LME –Environmental Effects & Coatings Branch

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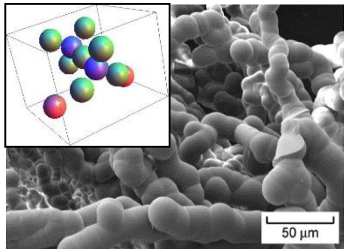
LME – Major Thrusts

High Temperature Behavior of Materials - Chemistry and Physics

- Oxidation, compatibility & diffusion, experimental & computational methods
- Identification (thermodynamics) and quantification (kinetics) of experiments for identification of degradation/failure modes

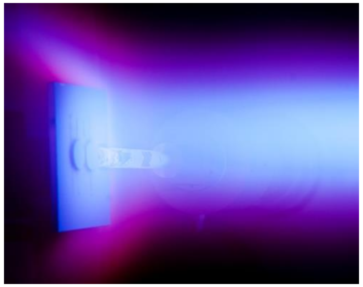


Advanced Coatings Development: Concepts and Processing

- TBCs, EBCs, multi-layer engineered coatings
- Develop coating compositions to mitigate environmental degradation
- Characterize and develop new coating processing methods

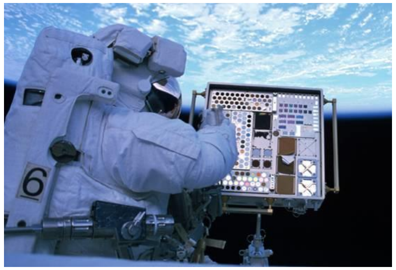
Durability testing in Extreme Environments

- Exposure to relevant conditions (thermal + mechanical + environmental)
- High temp, high heat flux, isothermal & cyclic, combustion, oxidation & corrosion, steam & water vapor, CMAS, erosion, impact



Space & Planetary Environments: Simulation & Analysis

- Flight experiments, durability testing, modeling, life prediction
- Atomic oxygen, vacuum ultraviolet radiation, lunar dust adhesion, extreme temperature electronics, Mars atmosphere



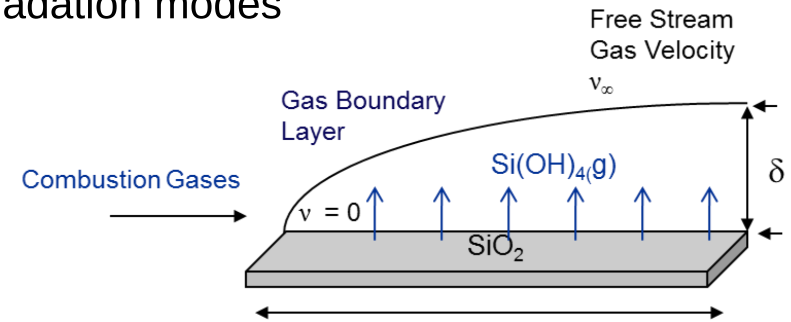
High Temperature Behavior of Materials

Experimental Thermodynamics & Kinetics Capabilities:

- Identify gaseous reaction products for unknown reactions
- Determine kinetic rate of candidate materials degradation modes

Knudsen Effusion Mass Spectrometer

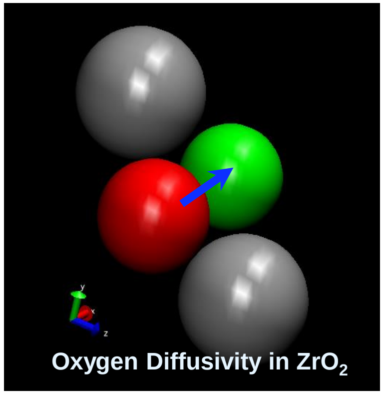
***Thermo-gravimetric Analysis** (air/water/vacuum)*



*GRC identified $Si(OH)_4$ product for reaction of SiC with moisture – **reaction is life limiting to SiC/SiC durability in turbine engines***

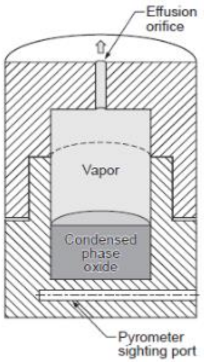
Computational Thermodynamics & Computational Models:

- Thermodynamics & kinetic approach
  - Identify degradation modes due to adverse reactions w/ adjoining materials and environment constituents
  - Code generated phase diagrams (FactSage / ThermoCalc / Dictra)
  - Modeling efforts complimented with in-house experimental capabilities
- Atomistic, nanoscale, and continuum DFT materials modeling
  - Molecular dynamics, Metropolis/Kinetic Monte Carlo, and particle statics/dynamics

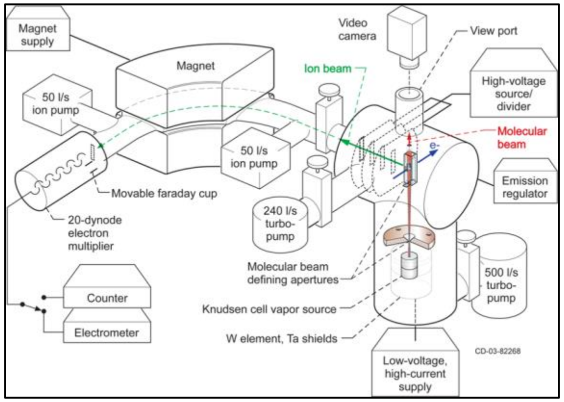


LME Mass Spectrometer Lab

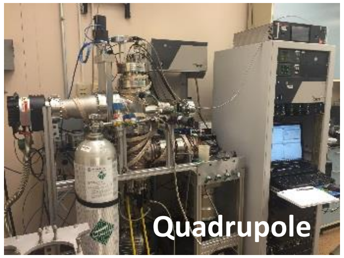
(3) unique instruments to identify gas and vapors at high temperatures. One-of-a-kind facility in US, only 2-3 worldwide.



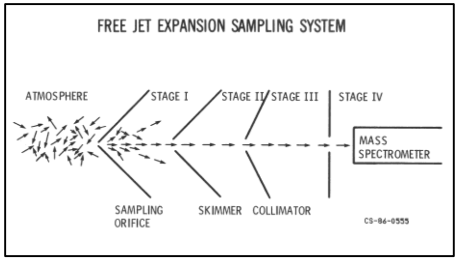
- **Vacuum studies based on Knudsen cell**
  - Typical 1cm dia x 1cm high, 1 mm orifice, establish equilibrium, vapor effuses
  - Wt loss rates relates to pressure



- **Knudsen Cell Mass Spectrometers**
  - Magnetic Sector KEMS
    - Magnet sorts ions by mass-to-charge ratio and ion intensity α vapor pressure
    - High stability / resolution
  - Fast Scanning Quadrupole KEMS
    - Electric field sorts the ions
  - Thermodynamic information provided:
    - Heats of Vaporization & composition of vapor phases
    - Activity measurements & phase diagram boundaries



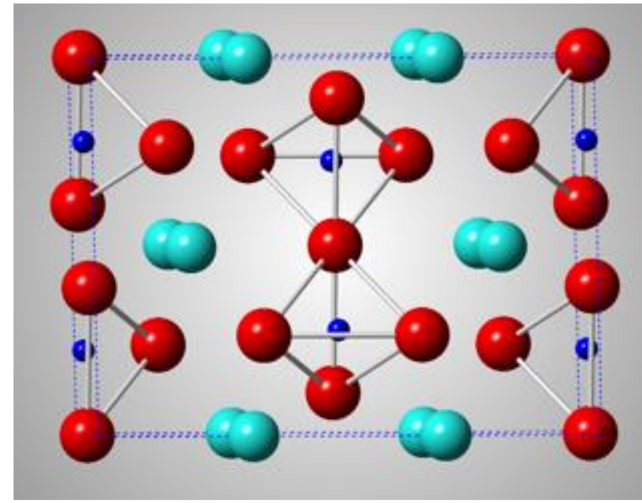
- **High Pressure Mass Spectrometer**
  - Free Jet Expansion
    - Allows (10^{-6} atm) sampling at 1 atm
    - Series of differential chambers
    - Eliminates cold surface condensate
    - Chemical & dynamic integrity of gases
    - More qualitative (approx. amts)



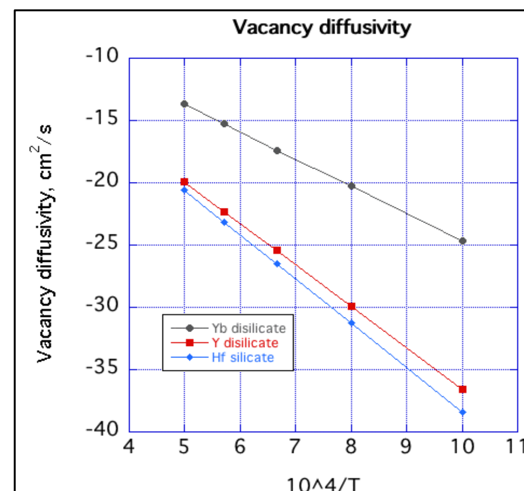
Computational Modeling

Overall Approach:

- Kinetic Monte Carlo (kMC) computer simulations of oxygen/H2O diffusion in candidate materials such as Yb2Si2O7, Y2Si2O7, and HfSiO4.
- Processes are assumed to be thermally activated.
- Consider vacancy and interstitial diffusion mechanisms.
- Migration barrier energies are computed using Density Functional Theory (DFT).
- Barrier energies are used to produce diffusivities using a kMC code developed in our laboratory.

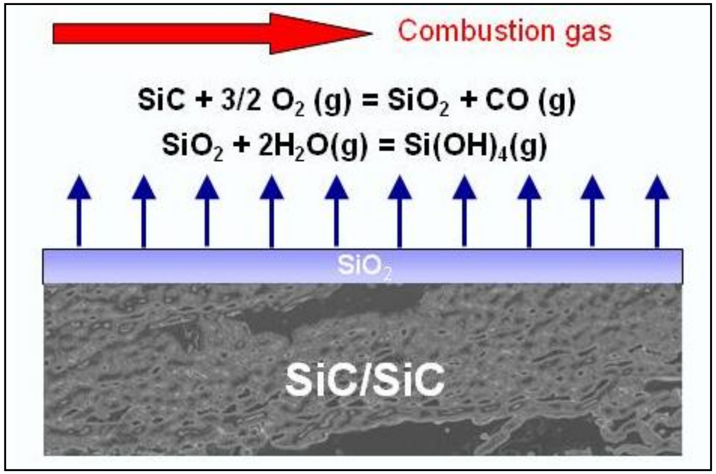


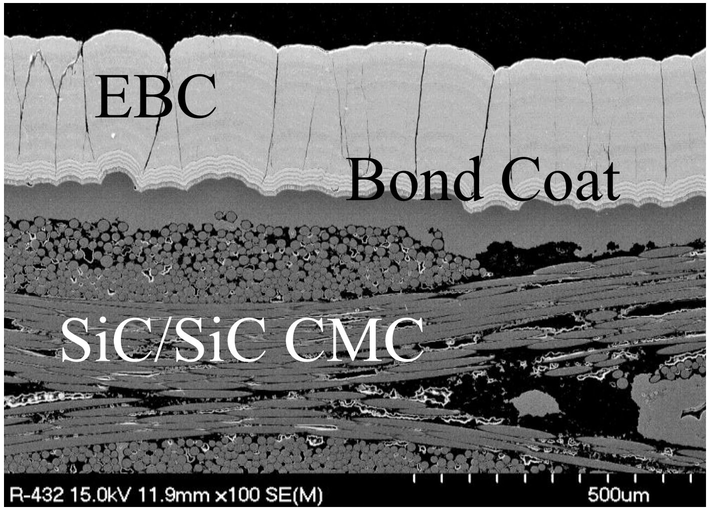
$Yb_2Si_2O_7$ Structure



Advanced Coatings: Concepts

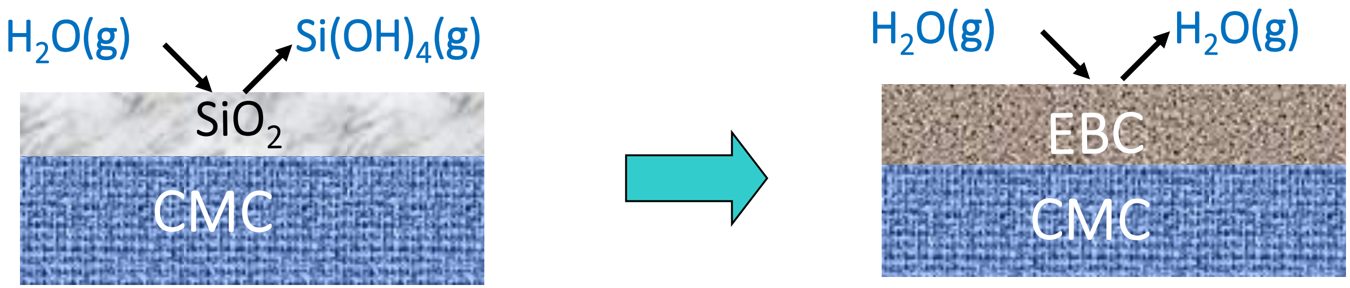
- Incorporation of Si-based ceramics into turbine hot section has substantial benefits
  - High temperature, low density
  - 1990's: Observation that SiC undergoes rapid recession in water vapor
- Environmental barrier coatings (EBCs) are necessary to protect the underlying ceramic
  - Chemical compatibility between layers
  - CTE match @ EBC/bond coat/substrate
  - Thermal stability w/ limited volumetric change
  - Limited O2/H2O ingress & weight loss, CMAS resistant, erosion toughness
  - Total thickness of 5-10 mil (125-250 micron)
  - 2000s: Development of coatings to minimize water vapor effects at 2400°F
- Current NASA goals require durable coating systems at 1482C (2700F) w/ reduced cooling
  - Limited recession and good adhesion
- Traditional processing methods may not be able to meet the requirements
  - Plasma Spray-Physical Vapor Deposition (PS-PVD)
  - Slurry casting



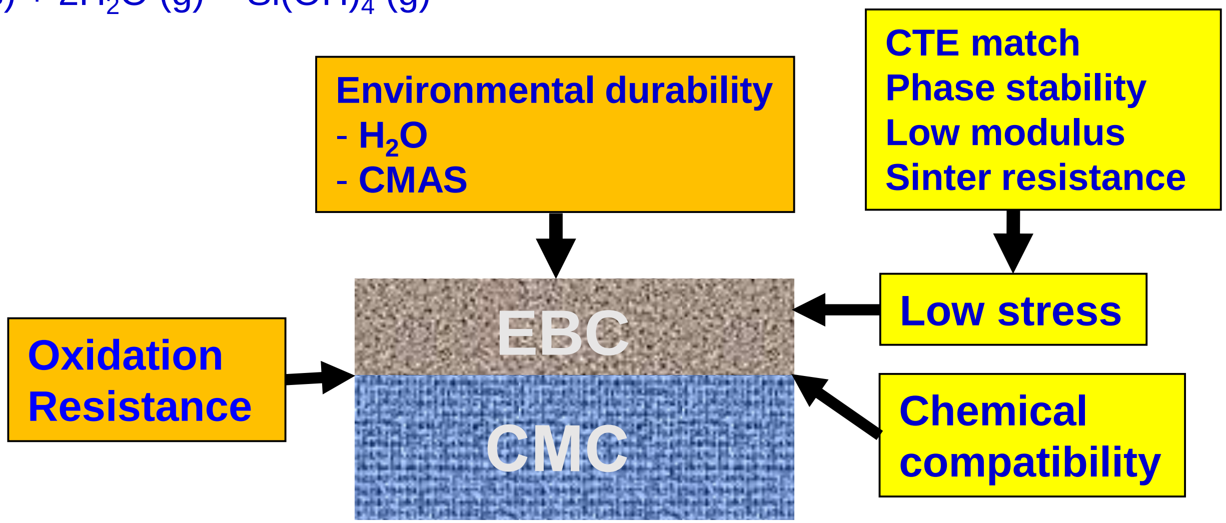


Environmental Barrier Coating (EBC)

An external coating to protect CMC from water vapor



SiO_2 (s) + $2H_2O$ (g) = $Si(OH)_4$ (g)



EBC is essential for CMC operation. Uncoated CMC suffers rapid recession.

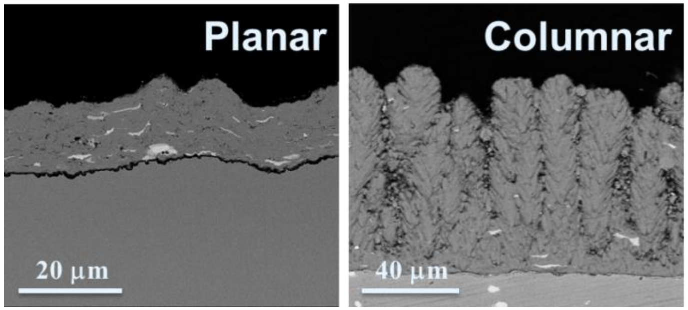
Advanced Coatings: Processing

Develop in-house new techniques and partner with outside contractors in parallel paths:

- Rich history of Thermal and Environmental Barrier Coatings
- In-house facilities include:
  - Ambient / High Temperature Plasma Spray
  - Plasma Spray-Physical Vapor Deposition (PS-PVD)
  - Slurry Coating Deposition (new)
- Partner externally for developing EB-PVD, CVD, DVD

Plasma Spray-Physical Vapor Deposition:

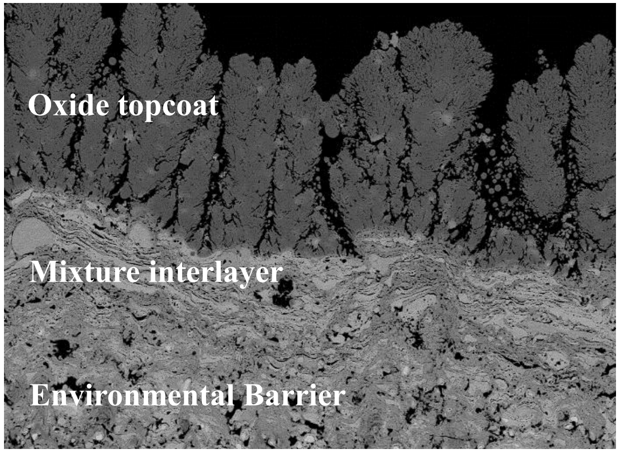
- One of 5 systems worldwide, online in 2010
- Relatively high deposition rate over other methods
- Non line of sight deposition
- Wide range of applications



Same material, different processing parameters

Multiple materials

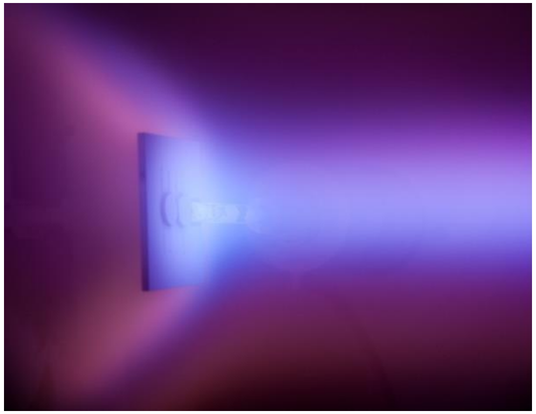
Different processing parameters

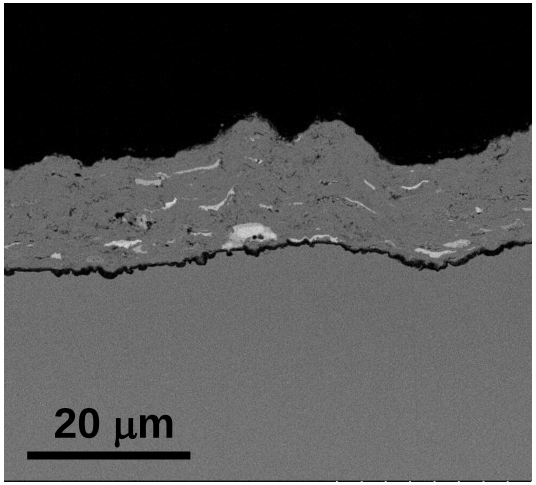


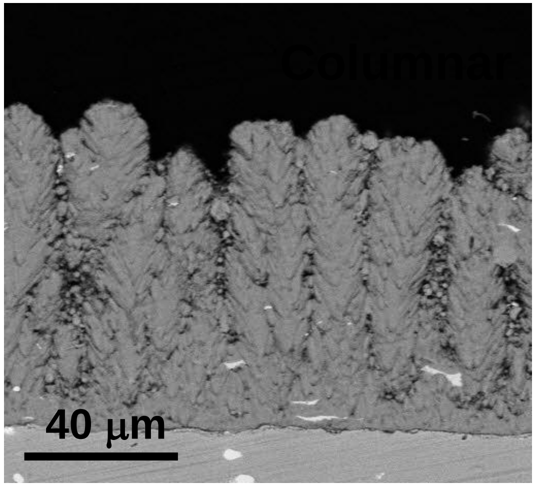
Plasma Spray-Physical Vapor Deposition (PS-PVD)

- Bridges the gap between plasma spray and vapor phase methods
  - Variable microstructure
  - Multilayer coatings with a single deposition
- Low pressure (70-1400 Pa)

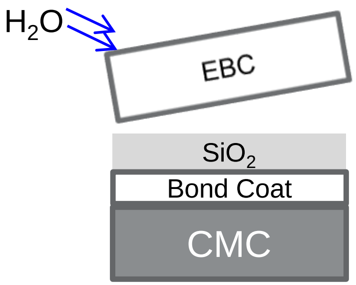
  High power (>100 kW)
  - Temperatures 6,000-10,000K
- High throughput[1]
  - 0.5 m^2 area, 10 mm layer in < 60s
- Material incorporated into gas stream
  - Non line-of-sight deposition
- Attractive for a range of applications
  - Solid oxide fuel cells, gas sensors, etc.



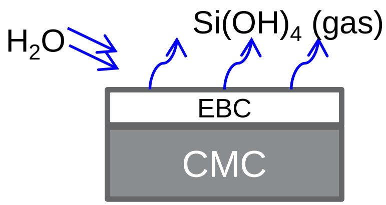




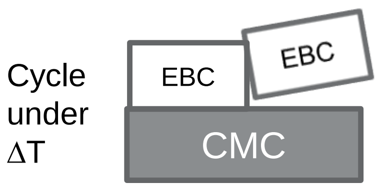
EBC Failure Modes



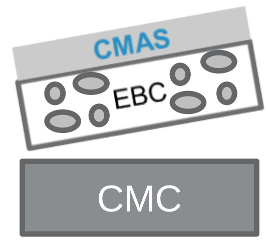
- Steam oxidation



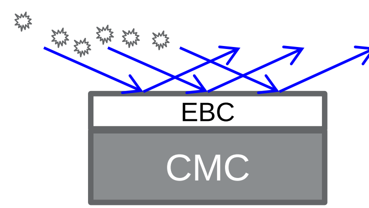
- Recession by water vapor



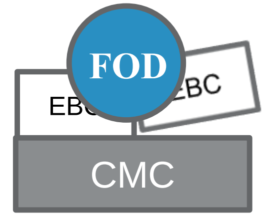
- Thermal fatigue
- Thermo-mechanical fatigue



- Sand/Volcanic Ash Ingestion
- CMAS: Calcium-Magnesium-Alumino-Silicate



- Erosion



- Foreign Object Damage

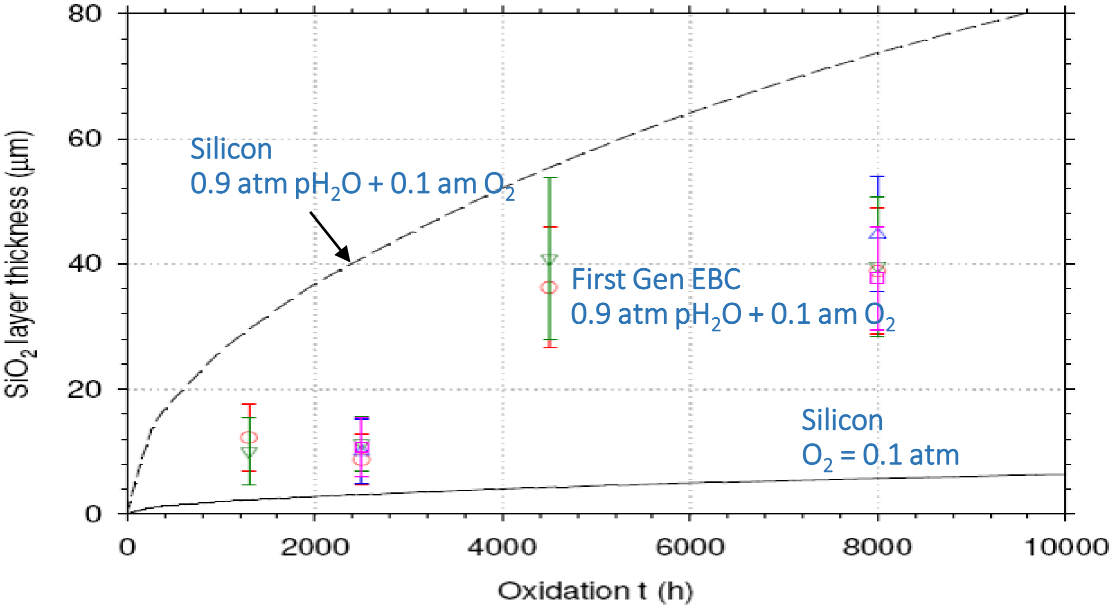
Synergies between failure modes lead to the ultimate EBC failure

K. N. Lee, "Environmental Barrier Coatings for CMC's"; in *Ceramic Matrix Composites*, Wiley, New York (2015)

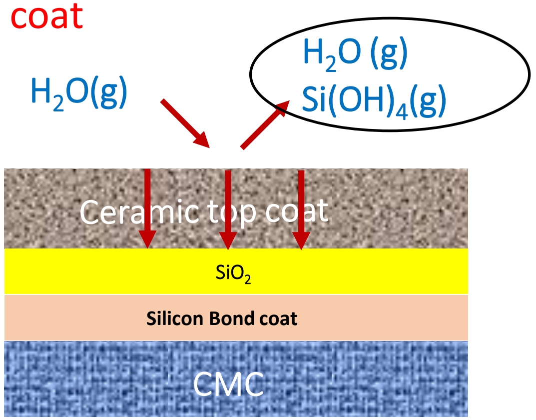
EBC Steam Oxidation

- Silicon oxidizes faster in $H_2O(g)$ than in air by an order of magnitude
- Attributed to high solubility of $H_2O(g)$ in SiO_2
- Ceramic top coat does not stop the transport of $H_2O(g)$ to Si bond coat

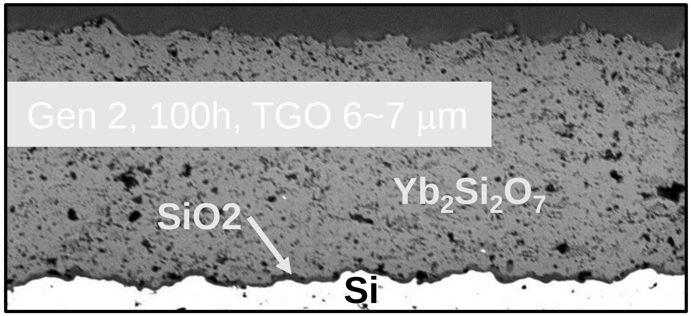
Isothermal Oxidation, T = 2200°F (1204°C)



GE Final Report – AMAIGT Program Dec. 2010



Cyclic Oxidation, 2400°F, 90% H_2O



NASA, Unpublished data

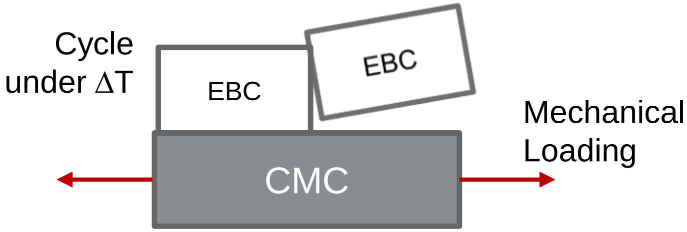
Oxidation of EBC/CMC system must be evaluated in H_2O environments

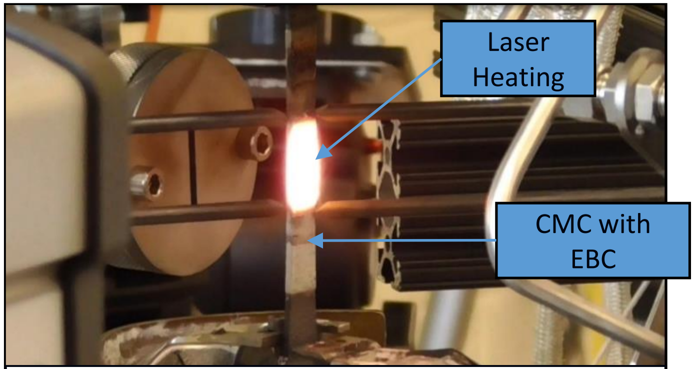
Thermomechanical Testing of CMC/EBC

- First integration and testing of NASA developed CMC with the NASA developed EBC system

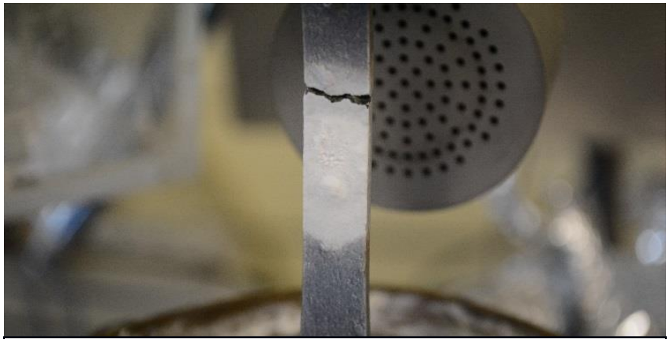
- Sustained peak low cycle fatigue (SPLCF) test with laser gradient heating for thermomechanical validation

- Milestones have been reached for desired temperature and loading conditions.





EBC coated CMC under stress heated by a high heat flux laser



After testing

NASA EBC Development Test Rigs

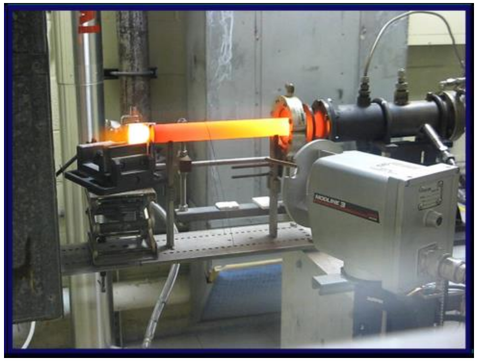
Rig	Capability	Failure modes to be tested
Mass Spectrometer	$P(H_2O)$ = N/A v = N/A P_{total} = N/A	Recession (High pressure measurement of reaction products and Low pressure measurement of activities)
Steam TGA	$P(H_2O)$ = up to ~0.5 atm v = a few cm/s P_{total} = 1 atm	Recession (Initial screening of candidate materials)
Mach 0.3 Burner rig	$P(H_2O)$ = ~0.1 atm v = 230 m/s P_{total} = 1 atm	CMAS, Erosion, FOD
Steam cycling rig	$P(H_2O)$ = up to ~1 atm v = a few cm/s P_{total} = 1 atm	Steam oxidation
High heat flux laser rig	$P(H_2O)$ = ambient air v = zero P_{total} = 1 atm	Thermal fatigue in temp gradient Thermo-mechanical fatigue in temp gradient
Natural gas burner rig	$P(H_2O)$ ~ 0.5 atm, v ~ 250m/s P_{total} = 1 atm	Recession Thermal fatigue in temp gradient (Coupons, Tensile bars, components)
CE-5 combustion rig	$P(H_2O)$ ~ 3 atm v ~ >30 m/s P_{total} ~ 30 atm	Steam oxidation w/ temperature gradient Recession (Coupons, Tensile bars, components)

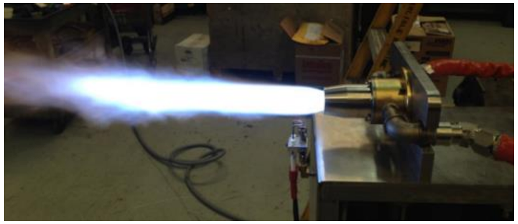
- **Combinations of rigs to investigate synergies between failure modes**
- **The only test vehicle that has all key variables is an engine**

Environmental Durability Testing

Materials evaluated in relevant conditions with a wide range of facilities:

- **High Heat Flux Laser Rigs**
  - (4) rigs capable of up to 315 W/cm^2
  - Thermal-mechanical capability
  - Isothermal, thermal gradient, steam
  - In Situ Thermal Conductivity
- **Mach 0.3 Burner Rigs**
  - Jet fuel / air combustors (Mach 0.3 - 0.7)
  - Tgas over 3000°F / Tsrf up to 2700°F
  - Automated, thermal cycling, impact, loading
- **Dedicated Erosion Burner Rigs**
  - Alumina erodent particulates (1-600 micron)
  - Adapted for CMAS compositions
  - Continuous/uniform feeding (.08-60 gm/hr)
- **Steam Cyclic Oxidation Testing**
  - 90% water vapor (9 atm total pressure)
  - Temperatures up to 2700°F (1482 C)
- **Natural Gas / O2 Burner Rig**
  - Natural gas / O2 combustion
  - 4200 F, 250 m/s, up to 58% H_2O, 160-215 W/m2
  - Versatile: water recession, full coverage high heat flux, complex geometries, film cooling, combine with erosion / CMAS

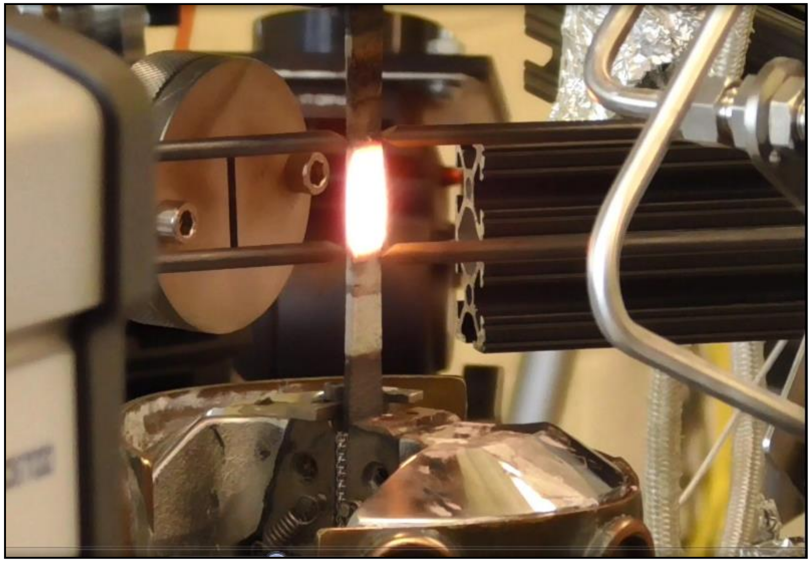




High Heat Flux Laser Rigs

<u>Typical Laser Test Rig:</u>

- Laser Heating (4000 W) on Front
- Backside Air Cooling
- Surface Temperature Measured with Pyrometers and/or IR Camera
- Surface Temperatures up to 3000 °F (Material Dependent)
- Thermal Fatigue and Combined Thermal Gradient and Axial Fatigue
- Uncoated / EBC Coated SiC/SiC CMCs

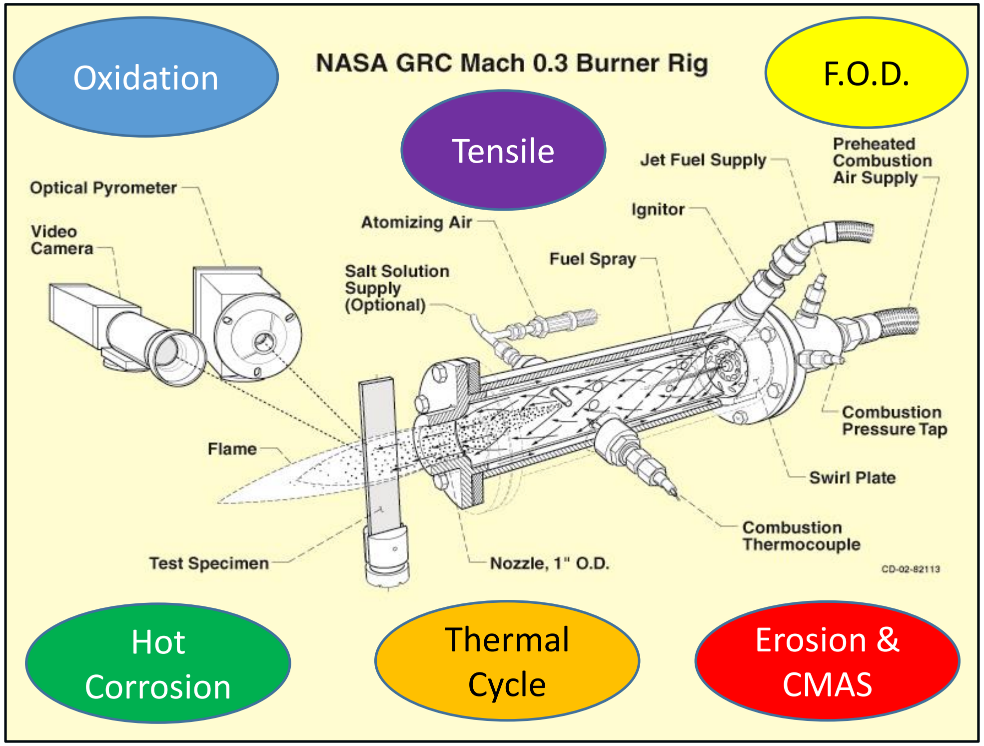


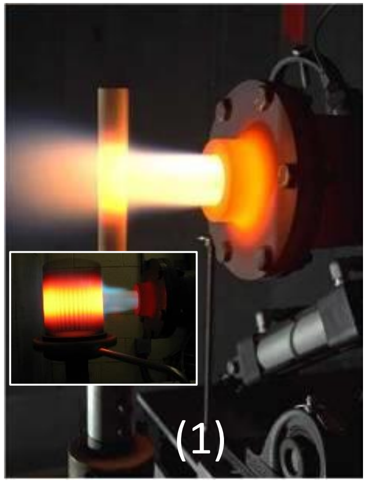
<u>Testing Features:</u>

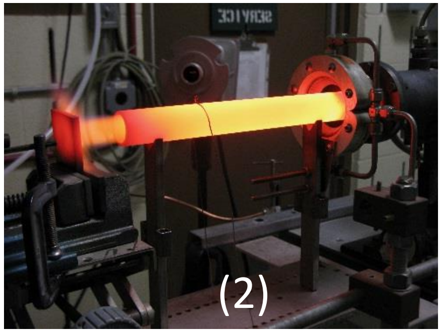
- Servo-hydraulic , 25 kN Load Cell
- Water-cooled Wedge Grips
- Two 1 in. Gage Length, Water-Cooled
- Extensometers; 6 in. Long Tensile Specimens
- Frequencies up to 30 Hz
- Load and Stroke Control
- Strain-Control capability in progress
- Tensile, flexural, HCF, LCF, SPLCF
- In situ thermal conductivity measurement

Mach 0.3 Burner Rig Facility

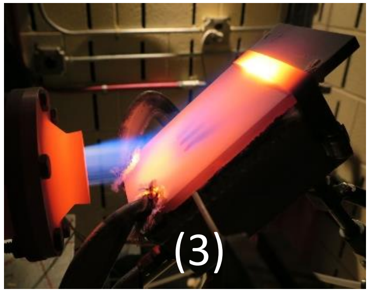
- 8 computer-controlled jet-fueled combustors in individual test cells Building 34
- Extremely efficient means of testing the durability of new jet engine materials
- Material test temperatures from 600° to 2700°F, flame temperatures to 3000ºF
- Creates the extremely hostile operating environment found in turbine engines
- Multiple or single samples tested using rotating carousels to compare materials
- Thermal cycling duplicates actual flight cycles: takeoffs, cruise, and landings

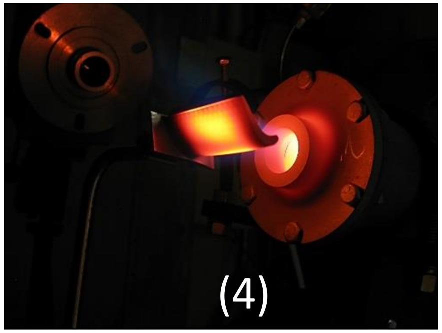






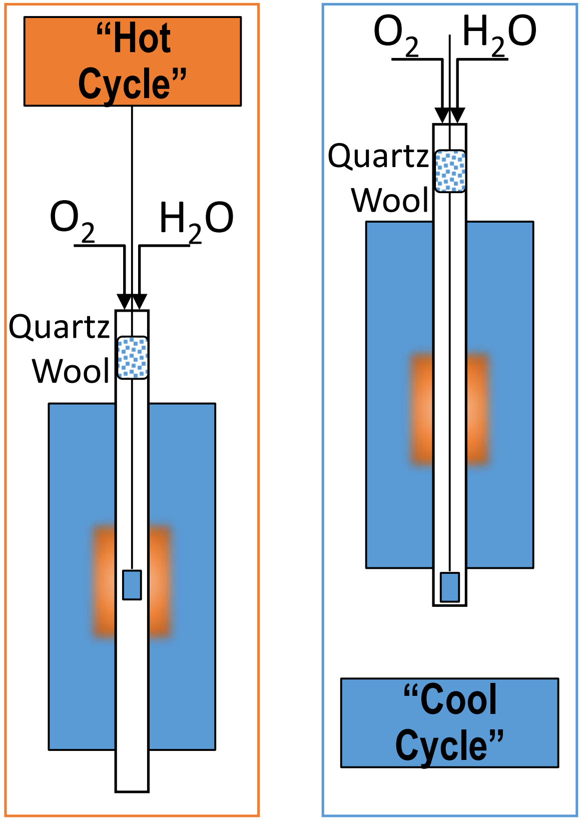
(1) TBC/Super-alloy, (2) Erosion, (3) Film-cooled monolithic ceramic, (4) Metal Turbine Blade

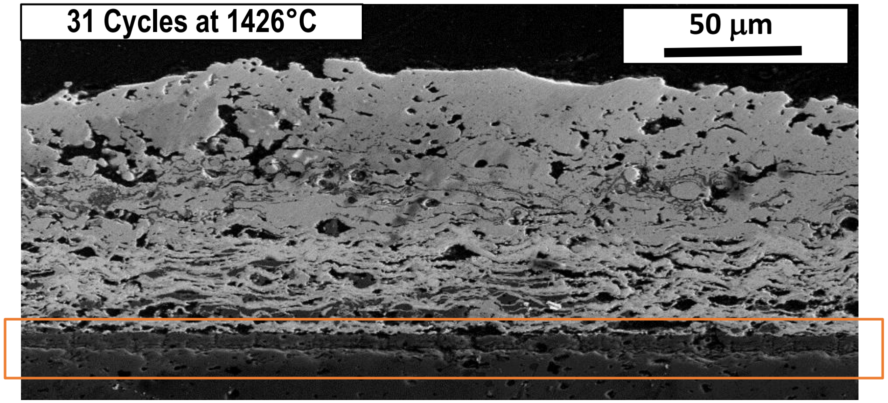




Cyclic Steam Oxidation Testing

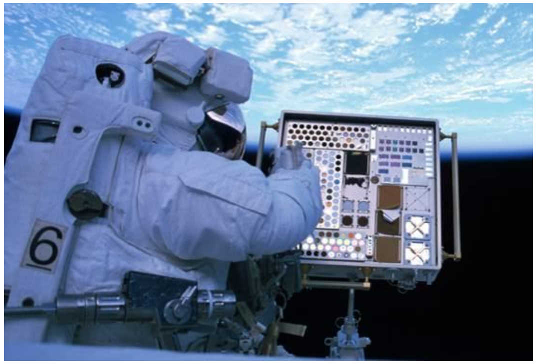
- Steam oxidation required to determine durability of EBC
  - Limitation of formation and growth of SiO_2 layer critical to lifetime
  - Oxidation of Si-based ceramics (including Si) is an order of magnitude or more in steam
- Steam oxidation performed at NASA
  - "Hot cycle" temperature 1426°C
  - 0.9 atm H_2O bal. O_2
  - 2.2 cm/sec flow rate
  - 1 hour hot followed by 20 minute cool



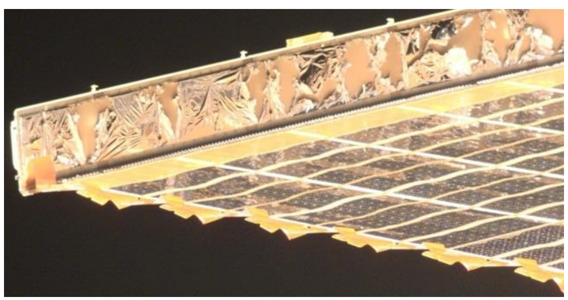


- Scales formed in cyclic steam oxidation are often much thicker and more porous
- TGO scales at coating interface lead to spallation failure

Space Environments



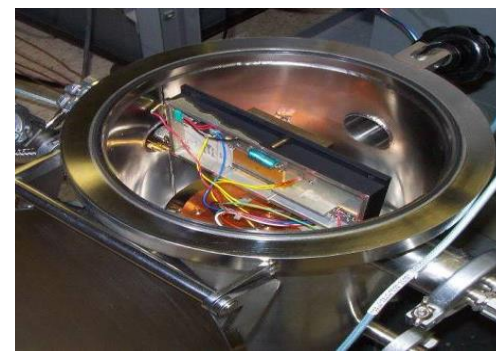
Space and Planetary Environment Durability and Performance Prediction



Environmental Degradation Abatement Technology Development

- **Environment Effects Knowledge**
  - Flight Experiments (EOIM-III, LDEF, Mir, MISSE 1-7, etc.)
  - Environment Simulation (Low Earth Orbit, Lunar, Mars …)
    - Atomic Oxygen, UltraViolet Radiation, Dust Adhesion
    - Atmosphere & surface interaction
  - Modeling (Erosion of Polymers, Sunspot Prediction)
- **Analysis and Life Prediction**
- **Abatement, High Performance Surfaces, and Component Development**
  - AO & UV resistant coatings
  - Orion docking seals / windows
  - High Emittance Radiator Coatings
  - Radiation Durable Solar Cells
  - Extreme Temperature Electronics
- **Technology Transfer of Developed Technologies** (for Aerospace, Medical, Industrial and Art Applications)

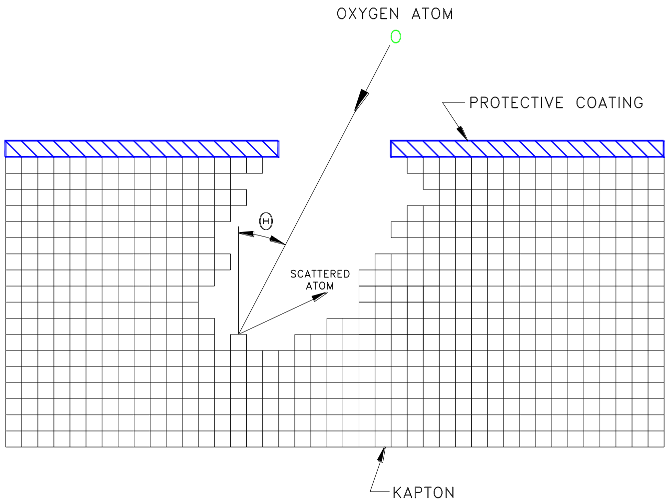
Space Environment Simulation

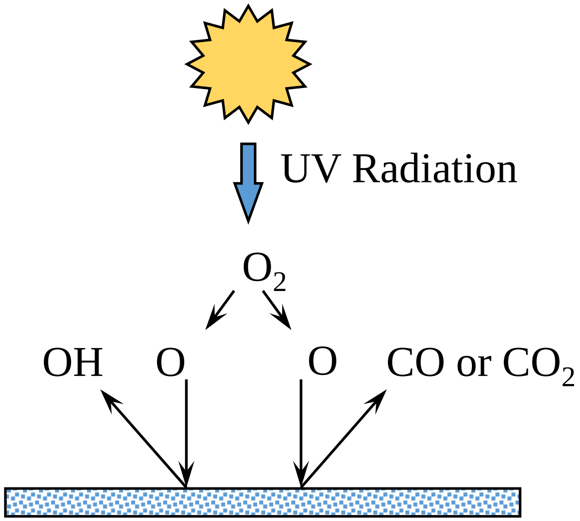


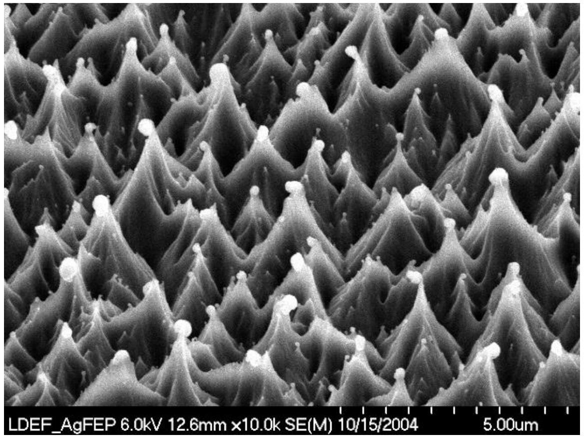
Environmentally Durable High Performance Surfaces and Component Development

Atomic Oxygen Modeling

- Monte Carlo Analysis: 2-D Computational modeling of atomic oxygen erosion of polymers based on observed in-space results
- Takes into account:
  - Energy dependence of reaction probability
  - Angle of impact dependence on reaction probability
  - Thermalization of scattered oxygen atoms
  - Partial recombination at surfaces
  - Atomic oxygen scattering distribution functions
- Modeling parameters tuned to replicate in-space erosion







LDEF Teflon FEP

AO F= 7.78×10^{21} atoms/cm^2

Atomic Oxygen Art Restoration

Fire damaged

Atomic oxygen restoration