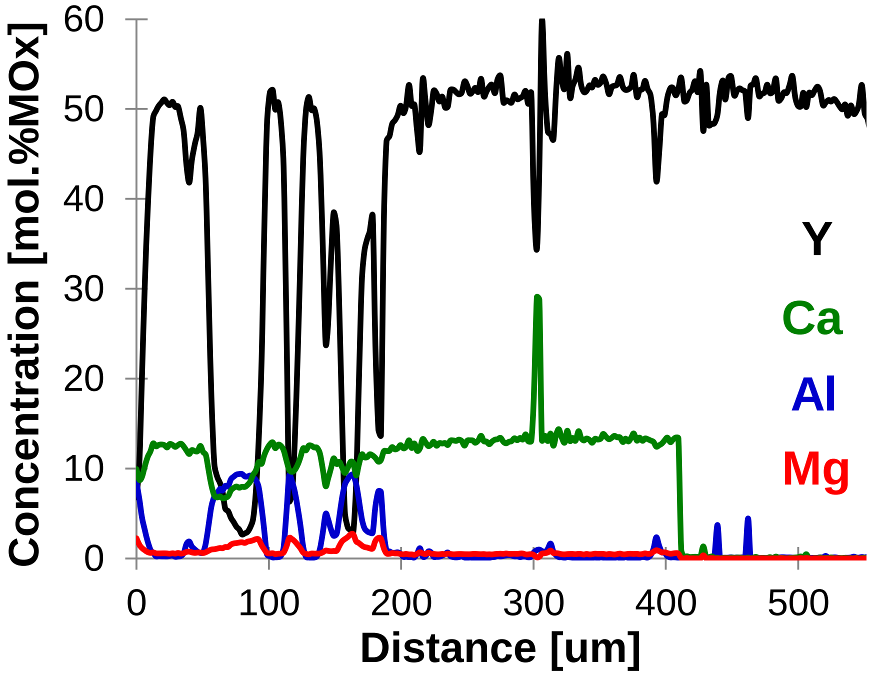
Quantification of Composition by EPMA

- Electron probe micro-analysis (EPMA)
  - Evaluate composition along line normal to substrate surface
  - Quantify variation in elemental composition from CMAS glass through $Y_2Si_2O_7$ substrate after various heat treatments

- Ca detected throughout CMAS glass and interaction region
- No Ca in substrate
- Minimal Al or Mg in interaction region

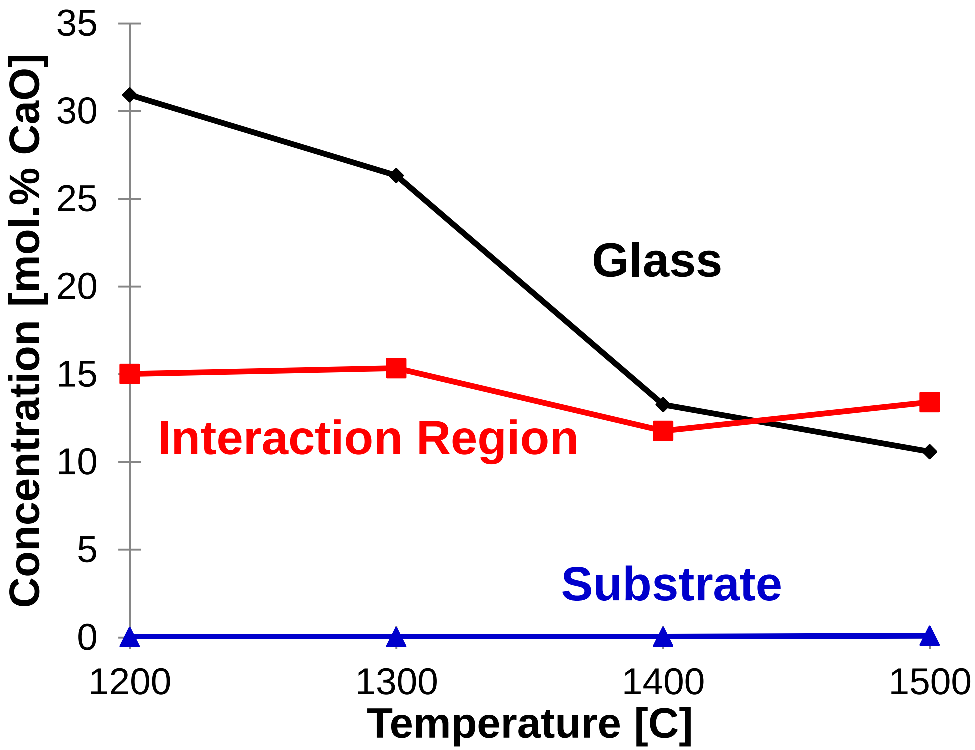
➢ Compare Ca content in specimens heat treated at different temperatures

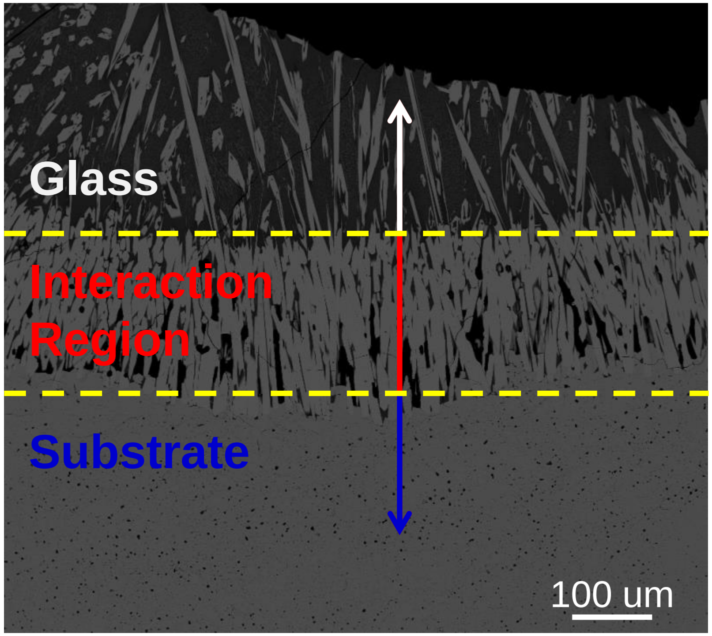




Average CaO Content by EPMA

- CaO content in glass decreases with temperature
- CaO content in interaction region constant
  - Depth of interaction region increases with temperature
- No CaO detected in substrate



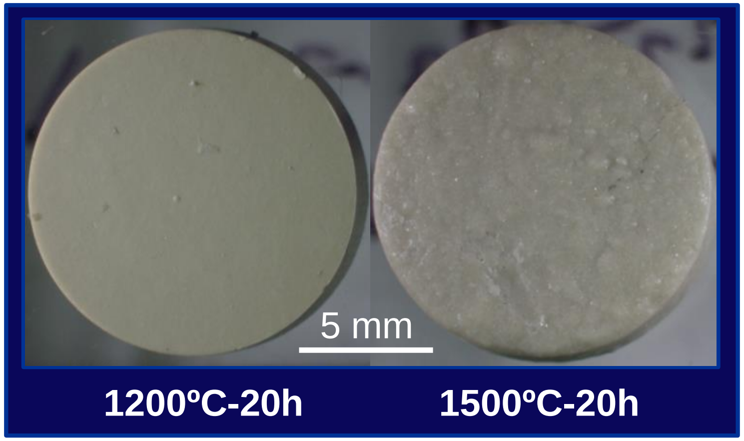


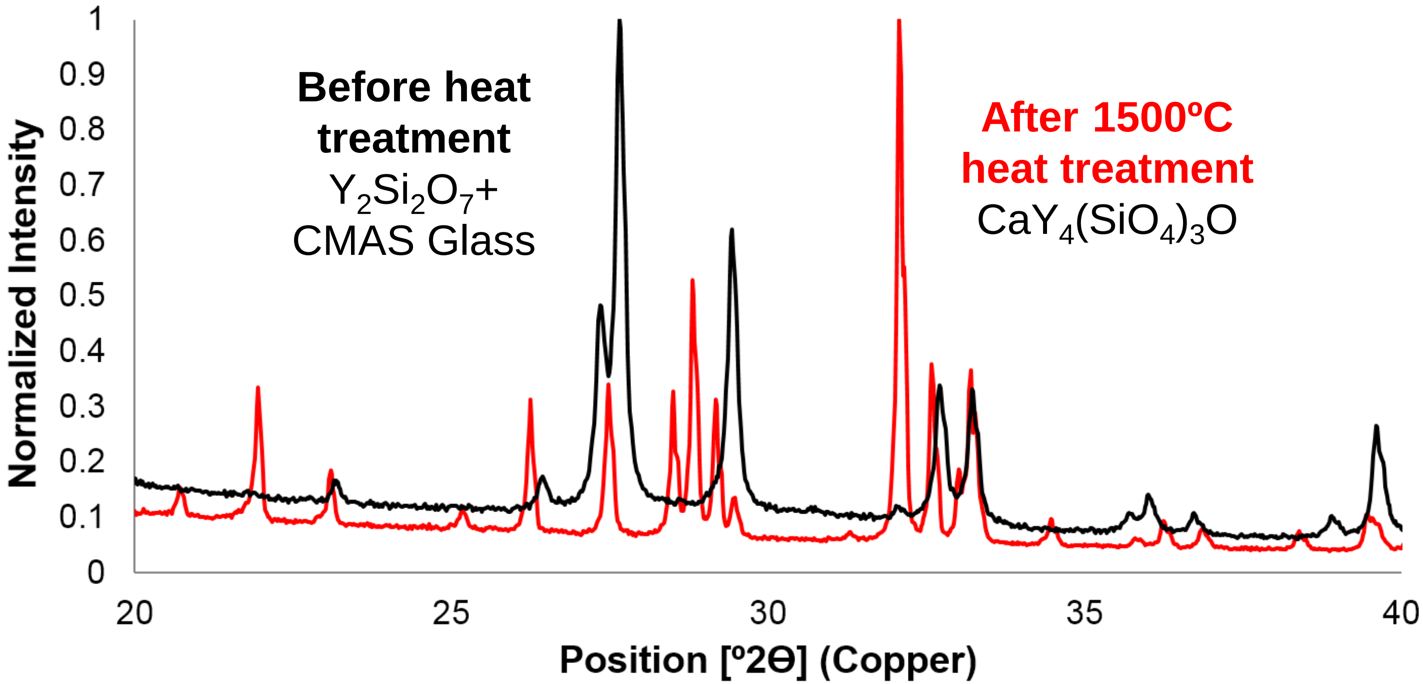
BSE image of CMAS glass/$Y_2Si_2O_7$ substrate after 1500ºC-20h

Identifying Alternate Phase using XRD

- Heat treat powder pellets containing 80 wt.% EBC powder ($Y_2Si_2O_7$) and 20 wt.% CMAS glass
- Evaluate reacted pellet using X-ray diffraction (XRD)

Alternate phase: $Ca_2Y_8(SiO_4)_6O_2$ oxyapatite silicate phase





Conclusions and Current Efforts

- Desert sand (CMAS) glass reacted with $Y_2Si_2O_7$ yielding $Ca_2Y_8(SiO_4)_6O_2$ oxyapatite silicate phase
  - Formed by dissolution of $Y_2Si_2O_7$ in CMAS glass followed by precipitation during cooling
  - Similar reaction observed for Y_2SiO_5
- Depth of CMAS infiltration increased with increasing heat treatment temperature
  - More significant pore formation and microstructural deformation in interaction region compared with Y_2SiO_5

➢ Evaluate other advanced EBC materials' high-temperature interaction with desert sand (CMAS) glass