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SCHOOL OF ENGINEERING

Abradable Sealing Materials for Emerging IGCC-Based Turbine Systems

2014 University Turbine Systems Research Workshop
Purdue University, West Lafayette, IN: October 21st, 2014

Daniel R. Mumm

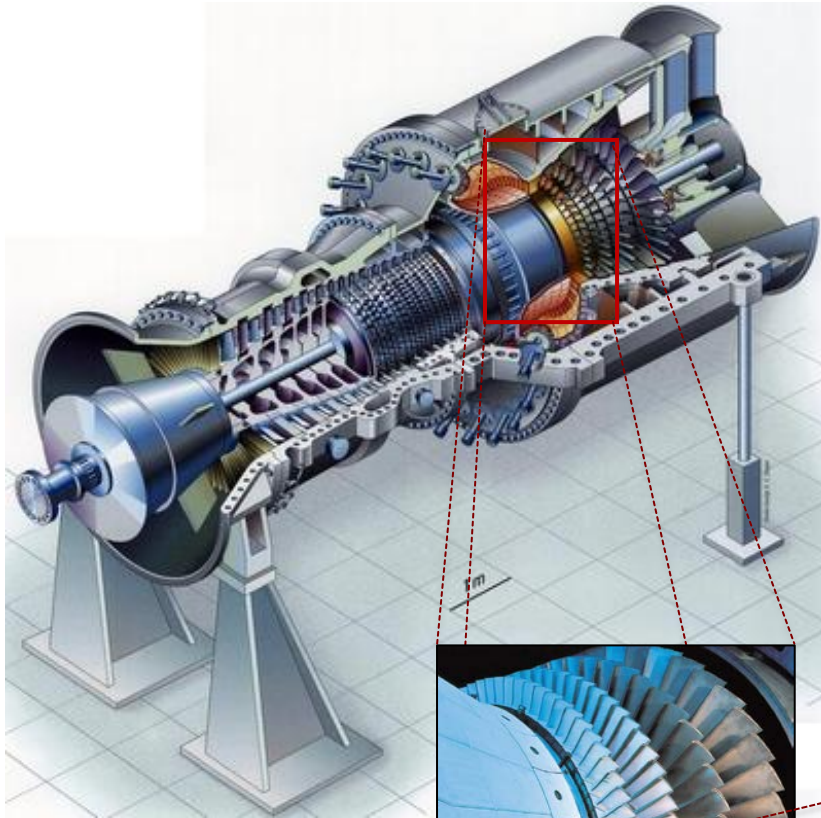
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Agreement # DE-FE0011929; Project Manager: Dr. Robin Ames

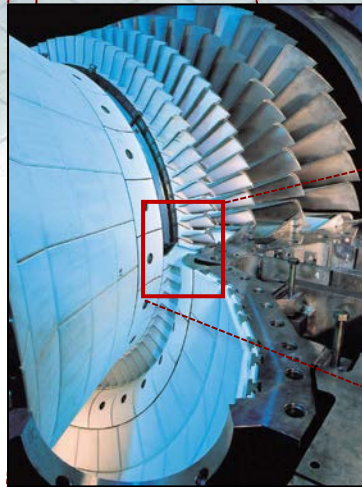


U.S. DEPARTMENT OF
ENERGY

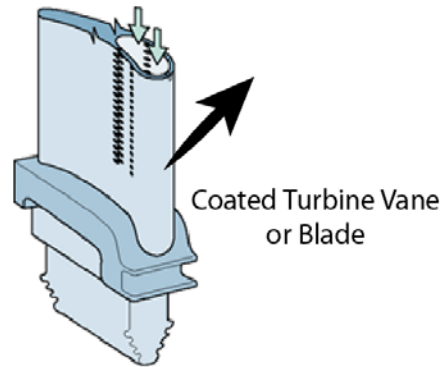
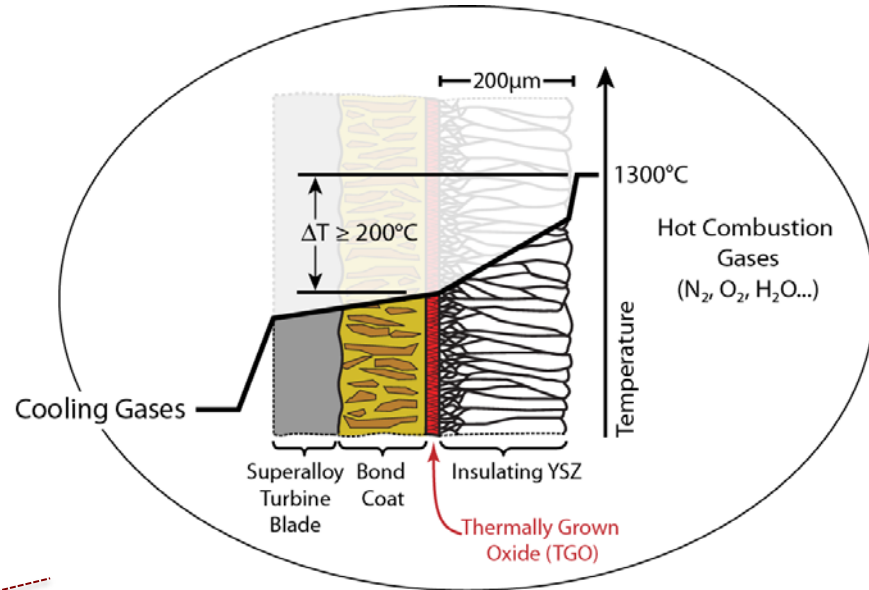
Turbine Hot-Section Materials



**Siemens
SGT5-4000F
Gas Turbine Engine
287 MW**



**TBCs and TGOs
Protect Hot-Section
Materials**

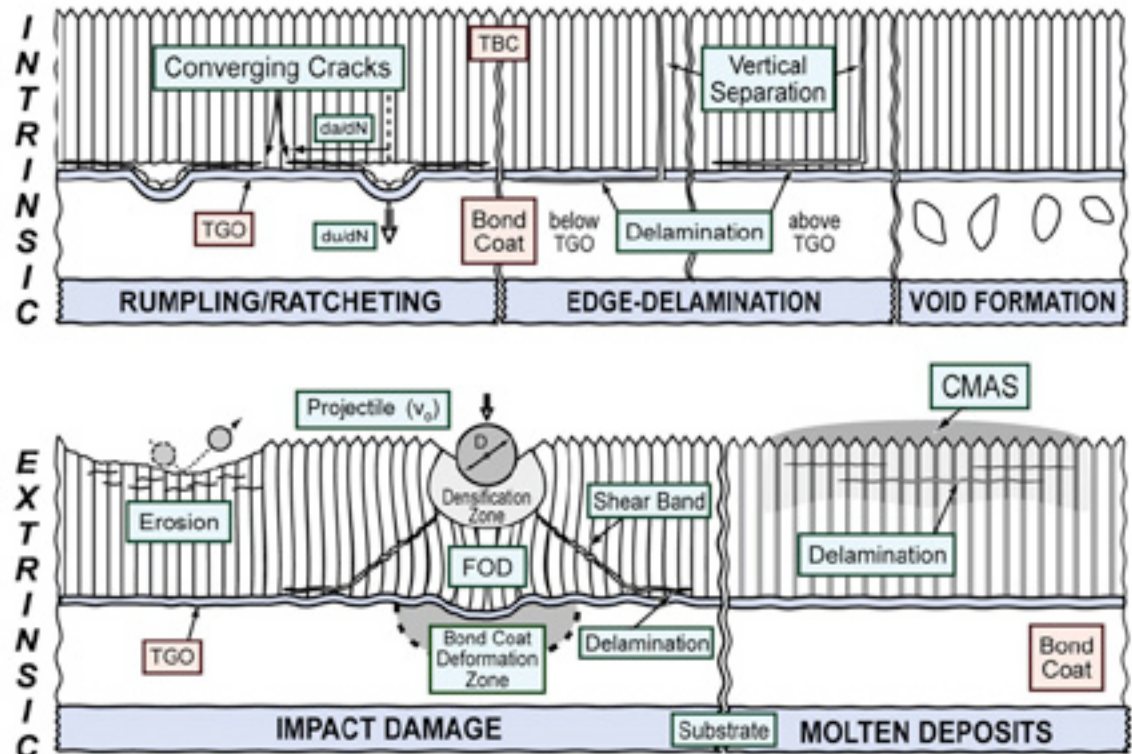


Potential Challenges in Transitioning to Alternative Fuels

A.G. Evans (2007)

There has been extensive research efforts directed toward the development and improvement of Thermal Barrier Coating (TBC) materials.

Our understanding of these Degradation Mechanisms forms a basis for understanding performance of Abradable Seals.

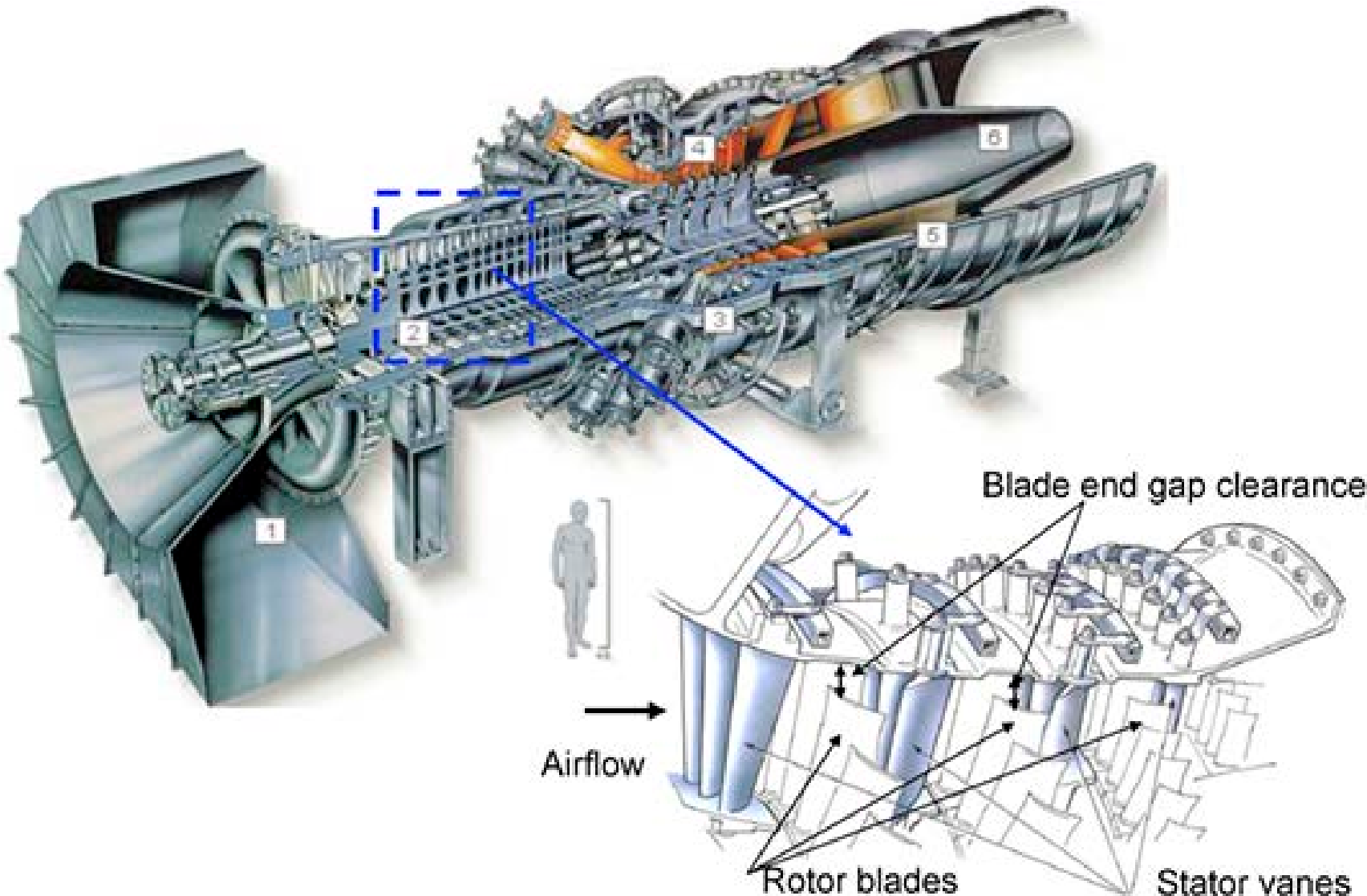


A.G. Evans, D.R. Clarke and C.G. Levi (2008)
Journal of the European Ceramic Society, 28, 1405-1419.

A.G. Evans, D.R. Mumm, J.W. Hutchinson, G. Meier and F.S. Pettit (2001)
Progress in Materials Science, 46, 505-53.

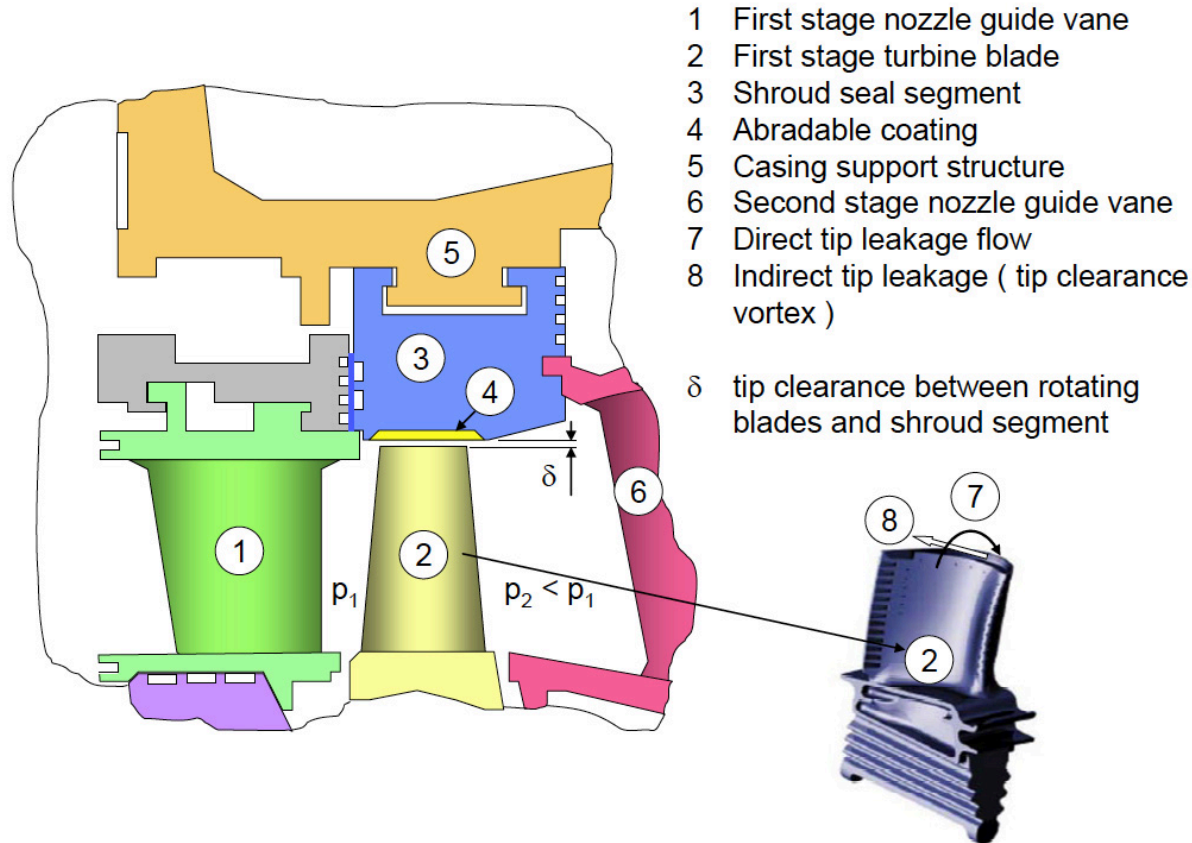


Abradable Seal Coatings



Abradable Seal Coatings

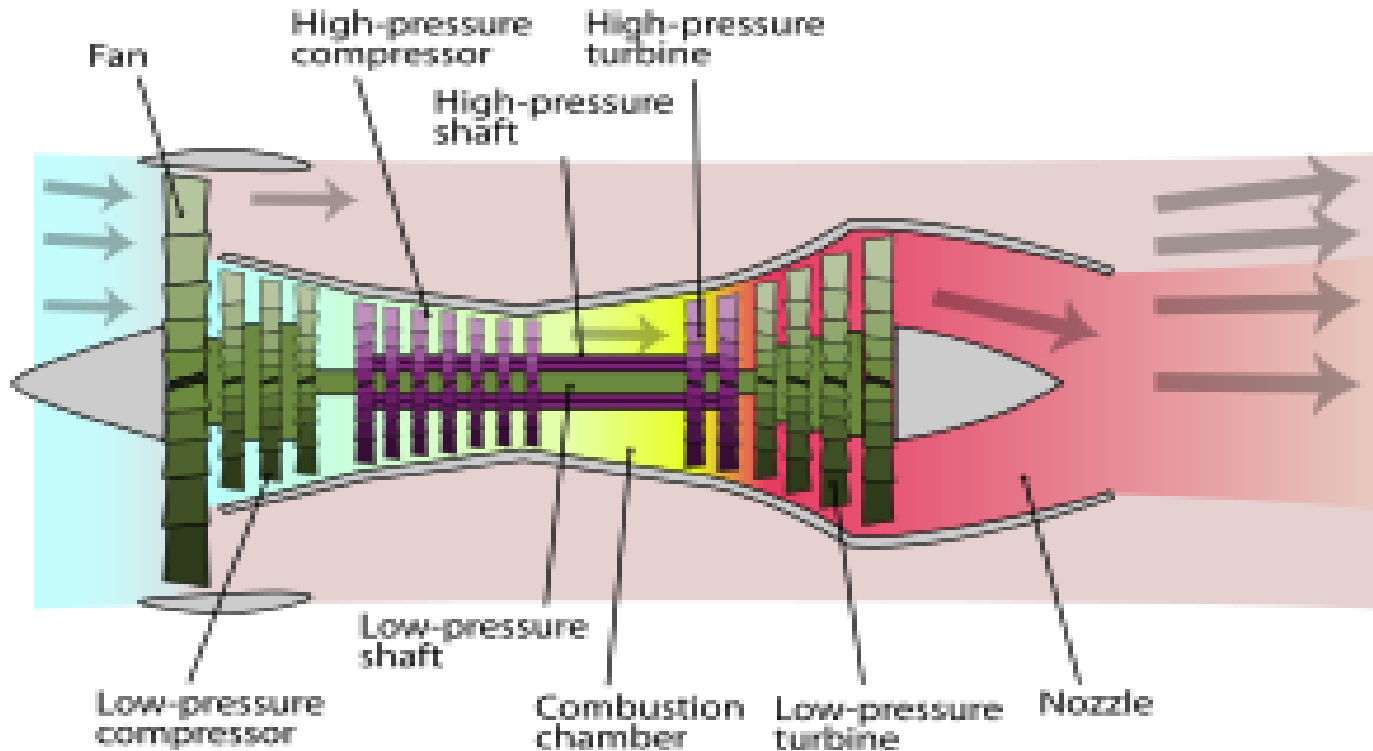
Schematic of a section through a gas turbine engine high pressure stage, showing where an abradable coating is used and how gas leaks through this seal leads to performance loss.



D. Sporer, S. Wilson and M. Dorfman, "Ceramics for Abradable Shroud Seal Applications." *Proceedings of the 33rd International Conference on Advanced Ceramics and Composites*, volume 3, 2009,

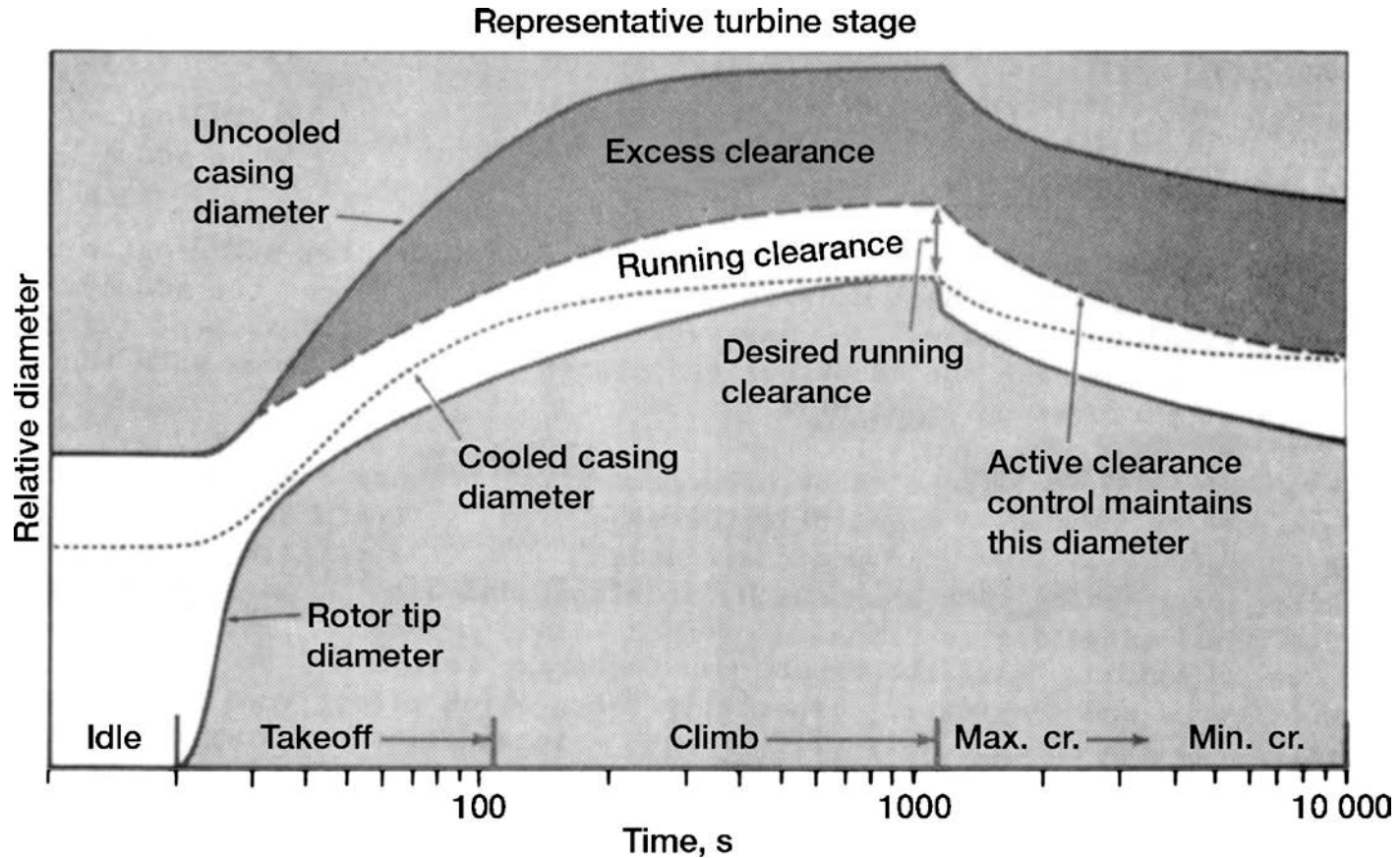


Abradable Seal Coatings



- Concerns include: excessive blade-tip wear, macrorupture in coatings, transfer of materials from blade to shroud.

Active Cooling Control for Clearance Control

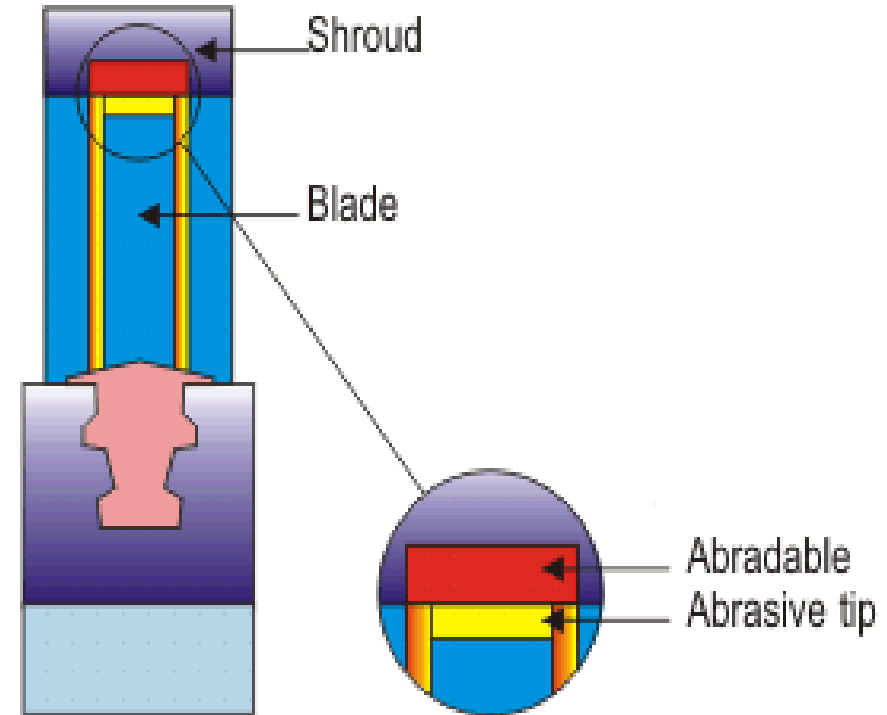


R.E. Chupp, et al., Journal of Propulsion and Power
Vol. 22, No. 2, March–April 2006



Abradable Materials for Clearance Control

- To reduce rotor-shroud clearance (an extra gap of .005" between the rotating blades and the engine casing can increase fuel consumption by as much as 0.5%).
 - Lower consumption of engine fuel
 - Improves engine-efficiency
- To achieve high temperature stability, low thermal conductivity, chemical stability, and erosion resistance at operating temperatures



Abradable Materials

- Metal matrix ($T < 700$ degree C)
- Ceramic materials ($T > 700$ degree C)
- Lubricant/dislocator agent
- Porous materials
- Ni/Graphite and AlSi/hBN for compressor
- CoNiCrAlY/hBN/Polyester for LP turbine sections of aircraft jet engines.
- YSZ or similar ceramics for HPT sections

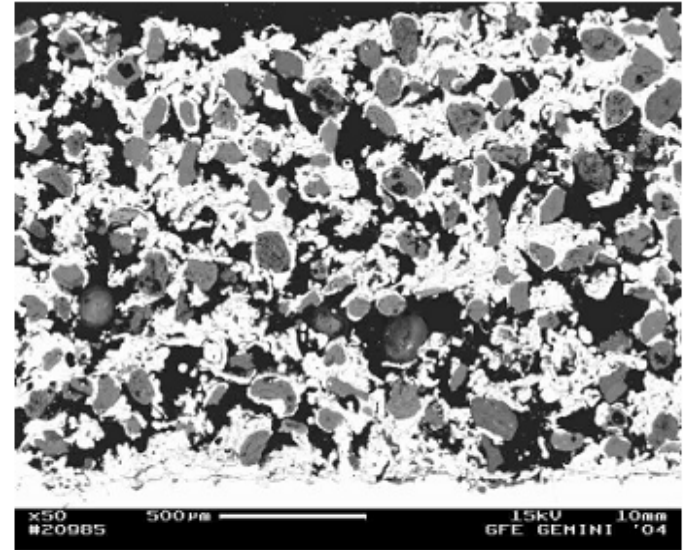
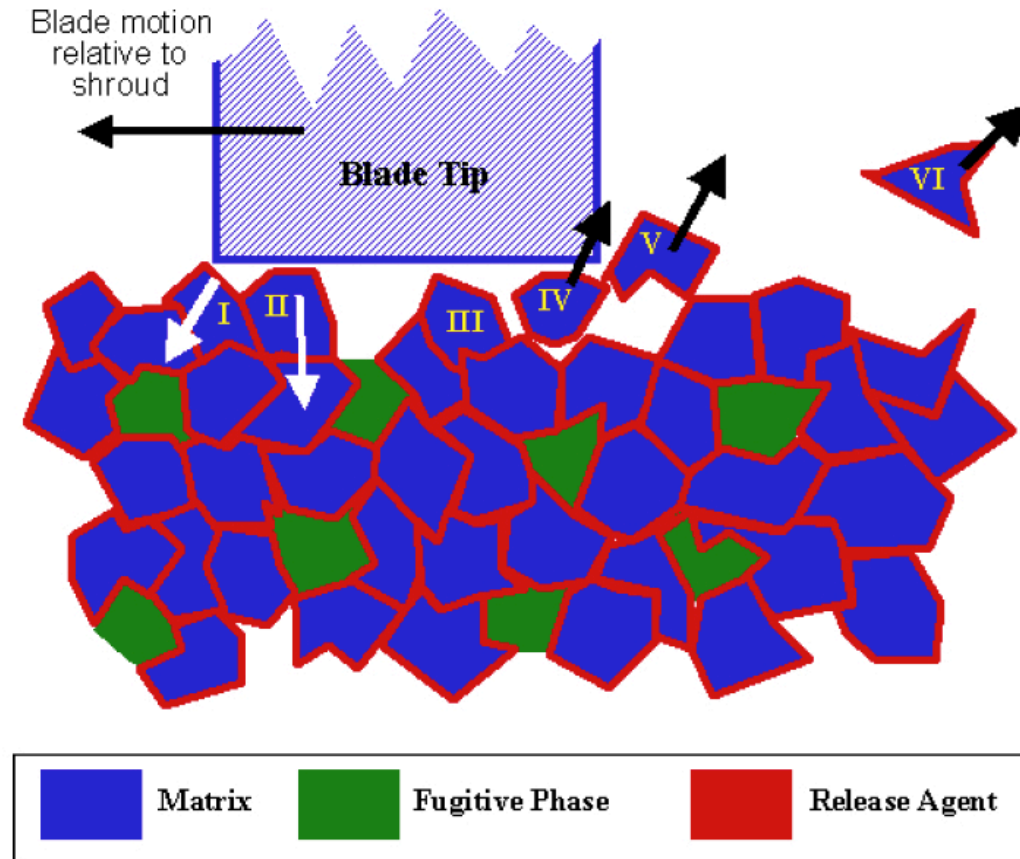


Figure 1: Cross section of a typical abrasion-resistant coating (dark phase: porosity, grey phase: bentonite, bright phase: NiCrAl metal matrix)

Abradable Seal Wear and Recession – A Balancing Act

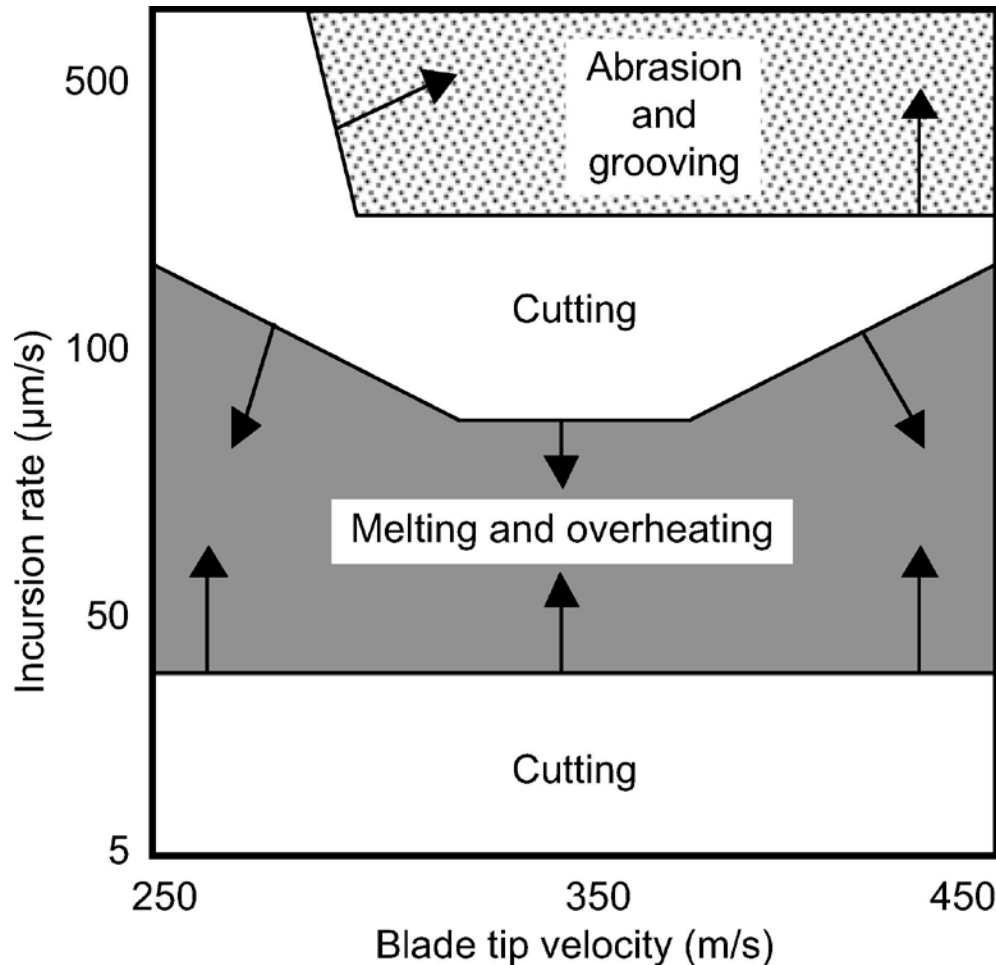
The ideal wear and recession behavior of an abradable coating consisting of a ceramic matrix (YSZ), a fugitive pore-forming phase, and a release agent that creates weak interfaces.



D. Sporer, S. Wilson and M. Dorfman, "Ceramics for Abradable Shroud Seal Applications." *Proceedings of the 33rd International Conference on Advanced Ceramics and Composites*, volume 3, 2009,



Abradable Seal Wear and Recession – A Balancing Act



Abradable Coating response is dependent upon operational parameters of the engine

R.E. Chupp, et al., Journal of Propulsion and Power, Vol. 22, No. 2, March–April 2006



Project Objectives

- ❑ Investigate the impacts of coal-derived syngas combustion environments on the performance, durability and degradation of existing abradable coatings used on turbine shroud structures.
- ❑ Assess the potential of alternative materials sets for improving performance of hot-section abradable seals in IGCC-based gas turbine power plants .
- ❑ Develop an improved mechanistic understanding of factors governing performance of high temperature abradable seals, and degradation mechanisms unique to coal-derived syngas and HHC-based combustion environments – ultimately with the goal of developing a knowledge base upon which the design of coatings that retain optimal sealing characteristics and are more resistant to the observed wear/attack mechanisms.

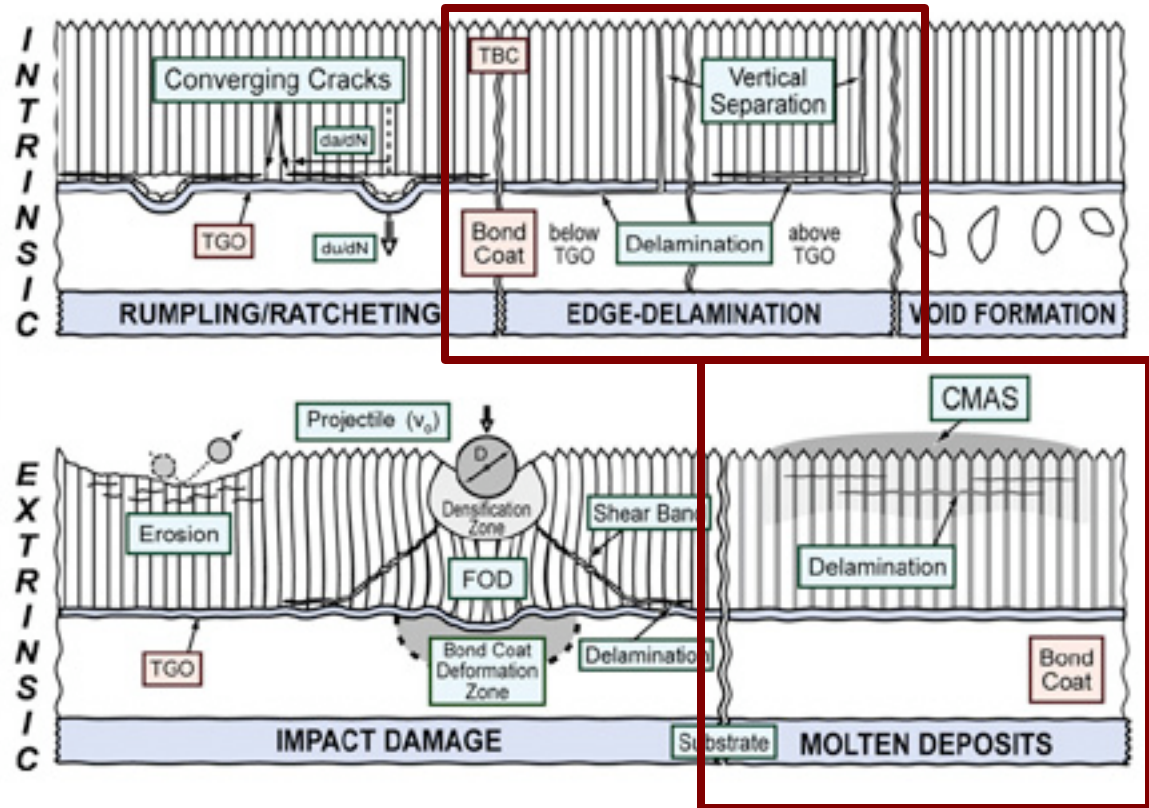


Deposit Formation Edge Delamination

What potential analogies to existing TBC

thermomechanical damage and CMAS degradation mechanisms arise for abradable coatings with use of syngas or HHC fuels and intrinsic combustion by products

?



Project Overview:

- Reducing the gap between rotating and stationary parts in gas turbine engines, and mitigating gas leakage via these paths, can significantly increase the performance and attendant efficiency. One approach to reducing this gap is to use abradable coatings on the stationary shroud components as seals.
 - Abradable coatings must be able to withstand high temperature oxidation, thermal cycling, and erosion, while providing optimal controlled abrasion and associated shape retention.
 - Preliminary testing of syngas and high-hydrogen-content (HHC) fired turbines has shown that the stability of hot-section materials (and presumably abradable sealing materials) may be substantially altered due to characteristic changes in the combustion by-products (partial pressures of water vapor, etc.) as well as characteristic impurities and particulate matter entrained in the fuel.



Project Overview: Goals and Objectives

- Derive a **mechanisms-based understanding** of factors controlling the performance and degradation of abradable seals used in the high-temperature turbine sections of gas turbine engines in relation to emerging IGCC-based combustion environments, and evaluate the potential of alternative materials as abradable seal coatings.
 - Investigate several classes of abradable coatings under simulated exposures to syngas-based combustion environments, evaluating the relevant wear behavior, hardness, stability under cyclic oxidation, and general thermo-mechanical behavior.
 - Develop of a combustion-based abradability test rig, leveraging existing activities and infrastructure (burner rig testing and associated materials exposure experiments), to correlate the measured thermo-mechanical behavior and controlled abrasive wear with the intrinsic properties of the multilayer coatings and processing-controlled microstructural features .
 - Investigate novel materials solutions to mitigate materials degradation.
 - Educate the next generation of scientists and engineers trained in materials design for advanced turbine systems.



- ❑ **Project has an overarching goal of developing a knowledge base upon which the design of coatings that retain optimal sealing characteristics and are more resistant to the observed wear/attack mechanisms .**
- ❑ **To achieve this goal, the project is organized around six inter-related themes:**
 - * **Evaluating** the performance of existing high temperature abradable seal coatings, and identify and materials evolution and degradation mechanisms specific to syngas and HHC combustion environments.
 - * **Assessing** oxidation behavior of abradable seal materials (in particular, those based on APS MCrAlY coatings) unique to HHC environmental exposures.
 - * **Carrying out** high-resolution imaging and microanalysis to explore how the abrasive recession and seal definition vary as a function of the combustion environment.
 - * **Assessing** thermo-mechanical drivers and thermal gradient effects on the needed abrasive wear processes, as well as seal failure through cracking and delamination (including testing under thermal gradient exposures).
 - * **Executing** experiments to study potential impacts of melting of particulate matter entrained in the gas flow stream, and infiltration into the seal coatings, and assist in identifying reaction products and evolving phases associated with molten phase corrosion mechanisms.
 - * **Identifying** alternative materials (including new compositions or bi-layer concepts) with potential for use as high temperature seals with improved performance in emerging aggressive combustion environments.



Assessment of the stability of current abradable seal materials in emerging HHC/syngas fueled turbine systems.

Non-Ideal oxide formation and water-vapor effects on TGO development

TBC stability studies in high p_{H_2O} environments

Mechanisms underpinning environment-dependent degradation (volatilization, etc.)

Characteristic surface deposits and CMAS-based degradation

New materials and processing approaches directed at mitigating damage evolution and optimizing system performance.

Development of improved thermo-mechanical models to guide development of abradable – but erosion resistant – seal coating systems.

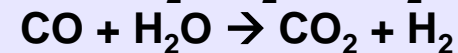
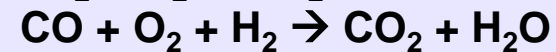
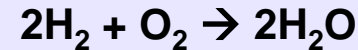


Motivation – Hot-Section Combustion Environment Modifications

Predicted Increased Water Content in Power Generation Turbines by the Use of Synthetic Gas (Syngas)

Flow Segment ID	Gas Composition				
	Units	GE Case 2	CoP Case 4	Shell Case 6	Range
Clean High-H ₂ Syngas	H ₂	91%	76%	86%	76-91%
	H ₂ O	0%	14%	3%	0-14%
	CO	2%	1%	3%	1-3%
	CO ₂	4%	2%	2%	2-4%
	Ar	1%	1%	1%	1%
	N ₂	1%	1%	5%	1-5%
Turbine Exhaust	N ₂	75%	74%	75%	74-75%
	H ₂ O	12%	14%	13%	12-14%
	O ₂	11%	10%	11%	10-11%
	CO ₂	1%	1%	1%	1%
	Ar	1%	1%	1%	1%

General Combustion Rxns:

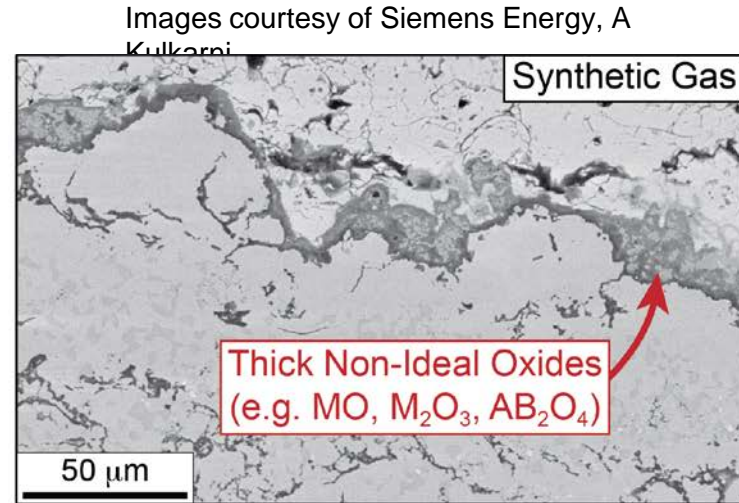
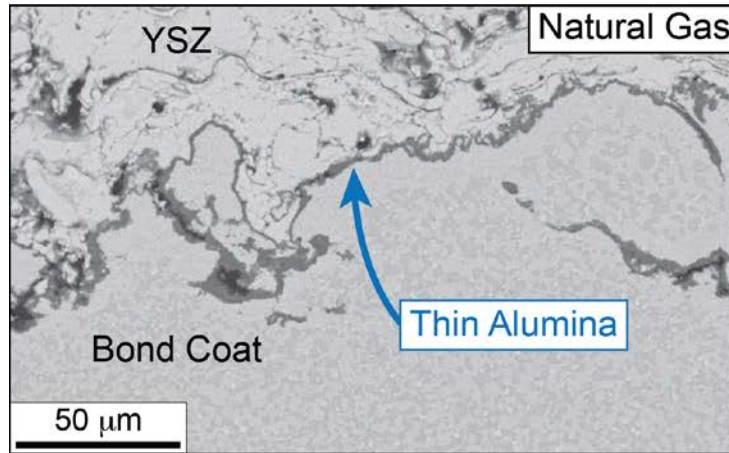


- 15-18 vol% H₂O in turbine exhaust when using dry, high-H₂ syngas fuel
- If steam is used for NO_x suppression, H₂O could run as high as 30%
- Represents a 2-4x increase over H₂O in natural gas combustion (5-7%)

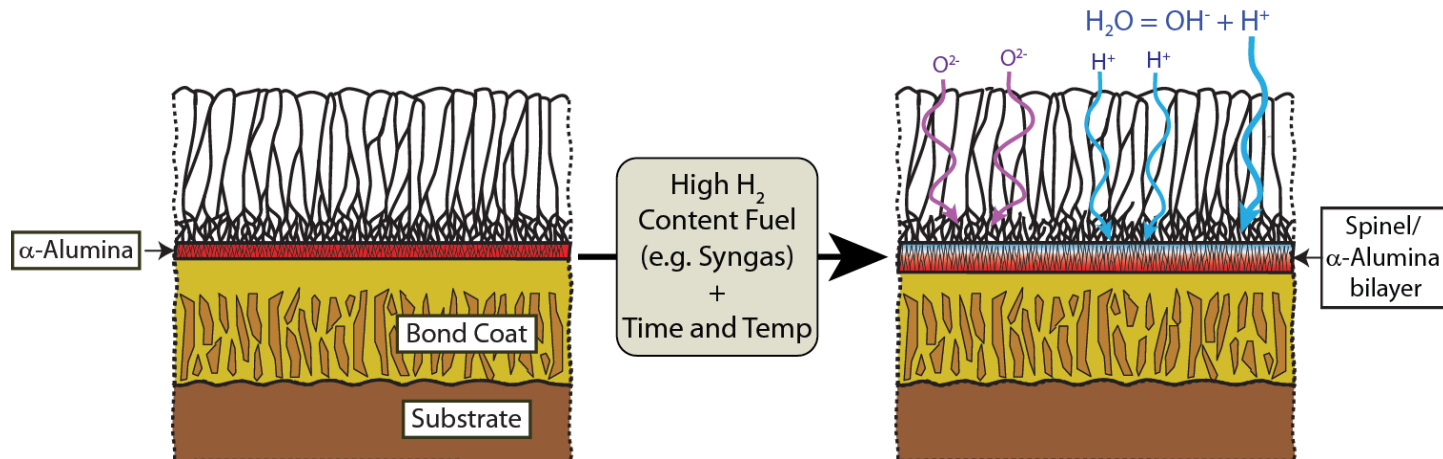
White, Ames and Burke. National Energy Technology Laboratory (NETL) Report, 2013.



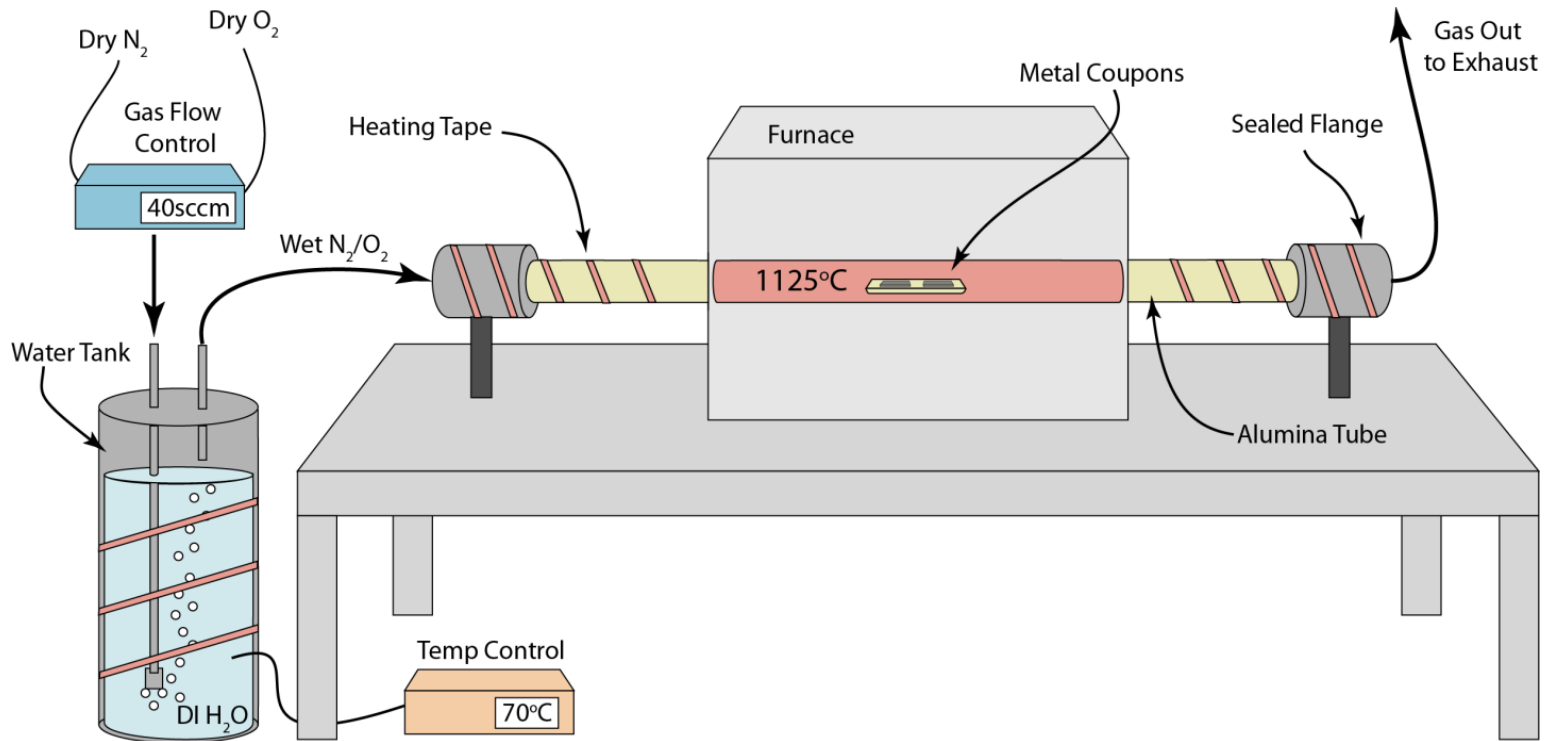
Project Motivation: Non-Ideal TGO Growth



- ✧ Note location of new (spinel) phases – above the alumina
- ✧ Suggests Ni transport **through** the alumina layer...



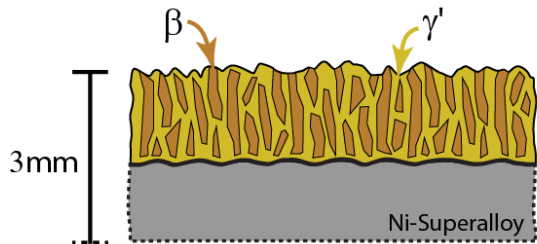
Observed spinel formation apparently not simply due to depletion of Al....



- Water tank temperature determines vol% H₂O via gas-liquid equilibrium exchange
- For 0% H₂O, the water tank is bypassed completely

Materials

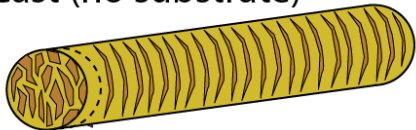
1) Sprayed (with substrate)



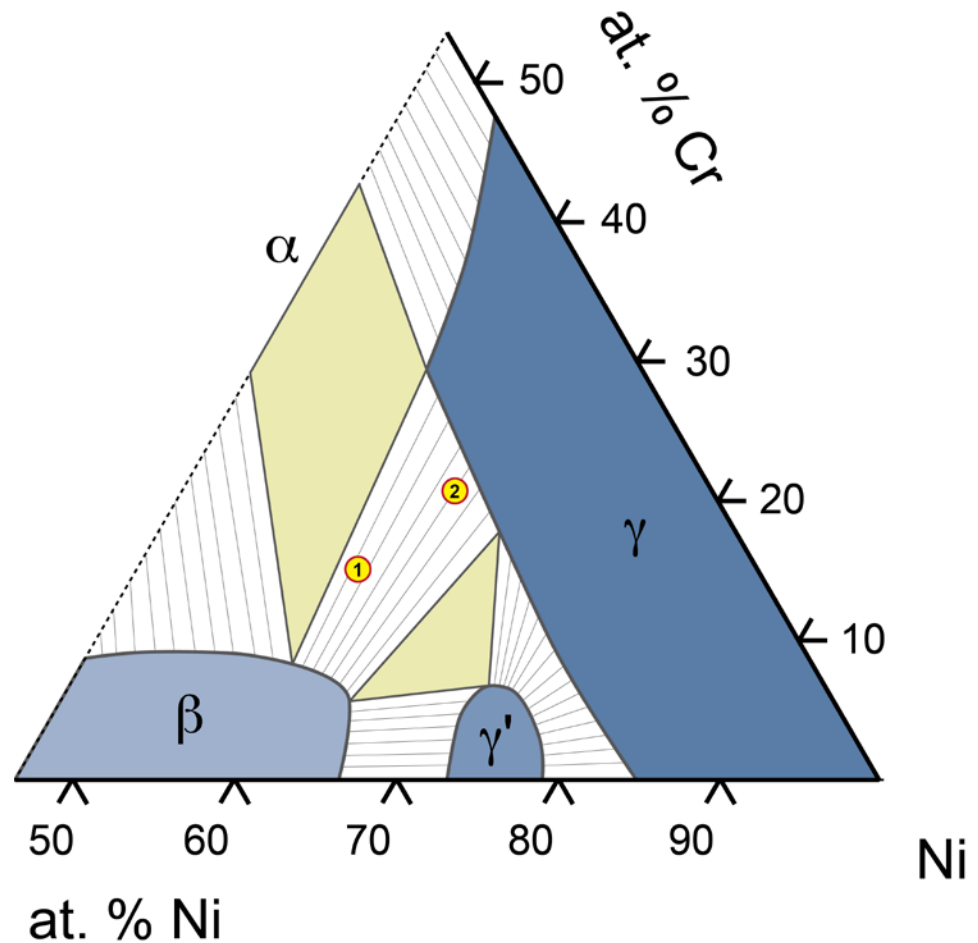
2) Sprayed (without substrate)



3) Cast (no substrate)



Ingot sectioned and polished



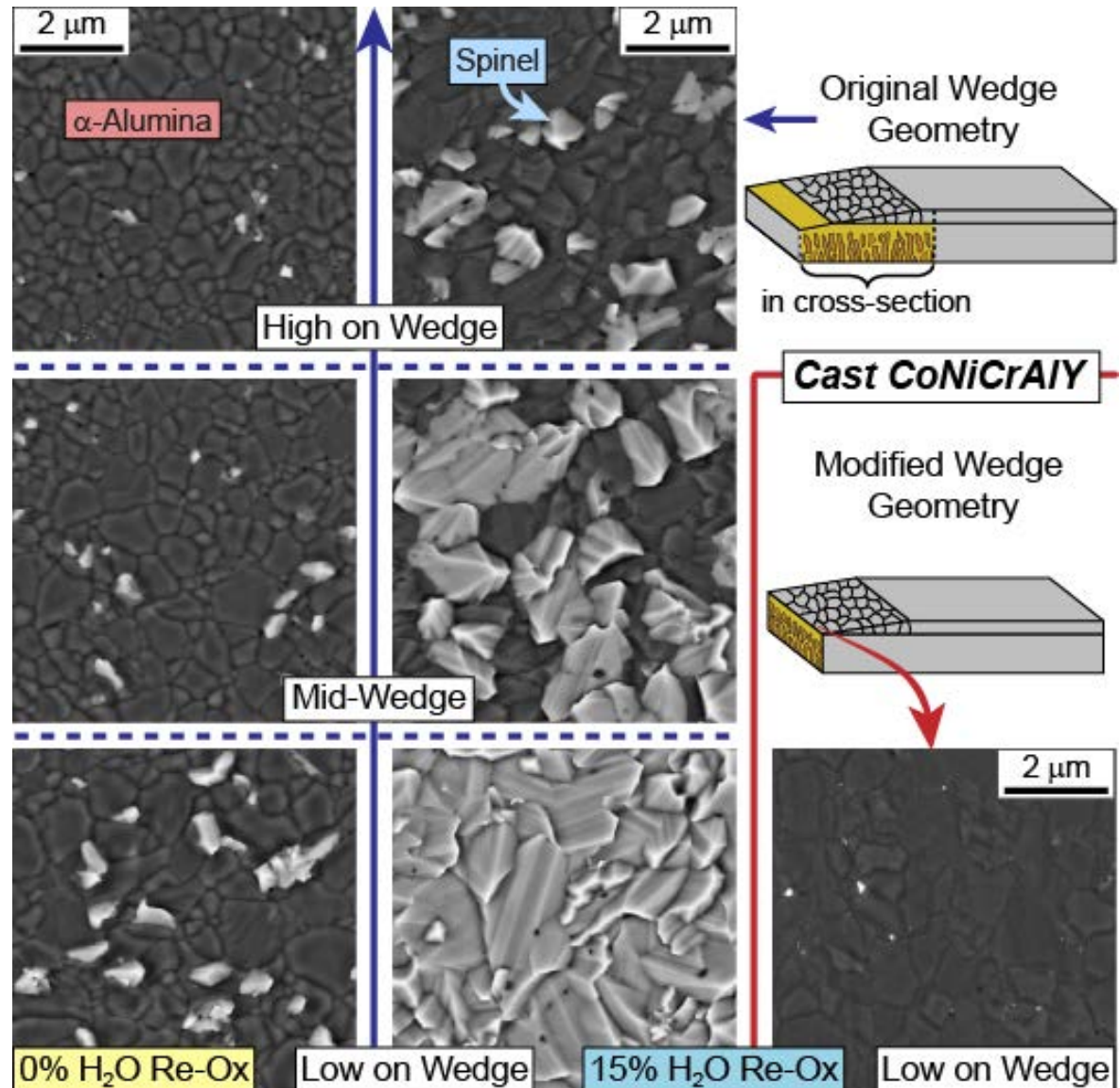
1 Ni-20Co-23Al-16.5Cr-0.3Y

2 Co-28.6Ni-15.6Al-21.2Cr-0.3Y

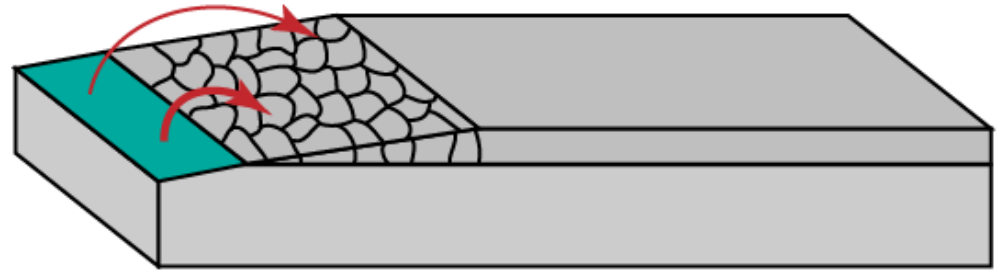
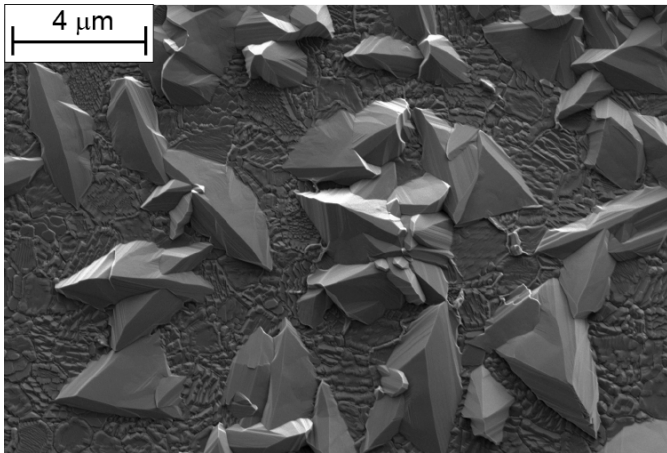


TGO Development Studies: Tapered Specimen Studies

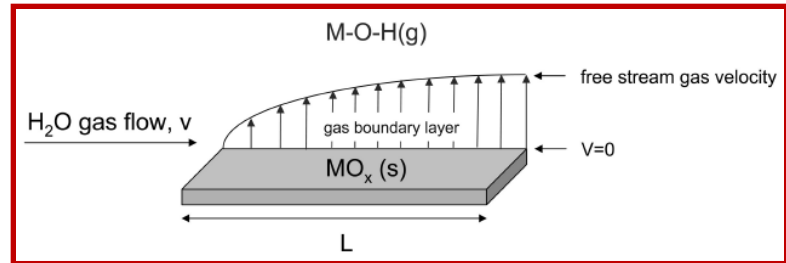
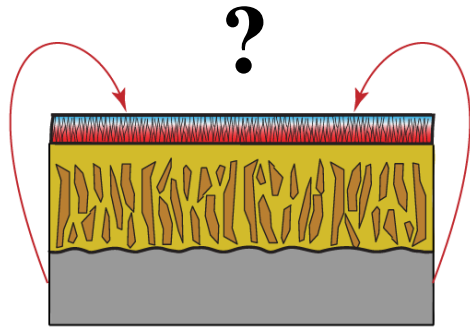
- Use the wedge polishing technique to evaluate growth of new oxide
- Spinel only grows on surface if Al-depleted section of alloy is exposed in front of wedge
- “Modified Wedges” do not grow any spinel at all



TGO Development Studies: Volatilization Effects



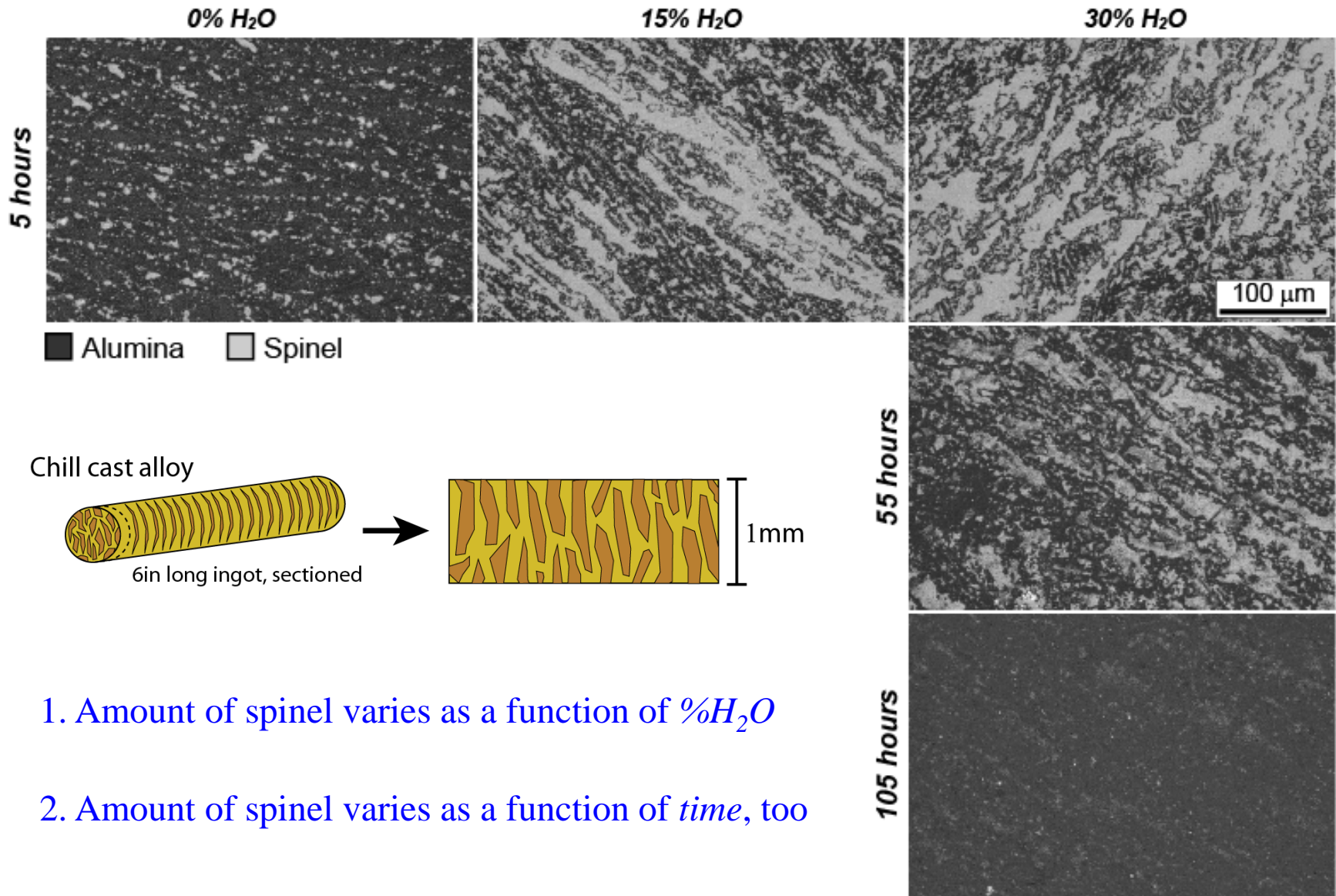
- The exposed, Al-depleted wedge (rich in Ni, Cr and Co) grows non-ideal oxides that volatilize and redeposit via vapor phase proportionately up the wedge



Opilia EJ, JOM (2006)

- Solid spinels can be synthesized via vapor phase combination of M^{2+} and M^{3+} metal hydroxides (e.g. Ni^{2+} and Cr^{3+})

TGO Development: Water Vapor and Volatilization

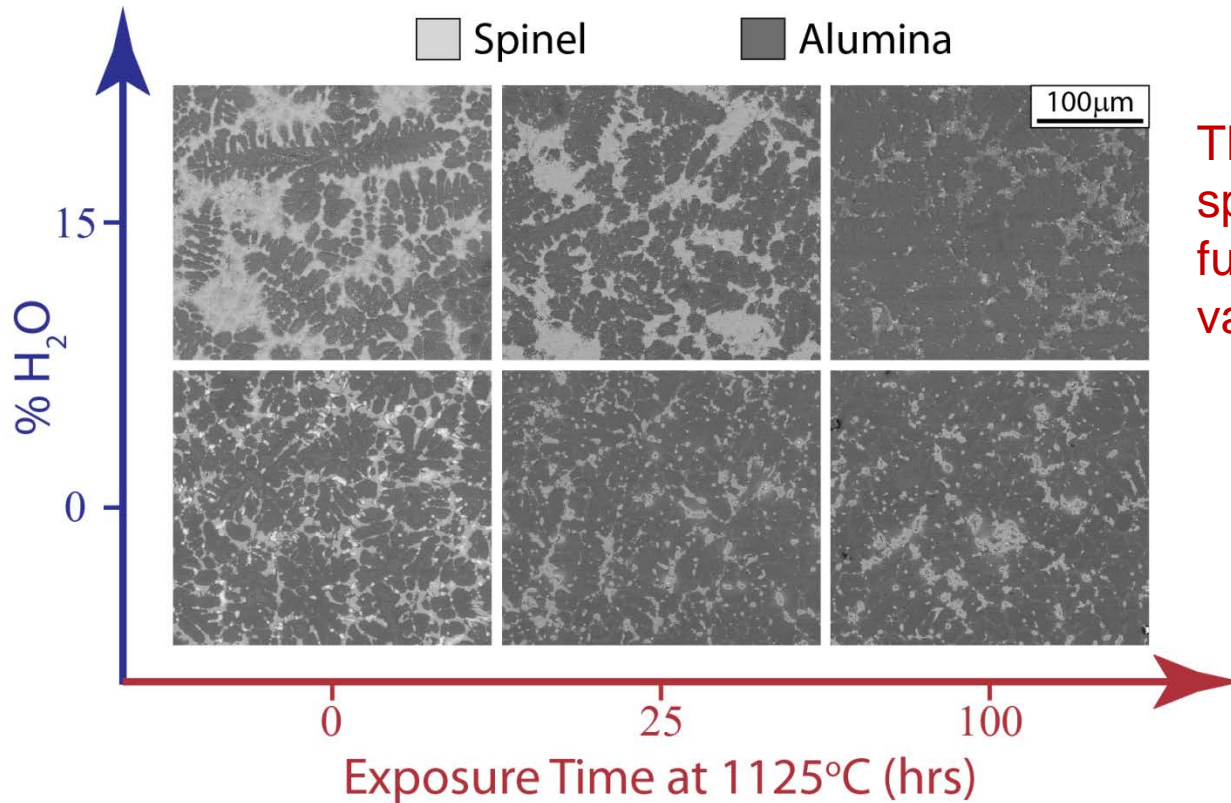


1. Amount of spinel varies as a function of $\%H_2O$
2. Amount of spinel varies as a function of *time*, too



TGO Development Studies: Evidence of Ni Volatilization

Visual inspection of low magnification, plan view, backscatter electron images shows the relative distribution of alumina and spinel on the surface of the TGO



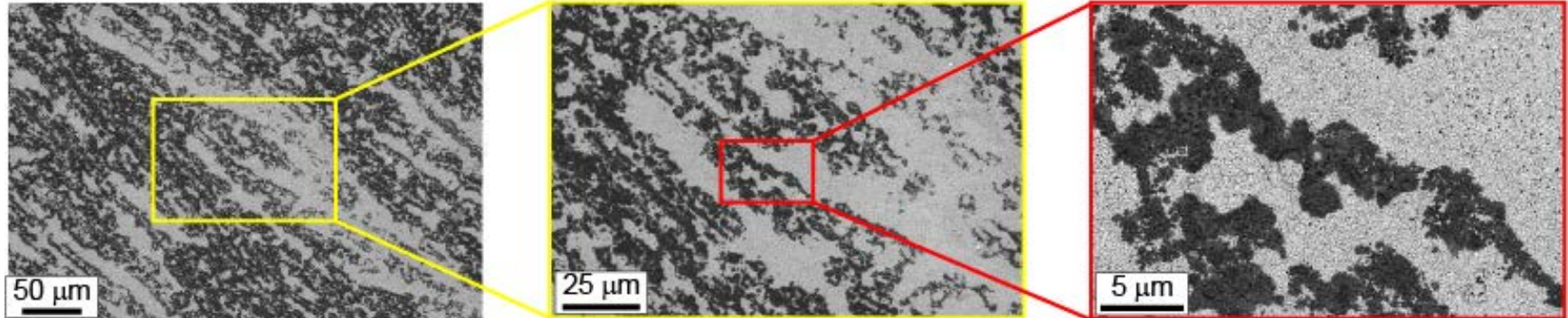
The amount of surface spinel *decreases* as a function of time in water vapor



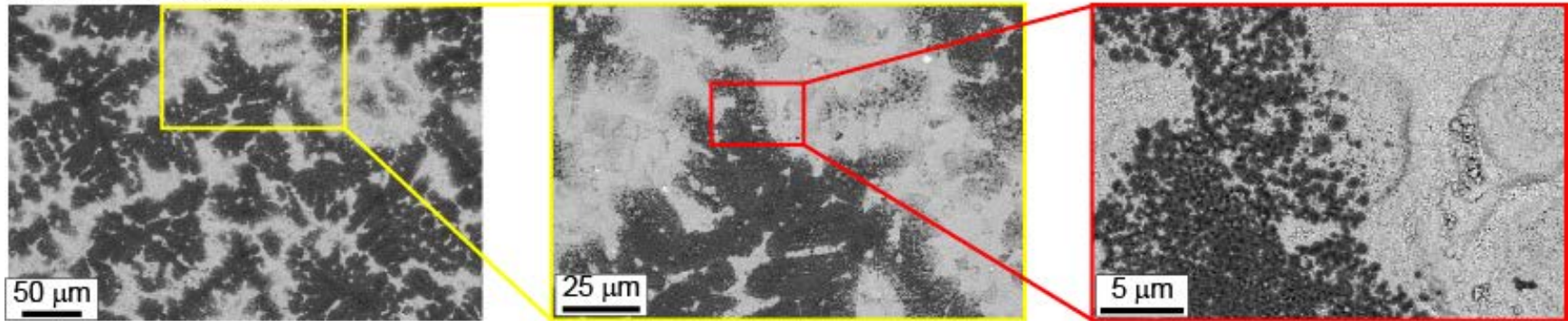
Water Vapor Effects on Transient Oxides

Spinel formation on MCrAlYs w/ elevated water vapor exposures

CoNiCrAlY: 15%H₂O, 5hrs



NiCoCrAlY: 15%H₂O, 5hrs



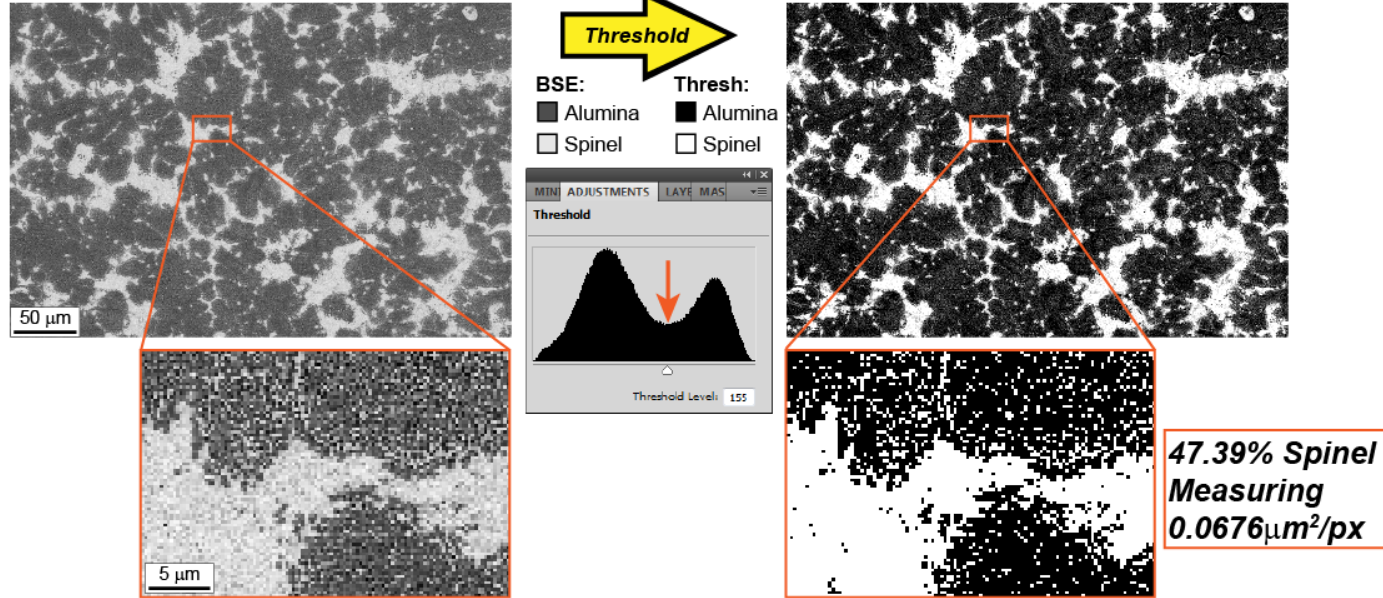
■ Alumina □ Spinel



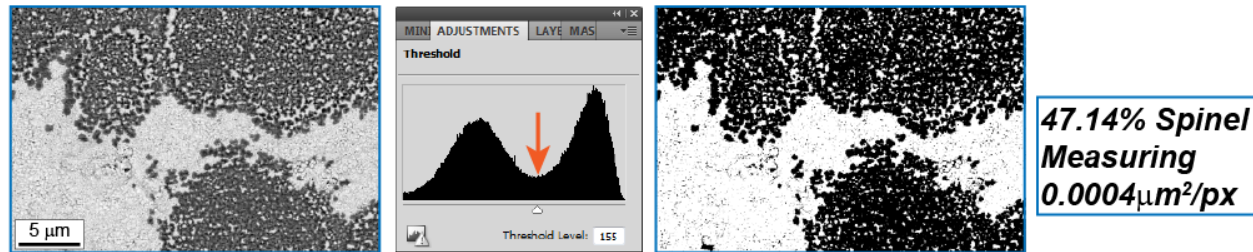
Thresholding Technique

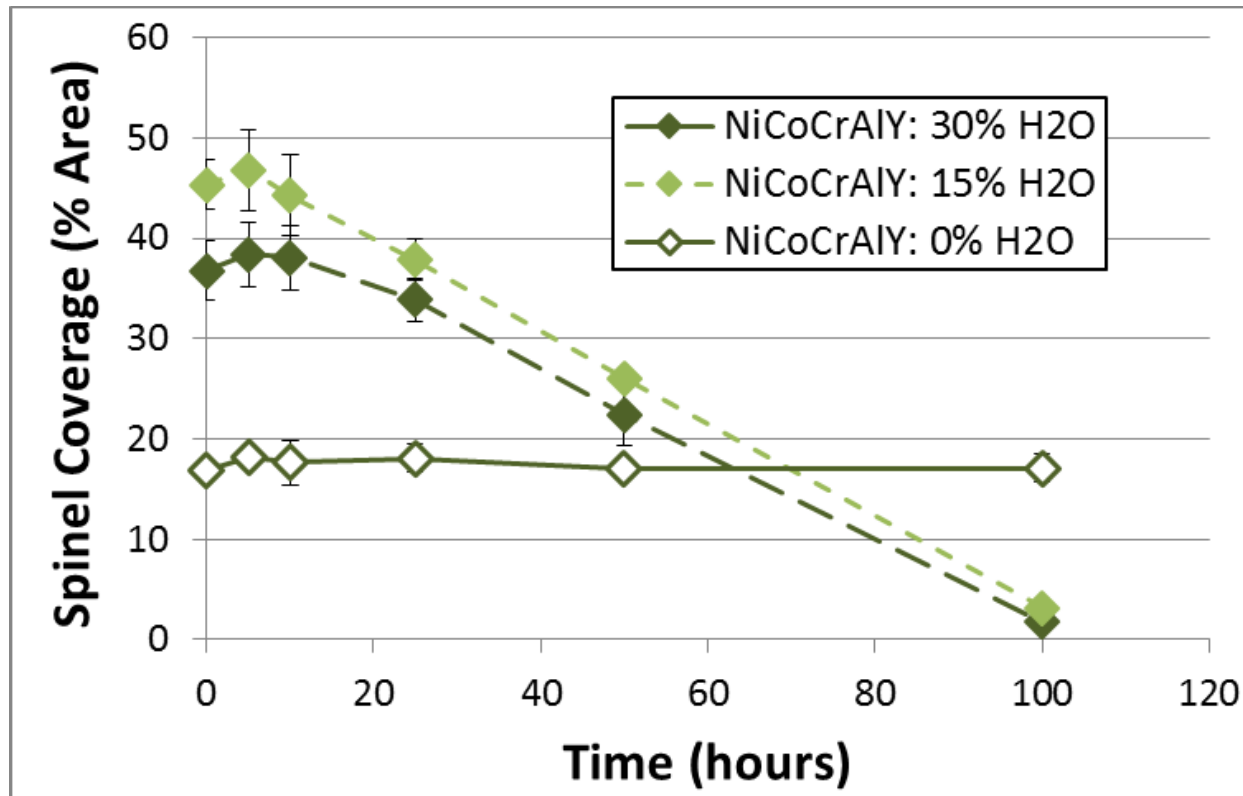
Low mag BSE images are used to accurately quantify the amount of surface spinel

A. HFW = 400 μm , 1536 x 768px



B. HFW = 30 μm , 1536 x 768px



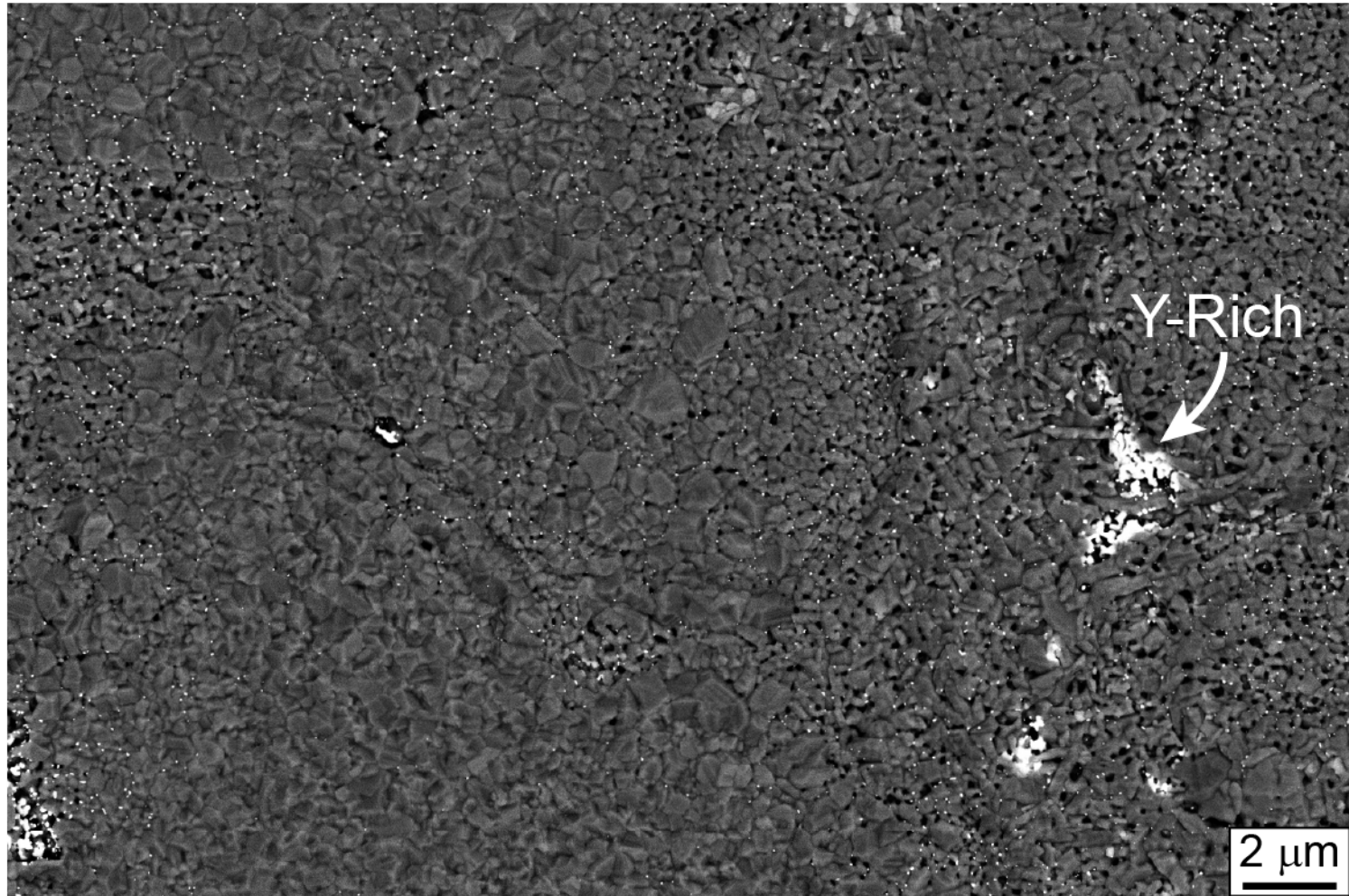


- In wet environments surface spinel decreases as a function of time and %H₂O
- In dry environments, spinel coverage is constant.



NiCoCrAlY: 15%H₂O

■ Alumina □ Spinel

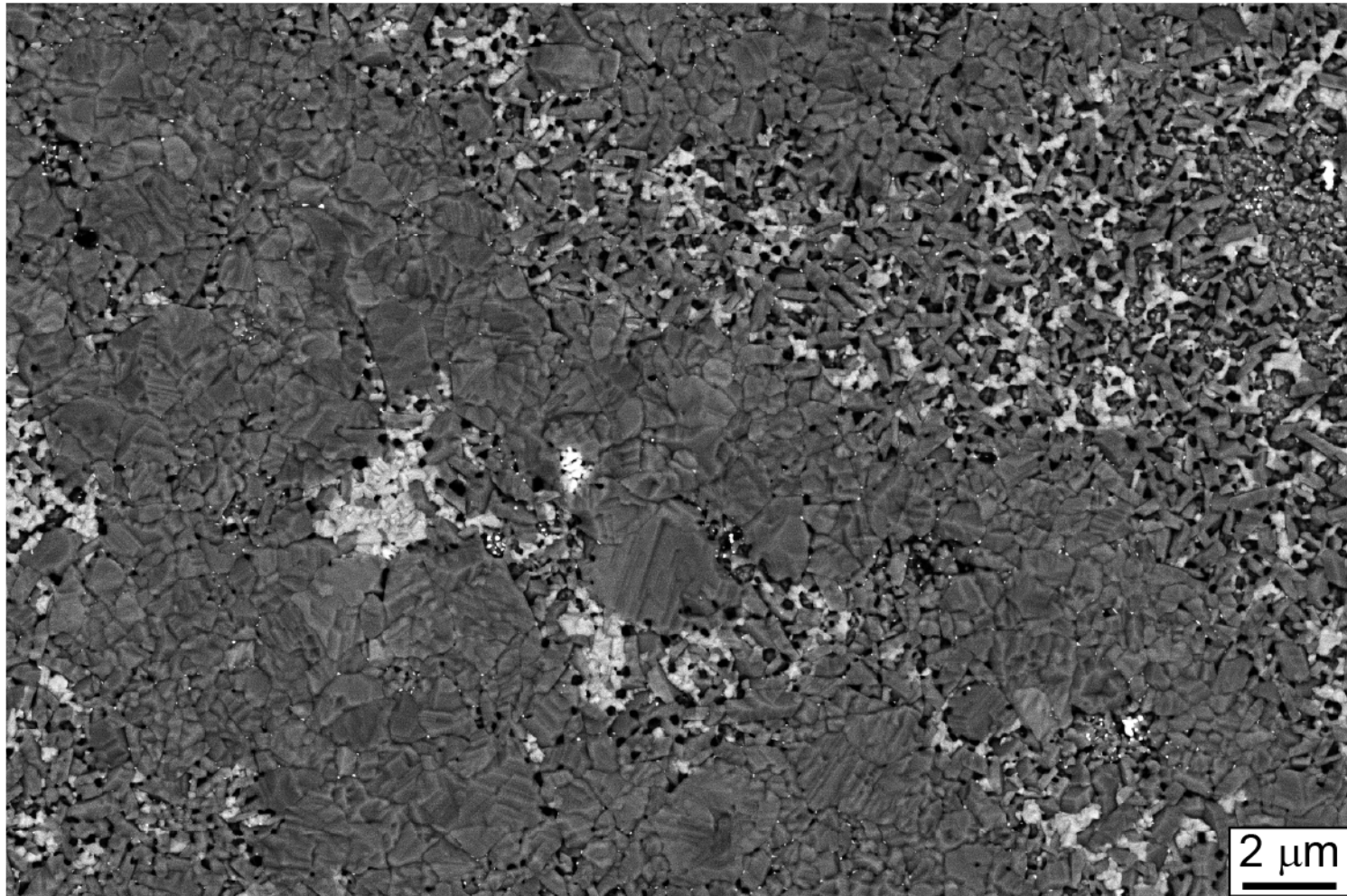


105 hours

- Spinel disappears from surface as a function of time in a wet environment

CoNiCrAlY: 15%H₂O

■ Alumina □ Spinel

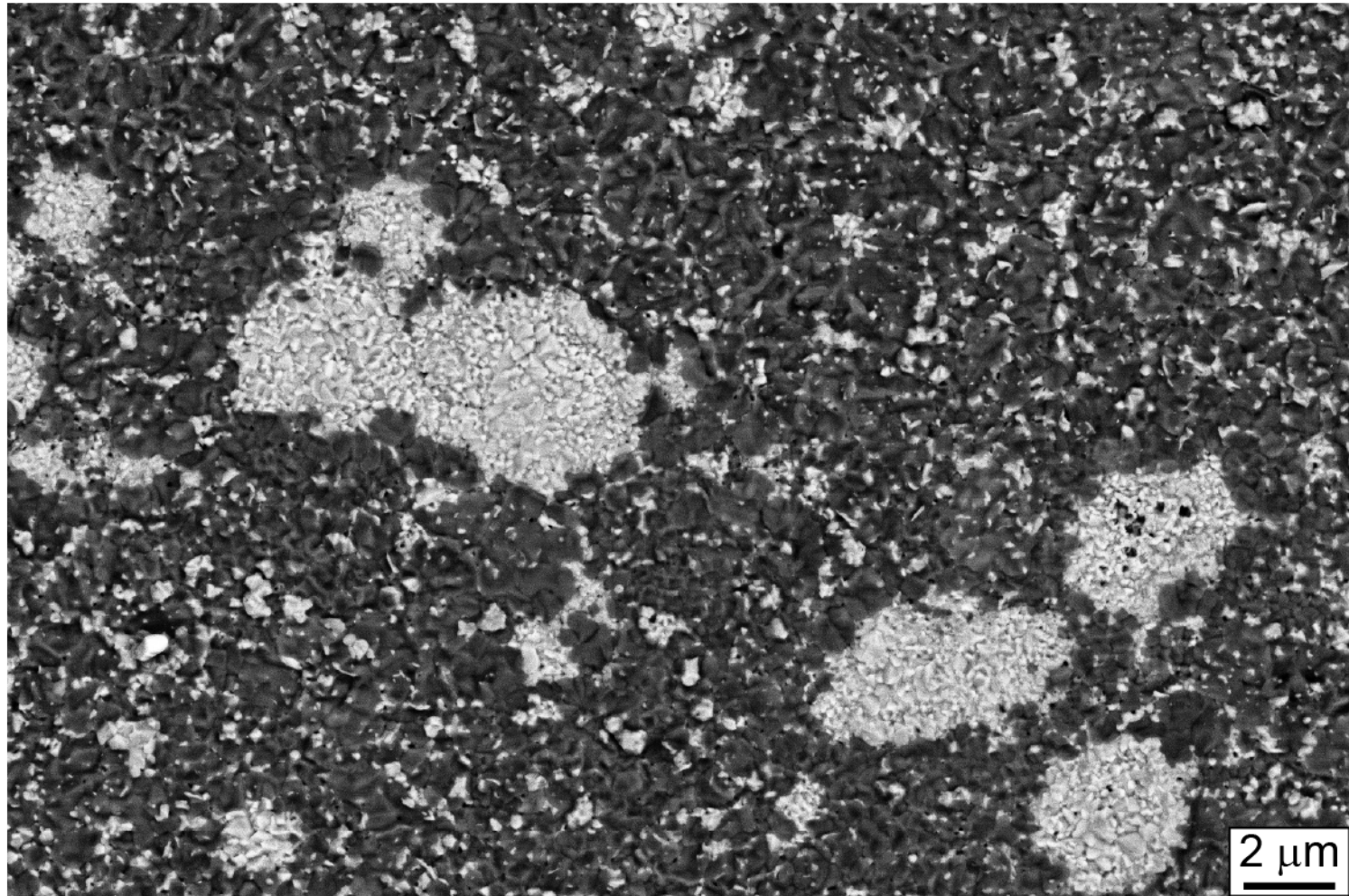


105 hours

- Spinel disappears from surface as a function of time in a wet environment

CoNiCrAlY: 0%H₂O

■ Alumina □ Spinel

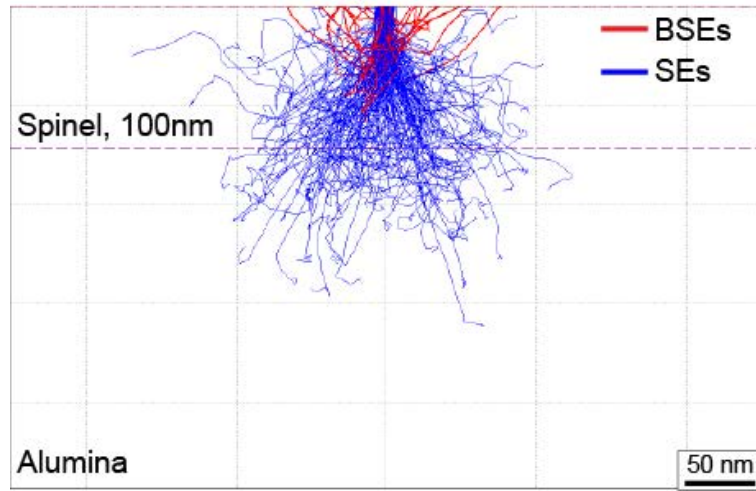


105 hours

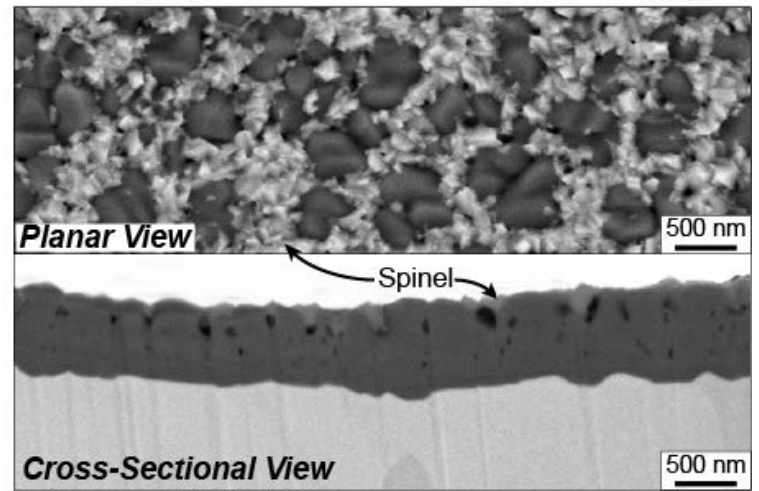
- Surface spinel is unchanged as a function of time in a dry environment

Water Vapor Effects on Transient Oxides - Volatilization

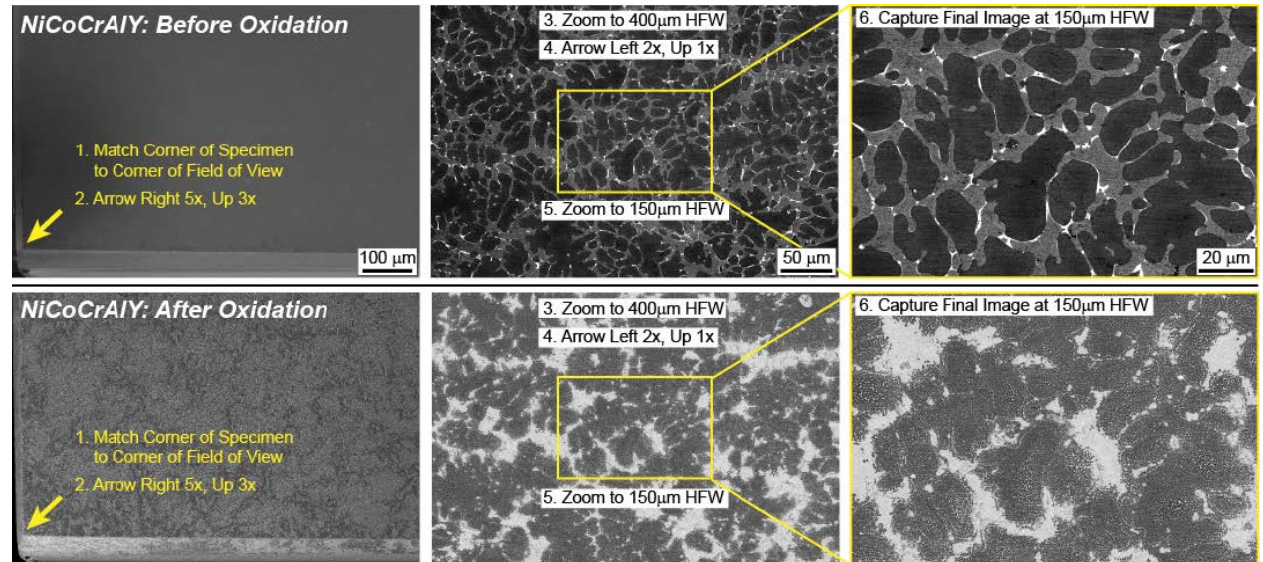
Monte Carlo Simulation, 5kV



BSE Images, 5kV



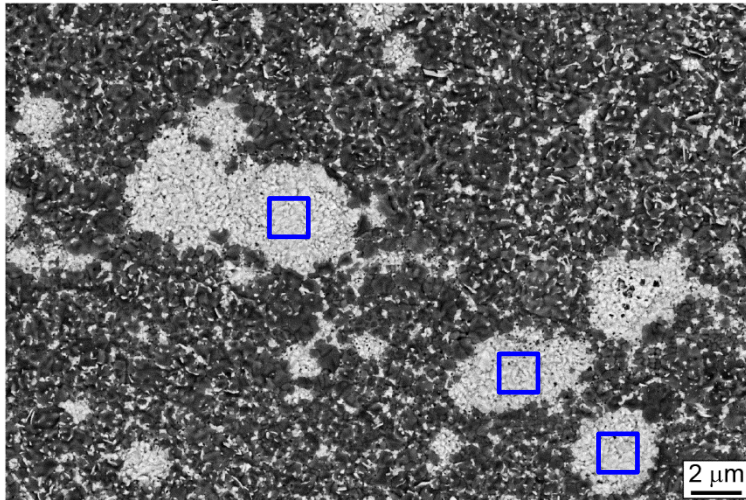
Technique development for site specific examinations of transient oxide formation and evolution:



Water Vapor Effects on Transient Oxides

CoNiCrAlY: 0%H₂O

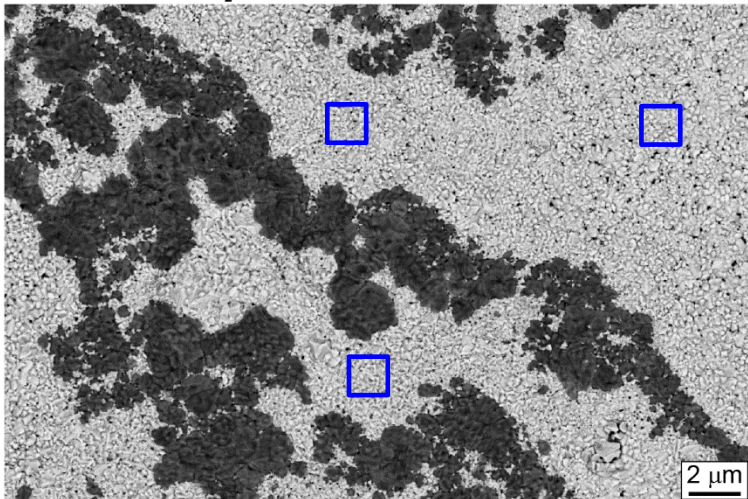
■ Alumina □ Spinel



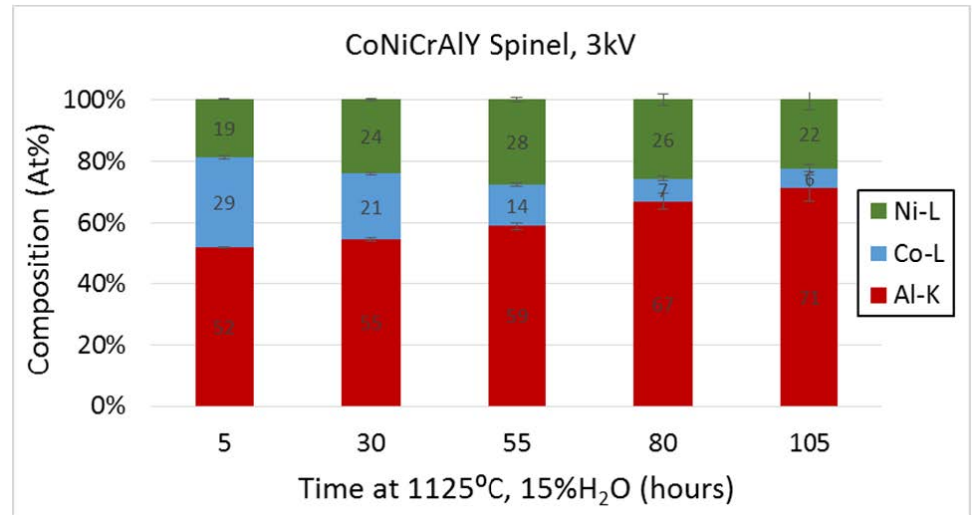
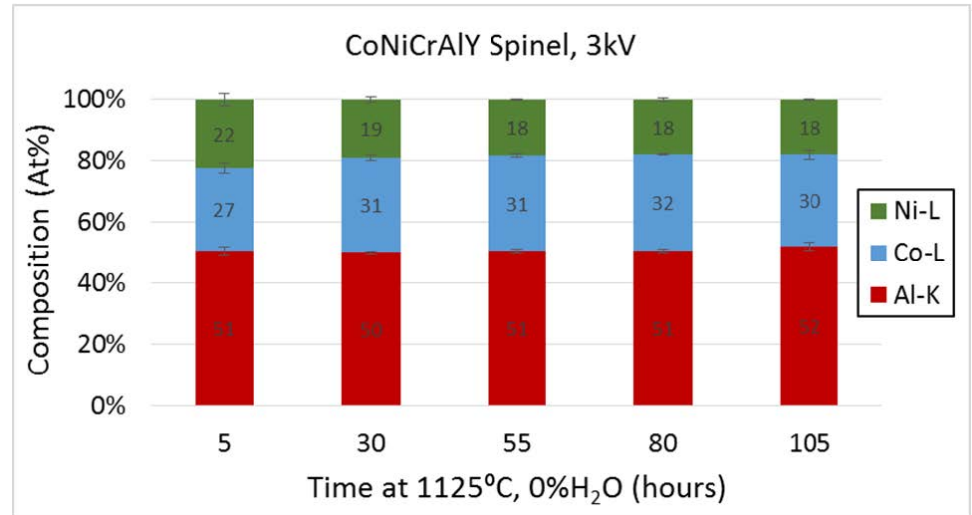
5 hours

CoNiCrAlY: 15%H₂O

■ Alumina □ Spinel



5 hours



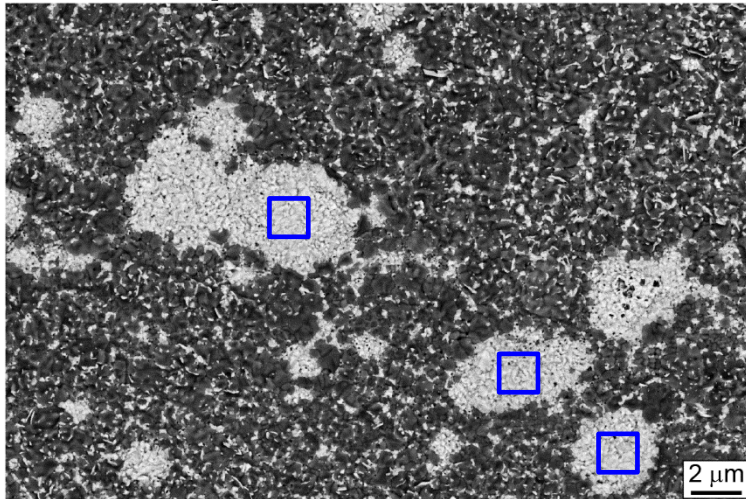
Spinel coverage evolution – EDS analysis



Water Vapor Effects on Transient Oxides

CoNiCrAlY: 0%H₂O

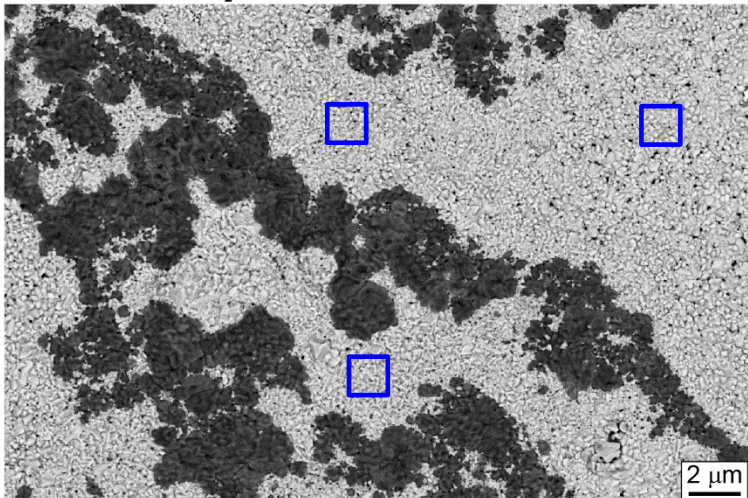
■ Alumina □ Spinel



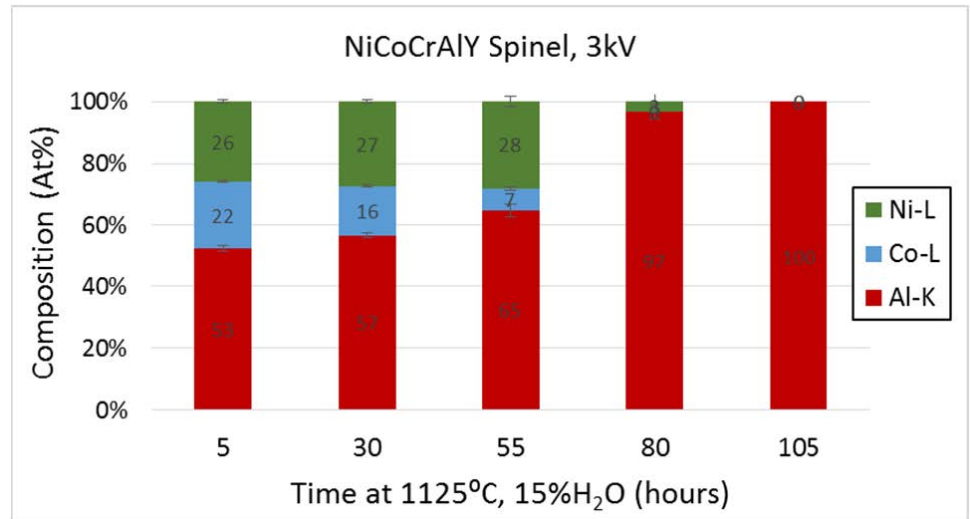
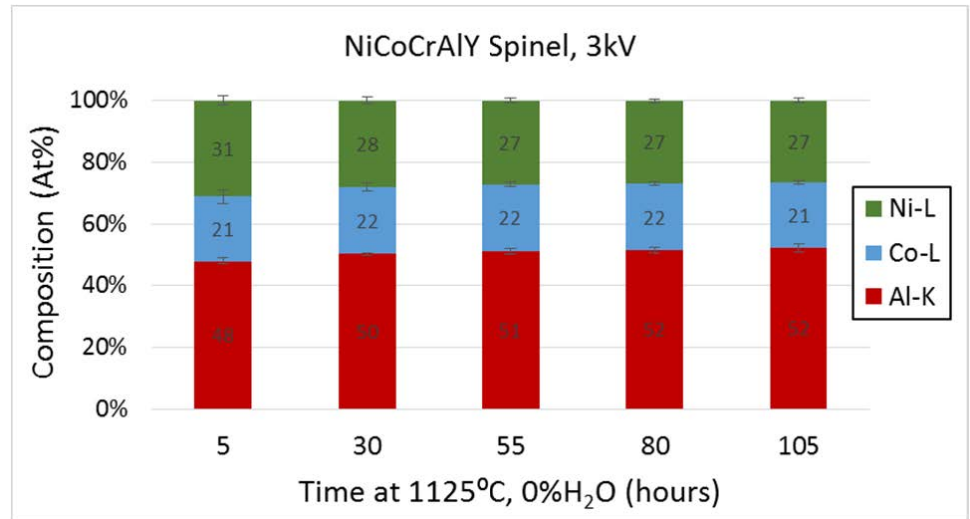
5 hours

CoNiCrAlY: 15%H₂O

■ Alumina □ Spinel



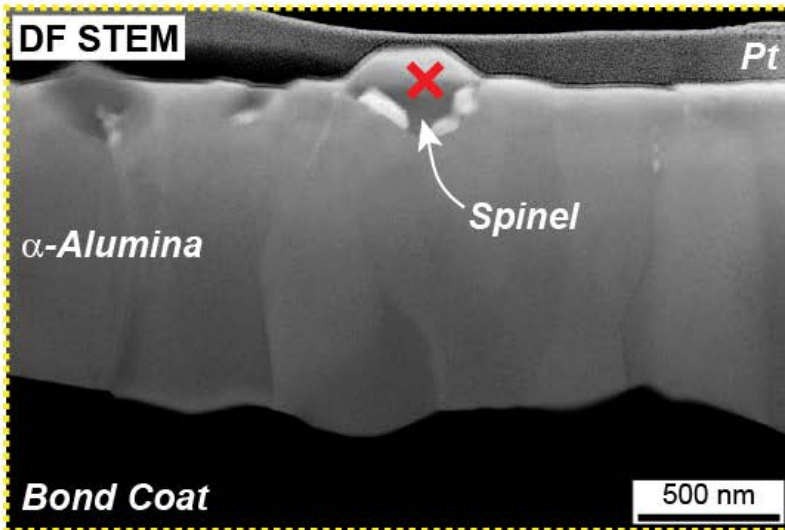
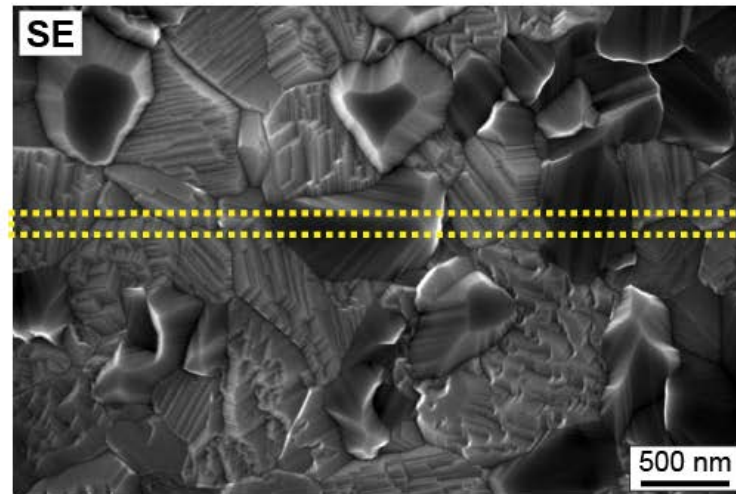
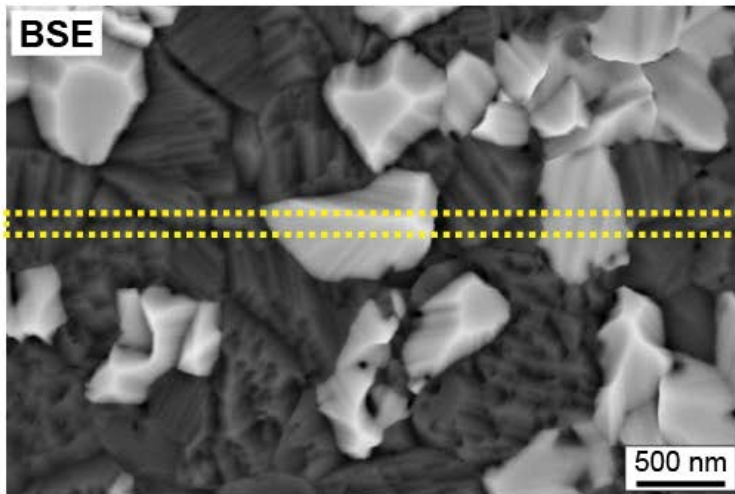
5 hours



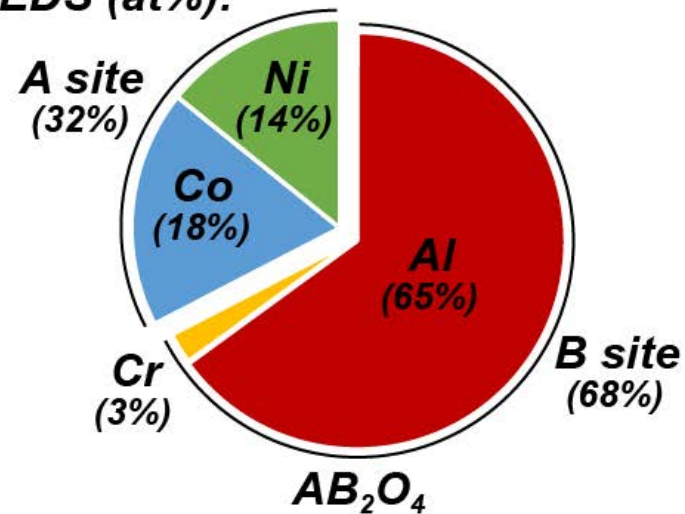
Spinel coverage evolution – EDS analysis



H₂O-Related Artifacts



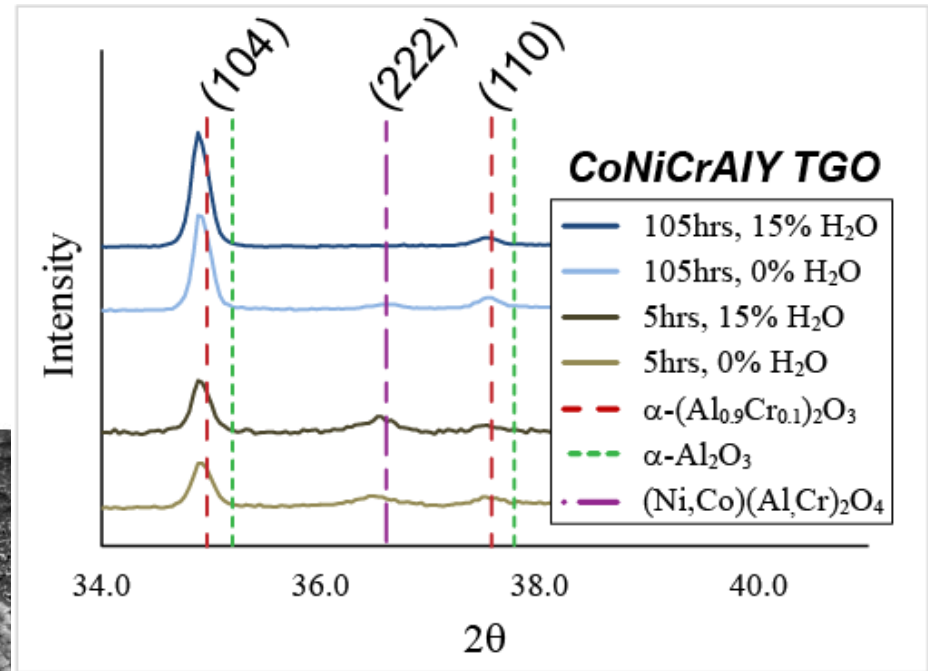
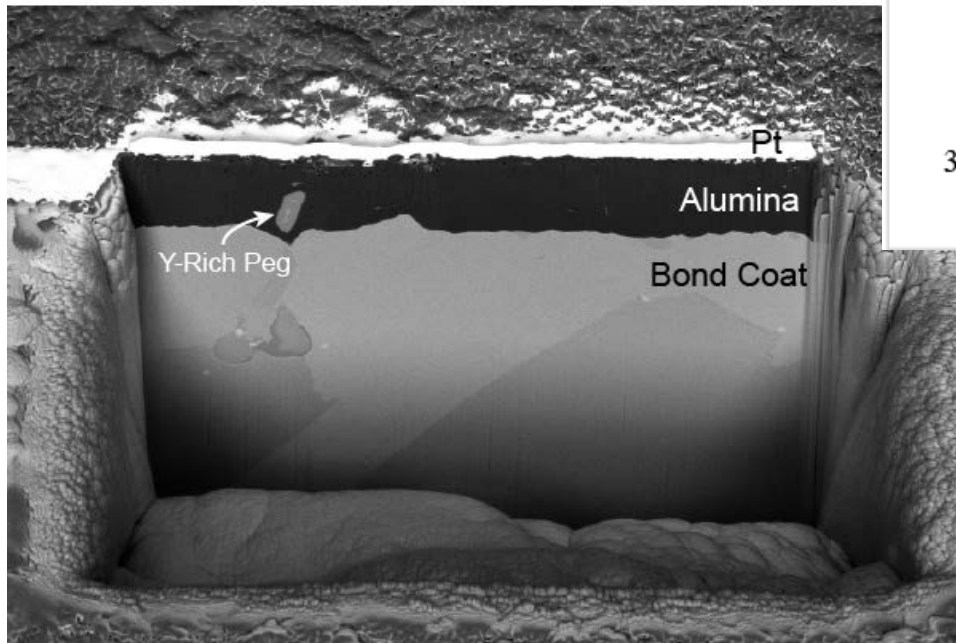
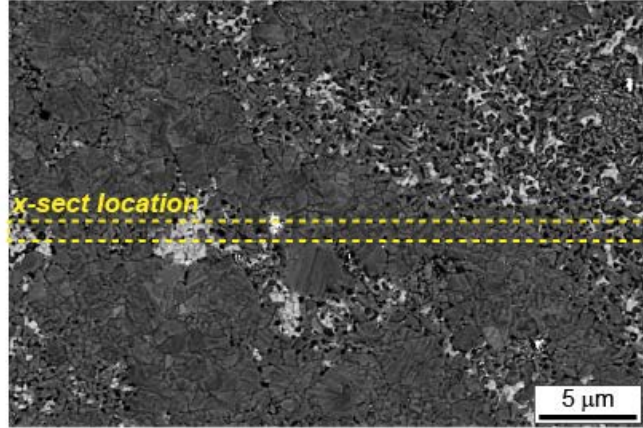
EDS (at%):



- EDS reveals a 1:2 atomic ratio of A to B, indicating this is an AB_2O_4 spinel

H₂O-Related Artifacts

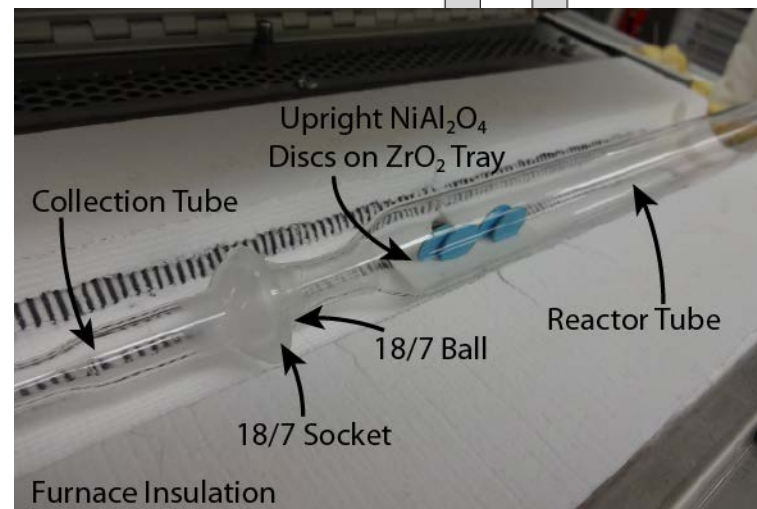
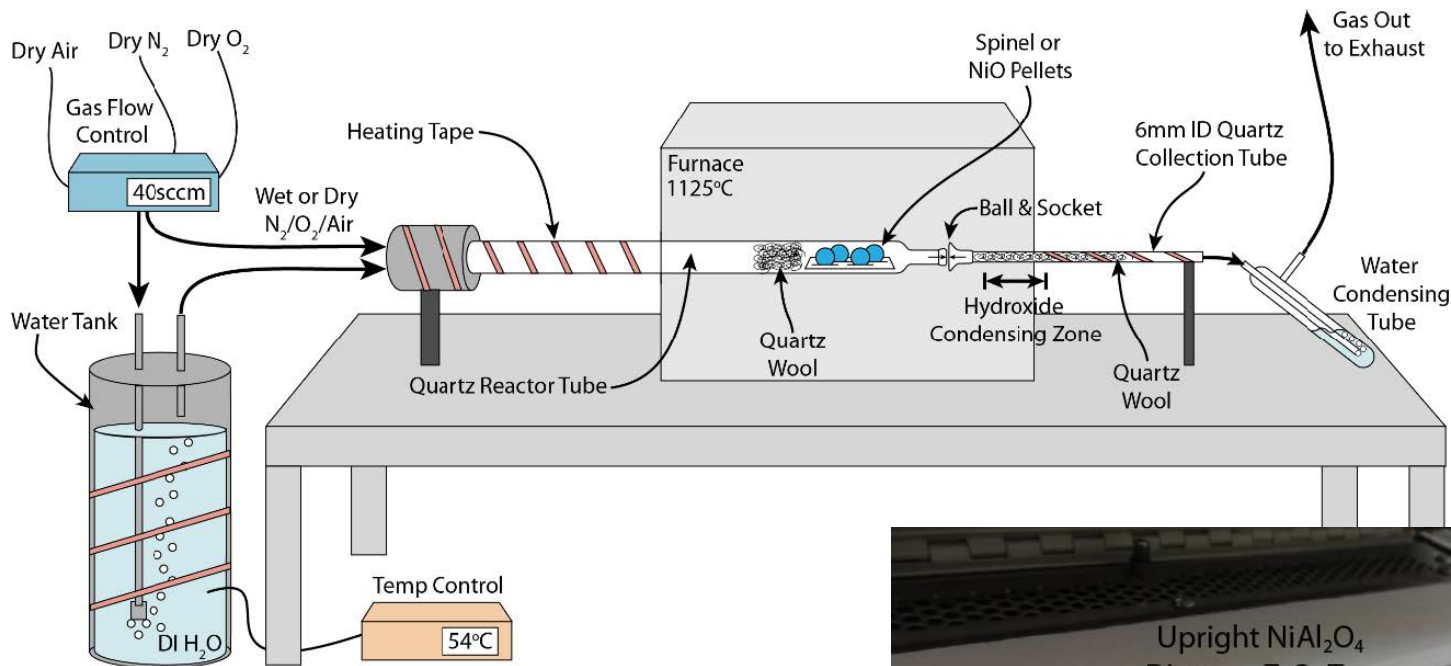
CoNiCrAlY: 15% H₂O, 105 hours



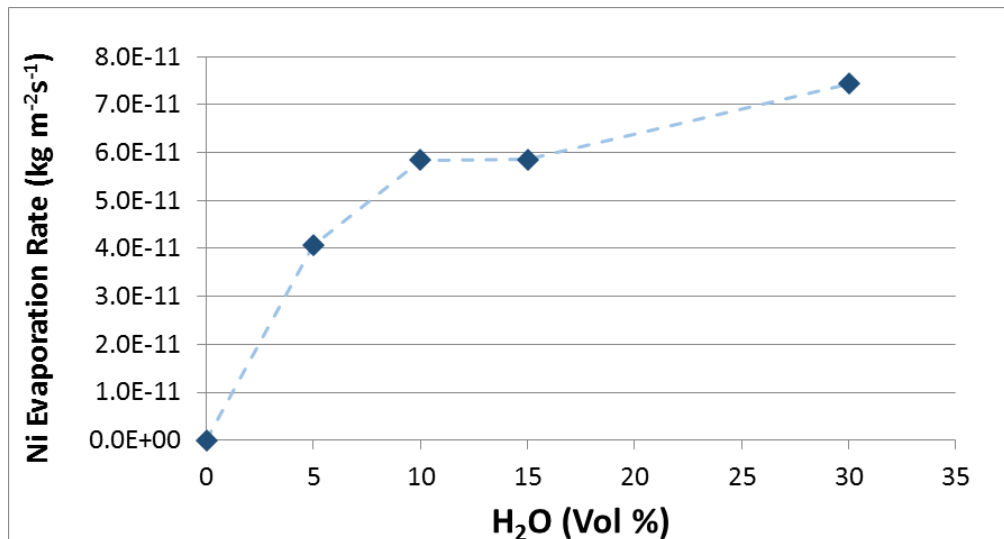
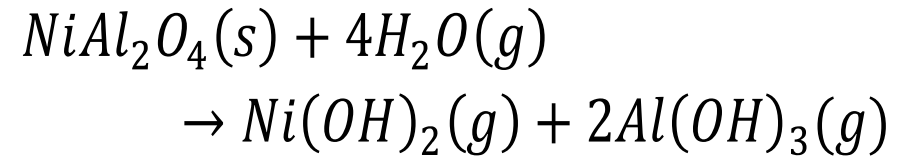
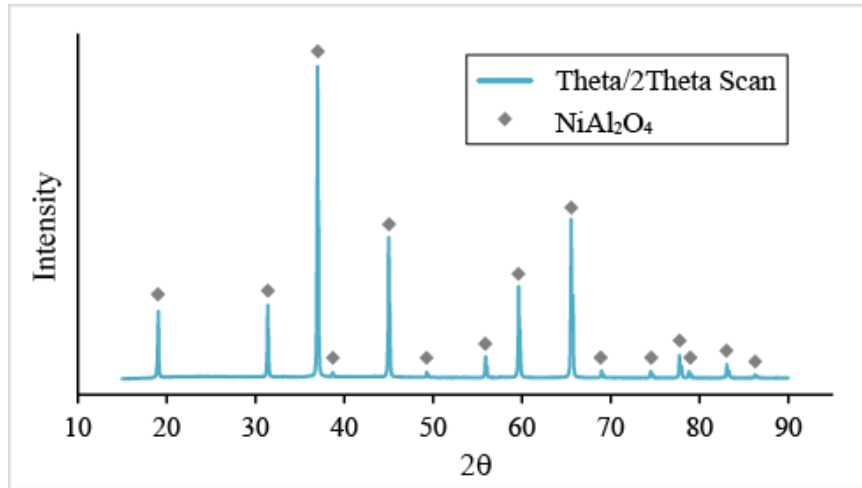
- Cross-section: No spinel found in oxide layer
- XRD: No heightened solubility for Ni, Co cations in wet vs. dry environment

Volatilization of NiAl_2O_4 Species

Modified transpiration experiments to verify volatilization of spinel cation species via atomic absorption spectroscopy:



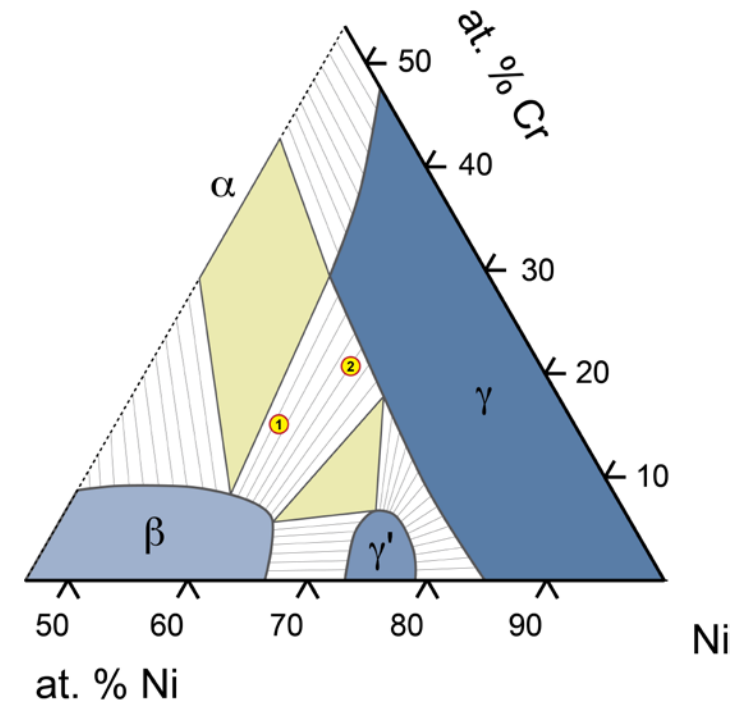
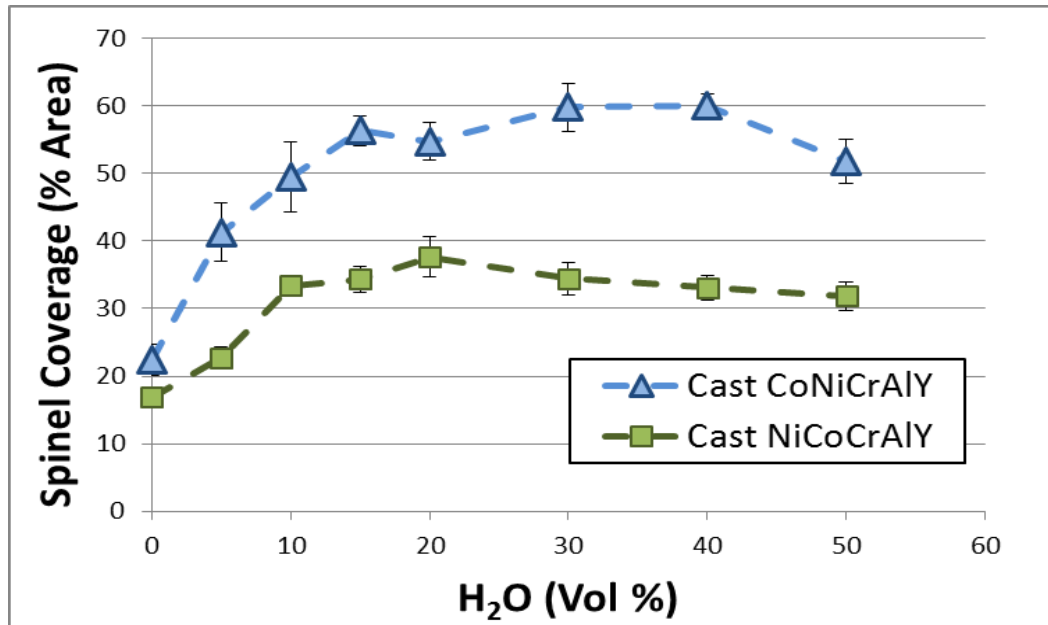
Volatilization of NiAl₂O₄ Species



- Evaporation of Ni from pure NiAl₂O₄ discs is confirmed
- Evaporation at 1125^oC is about an order of magnitude less than Cr at 800^oC



Transient Oxidation



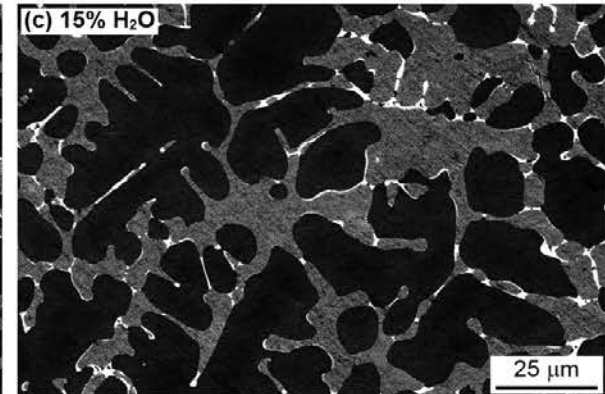
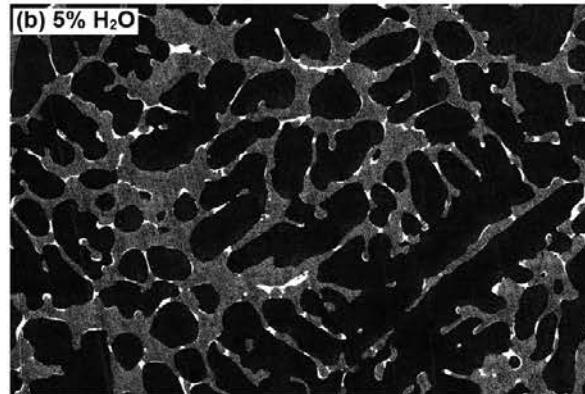
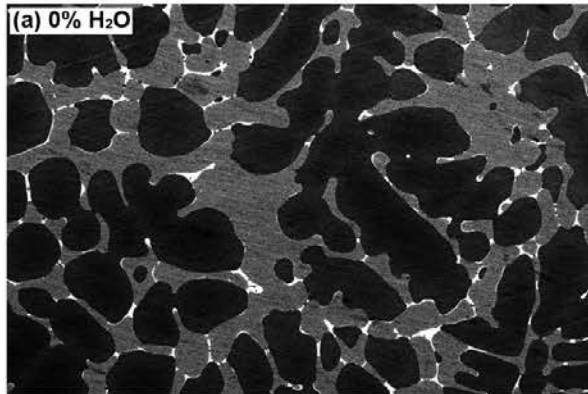
- Using the thresholding technique again on low magnification images, spinel coverage in transient stage is found to peak between 15-30% H₂O

Transient Oxidation

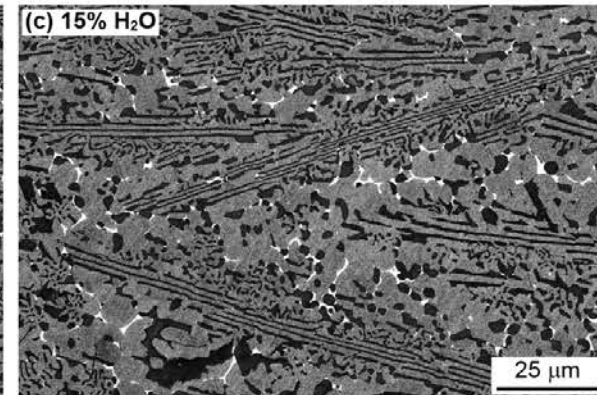
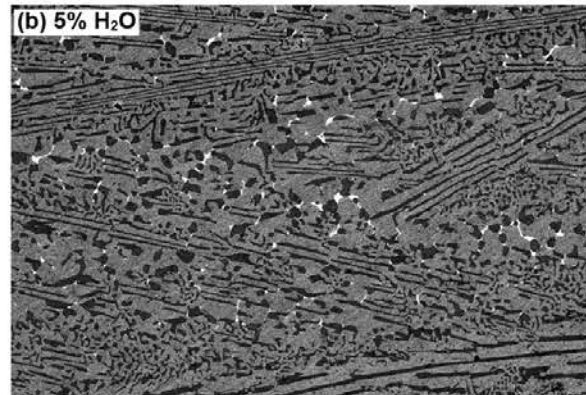
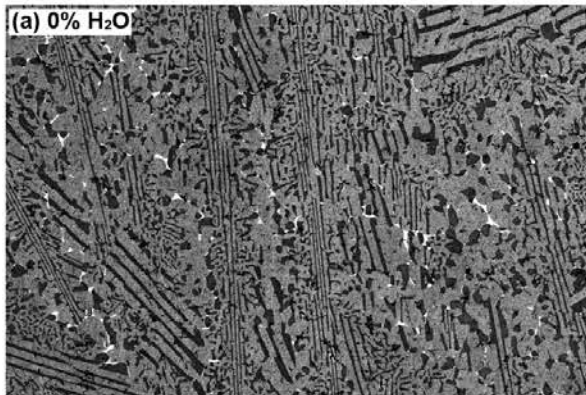
0%H₂O

5%H₂O

15%H₂O



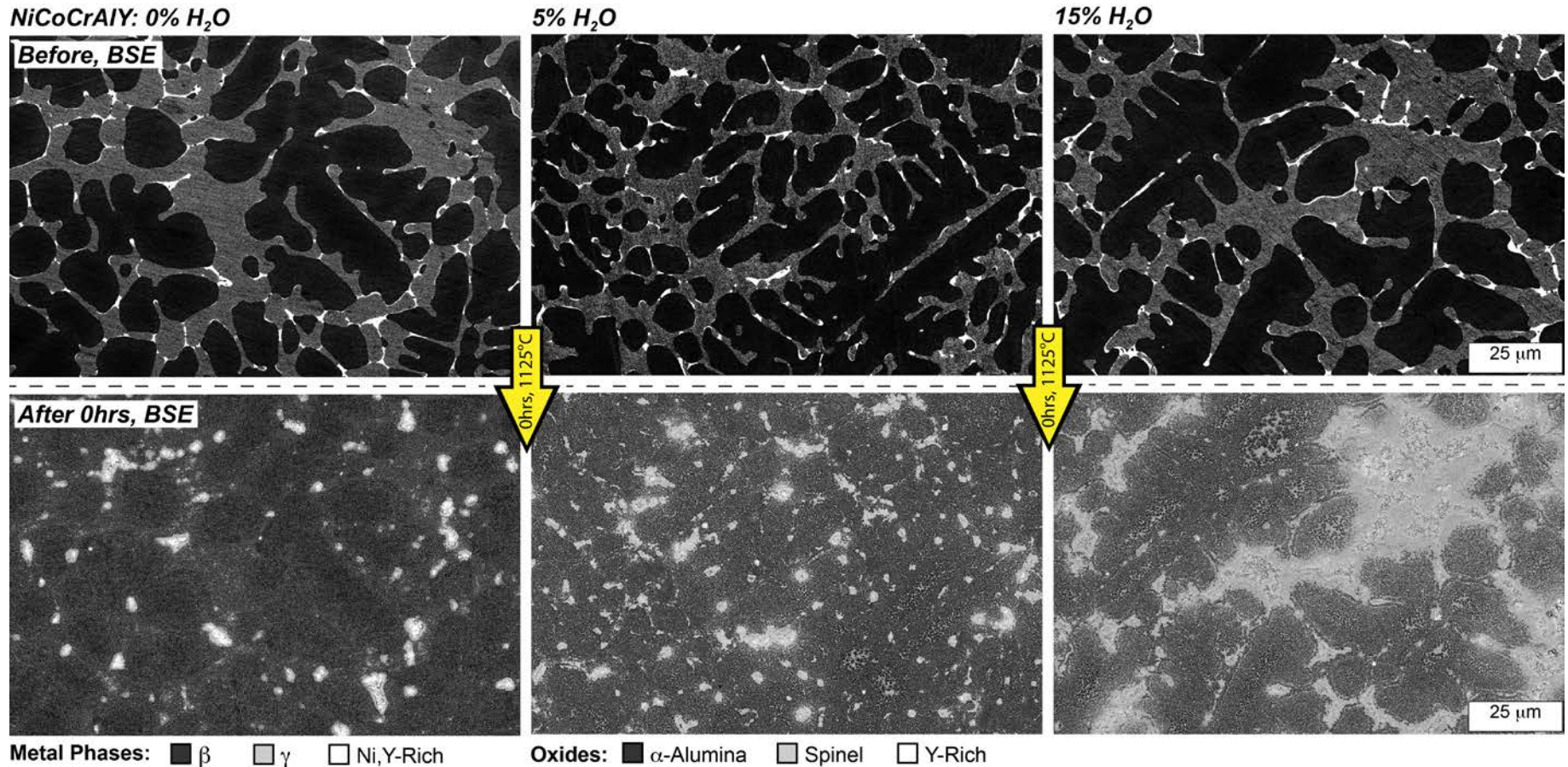
BEFORE → Cast NiCoCrAlY Alloy Phases: β (Al-Rich) γ (Al-Poor) Ni,Co,Y-Rich
 Oxide Phases (0 hrs, 1125°C): α-Alumina Spinel NiO



BEFORE → Cast CoNiCrAlY Alloy Phases: β (Al-Rich) γ (Al-Poor) Ni,Co,Y-Rich
 Oxide Phases (0 hrs, 1125°C): α-Alumina Spinel NiO



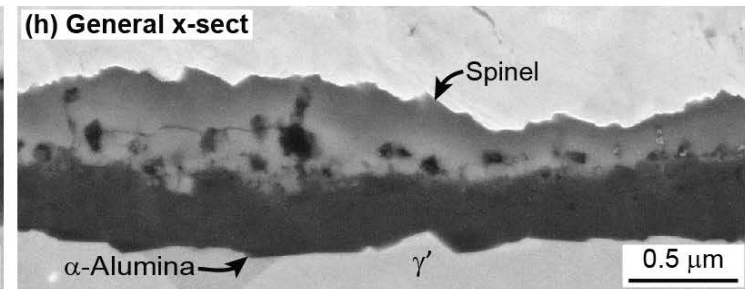
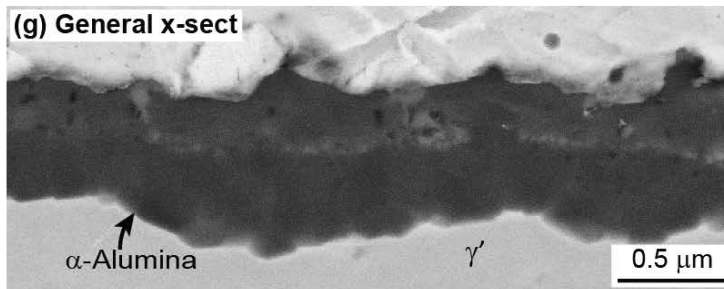
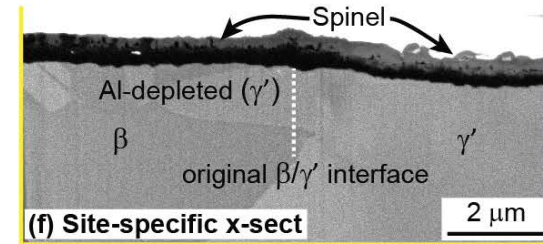
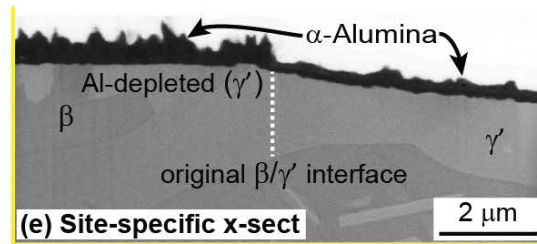
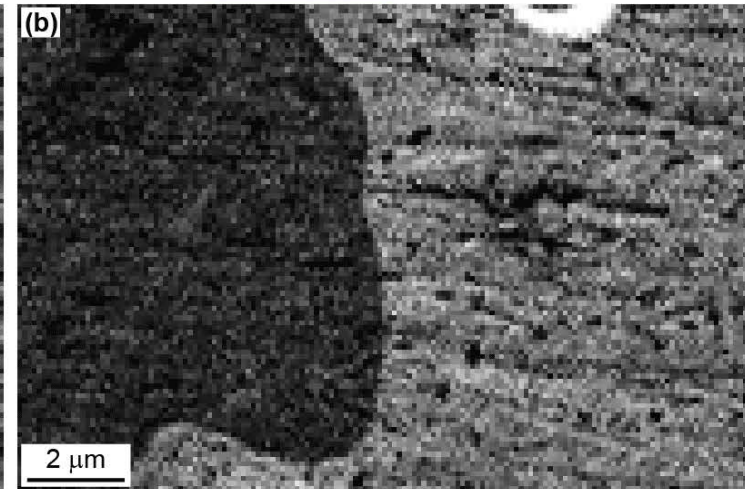
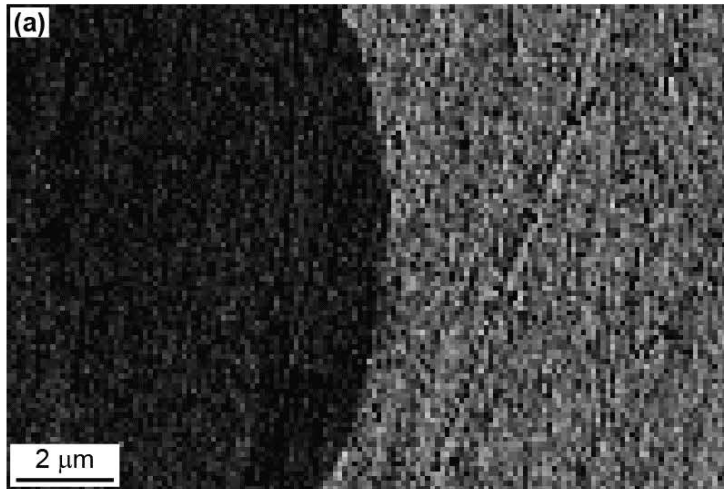
Transient Oxidation



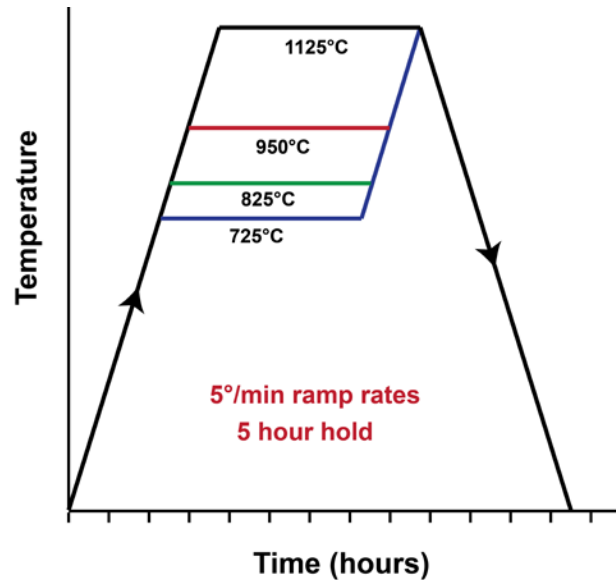
- Increasing H₂O allows for spinel to grow over progressively more Al-rich alloy phases

Transient Oxidation – Site Specific

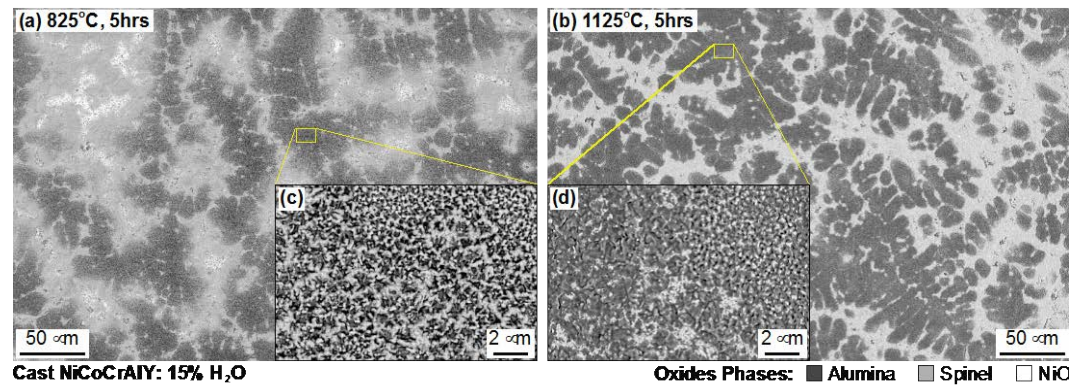
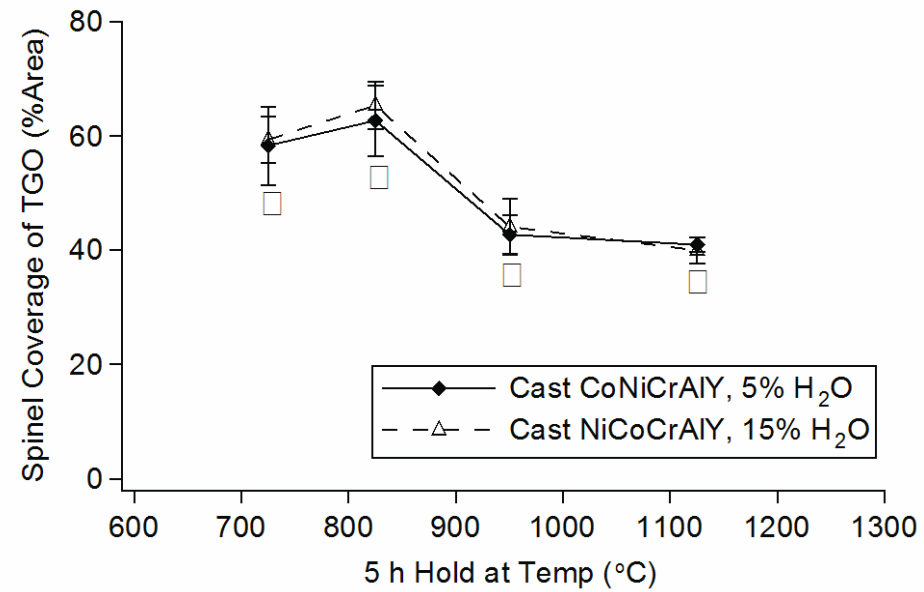
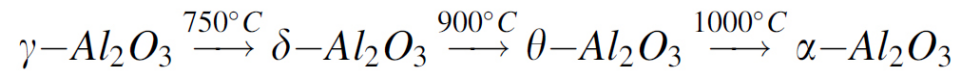
BEFORE → Cast NiCoCrAlY Alloy Phases: β (Al-Rich) γ' (Al-Poor) M,Y-Rich
 Oxide Phases (0 hrs, 1125°C): α -Alumina Spinel x-sect location



Metastable Alumina Phases and Transient Spinel Formation



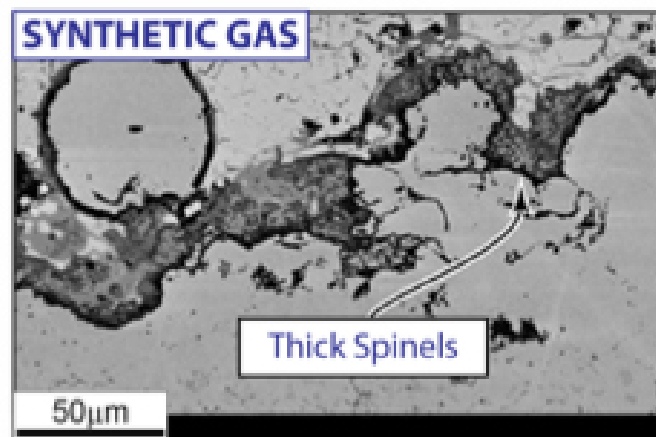
By staging lower temp holds – when alumina exists as a cubic or tetragonal, defect-spinel structure – cations more readily participate in the formation of spinel during the transient stage. Water vapor prolongs the lifetime of the defect-spinel metastable aluminas (γ and δ), in particular.



Worst Case Scenarios

- Given what we've learned, what is the “worst” we can do, to try to match the motivating figure?

Flow Segment ID	Gas Composition				
	Units	GE Case 2	CoP Case 4	Shell Case 6	Range
Clean High-H ₂ Syngas	H ₂	91%	76%	86%	76-91%
	H ₂ O	0%	14%	3%	0-14%
	CO	2%	1%	3%	1-3%
	CO ₂	4%	2%	2%	2-4%
	Ar	1%	1%	1%	1%
	N ₂	1%	1%	5%	1-5%
Turbine Exhaust	N ₂	75%	74%	75%	74-75%
	H ₂ O	12%	14%	13%	12-14%
	O ₂	11%	10%	11%	10-11%
	CO ₂	1%	1%	1%	1%
	Ar	1%	1%	1%	1%

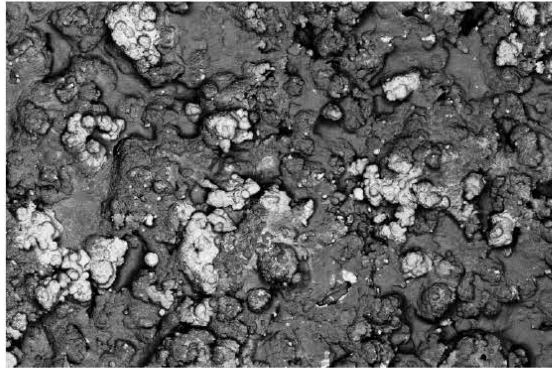


1. CoNiCrAlY has the worst oxidation resistance
2. 30% H₂O, the high for syngas (when steam is added to 12-14% baseline for NO_x reduction) yields most spinel for CoNiCrAlY
3. Low O₂ (10%) yields most spinel for CoNiCrAlY and is the syngas guidance
4. Add CO₂ to simulate syngas combustion

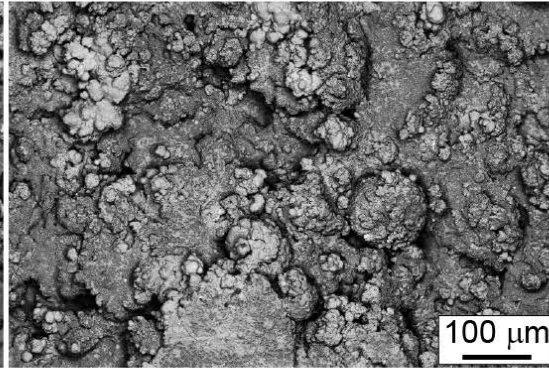
Transient Oxidation

Sprayed CoNiCrAlY: 0 hours

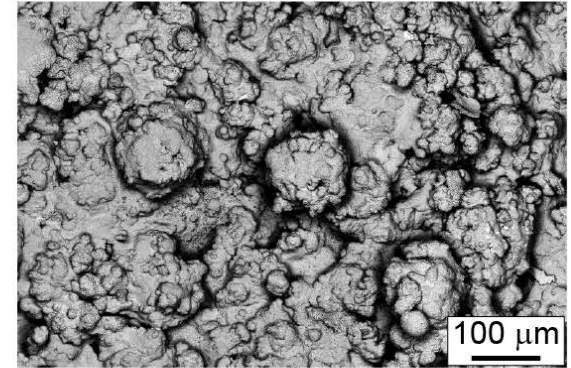
■ α -Alumina □ Spinel



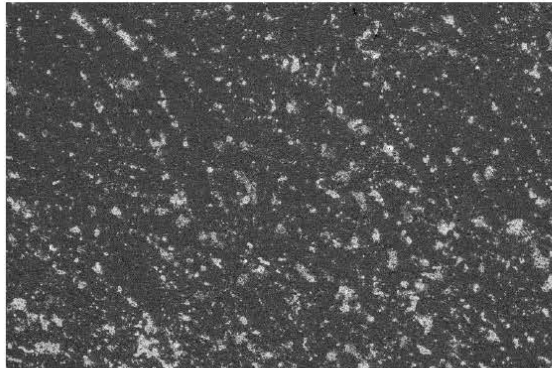
0% H₂O



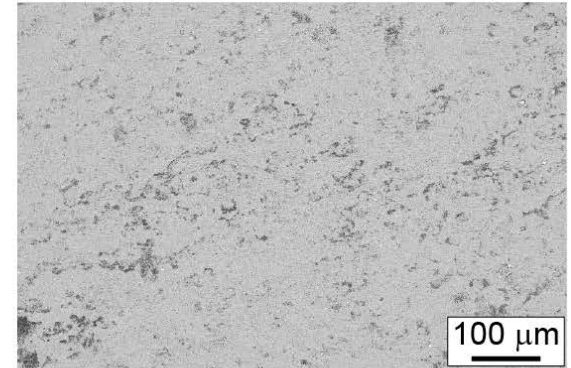
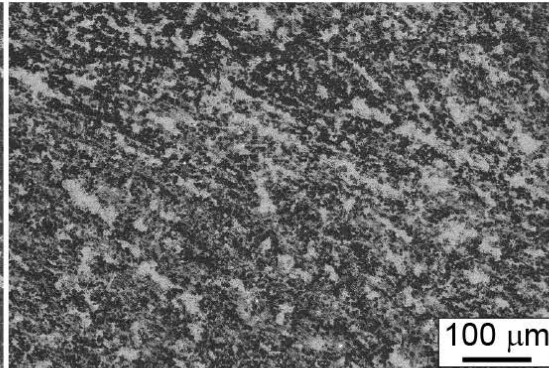
15% H₂O



30% H₂O, 10% O₂, 2% CO₂ (5 hours)



Cast CoNiCrAlY: 0 hours

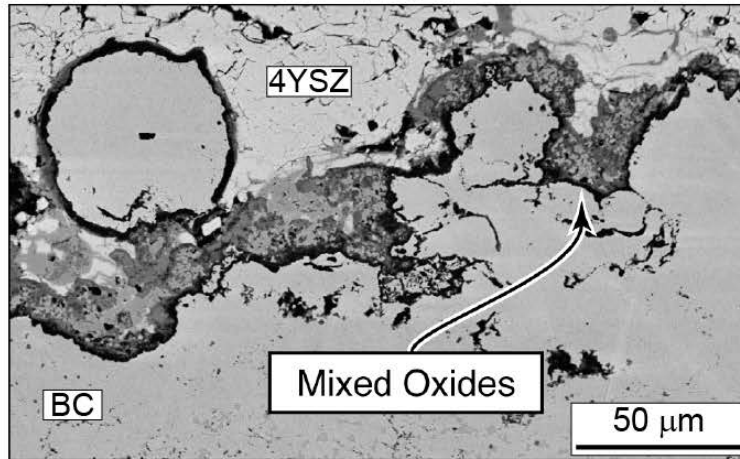


- The highest H₂O scenario with syngas combustion matches the worst-case spinel coverage scenario in the lab.
- Adding 2% CO₂ to the worst case scenario yields *complete* spinel coverage.

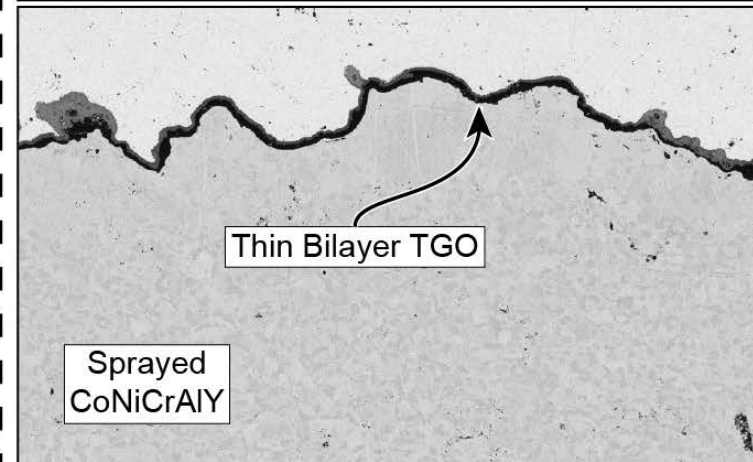
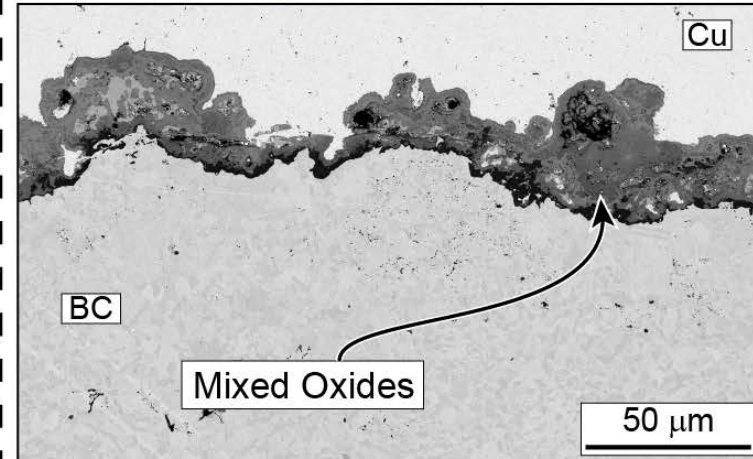


Transient Oxidation

a. In Service TGO via Syngas Combustion



b. In Lab: 30% H₂O, 10% O₂, 2% CO₂, 5 hours



- The problematic TGOs from syngas combustion can be recreated in very short laboratory exposures.
- Transient layer development is strongly dependent upon combustion environment.
- All of the spinel problems likely stem from the first several hours of exposure – transient stages of TGO development

□ Effects of Water Vapor on the Stability of YSZ Coatings

Controlled environment tube furnace systems are utilized to run long-duration exposures of sample coupons to water vapor contents that bracket the behavior of syngas and high hydrogen content turbine systems. Will further examine phase changes, volatilization and erosion of seal coating materials as a function of the water vapor content, temperature and the type of coating microstructure. Baseline tests will be performed at identical temperatures but without water vapor, in an effort to elucidate the contribution of environment to de-stabilization.

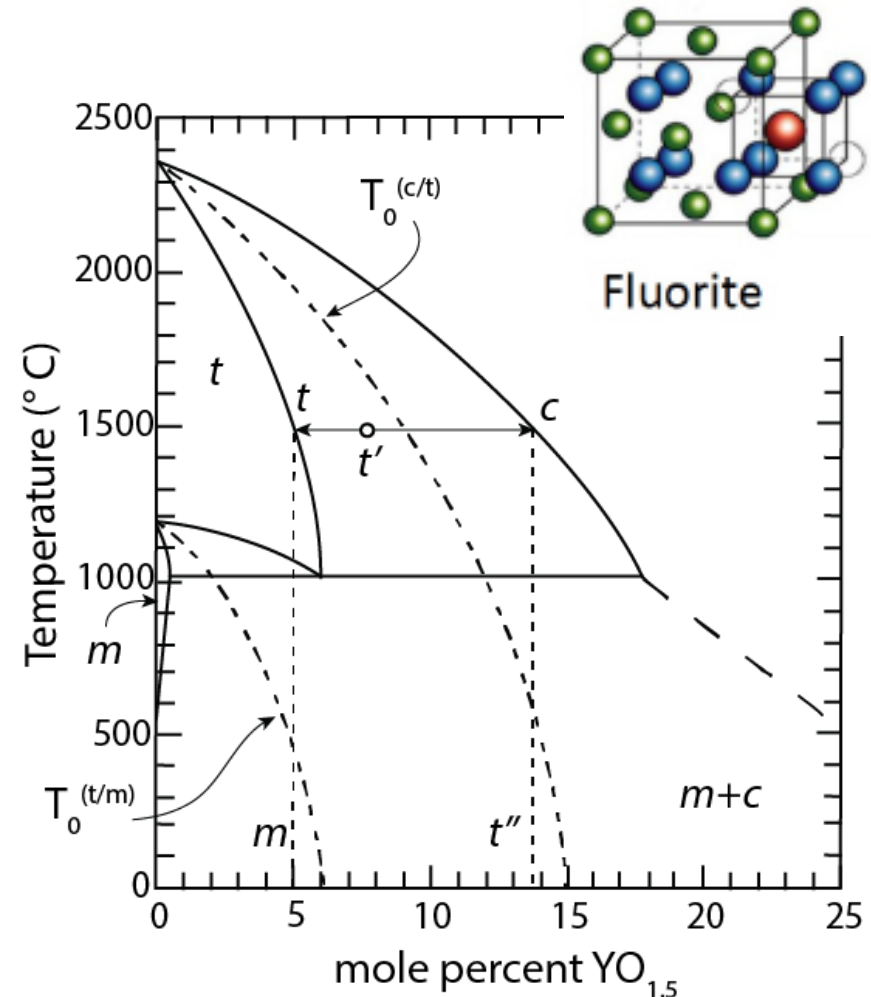
- * Developed test protocols to run tube furnace thermal exposures with simulated syngas and HHC fuel environments, based on ongoing research procedures, in comparison to simulated natural gas combustion environments.
- * The degradation, associated volumetric changes and degradation in potential sealing behavior will be evaluated in relation to the combustion environment, as preliminary work for performance as an abradable material.
- * Spectroscopic analysis will be performed of phase destabilization processes (using Raman spectroscopy and XRD analysis).



Aging of YSZ under IGCC-Relevant Environments

- Composition of yttria puts the 8YSZ in the tetragonal + cubic phase field
- t' phase will eventually decompose to these equilibrium phases
- Rate is dictated by ageing time and temperature
- Can normalize the influence of time and temperature by the use of the Larson-Miller or the Hollomon-Jaffe Parameter of the form:

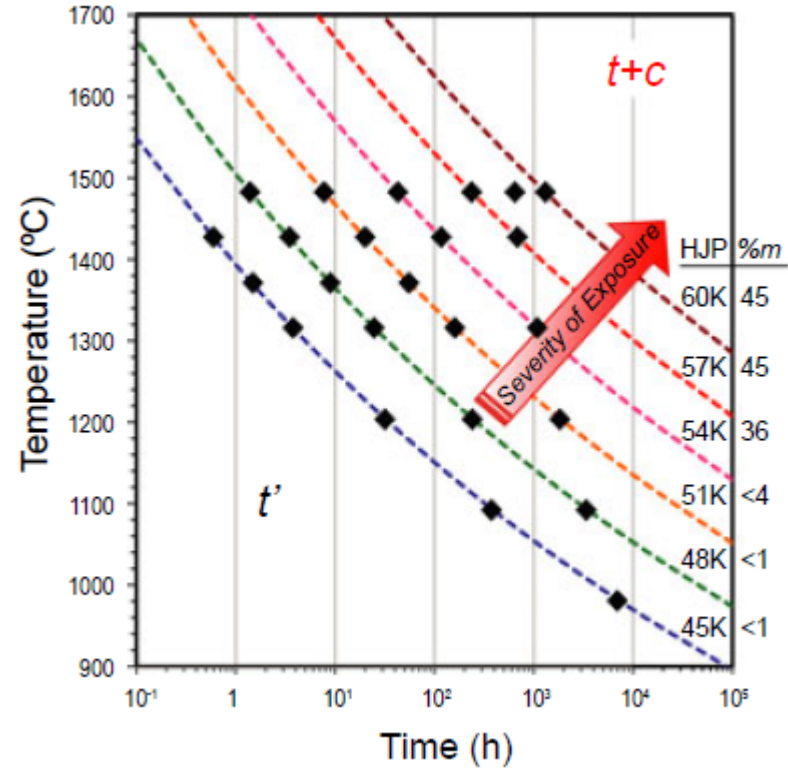
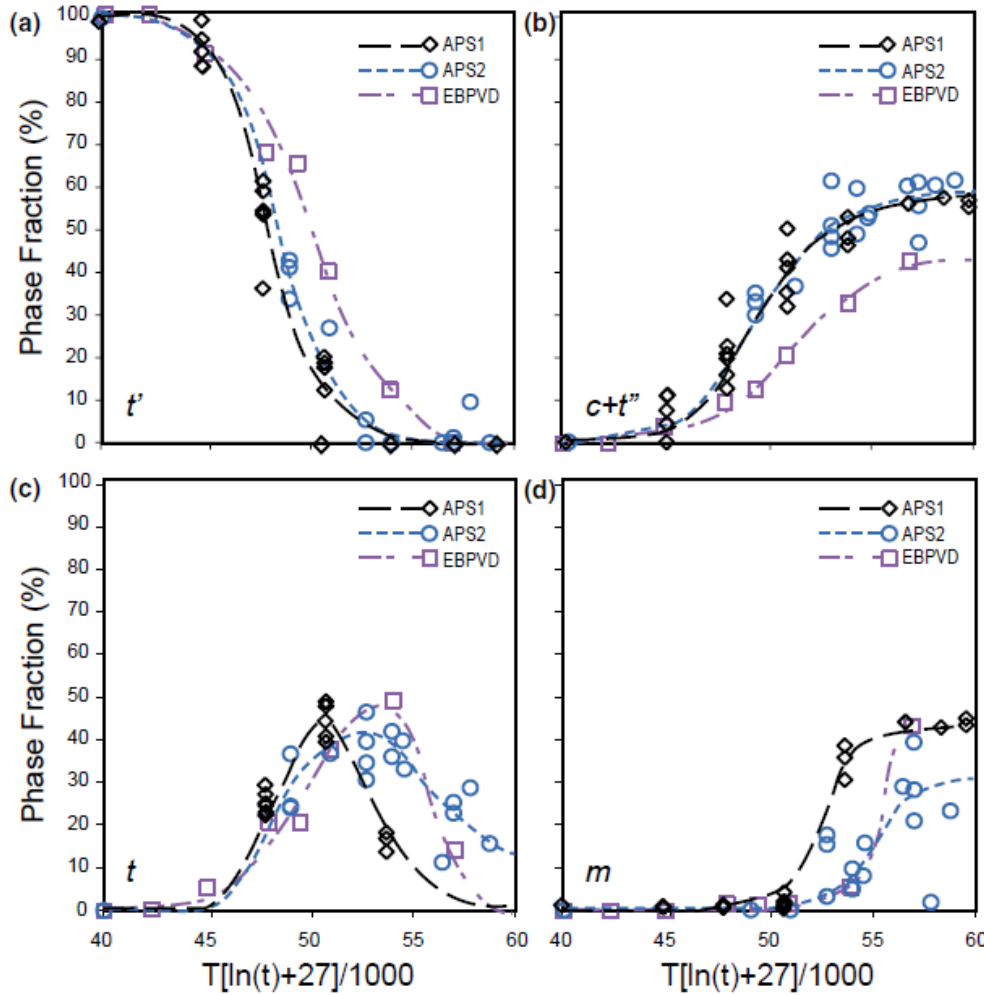
$$T[C + \ln(t)]$$



Redrawn from Levi *et al.*,
J. Am. Ceram. Soc., 96 [1] 290-298 (2013)



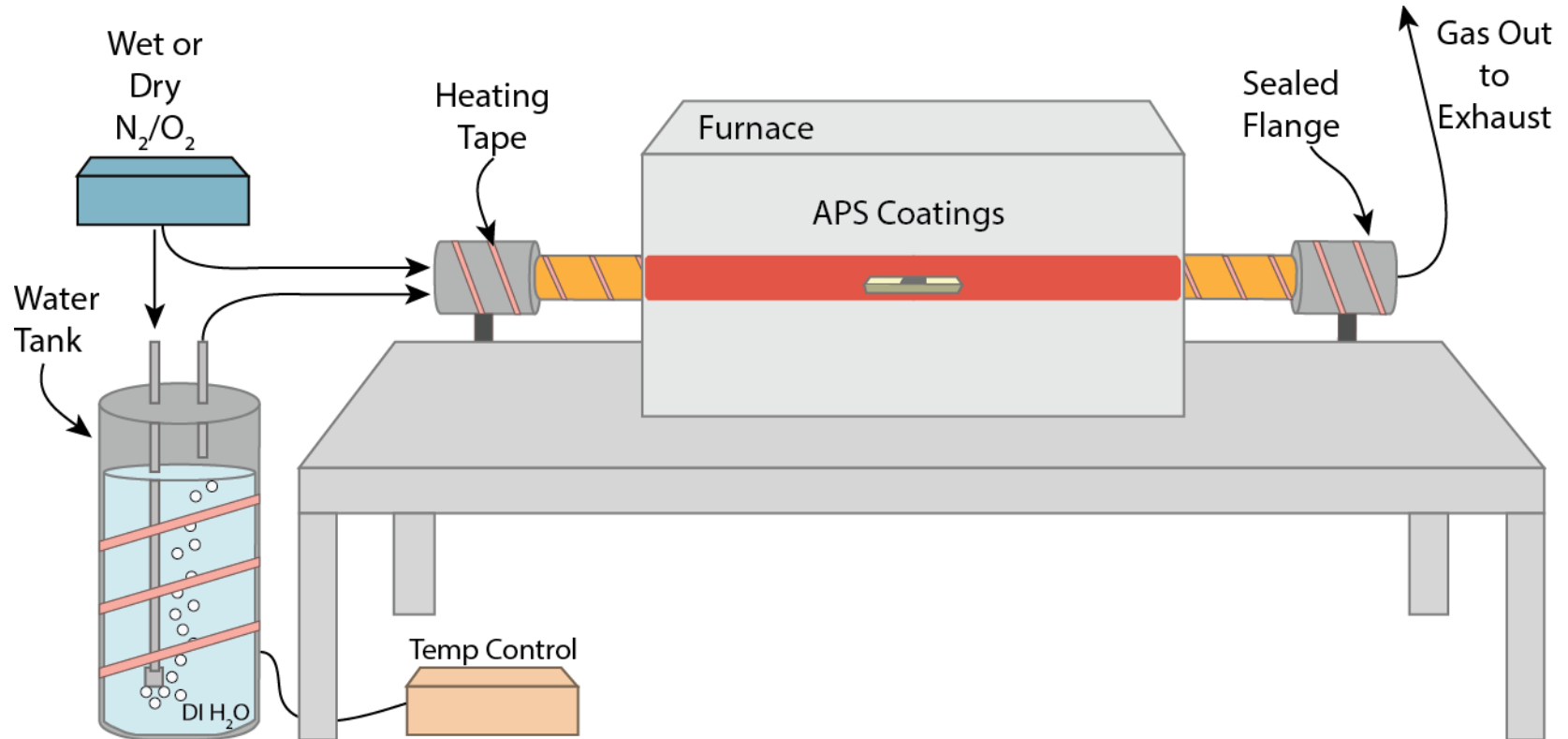
Prior Research Findings



How does a humid environment influence the destabilization of the t' phase during ageing?

Levi *et al.*, *J. Am. Ceram. Soc.*,
96 [1] 290-298 (2013)

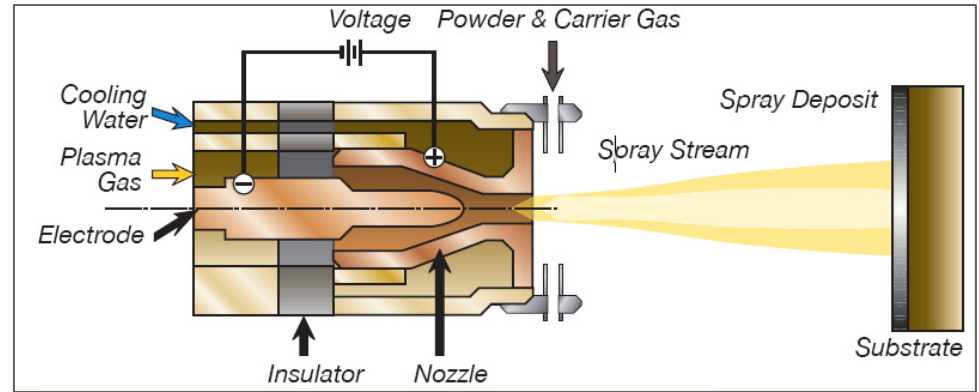
Experimental Methods



- Water tank temperature determines vol% H₂O via gas-liquid equilibrium exchange
- For 0% H₂O, the water tank is bypassed completely
- Exposed to dry or humid ageing (45 vol. %) in a controlled environment
- Air plasma spray, 8 wt.% Ytria-Stabilized Zirconia

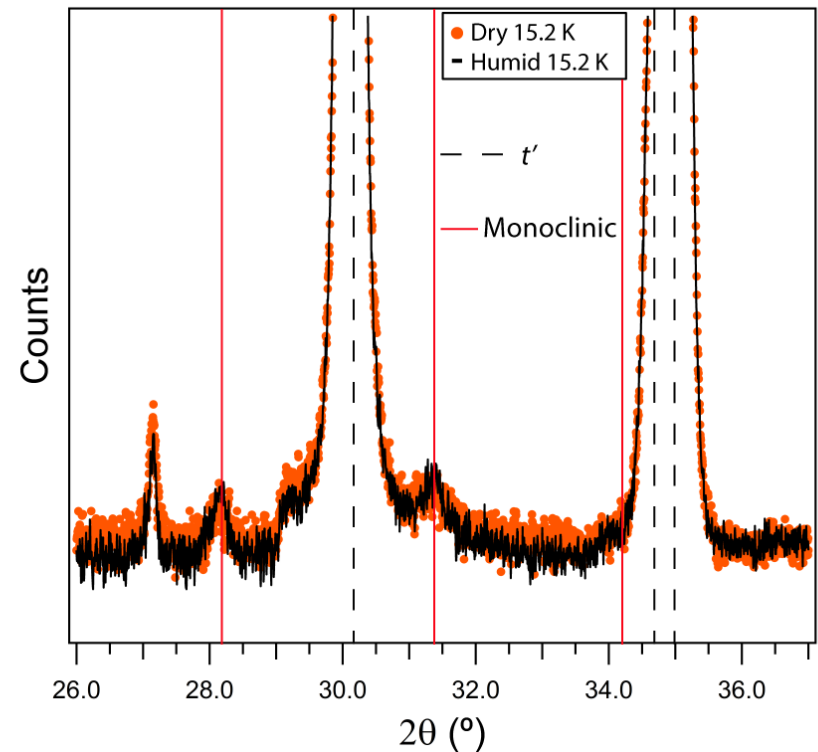
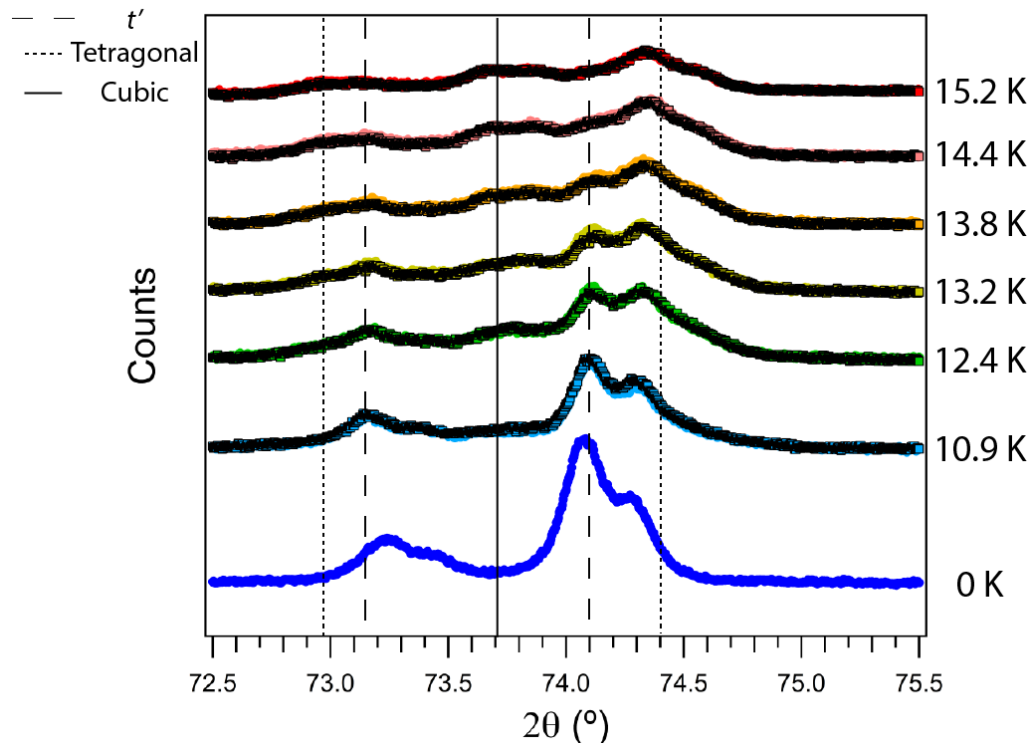


TBC Degradation Studies: Materials Preparation



Aging to a Max LMP of 15.2k (88 hours at this Temp)

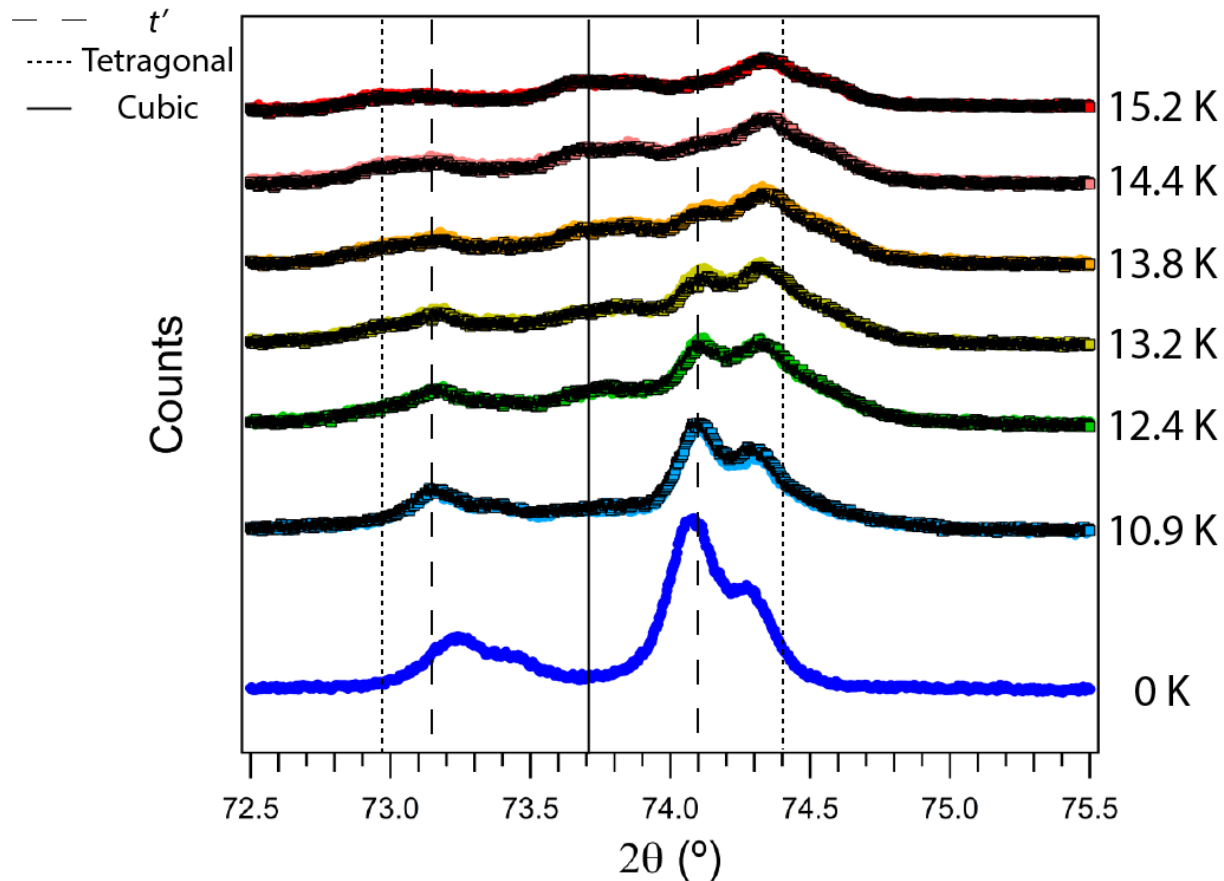
- Dry and humid environments decompose the t' phase at the same rate up to this point



- XRD results overlap for the (004) and (220) tetragonal planes and the (004) cubic plane on the left as well as the three main peaks of the monoclinic phase—(), (111), and (002) on the right



Evidence in support of Spinodal Decomposition

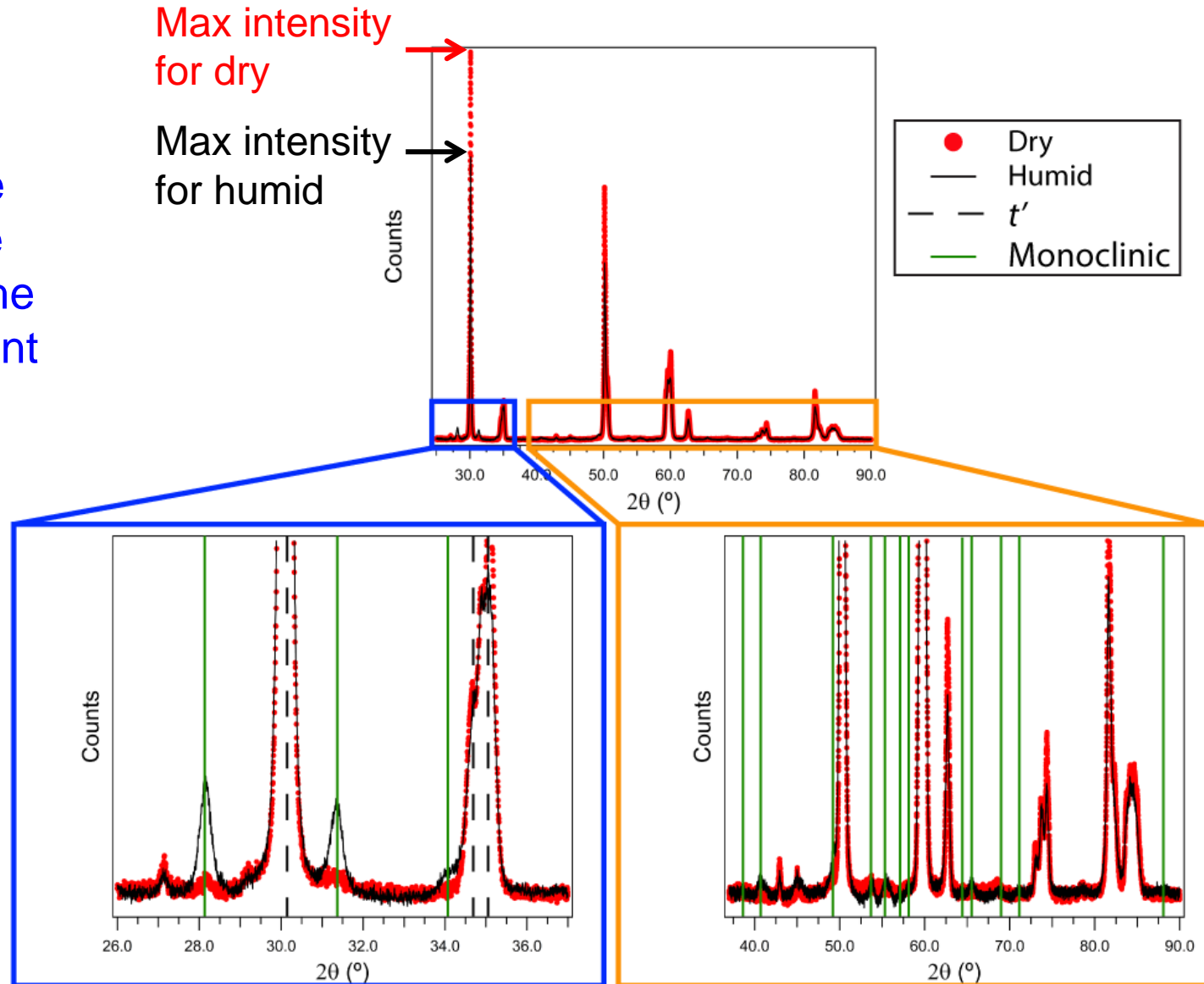


- Gradual shifting of the XRD peaks
- Indicates a range of lattice parameters as the t' destabilizes to the tetragonal and cubic phases

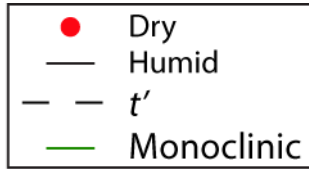


Further Aging to LMP = 15.9k [Environmental Dependence Observed]

The growth of the monoclinic phase occurs faster in the humid environment (after 144 Hrs)



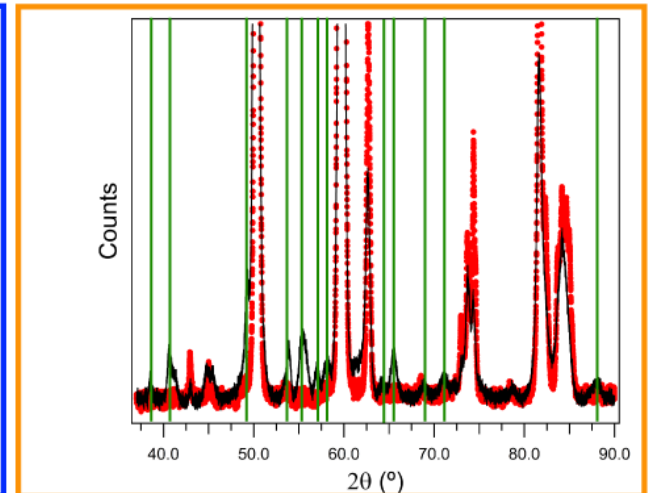
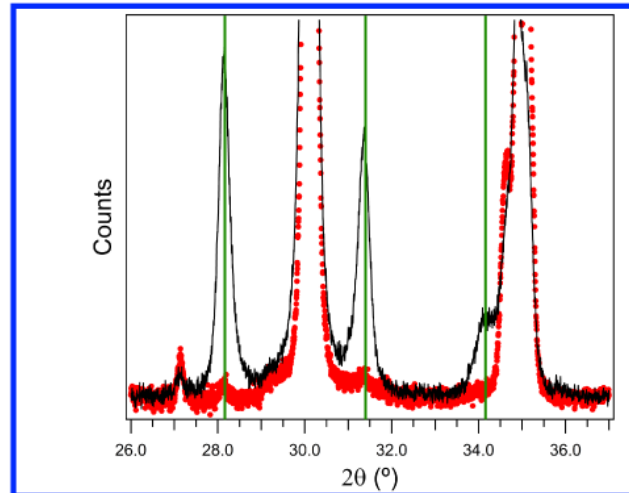
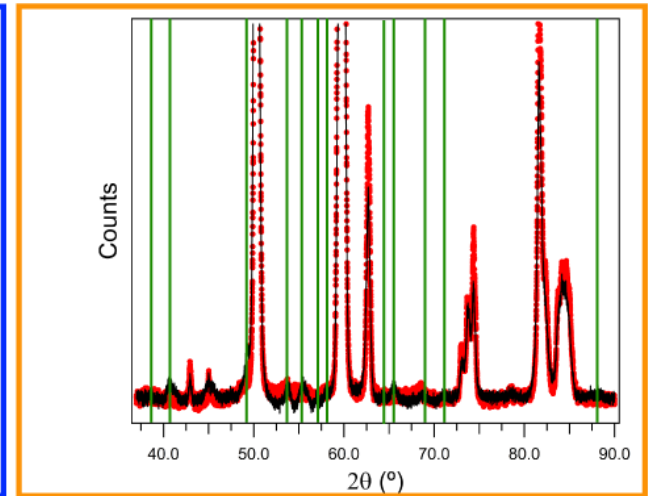
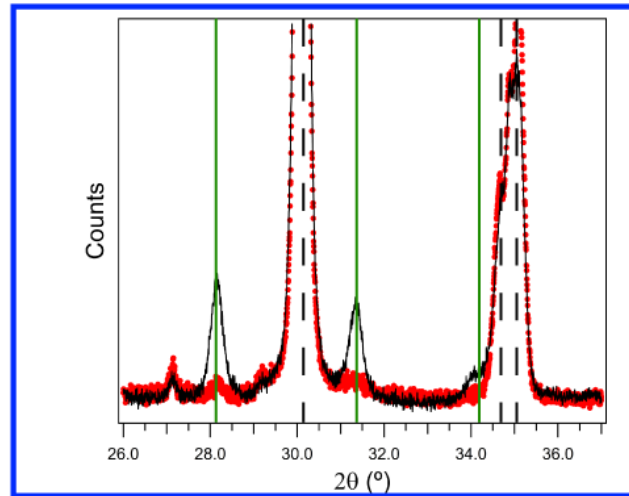
Further Aging to LMP = 16.7k (224 Hrs)



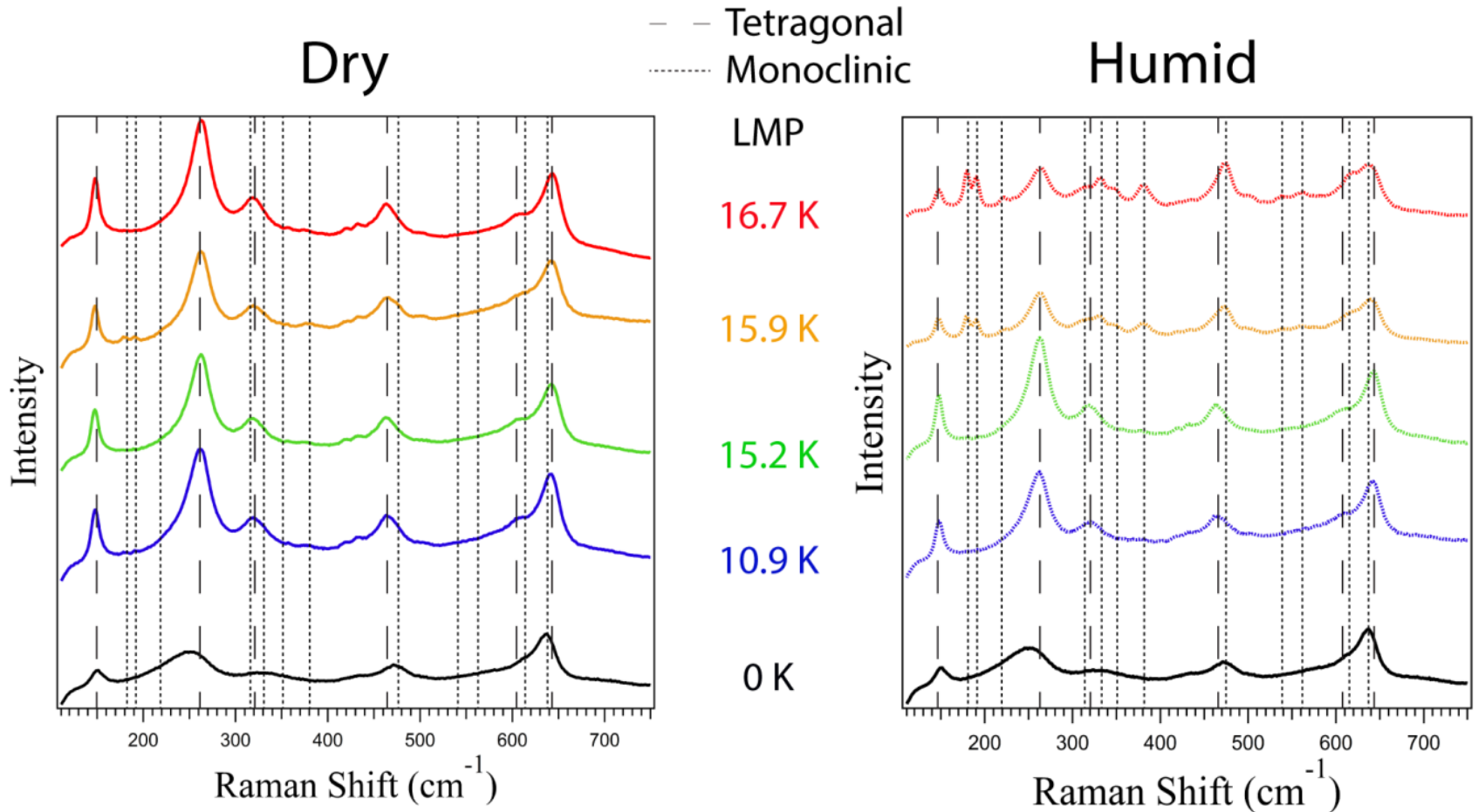
LMP of 15.9 K →

- After the next ageing step, the trend of enhanced $t \rightarrow m$ phase transformation continues

LMP of 16.7 K →



Raman Spectroscopy Confirms XRD Results

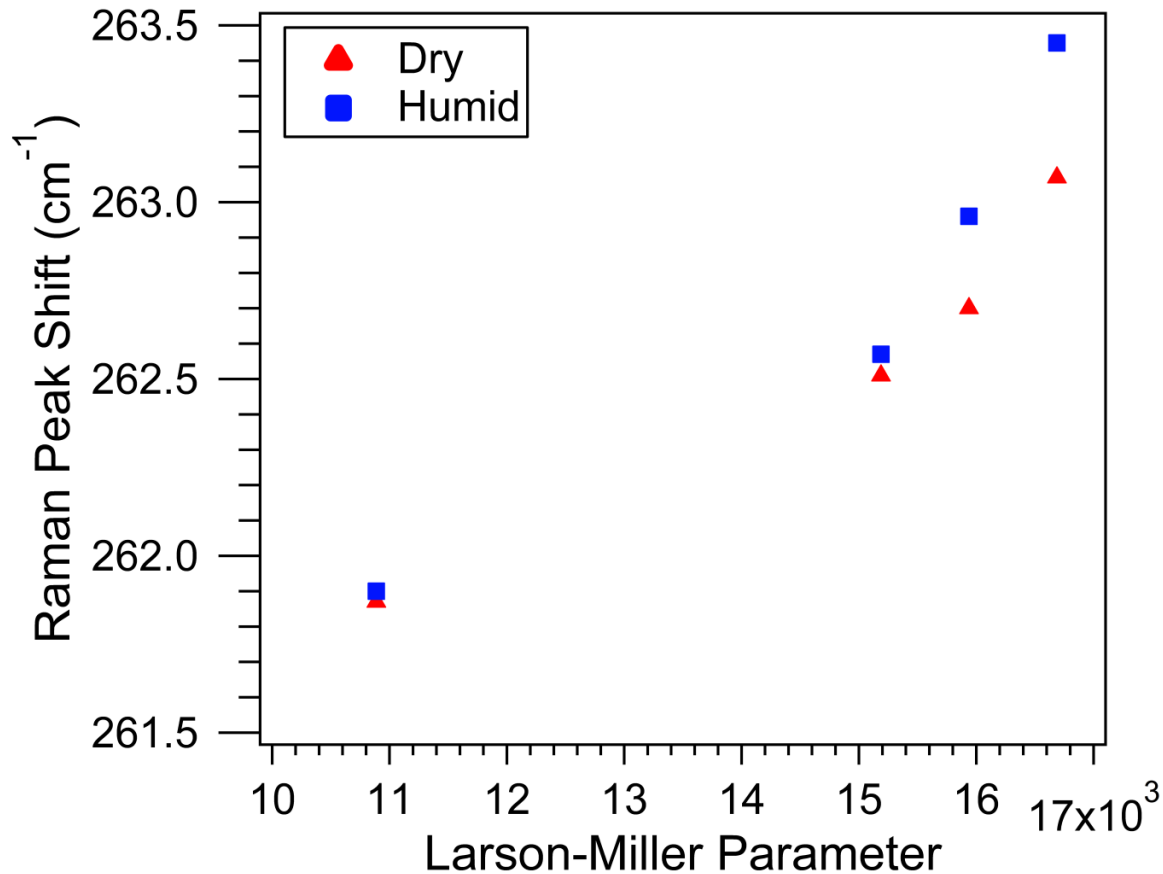


Raman results corroborate the XRD results

- Only the tetragonal modes are observed up to a LMP of 15.2 K
- For LMPs 15.9 K and 16.7 K, the monoclinic modes are now present



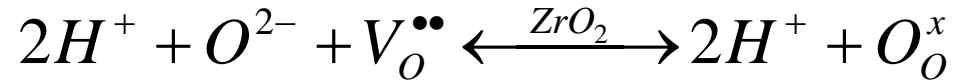
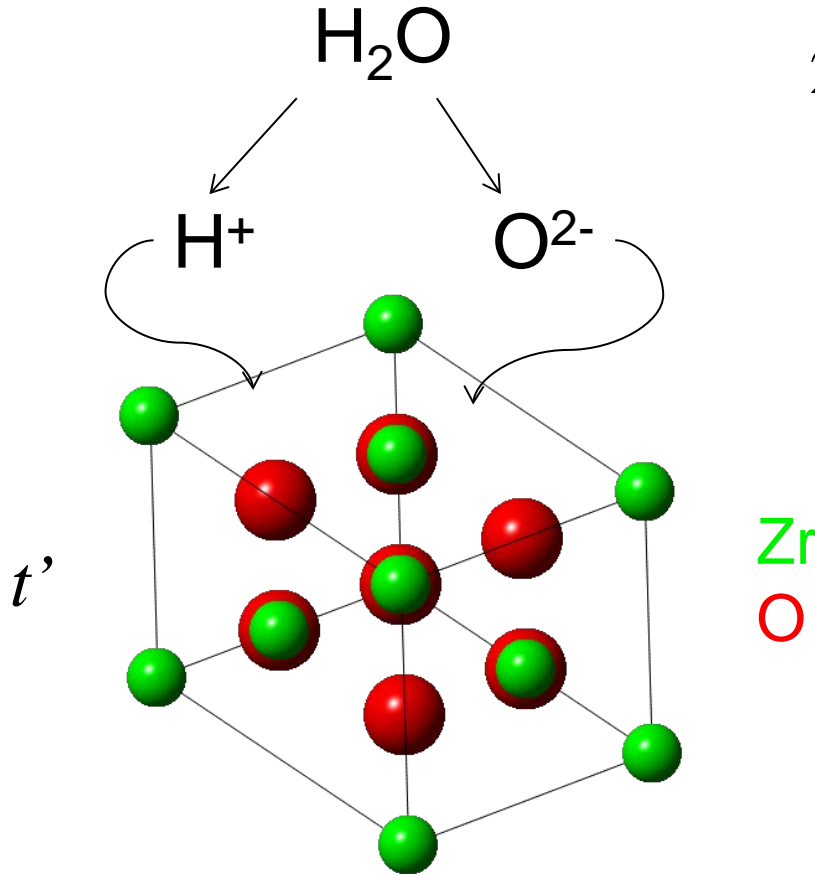
Raman Spectroscopy Supports Spinodal Decomposition Mechanism



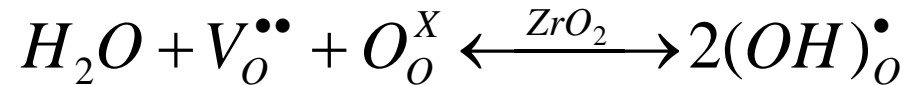
- Peak fitting of the A_{1g} mode shows peak shifting in both environments
- Indicates a continuous change in the lattice parameters
 - Peak shifts faster for humid-ageing condition



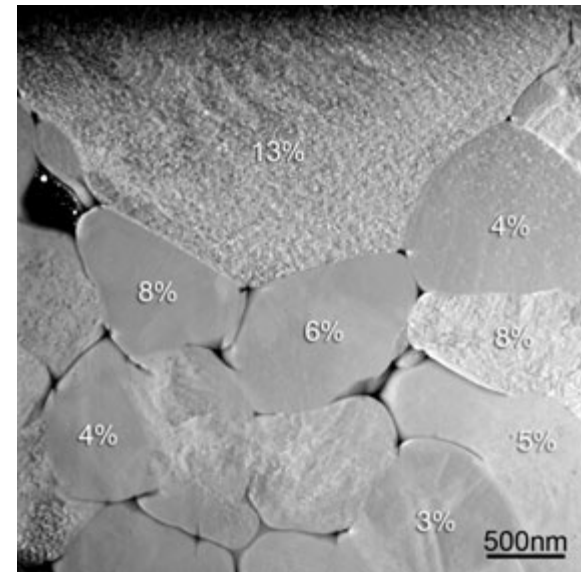
Possible Underpinning Mechanisms for Observed pH₂O Dependence



And/Or



Clarke *et al.*, *J. Am. Ceram. Soc.*, 92 [9] 1901-1920 (2009)



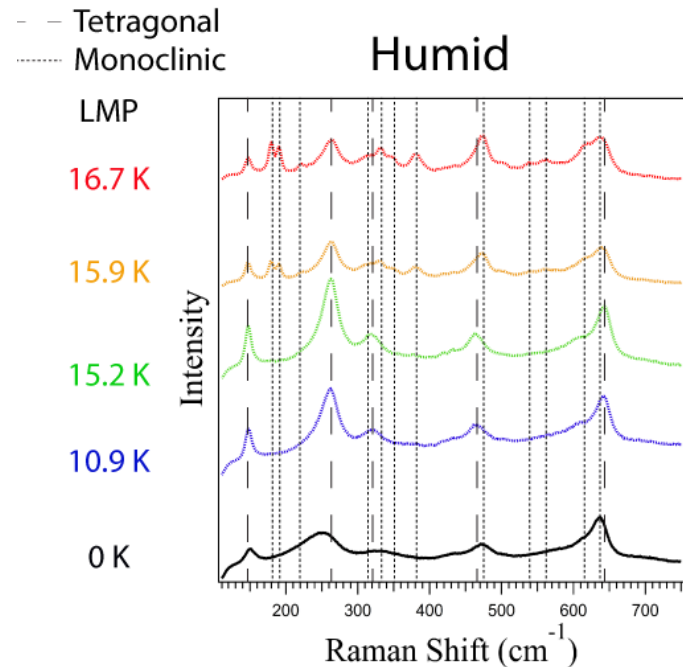
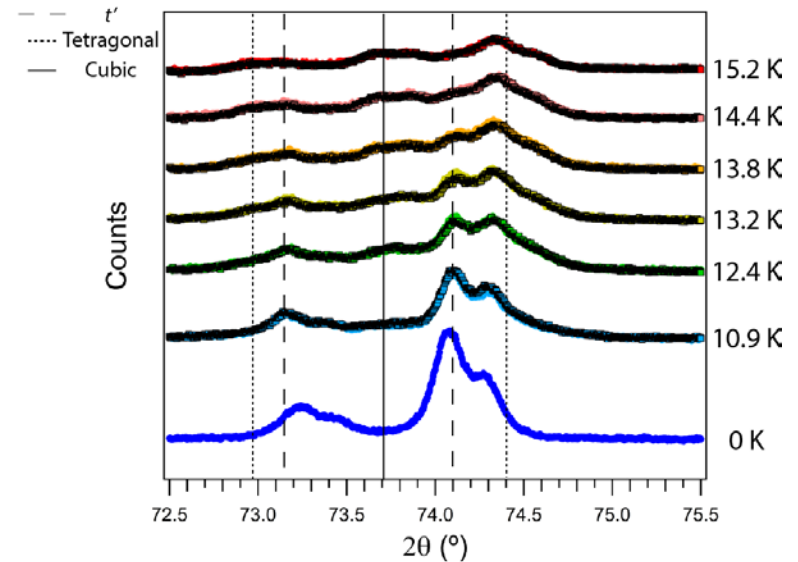
Levi *et al.*, *J. Am. Ceram. Soc.*, 96 [1] 299-307 (2013)

Silica impurities at grain boundaries may incorporate water species and enhance yttrium diffusion



Conclusions on YSZ Aging in IGCC-Relevant Environments

- Ageing of APS 8YSZ in controlled environments
- Evidence of spinodal decomposition from t' to tetragonal and cubic phases
- Humid ageing enhances the destabilization of the t' phase



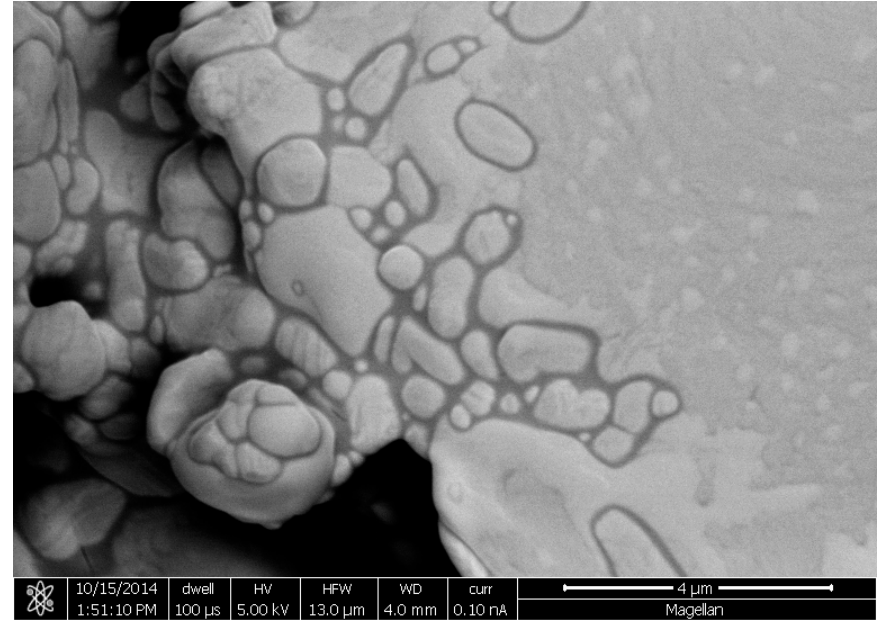
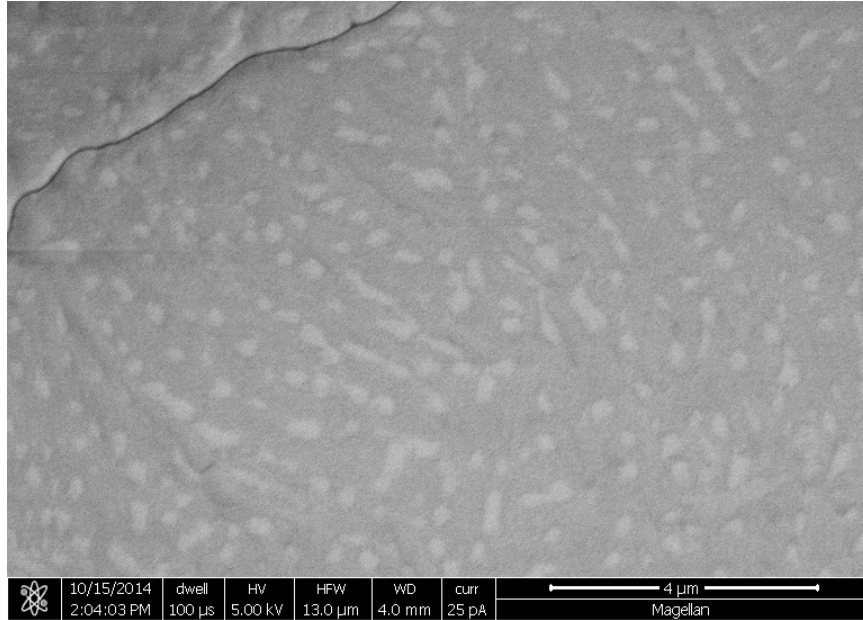
□ **Baseline Evaluation of Existing HT Abradable Seal Materials**

High temperature X-ray diffraction analysis, Raman spectroscopy, SEM characterization, weight change measurements and cross-sectional examination using the focused ion beam (FIB) will be used to quantify material removal and phase destabilization processes. Baseline tests will be performed in parallel in dry air, in an effort to elucidate the contribution of fuel-specific environments to TGO development. This task will identify the drivers for thermo-mechanical failure of the systems, and correlate the observed damage development with fuel-dependent aspects of the exposure environment.

- * Carry out extended oxidation experiments (100 hours) in simulated fuel environments at two target temperatures and under varying water vapor contents (current typical temperatures, and a higher temperature indicative of higher heat transfer with water vapor content environments).



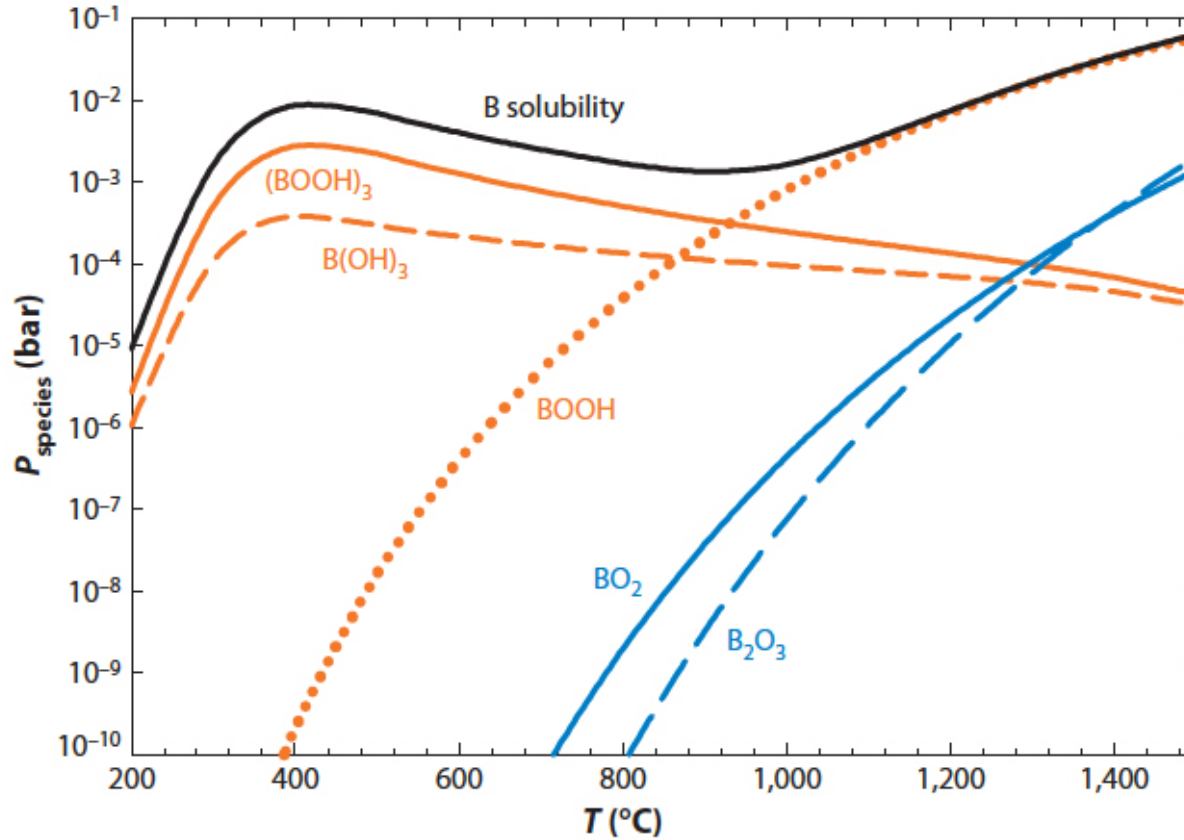
YSZ/hBN/Polyester Abradable System Behavior



- As processed materials show uniform distribution of BN phase
- Exposed samples show development of boron oxide or boro-silicate glass phases (developed in dry air exposure).
- Potential volatility with elevated $p\text{H}_2\text{O}$ levels



YSZ/BN System Behavior



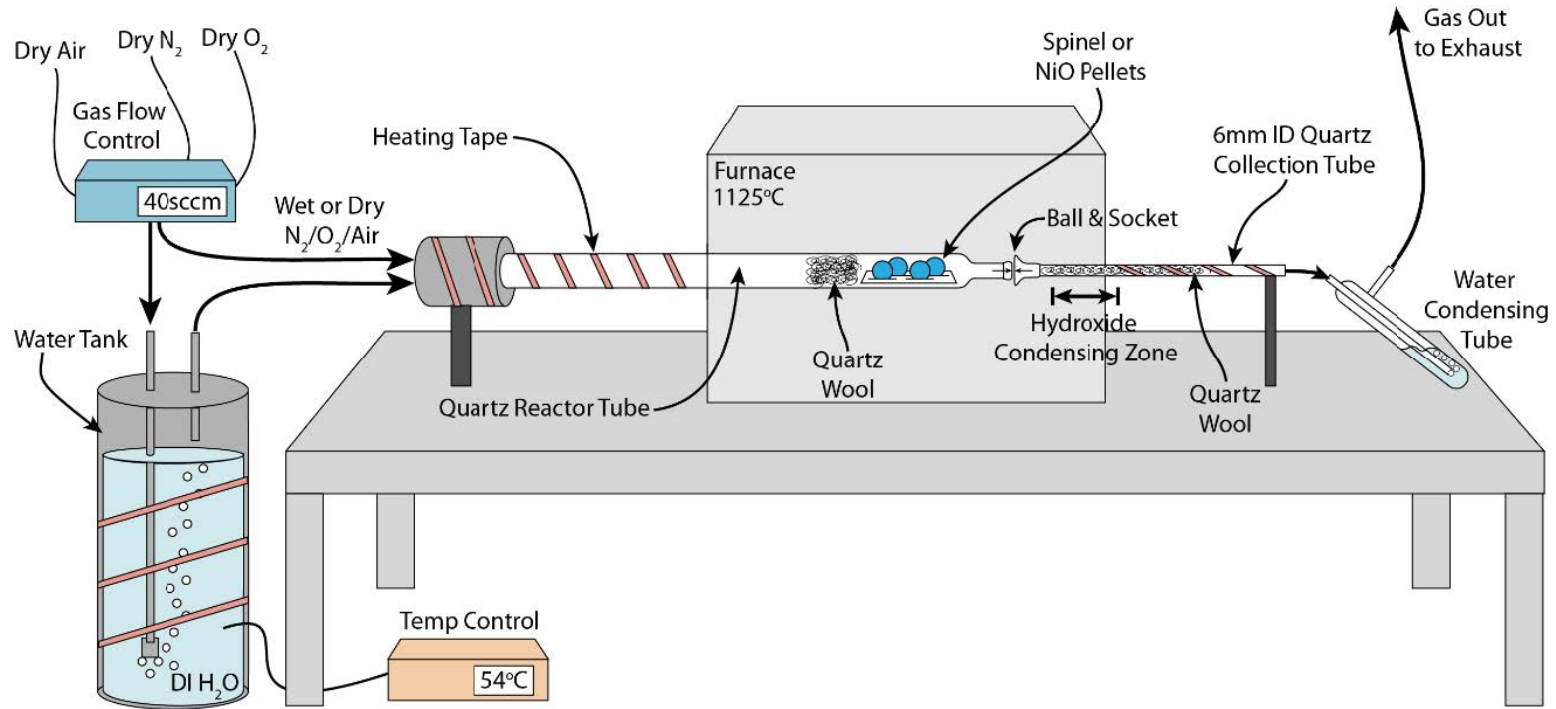
- Development of significant volatility in water vapor containing environments

P.J. Meschter, E.J. Opila and N.S. Jacobson,
Annual Review of Materials, 2013



Water Vapor Effects: Preferential Volatilization of Lubricious Phases?

Transpiration experiments will be used to verify volatilization of second phase constituents (hBN species) via atomic absorption spectroscopy:

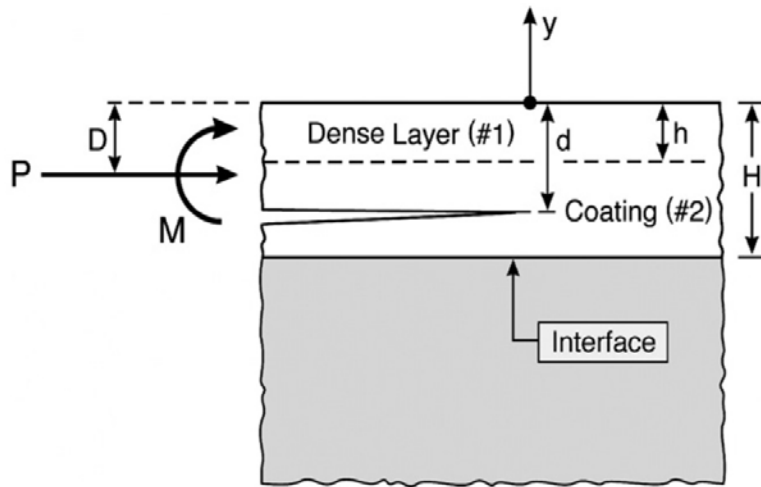


□ Nature of Sintering and Microstructural Evolution in Relation to Combustion Environment and Abradable Performance

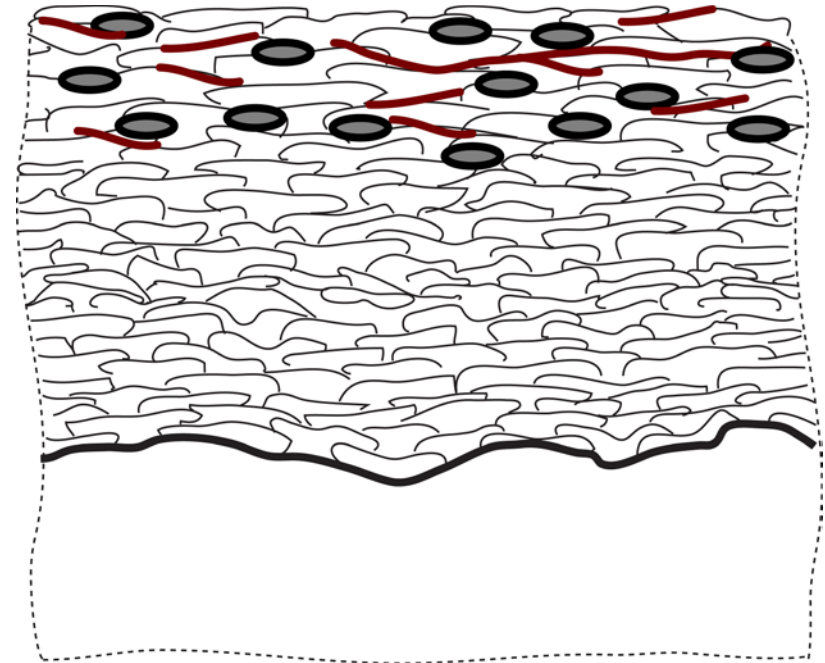
Characterize any enhancement of sintering rates associated with the syngas environments by making use of in-situ curvature measurements. The progression of microstructural change will be tracked and related to an underlying mechanistic driver. A key component of this effort is to evaluate spatial relationships between cracks, and the crack morphology, to evaluate what crack drivers existed prior to propagation.

- * Test coupons of coated systems will be subjected to simulated syngas and HHC environments, with elevated water vapor (8.5% and 30%) to assess sintering and microstructural evolution effects in the abradable seal systems.
- * The nature of the abrasive material removal will be characterized in relation to the exposure environment.



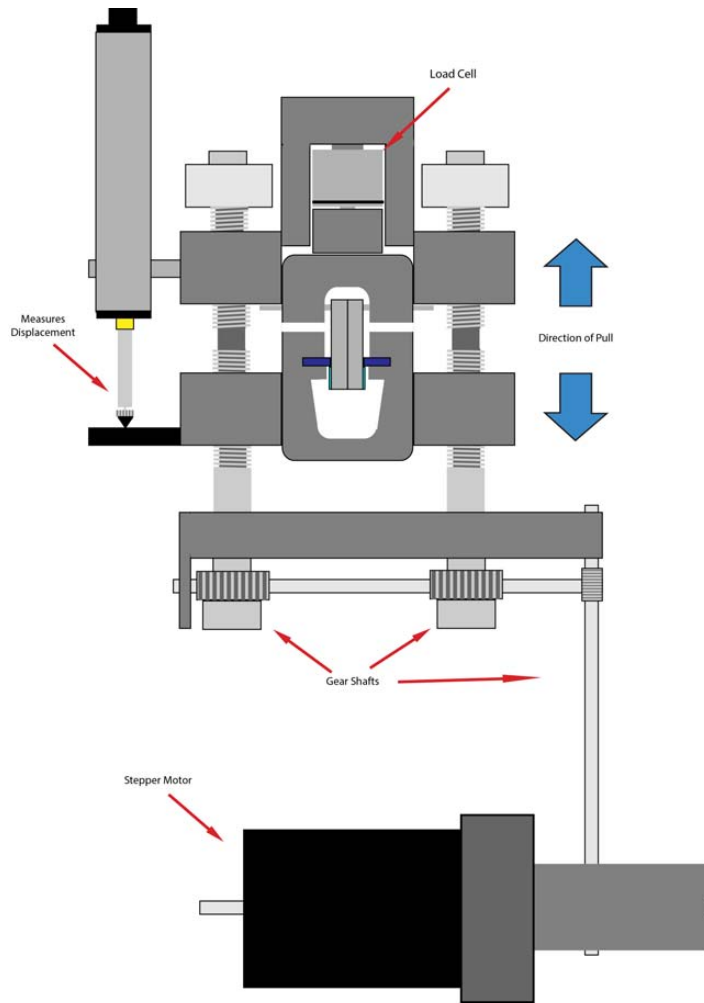


Sintering Effects

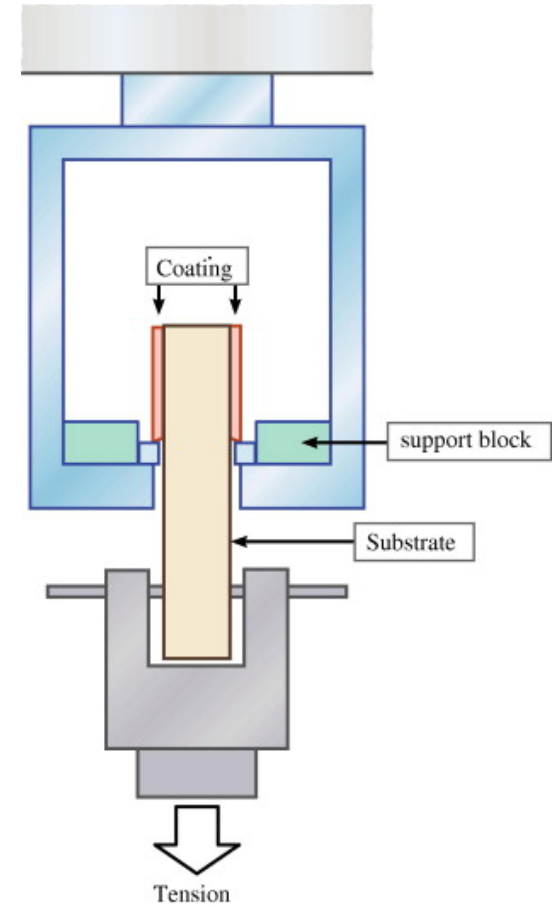


Pore Evolution,
Volatilization and
Interface Degradation

Mechanical Property Evaluations



Full Barb Pullout Test Rig With Loaded Sample and Gauges

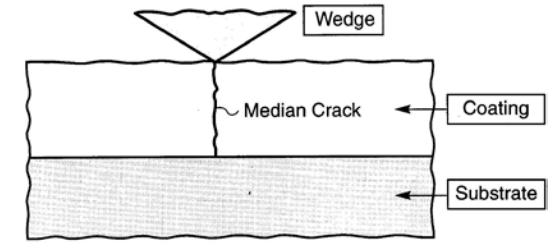
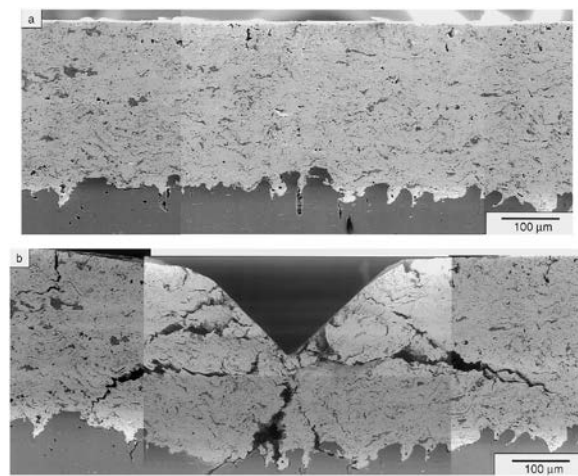
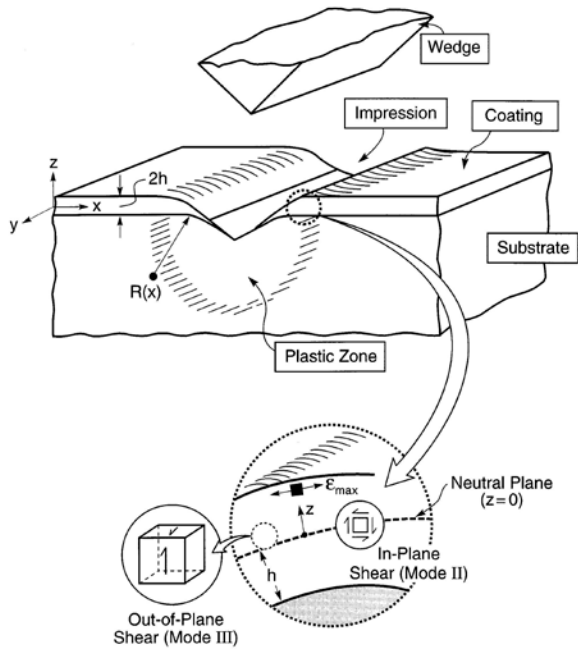


'Barb' Test

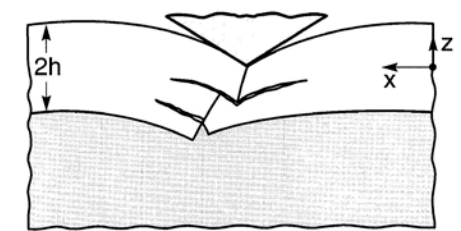


Mechanical Property Evaluations

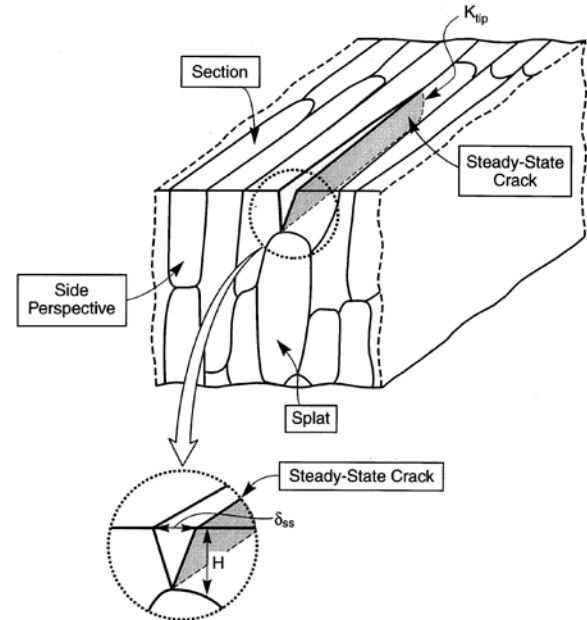
WEDGE IMPRESSION TEST



a) Median Crack



b) Bending And Lateral Cracking



Wedge Impression Test

A. Rabiei, et al, Materials Science and Engineering A, 369 (1999) 152



❑ Development of a Combustion-Based Abradability Test Rig

Leveraging existing burner rig capabilities, a module will be developed to carry out laboratory-scale assessments of the abradable behavior of shroud seal coatings under representative combustion environments. Design features will be vetted with the OEMs and coating vendors with experience in carrying out such tests, to ensure that representative wear behavior and high temperature seal material behavior can be assessed.

- * System design and component fabrication, to develop a rudimentary combustion-based high temperature tribological/abradability test facility that allows qualitative comparisons of erosion and fuel specific impurity deposition.
- * Baseline experiments with industry standard specimens (metal-based and ceramic based abradables) to confirm suitability of the test rig to carry out future experiments to evaluate performance of abradable seal systems as a function of the fuel-specific combustion environment.

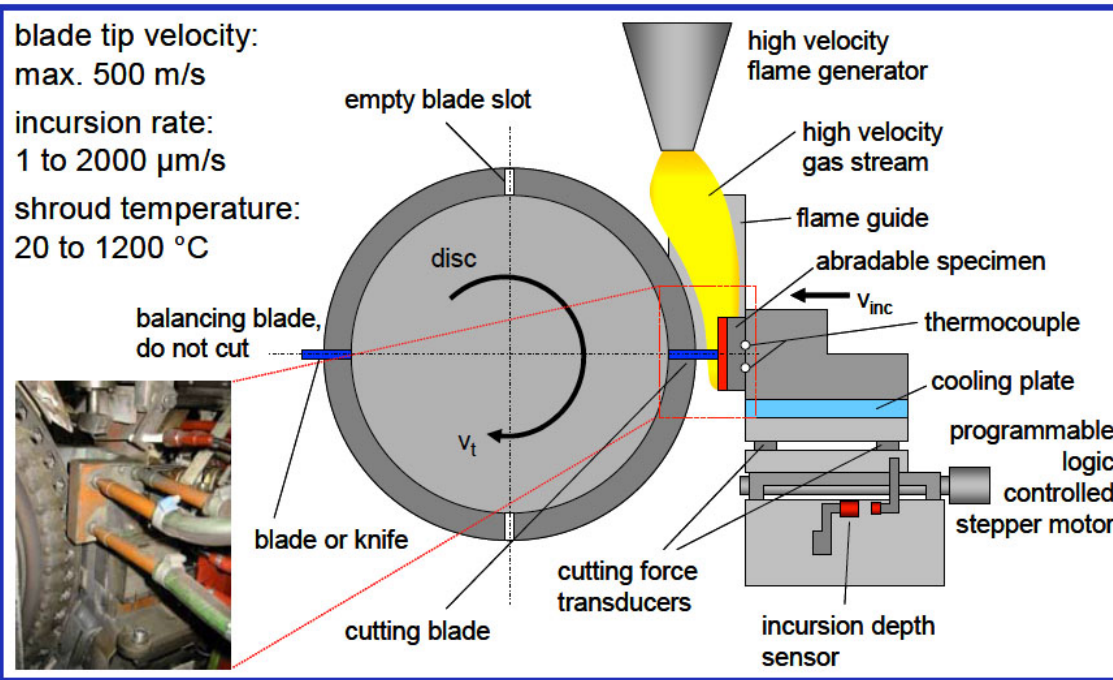


Abradability Test Rig

blade tip velocity:
max. 500 m/s

incursion rate:
1 to 2000 $\mu\text{m/s}$

shroud temperature:
20 to 1200 $^{\circ}\text{C}$

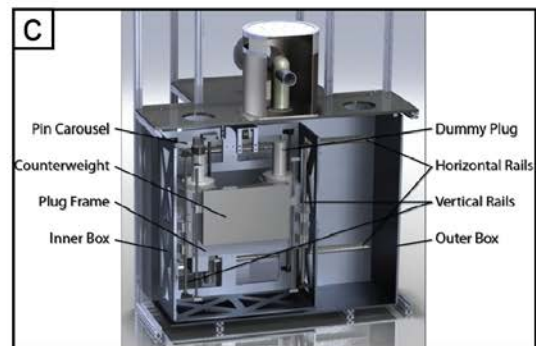
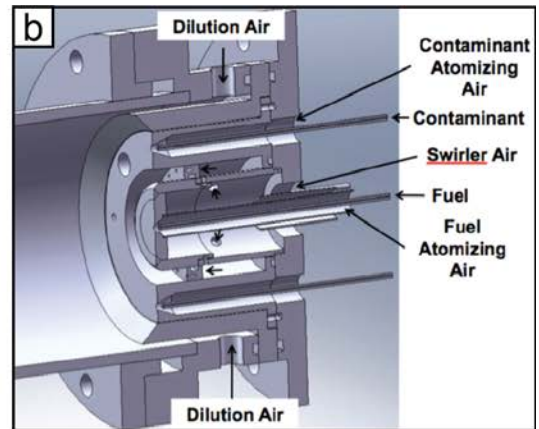
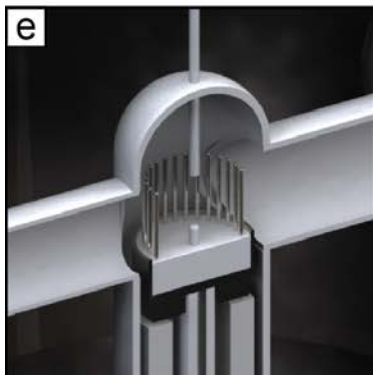
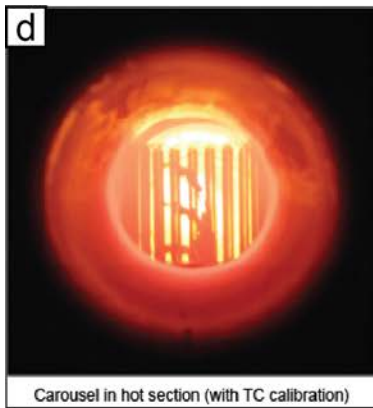


- Simulates the rubbing of blade tips against the coated casing as occurs during engine service.
- Stepper motor force the coated coupon into the moving rotor.
- Incursion rates can be accurately controlled.
- Abradability results are determined by measuring incursion depth of the blade into the coating, blade wear and abratable roughness.

Design features will be vetted with the OEMs and coating vendors with experience in carrying out such tests, to ensure that representative wear behavior and high temperature seal material behavior can be assessed

D. Sporer, S. Wilson and M. Dorfman, "Ceramics for Abradable Shroud Seal Applications." *Proceedings of the 33rd International Conference on Advanced Ceramics and Composites, volume 3, 2009,*





UC Irvine Low Velocity Burner Rig
(Currently optimized for Hot Corrosion Studies)



❑ Impurity Deposition and Corrosion, Infiltration and Phase Destabilization

For analysis of the thermo-mechanical response of the abradable coatings under simulated high temperature seal environments thermo-chemical reactions and phase development, we will utilize advanced electron imaging, focused ion beam sectioning routines, 3D serial sectioning and volume reconstruction, full spectrum EDS mapping, crystallographic texture mapping, and newly acquired x-ray tomography capabilities. These techniques will be applied to track material removal processes during abradable seal contact, as well as the development of damage zones.

- * Measurements and testing on metal-based seal materials will conclude, examining such materials after long-duration exposures to hardness and abradability testing. Microanalytical characterization will be concluded, quantifying the composition of oxides formed under each test condition, and correlating this with the thermo-mechanical testing results (hardness, spalling behavior, and abrasion resistance).
- * Consultations with OEMs to assess expectations on the role of fuel-specific particulates on seal integrity, and the appropriateness of planned laboratory procedures to capture relevant behavior, and accurately represent what may be expected for IGCC engine hardware.
- * Additional test coupons of coated standard coating systems will be subjected to Impurity deposition tests, using periodic spray deposition of suspensions of representative particulate matter, with assessments of the role of these gas phase constituents on the erosion and degradation of the seal surfaces.



▣ Studies of Alternative Composite Ceramic Seal Materials and Failure Mechanisms Under Thermal Gradient Conditions

The focus then shifts to identifying alternative materials (including new compositions or bi-layer concepts) with potential for use as high temperature seals with improved performance in emerging aggressive combustion environments, and their performance under more realistic operational thermal gradient based environments.

- * Develop feedstocks for fabricating seal materials from composites of zirconia and rare-earth phosphates known for attractive machinability properties. Fabricate test coupons for abrasability testing under IGCC-relevant testing conditions.
- * Studies paralleling earlier isothermal exposure tests (syngas fuel and high water vapor content) will be performed to assess the role of thermal gradients on the performance of alternative seal materials. A mechanisms-based assessment of the thermo-mechanical processes dictating the abrasability performance of alternative high temperature seal materials will be developed.



Summary and Key Developments and Conclusions

- Evaluated effects of elevated water vapor environments on TGO development for alumina-forming alloys. A strong correlation between $p\text{H}_2\text{O}$ and *transient* spinel formation is observed. Furthermore, there is a strong dependence of TGO and spinel growth kinetics on $p\text{H}_2\text{O}$, but *volatilization effects counteract* these effects and complicate analysis of mechanisms.
- YSZ aging appears accelerated in higher $p\text{H}_2\text{O}$ environments.
- CMAS degradation studies relevant to abradable coatings (hBN included) are underway.
- Mechanical and thermomechanical test approaches– for evaluation of abradable coating performance in relation to IGCC systems – are also under development
- **Use of IGCC combustion systems brings up additional issues in oxidation, corrosion volatilization and deposit-based degradation; the underlying mechanisms must be better understood in order to develop effective materials design strategies.**



Questions?

