Flexural Fatigue Behavior of an EBC/CMC Composite System In Air and Steam at High Temperature

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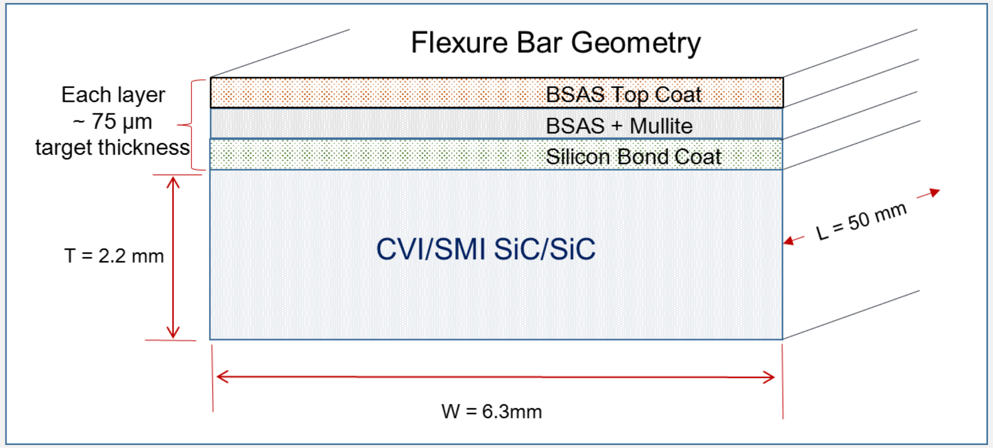
Evaluate Flexural Fatigue Behavior of EBC/CMC System in Steam or Air

Overall Goal:

- Investigate failure modes in an EBC/CMC system tested under fatigue conditions in steam and air.
- Focus on interaction of coating and substrate during failure event.



Approach:

➢ Coating Application –Barium Strontium Aluminosilicate (BSAS) coatings applied in-house by an air plasma spray method (APS).

➢ Mechanical Testing
  – Fast fracture flexure testing of coated and uncoated samples at room and elevated temperature to determine the proportional limits for the system.
  - Incremental flexure loading of the samples was performed to track the development and propagation of cracks up to the proportional limit at both room and elevated temperature
  - Sustained peak low cycle fatique (SPLCF) tests were performed in air and steam for both coated and uncoated samples

➢ Characterization
  - Computed tomography (CT) scans were performed before and after testing of coated samples.
  - Electron and optical microscopy were used to identify crack propagation in the system.

EBC/CMC System

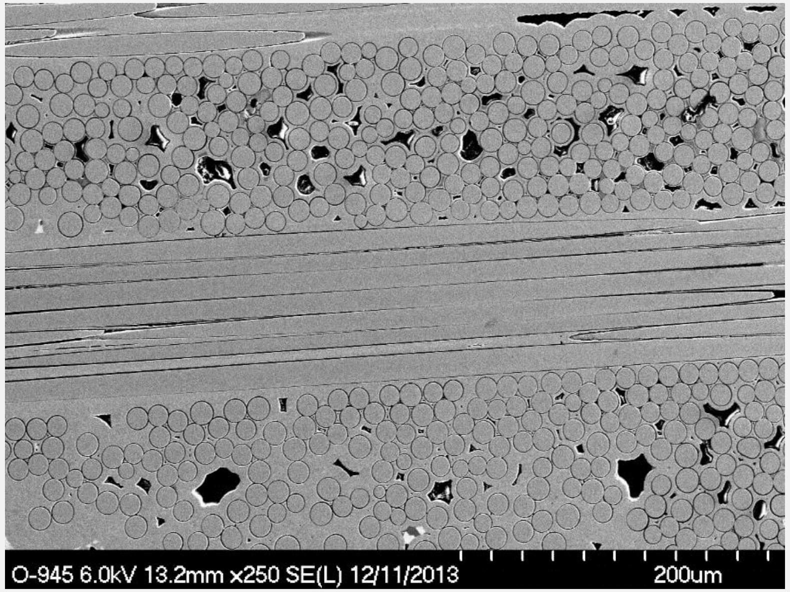
CMC

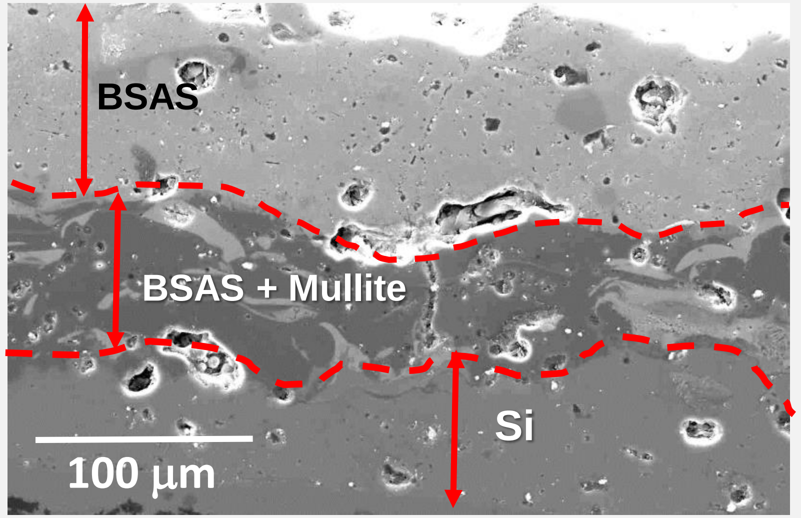
- Hi Nicalon™ Type-S SiC fibers
- 2D cloth lay up with 5HS weave
- CVI / SMI matrix
- 0.5 micron BN coating on fibers
- All samples taken from the same panel:

Fiber Vol	CVI SiC Vol	SMI SiC Vol	Res. Porosity
36.1%	32.6%	23.70%	7.6%

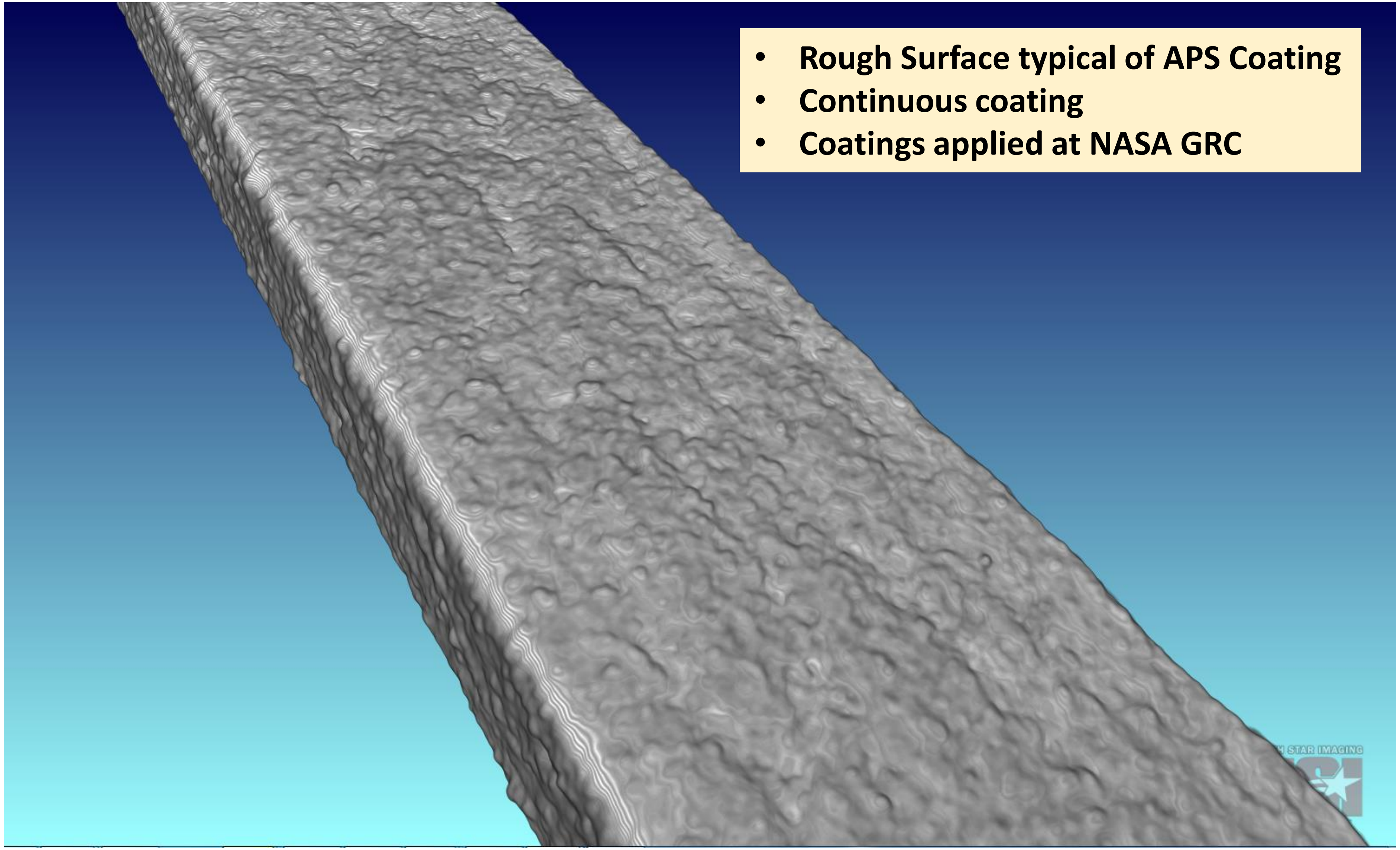
EBC

- BSAS coating applied at NASA Glenn
- Multilayer air plasma spray coating
- CMCs lightly grit blasted with alumina before coating to improve bonding
- CMC heated above 1000°C during coating
- Target 75 μm thickness layers
- Hexacelsian topcoat transformed to more stable Celsian phase with heat treatment at 1300°C / 20hrs / Air
- Large pores remain in all EBC layers



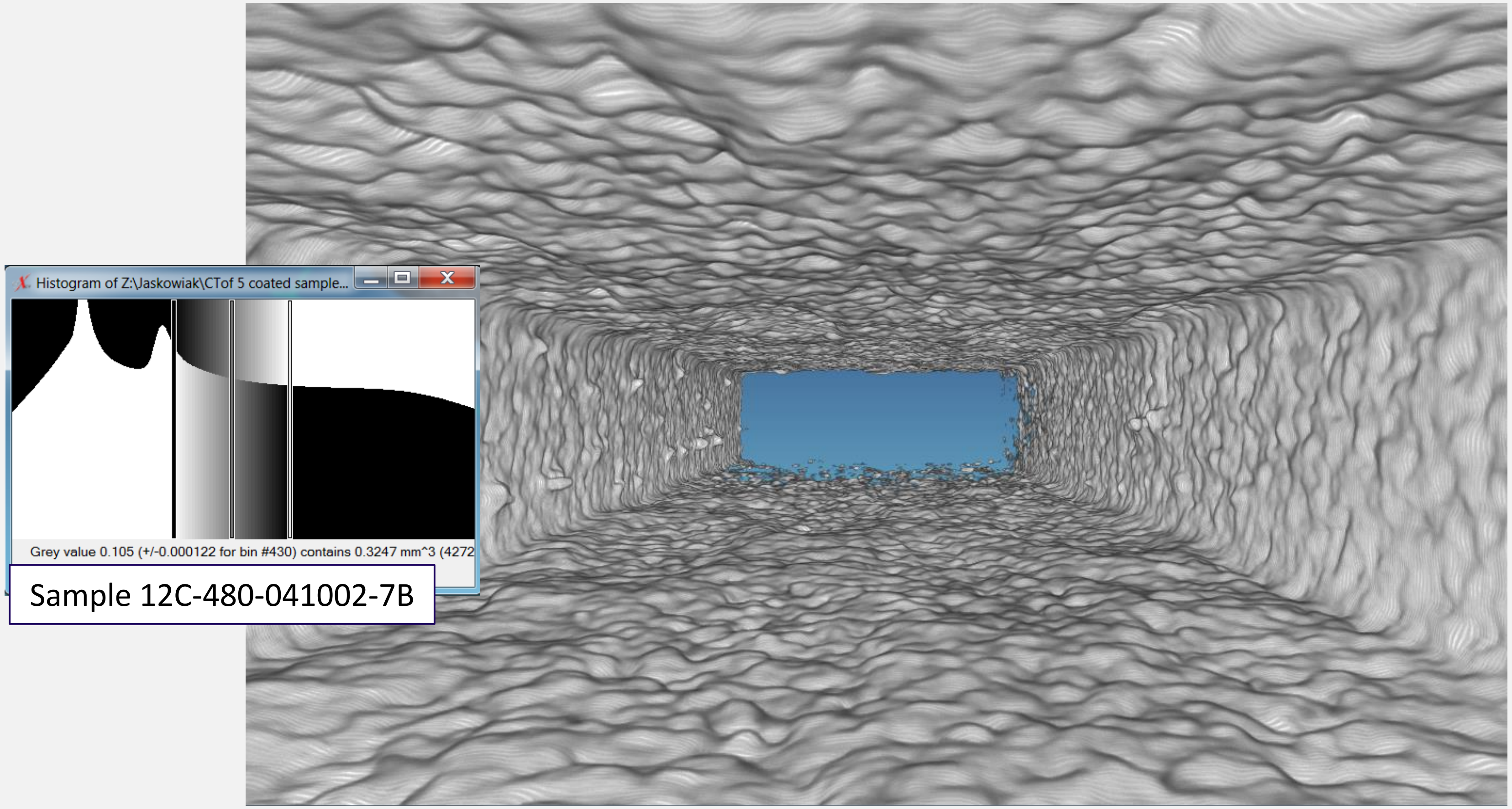


CT Scan of As-deposited BSAS Coating on CVI/SMI SiC/SiC



- Rough Surface typical of APS Coating
- Continuous coating
- Coatings applied at NASA GRC

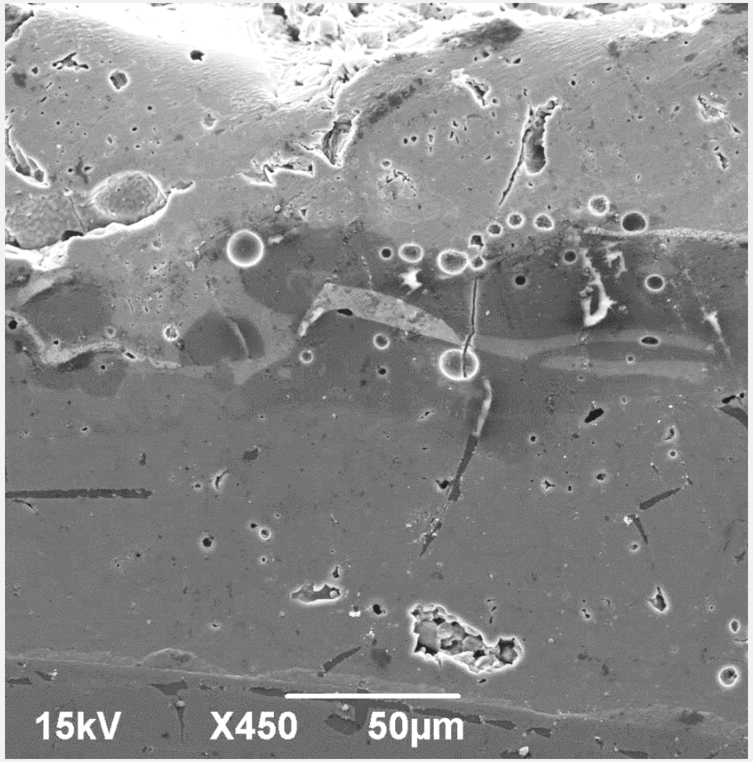
CT Scan of Pre-test BSAS Coating on CVI/SMI SiC/SiC

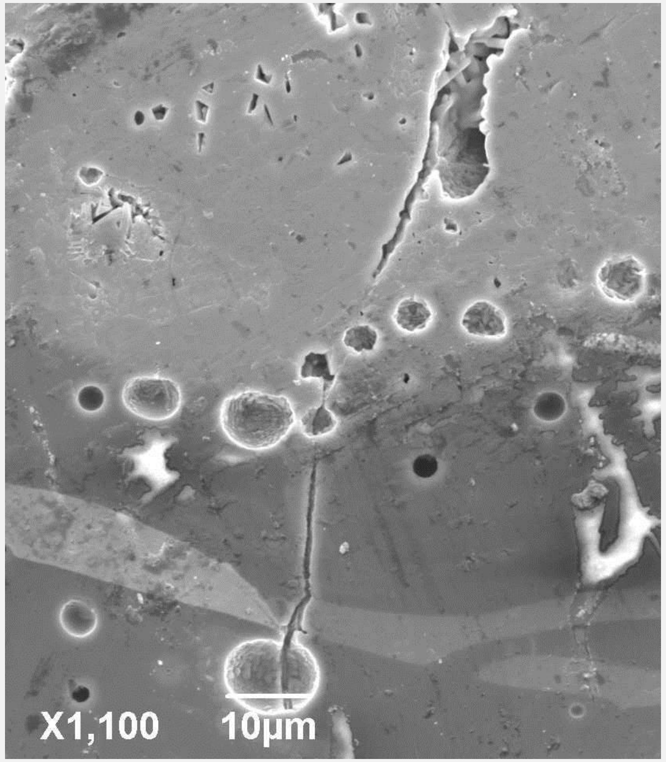


- NDE software allows for different density materials to be selectively removed from image
- With CMC removed, internal surface of the coating is also observed to be quite rough
- Have not studied effects of degree of grit blasting and mechanical bonding on the failure mechanism for this EBC/CMC system
- CT NDE not capable of identifying crack paths at this time

Cross Section of Heat Treated EBCs After Heat Treatment

Heat treatment at 1300°C / 20 hrs / Air





- Samples heat treated to 1300°C to transform BSAS to more the stable Celsian phase.
- Vertical cracks are present in as-coated material that heal partially upon heat treatment, these cracks are formed in cooling cycle during coating application
- None of these cracks propagated to CMC, mostly in top coat and intermediate layer
- Can easily distinguish between blunt cracks formed during coating application and cracks that are formed later from mechanical loading

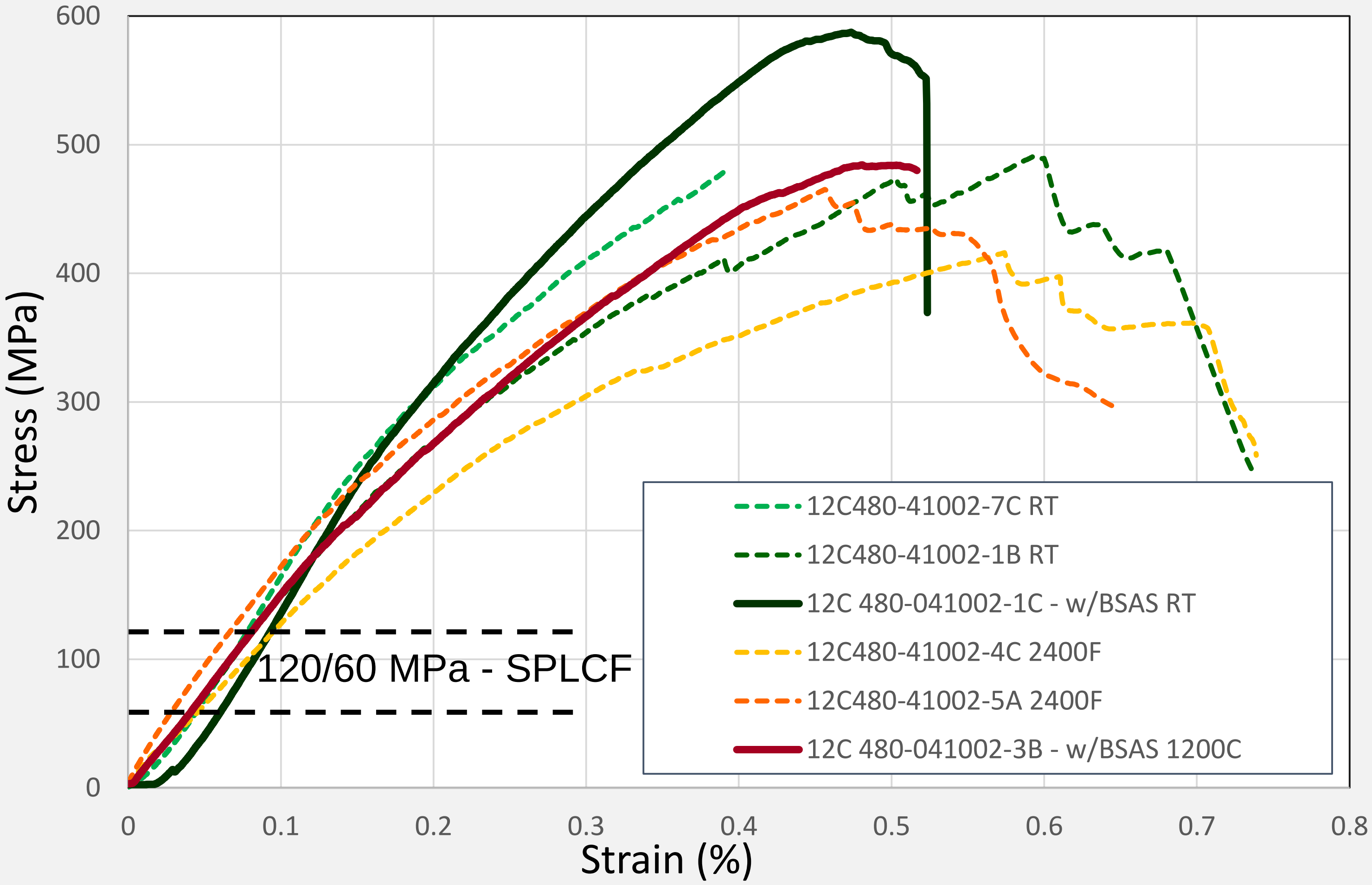
Mechanical Test Matrix

All Flexural Samples - All from the Same Panel

CMC	Coating	Test Type	Temperature	Environment	Stresses
SiC/SiC	None	Fast Fracture	Room Temp	Air	To Failure
SiC/SiC	None	Fast Fracture	1315°C	Air	To Failure
SiC/SiC	BSAS	Fast Fracture	Room Temp	Air	To Failure
SiC/SiC	BSAS	Fast Fracture	1315°C	Air	To Failure
SiC/SiC	BSAS	Incremental Loading	Room Temp	Air	To PL
SiC/SiC	BSAS	Incremental Loading	1200°C	Air	To PL
SiC/SiC	None	SPLCF - 100 hrs	1200°C	Air	120-60 Mpa
SiC/SiC	None	SPLCF - 100 hrs	1200°C	50% Steam	120-60 Mpa
SiC/SiC	BSAS	SPLCF - 100 hrs	1200°C	Air	120-60 Mpa
SiC/SiC	BSAS	SPLCF - 100 hrs	1200°C	50% Steam	120-60 Mpa

Fast Fracture Bend Test

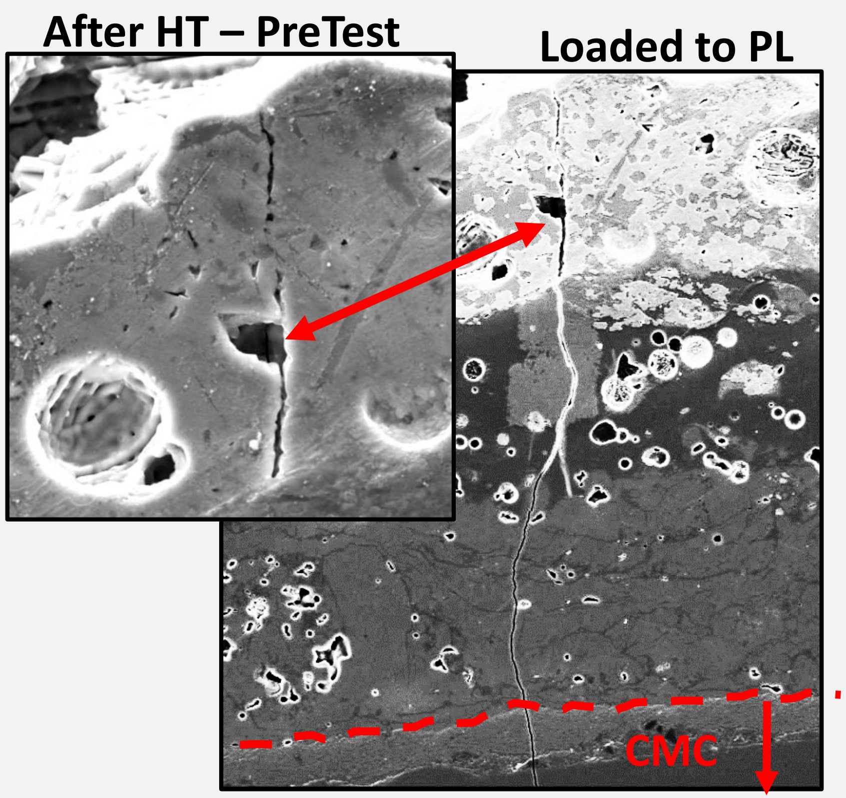
From single panel of RR-HTC CVI/SMI Panel # 12C480-41002



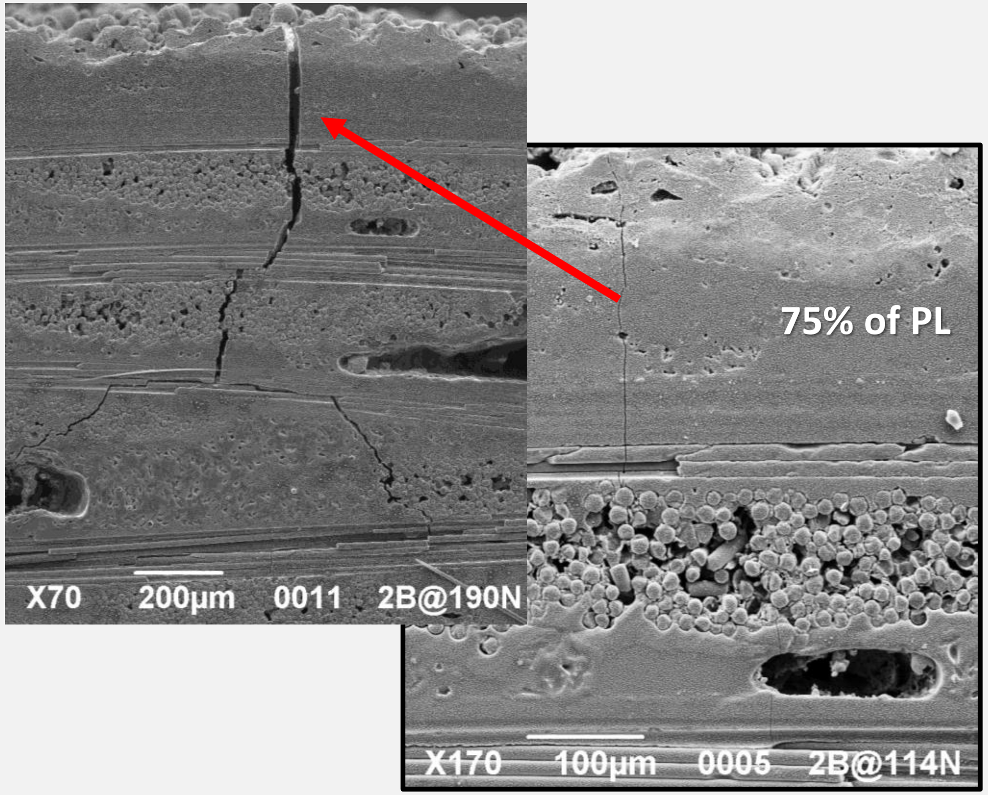
- Fast fracture used to determine proportional limit for incremental loading and SPLCF testing
- All samples used throughout the study were machined from the same panel
- Samples with all sides coated exhibited slightly higher proportional limit

Flexural Failure – Incremental Loading

Flexural Failure at Room Temp

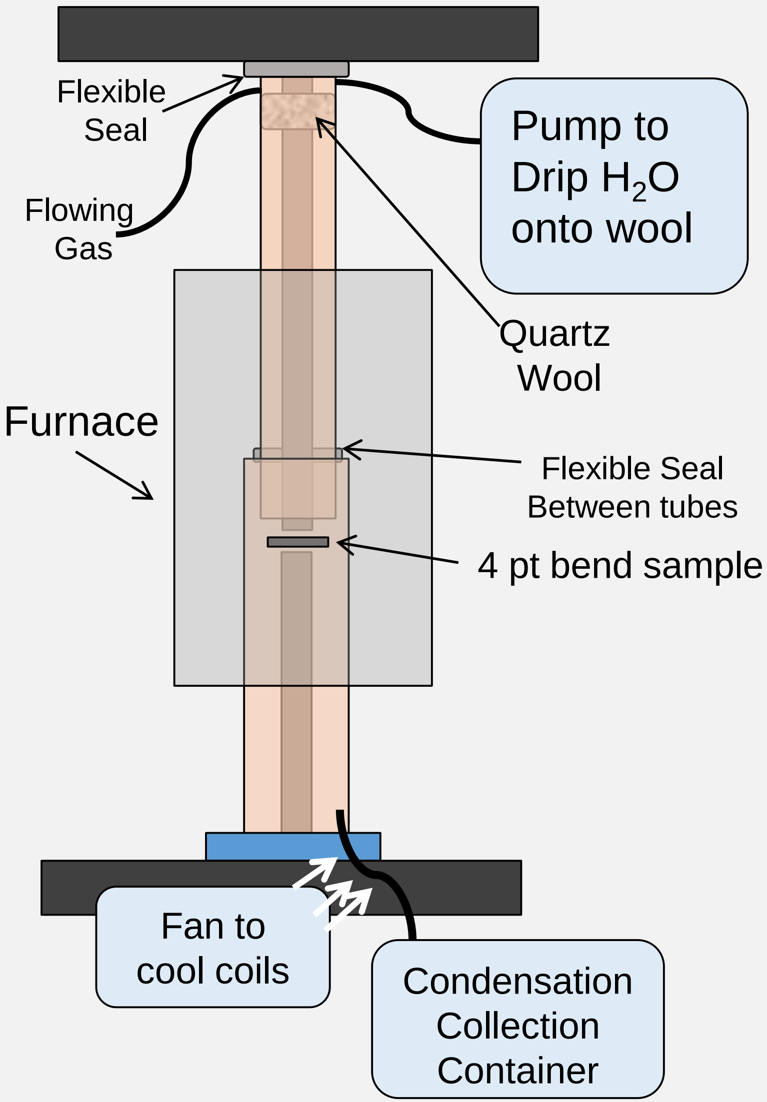


Flexural Failure at 1200^{o}C



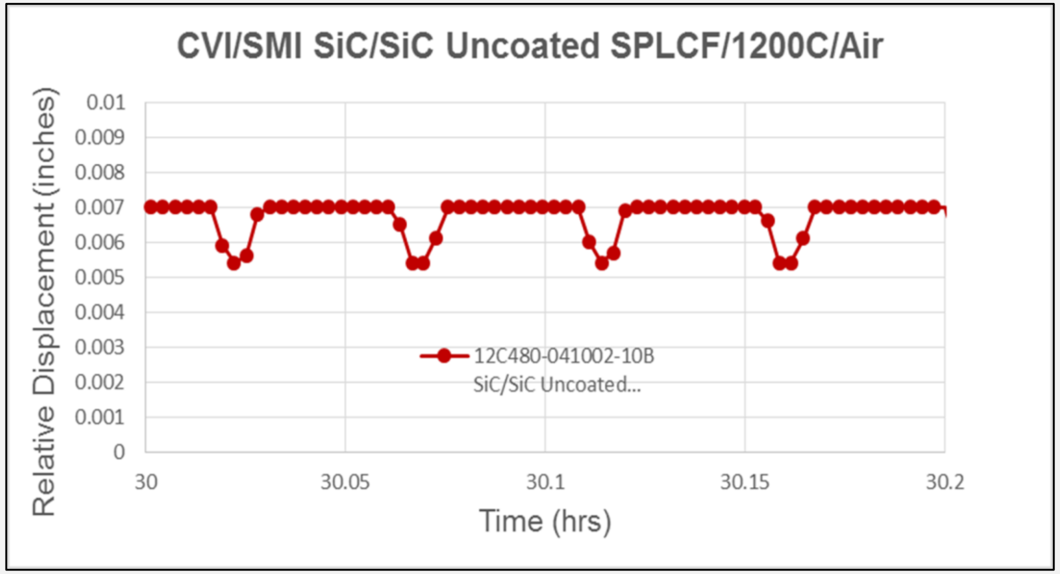
- With incremental loading up to the proportional limit, cracks initiate in the coating near the tensile surface and propagate into the CMC without deflection within the coating layers or any delamination of coating.
- Crack deflection only occurred within the composite at failure loads for both HT or RT loading

Flexural Fatigue Test Set-up

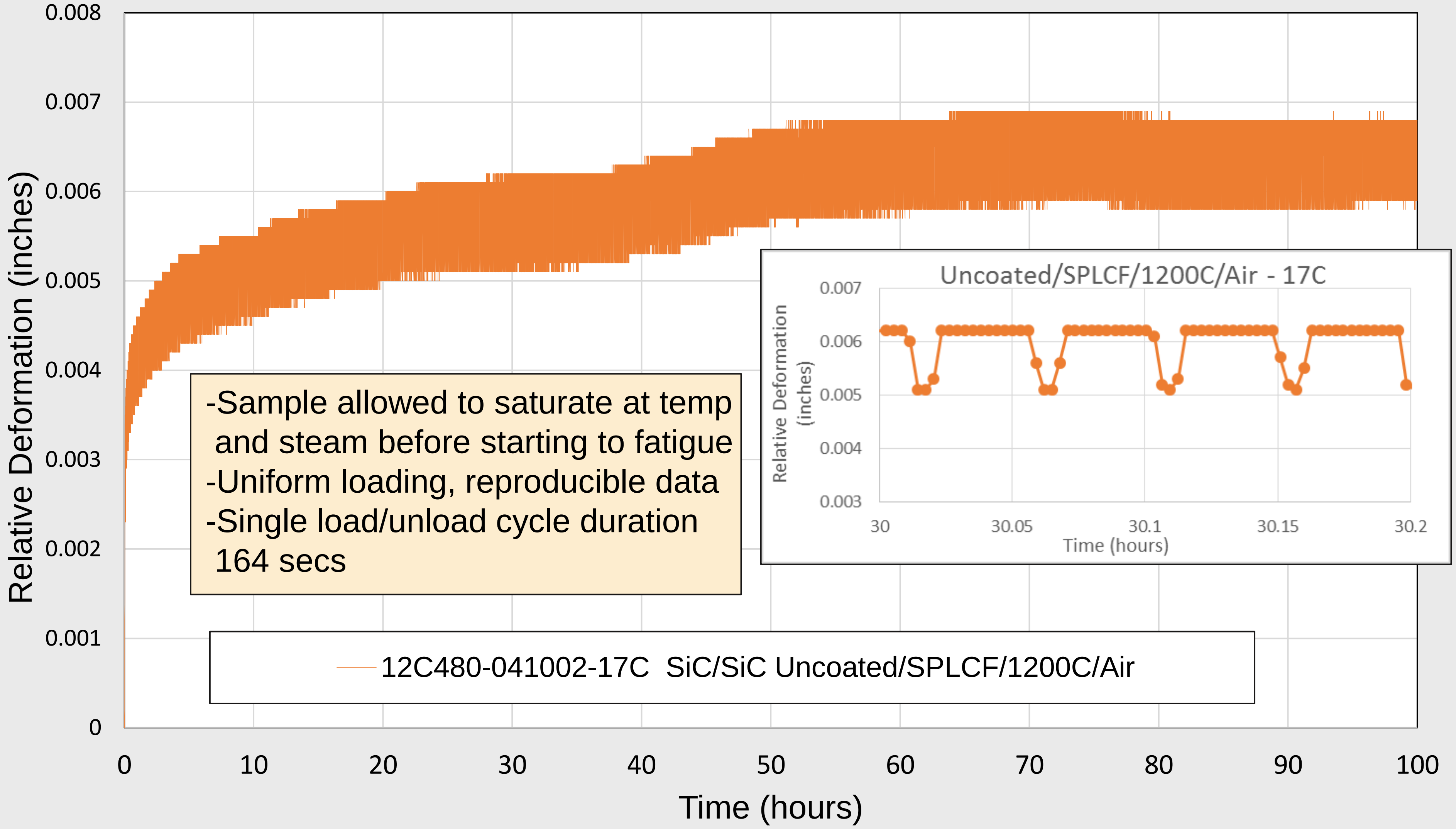


<u>Lever Arm Creep Frame</u>

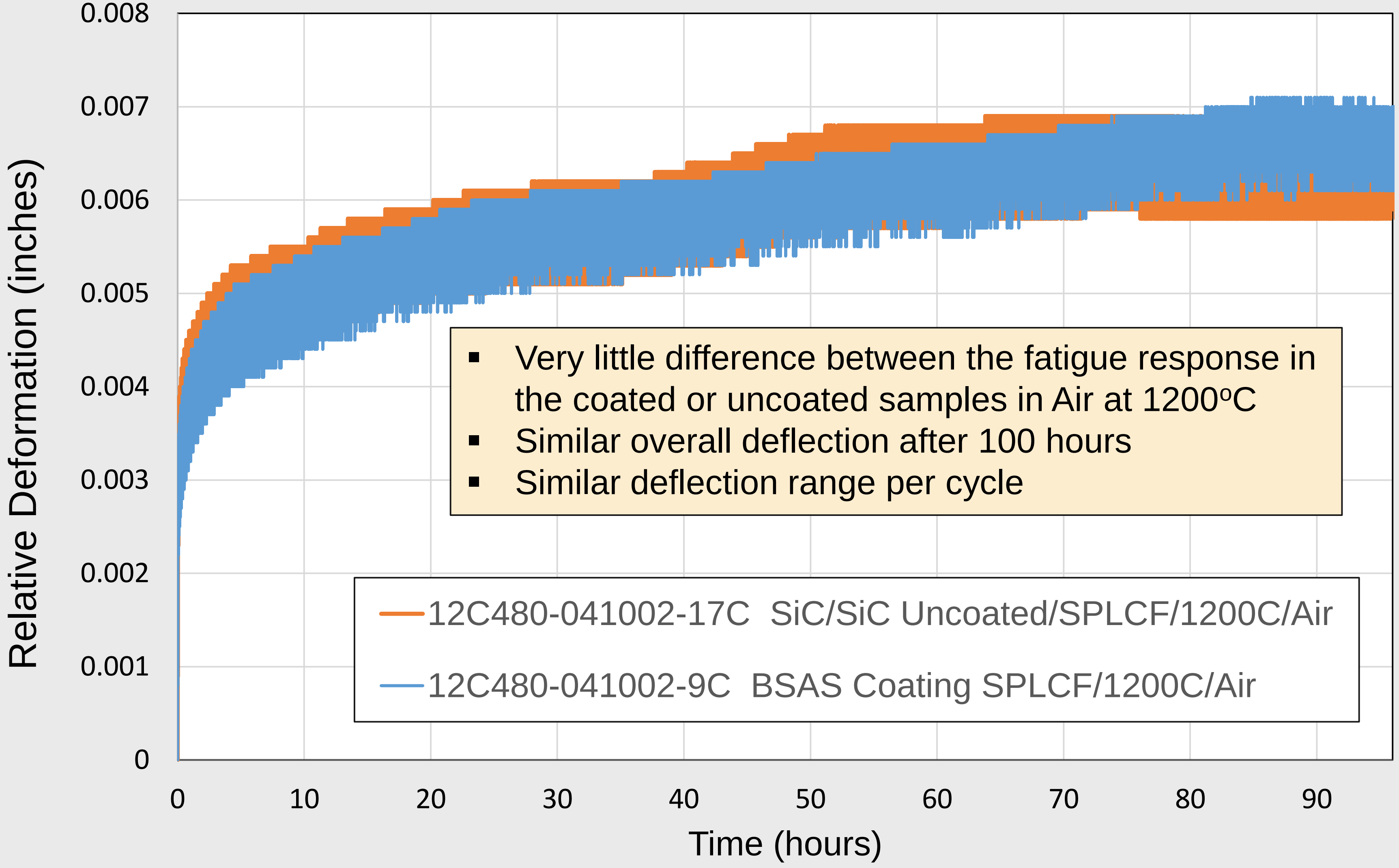
- Testing in 50% steam, atmospheric pressure
- 4 pt bend test
- 50 mm flex bars, 20/40mm load/support span
- Sustained-peak Low-cycle Fatigue (SPLCF) - two minutes at peak load, 10 sec at reduced load
- R = 0.5, cycle between 120MPa and 60 MPa



Uncoated SiC/SiC - SPLCF/1200ºC/Air

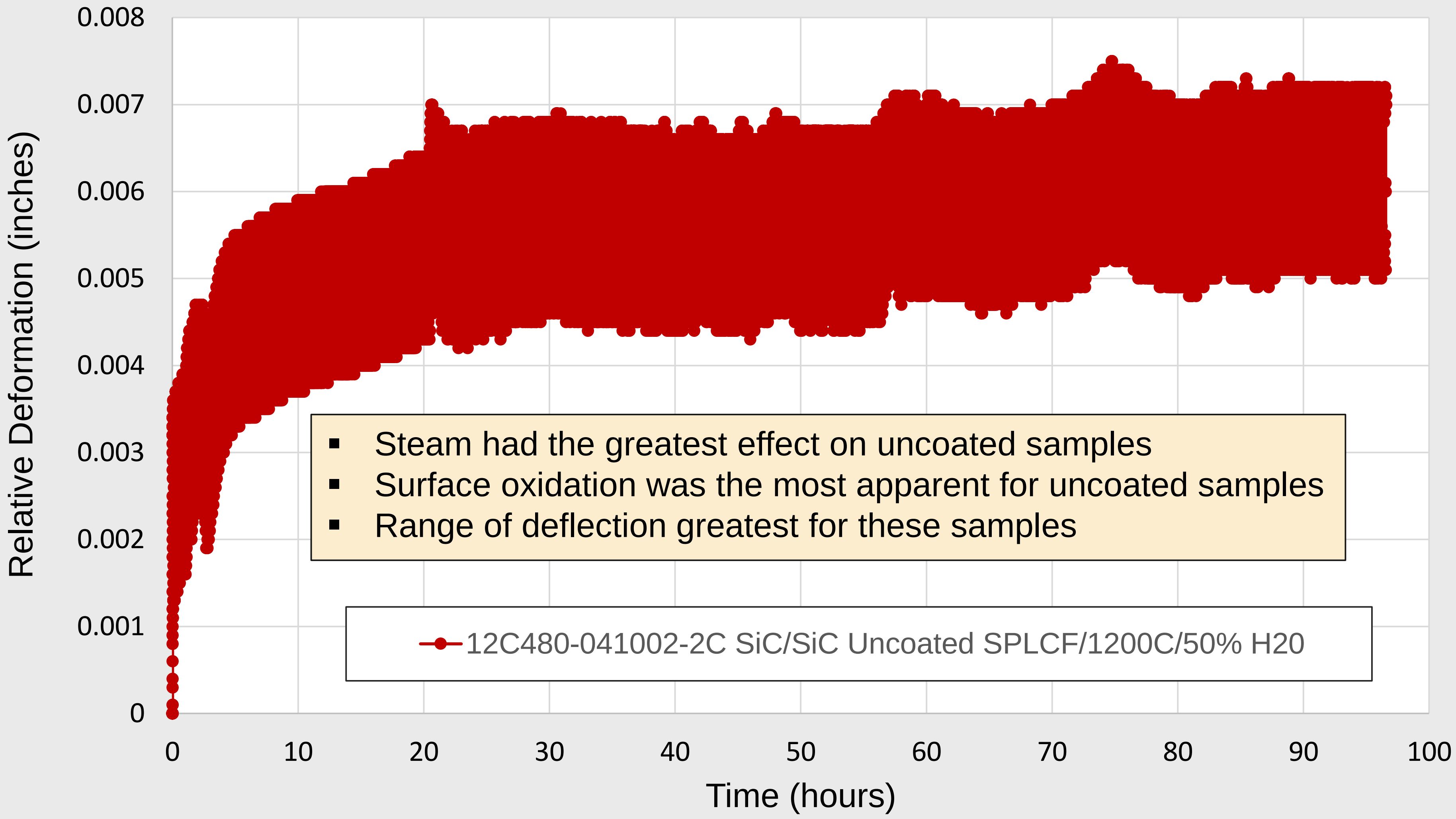


Uncoated vs Coated SiC/SiC - SPLCF /1200ºC / Air



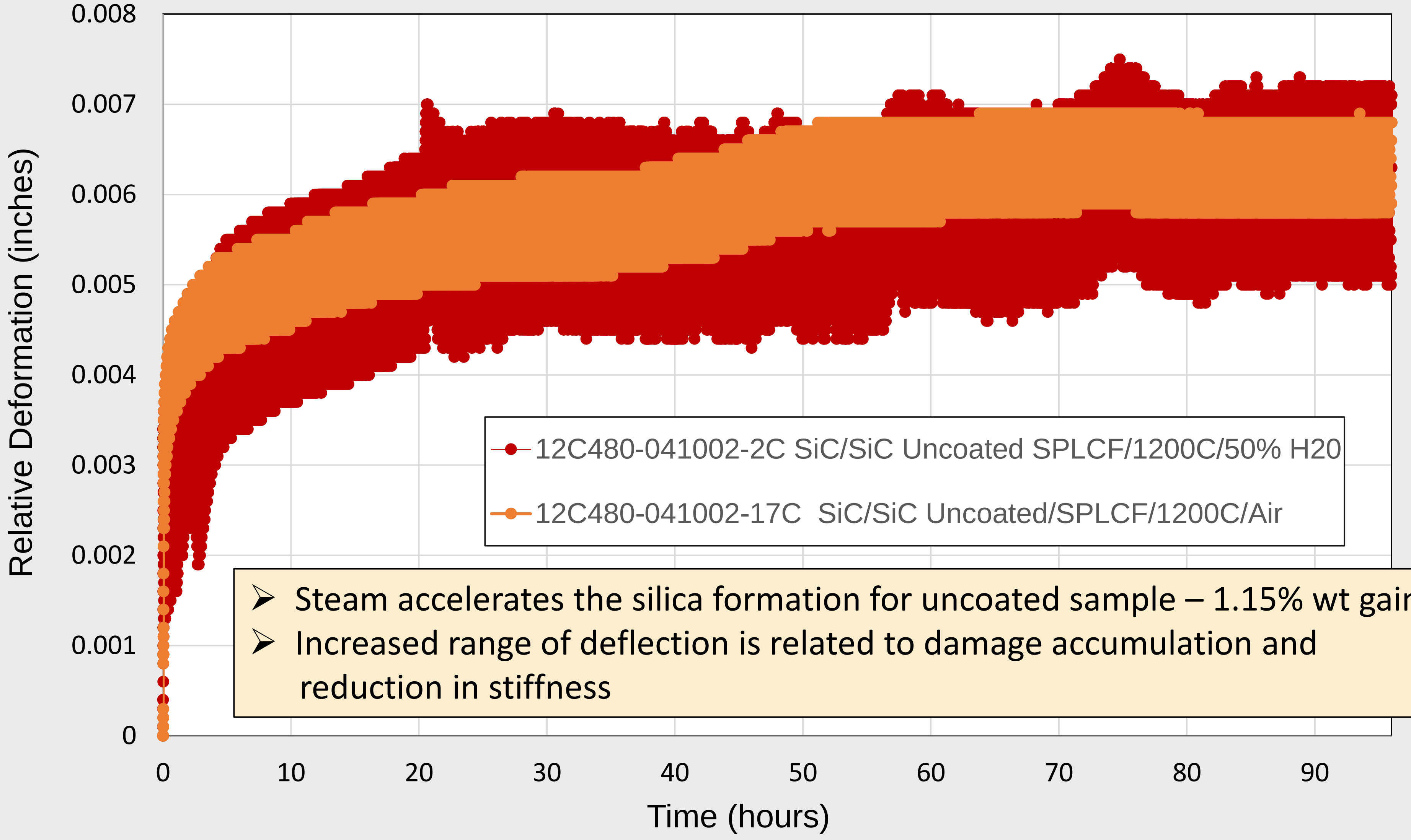
- Very little difference between the fatigue response in the coated or uncoated samples in Air at 1200ºC
- Similar overall deflection after 100 hours
- Similar deflection range per cycle

Uncoated SiC/SiC - SPLCF/1200ºC/50% H_20



- Steam had the greatest effect on uncoated samples
- Surface oxidation was the most apparent for uncoated samples
- Range of deflection greatest for these samples

Uncoated SiC/SiC - SPLCF/1200ºC in Air or Steam

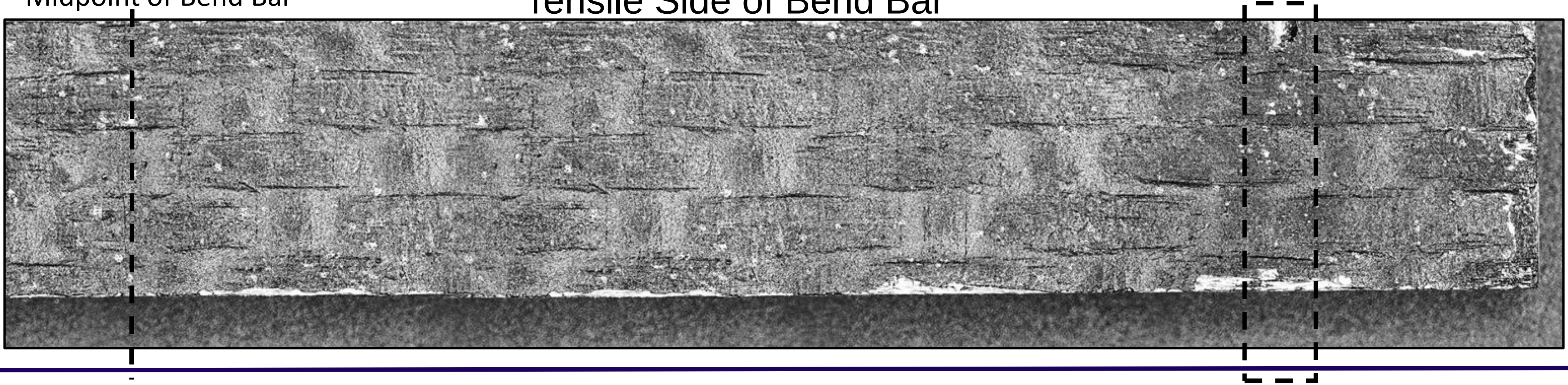


- Steam accelerates the silica formation for uncoated sample – 1.15% wt gain
- Increased range of deflection is related to damage accumulation and reduction in stiffness

Optical Microscopy of Tensile Surface
Uncoated SiC/SiC – SPLCF / 1200°C / Steam

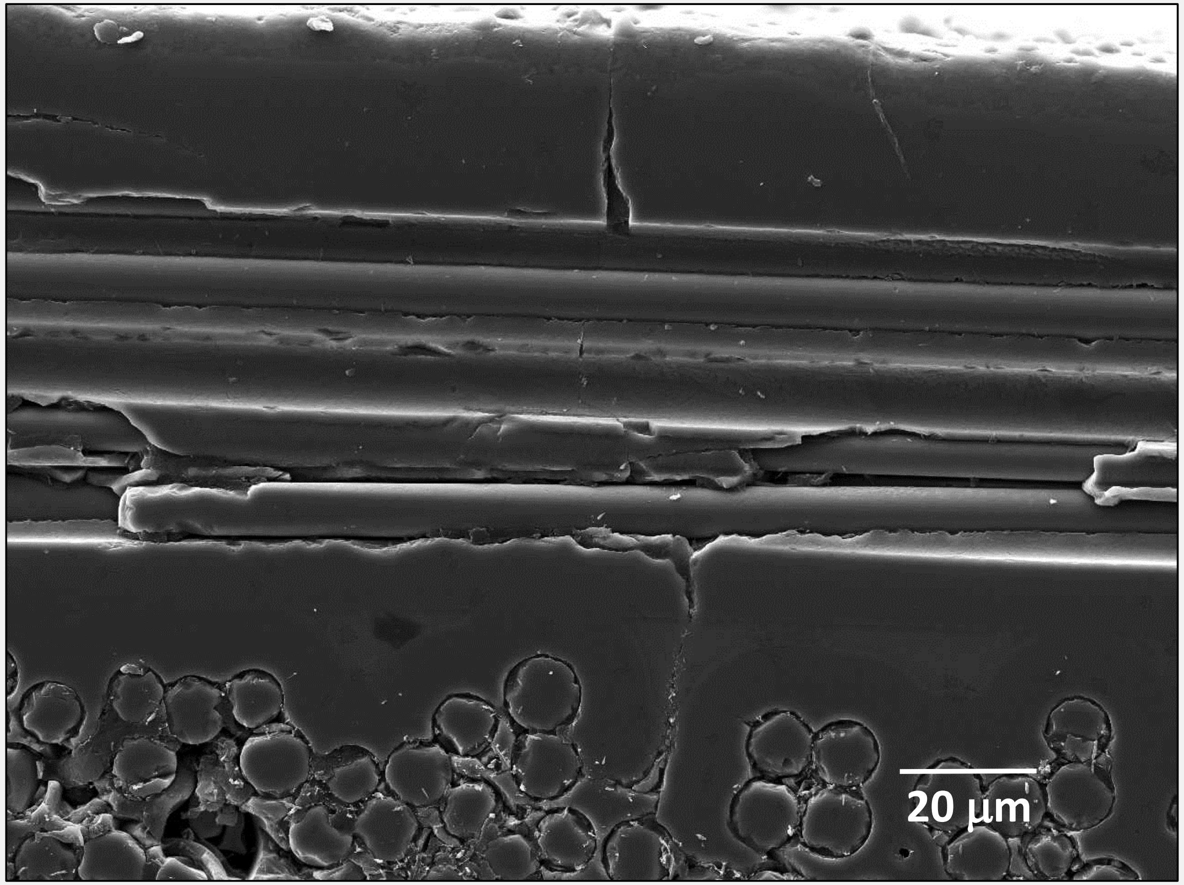
- Extensive transverse surface cracking on tensile side of uncoated bend bar fatigued in steam
- Oxidation and glass formation apparent on all sides of sample
- Little or no bonding between bend fixture and bend bar
- No discernible cracking in uncoated samples fatigued in air

Tensile Side of Bend Bar



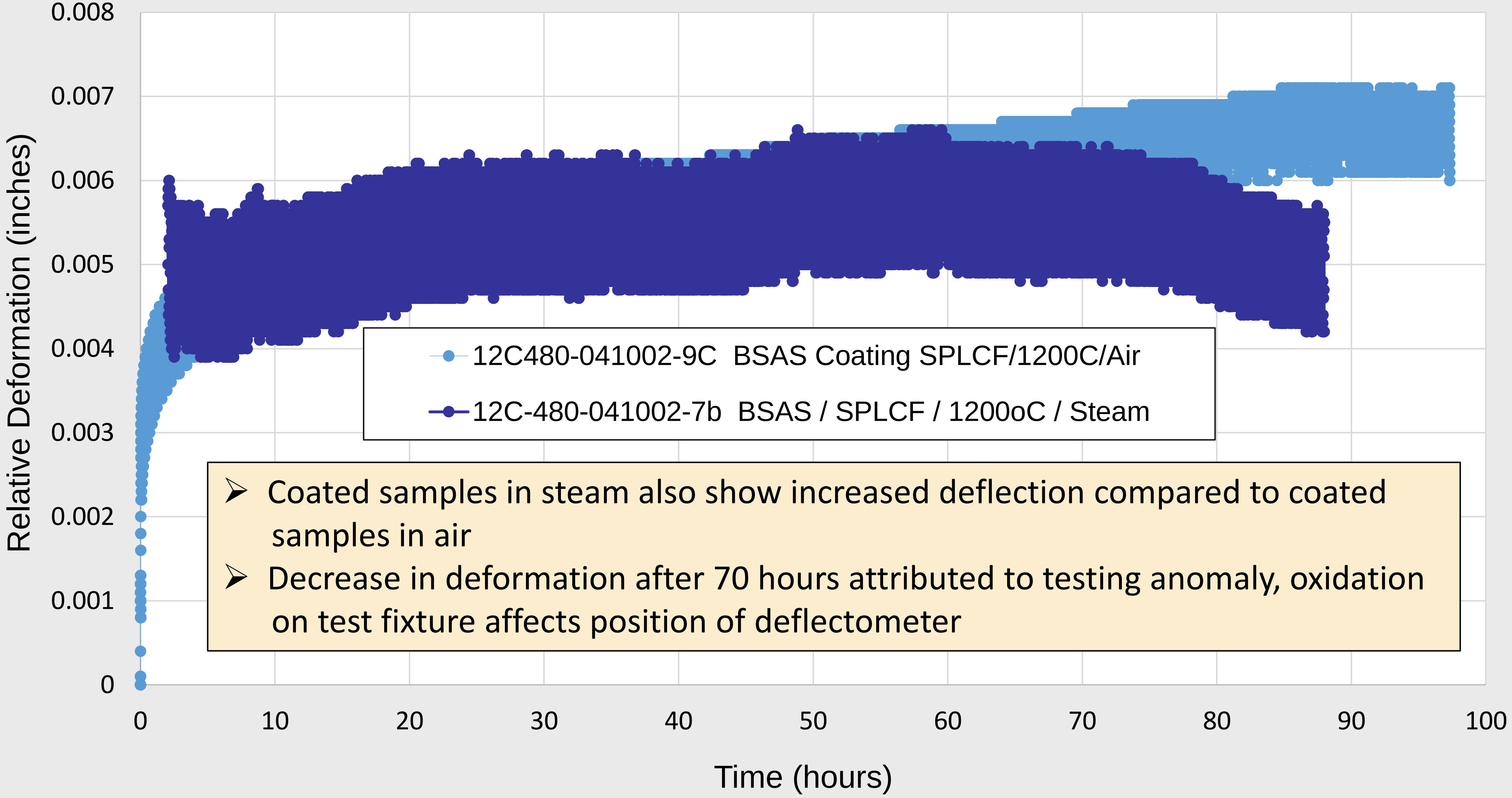
Uncoated SiC/SiC – SPLCF / 1200ºC / Steam

SEM Cross-Section



- Cracks on tensile side of bend bar extend into matrix
- Fibers appear to remain intact

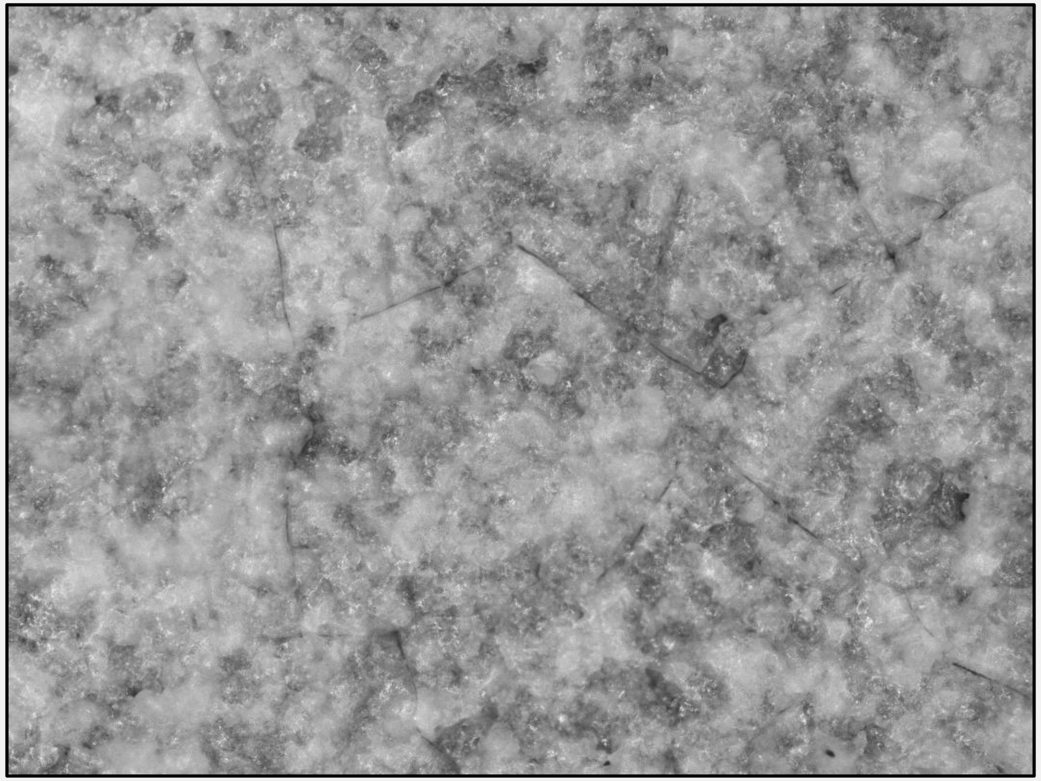
Coated SiC/SiC - SPLCF/1200ºC in Air or Steam



- Coated samples in steam also show increased deflection compared to coated samples in air
- Decrease in deformation after 70 hours attributed to testing anomaly, oxidation on test fixture affects position of deflectometer

Optical Microscopy of Tensile Surface

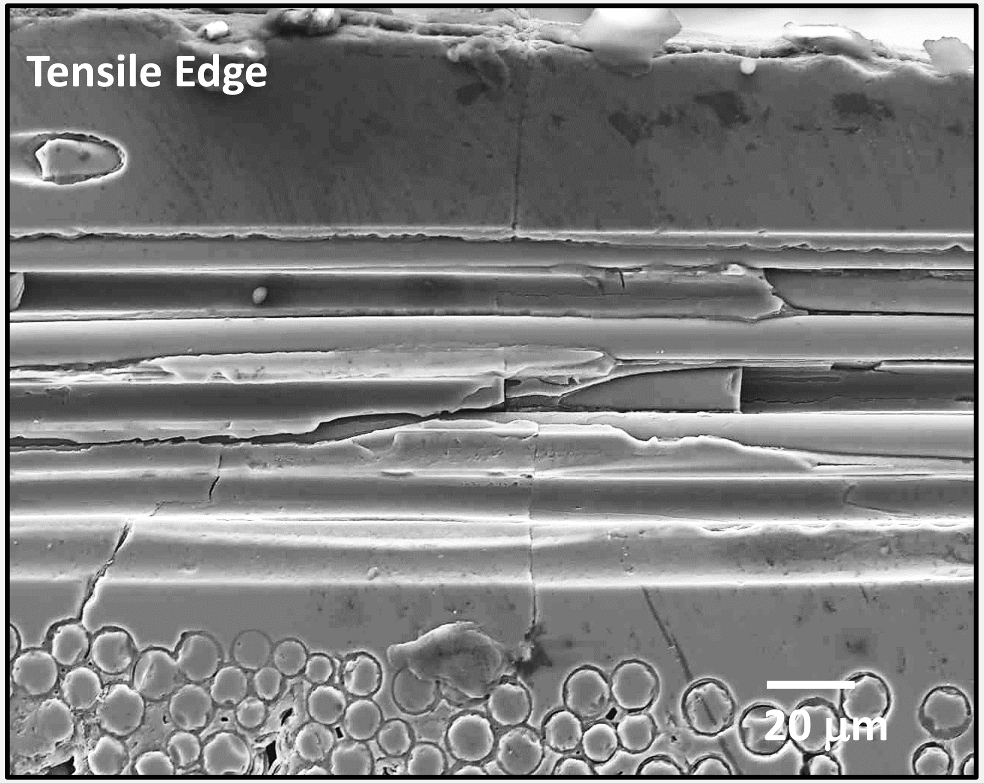
BSAS Coated SiC/SiC – SPLCF / 1200^{o}C / Steam



- Difficult to find any transverse surface cracking on coated samples fatigued in air, small amount of random surface cracks
- Easier to identify random surface cracking in the coated samples fatigued in steam vs. coated samples fatigued in air
- Crack patterns may be left from phase transformation/heat treatment step

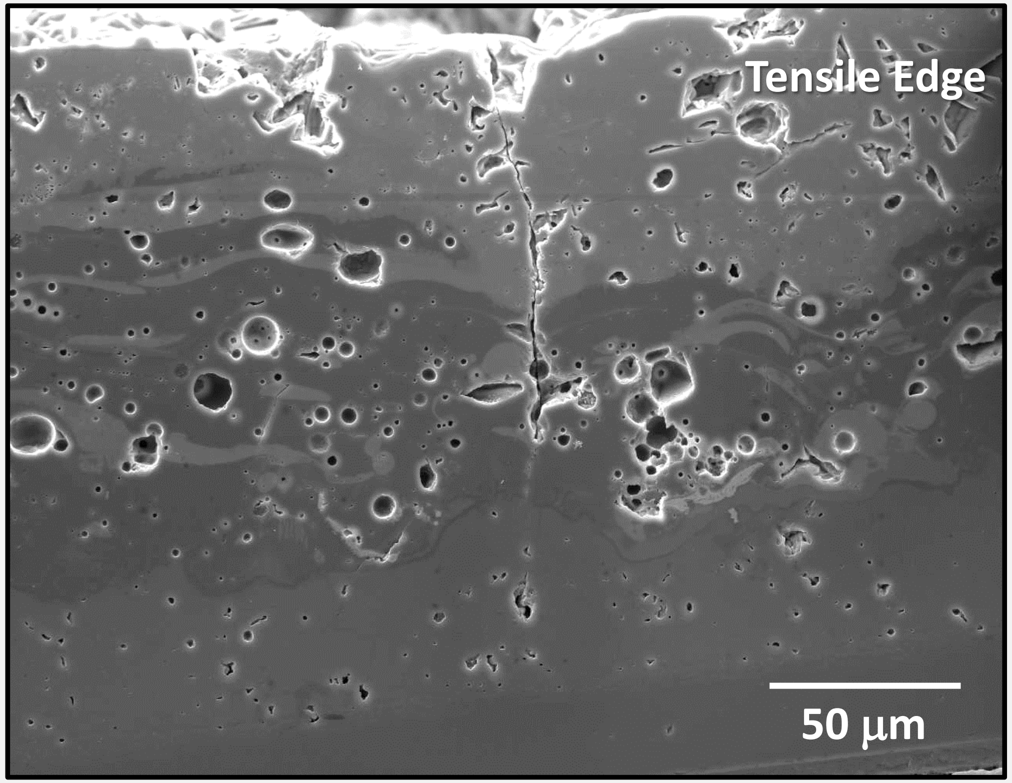
SPLCF / 1200ºC / Steam
SEM Cross-Section

Uncoated SiC/SiC Steam



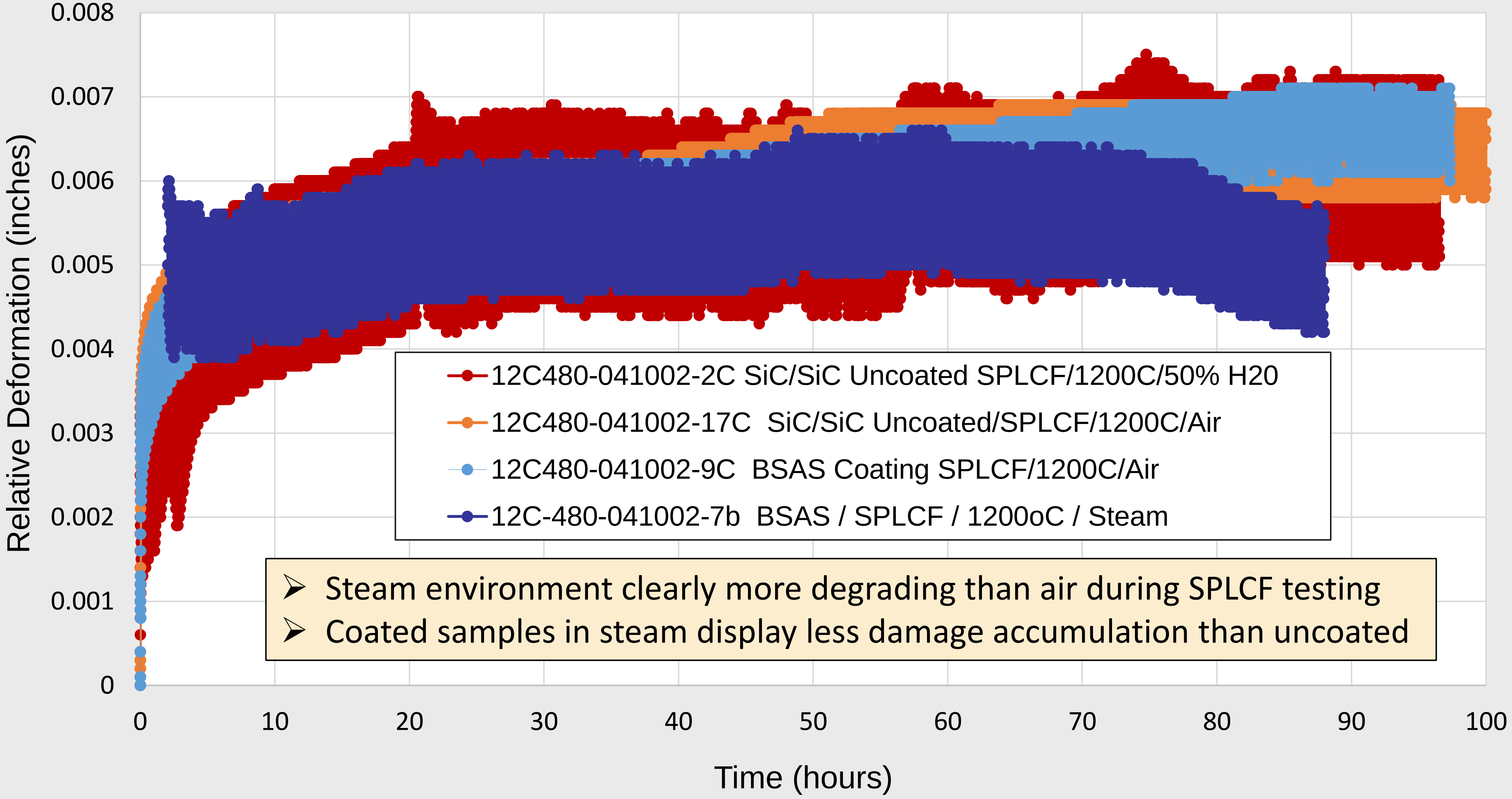
- Surface cracking - tensile side of bend bar
- Cracks propagate thru the matrix and around fibers
- Fibers appear to remain intact

BSAS Coated SiC/SiC Steam



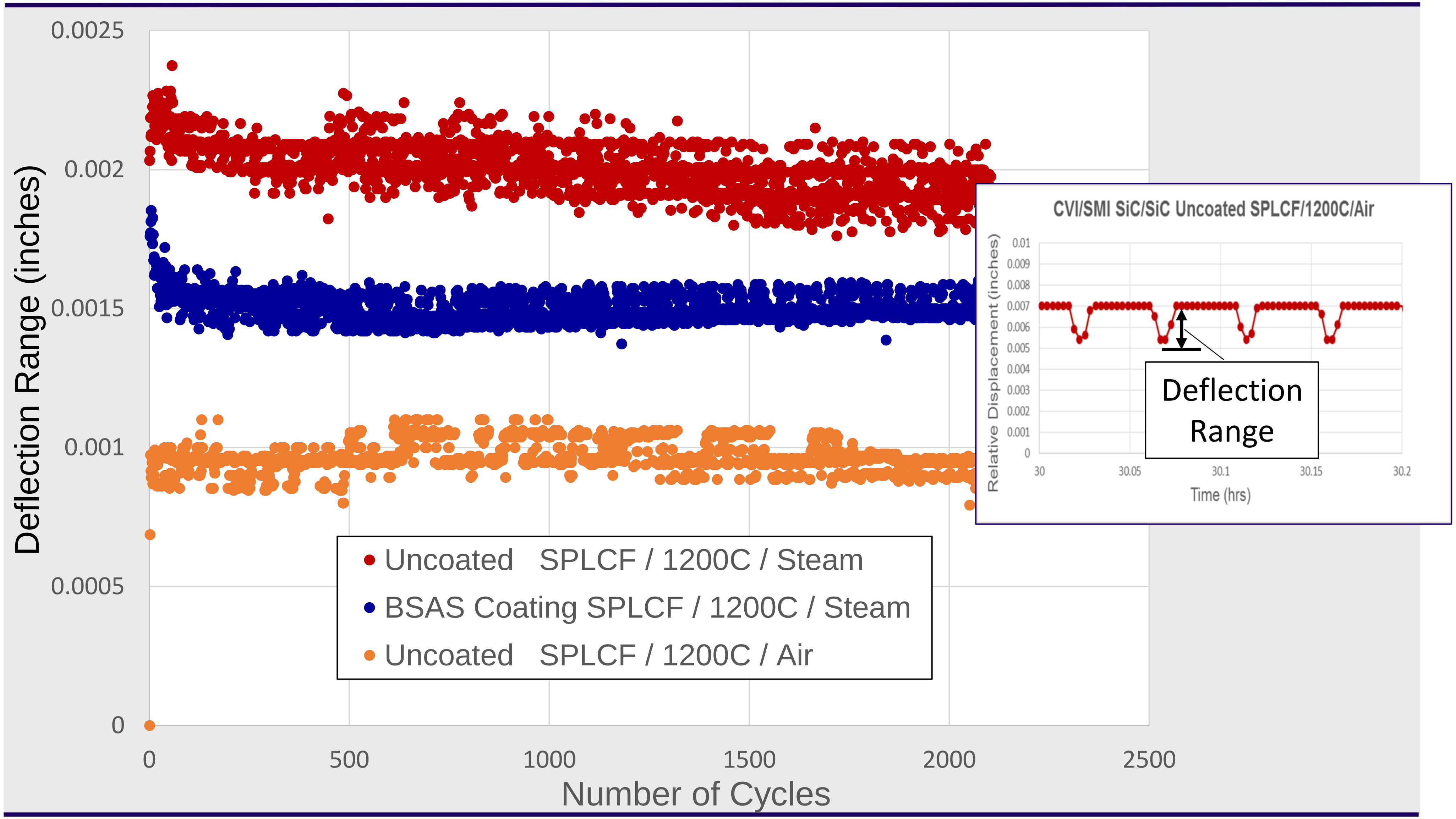
- Mud flat type of cracking at surface leads to vertical cracks in coating
- Cracks tend to bridge large flaws
- Vertical cracks create oxidation pathways to the CMC

Coated vs Uncoated in Steam or Air

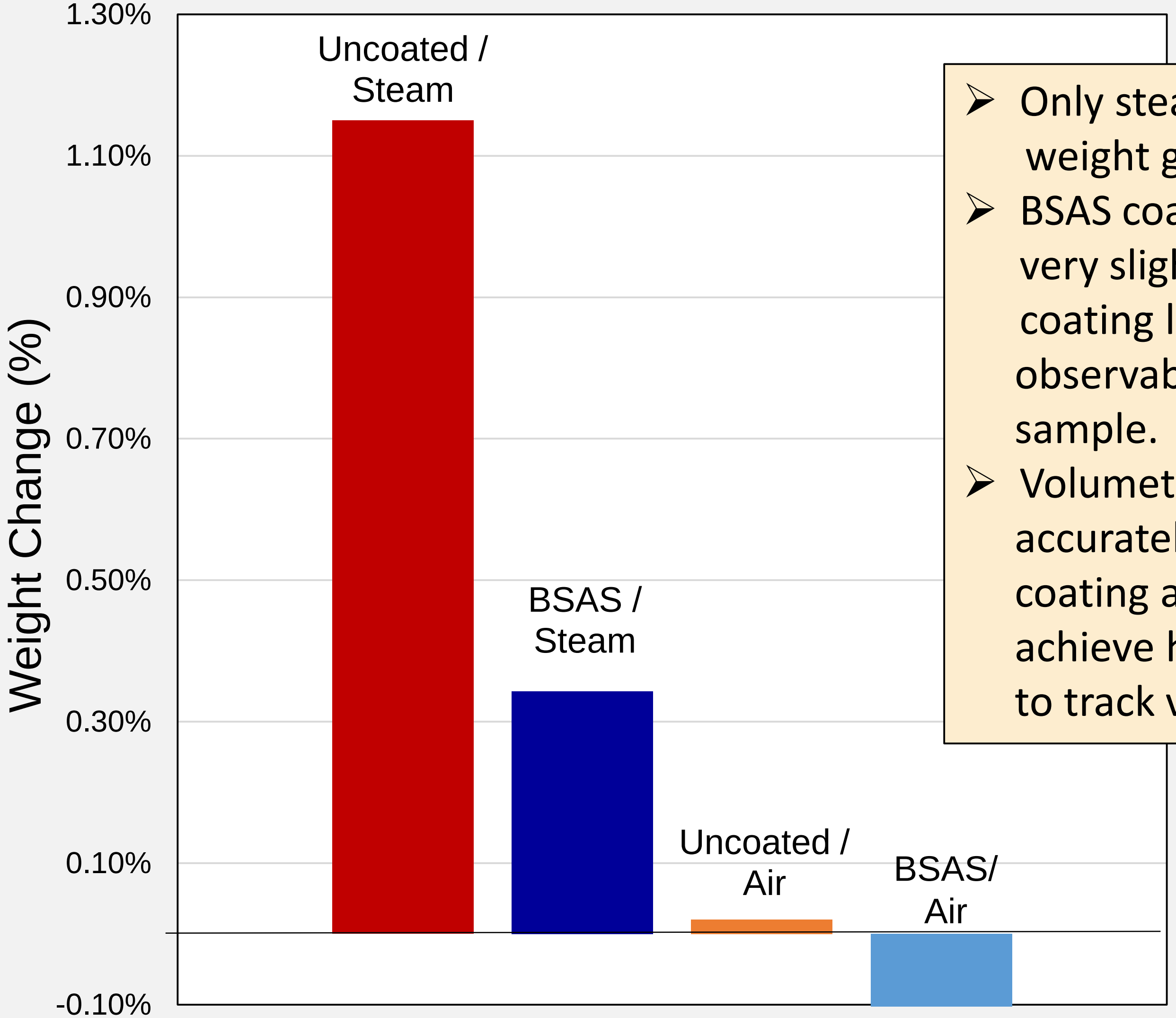


- Steam environment clearly more degrading than air during SPLCF testing
- Coated samples in steam display less damage accumulation than uncoated

Change in Deflection Range with Cycles



% Weight Change after SPLCF in Air or Steam

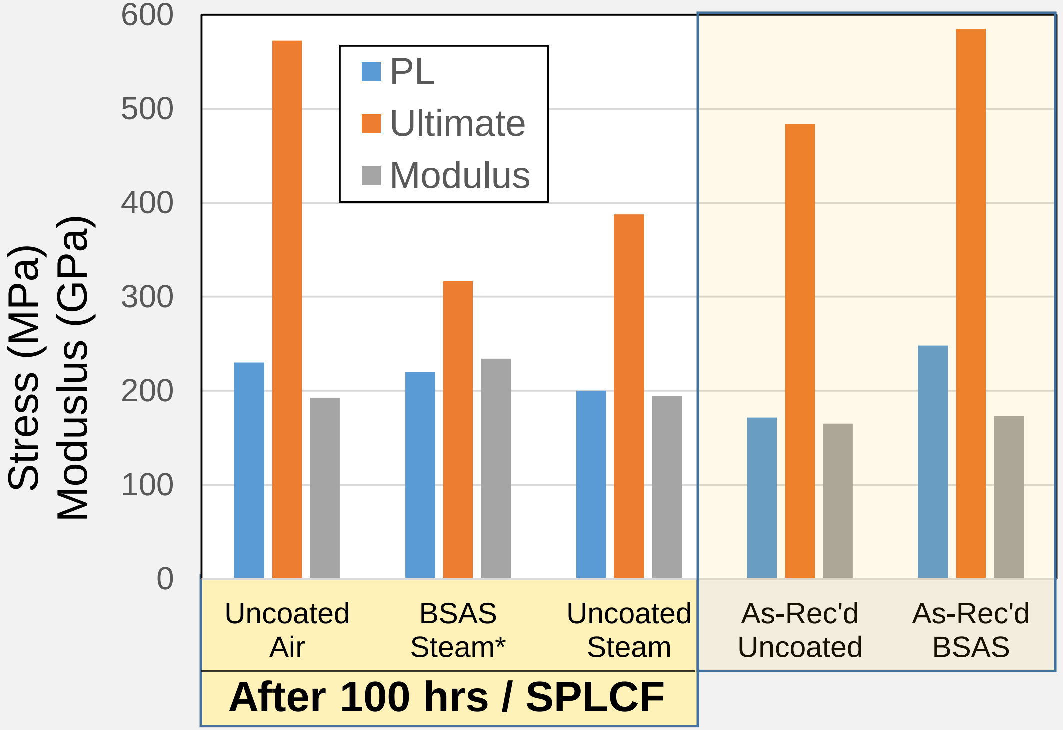


- Only steam runs showed any significant weight gain due to oxidation
- BSAS coated sample tested in air showed very slight weight loss, possibly due to coating loss during handling or testing. No observable spalling of coating on any sample.
- Volumetric changes too small to measure accurately, surface roughness of both the coating and composite make it difficult to achieve high fidelity measurements needed to track volume changes

Residual Strength after Fatigue Tests

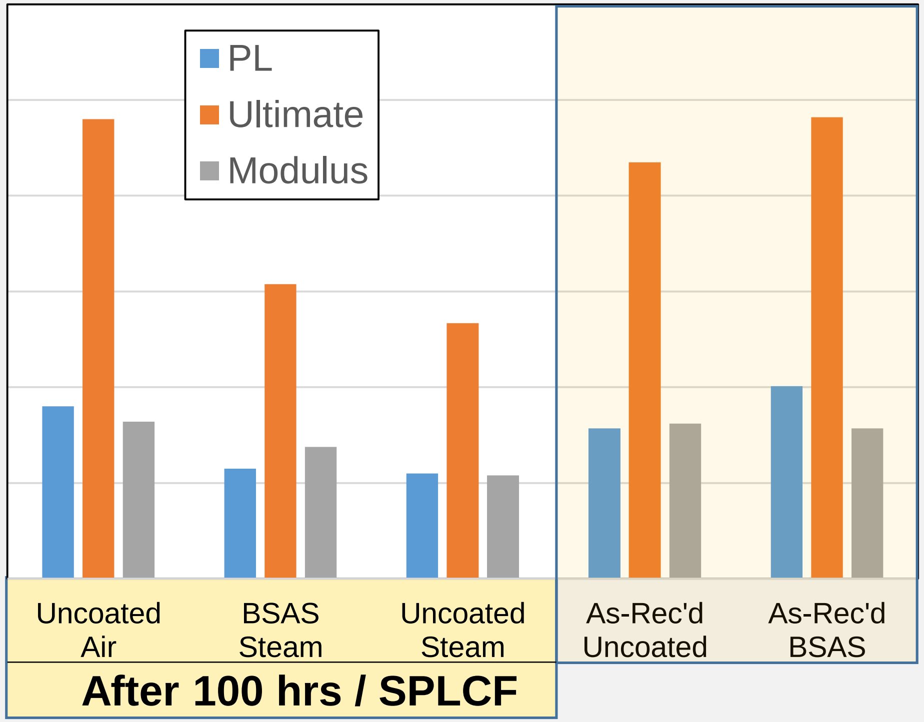
Room Temp Tests

Residual Bend Properties after SPLCF
vs As-Received Bend Properties



1200°C Tests

Residual Bend Properties after SPLDF
vs As-Received Bend Properties



- Fatigued in air - 1200°C for 100 hours, 120 / 60 Mpa did not result in strength loss
- Fatigued in steam - resulted in lower residual tests compared to air tests
- BSAS/Steam* RT test -- SPLCF test for this sample ran longer, 130 hours total

Summary

- A test method was developed to evaluate the effects of fatigue and steam on the failure behavior of an EBC/CMC system.
- Tests to date: 100 hrs under SPLCF conditions, max stress of 120 Mpa, in air or 50% H_2O
- No spalling of BSAS coating or deflection of cracks within the coating layers for any of the test conditions, either incremental loading or SPLCF
- Only the uncoated samples tested in steam showed transverse cracks on the surface, these cracks propagated into the matrix without damaging fibers
- Coated samples showed random cracking on the surface, crack propagation into matrix was not observed
- Uncoated samples fatigued in steam showed the greatest degree of oxidation and matrix cracking

Conclusions and Future Efforts

- Recent SPLCF tests in steam or dry air agree with results of incremental loading tests; no spalling or deflection of cracks within the coating for any of our test conditions.
- Steam environment accelerated the oxidation of both the coated and uncoated samples and resulted in residual strength and modulus loss for all samples.
- BSAS coatings slowed the oxidation in steam and limited the matrix cracking, but flaws produced during coating process lead to cracking in coating and provide oxidation paths to the CMC
- Current fatigue/steam rig and test fixture needs to be modified.
- Need to further investigate methods of dye infusion with NDE for ease of crack path observation throughout the sample.
- Expand test matrix; increase loads and times.
- Develop an empirical model showing the effects of steam on the degradation of an EBC/CMC system.