Advanced Thermal Barrier Coatings for Next Generation Gas-Turbine Engines Fueled by Coal-Derived Syngas

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Support
DoE NETL University Coal Research Program

DoE Project Officer
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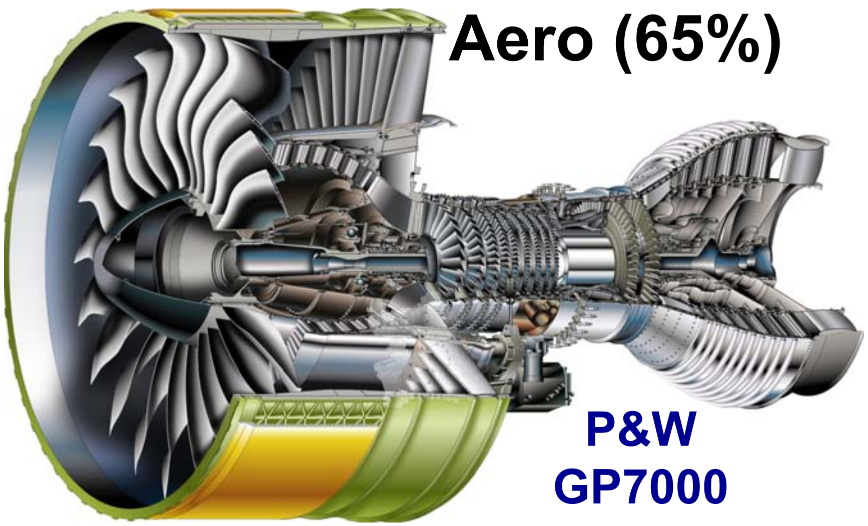
Grant No.
DE-FE0008933

Outline

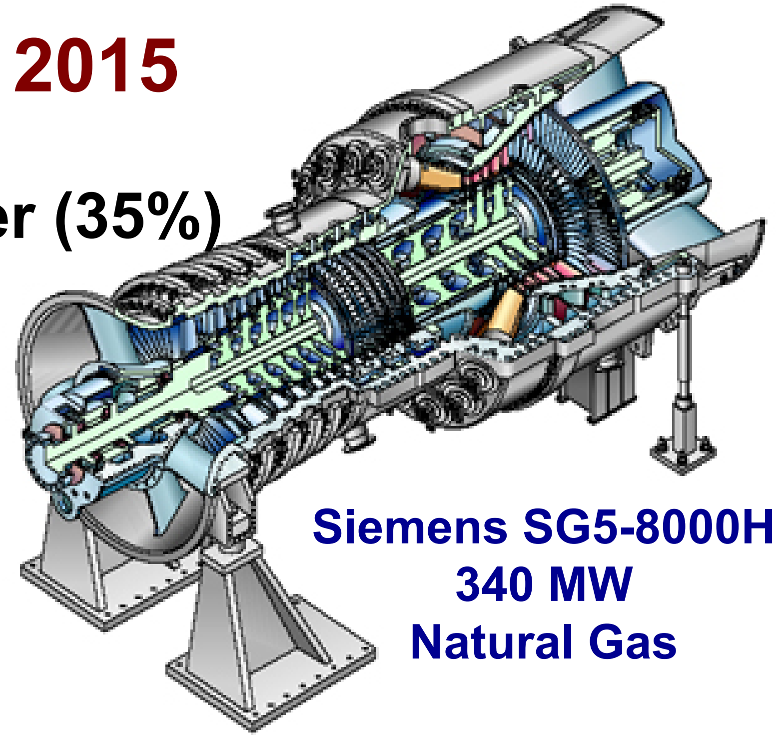
* Thermal Barrier Coatings (TBCs)
    - Background and Motivation (Supported by ONR and DoE UTSR)
    - Molten Deposits: Calcium-Magnesium-Alumino-Silicate (CMAS)
        > Sand, Volcanic Ash, Fly Ash
    - Damage to Air-Plasma Sprayed (APS) 7YSZ TBCs by Molten CMAS
        > Understanding Mechanisms
    - Damage Mitigation via TBCs Composition Engineering
        > APS 7YSZ TBCs with Al^{3+} and Ti^{4+} in Solid Solution
        > APS $Gd_2Zr_2O_7$ TBCs
* Porous TBC Ceramics/CMAS Interactions: "Model" Studies
    - Effect of "Solute" Type and Concentration in ZrO_2
    - Guidelines for Design of Future CMAS-Resistant TBCs
* APS $2ZrO_2 \cdot Y_2O_3$ (48YSZ) TBCs
    - New Coatings from Commercially Developed Powders
        > CMAS Interaction Studies
        > Graded 48YSZ-7YSZ TBCs
* Ongoing and Future Work
* Outlook

Gas-Turbine Engines: $55B by 2015

Aero (65%) **Power (35%)**



P&W
GP7000



Siemens SG5-8000H
340 MW
Natural Gas

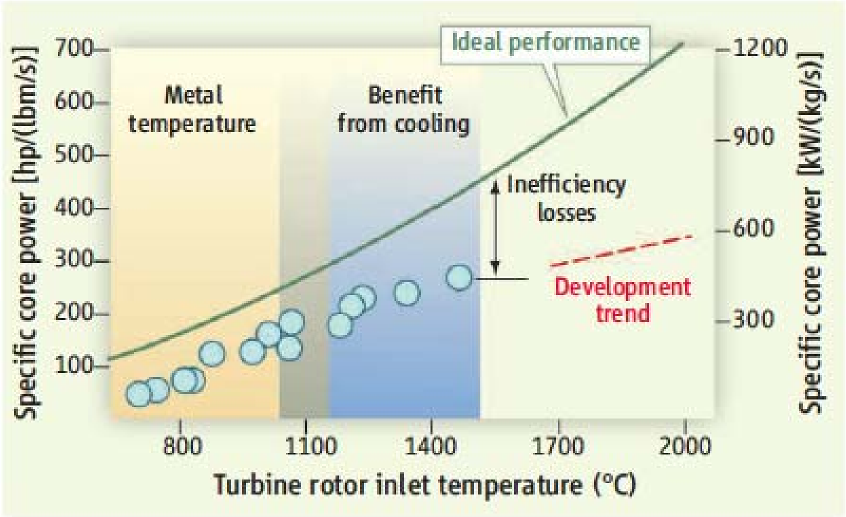
Langston, 2011

Motivation

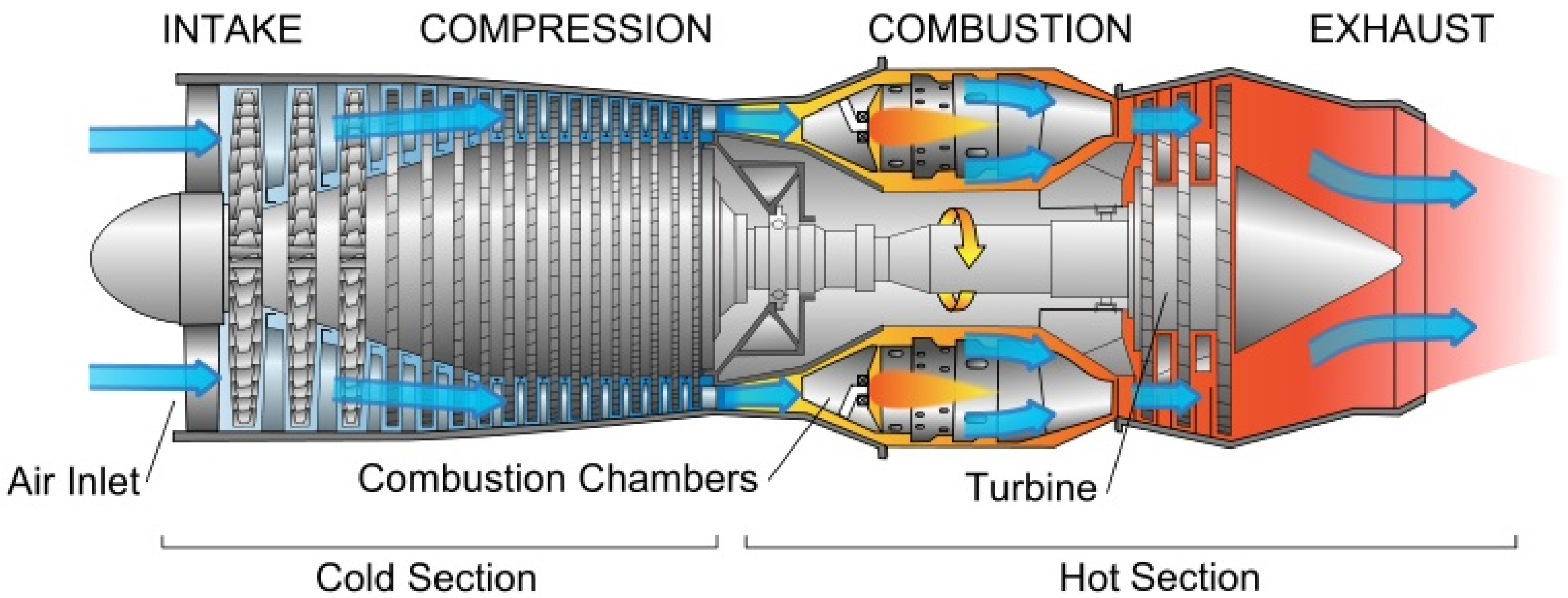
* Need for Higher Power and Efficiency
    - Aircraft Propulsion
    - Electricity Generation

* Need for Higher Hot-Section Temps.

* Materials "Bottleneck":
    - Improved Structural Alloys
    - Ceramic Matrix Composites
    - Ceramic Thermal Barrier Coatings (TBCs)



Perepezko, 2009



Ceramic Thermal Barrier Coatings (TBCs)

* **Engines**
  - **Aero**
  - **Power**

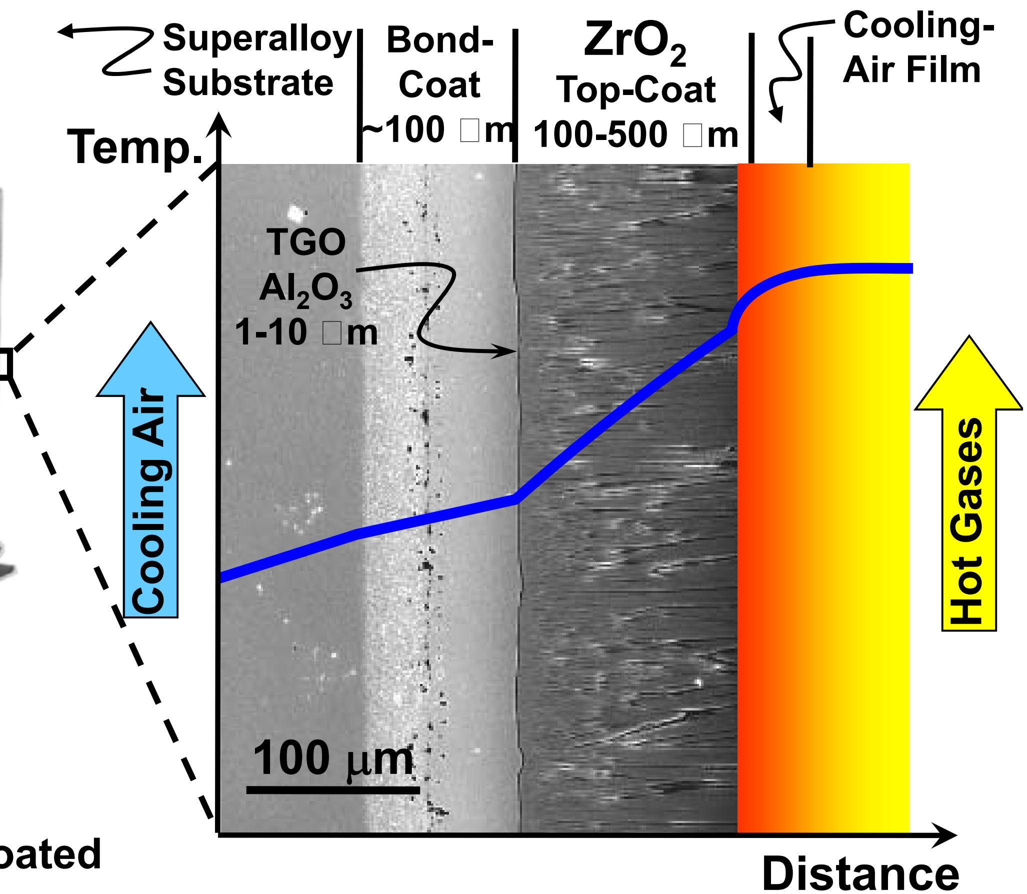
* **Blades/Vanes, Combustors, Shrouds**

* **Strain-Tolerant, Low Th. Cond.**

* **Up to 300 ˚C Temp. Reduction**

* **Improved**
  - **Performance**
  - **Efficiency**
  - **Durability**

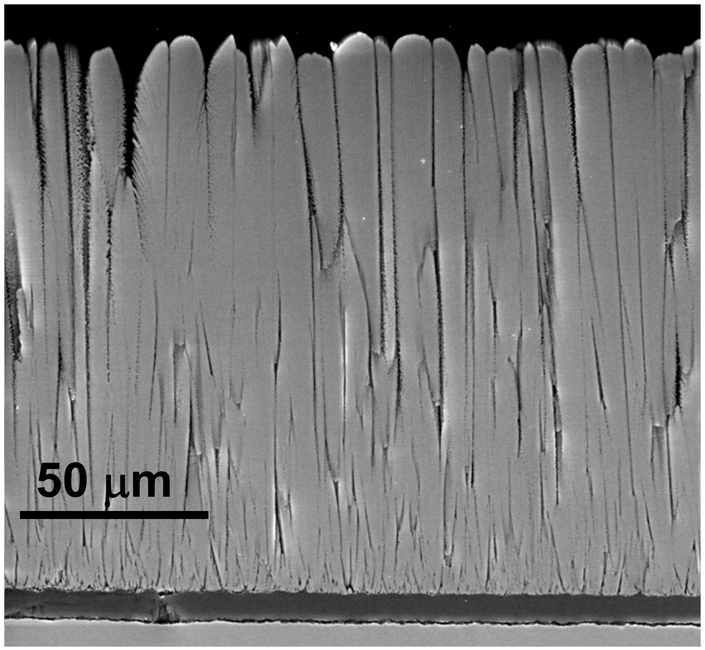
Thermal-Barrier-Coated Turbine Blade



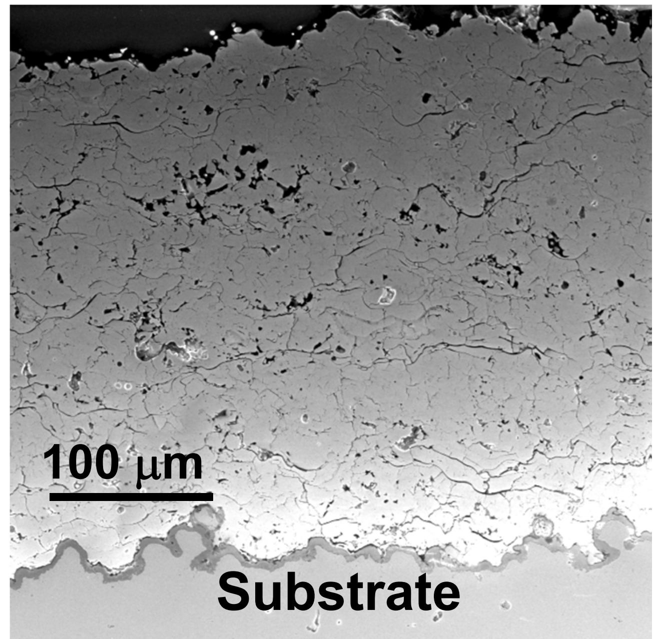
Science, 2002; *MRS Bull.*, 2012

Ceramic TBCs

Electron-Beam Physical Vapor Deposition



Air Plasma Spraying (APS)

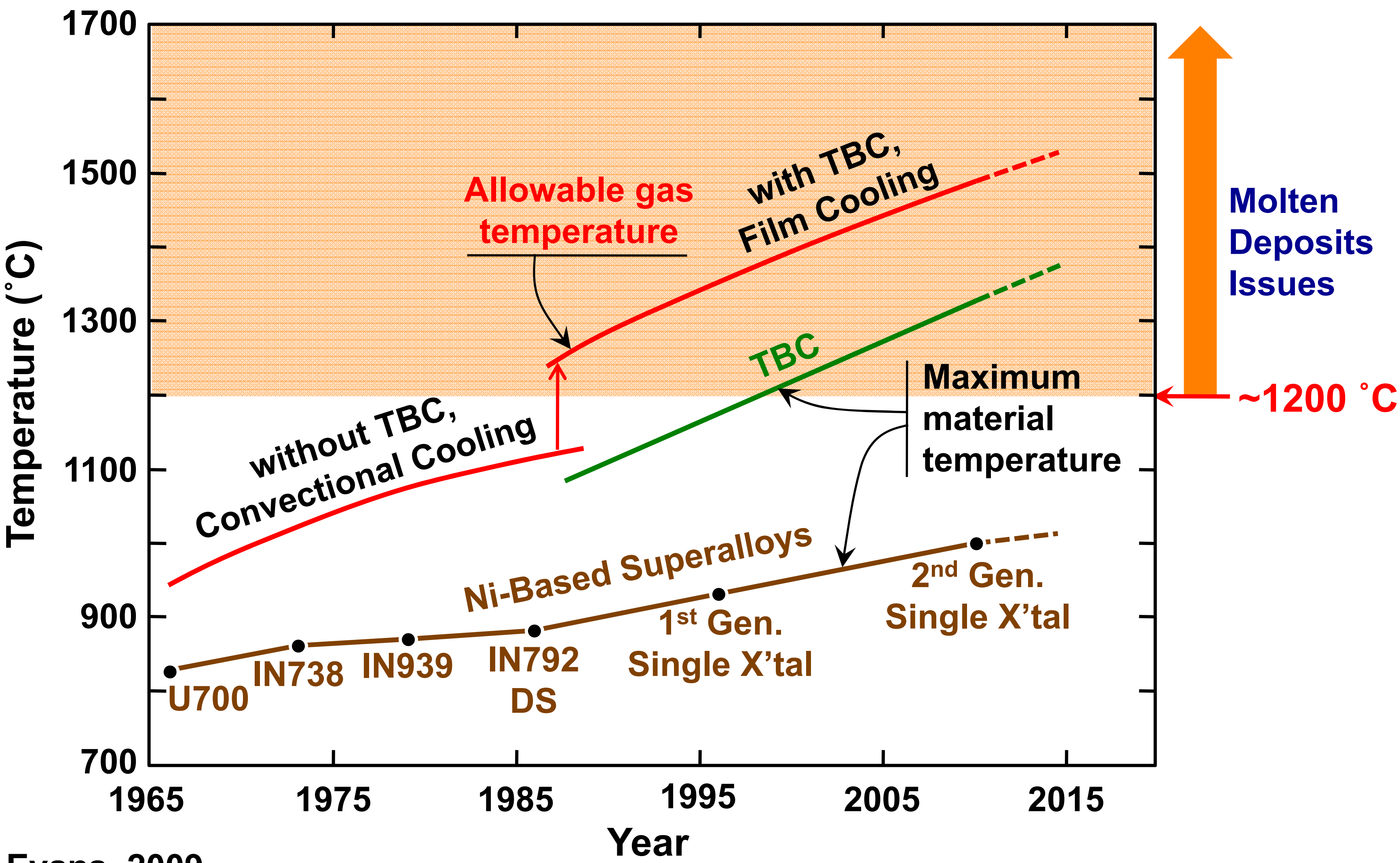


* Low Thermal Conductivity (ZrO_2 + 7 wt% Y_2O_3 Solid Soln.: 7YSZ)

* High Porosity (15 - 20%); Thickness 100 to 500 μm

* "Strain Tolerant" to Accomodate Th. Exp. Mismatch with Metal

Thermal Barrier Coatings (TBCs)



After Evans, 2009

Push for Higher Temperatures => New Materials Issues

Sources of Silicate Deposits in Aero Engines

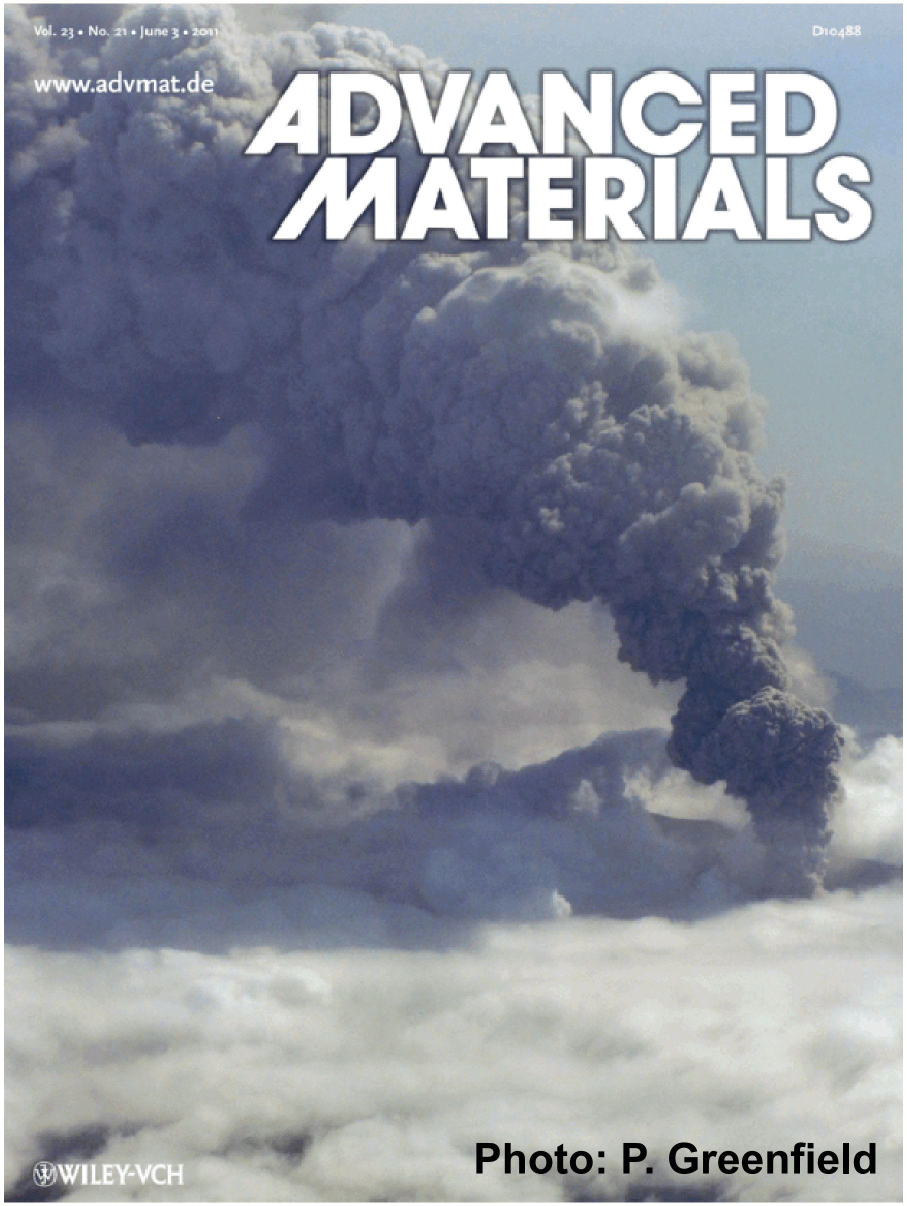
Calcium-Magnesium-Alumino-Silicate (CMAS) Sand

* Sandstorms: ~0.1 mg/m^3
  (Ansmann, *et al*. 2003)
* Ambient: ~0.01 mg/m^3
* Runways: >1.0 mg/m^3 (?)
* Sand Ingested by Engines: 1 to 100 g/h
  (Depends on Engine, Bypass Ratio...)

Can Cause Severe Damage to TBCs and Engines

Sources of Silicate Deposits in Aero Engines

CMAS Volcanic Ash



* Eyjafjallajökull Eruption in Iceland in 2010

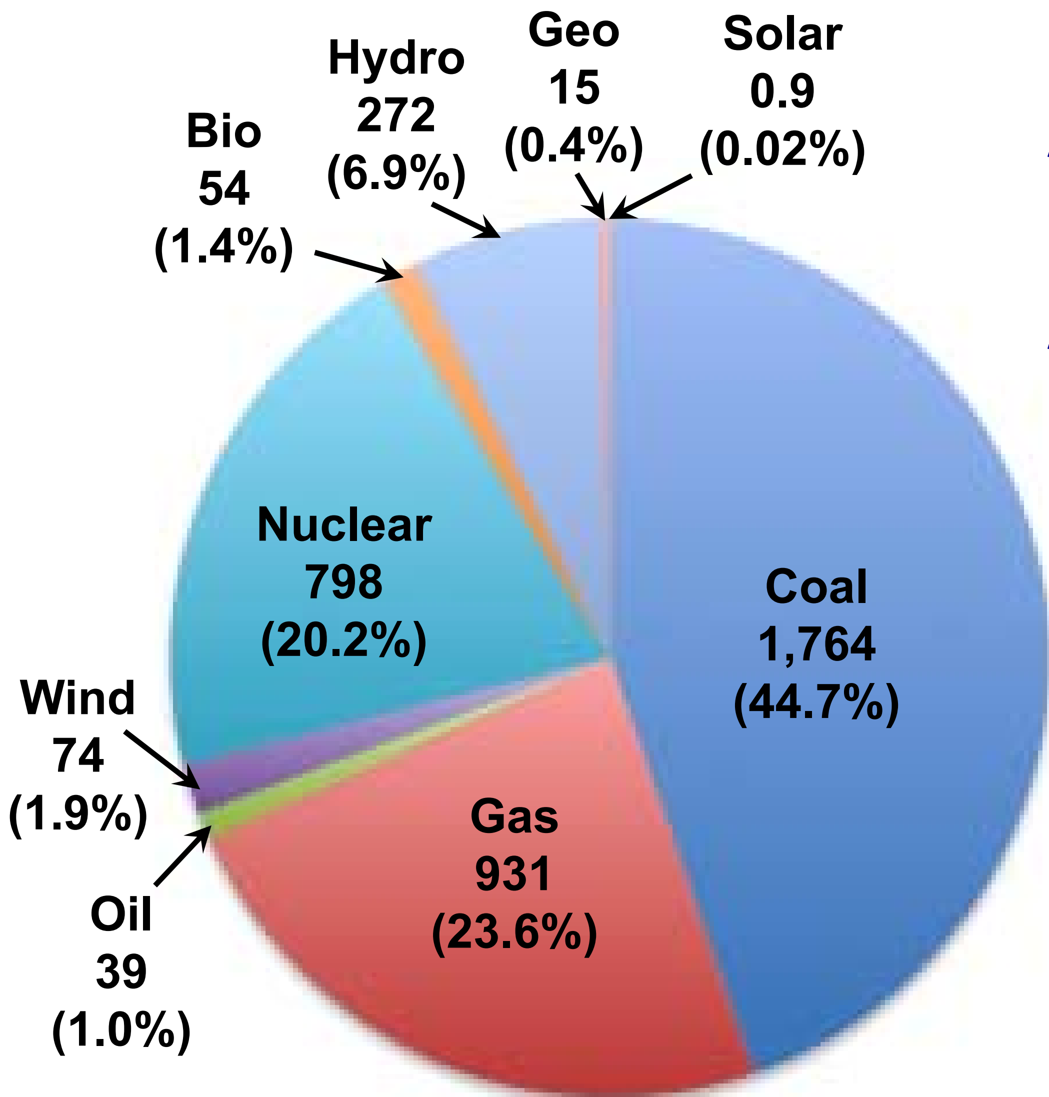
* Shut Down of Vast European Air Space for Several Days

* Economic Loss Approaching $2B

* Conc.: No-Fly Zone: >4.0 mg/m^3
Limited-Fly: 2.0-4.0 mg/m^3
Unrestricted: <2.0 mg/m^3
(Sultana, 2010)

Can Cause Severe Damage to TBCs and Engines

Sources of Silicate Deposits in Power Engines



* USA Electricity Generation by Source in 2009

* USA Total ~3,950 Billion KWh (World ~19,100 B KWh)

Need for Environmentally Responsible, Efficient Way of Using Available Coal

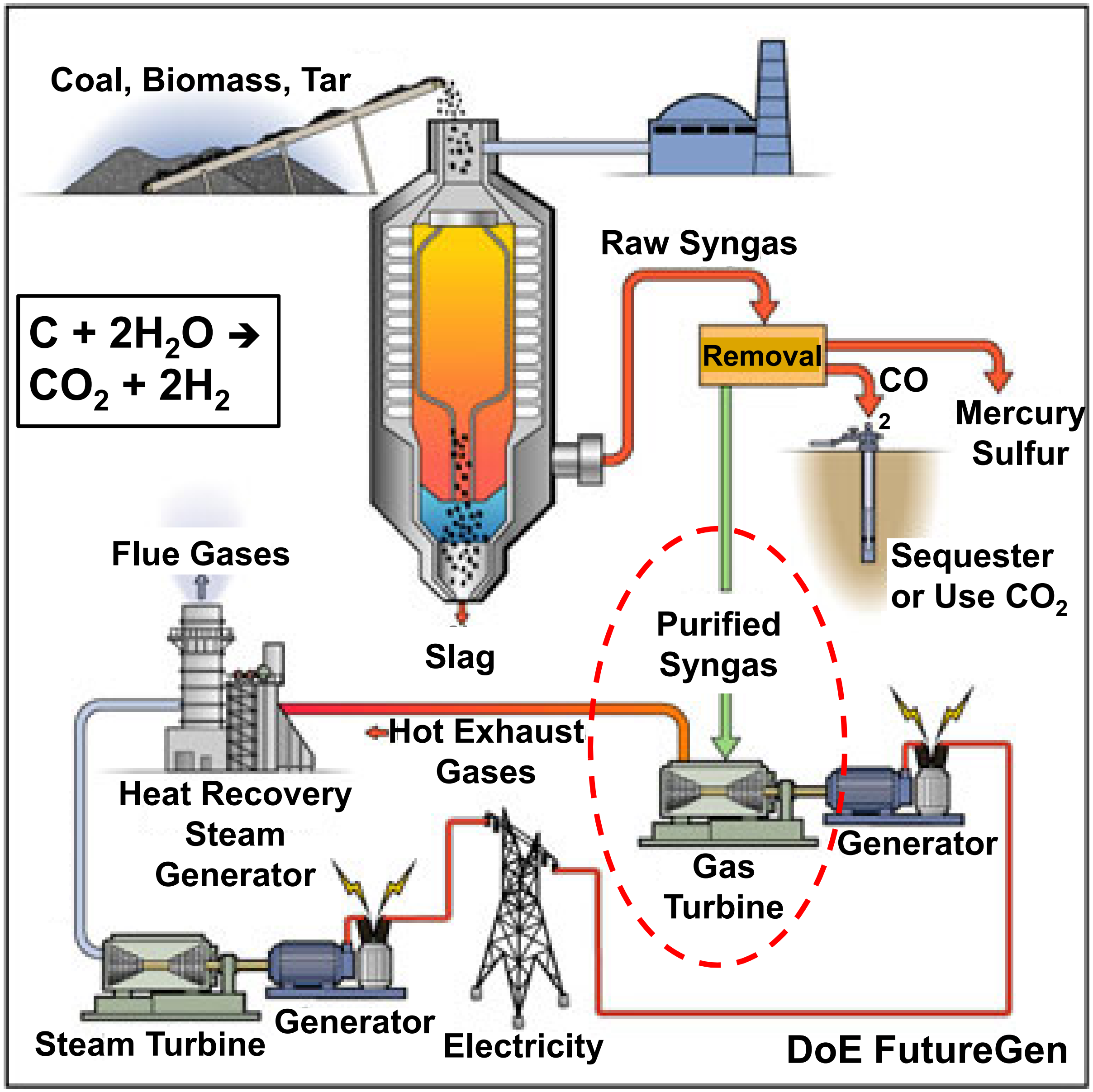
US Energy Info. Admin.
http://www.eia.doe.gov/fuelelectric.html

Sources of Silicate Deposits in Power Engines

- * Natural Gas (~40% Eff.)
- * Syngas Produced from Abundant Coal + H_2O
- * CO_2 Capture/Sequester
- * IGCC Plants Highly Efficient (~55%)
- * H_2-Rich Syngas-Fired: Higher Temps., Water
  - * F-Class: 1370 ˚C
  - * H-Class: 1430 ˚C
  - * J-Class: 1480 ˚C
  - * X-Class: 1700 ˚C
- * Syngas has Fly Ash (0.4 mg/m^3) (R. Wenglarz)
- * Amb. Dust (0.01 mg/m^3)
- * Kgs/day

Severe Damage to TBCs and Engines

Integrated Gasification Combined Cycle (IGCC)

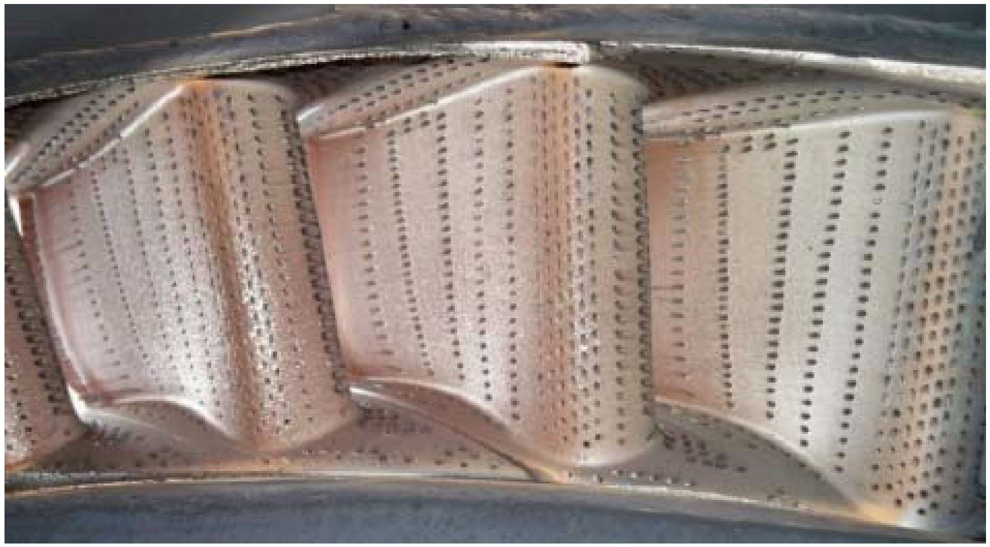


IGCC Power Plants

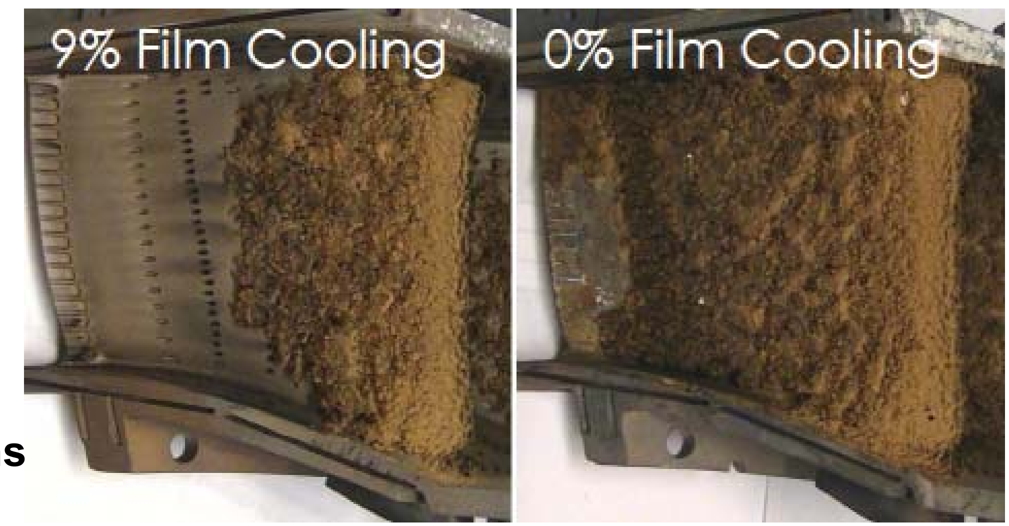
DoE

Sources of Silicate Deposits in Power Engines

Fly Ash (Lignite) Injection Tests on Hot Vanes (without TBC)



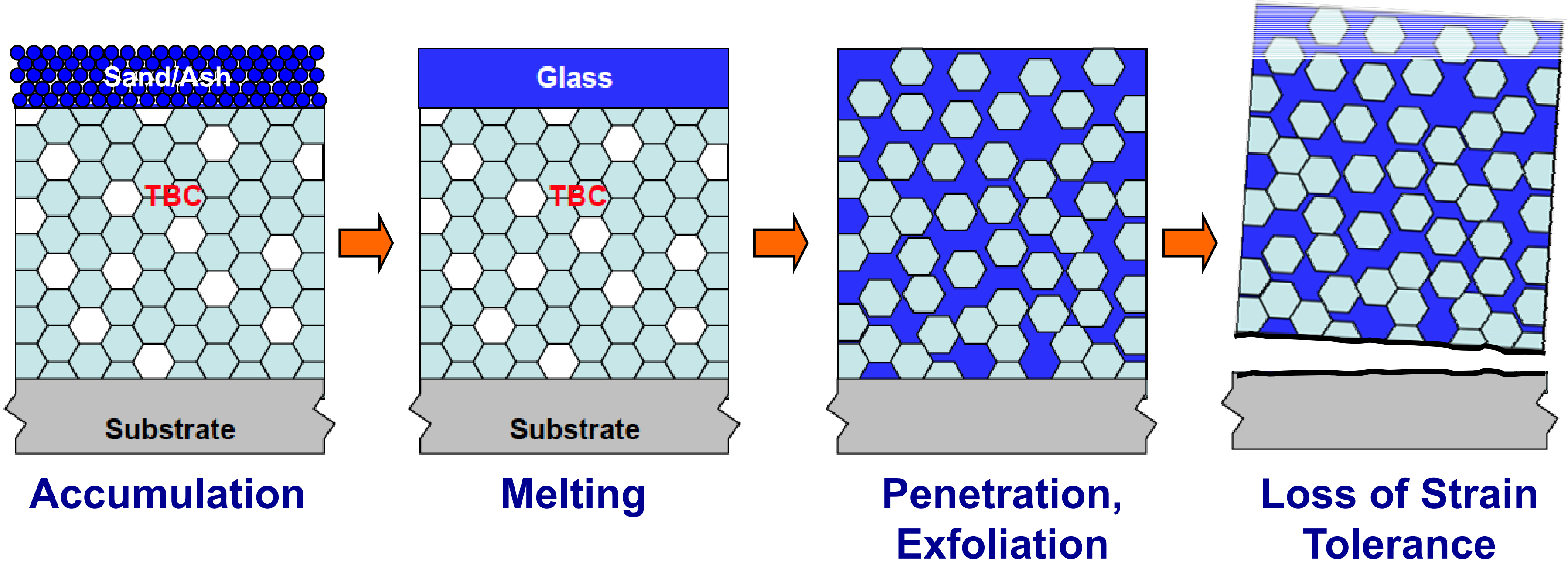
1010 ˚C
1 h Fly Ash Injection

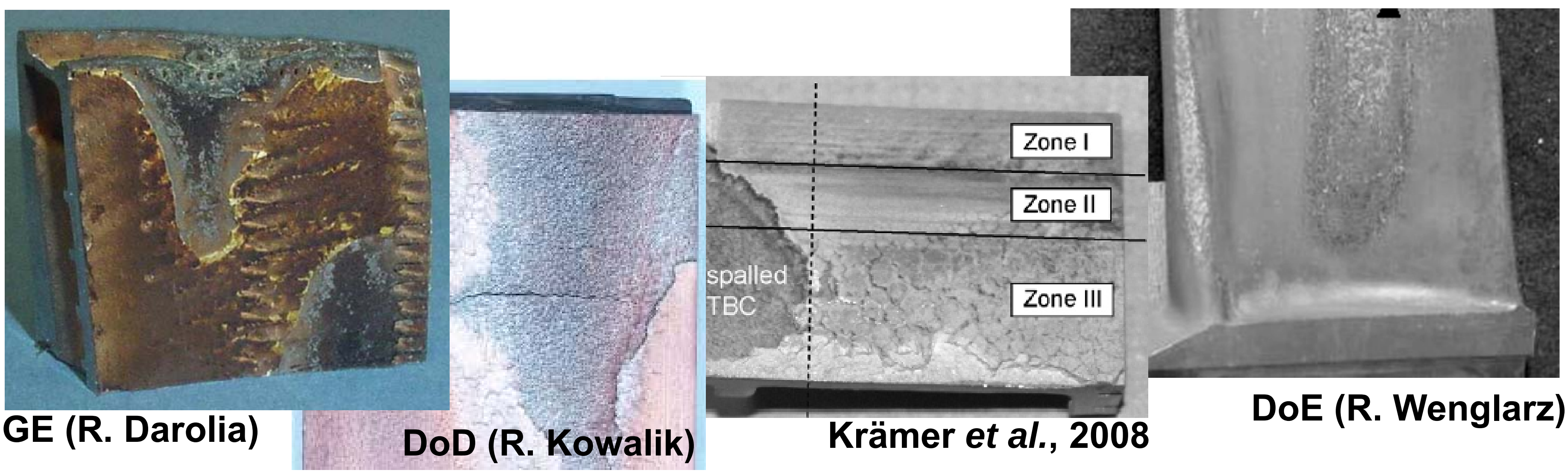


1066 ˚C
0.5 h Fly Ash Injection

J. Bons

Thermo-Chemo-Mechanical Damage of TBCs

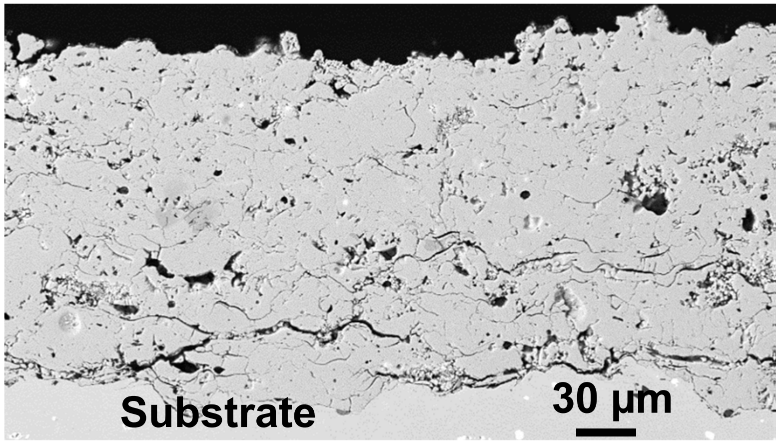


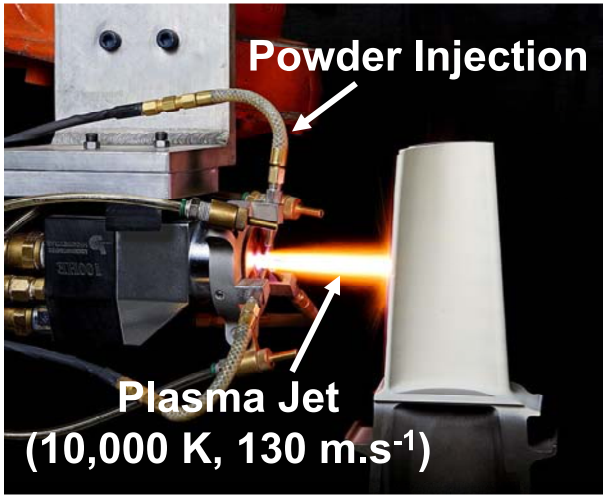


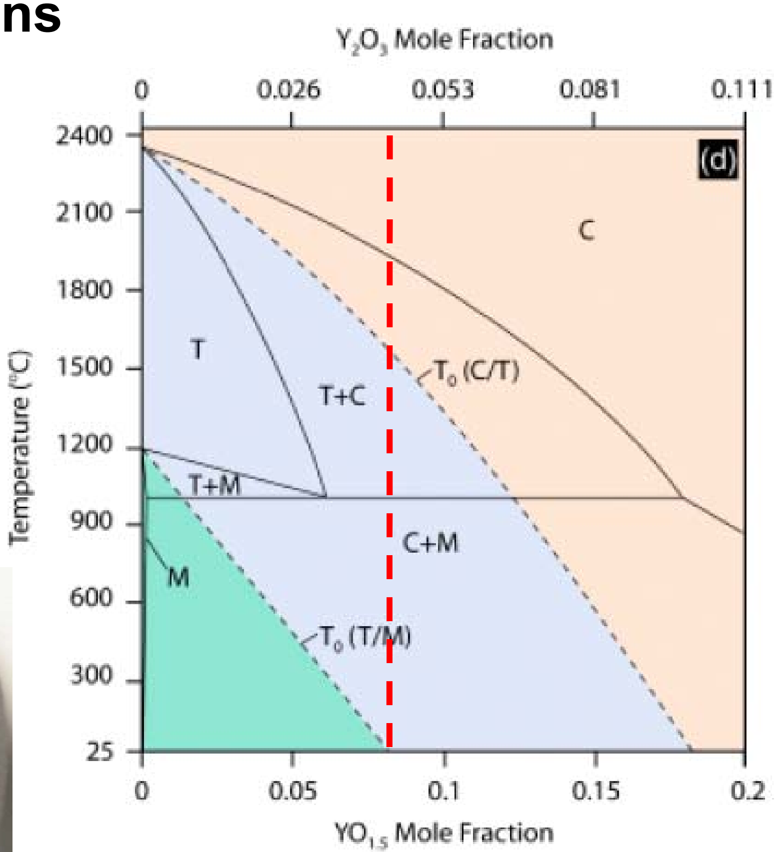
GE (R. Darolia) DoD (R. Kowalik) Krämer *et al.*, 2008 DoE (R. Wenglarz)

Molten Silicates Damage to Conventional TBCs

Air Plasma Spray (APS)
7 wt% Y_2O_3 Stabilized ZrO_2 (7YSZ) TBC
on Haynes 214 Ni-Based Superalloy Buttons



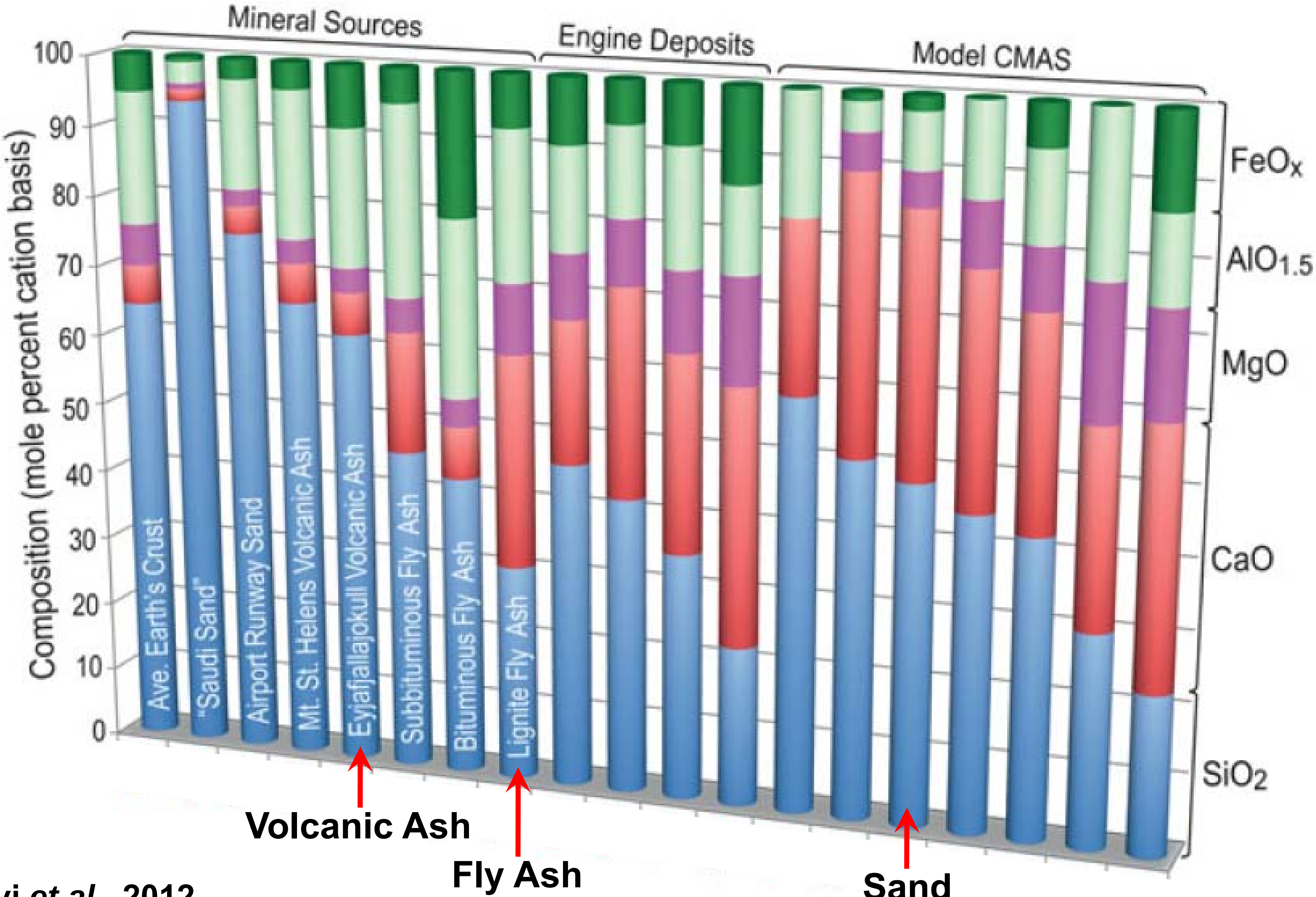




Chevalier *et al.* 2009

S. Sampath

Compositions of Silicate Deposits



Levi *et al.*, 2012

Silicate Deposits: CMAS Sand

Composition of Field Iraqi Sand Fines by XRF (wt.%)

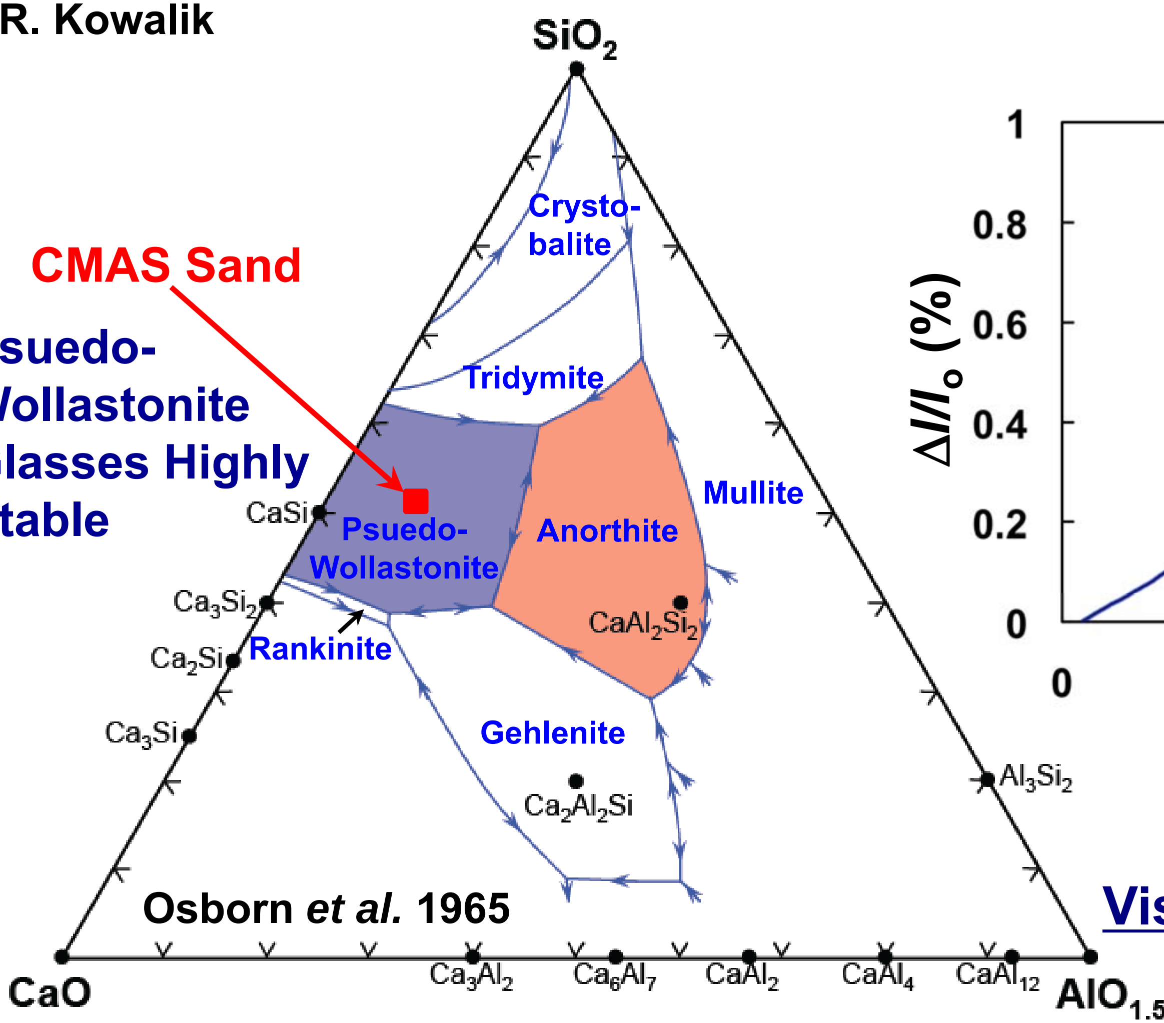
SiO_2	CaO	Al_2O_3	MgO	FeO	K_2O	Na_2O
49.7	35.3	6.7	3.3	2.4	1.6	1.0

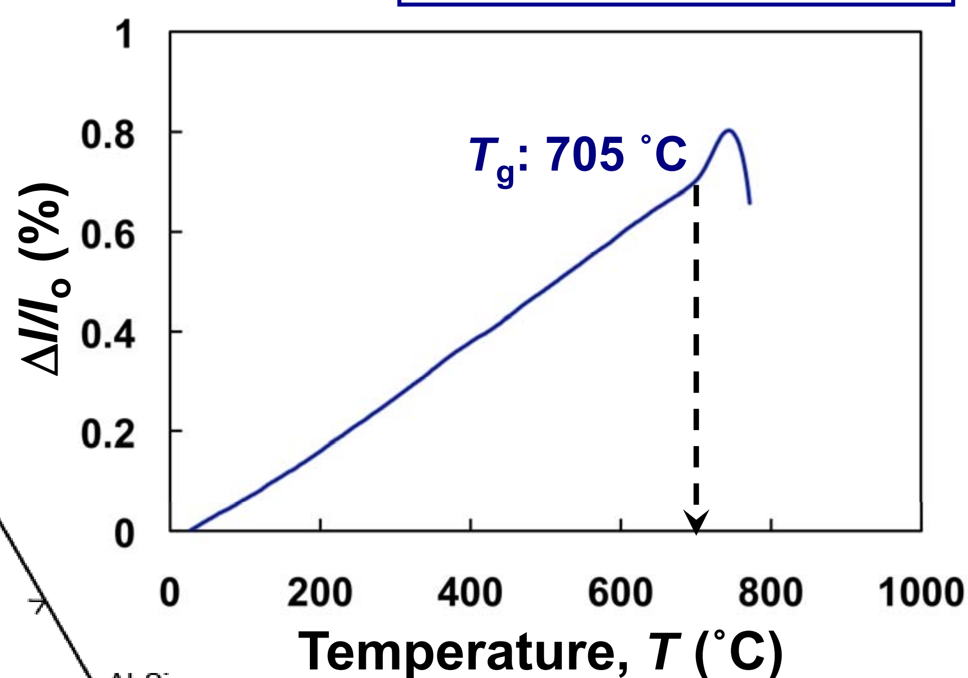
R. Kowalik

Glass Melted from Mixture of Oxides to Simulate Composition and Ball-Milled

CMAS Sand

Psuedo-Wollastonite Glasses Highly Stable

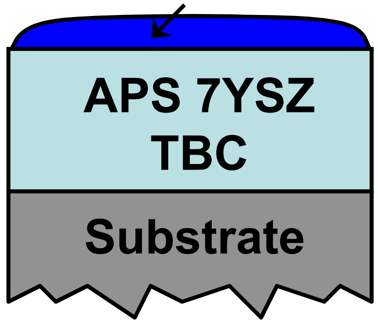


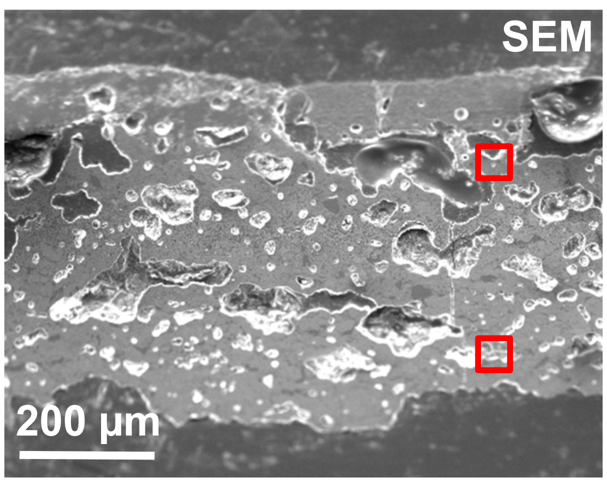


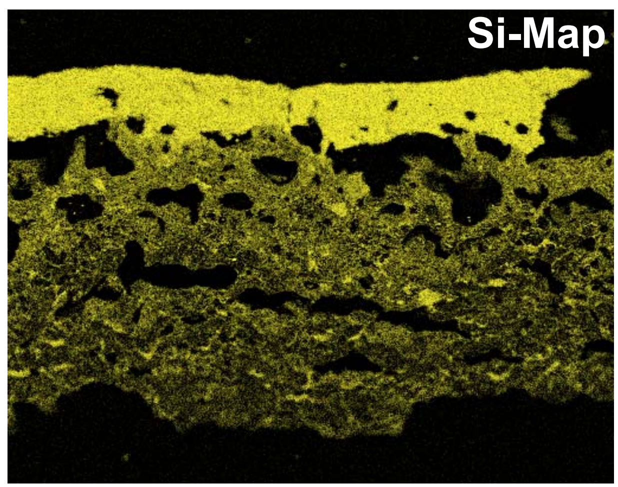
CTE: 10.4×10^{-6} ˚C^{-1}

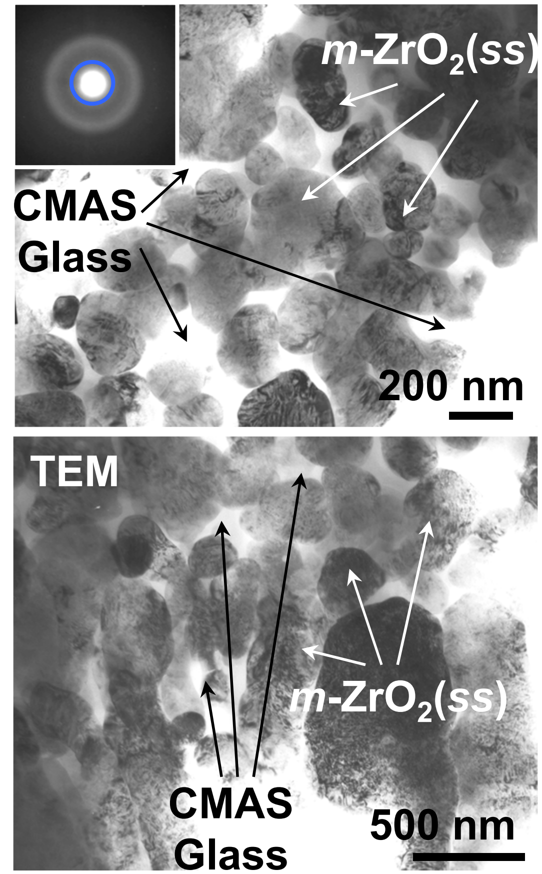
Viscosity: ~20 Pa.s@1200 ˚C

Molten Silicates Damage to APS 7YSZ TBCs: CMAS Sand









TBC Fully Penetrated

Acta Mat. 2007; *Acta Mat.* 2010

Silicate Deposits: Volcanic Ash

Eyjafjallajökull Ash (wt.%): Aero Engines

SiO_2	Al_2O_3	FeO	CaO	Na_2O	MgO	TiO_2	K_2O	P_2O_5	MnO
58.0	14.9	9.8	5.5	5.0	2.3	1.8	1.8	0.5	0.2

Óskarsson *et al.*, 2010

Plinian-Style
(Explosive)

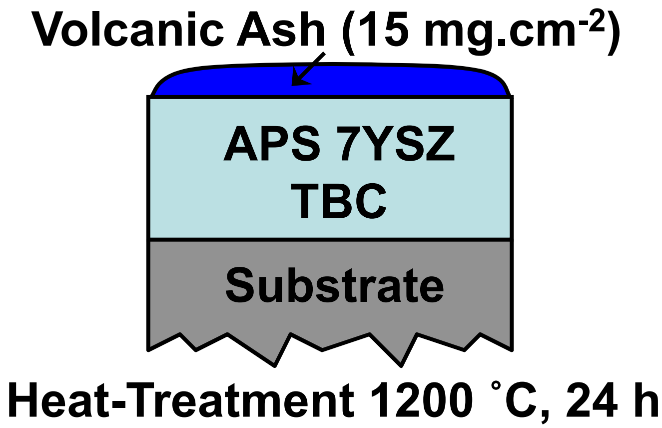
SiO_2(wt%)	Magma
68-77%	Rhyolite
63-68%	Dacite
52-63%	Andesite
48-52%	Basalt

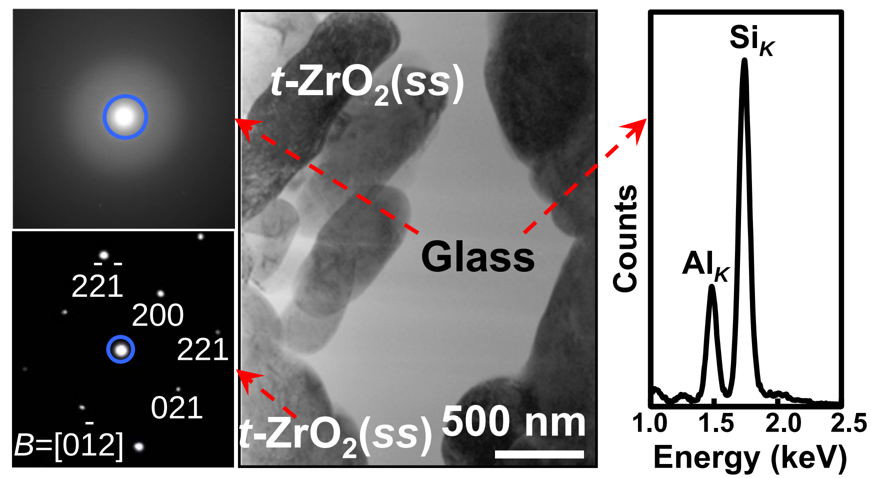
Hawaiian-Style
(Effusive)

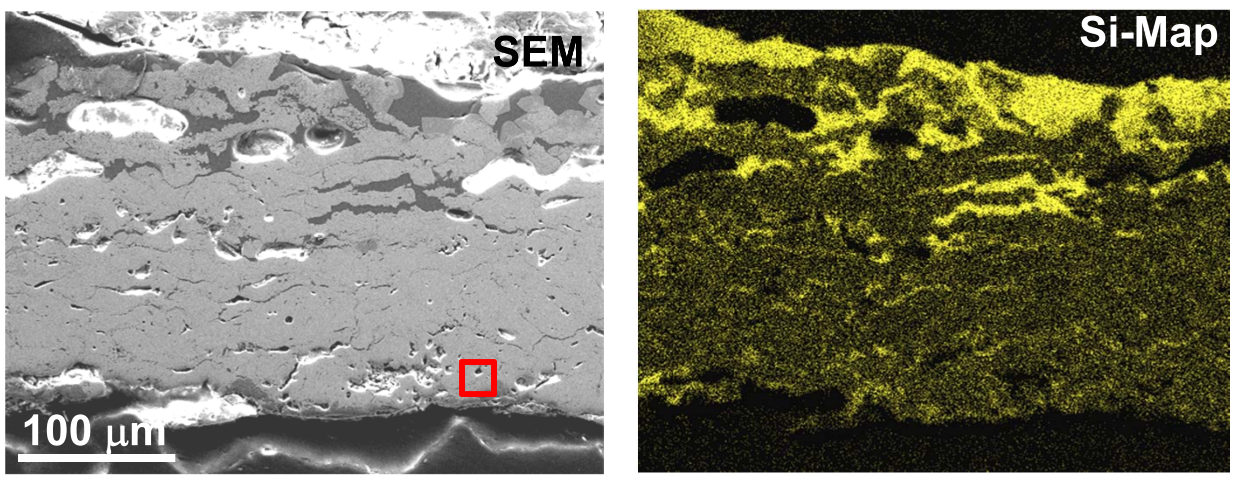
USGS

Schmincke, 2004

Molten Silicates Damage to APS 7YSZ TBCs: Eyjafjallajökull Ash







TBC Fully Penetrated

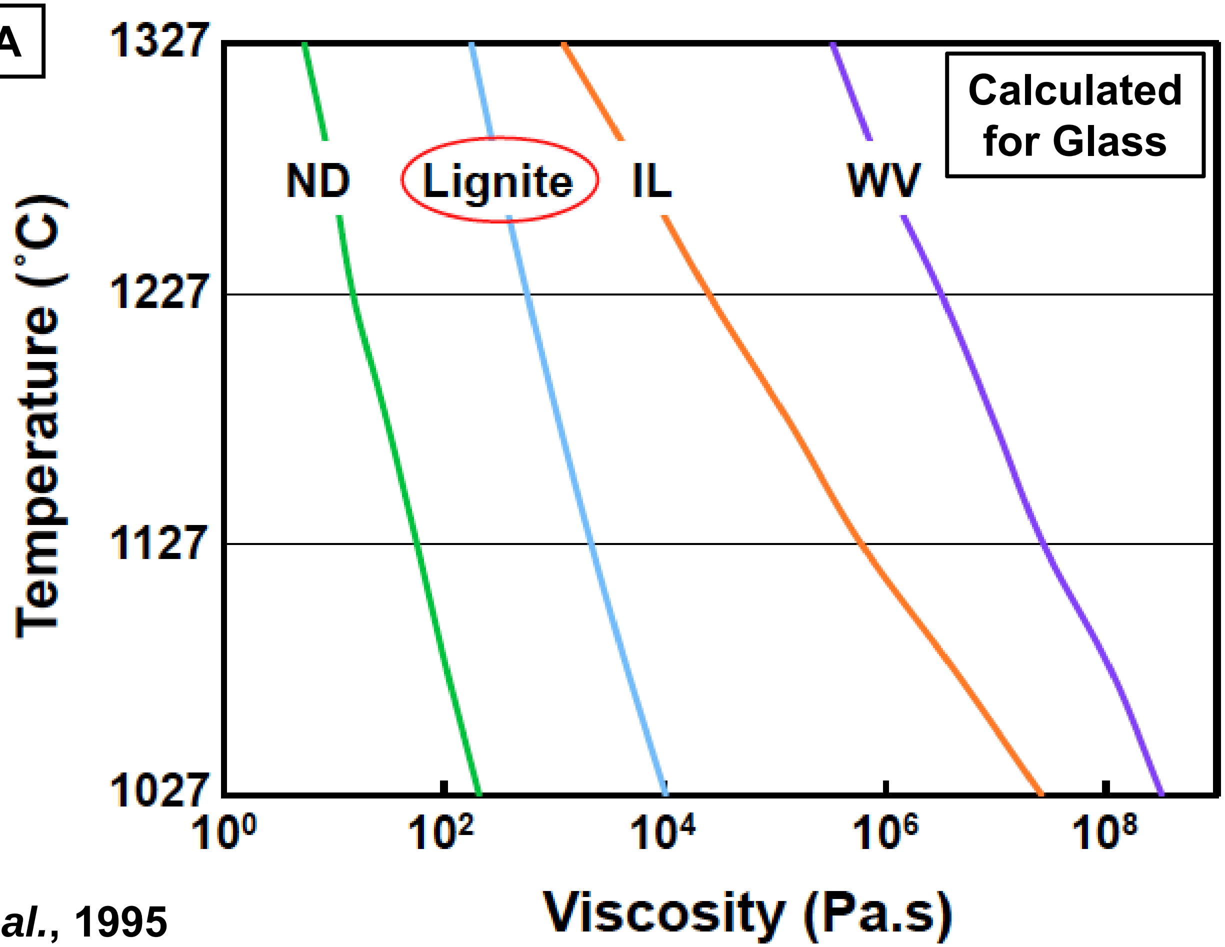
Adv. Mat. 2011

Fly Ash

Composition of Field Lignite Fly Ash by XRF (wt.%)

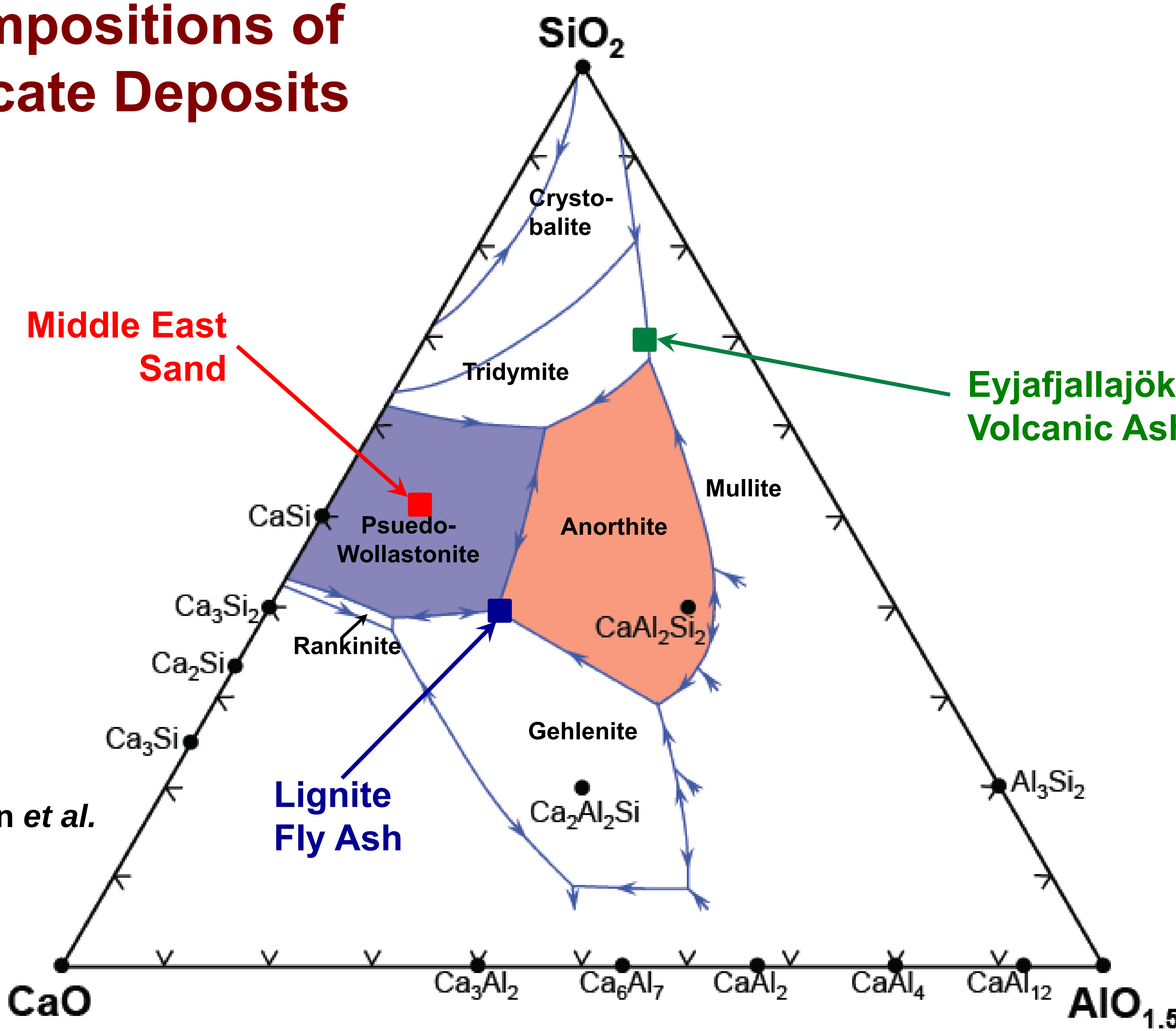
SiO_2	CaO	FeO	Al_2O_3	Cr_2O_3	MgO	SO_3	TiO_2	SrO	MnO
29.7	25.4	14.8	14.7	5.1	3.6	1.8	1.1	1.0	0.9

M.P. ~1180 ˚C by DTA



Senior *et al.*, 1995

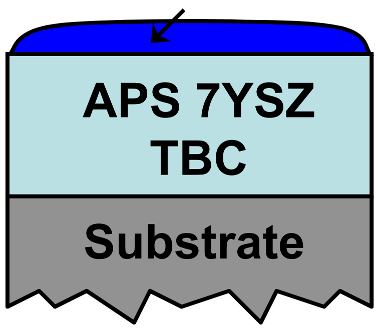
Compositions of Silicate Deposits



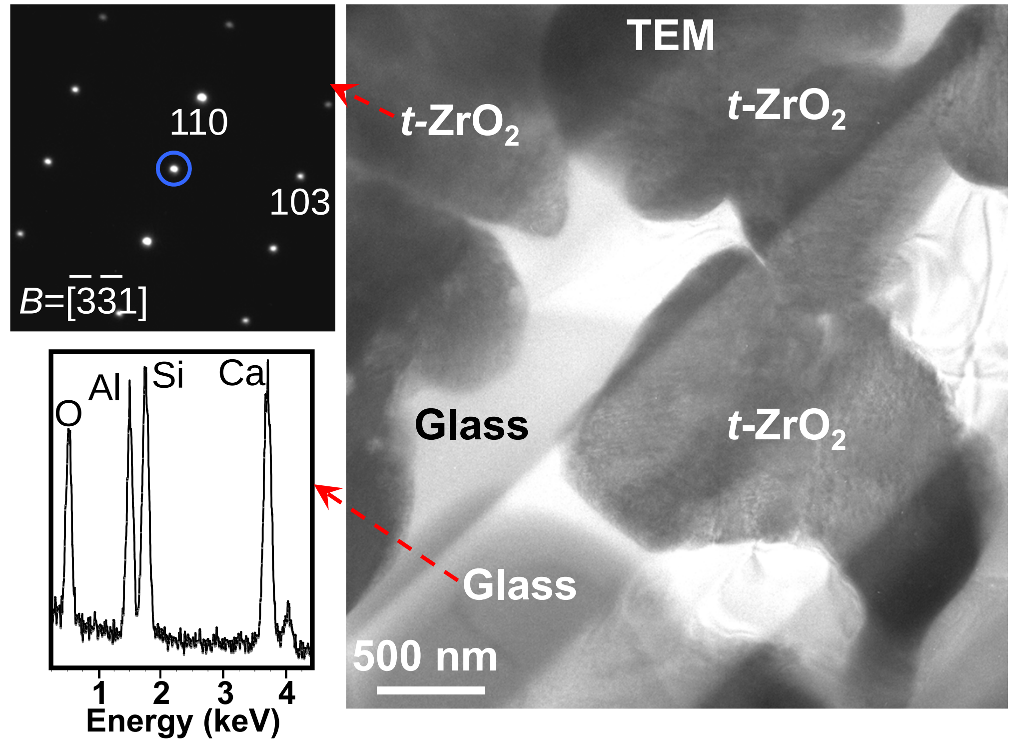
Osborn *et al.* 1965

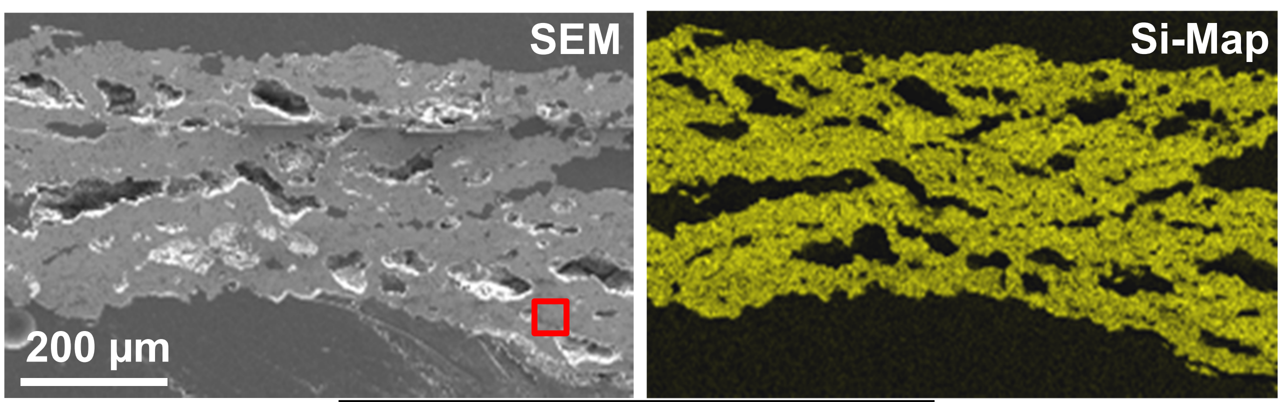
Molten Silicates Damage to APS 7YSZ TBCs: Lignite Fly Ash — Power Engines

Ball-Milled Fly Ash
(35 mg.cm^{-2})



Heat-Treatment 1200 ˚C, 24 h





Mat. Sci. Engr. A **2011**

TBC Fully Penetrated

Molten Deposits Damage Mechanisms: 7YSZ

* CMAS/Ash Melts into a Glass

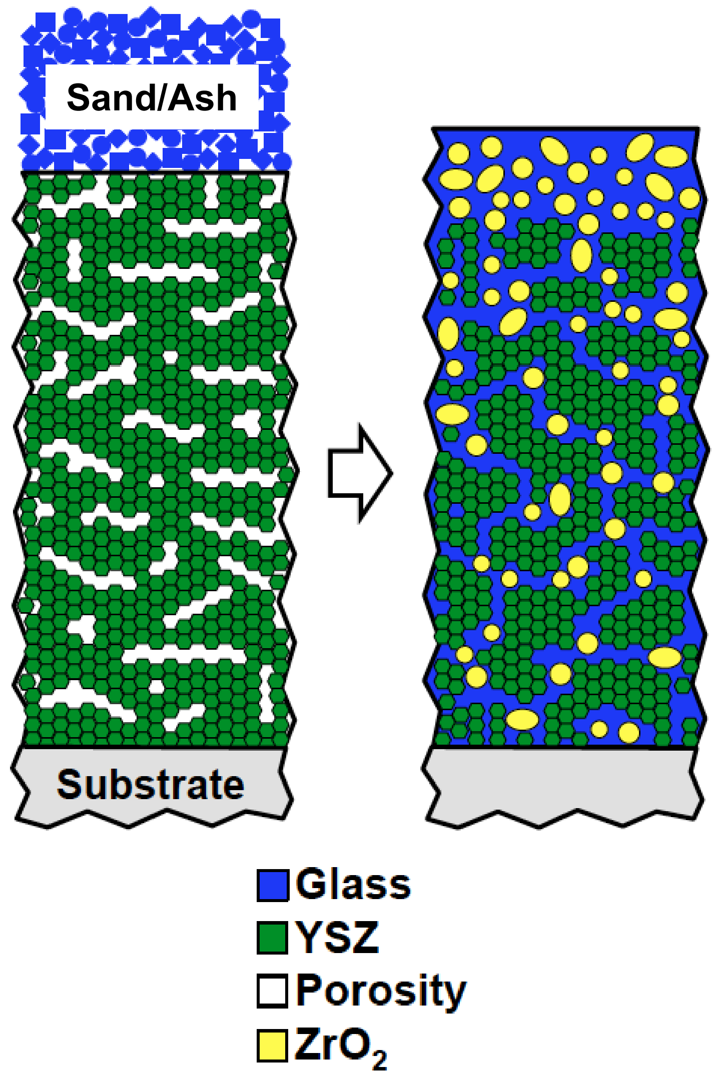
* Infiltrates Pores/Cracks

* Penetrates 7YSZ Grain Boundaries

* 7YSZ Dissolution, Reprecipitation as Y-Depleted ZrO_2 (C. Levi)

* Dilatation and Exfoliation

* Little Effect on Glass Behavior, Small Amount of Solute (Y_2O_3) in TBC: Solute:Zr :: 0.083:1



Damage Mitigation: APS 7YSZ+Al+Ti TBCs

* Engineered TBCs Modify Glass in Contact *In Situ*

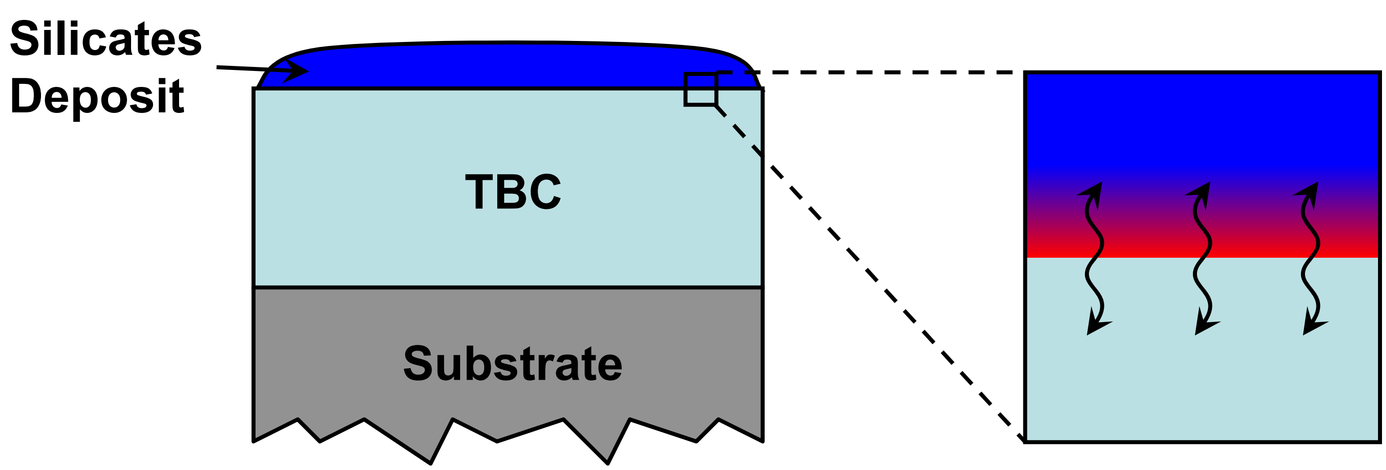
* TBCs "Reservoirs" of Al^{3+}, Ti^{4+} in Solid Solution

* Al^{3+} Incorporation in Glass => Shifts Comp., Crystallizes Glass

* Makes Glass Immobile at Operating Temps.

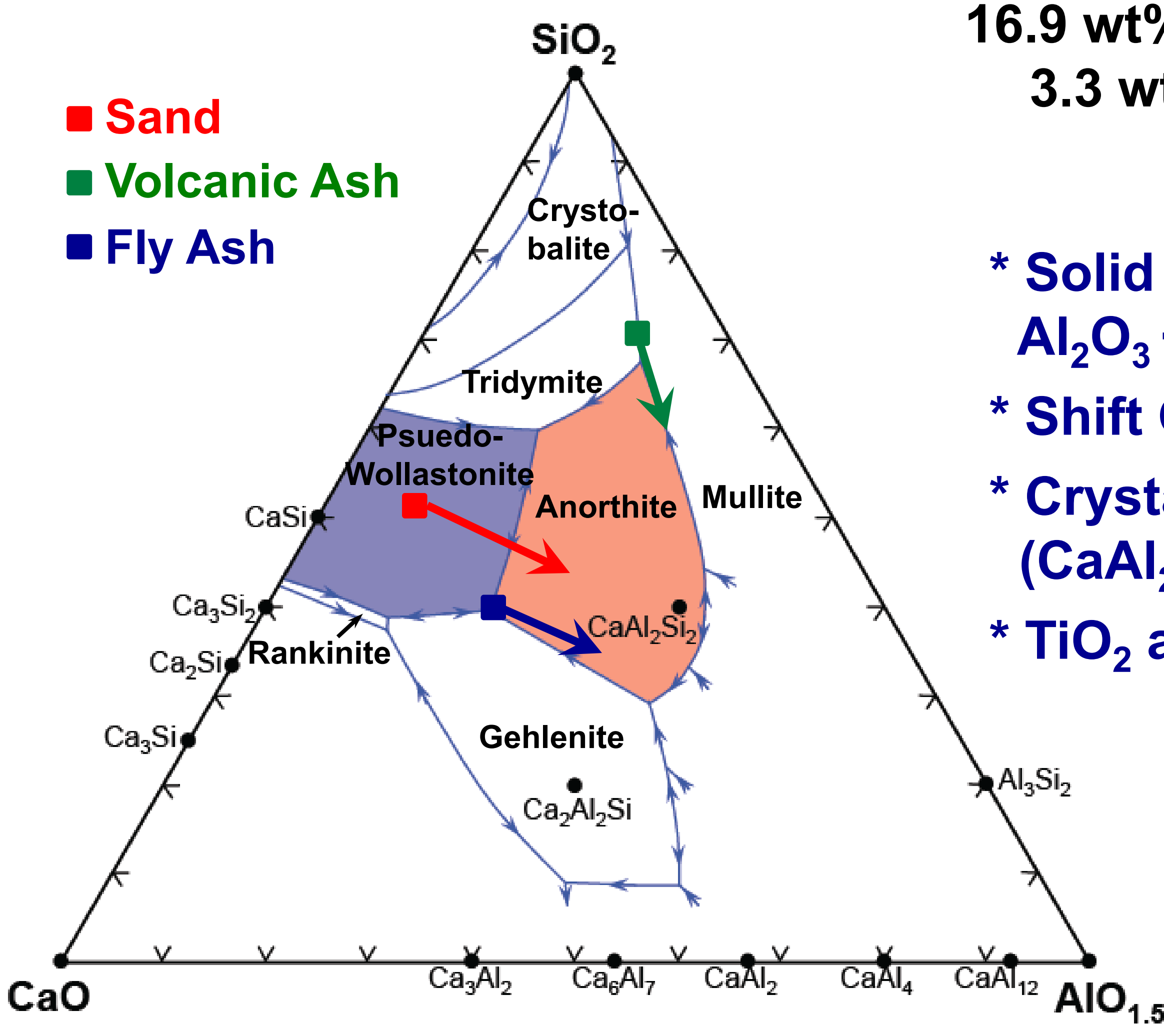
* Al^{3+}, Ti^{4+} in Solid Solution (Metastable) in 7YSZ TBC

  - Avoids Second Phase, Th. Exp. Mismatch, Cracking
  - Easier dissolution
  - Ti^{4+} Serves as Nucleating Agent



Acta Mat. 2007; *Surf. Coat. Tech*. 2009; *Acta Mat.* 2010; *Adv. Mat.* 2011; *Acta Mat.* 2012

APS 7YSZ+Al+Ti TBCs: New Composition

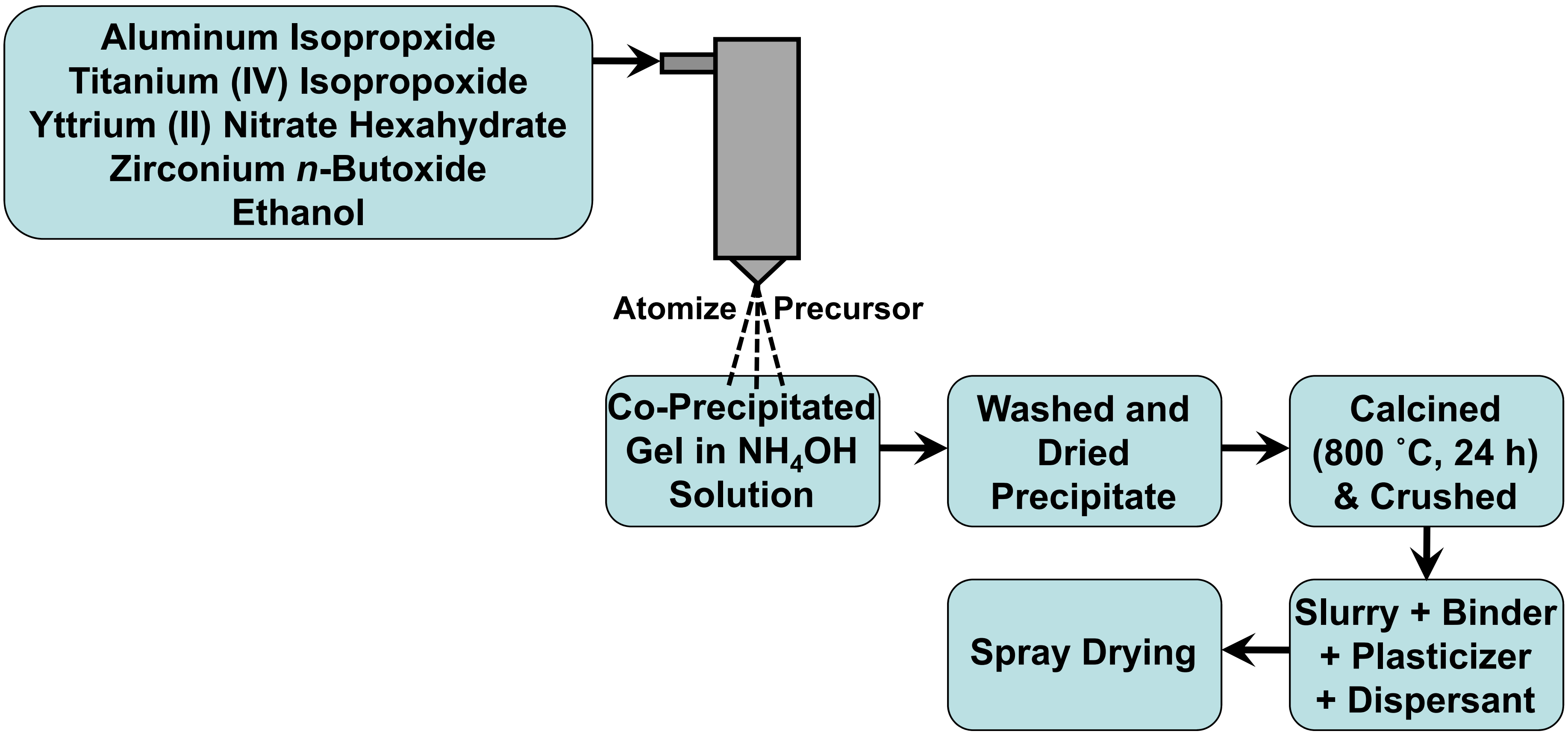


7YSZ +
16.9 wt% (20 mol%) Al_2O_3 +
3.3 wt% (5 mol%) TiO_2

- Solid Sol'n with Metastable $Al_2O_3 + TiO_2$
- Shift Glass Comp. to Al-Rich
- Crystallize Anorthite ($CaAl_2Si_2O_8$)
- TiO_2 as Nucleating Agent

APS 7YSZ+Al+Ti TBCs: Processing

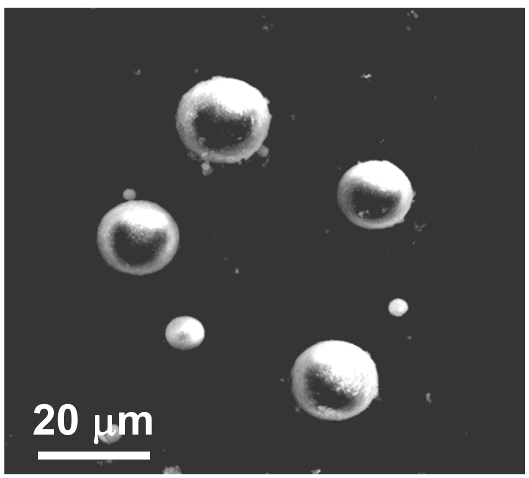
* **Special Sprayable Powders Synthesized**
  - 7YSZ + 16.9 wt% (20 mol%) Al_2O_3 + 3.3 wt% (5 mol%) TiO_2



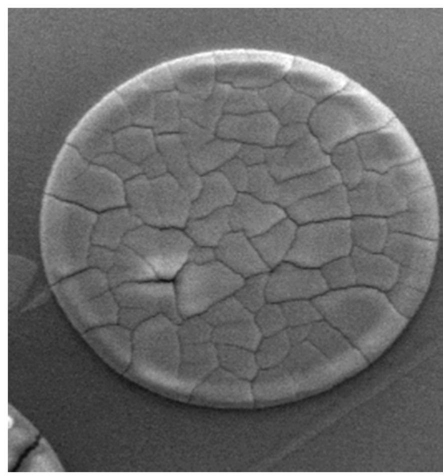
APS 7YSZ+Al+Ti TBCs: Processing

* Spray-Dried Single-Phase Powder: ~20 μm

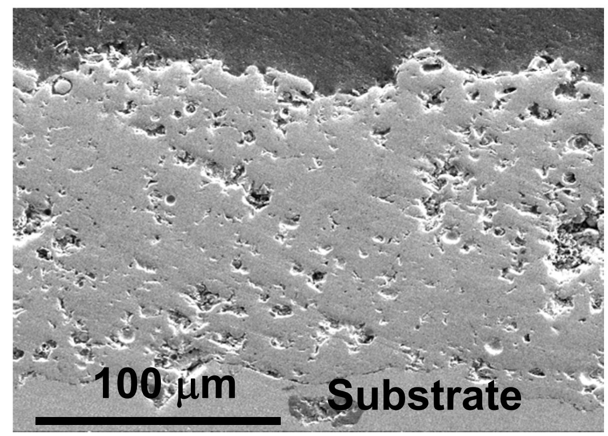
* Air Plasma Spray Development
  - Single-Splat Experiments
  - Process Optimization Through Diagnostics
  - Coatings on Haynes 214 Substrates



Spray-Dried Powder



Single-Splat Experiments



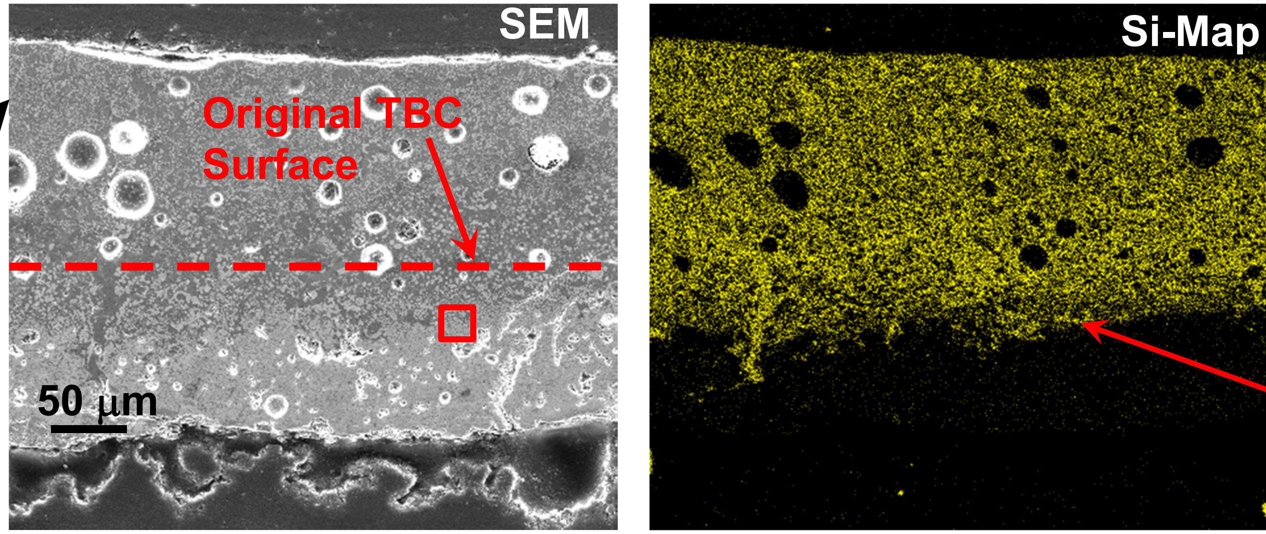
APS Coatings

S. Sampath

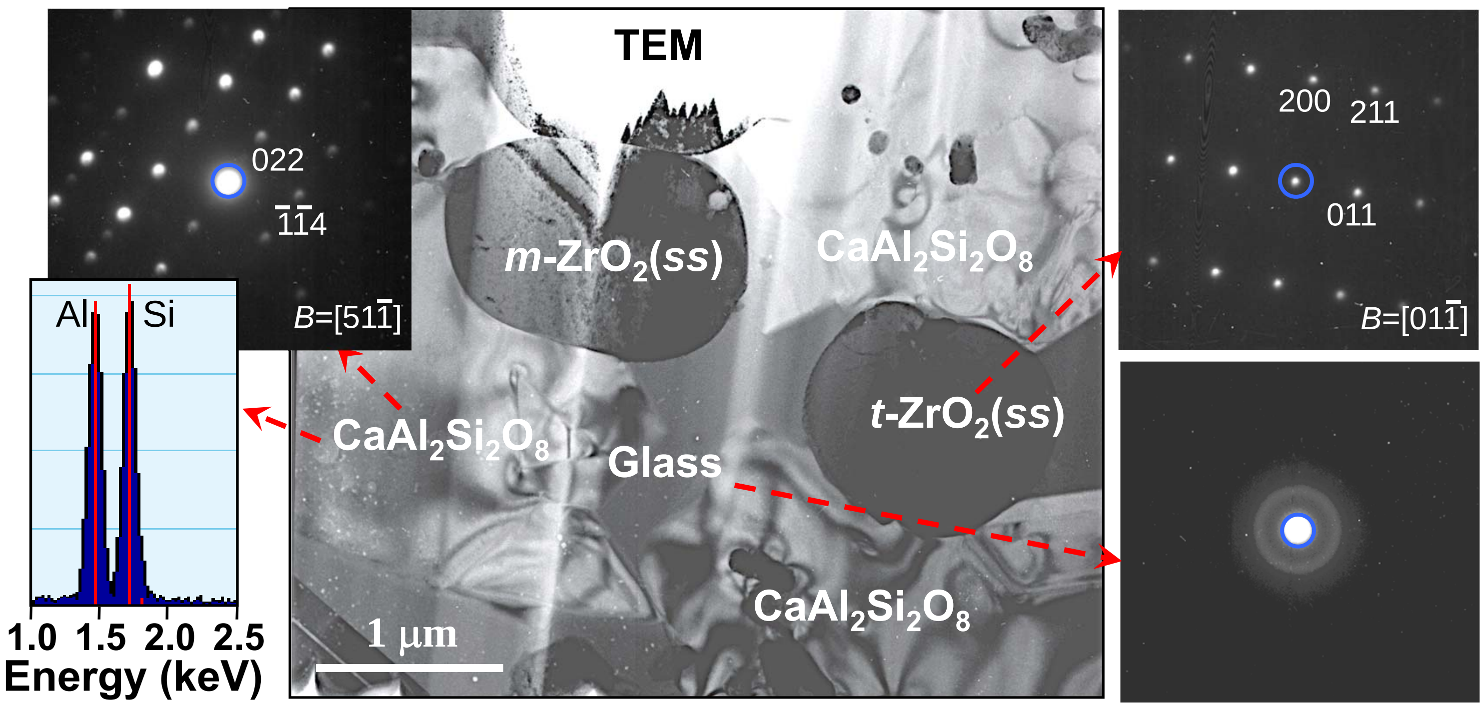
APS 7YSZ+Al+Ti TBCs: CMAS Sand

CMAS Sand Glass Frit ($35\ mg.cm^{-2}$)

1200 ˚C, 24 h



CMAS-Front Arrested (~25%)

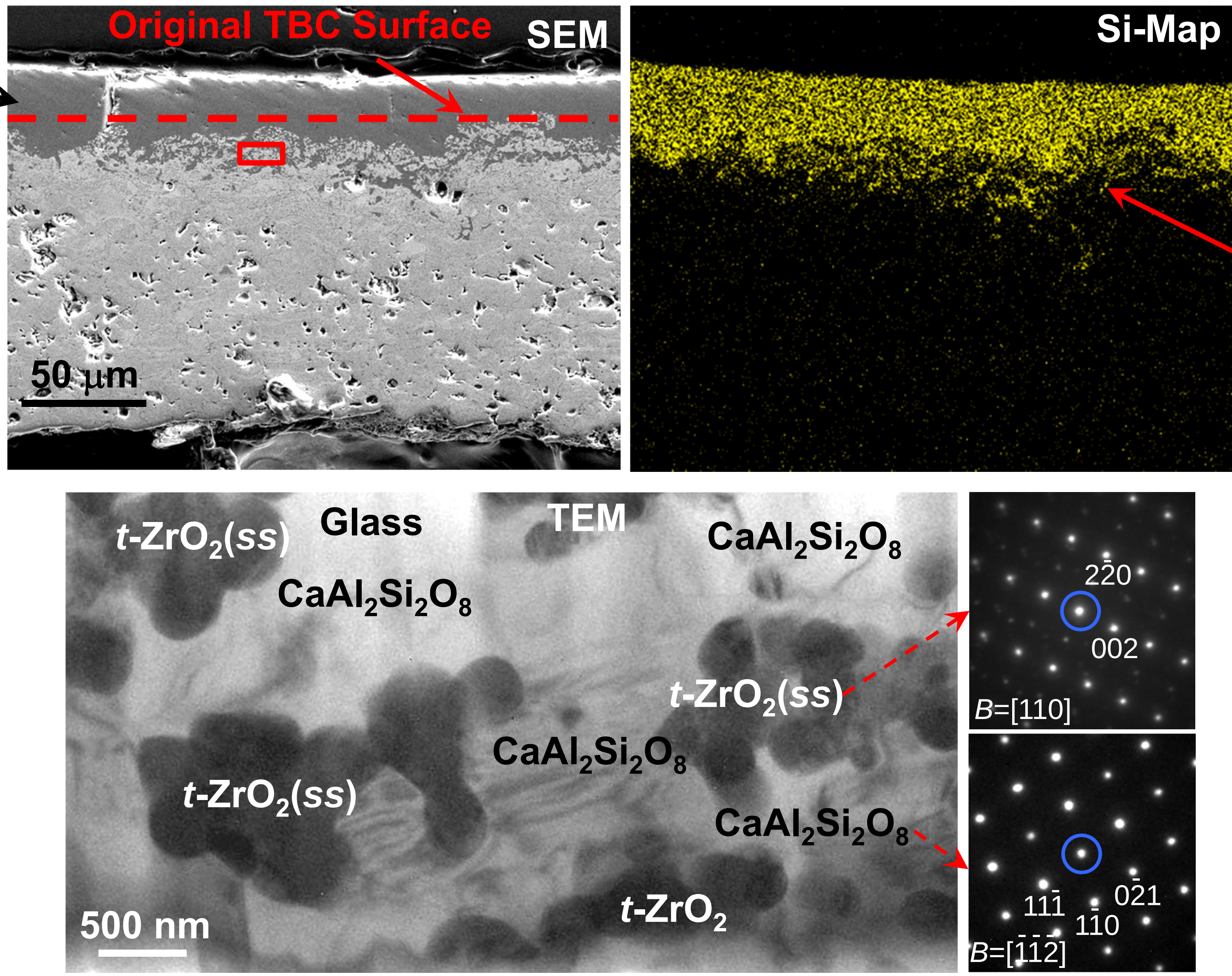


Acta Mat. 2010

APS 7YSZ+Al+Ti TBCs: Eyjafjallajökull Ash

Ball-Milled Volcanic Ash ($15\ mg.cm^{-2}$)

1200 ˚C, 24 h



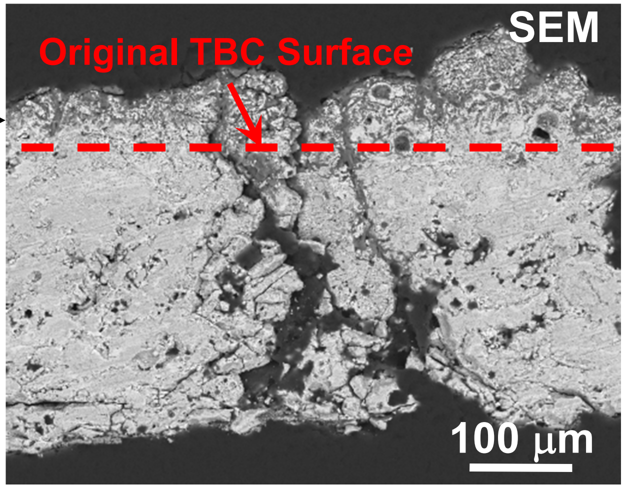
Ash-Front Arrested (~17%)

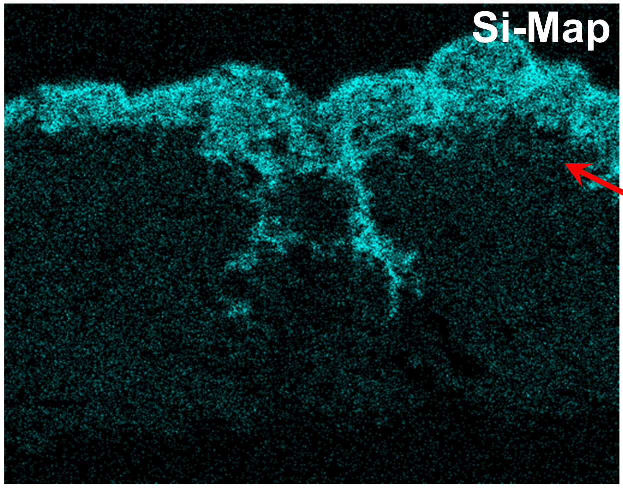
Adv. Mat. 2011

APS 7YSZ+Al+Ti TBCs: Lignite Fly Ash

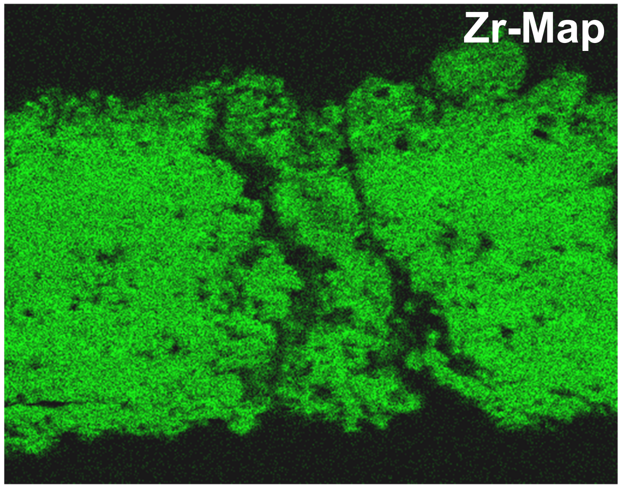
Ball-Milled Fly Ash (35 mg.cm^{-2})

1200 ˚C, 24 h





Ash-Front Partially Arrested



Mitigation Mechanisms: APS 7YSZ+Al+Ti TBCs

* Sand/Ash Melts into a Glass

* Infiltrates Pores/Cracks

* Penetrates Grain Boundaries

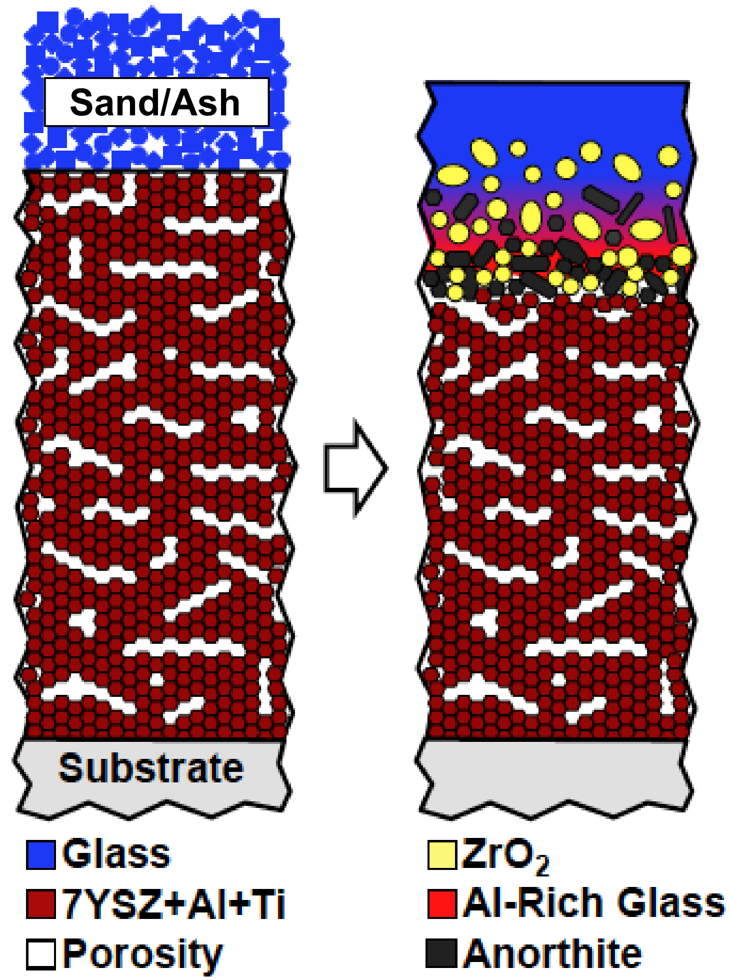
* Dissolution-Reprecipitation of Solute-Depleted ZrO_2

* Glass Accumulates Al and Ti, Large Amount of Solute (Y_2O_3, Al_2O_3, TiO_2) in TBC: Solute:Zr :: 0.73:1

* Shifts to Anorthite Composition

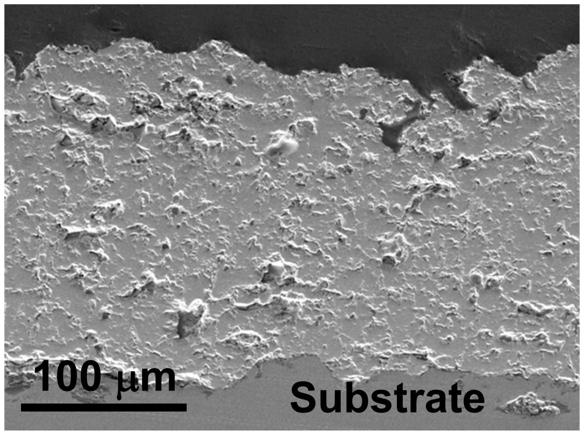
* Crystallization of Anorthite (MP 1550 ˚C) Aided by TiO_2 Nucleating Agent

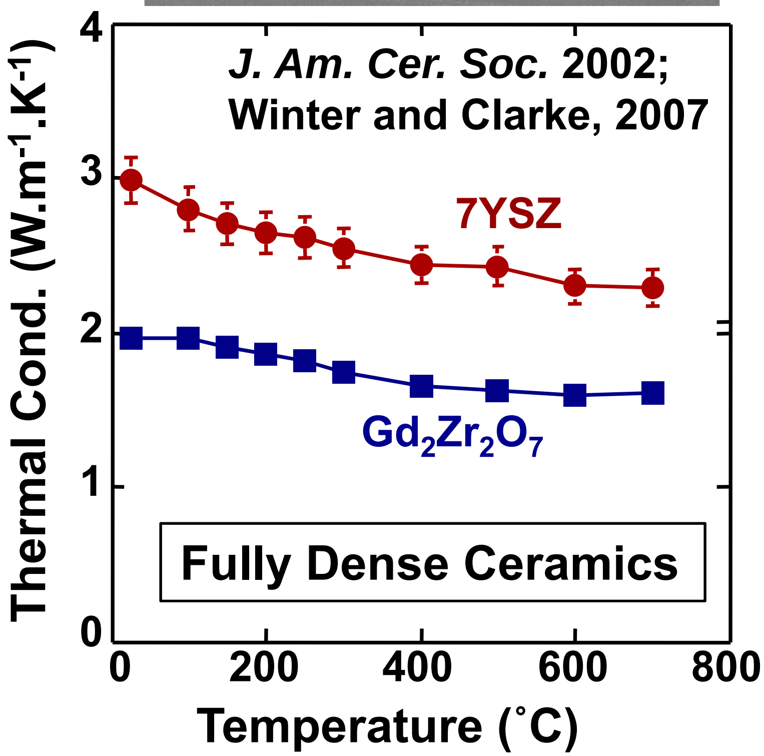
* Arrests Front



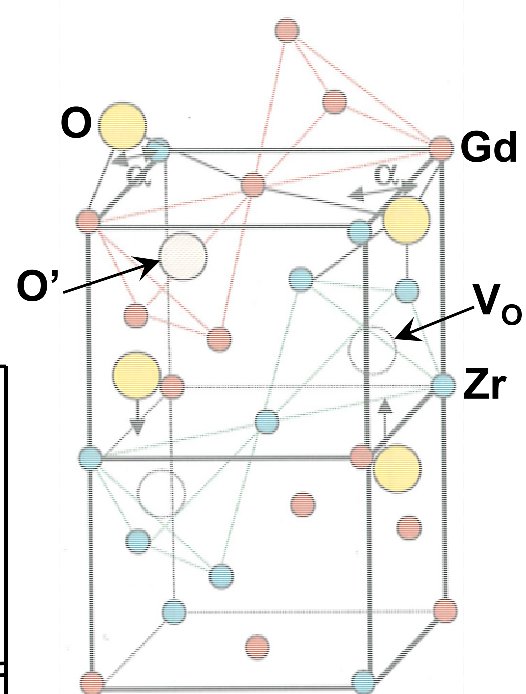
APS $Gd_2Zr_2O_7$ TBCs

$Gd_2Zr_2O_7$ APS TBC





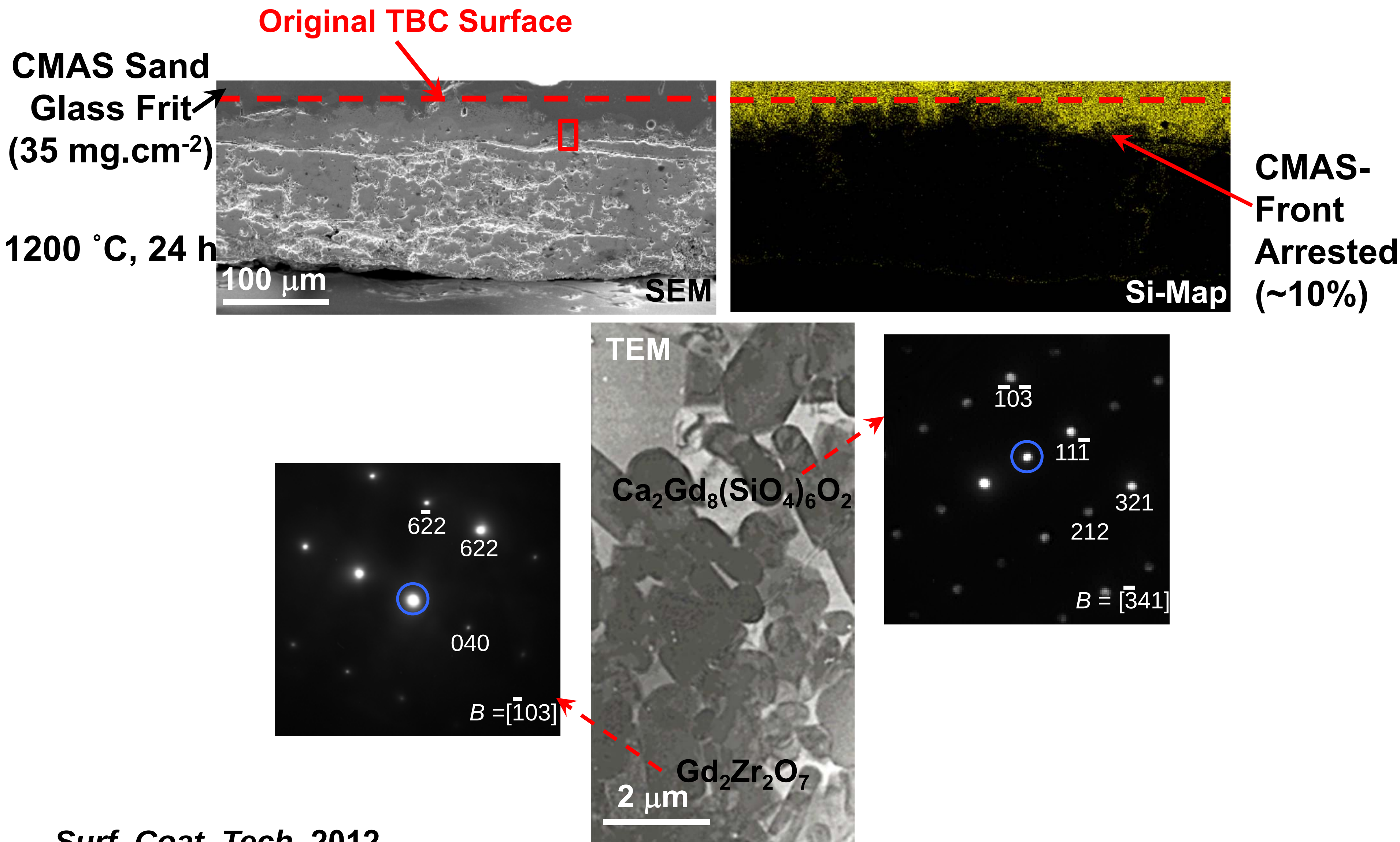
Pyrochlore/Fluorite



- * Low Th. Cond.
- * CMAS Resistance in EB-PVD TBCs Demonstrated (Maloney *et al.*, Litton *et al.*, Levi *et al.*)

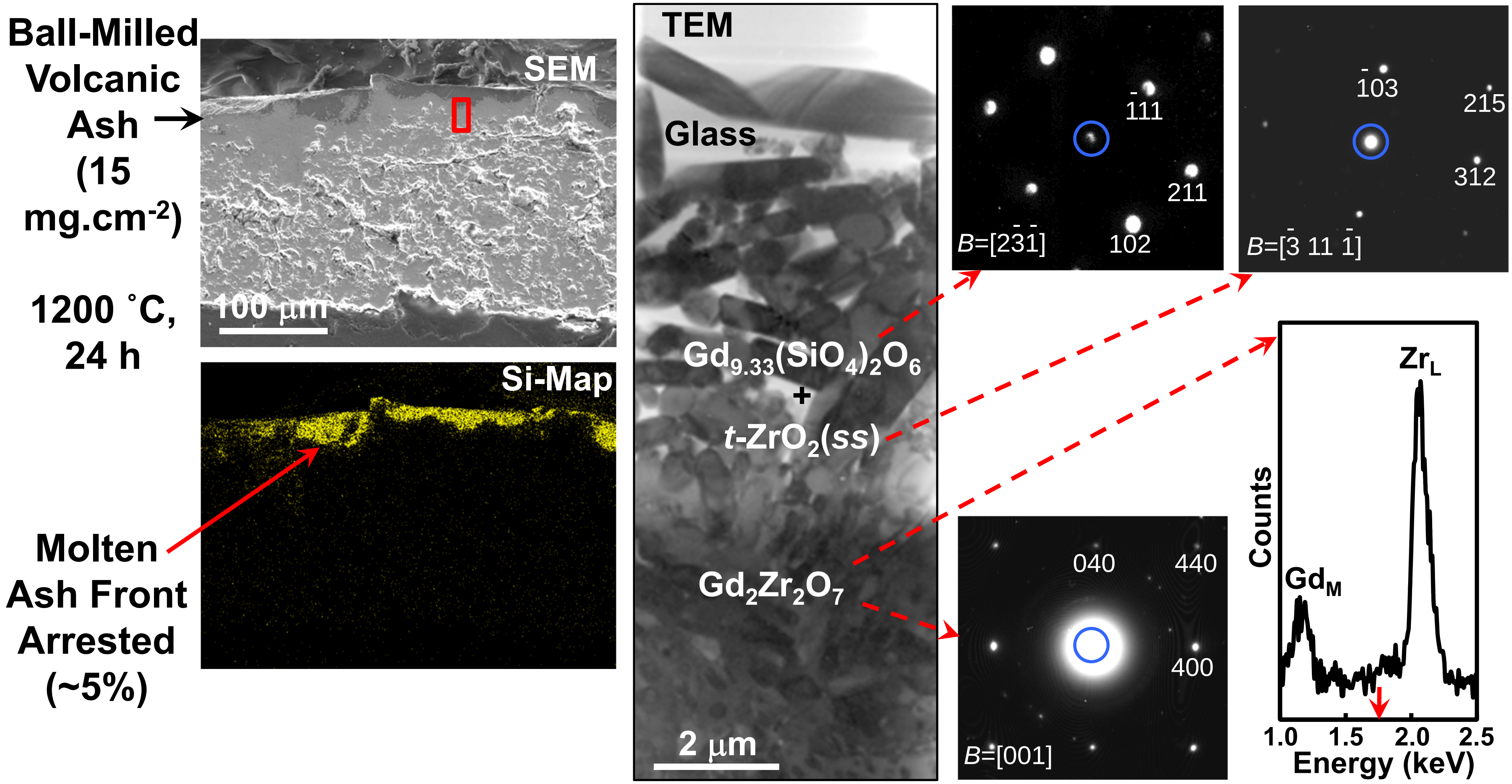
Adv. Mat. 2011; *Mat. Sci. Engr. A* 2011; *Surf. Coat. Tech.* 2012

APS $Gd_2Zr_2O_7$ TBCs: CMAS Sand



Surf. Coat. Tech. **2012**

APS $Gd_2Zr_2O_7$ TBCs: Eyjafjallajökull Ash

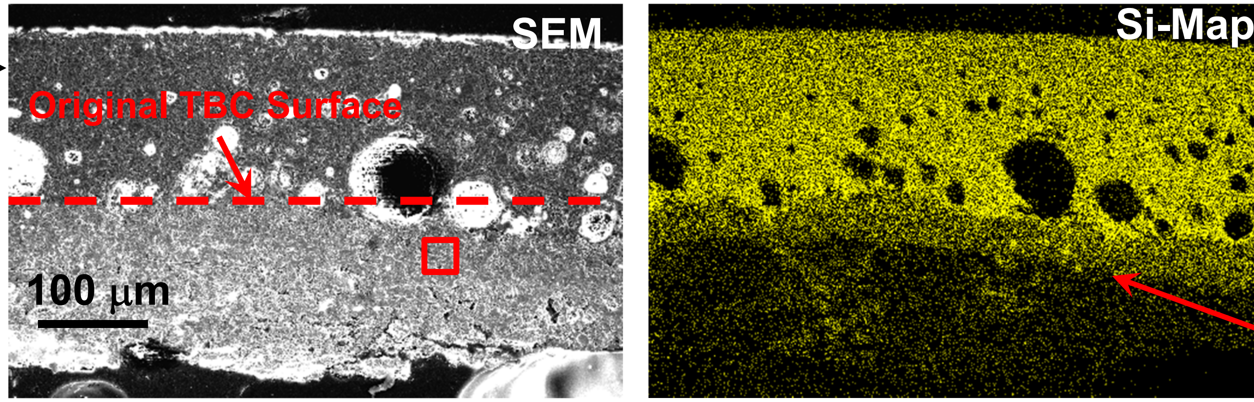


Adv. Mat. 2011

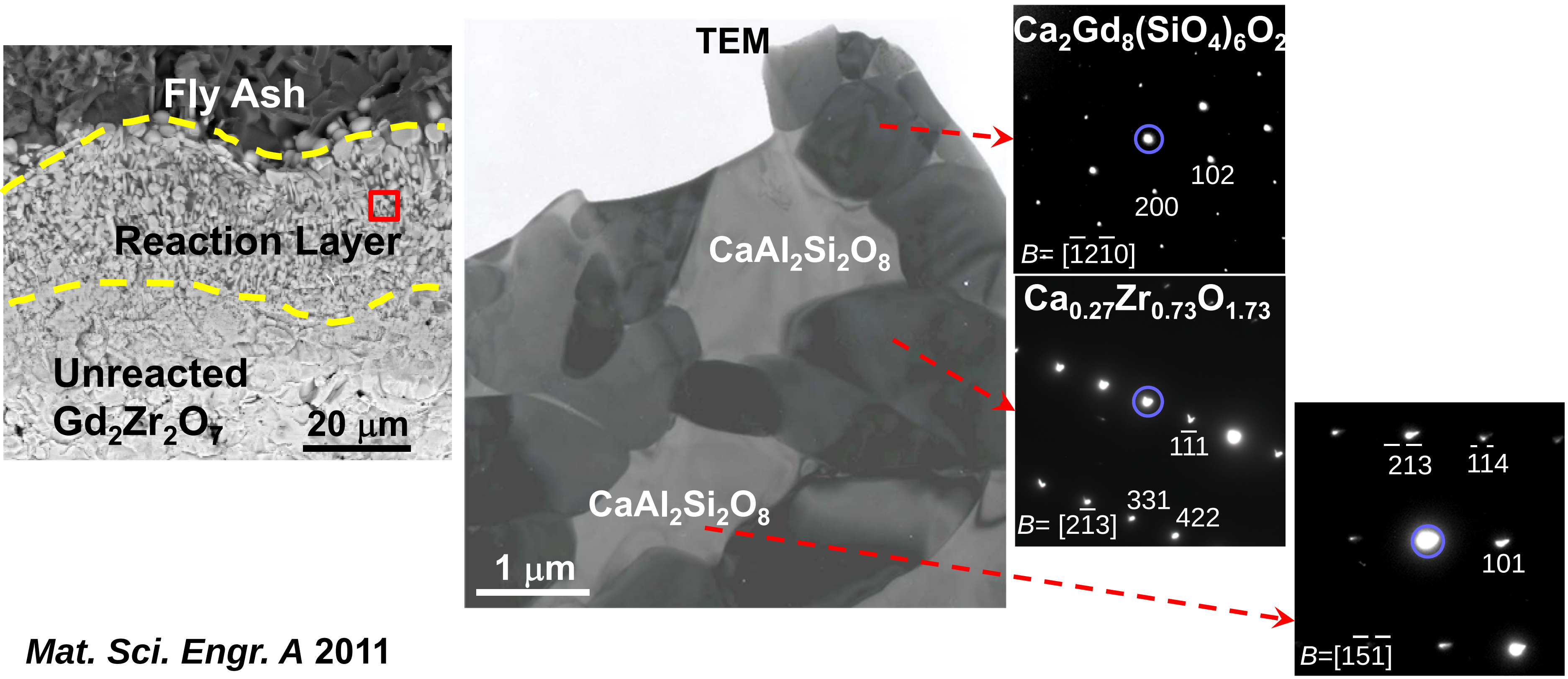
APS $Gd_2Zr_2O_7$ TBC: Lignite Fly Ash

Ball-Milled Fly Ash ($15\ mg.cm^{-2}$)

1200 ˚C, 24 h



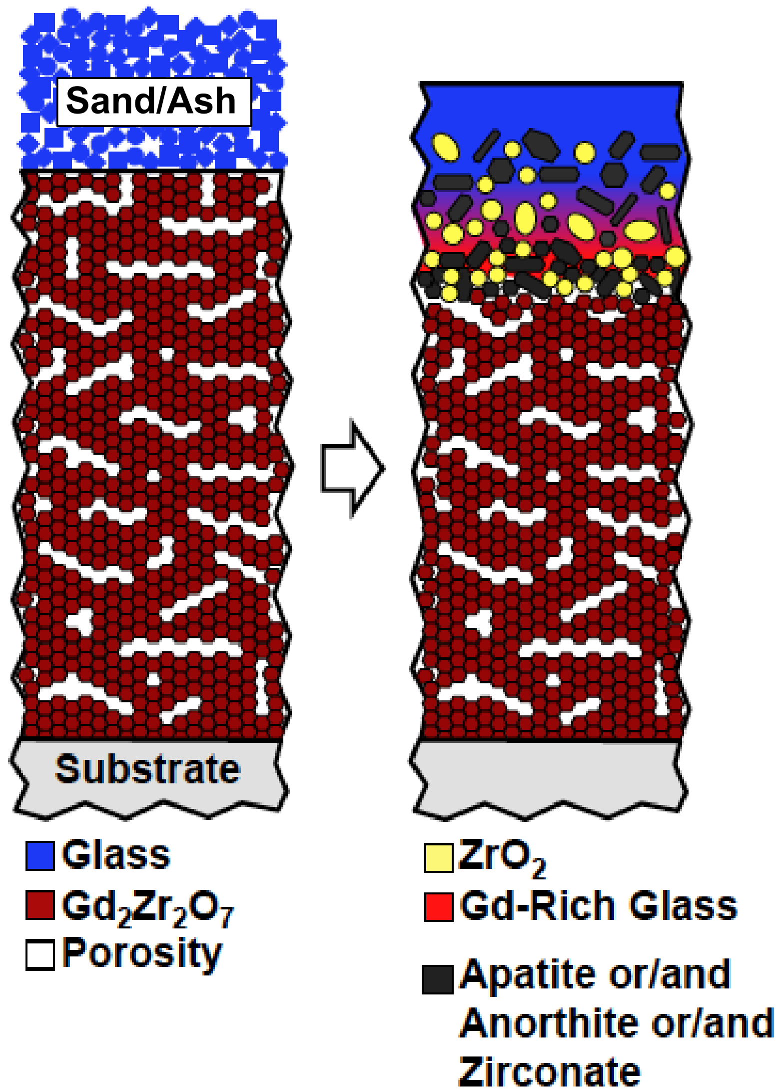
Molten-Ash Front Arrested (~30%)



***Mat. Sci. Engr. A* 2011**

Mitigation Mechanisms: $Gd_2Zr_2O_7$ TBCs

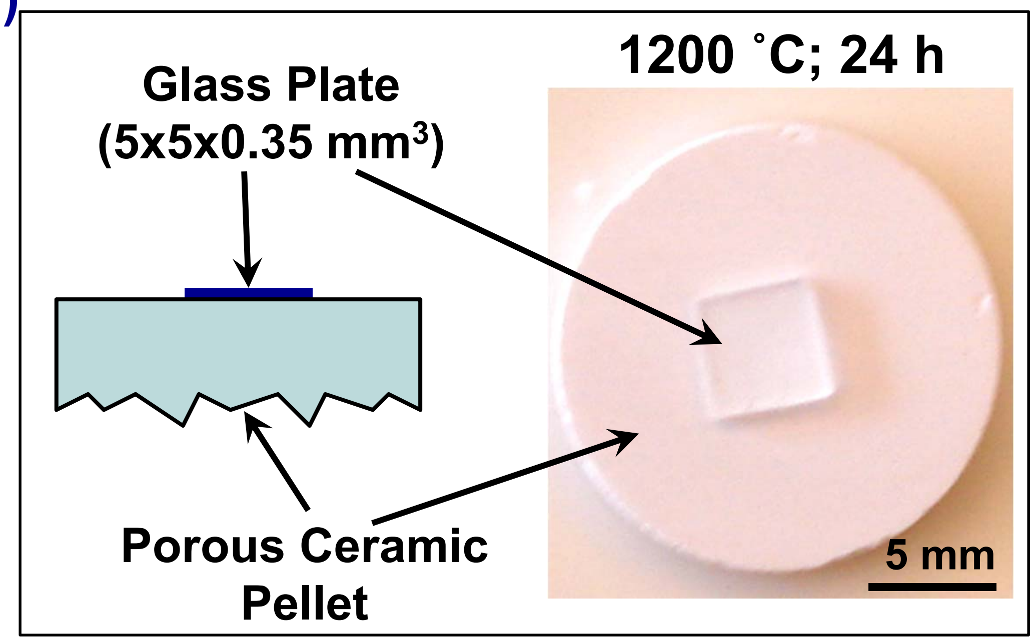
- Sand/Ash Melts into a Glass
- Infiltrates Pores/Cracks
- Penetrates Grain Boundaries
- Dissolution of $Gd_2Zr_2O_7$ and Reprecipitation of ZrO_2(*ss*)
- Glass Accumulates Gd, Large Amount of "Solute" (Gd_2O_3) in TBC: Gd:Zr :: 1:1
- Shifts Glass Composition
- Rapid Crystallization of Apatite Phases
- Arrests Front



TBC Ceramics/CMAS Glass Interactions: "Model" Study

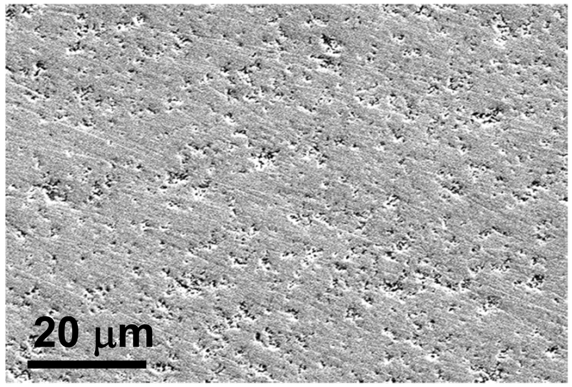
* Effects of "Solute" Type, Concentration in ZrO_2-Based TBC Ceramics
  - "Solute" Type: Y, Gd, Yb, Y+Al+Ti
  - Concentration: Low, High
* Partially Sintered Ceramic Pellets (~15% Porosity)
  - No Substrate: Higher Heat Treatment Temperatures Possible
  - Porous: Avoid Possible Dilatation Issues in Dense Ceramics
  - Better Control Over Composition, Microstructure
* CMAS Sand Glass Plate (wt%)

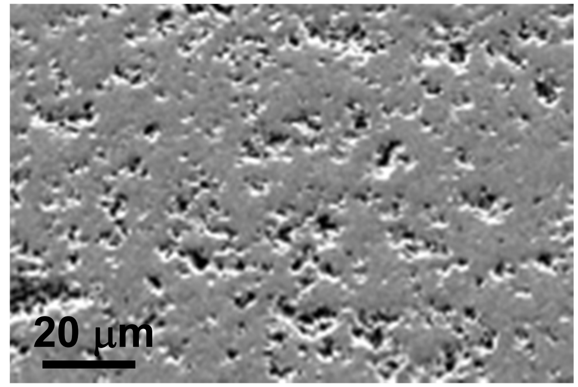
SiO_2: 53.3
CaO: 37.1
Al_2O_3: 7.1
MgO: 3.5



TBC Ceramics/CMAS Glass Interactions

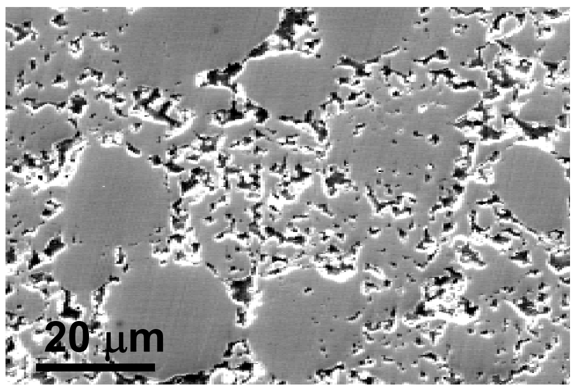
7YSZ
3.9 m% Y_2O_3
Y:Zr::0.08:1

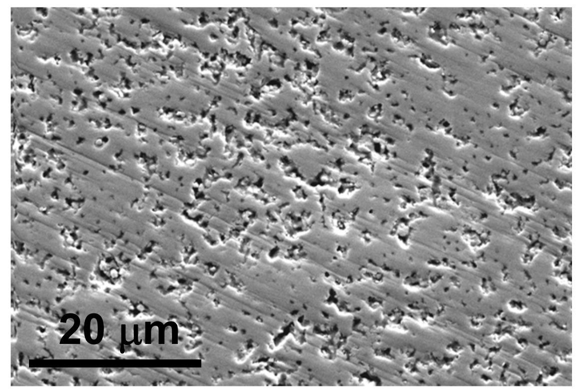




48YSZ
33.3 m% Y_2O_3
Y:Zr::1:1

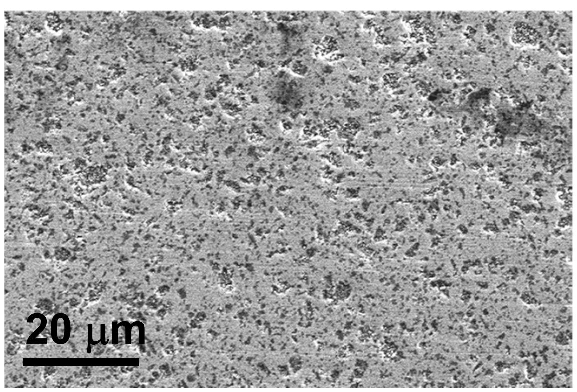
6.8GdSZ
3.9 m% Gd_2O_3
Gd:Zr::0.08:1

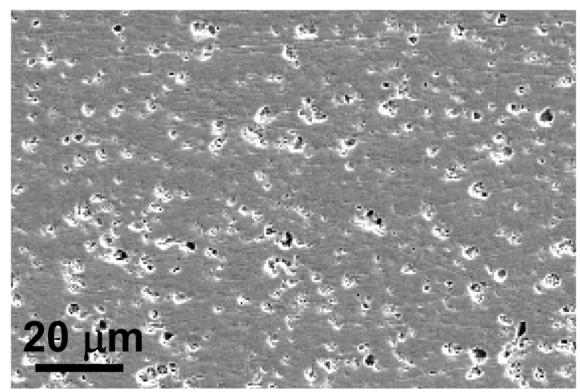




$Gd_2Zr_2O_7$
33.3 m% Gd_2O_3
Gd:Zr::1:1

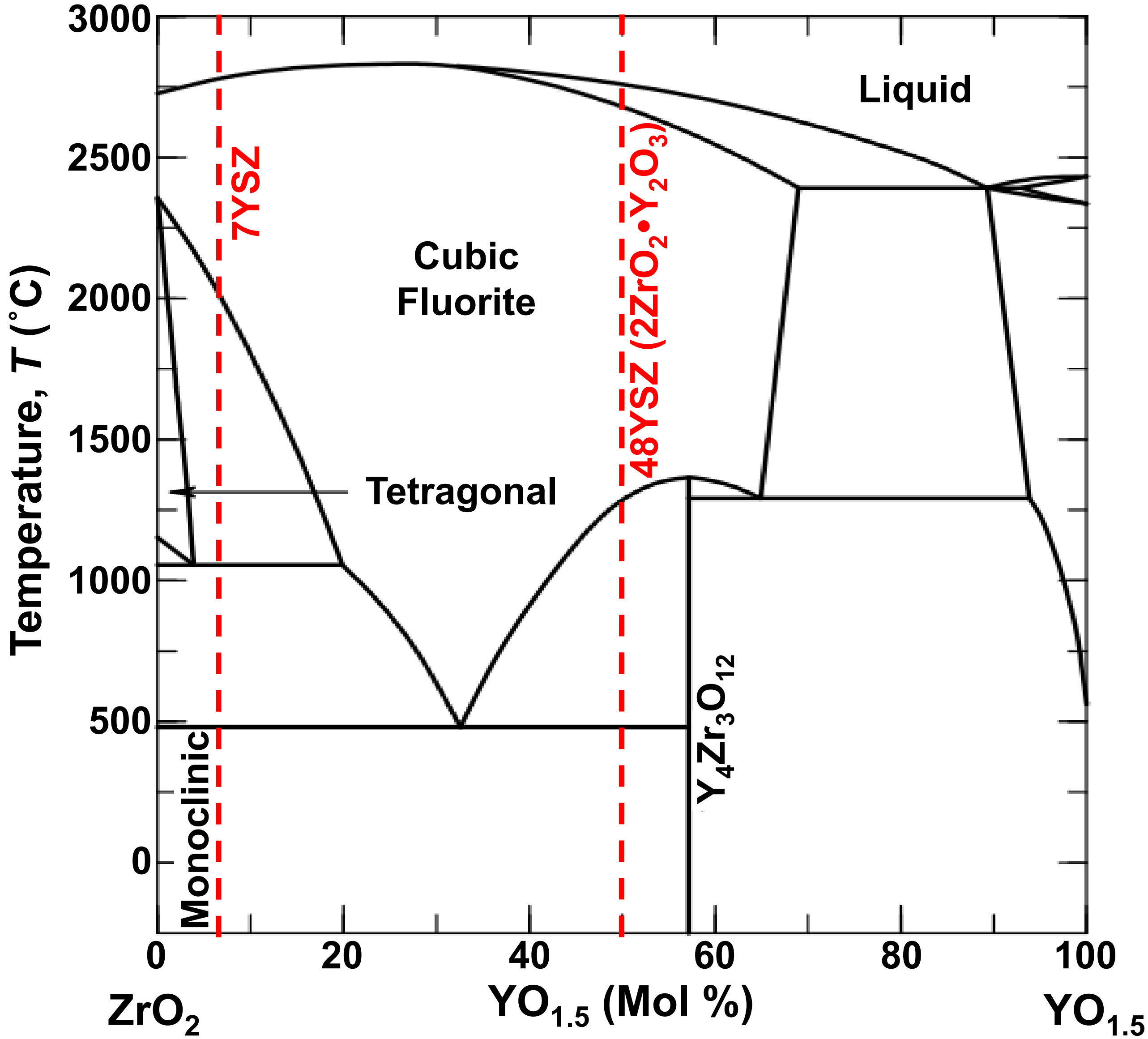
7YSZ+Al+Ti
Y+Al+Ti:Zr::
0.73:1





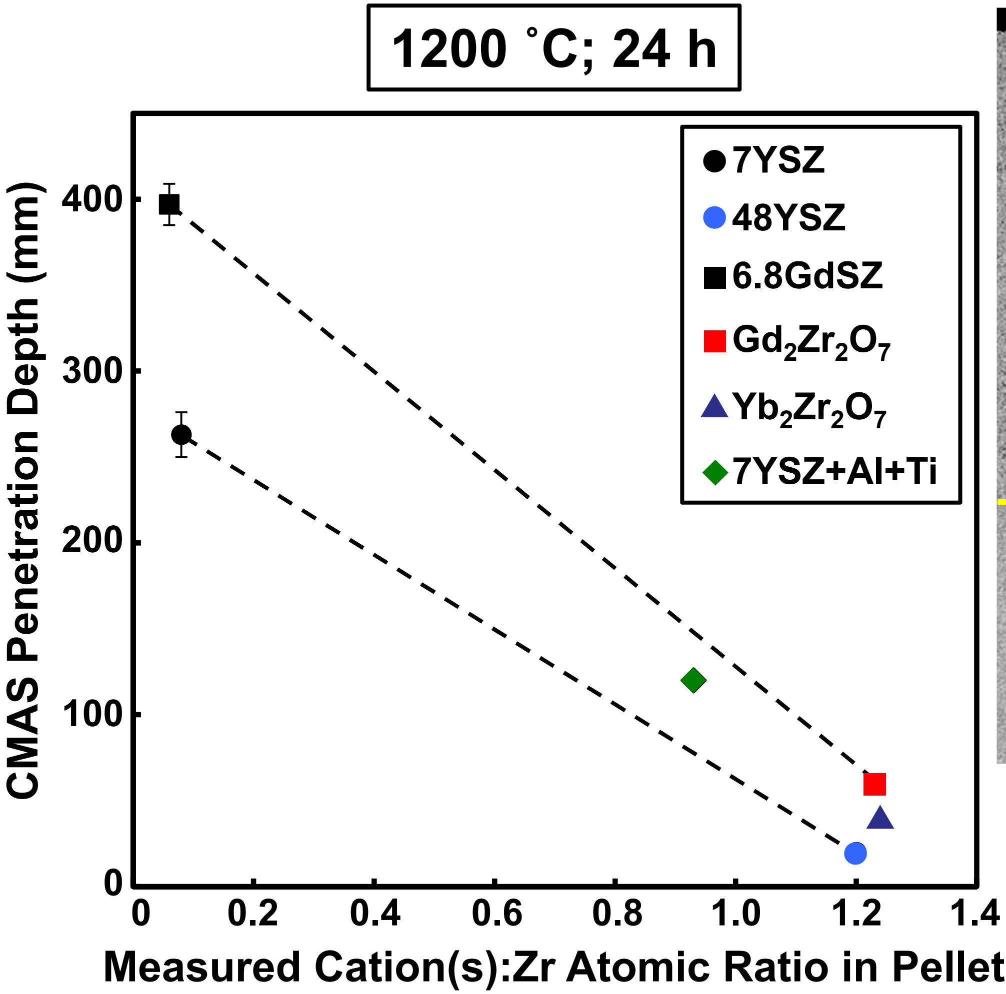
$Yb_2Zr_2O_7$
33.3 m% Yb_2O_3
Yb:Zr::1:1

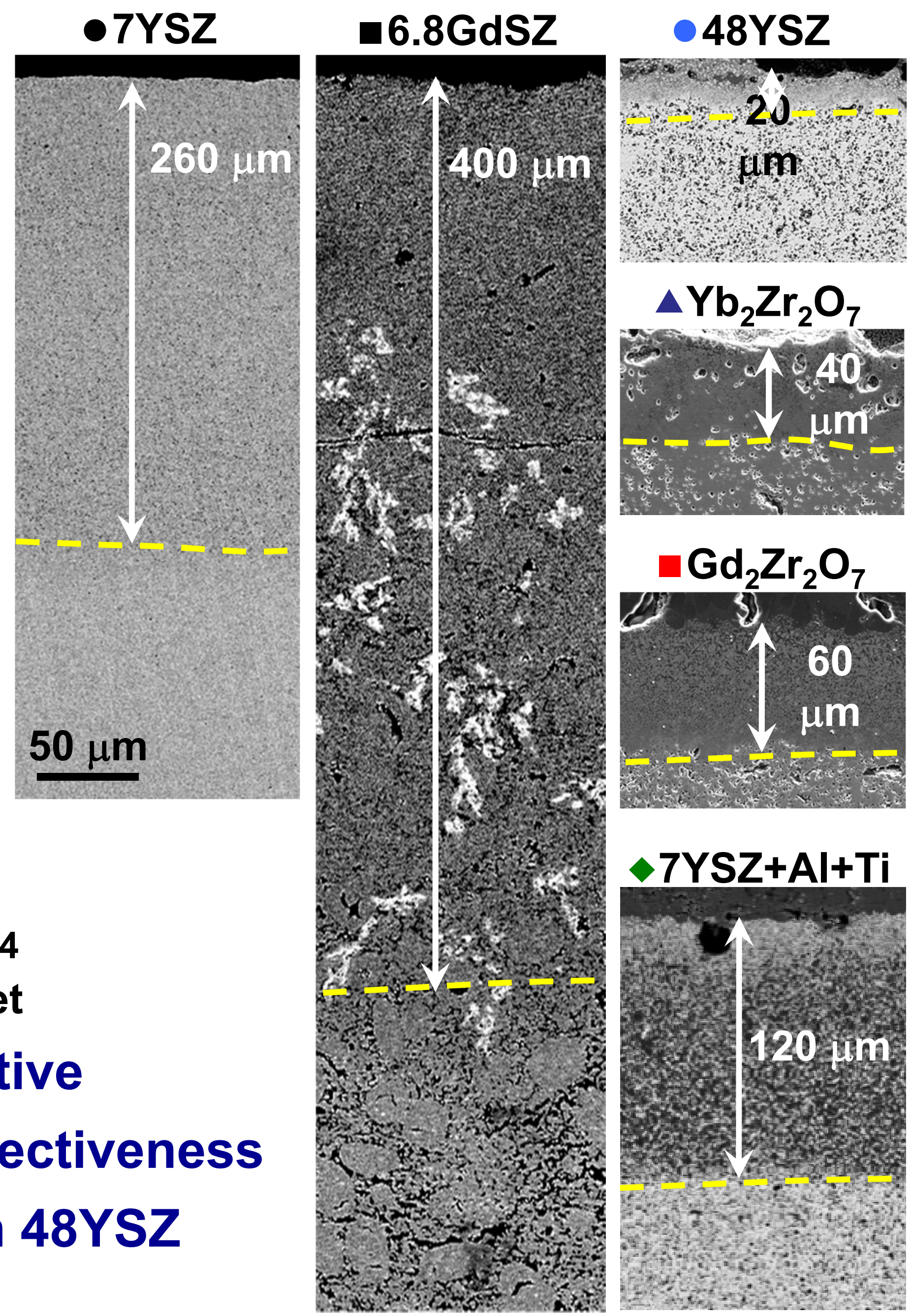
48YSZ ($2ZrO_2 \cdot Y_2O_3$)



Yokokawa *et al.*, 2001

TBC Ceramics/CMAS Glass Interactions





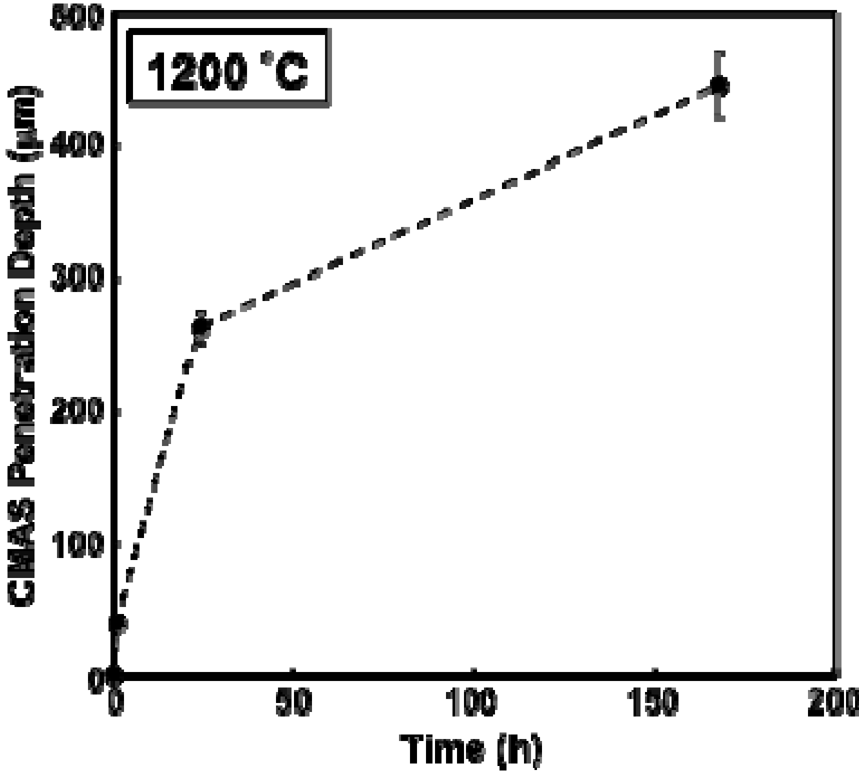
* Solute Concentration Most Effective

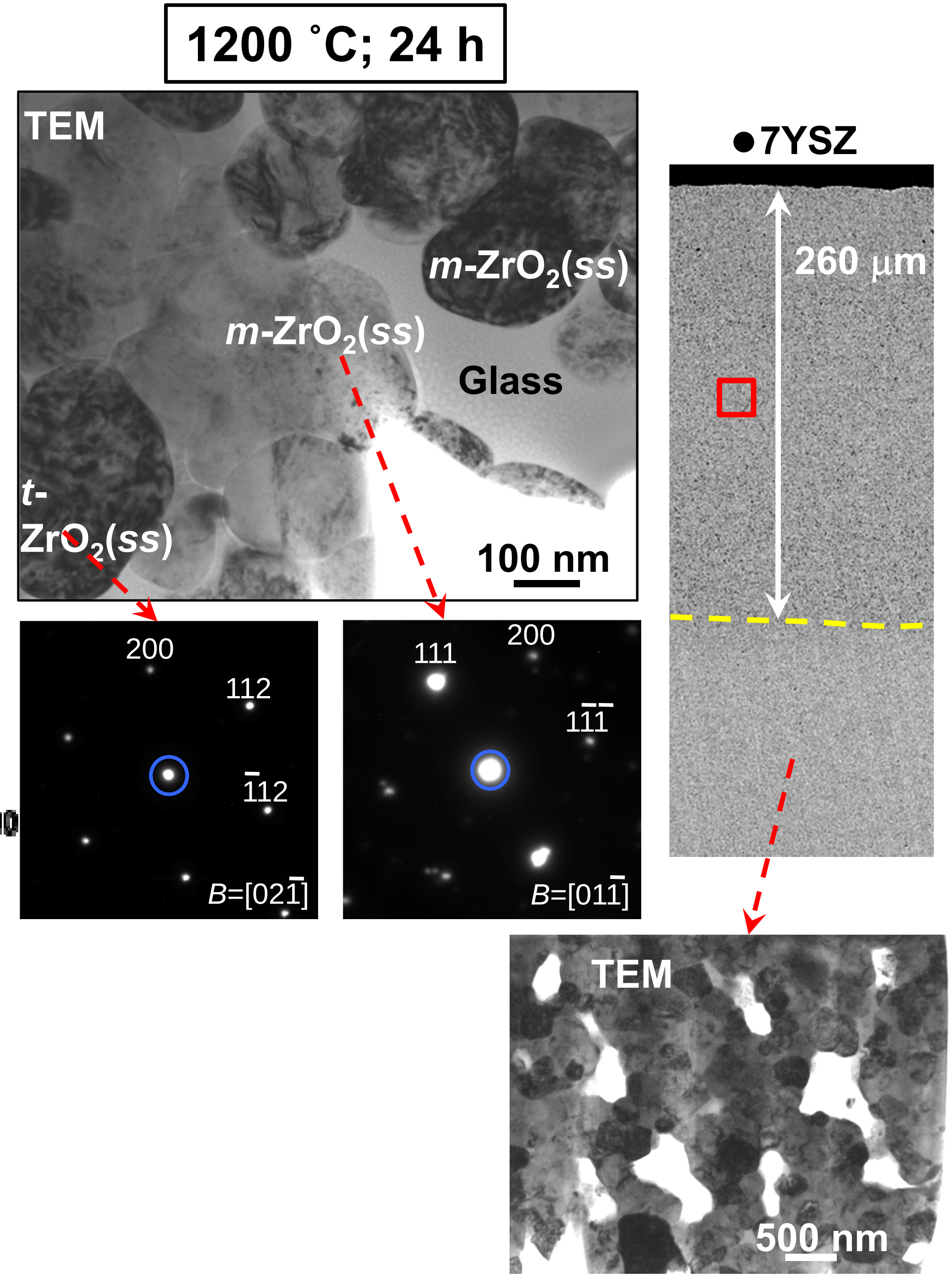
* At High Conc.: Gd < Yb < Y in Effectiveness

* Almost Complete Suppression in 48YSZ

Acta Mat. 2012

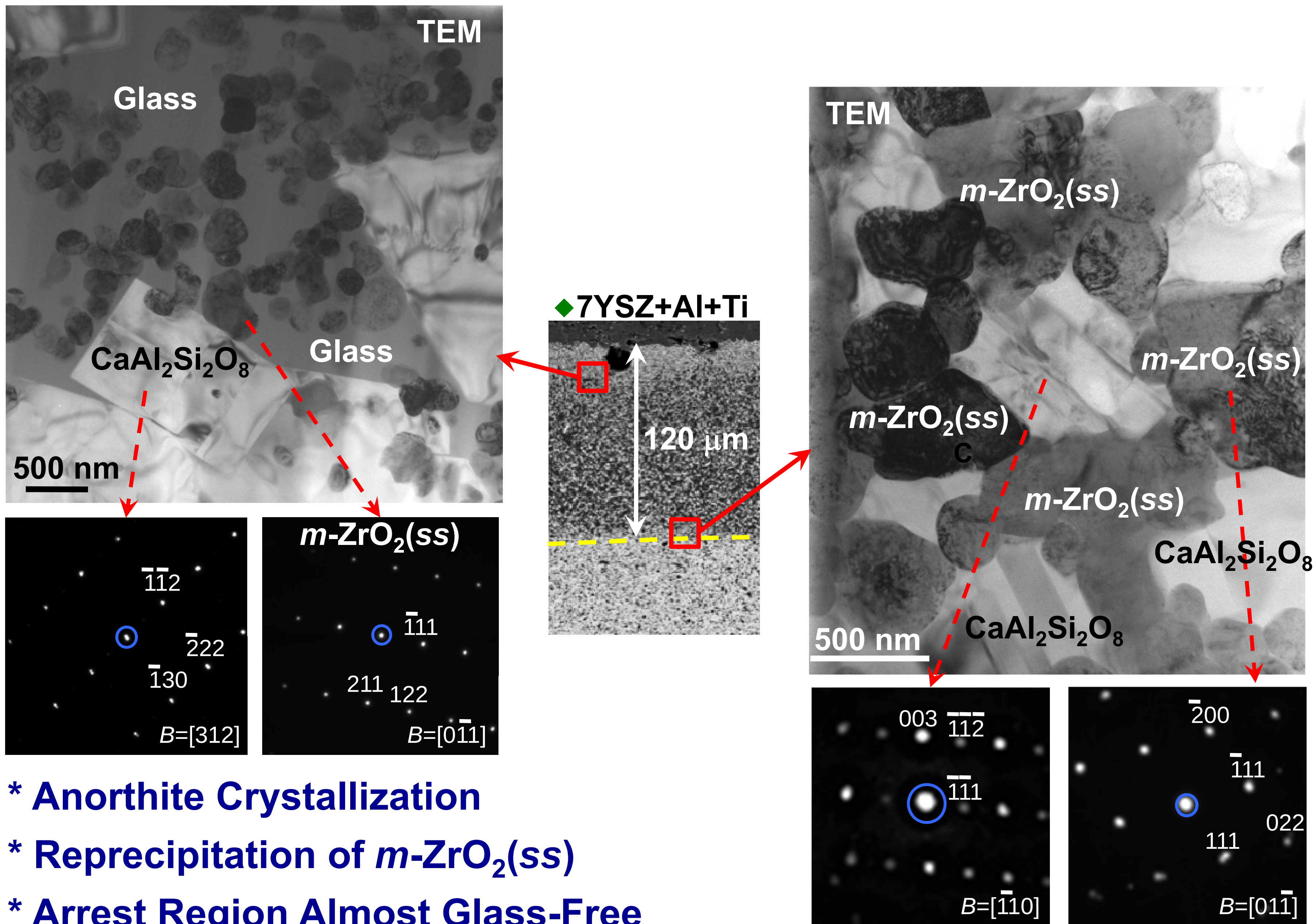
7YSZ





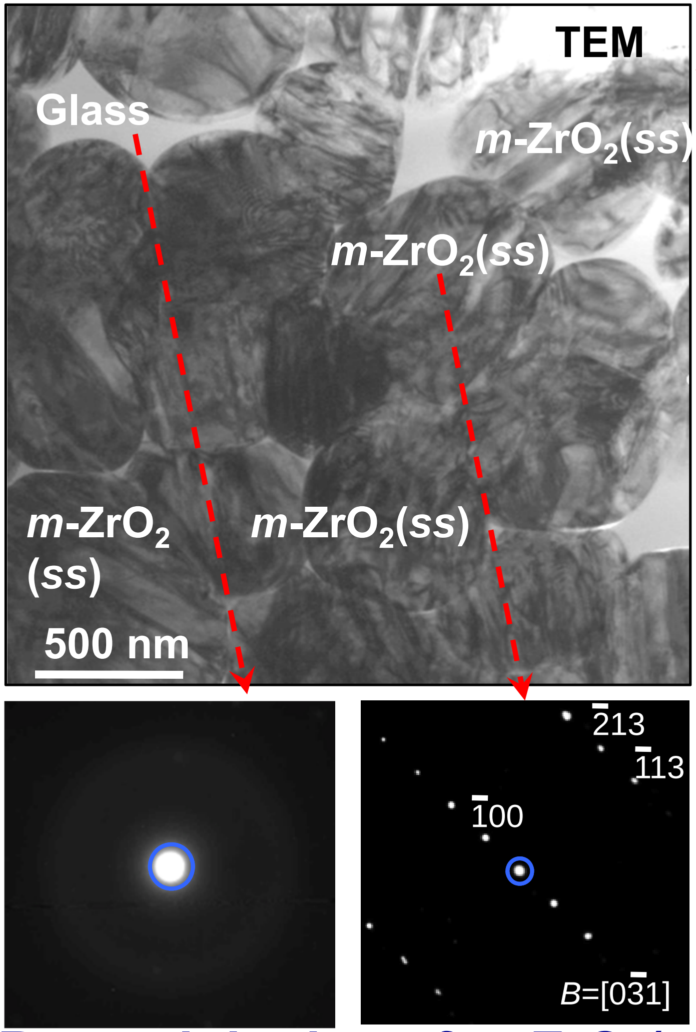
* **CMAS Penetration Continues**
* **No Detectable Other Phases**

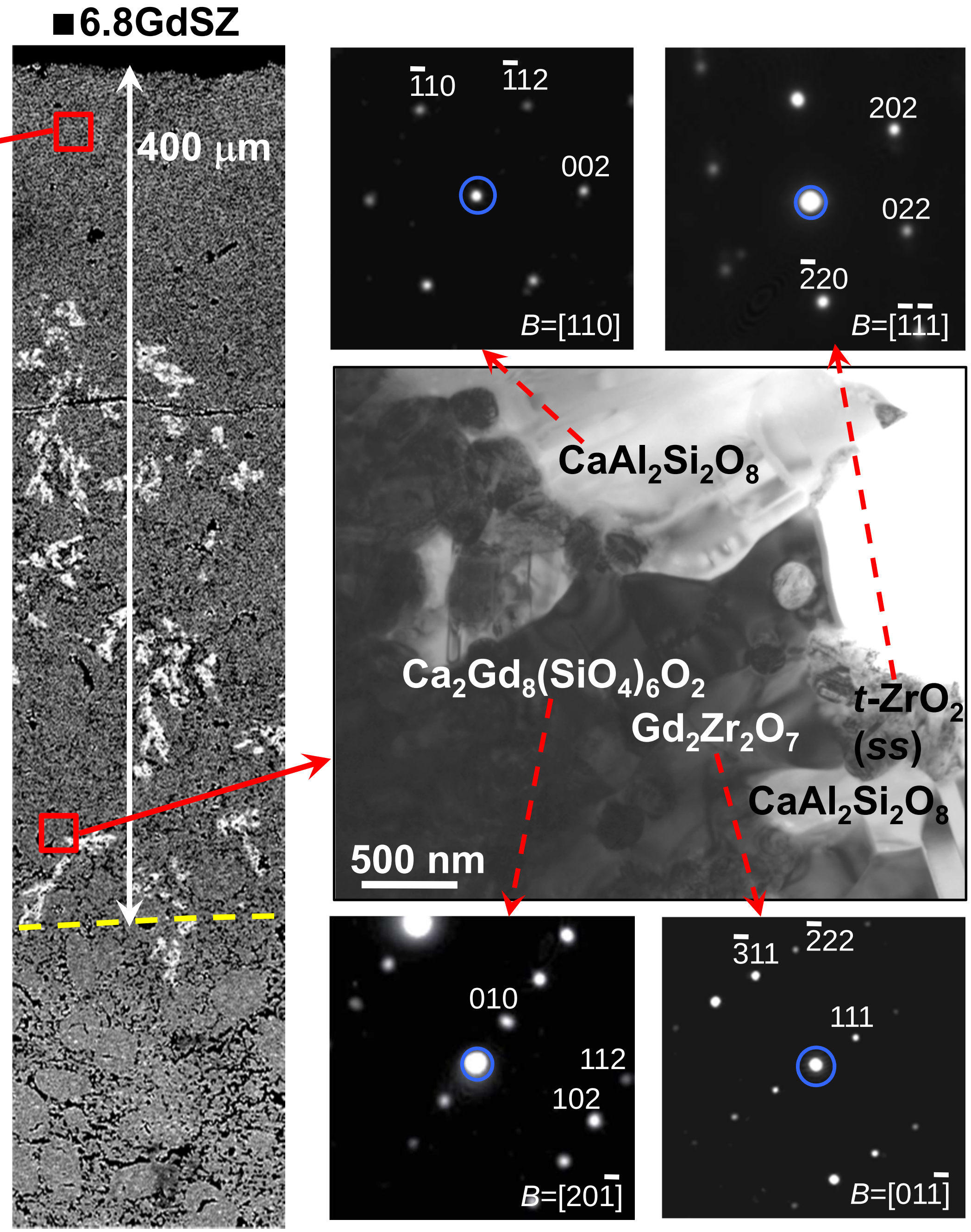
7YSZ+Al+Ti



* **Anorthite Crystallization**
* **Reprecipitation of *m*-ZrO_2(*ss*)**
* **Arrest Region Almost Glass-Free**

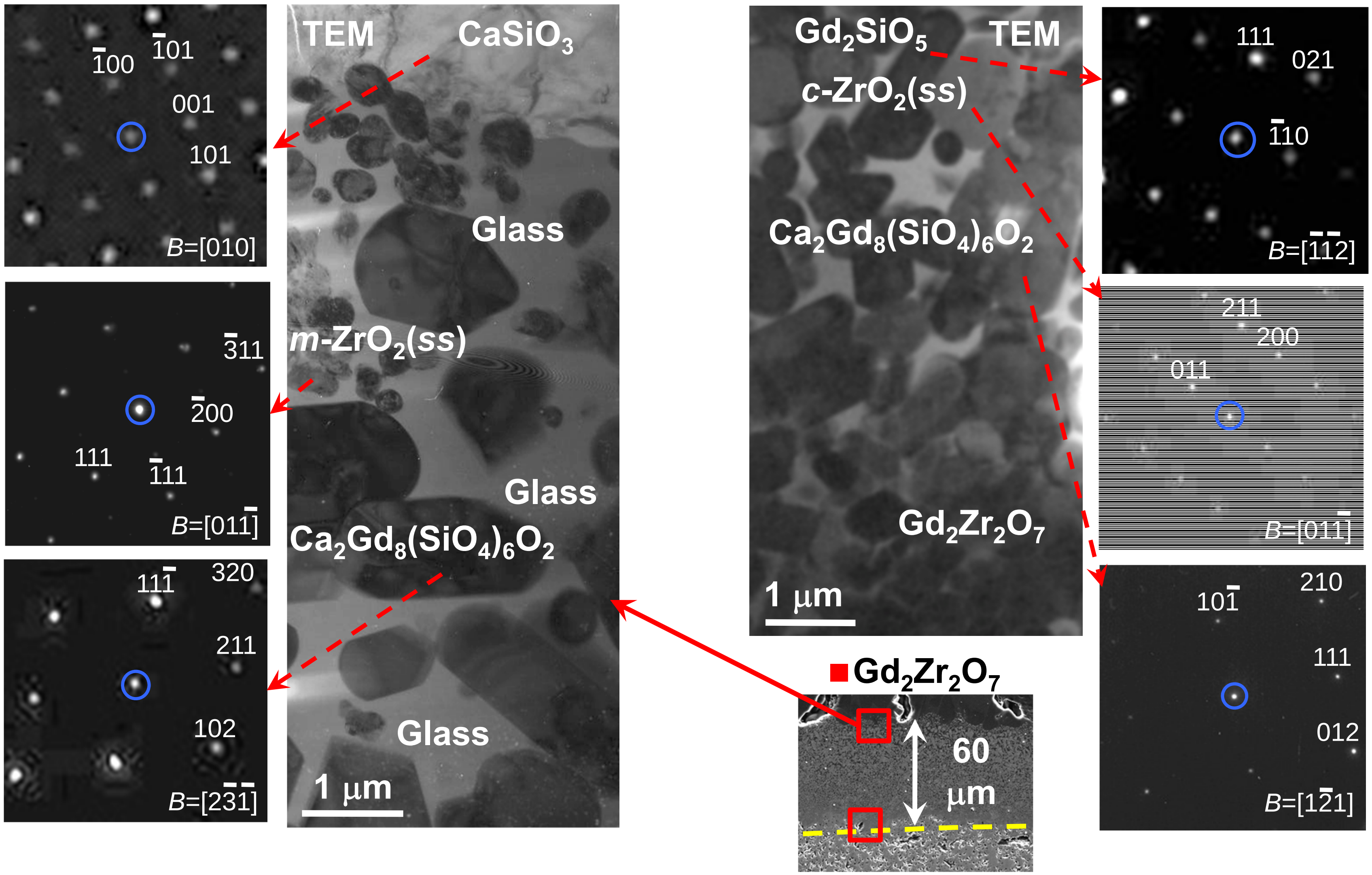
6.8GdSZ





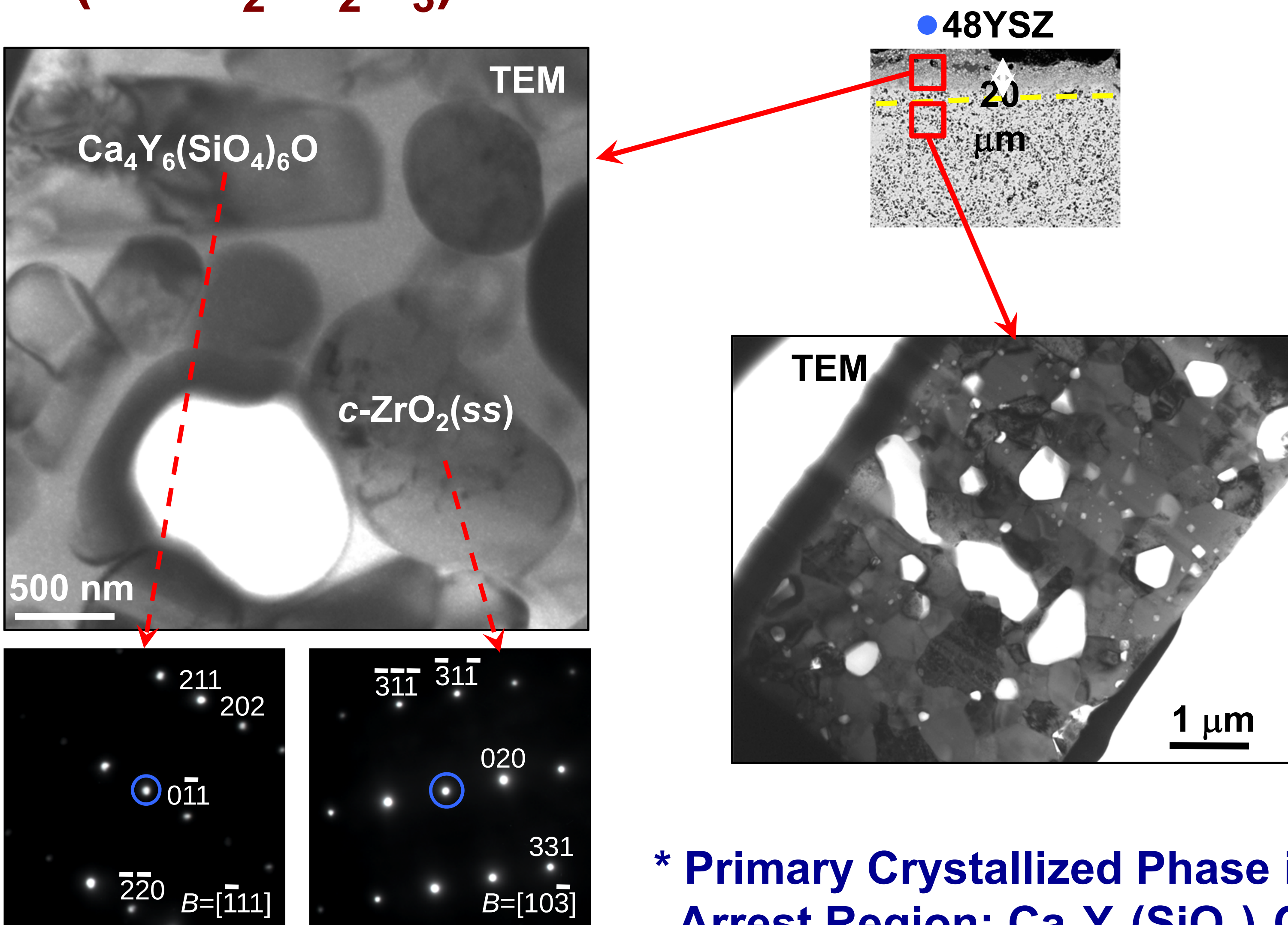
* Reprecipitation of m-ZrO_2(ss)
* Crystallization of Gd-Apatite and Anorthite
* Gd Decreases Glass Viscosity

$Gd_2Zr_2O_7$



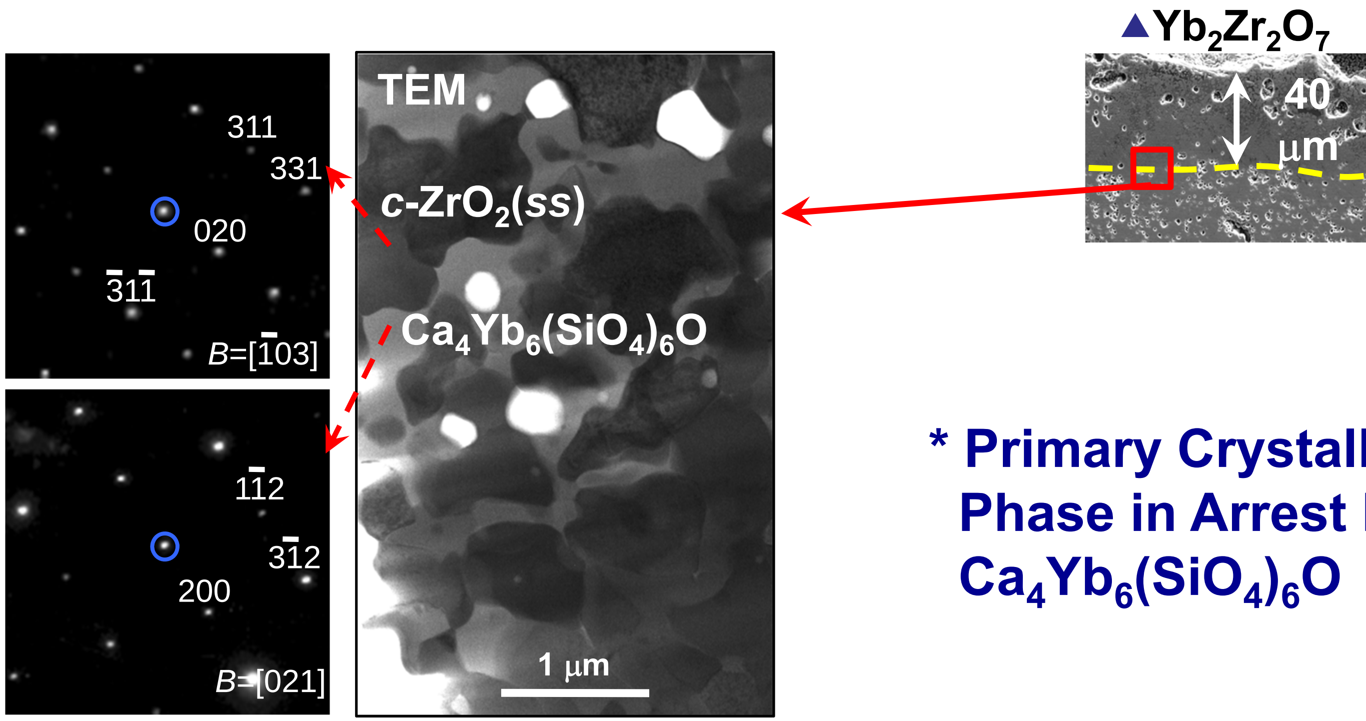
*** Primary Crystallized Phase in Arrest Region: $Ca_2Gd_8(SiO_4)_6O_2$**

48YSZ ($2ZrO_2 \cdot Y_2O_3$)



*** Primary Crystallized Phase in Arrest Region: $Ca_4Y_6(SiO_4)_6O$**

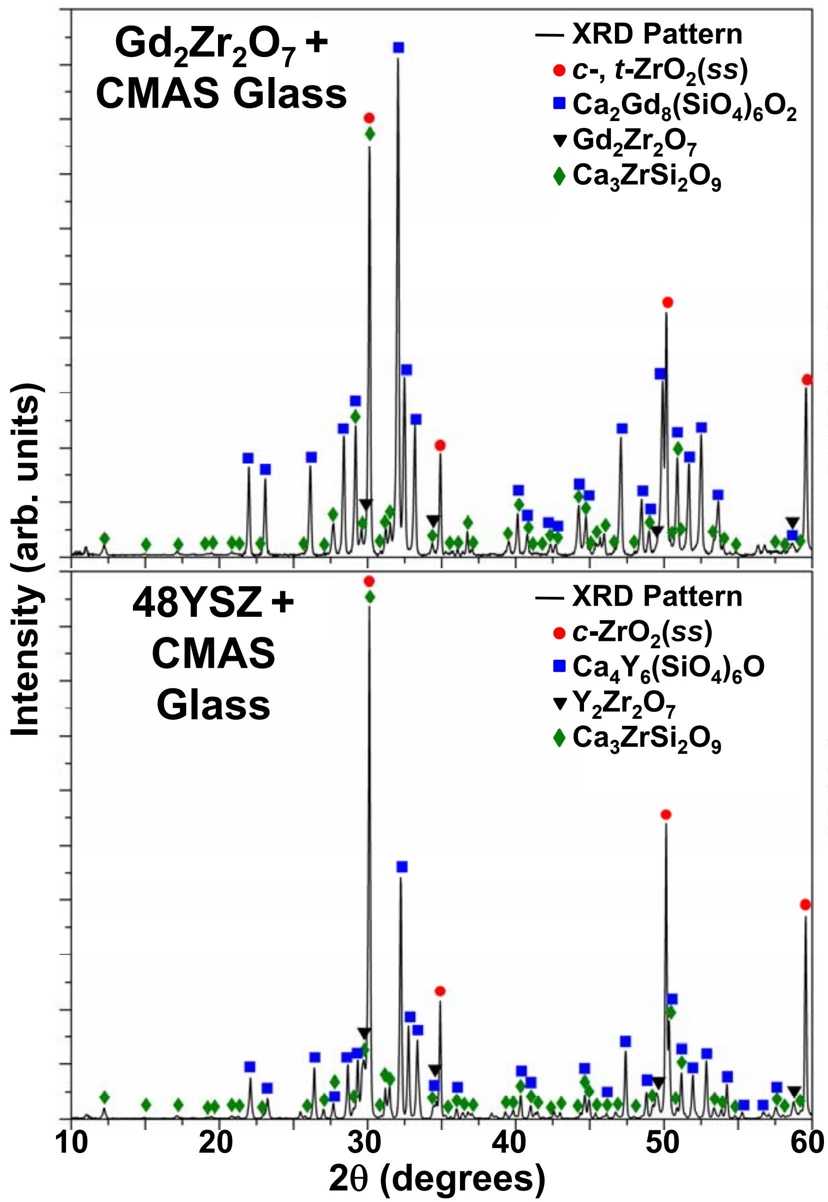
$Yb_2Zr_2O_7$

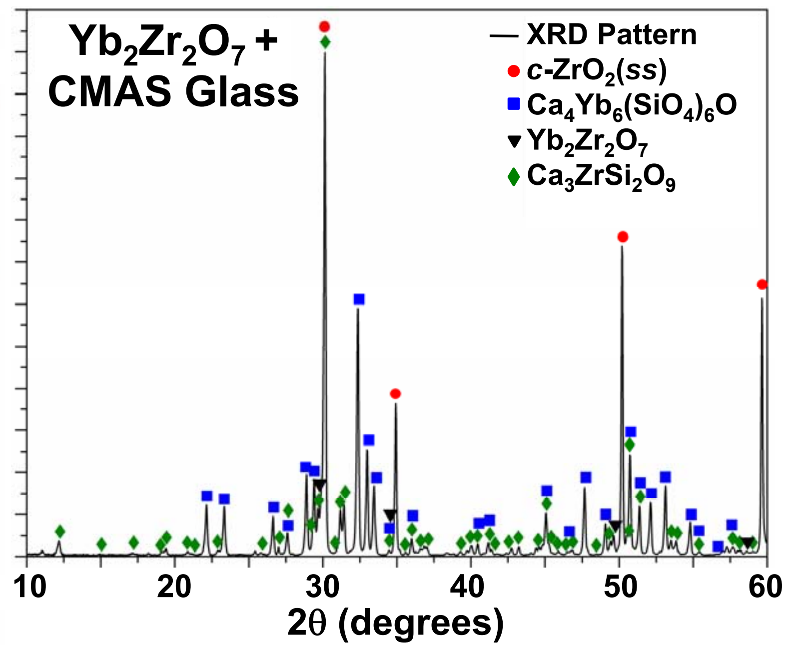


*** Primary Crystallized Phase in Arrest Region: $Ca_4Yb_6(SiO_4)_6O$**

TBC Ceramics/CMAS Glass Interactions

* XRD of Powder Mixtures Heat-Treated at 1200 ˚C, 24 h
* Confirm Primary Crystallized Phases





Silicate Apatites

CN=9 CN=7

$$A^{I}_{4}A^{II}_{6}(SiO_4)_6O_x$$

* $Gd_2Zr_2O_7$
  - Forms $Ca_2Gd_8(SiO_4)_6O_2$
  - A^I: All (2) Ca^{2+} + 2 Gd^{3+}
  - A^{II}: 6 Gd^{3+}
  - $(Ca_2Gd_2)Gd_6(SiO_4)_6O_2$
  - Need 8 Gd Atoms
* 48YSZ
  - Forms $Ca_4Y_6(SiO_4)_6O$
  - A^I: All (4) Ca^{2+}
  - A^{II}: All (6) Y^{3+}
  - Need 6 Y Atoms
* $Yb_2Zr_2O_7$
  - Forms $Ca_4Yb_6(SiO_4)_6O$
  - A^I: All (4) Ca^{2+}
  - A^{II}: All (6) Yb^{3+}
  - Need 6 Yb Atoms, But Apatite Crystallization Propensity Decreases with RE Size

(Quintas *et al.*, 2008)

Cation "A"	A^I Site (pm)	A^{II} Site (pm)
Lu^{3+}	103	-
Yb^{3+}	104	93
Er^{3+}	106	95
Y^{3+}	108	96
Gd^{3+}	111	100
Sm^{3+}	113	102
Nd^{3+}	116	-
Ca^{2+}	118	106
Ce^{3+}	120	107
La^{3+}	122	110

Harder to Crystallize Apatite ↑

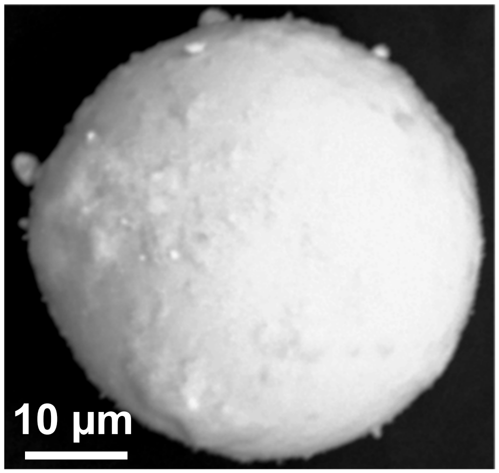
↓ Need More RE^{3+} to Form Apatite

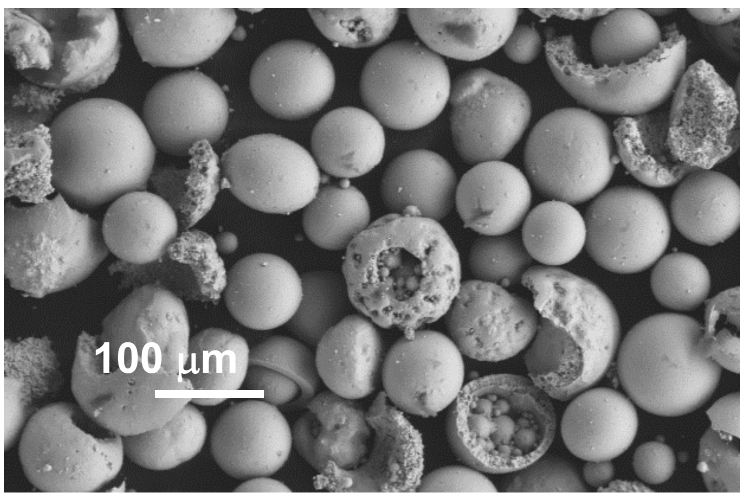
Shannon, 1976

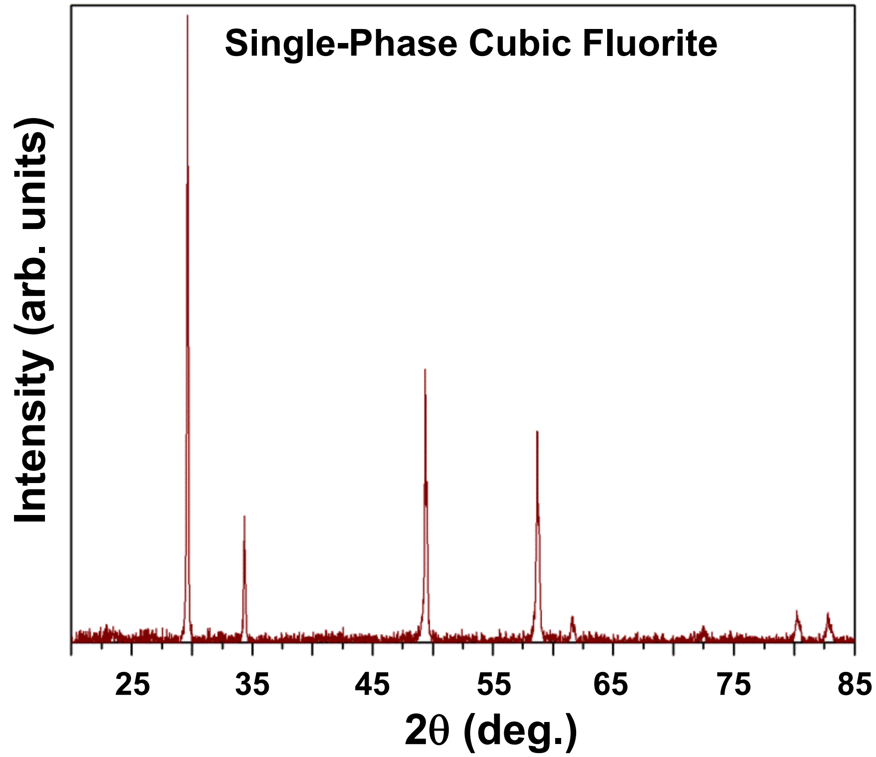
APS 48YSZ ($2ZrO_2 \bullet Y_2O_3$) TBCs: Processing

*** Developed Powders in Collaboration with Sulzer Metco**

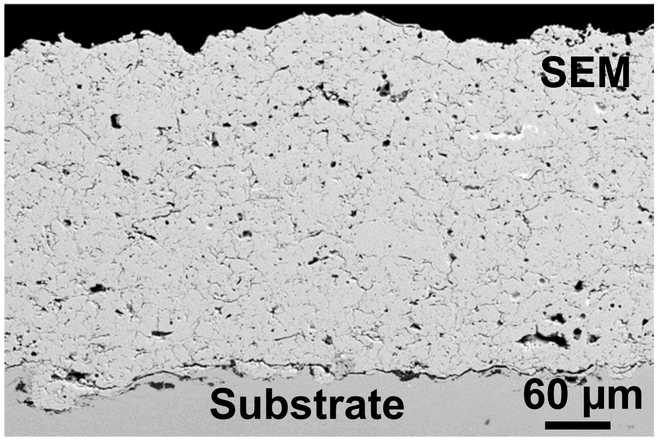
- Highly Sprayable Powders (Kilograms)

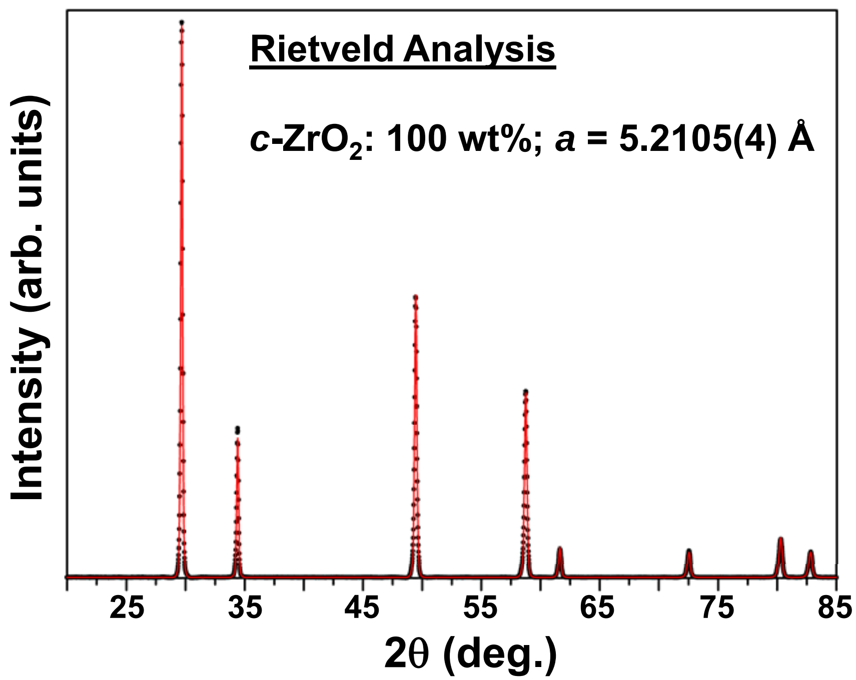


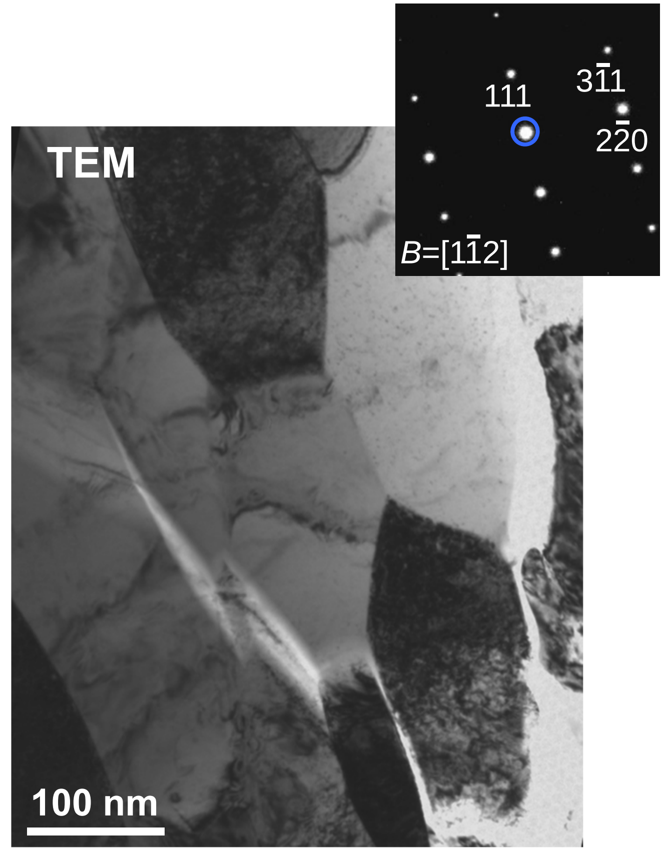




APS 48YSZ ($2ZrO_2 \bullet Y_2O_3$) TBCs: Processing

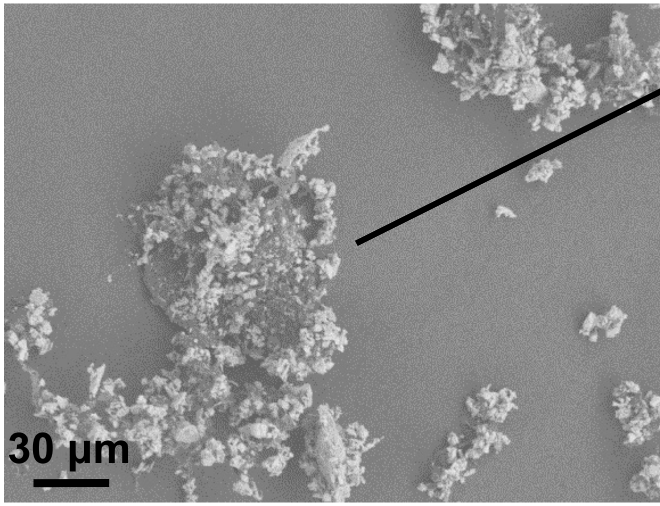






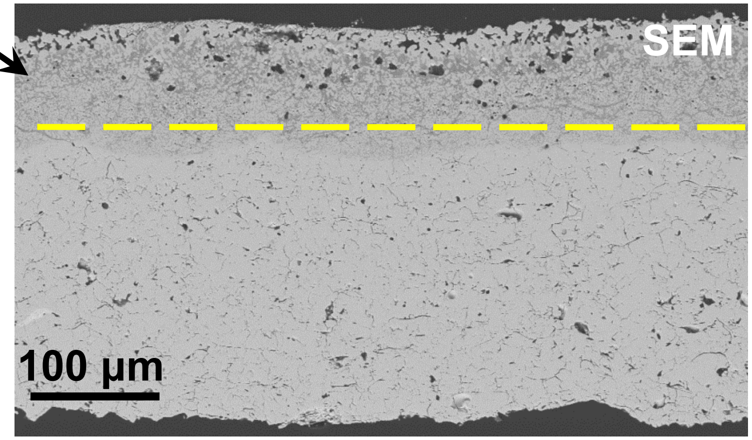
- * Thermal Conductivity (25 ˚C): 0.86 $W.m^{-1}.K^{-1}$
- * Toughness Measurements on Dense Ceramics Underway

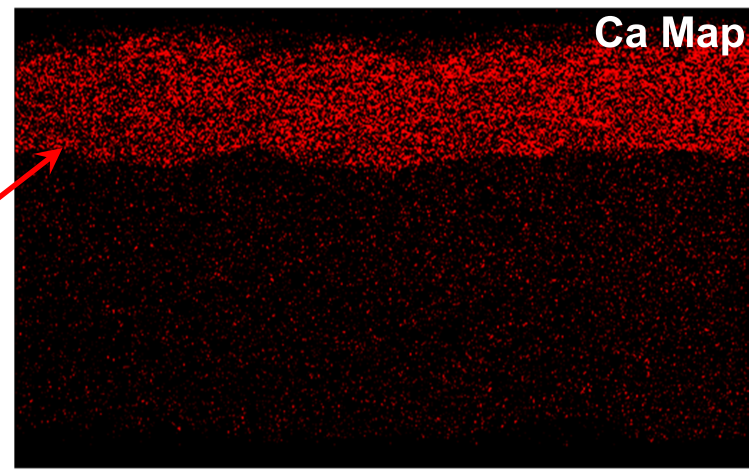
APS TBCs 48YSZ: Lignite Fly Ash



Ball-Milled Fly Ash ($30\ mg.cm^{-2}$)

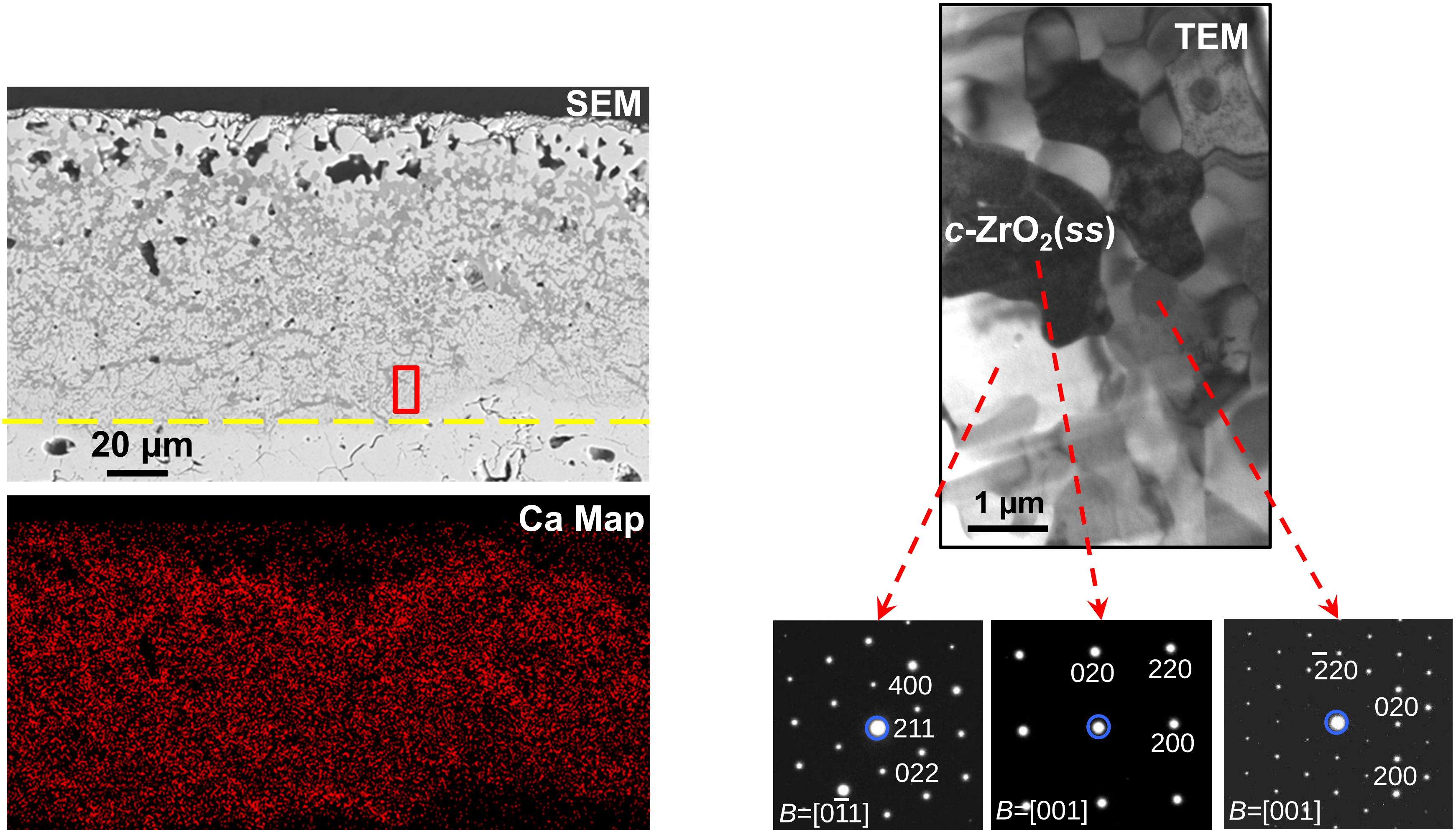
1300 ˚C, 24 h





Ash-Front Arrested (~5%)

APS TBCs 48YSZ: Lignite Fly Ash

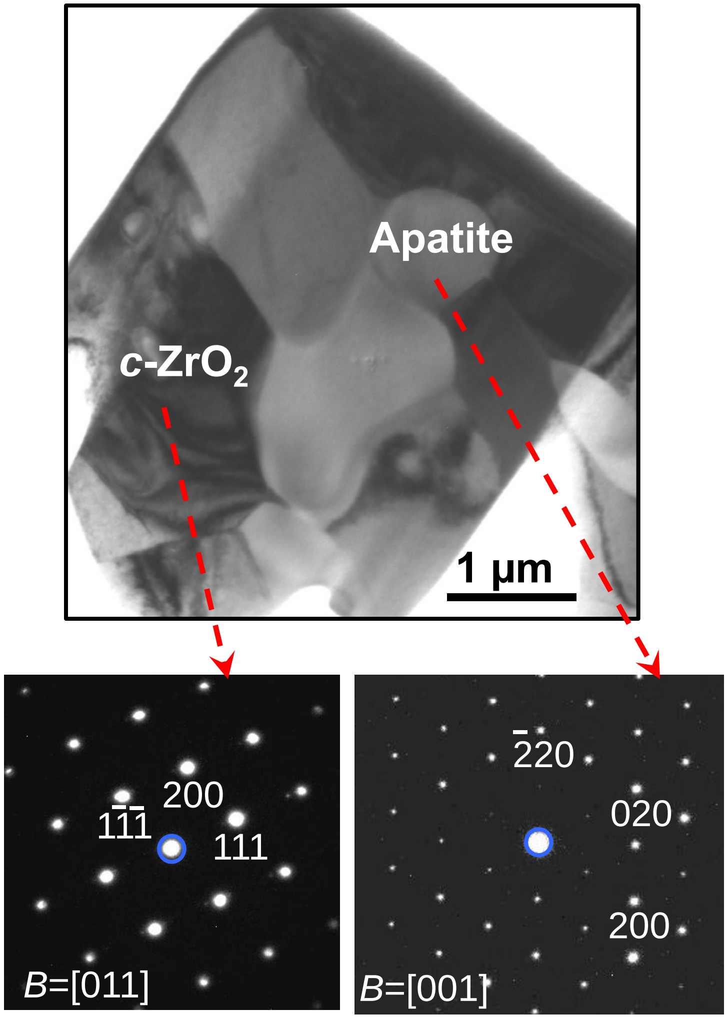


$Ca_{12}Al_{14}O_{33}$ (Mayenite)

c-ZrO_2(*ss*) (Fluorite)

$Ca_4Y_6(SiO_4)_6O$ (Apatite)

APS TBCs 48YSZ: Lignite Fly Ash

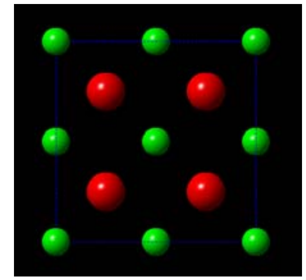


c-ZrO_2(ss)
(Fluorite)

$Ca_4Y_6(SiO_4)_6O$
(Apatite)

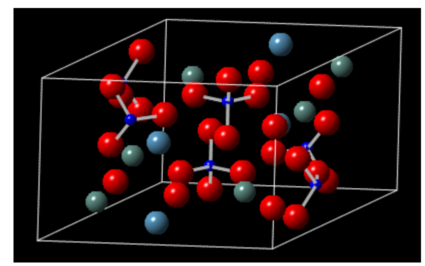
c-ZrO_2

Elemen t	Th. At.%	Actual At.%
Ca		2.9
Y	50.0	35.9
Zr	50.0	61.2



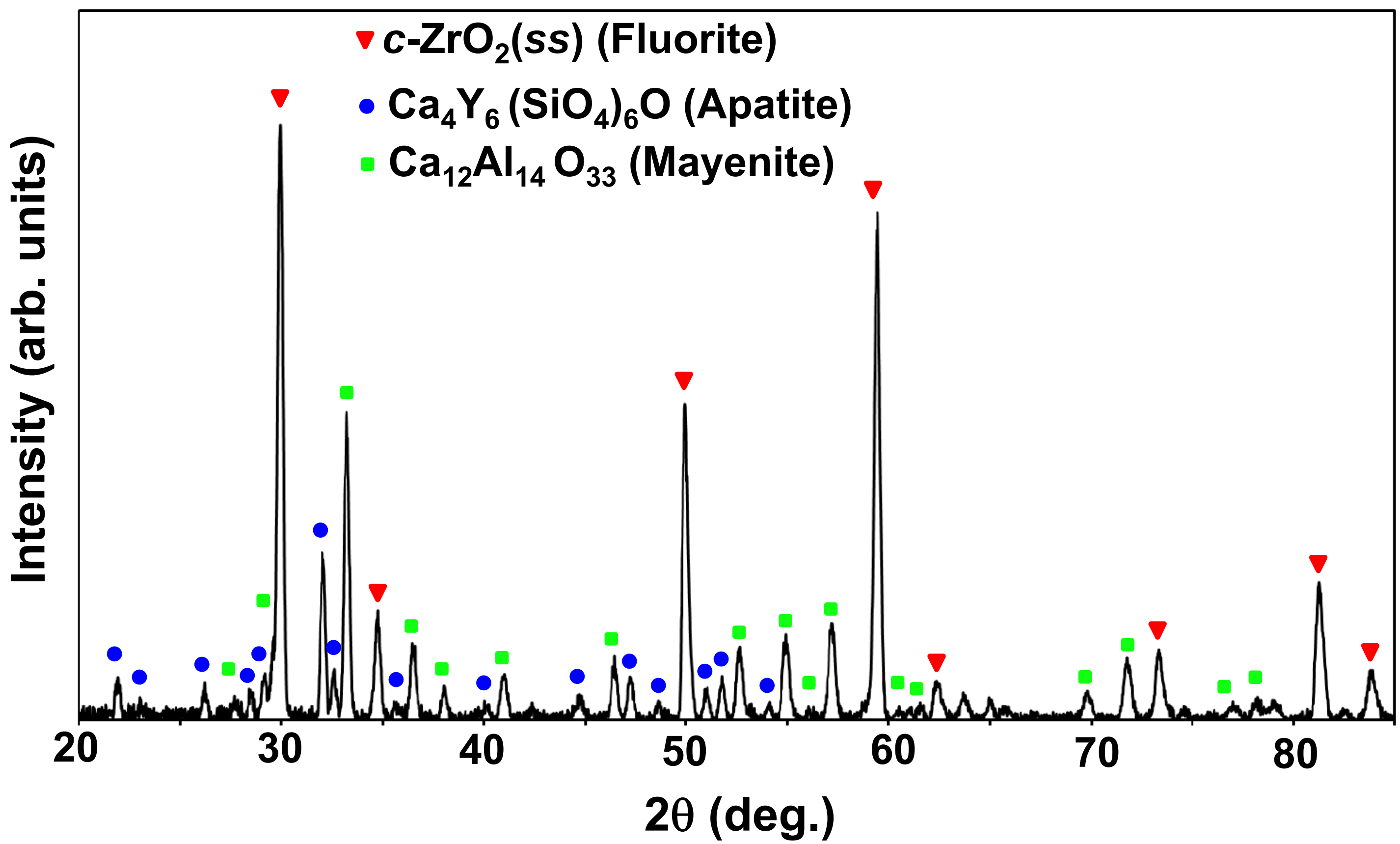
$Ca_4Y_6(SiO_4)_6O$

Elemen t	Th. At.%	Actual At.%
Si	37.5	43.5
Ca	25.0	14.4
Y	37.5	42.1



48YSZ - Lignite Fly Ash Interaction

* XRD of Powder Mixtures (50:50) Heat-Treated at 1200 ˚C, 24 h
* Confirmed Primary Crystallized Phases



Summary

* Molten CMAS attack of TBCs is a growing issue with rising temperatures and environmental severity in gas-turbine engines

* 7YSZ TBCs are not suited to repel CMAS attack
  - Low Y:Zr atomic ratio of 0.083:1 does not alter CMAS

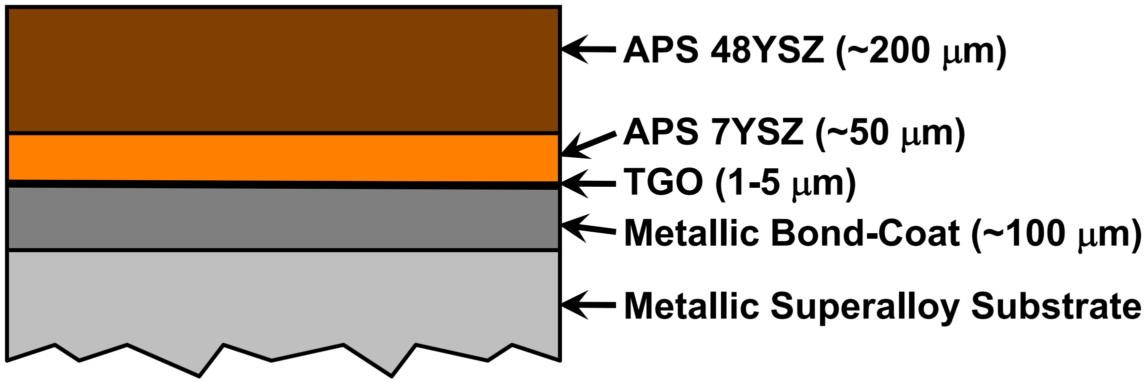
* Damage mitigation via TBCs composition engineering
  - New APS TBCs of 7YSZ with Al^{3+} and Ti^{4+} (Y"Al+Ti:Zr::0.73:1)
  - APS TBCs of $Gd_2Zr_2O_7$ (Gd:Zr::1:1)

* Porous TBC ceramics/CMAS interactions: "model" studies
  - Effect of "solute" type in ZrO_2: Y, Gd, Yb, Y+Al+Ti
  - Effect of concentration: low, high
  - Near-complete CMAS penetration suppression in $2ZrO_2{\bullet}Y_2O_3$ (48YSZ) solid solution composition
  - Guidelines for design of future TBCs

* New APS 48YSZ TBCs from commercially developed powders
  - CMAS interaction studies show arrest of penetration front

Ongoing and Future Work

* Two-Layer and Graded APS 48YSZ-7YSZ TBCs Optimization
    - CMAS-Resistant Top Layer, High Toughness Bottom Layer



* Toughness, High-Temperature Thermal Conductivity Measurements

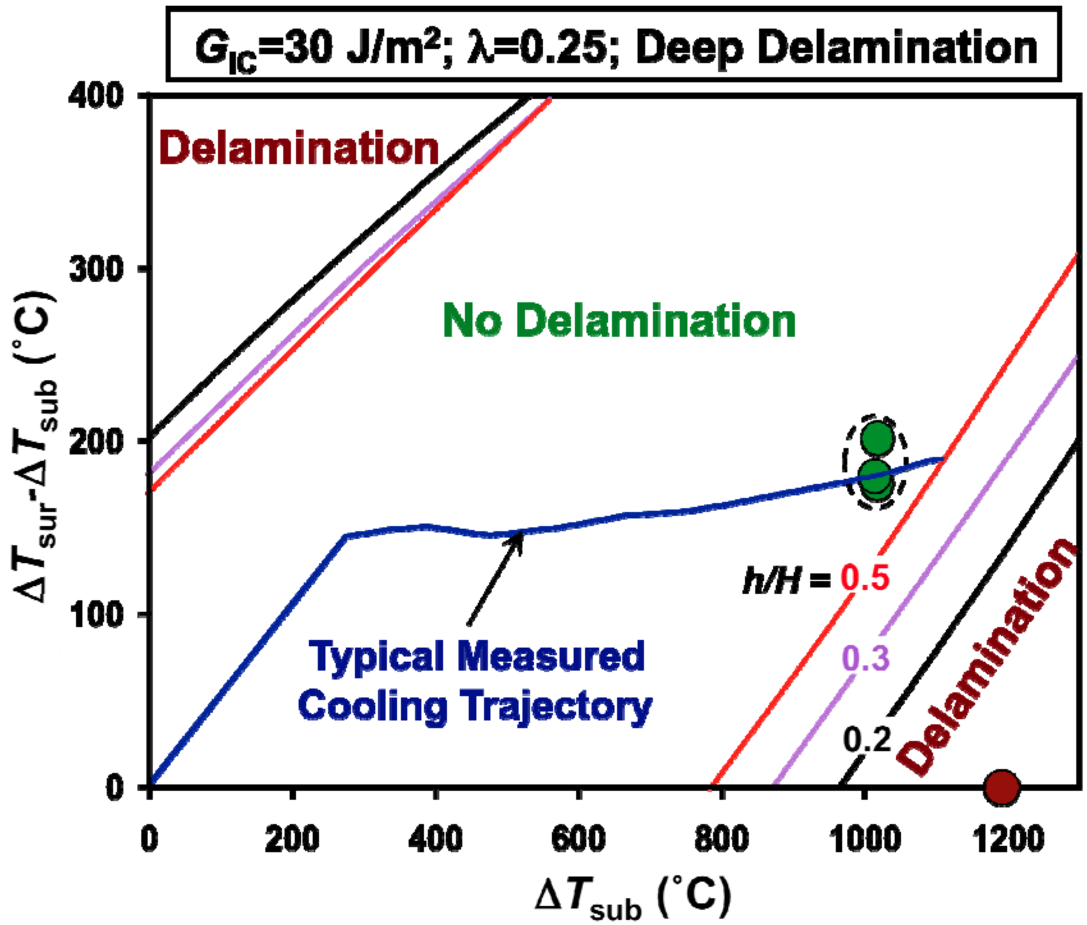
* Lignite Coal Fly Ash Testing Against Optimized TBCs

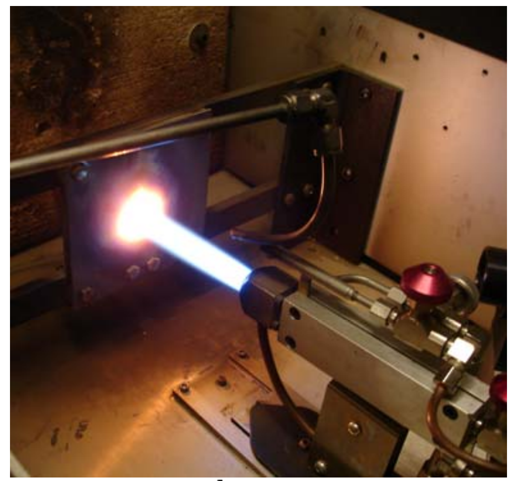
* Understanding Mitigation Mechanisms
    - *In Situ* Raman Spectroscopy Studies
    - DTA/DSC Studies of CMAS/TBCs Interactions and Crystallization

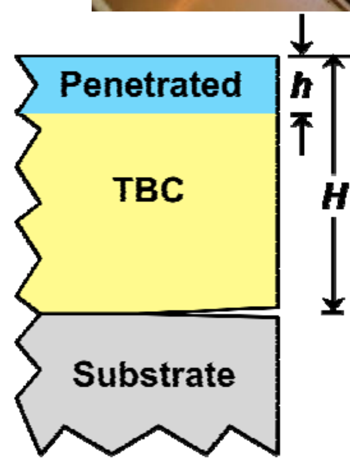
Ongoing and Future Work

* 7YSZ, 48YSZ, and Layered/Graded TBCs Testing Under Gradient with CMAS

* Mechanisms-Based Analysis of CMAS-Induced TBC Failure Under Thermal Gradient

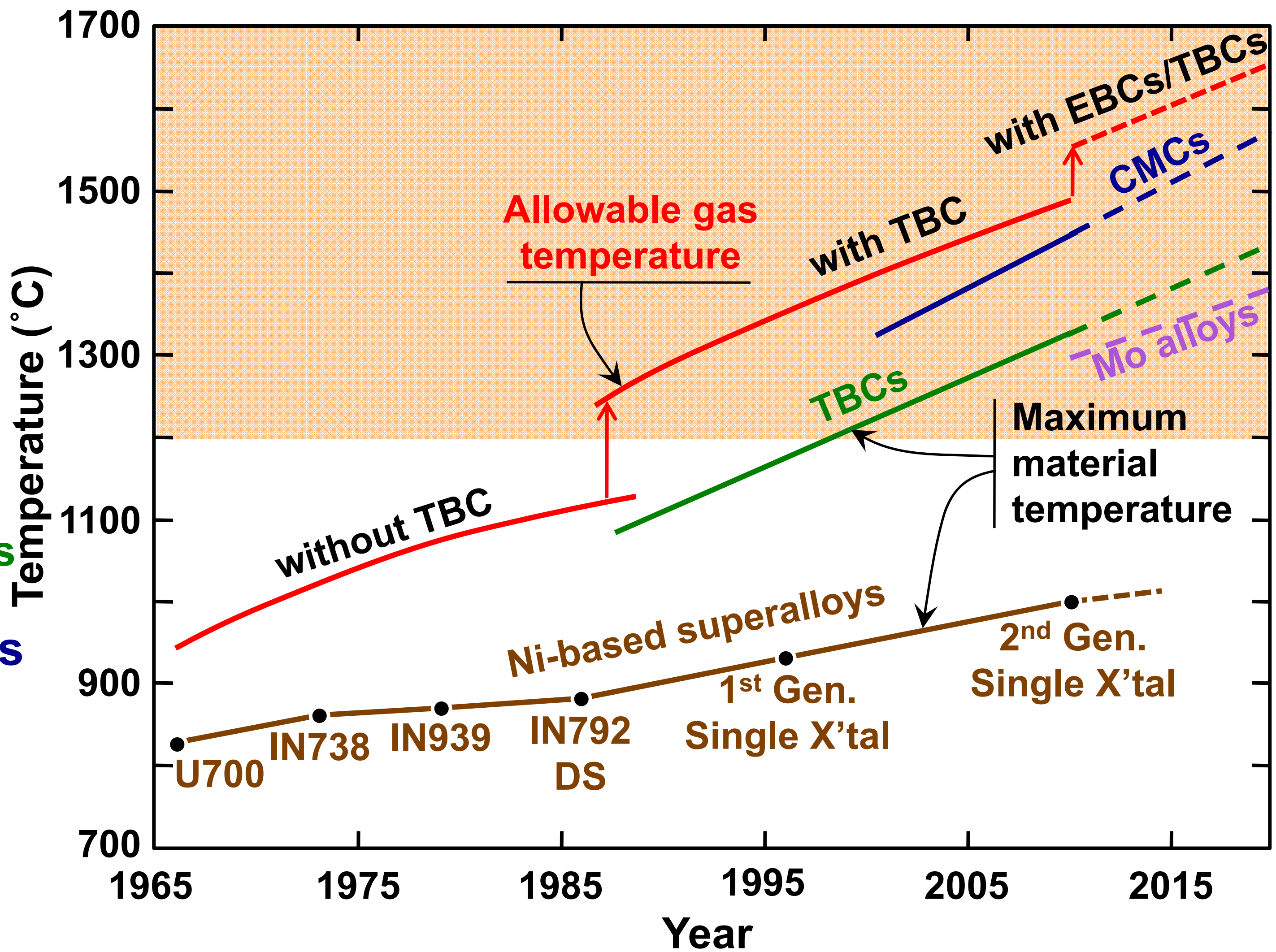






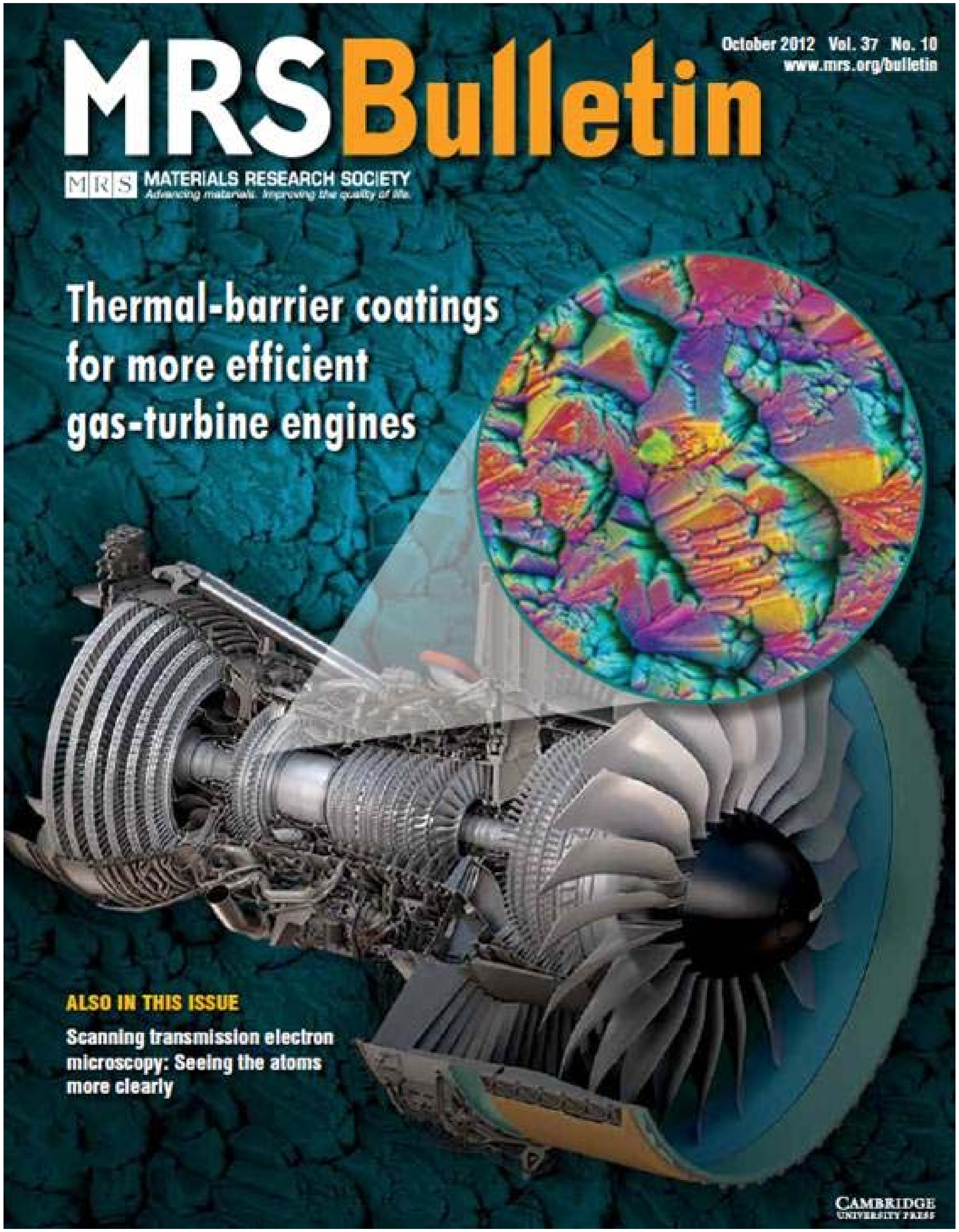
Outlook

* TBCs
  - Higher Temps.
  - Lower Th. Cond.
  - Th. Radiation
  - Higher Toughness
* Metals: Mo Alloys
  - TBCs, EBCs
* CMCs
  - EBCs



After Evans, 2009

Resistance to Molten Deposits



October, 2012

Thermal-barrier coatings for more efficient gas-turbine engines

David R. Clarke, Matthias Oechsner, and Nitin P. Padture, Guest Editors

Processing science of advanced thermal-barrier systems

Sanjay Sampath, Uwe Schulz, Maria Ophelia Jarligo, and Seiji Kuroda

Testing and evaluation of thermal-barrier coatings

Robert Vaßen, Yutaka Kagawa, Ramesh Subramanian, Paul Zombo, and Dongming Zhu

Low thermal conductivity oxides

Wei Pan, Simon R. Phillpot, Chunlei Wan, Aleksandr Chernatynskiy, and Zhixue Qu

Multifunctional coating interlayers for thermal-barrier systems

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Environmental degradation of thermal-barrier coatings by molten deposits

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Acknowledgements

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Chunhu Chen (Postdoc; NSYS Univ., Taiwan)
Julie Drexler (PhD; Boeing)
Andrew Gledhill (PhD; Diamond Innov.)
Dongsheng Li (Post-Doc; LBNL)
Madhu Reddy (Postdoc; CVD Equip. Corp.)
Alexander Vasiliev (Postdoc; Russ. Acad. Sci.)

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