Tensile Creep and Fatigue of Sylramic-iBN Melt-Infiltrated SiC Matrix Composites: Retained Properties, Damage Development, and Failure Mechanisms

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Abstract

An understanding of the elevated temperature tensile creep, fatigue, rupture, and retained properties of ceramic matrix composites (CMC) envisioned for use in gas turbine engine applications are essential for component design and life-prediction. In order to quantify the effect of stress, time, temperature, and oxidation for a state-of-the-art composite system, a wide variety of tensile creep, dwell fatigue, and cyclic fatigue experiments were performed in air at 1204oC for the SiC/SiC CMC system consisting of Sylramic-iBN SiC fibers, BN fiber interphase coating, and slurry-cast melt-infiltrated (MI) SiC-based matrix. Tests were either taken to failure or interrupted. Interrupted tests were then mechanically tested at room temperature to determine the residual properties. The retained properties of most of the composites subjected to tensile creep or fatigue were usually within 20% of the as-produced strength and 10% of the as-produced elastic modulus. It was observed that during creep, residual stresses in the composite are altered to some extent which results in an increased compressive stress in the matrix upon cooling and a subsequent increased stress required to form matrix cracks. Microscopy of polished sections and the fracture surfaces of specimens which failed during stressed-oxidation or after the room-temperature retained property test was performed on some of the specimens in order to quantify the nature and extent of damage accumulation that occurred during the test. It was discovered that the distribution of stress-dependent matrix cracking at 1204oC was similar to the as-produced composites at room temperature; however, matrix crack growth occurred over time and typically did not appear to propagate through thickness except at final failure crack. Failure of the composites was due to either oxidation-induced unbridged crack growth, which dominated the higher stress regime ($\geq$ 179 MPa) or controlled by degradation of the fibers, probably caused by intrinsic creep-induced flaw growth of the fibers or internal attack of the fibers via Si diffusion through the CVI SiC and/or microcracks at the lower stress regime ($\leq$ 165 MPa).

Outline:

- Approach and methodology
- Examination of standard specimens after air testing
  - Residual properties: what's left in the material
  - Optical microscopy: extent of matrix cracking
  - Fracture surface microscopy: nature of failure
  - Life-degrading mechanisms
- Conclusions

Damage Evolution

Approach and Methodology

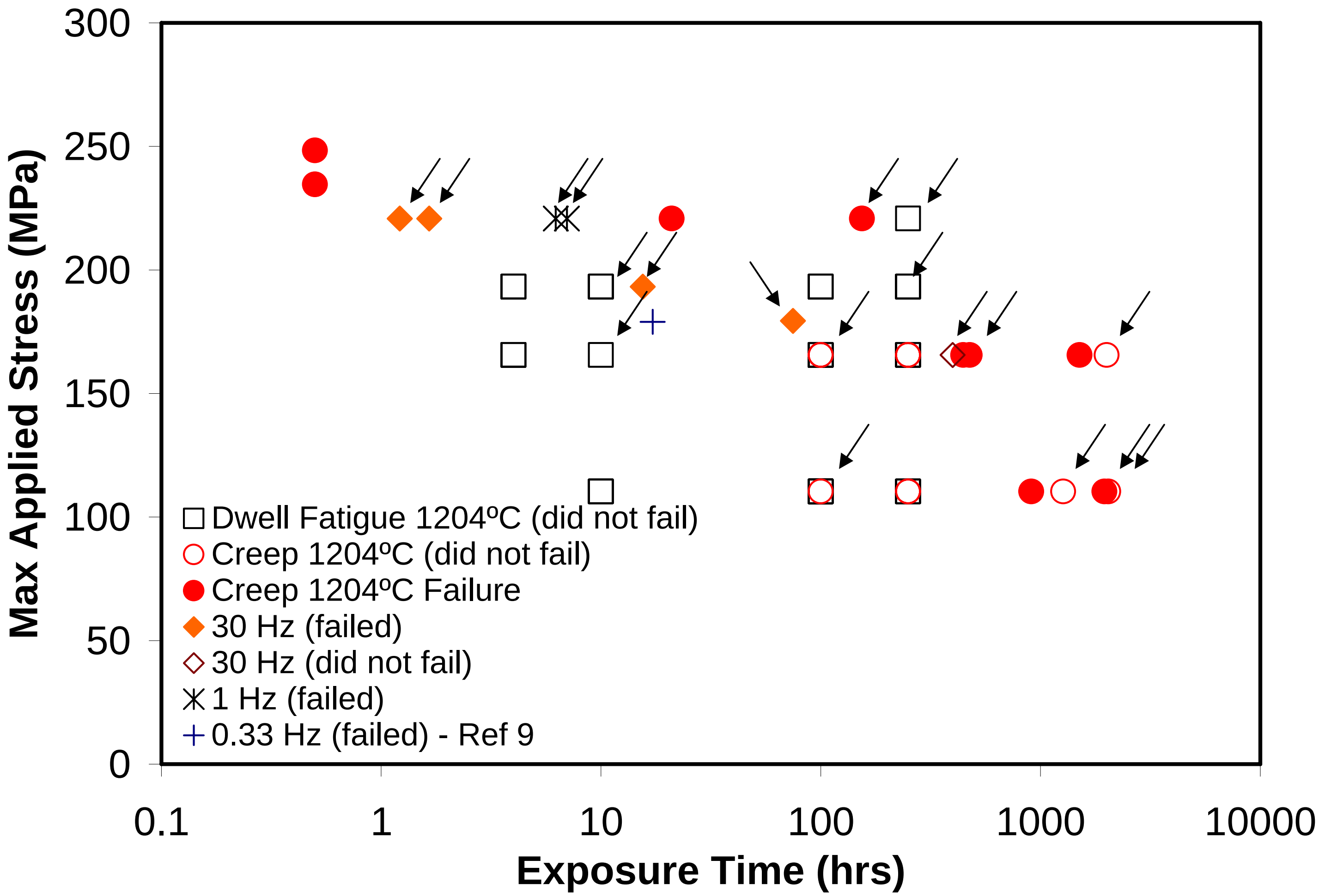
- Specimens were examined after creep or fatigue test if failed, or after a post-test room temperature failure if the specimen survived creep/fatigue condition. A few specimens which had survived the creep/fatigue condition were polished without post-test failure.
- ***Post-test room temperature fast fracture tests*** were often performed with acoustic emission to assess damage evolution and history dependent σ/ε behavior
- One section of the failed specimen was cut and ***polished along the length*** (edge or face alignment) for ***optical microscopy*** in order to determine the extent and amount of matrix cracking
- One section of the failed specimen was cut in order to examine the ***fracture surface*** (for most tests, the furnace was shut off with failure of the specimen which minimized the amount of oxidation on the fracture surface post-failure resulting in a fairly pristine fracture surface crack surface) via ***FESEM***

Damage Evolution: Standard Specimens Examined

Specimen (1300-01-)	Test	Stress, MPa/ksi	Cycles	Time, hr	Failure at Temperature	FESEM	RT Ret Test
002-p03	DF	165/24	5	10	No	--	--
005p06	DF	193/28	5	10	No	--	--
006-p01	Creep	165/16	--	*1269*	No	Yes	Yes
006-p02	Creep	110/16	--	2036	No	Yes	Yes
006-p05	creep	165/24	--	*478*	Yes	Yes	--
006-p06	creep	165/24	--	1508	Yes	--	--
006-p07	DF	193/28	5	10	No	--	Yes
006-p08	DF	193/28	125	250	No	--	Yes
006-p10	creep	110/16	--	*1953*	Yes	Yes	--
007-p03	creep	110/16	--	100	No	--	Yes
007-p04	creep	165/24	--	100	No	--	Yes
007-p07	DF	220/32	134	168	No	Yes	Yes
016-p05	Creep after RT HCF (1/30hz)	220/32	--	16	Yes	Yes	--
017-p05	HCF(1hz)-43ksi overload	220/32	18,457	*5*	Yes	Yes	--
017-p07	HCF(1/30hz)	220/32	154,546	*1*	Yes	--	--
017-p09	HCF(1hz)	220/32	25,217	*7*	Yes	Yes	--
018-p06	HCF(1/30hz)	220/32	131,100	*1*	Yes	Yes	--
018-p09	HCF(1/30hz)	220/32	178,493	*2*	Yes	Yes	--
018-p12	HCF(1/30hz)	165/24	4.2 E07	*389*	No	Yes	Yes
018-p13	HCF(1/30hz)	193/28	1,672,011	*15*	Yes	Yes	--
018-p14	HCF(1/30hz)	179/26	8,073,478	*75*	Yes	Yes	--

Damage Evolution: Standard Specimens

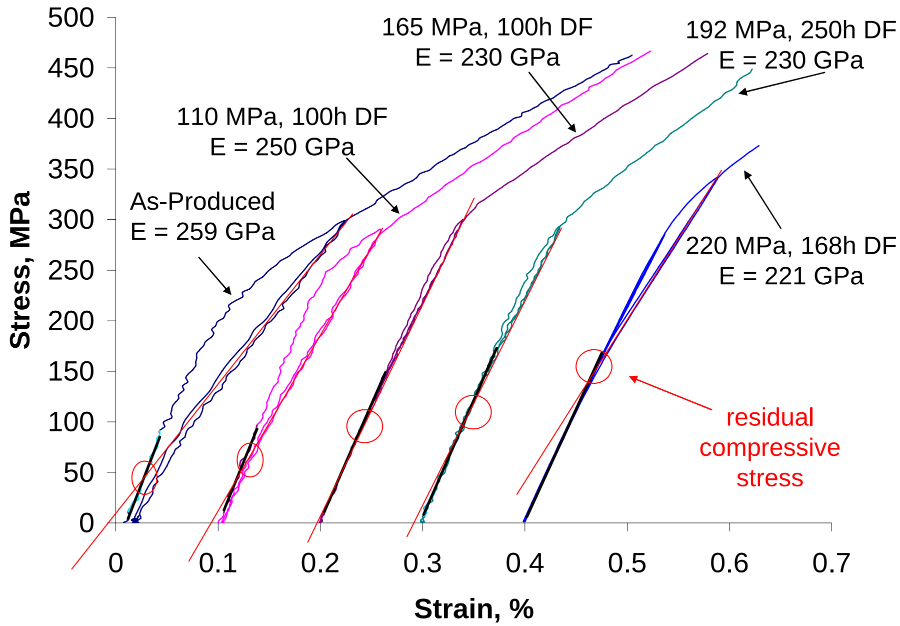
Specimens subjected to a wide range of conditions were examined (arrows)



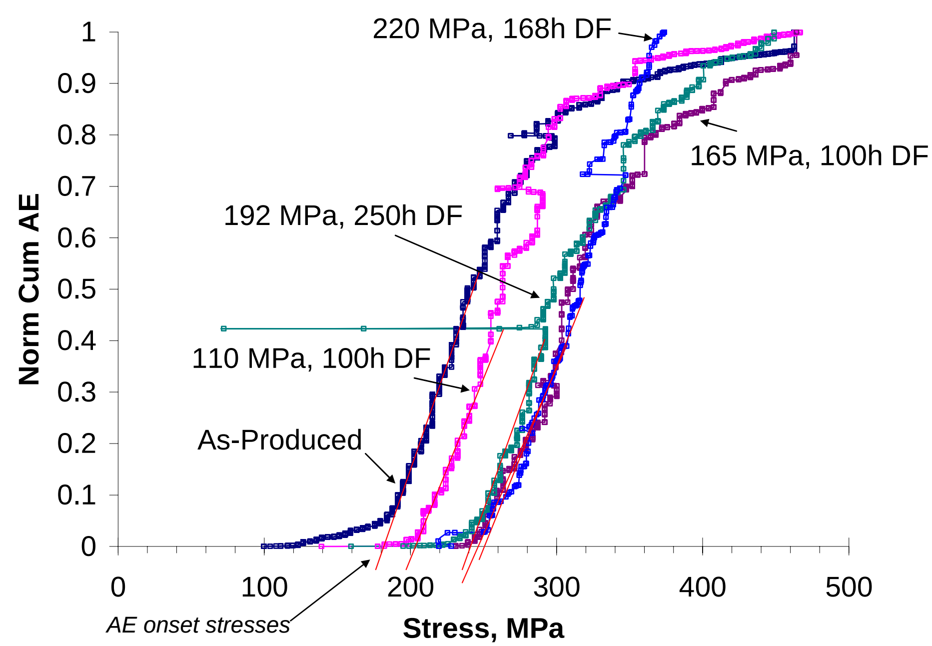
Damage Evolution: RT Retained Properties

With creep/fatigue comes increases in residual compressive stress and increased stress to cause matrix cracking

- For the case of 192 and 220 MPa, matrix cracks formed during fatigue; however, further matrix cracking required much higher stresses than creep or fatigue condition due to matrix relaxation



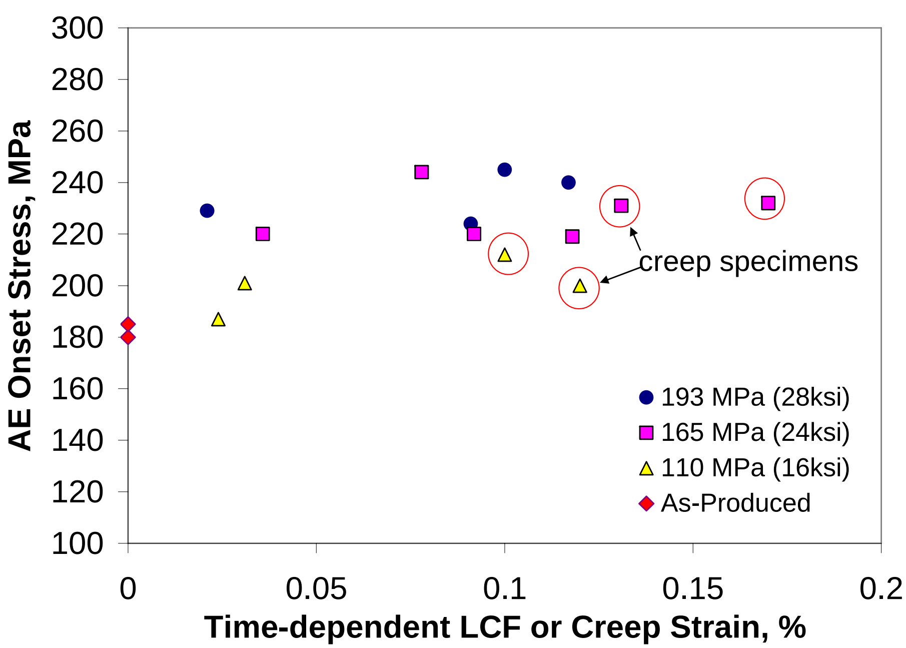
Strain offset for clarity

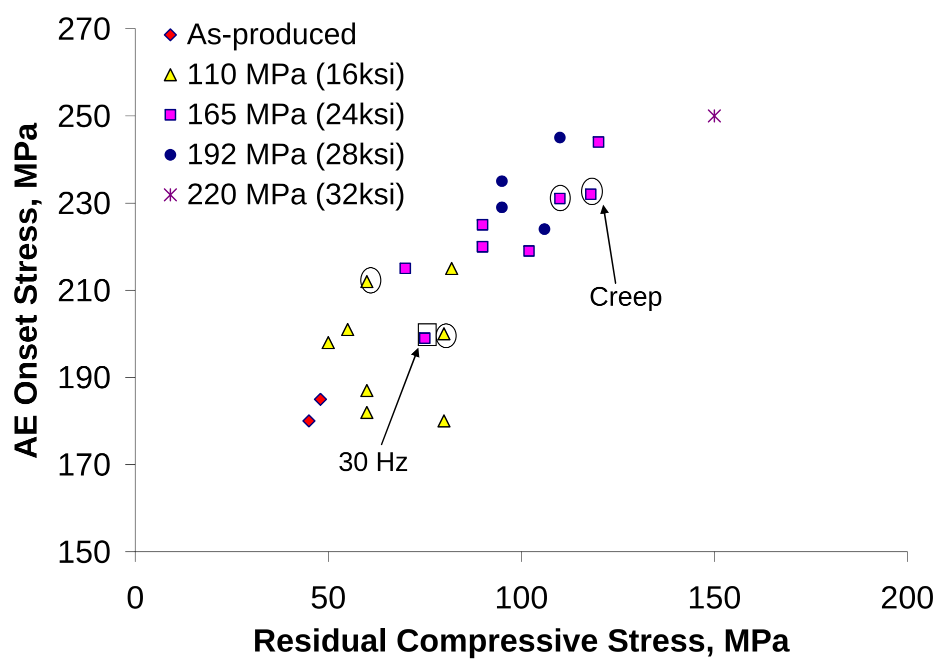


Damage Evolution: RT Retained Properties

Effect of accumulated strain and residual stress on matrix cracking stress

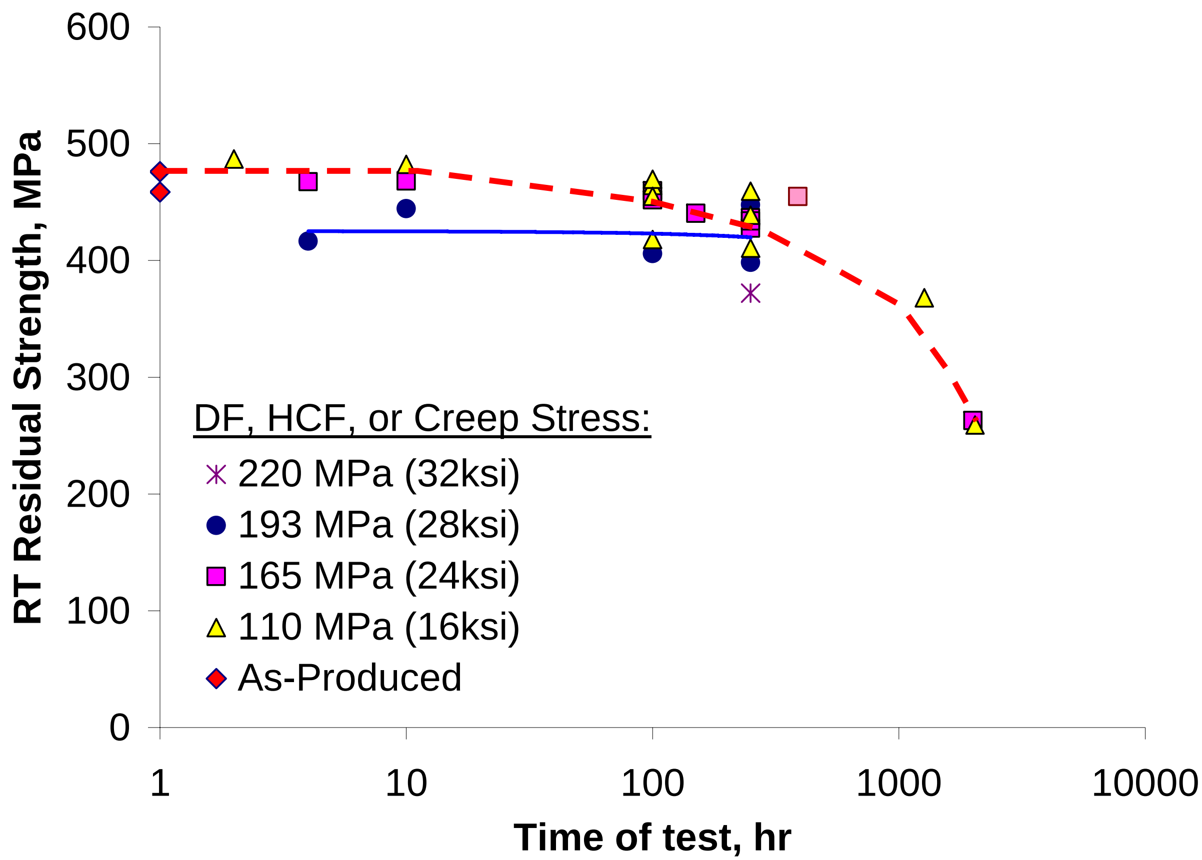
- Matrix cracking stress increases nearly proportionately with increases in residual compressive stress



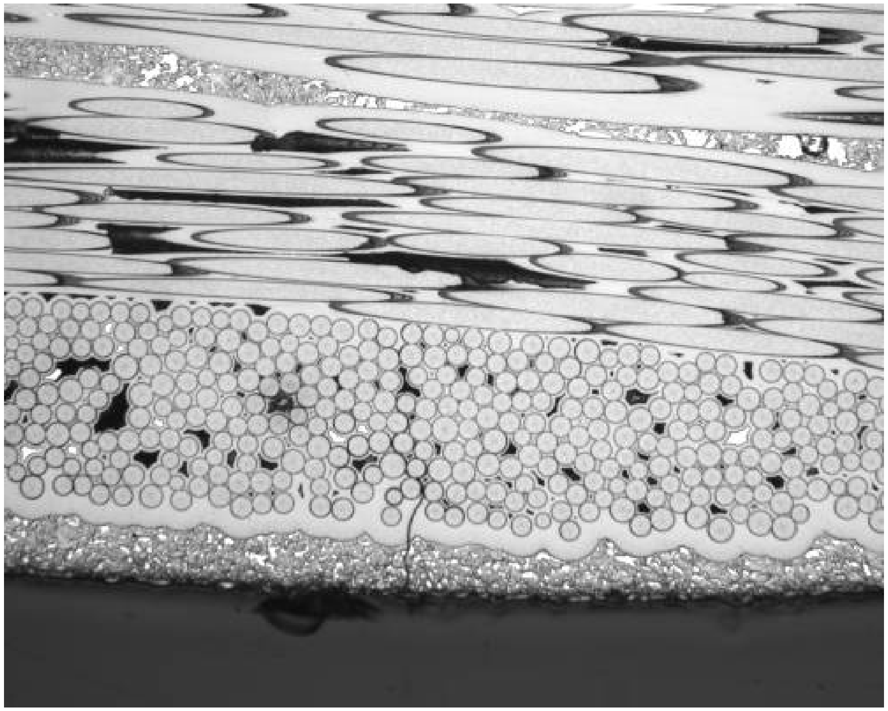


Damage Evolution: RT Retained Properties

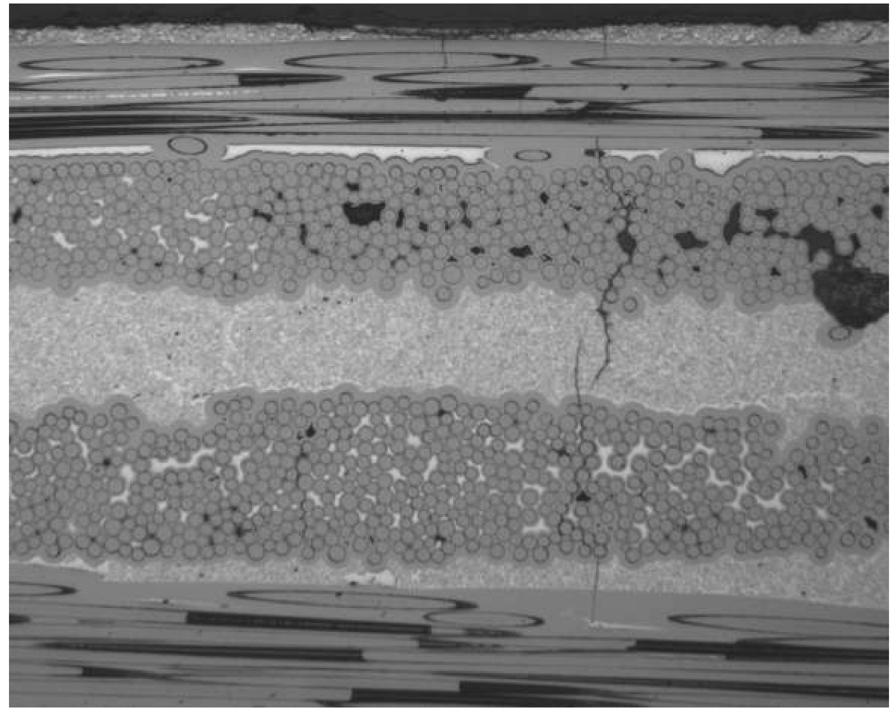
Residual strength with time of test… note significant degradation did not occur until longest time/low stress conditions



Damage Evolution: Optical Microscopy

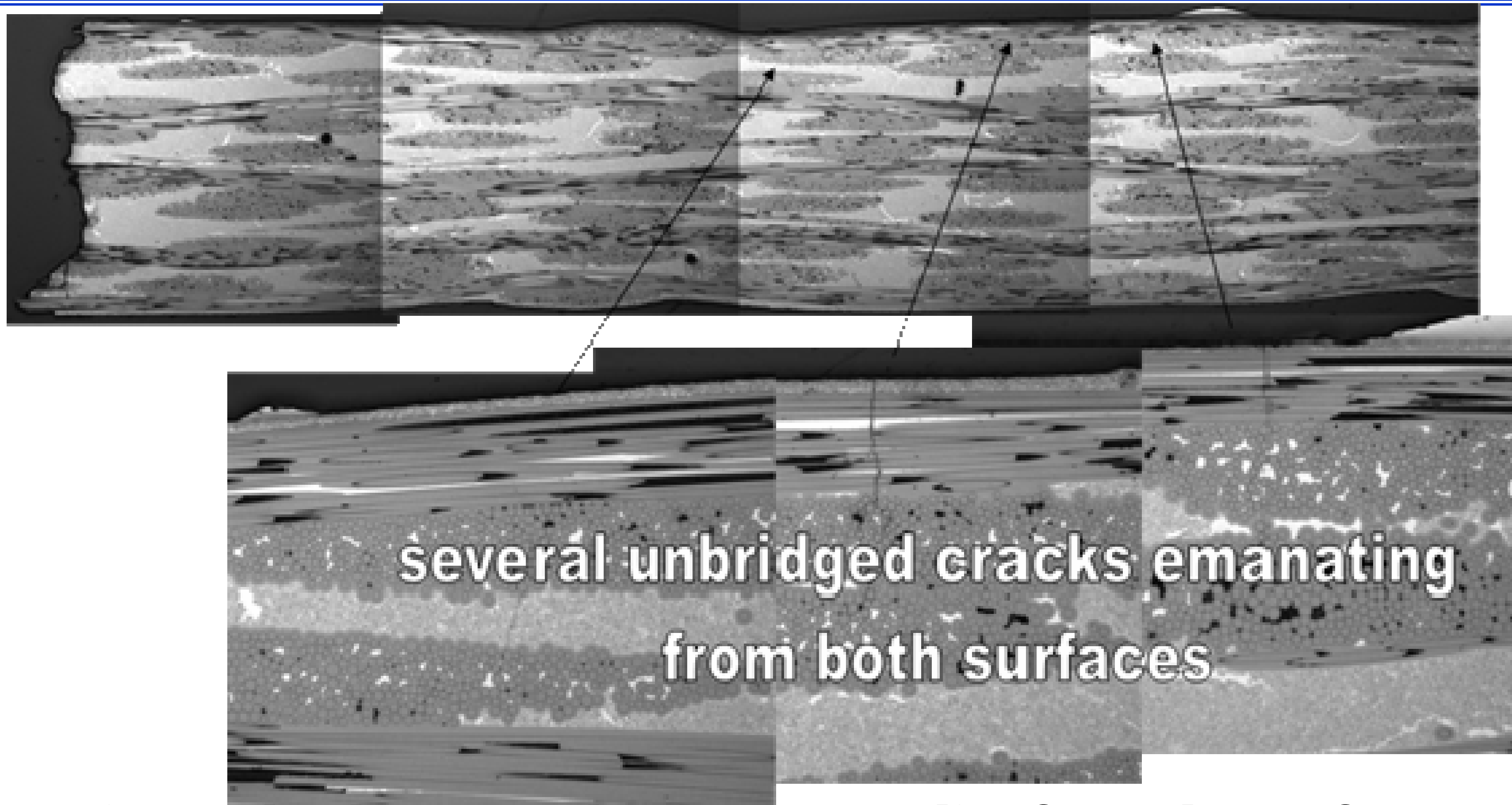


Surface 90 minicomposite (192 MPa, 10 hours, did not fail in rupture)



Back-to-back 90 minicomposite cracks which extended to the surface through a 0 minicomposite (165 MPa; 1508 hr creep rupture)

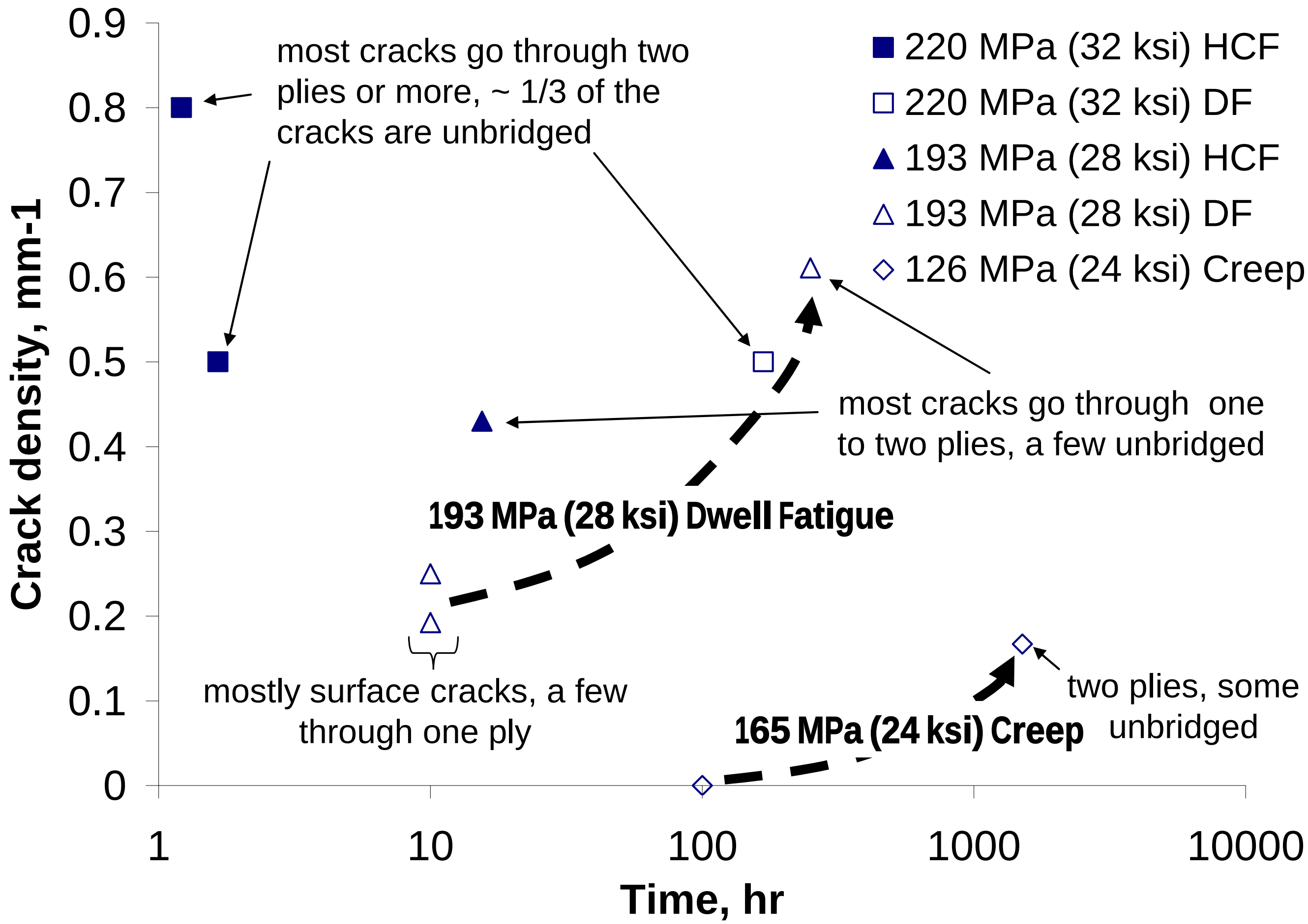
Damage Evolution: Optical Microscopy



Matrix cracks that extend at least two plies from the surface and in some cases have fractured fibers in the matrix crack wake. This specimen had undergone 220 MPa 30 Hz fatigue and lasted approximately 1.2 hours at 1200C.

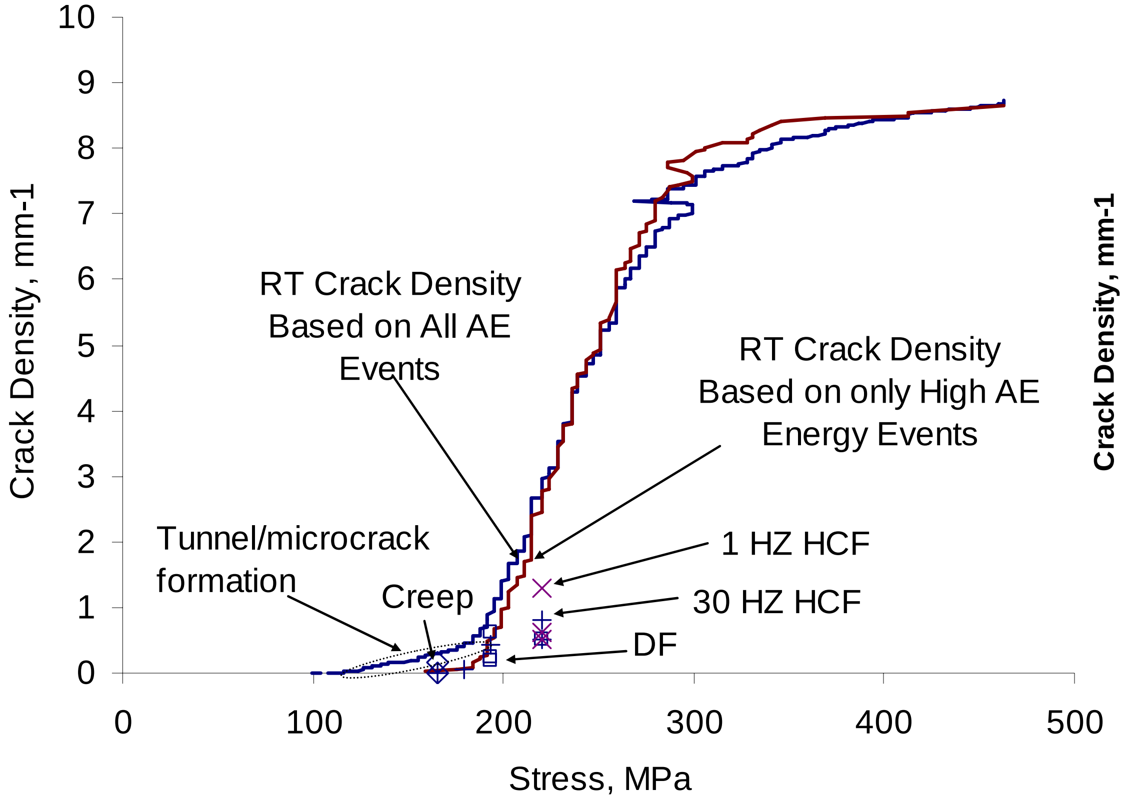
Damage Evolution: Optical Microscopy

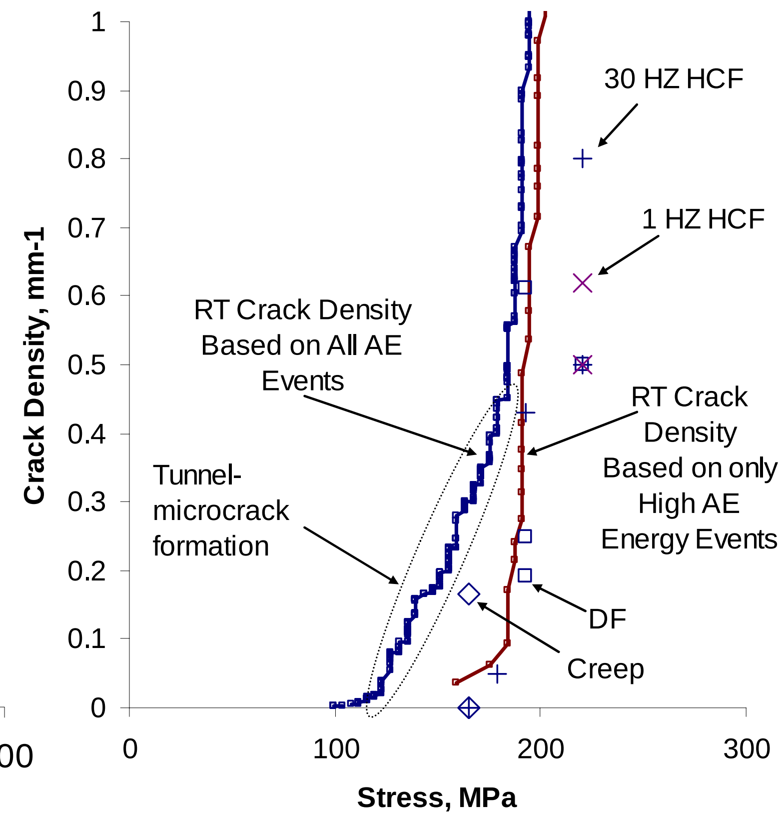
With increasing stress and/or time, fiber-bridged (traverses a 0^o minicomposite) matrix crack density increases



Damage Evolution: Optical Microscopy

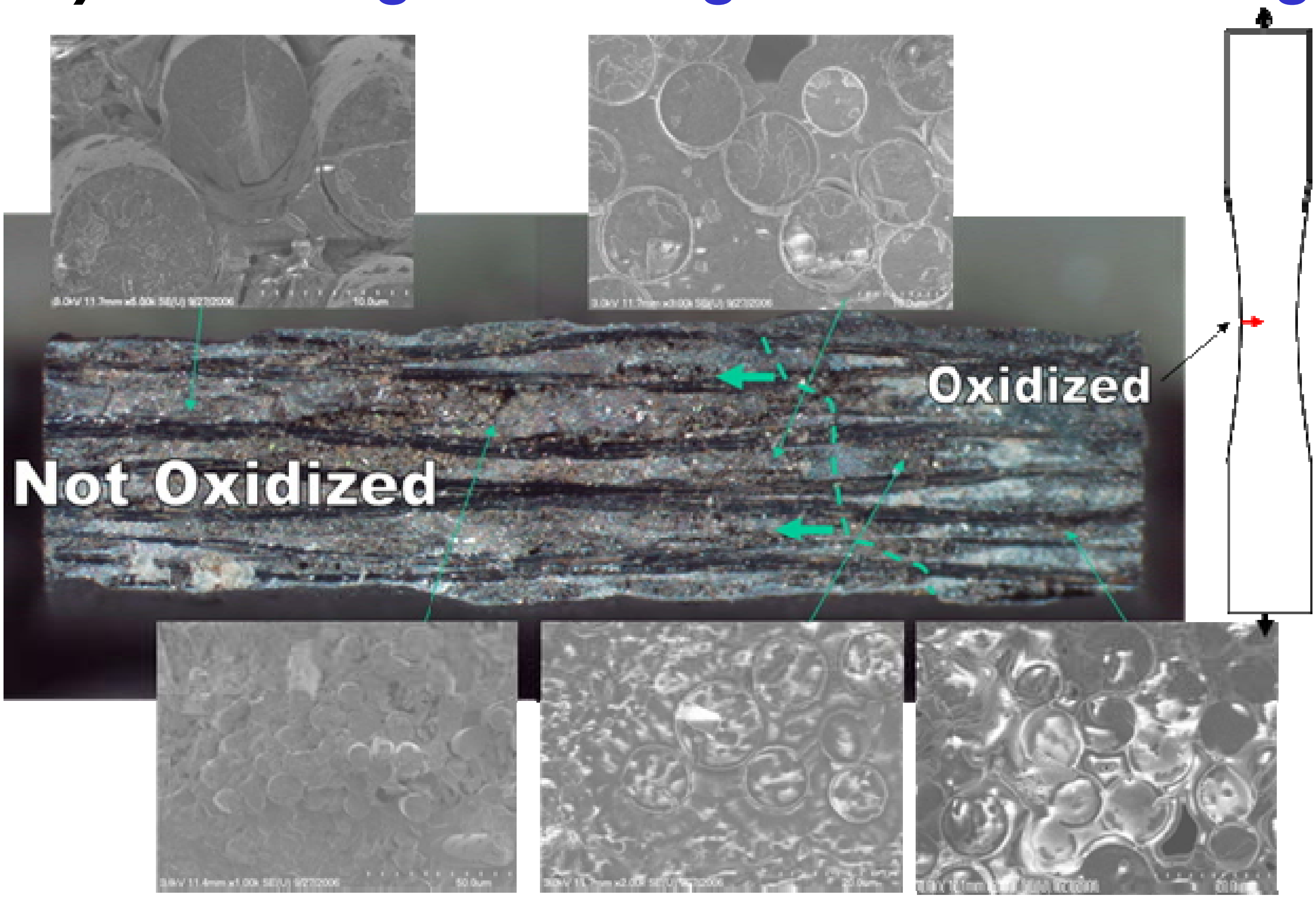
Matrix crack density at 1200ºC has similar dependence on stress as RT; however, cracks are not through-the-cross-section





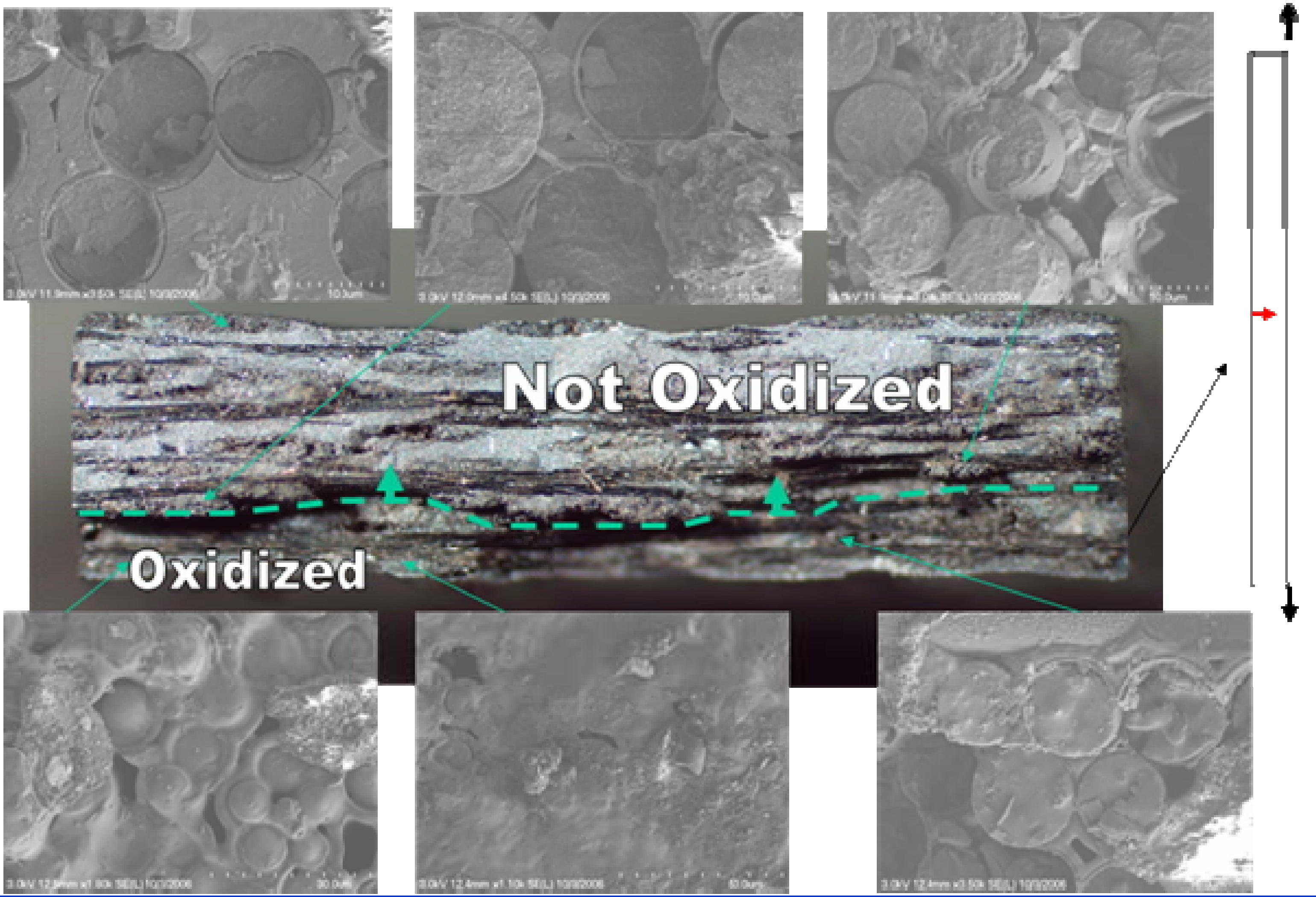
Damage Evolution: Fracture Surface *High Stress*

30 hz fatigue, 179 MPa for 8.1x106 cycles (~ 75 hrs) – unbridged crack growth from an edge



Damage Evolution: Fracture Surface *High Stress*

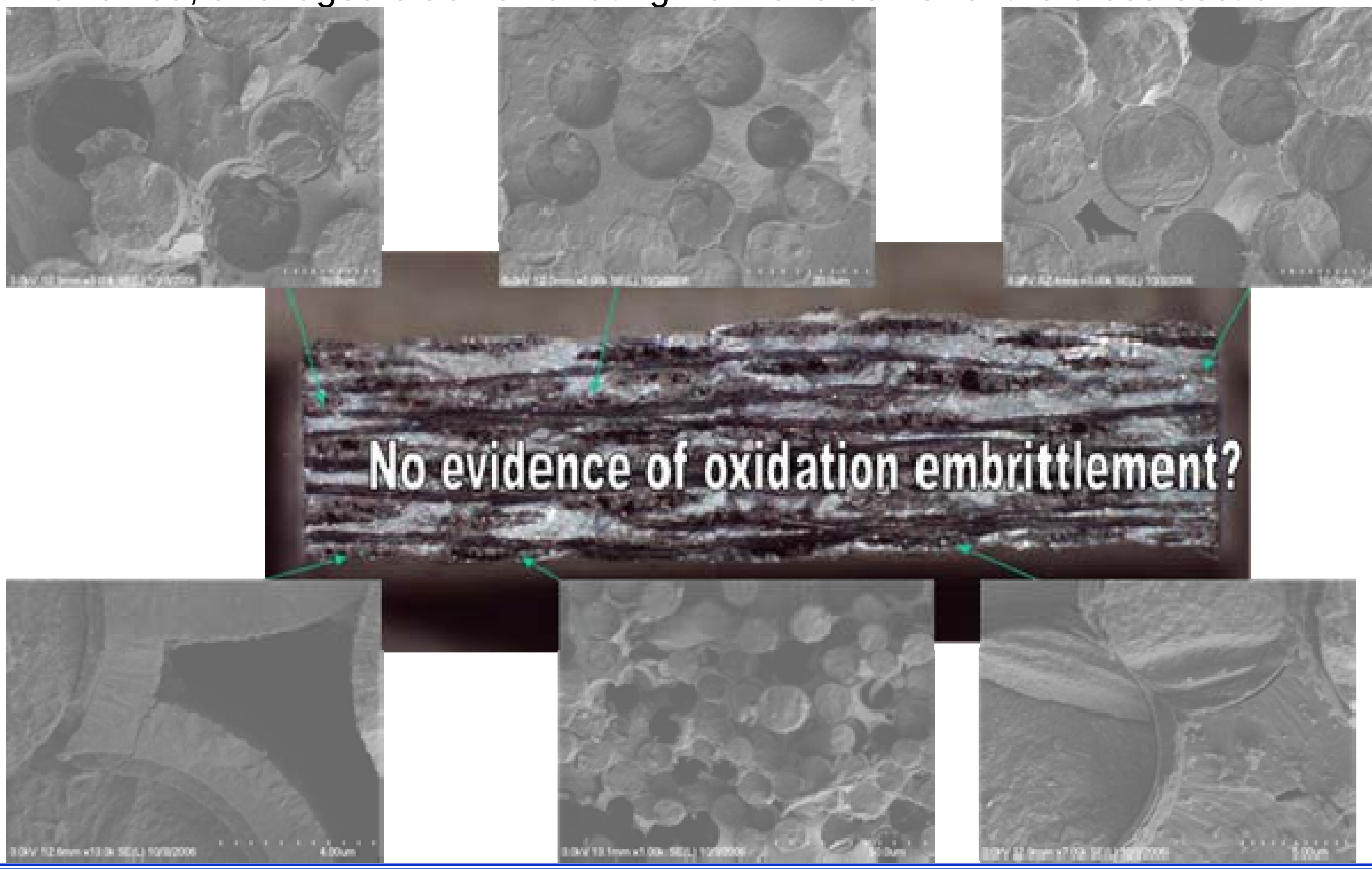
30 hz fatigue, 220 MPa for 178,493 cycles (~ 1.6 hrs) – unbridged crack growth from a face



Damage Evolution: Fracture Surface *Low Stress*

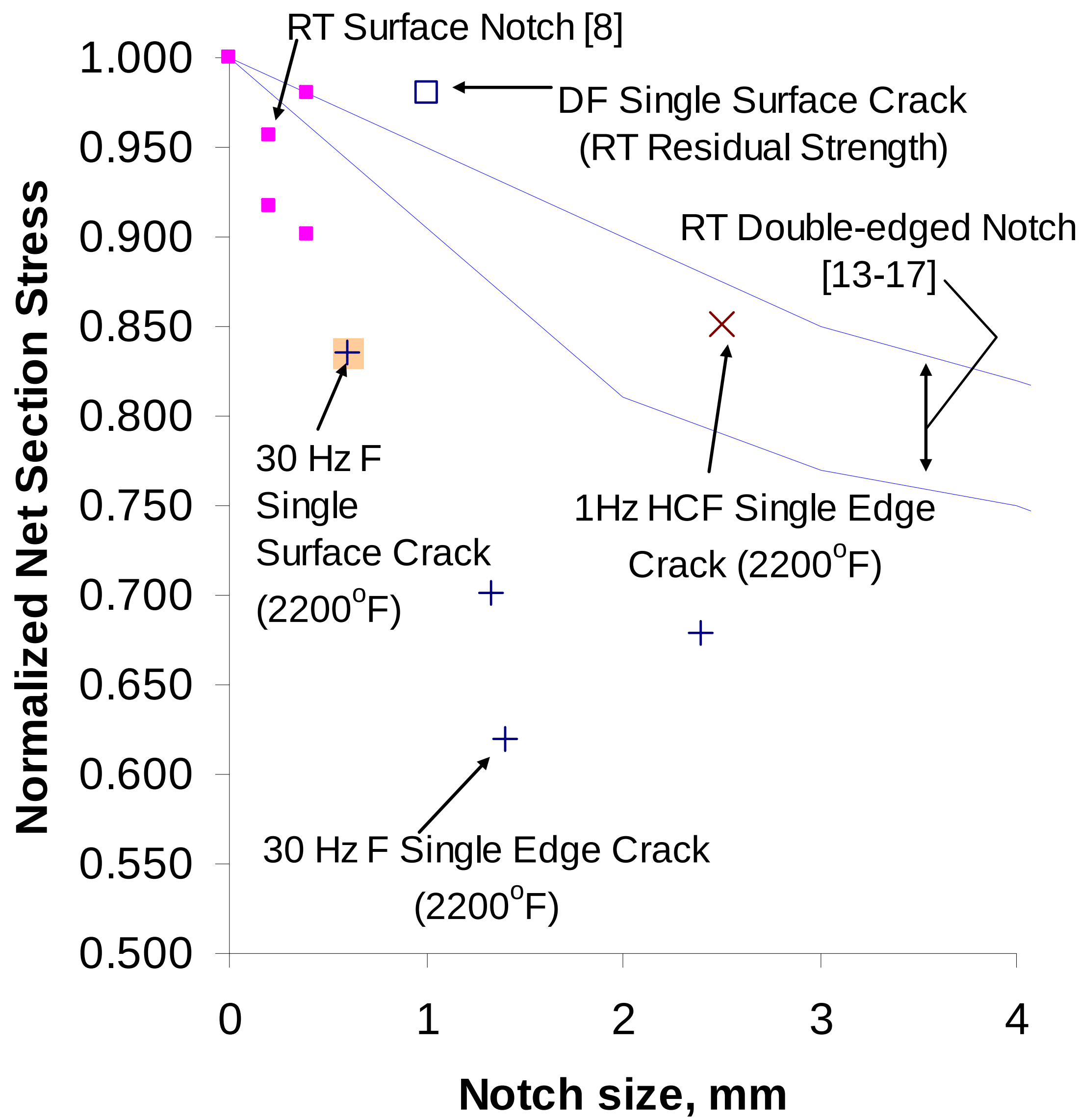
1300-01-006-p02, <u>110 MPa</u> creep for 2036 hours followed by room temperature residual strength test – No evidence of oxidation-assisted fiber failure

- Two specimens that did fail at 110 & 165 MPa showed a small triangular region of oxidized, unbridged crack emanating from one corner of the cross-section



Life Degrading Mechanisms (≥ 179 MPa)

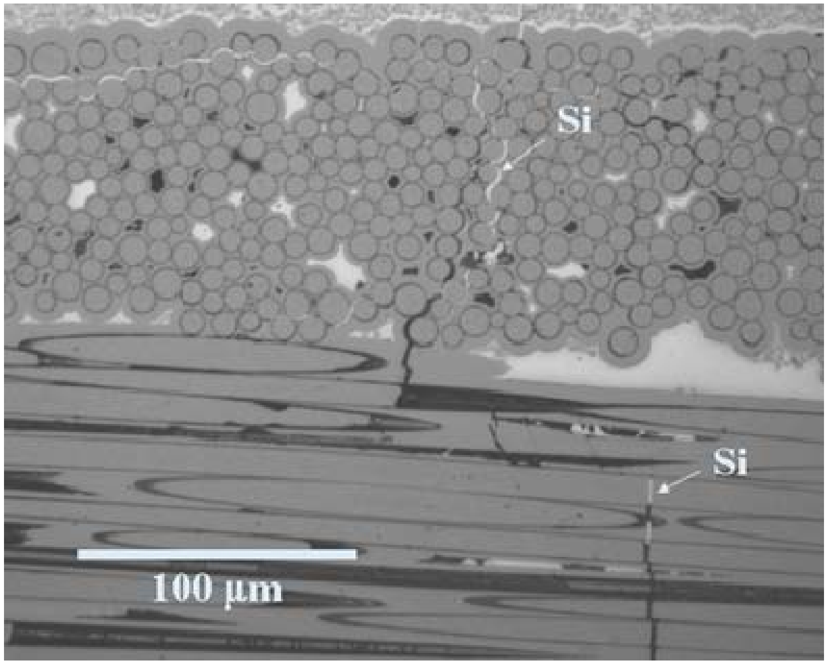
- Effective notch-size effect considering the unbridged crack as a notch



Life Degrading Mechanisms (≤ 165 MPa)

For low stress, long-time conditions, strength-degradation not apparently due to oxidation

- Intrinsic creep-degradation of fibers
- Si attack of fibers from free Si



165 MPa creep specimen that failed after 1500 hours

Conclusions

- Creep at 1204^oC of specimens resulted in matrix relaxation and higher proportional limits
- Matrix crack density at 1204^oC similar to RT → Good basis for starting point of life-model
  - However, matrix cracks were not through-the-cross-section
- ***Degradation Mechanism: $\geq$ 179 MPa*** (~ M.C. Stress), stressed-oxidation induced unbridged crack growth led to failure
- ***Degradation Mechanism $\leq$ 165 MPa***, reduction in retained strength at RT not usually due to oxidation → fiber-degradation due to intrinsic creep-degradation or Si attack
  - Note, two specimens that did fail showed unbridged crack at a corner of the cross-section