An Investigation On The Use Of A Laser Ablation Treatment On Metallic Surfaces And The Influence Of Temperature On Fracture Toughness Of Hybrid Co-cured Metal-pmc Interfaces

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Presentation Outline

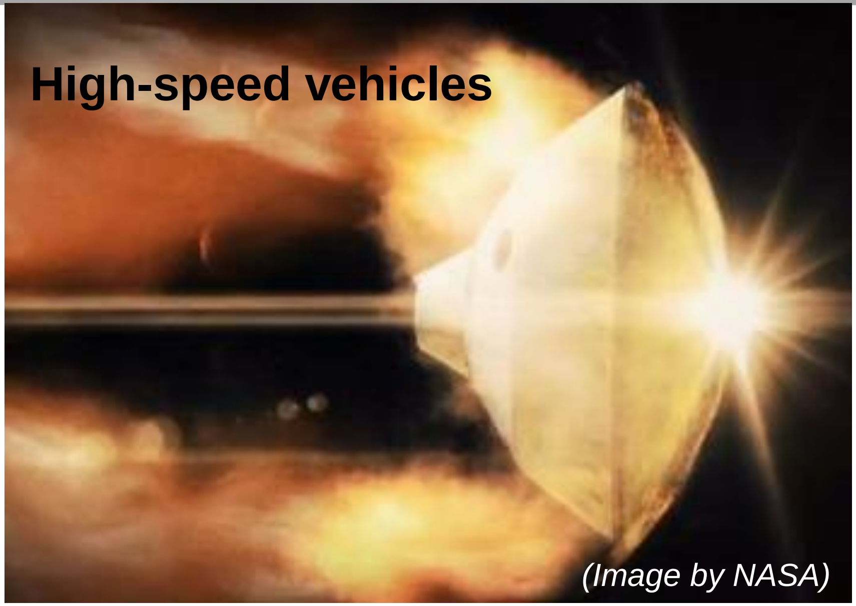
- Motivation
- Objectives
- Processing and Experimental Approach
  - Surface Treatment
  - Hybrid Laminate Fabrication
  - Fracture Toughness Tests with In-situ Digital Image Correlation (DIC)
- Results
- On-going Work and Conclusions

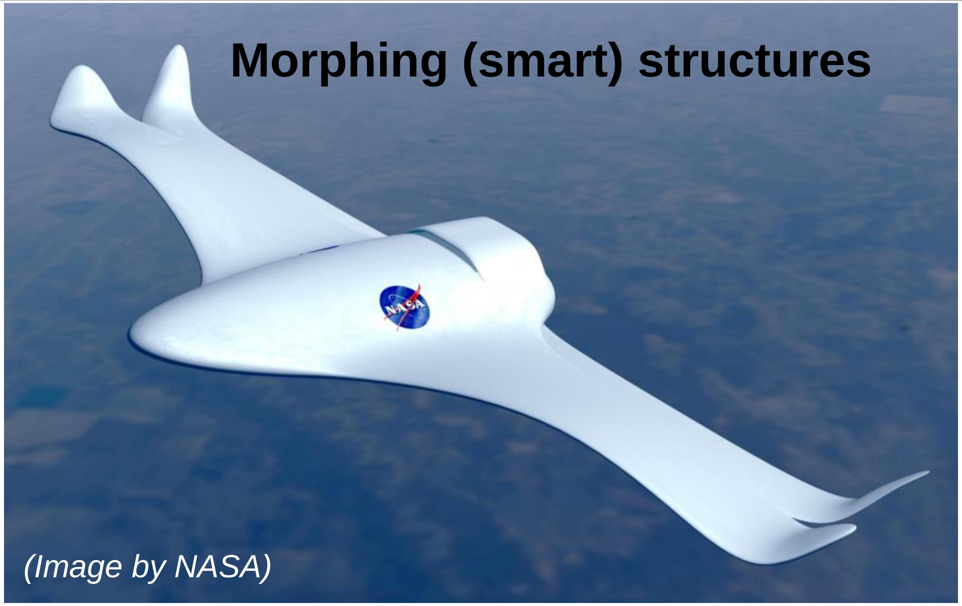
Objective and Approach

Objective: Investigate the effect of laser surface treatment and surface chemistry on the performance of Ti/polyimide hybrid laminate interfacial properties at elevated temperatures.

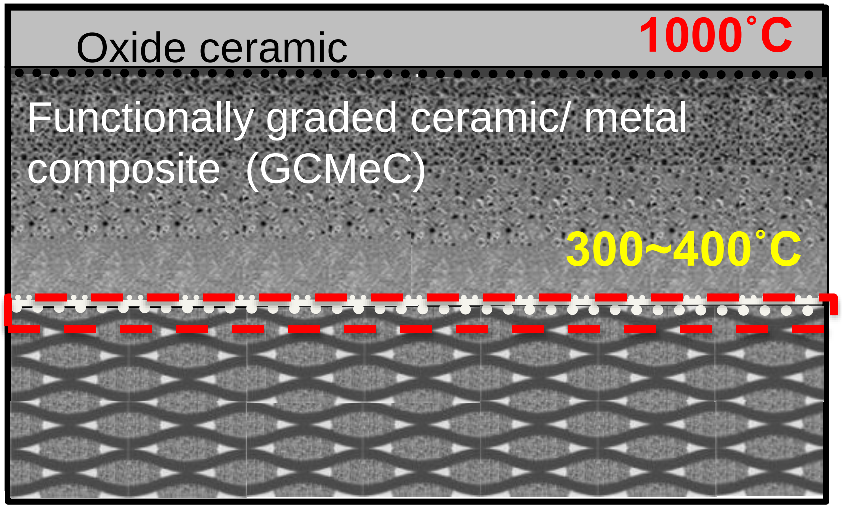
Approach: Fabricate double cantilever beam (DCB) and end notched flexure (ENF) specimens and test at elevated temperatures.

Motivation



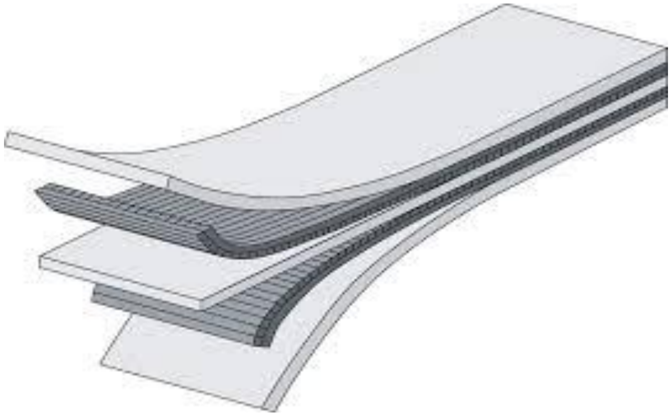


Hypersonic flight: frictional heating



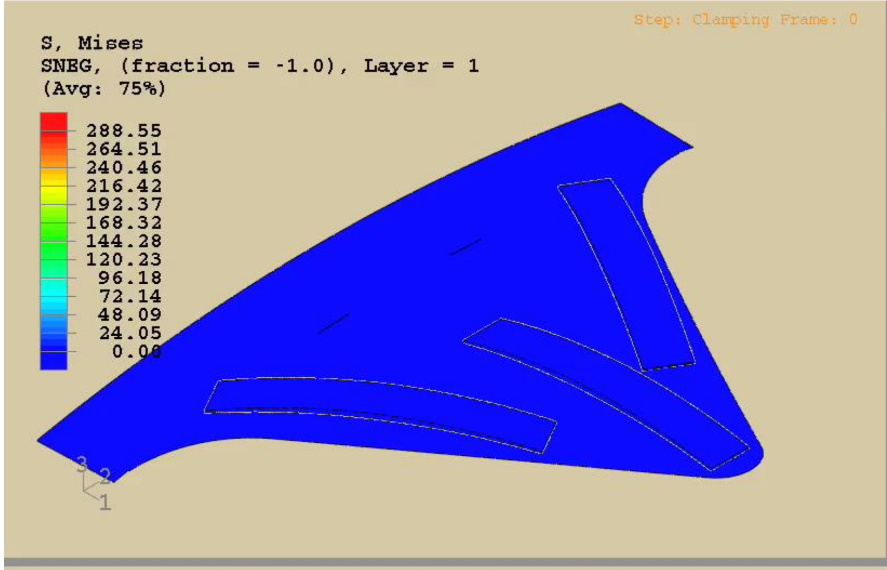
(Courtesy of AFOSR-MURI18 -2009)

Fiber metal (hybrid) laminates

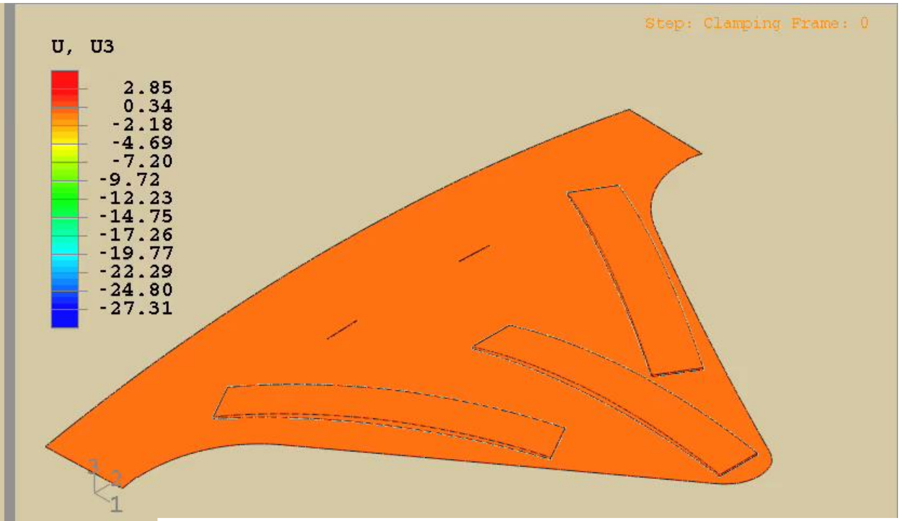


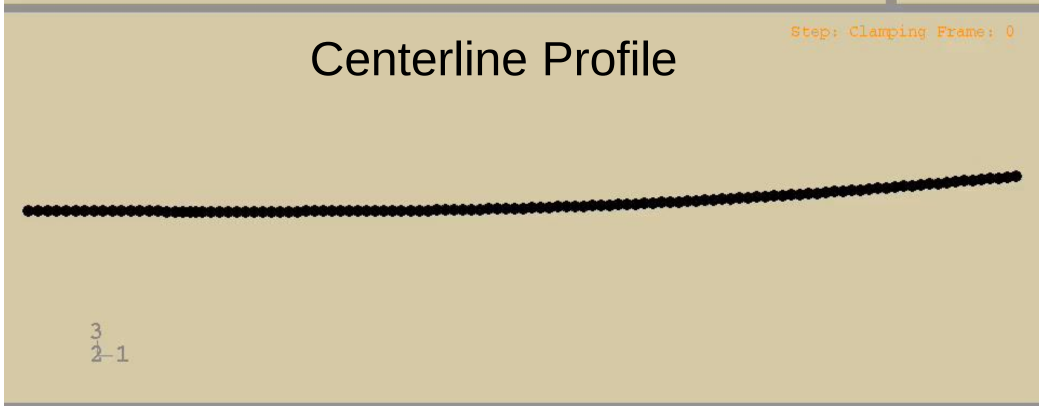
Thermally Actuated Variable Geometry Chevron (VGC)

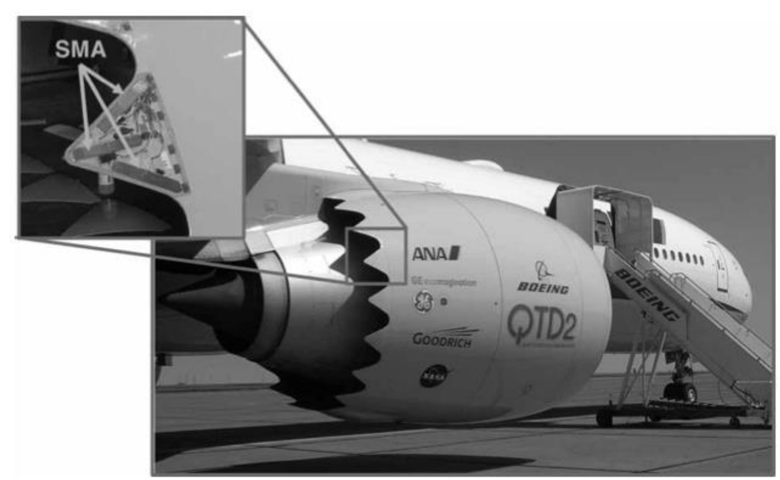
Stress (VM) Contours



Deflection Contours





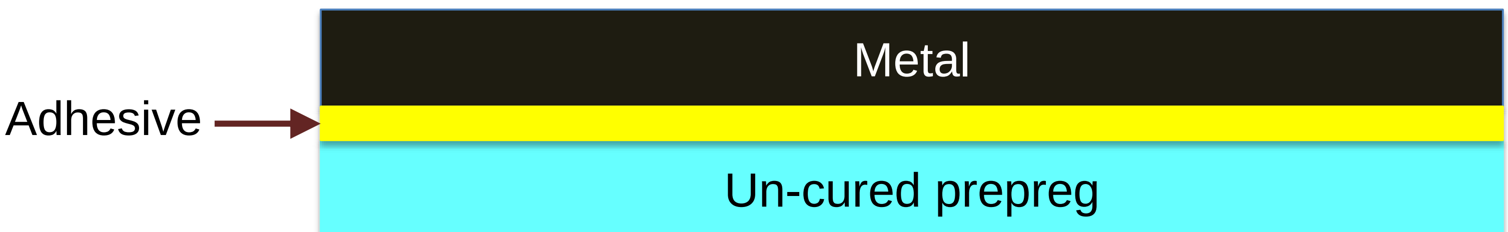


Example of smart material on morphing structures: adhesively bonded SMA beams on Boeing variable geometry chevron (VGC) *(Hartl et al., 2010)*

Composite Bonding Classifications

- Secondary bonding

- Co-bonding



- Co-curing

➡ Metal-composite interfaces in this work will be created by **co-curing**.

Criteria for Success

- Develop strong **hybrid interfaces** between metal/alloy and polymer matrix composites (PMC) in hybrid laminates for high temperature (200-300 ^{o}C) applications
  - Surface chemistry and topography
  - Reinforcement architecture
  - Damage initiation and delamination
  - Thermal degradation
- Experimentally and computationally investigate the "**fracture toughness**" as a function of temperature and interfacial architecture

Hybrid Laminates

Materials:

- Reinforcement
  - Plain weave T300 carbon fabric
  - ***8-HS T650 carbon fabric***
- Matrix
  - Epikote-Epikure 04908 (T_g = 82°C)
  - ***AFR-PE-4*** (T_g = 390°C)
- Metallic foil
  - **Ni/Ti (127 µm thick)**
  - **Ti (127 µm thick)**
  - Al (400 µm thick)

Metallic surface treatment:

- Sanding
- Acid etching
- ***Laser ablation***
- ***Sol-gel***
- Chromic anodization and primer

Layup: $[0^o/90^o]_2$/Metal/$_{\text{pre-crack}}$/$[90^o/0^o]_2$

Current work: Layup and Sol-Gel Chemistry

Layup: $[0^o/90^o]_2$/Ti/$[90^o/0^o]_2$ (0^o: parallel to warp direction, 90^o: parallel to fill direction)

PMC:

- Reinforcement: T650 carbon fiber, 8-harness satin weave
- Matrix: AFR-PE-4 polyimide resin

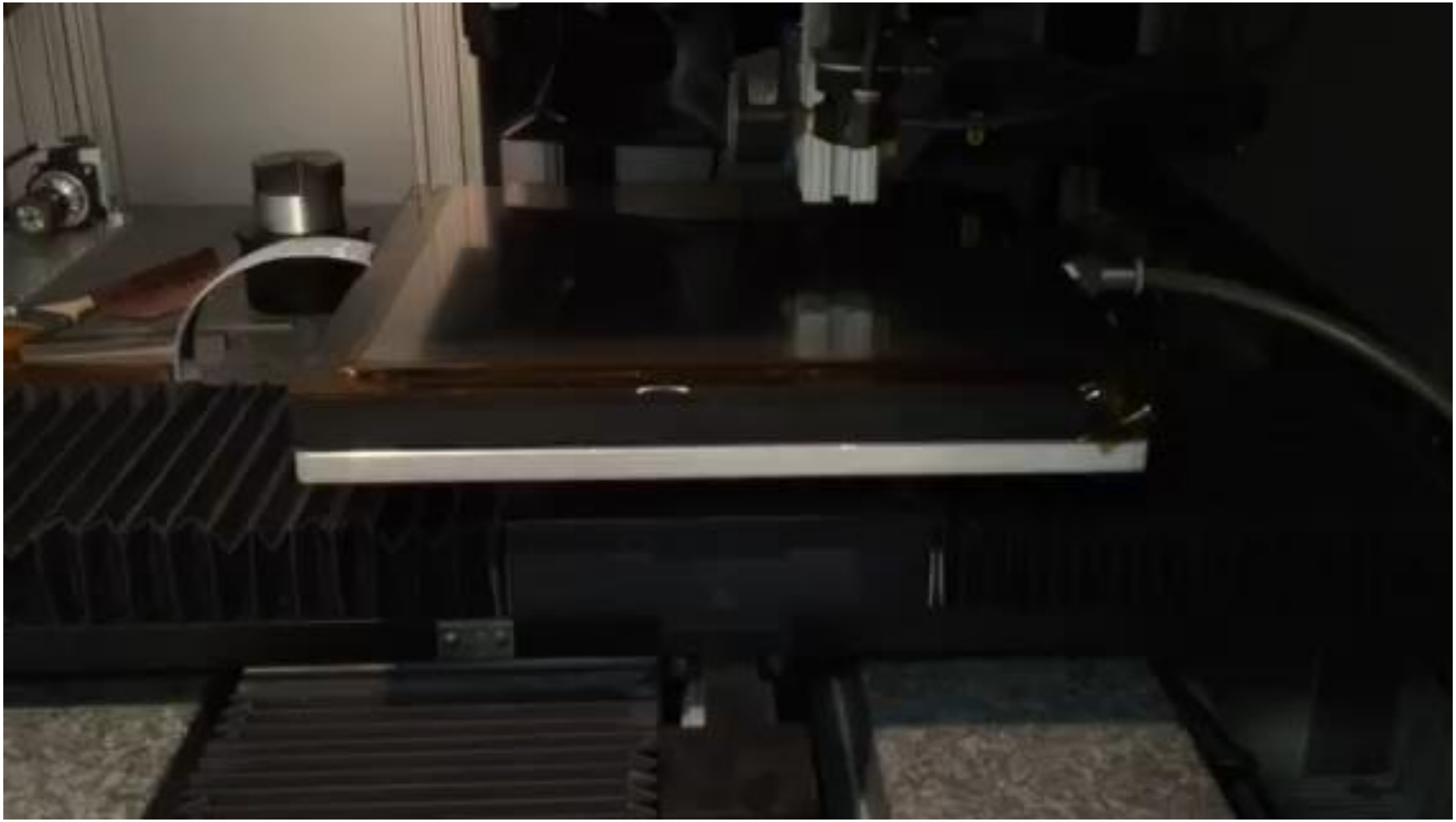
(AFR-PE-4-T650-35-3K-DEZ-8HS-50 from Renegade Materials Corp.)

Ti foil treated with laser ablation and different sol-gel chemistries

- LaRC sol-gel: mixture of pendent phenylethynyl imide oligomeric di(tetramethoxysilane) and tetraethoxysilane

- Amino phenyl sol-gel: mixture of zirconium n-propoxide, acetic acid and aminophenyltrimethoxysilane
- EPII sol-gel: mixture of zirconium n-propoxide, acetic acid and glycidoxypropyltrimethoxysilane

Laser Ablation of Ti and Ni/Ti foils



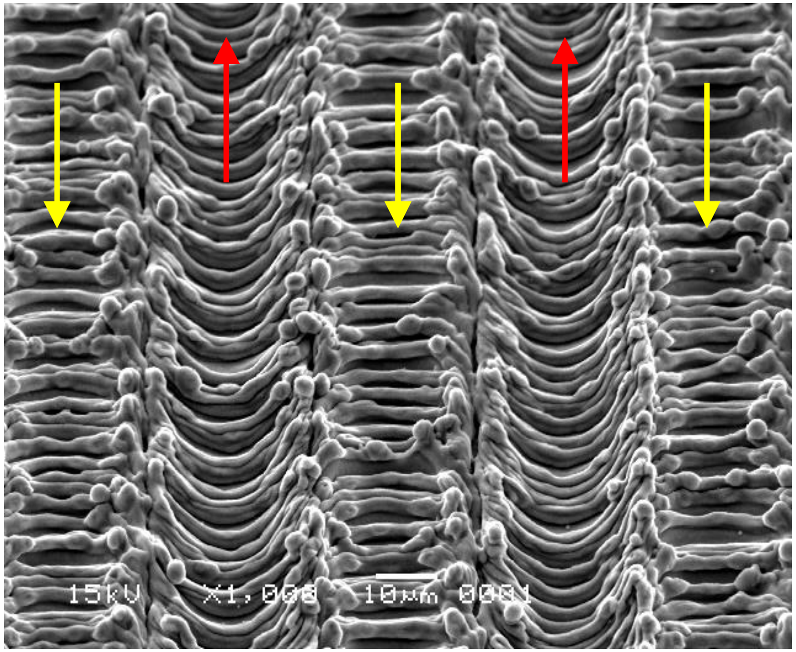
Selected laser ablation parameters: 80 kHz, 1.5W, 1 mil pitch

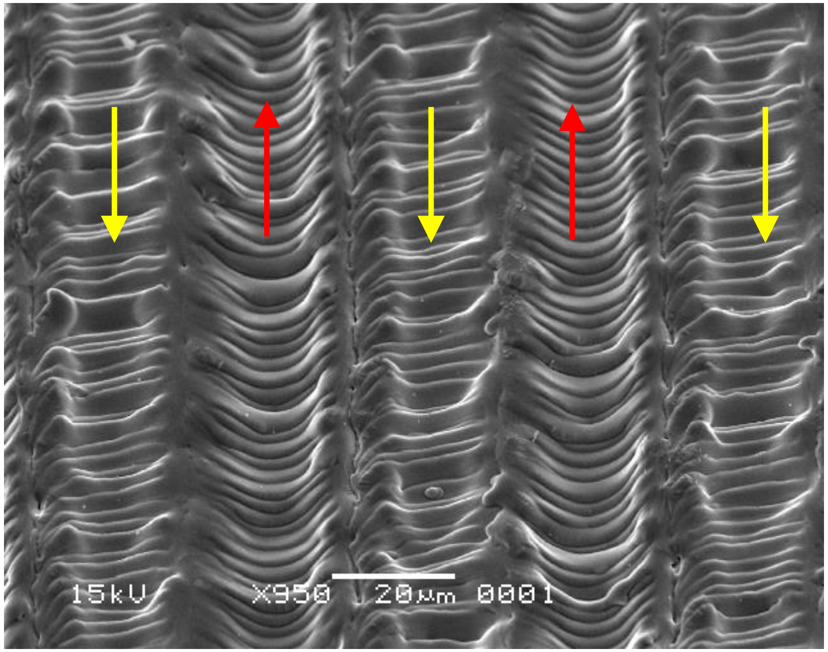
Surface of Ti and Ni/Ti Foil After Laser Ablation

Laser ablated Ti surface

Laser ablated Ni/Ti surface

Arrows denote laser path direction



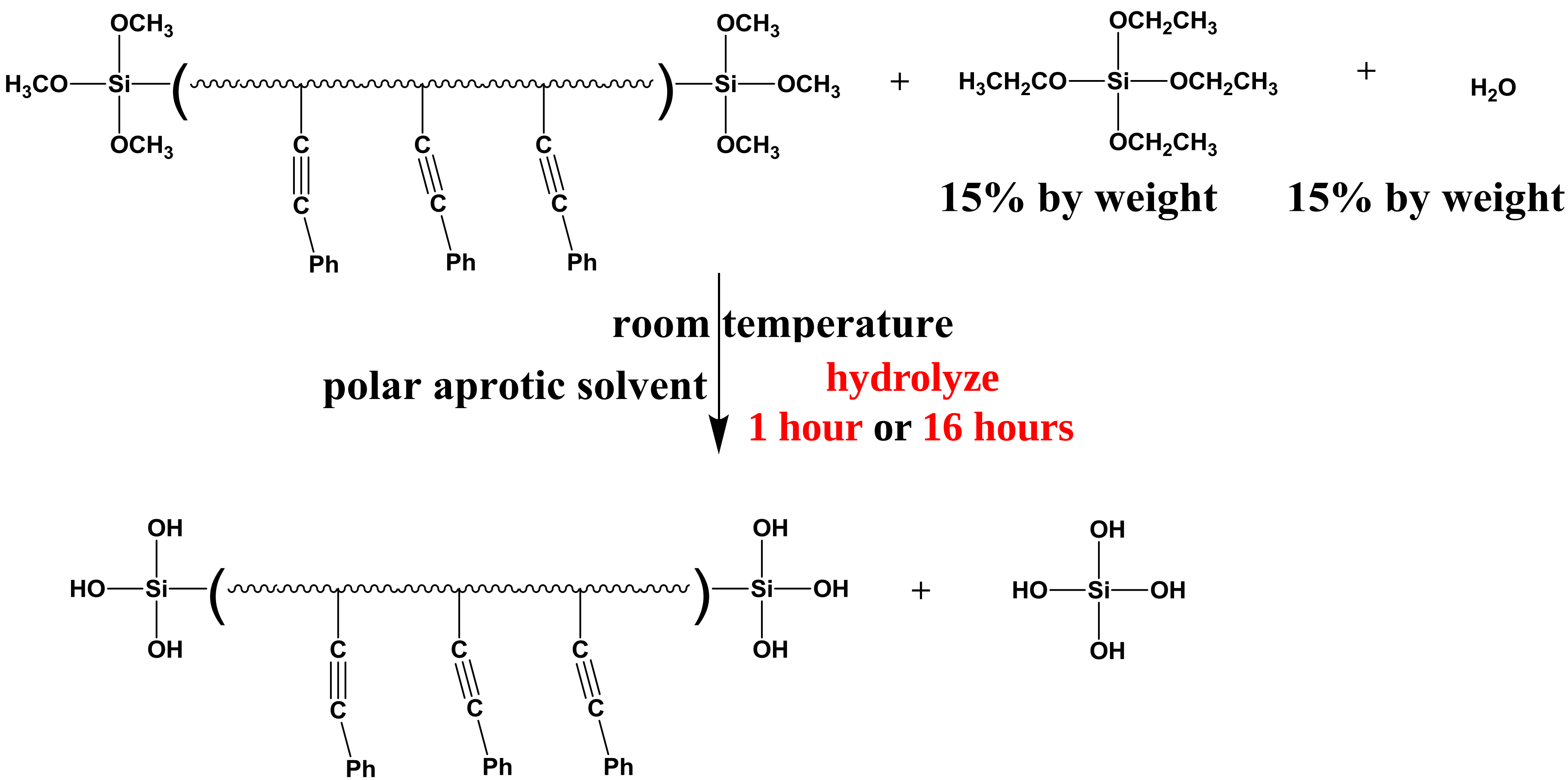


Laser ablation performed on Ti and Ni/Ti surfaces:

- Clean oxide layer on the metal surfaces was formed
- Created patterned micro-roughness on the metal surface
- Increased surface wettability
- No detrimental morphology or chemical species was formed

Frank L. Palmieri, Kent A. Watson, Guillermo Morales, Thomas Williams, Robert Hicks, Christopher J. Wohl, John W. Hopkins, and John W. Connell, "Laser Ablative Surface Treatment for Enhanced Bonding of Ti-6Al-4V Alloy", ACS Applied Materials and Interfaces, 5(4), 1254-1261 (2013).

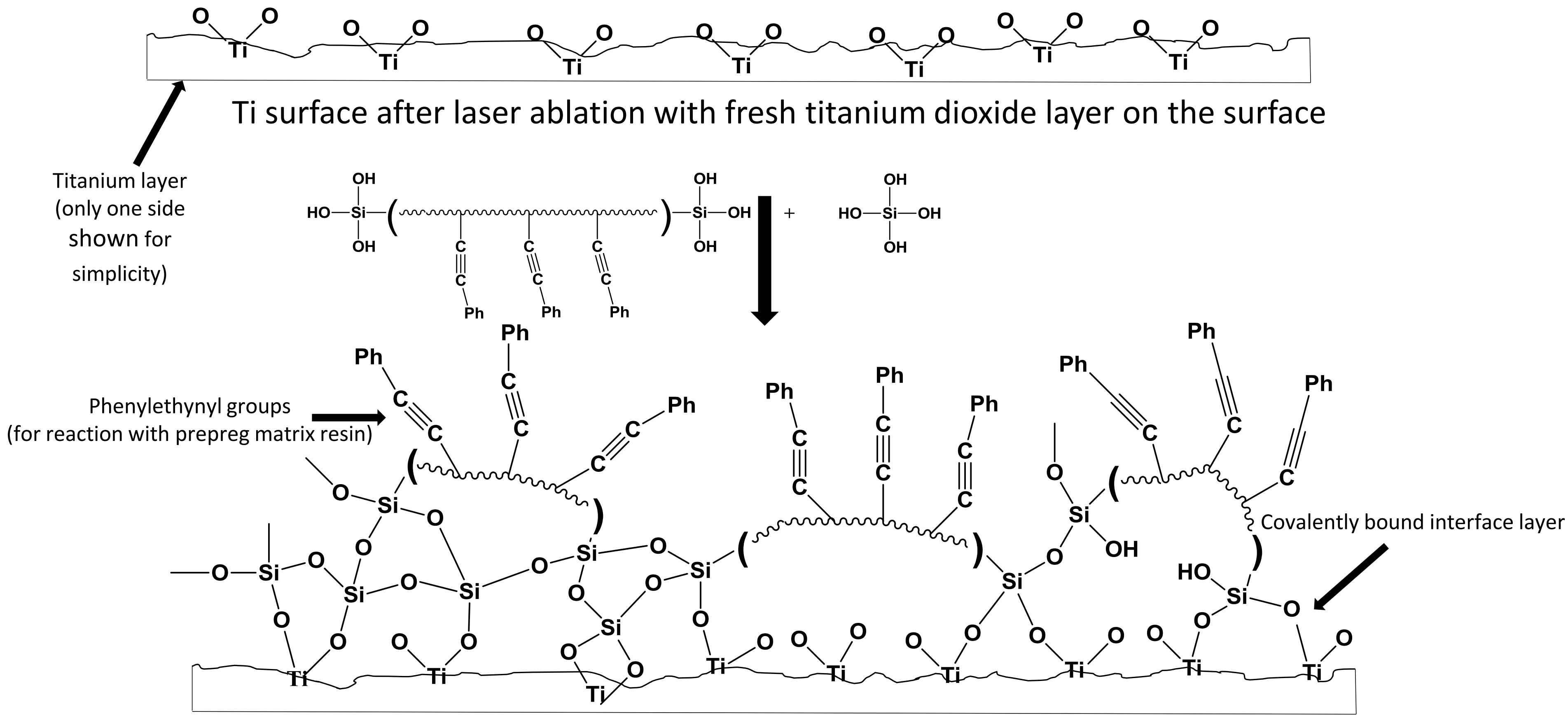
Synthesis of Amide Acid Sol-Gel Surface Treatment



surface treatment solution

Park, C., Lowther, S.E., Smith, J.G., Connell, J.W., Hergenrother, P.M., and St. Clair, T.L.; "Polyimide-Silica Hybrids Using Novel Phenylethynyl Imide Silanes as Coupling Agents for Surface-Treated Alloy" International Journal of Adhesion and Adhesives, 20, 457-465 (2000).

Representation of Ti Surface Treatment Chemistry

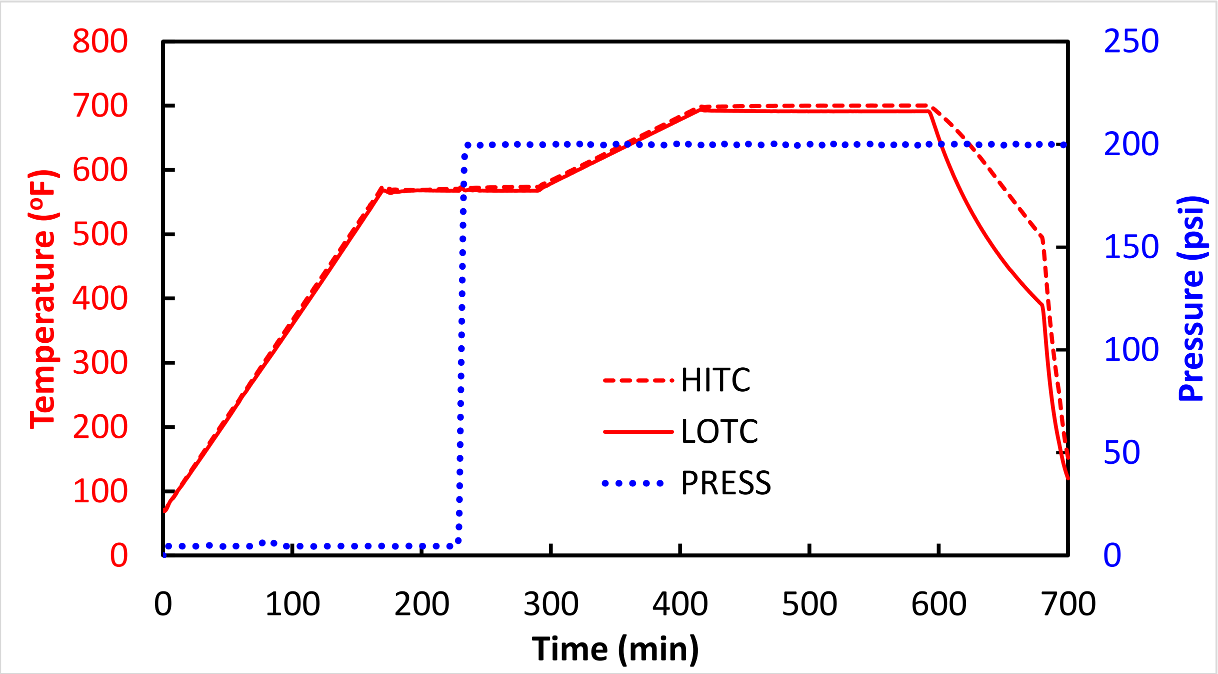


Frank L. Palmieri, Kent A. Watson, Guillermo Morales, Thomas Williams, Robert Hicks, Christopher J. Wohl, John W. Hopkins, and John W. Connell, "Laser Ablative Surface Treatment for Enhanced Bonding of Ti-6Al-4V Alloy", ACS Applied Materials and Interfaces, 5(4), 1254-1261, 2013.

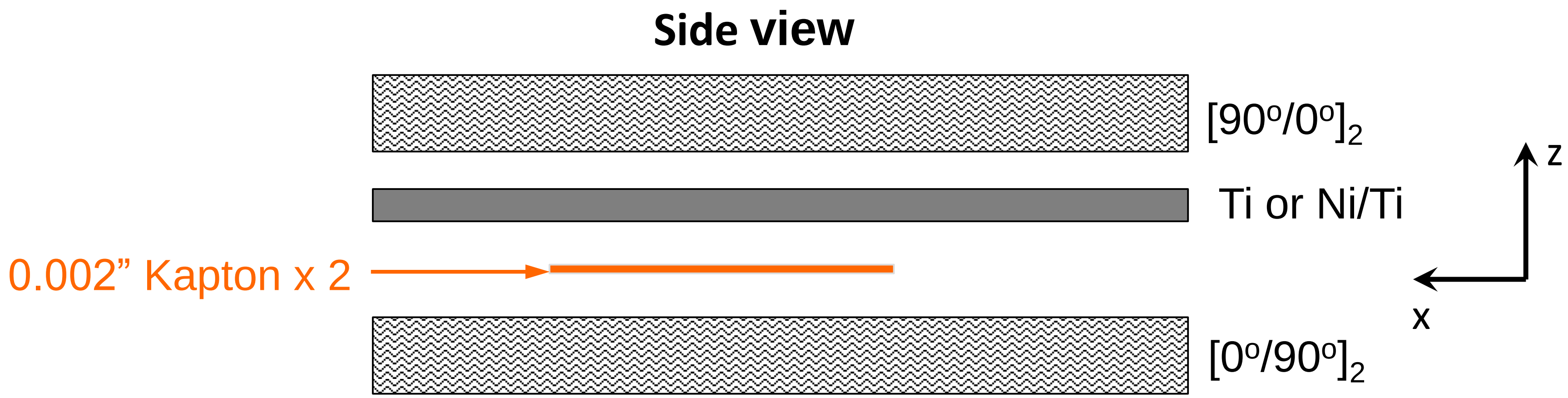
Fabrication Procedure

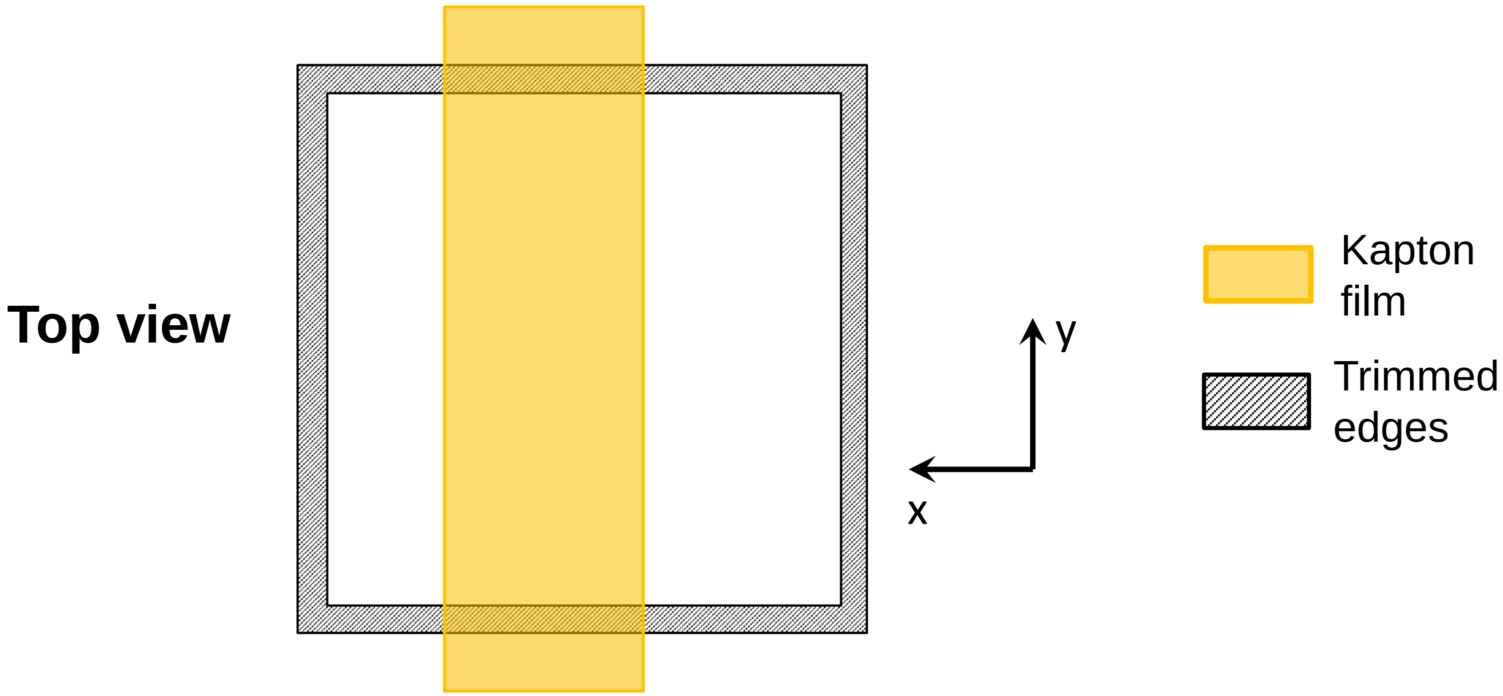
- Ti and Ni/Ti foil (127 μm thick) was laser ablated on both sides
- After hydrolysis of the sol-gel solution (either **1 hour** or **16 hours**), the solution was sprayed on both sides of the laser ablated foil, dried at RT in air for 1 hour, then cured at 110°C and 220°C each for 30 min
- Panel preform was vacuum-bagged over night
- Curing was done in a Wabash vacuum press (12 hours run)

T650/AFR-PE-4 curing cycle



Hybrid Panel Schematic

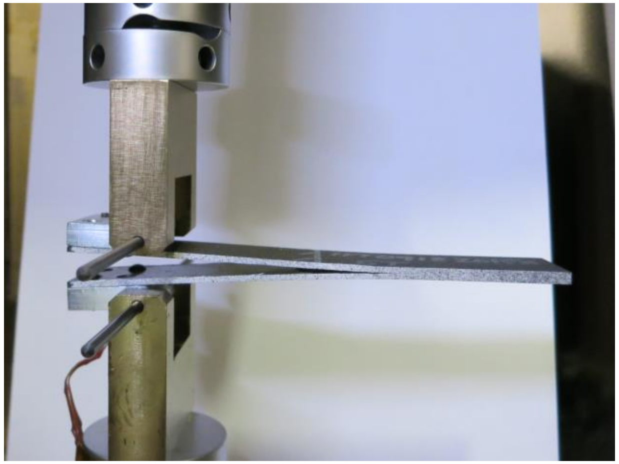




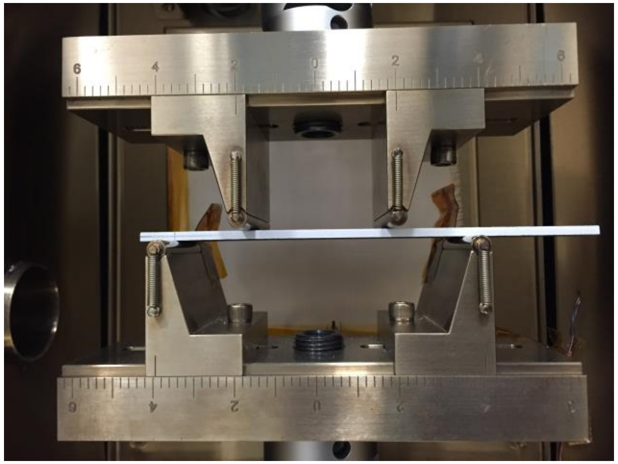
Characterization

Mode I and Mode II fracture toughness tests

DCB Test



4-ENF Test



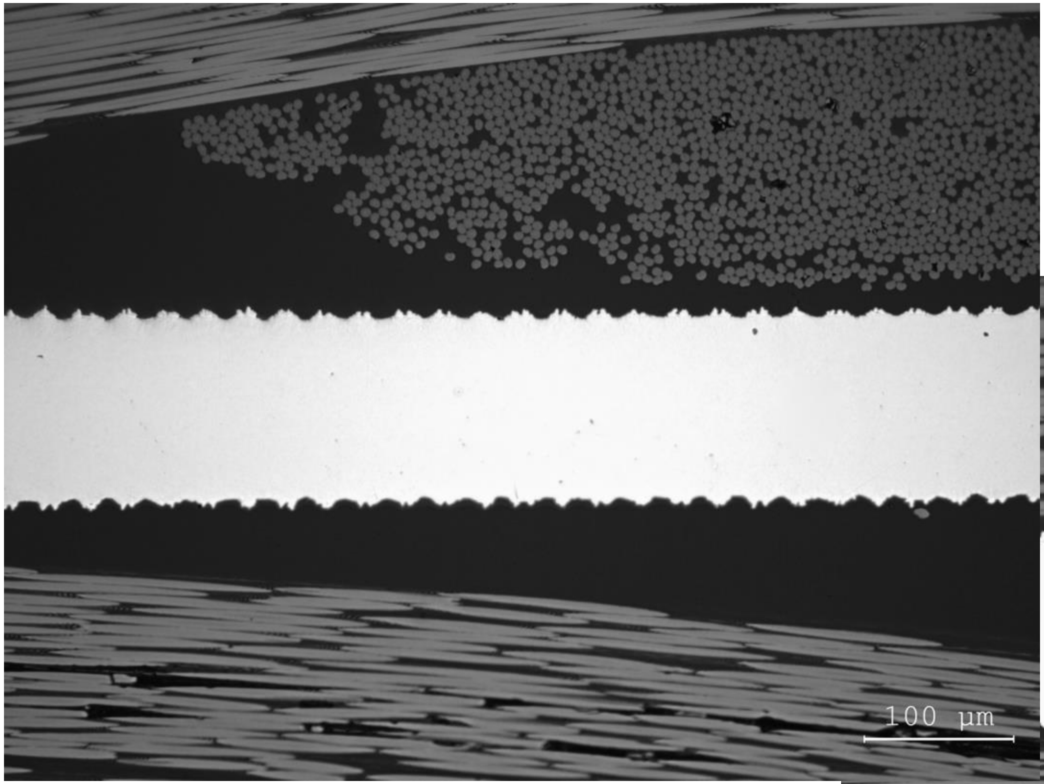
Fracture toughness tests carried out at RT, 150 ^{o}C, 250 ^{o}C and 315 ^{o}C

Monitor delamination growth with ultrasonic scanning

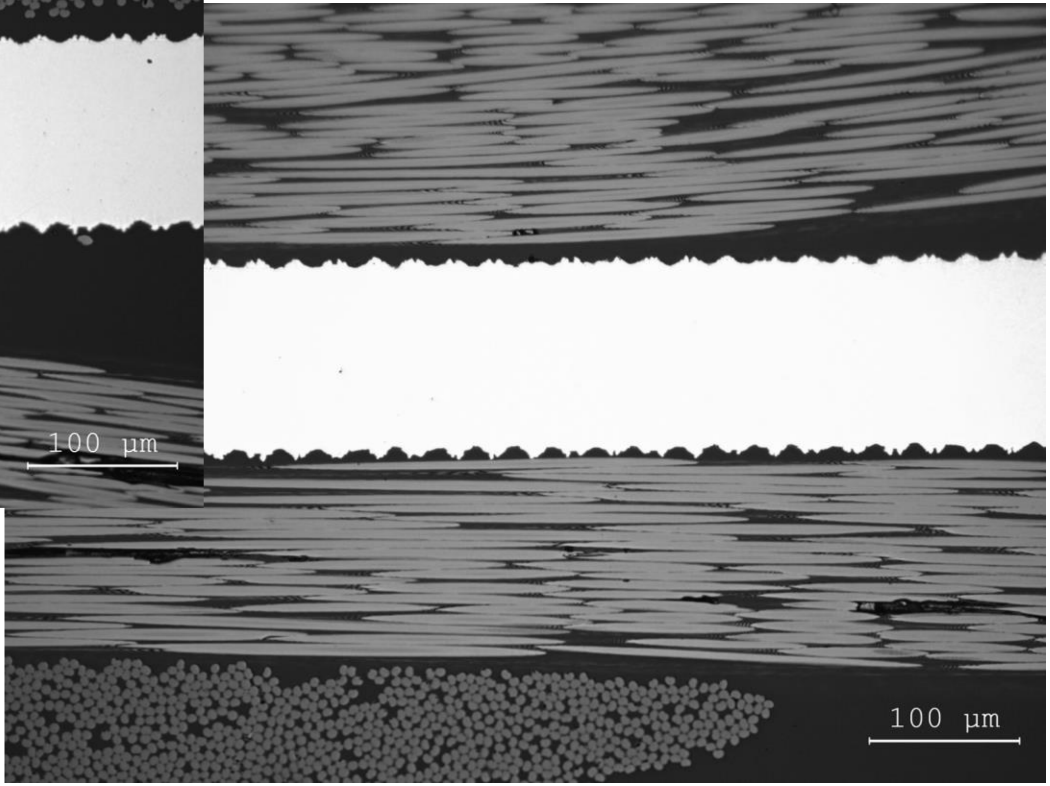
Other characterization techniques: TMA, DMA, FTIR, optical and electron microscopy (OM, SEM/EDS)

Cross-section of Laser Ablated Ti-PMC Interface

Interfaces near resin-rich regions



Interfaces where fiber tows are adjacent to Ti surfaces

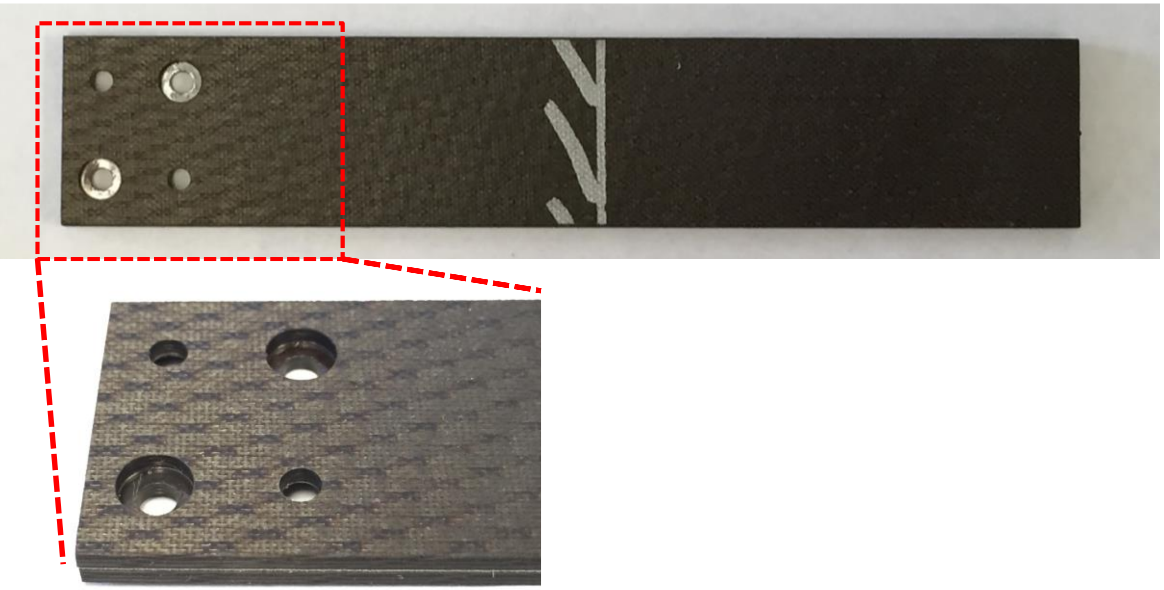


Mode I Fracture Toughness Test: Double Cantilever Beam Test with In-situ Digital Image Correlation (DIC)

DCB Specimens for High Temperature Tests

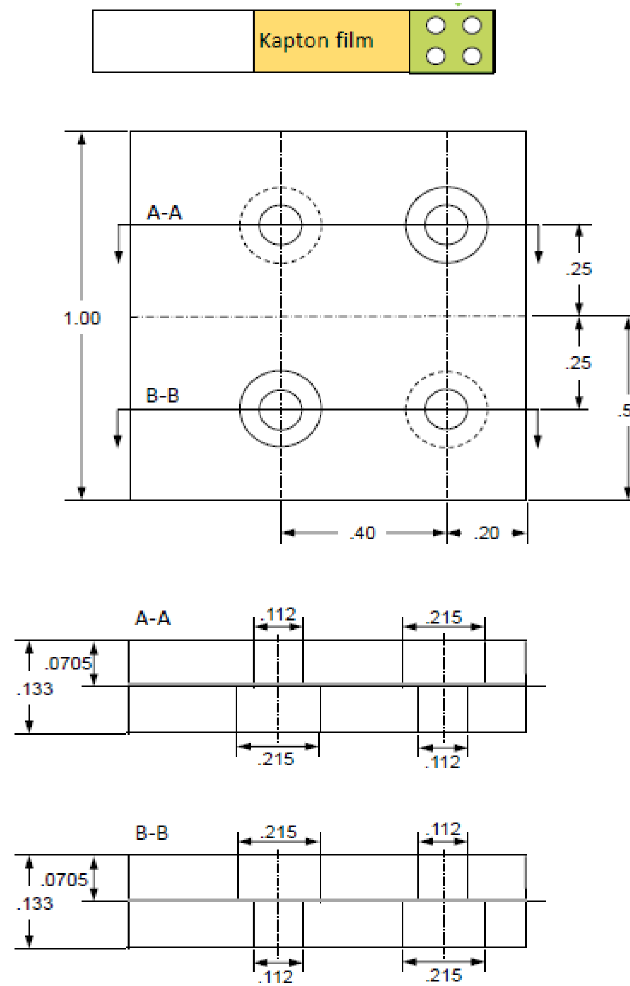
Hinges are mechanically attached to DCB using screws to facilitate high temperature testing

Top view of drilled DCB specimens



DCB specimen with hinges attached

Drilling pattern of DCB specimens



Units are in inches

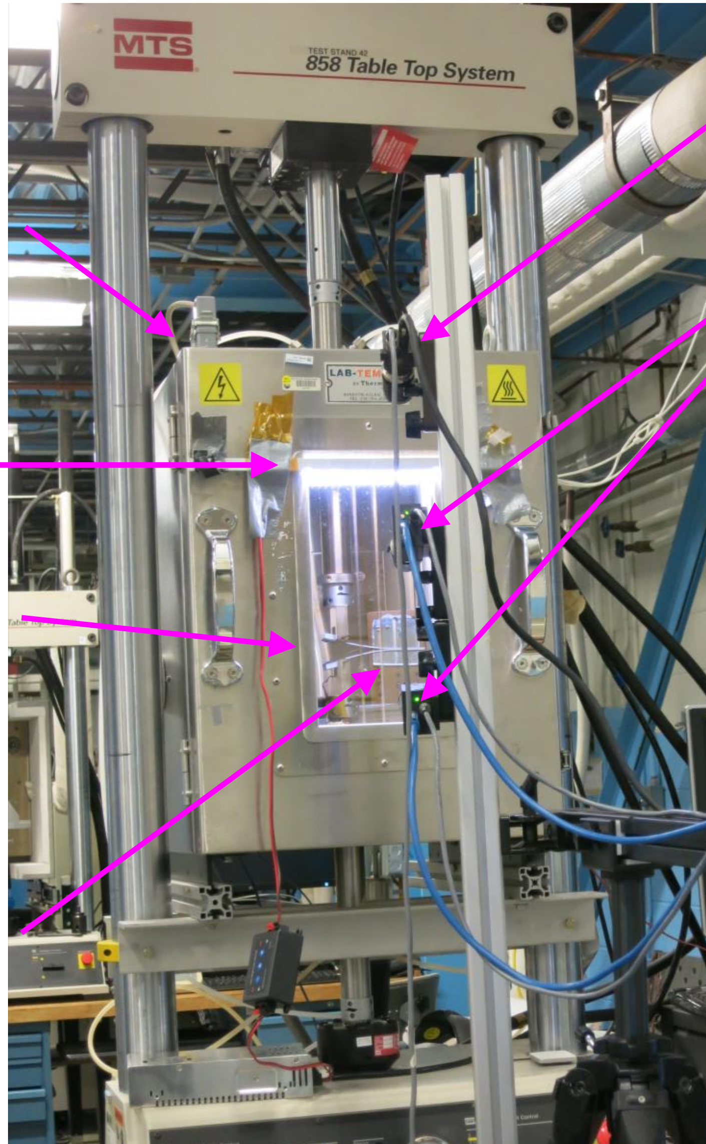
DCB Test Setup with In-situ DIC

Environmental chamber

Light source

DCB specimens being loaded

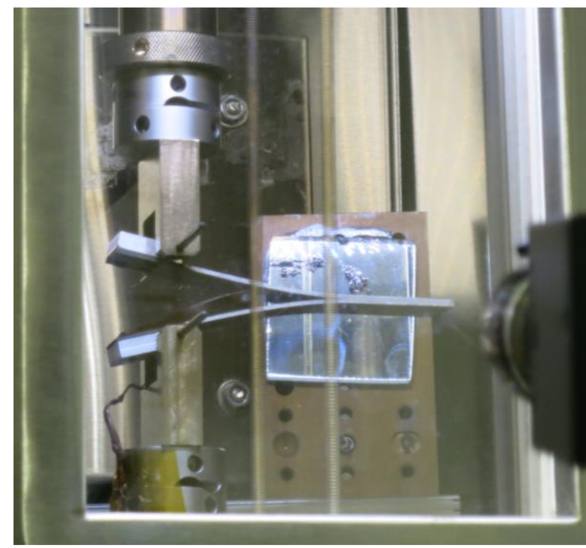
Mirror for reflection of crack path on back side of DCB specimen



Camera for crack growth monitoring (capturing images of the mirror during the test)

Cameras for 3-D DIC

A DCB specimen being tested in the environmental chamber

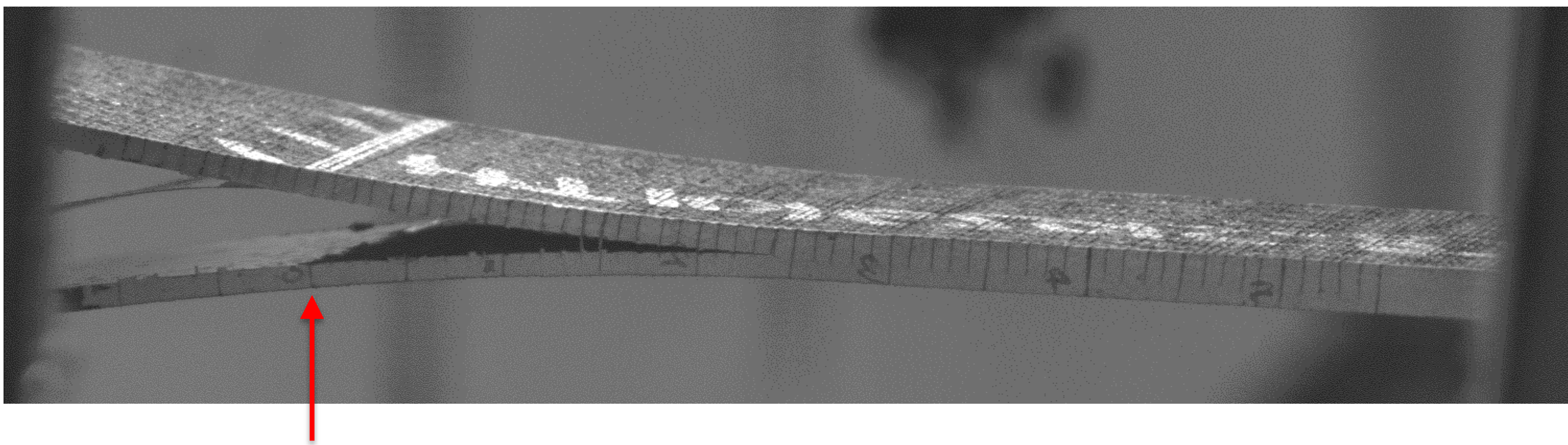


DCB test was performed by displacement controlled loading to 1 in followed by unloading to zero displacement (loading rate: 0.04 in/min & unloading rate: 0.2 in/min)

Crack Growth Visual Monitoring

The back side of the specimen was painted white and a scale was either adhesively bonded on the top edge or marked directly on the specimen.

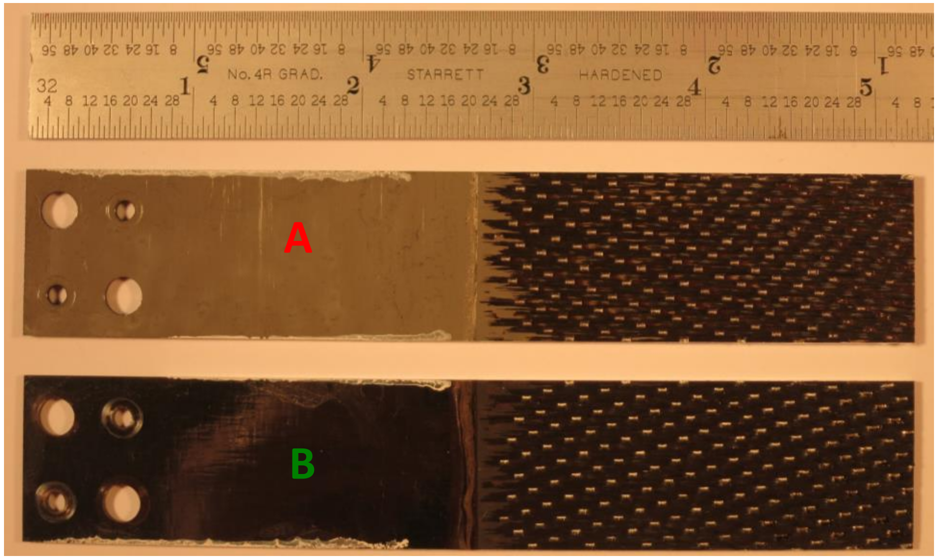
Images for crack growth monitoring were taken by capturing the image on the mirror placed at the back of the environmental chamber, reflecting the image of the specimen's back side.



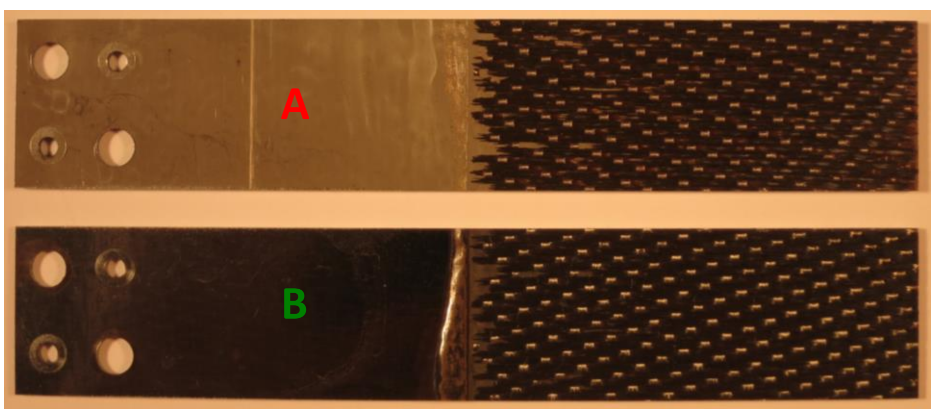
End of pre-crack location (measured before the test by UT scan analysis)

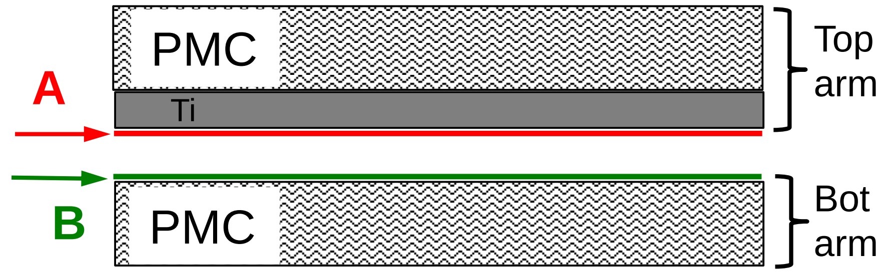
Fracture Surfaces (Sol-gel Hydrolyzed 1 hr)

RT DCB

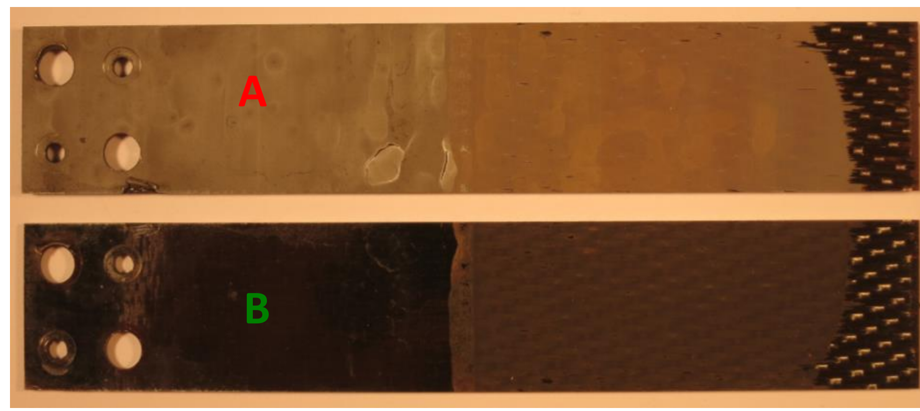


150°C DCB





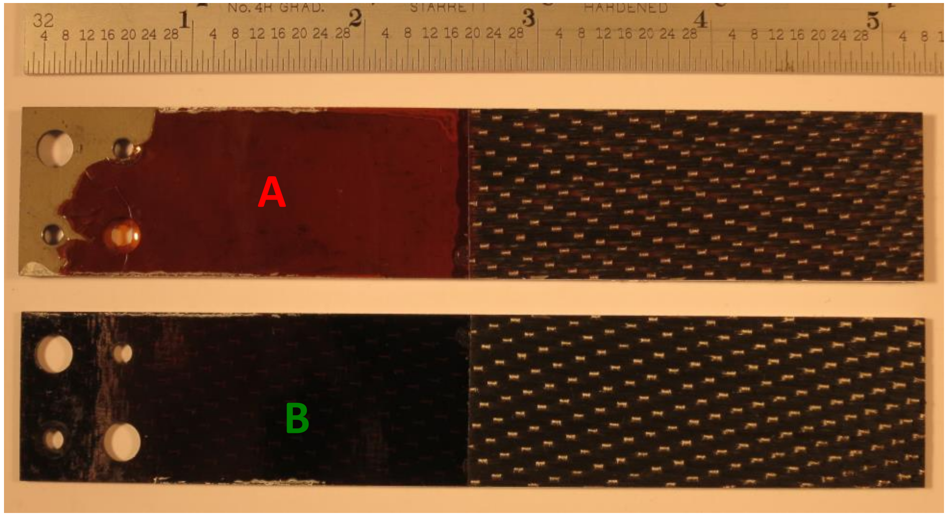
250°C DCB



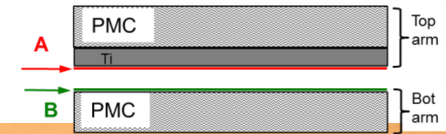
- Adhesive failure at 250 °C
- Cohesive failure at RT and 150 °C
  - Crack initiated at Ti surface then migrated to PMC

Fracture Surfaces (Sol-gel Hydrolyzed 16hr)

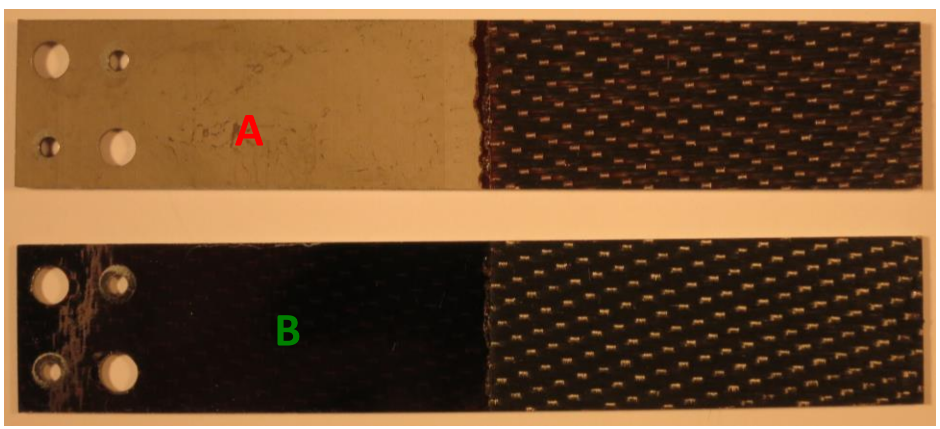
RT DCB



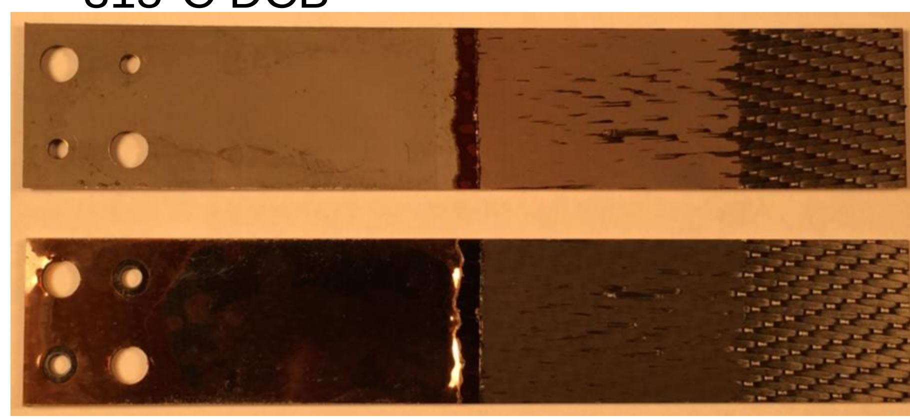
250^{o}C DCB



150^{o}C DCB



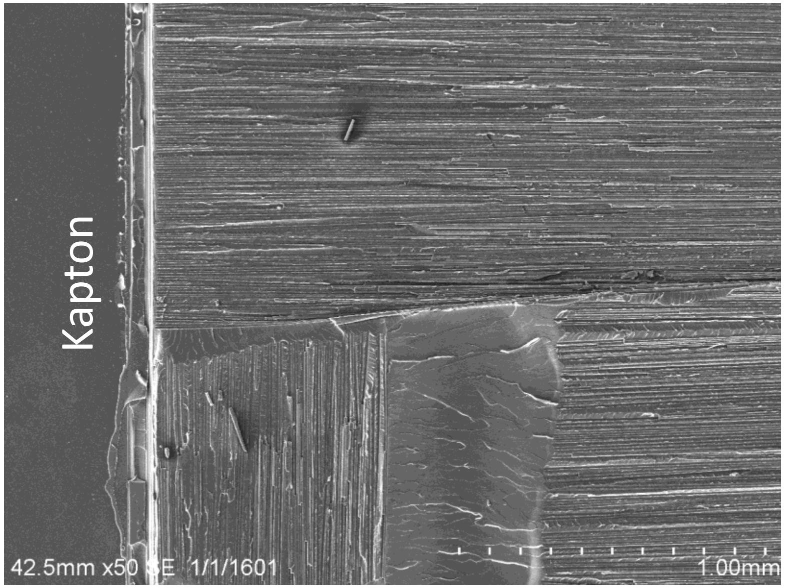
315^{o}C DCB

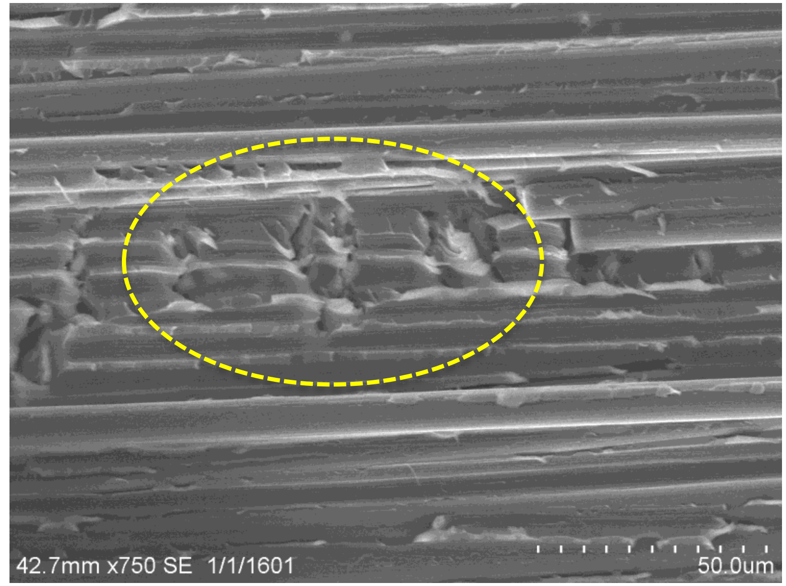


- Adhesive failure at 315 ^{o}C
- Cohesive failure at RT, 150 ^{o}C and 250 ^{o}C
  - Crack initiated in PMC side and remained in the same interface
  - No adhesive failure observed

SEM Fracture Surface (Ti side–RT and 150ºC DCB) (16 hr Hydrolyzed)

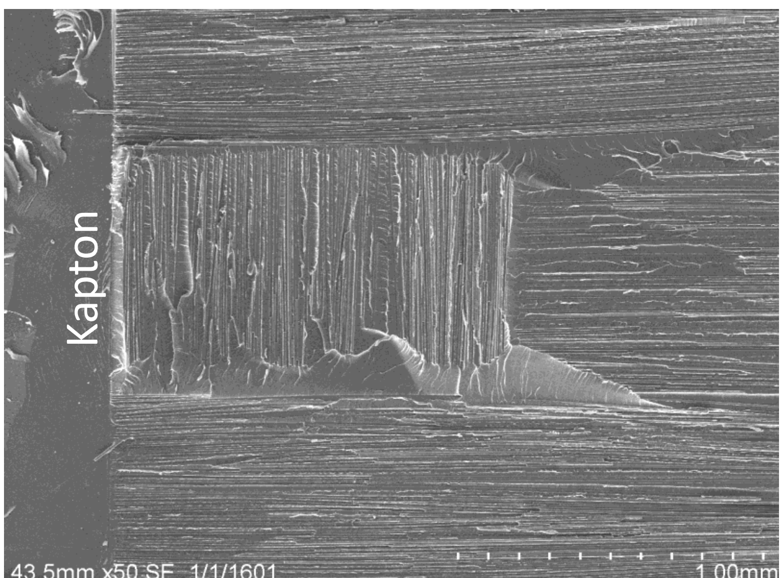
RT DCB

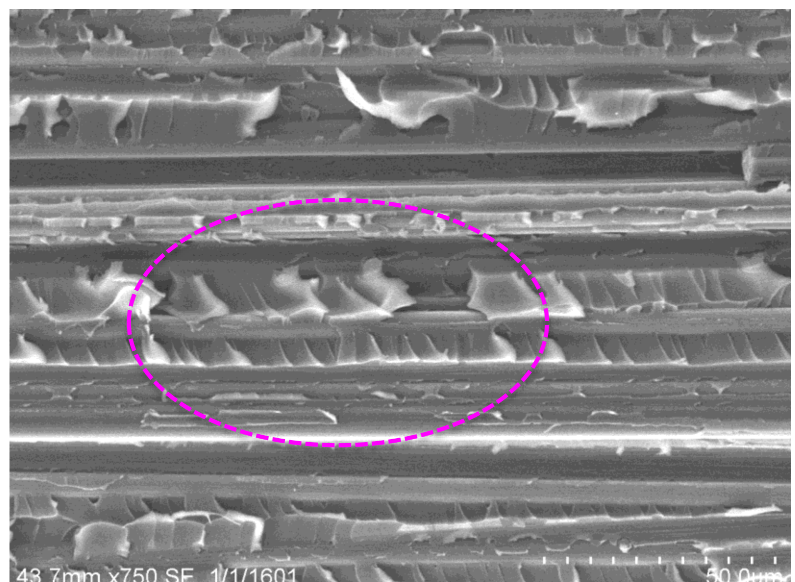




Micro-crack due to stress concentration at the troughs of laser ablated pattern on Ti surface

150ºC DCB



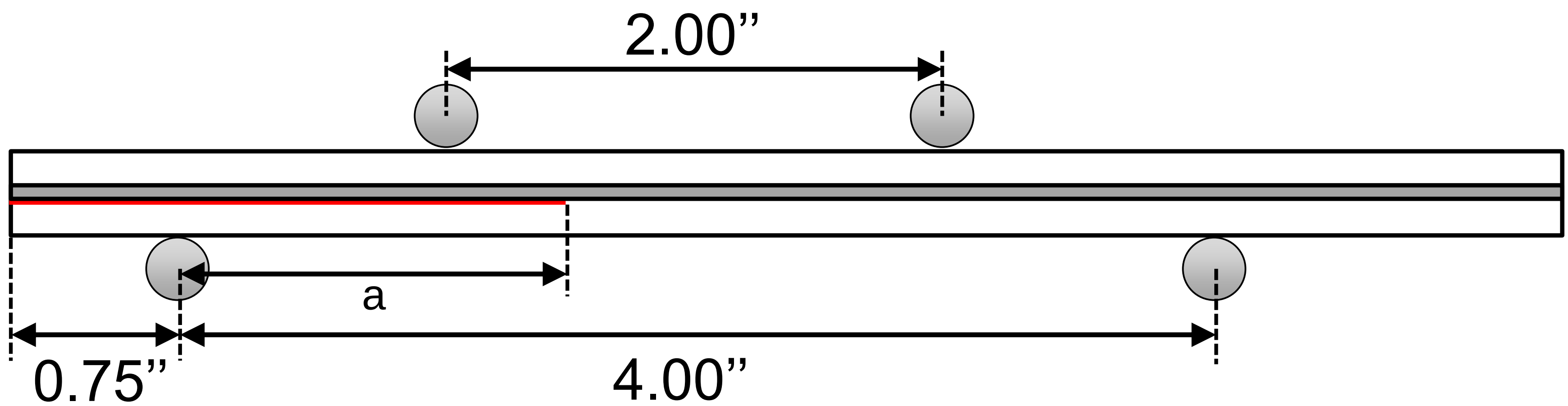


More upright cusps at HT due to matrix softening and shearing at fiber-matrix interface

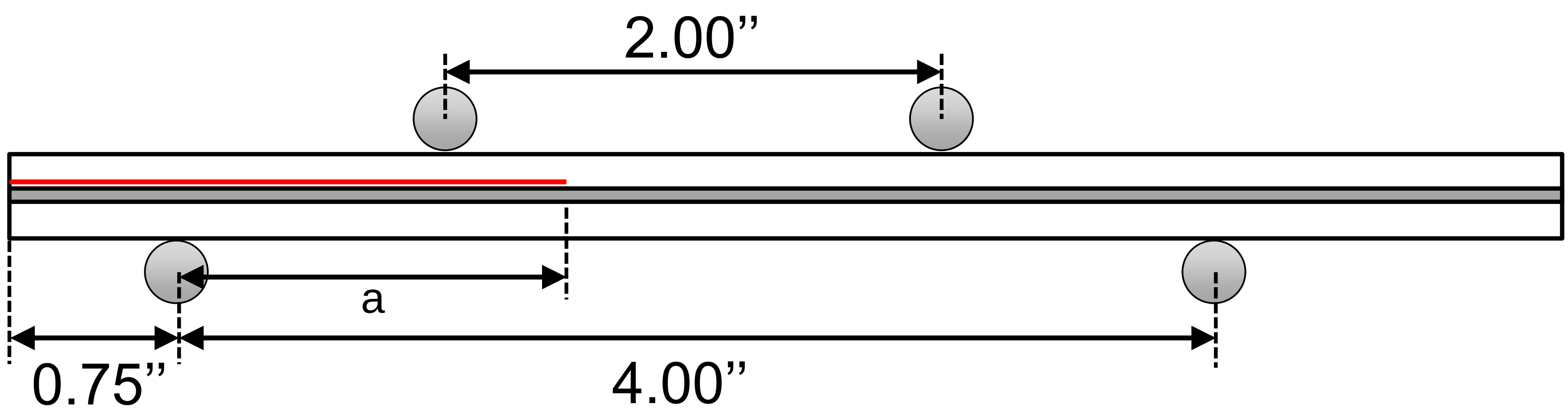
Mode II Fracture Toughness Test: 4-point End Notch Flexure Test

Schematic of 4-ENF Test

Crack growth on tension side: Kapton film is on the bottom of Ti foil



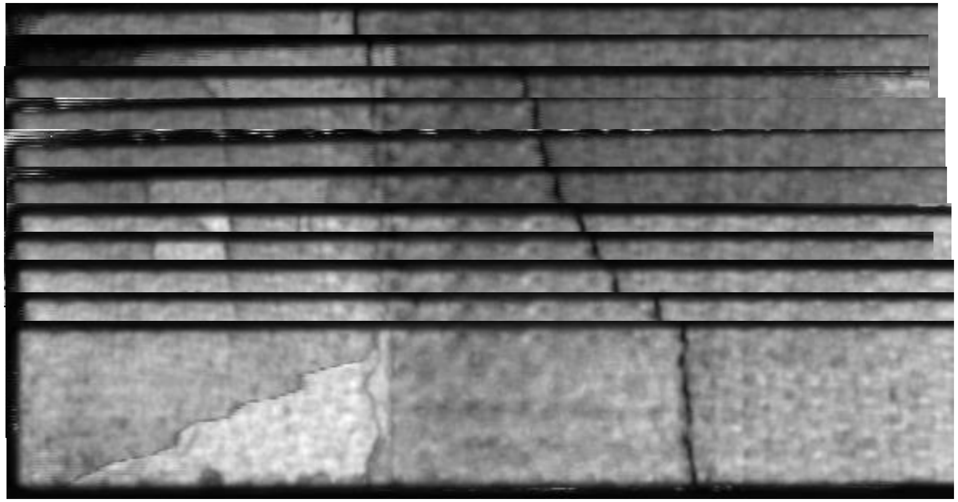
Crack growth on compression side: Kapton film is on the top of Ti foil



Kapton film   a: crack length

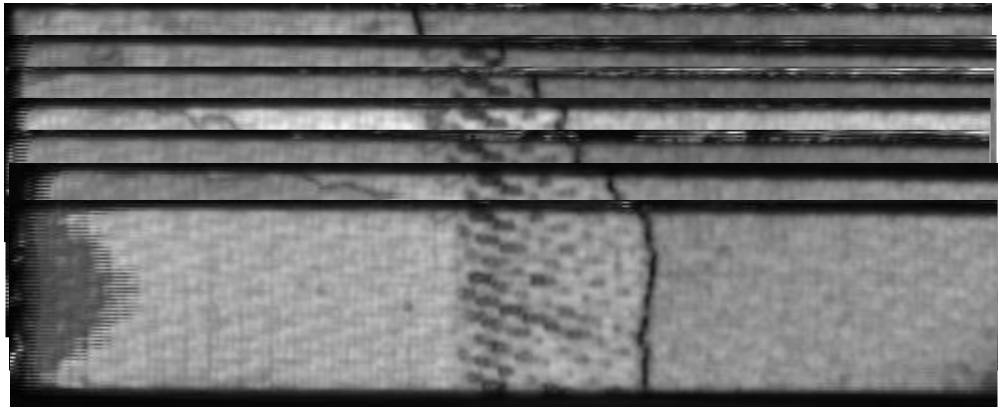
Monitoring Crack Growth using UT Scan

Specimen 17 (4-ENF test crack on tension side)



UT images show clean interface, indicating adhesive failure.

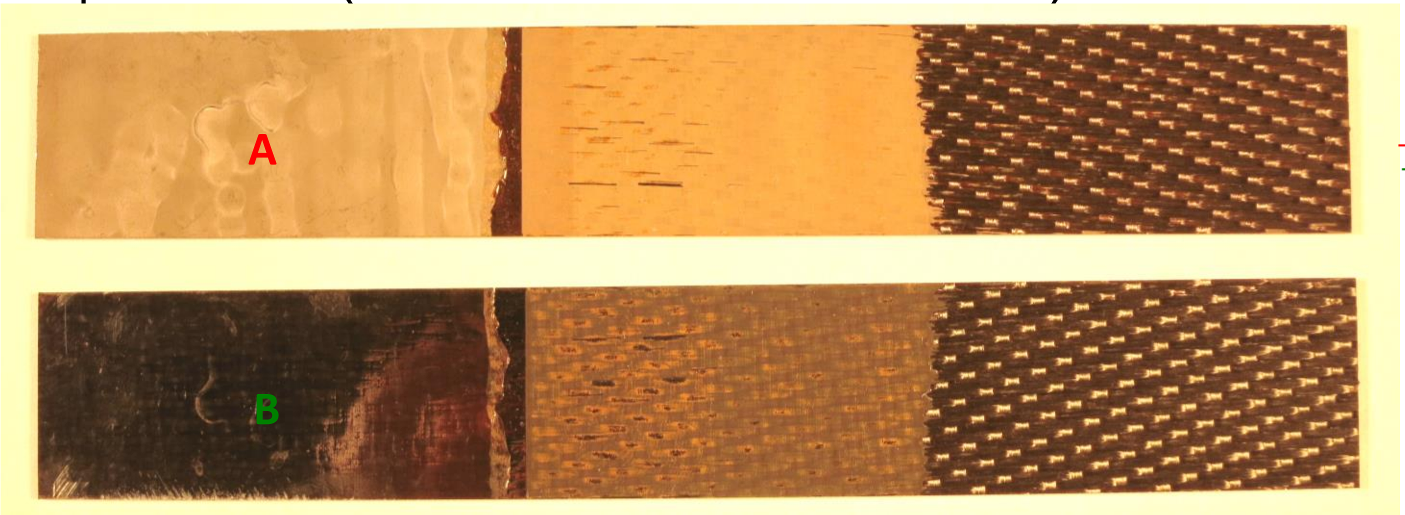
Specimen 11 (4-ENF test crack on compression side)

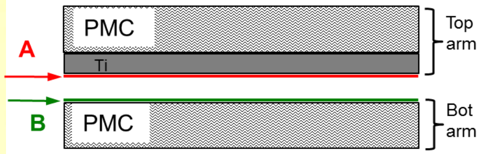


UT images at the interface show patterns from the carbon fabric bonded on Ti foil surface, indicating some cohesive failure.

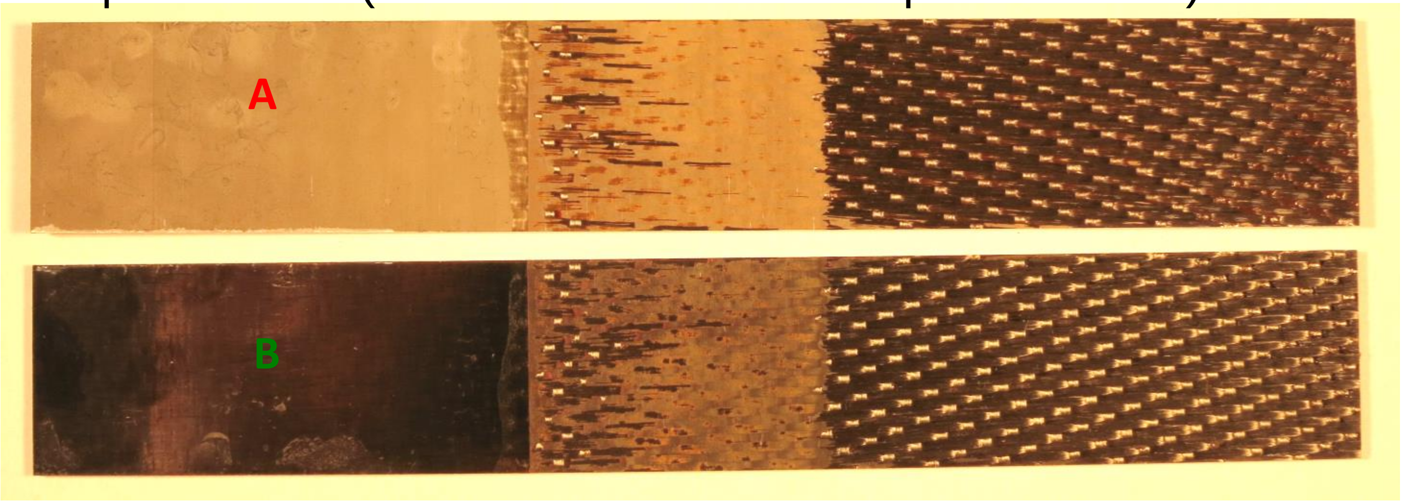
Fracture Surfaces After 4-ENF Tests at RT

Specimen 17 (4-ENF test crack on tension side)



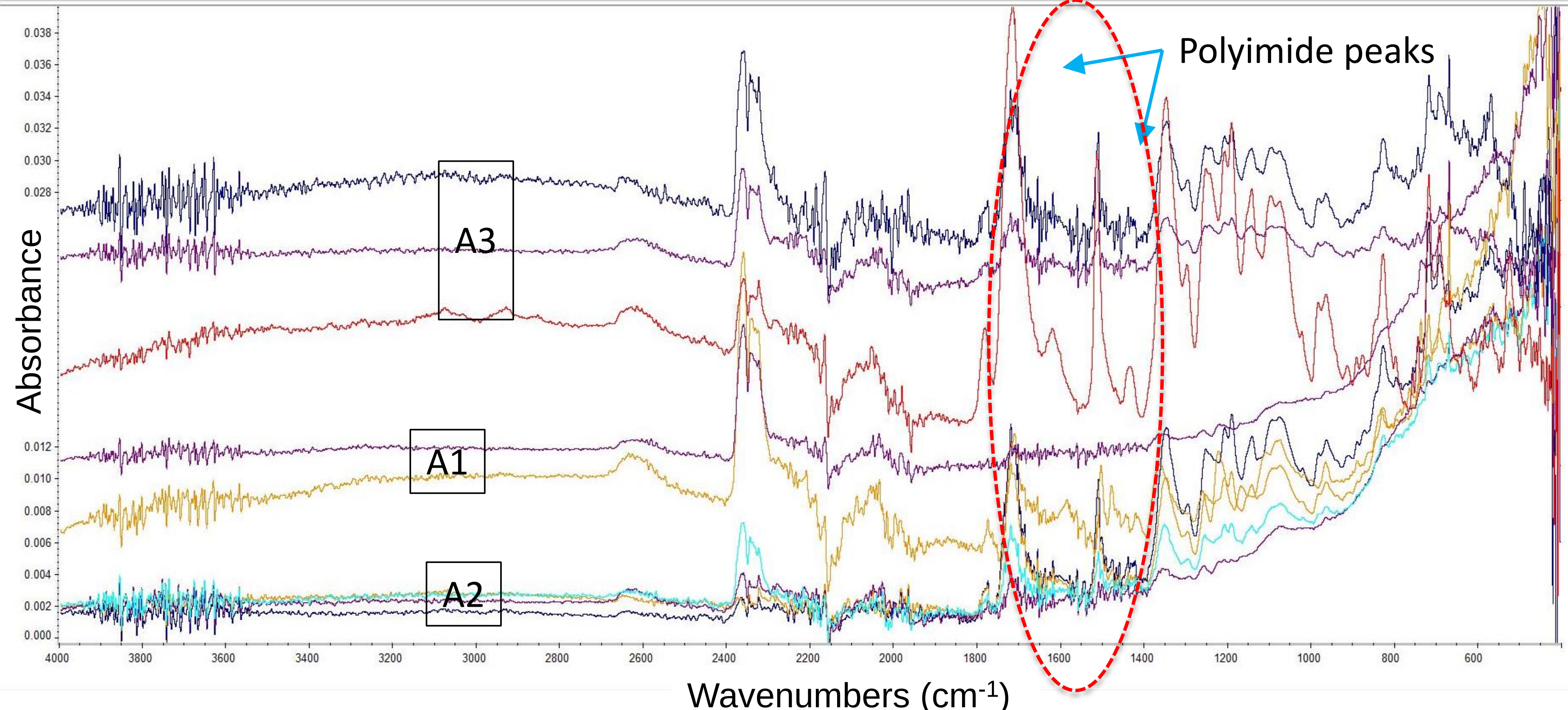


Specimen 11 (4-ENF test crack on compression side)

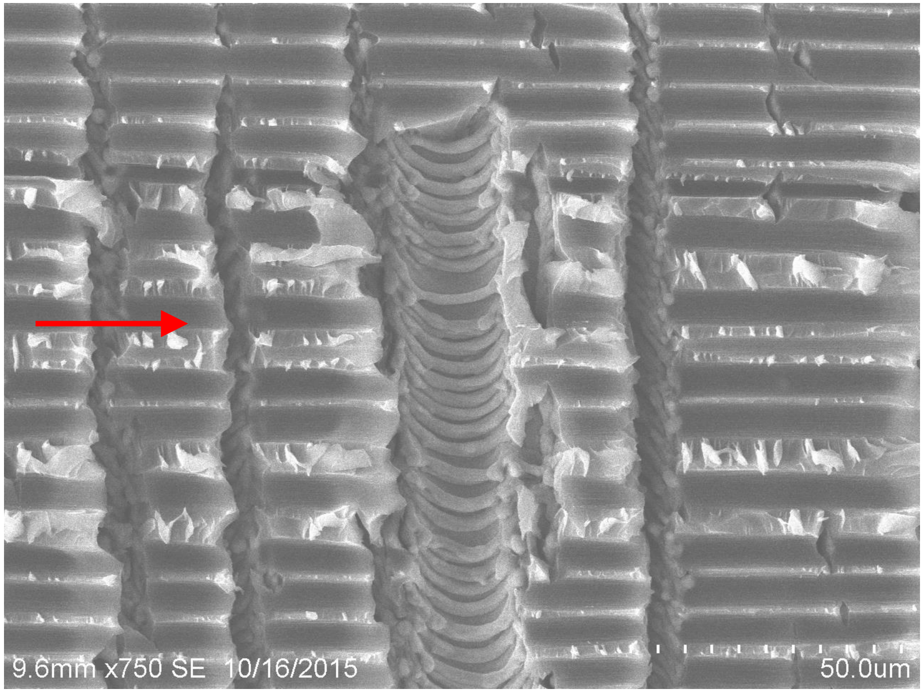


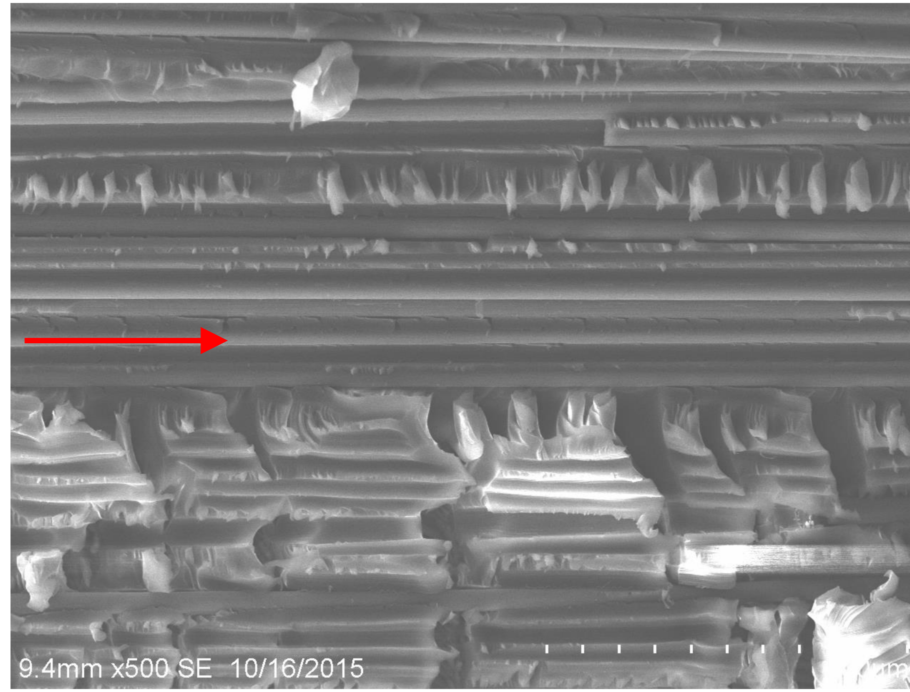
FTIR Spectra

Ti side of Sample 17 (4-ENF test crack on tension side)



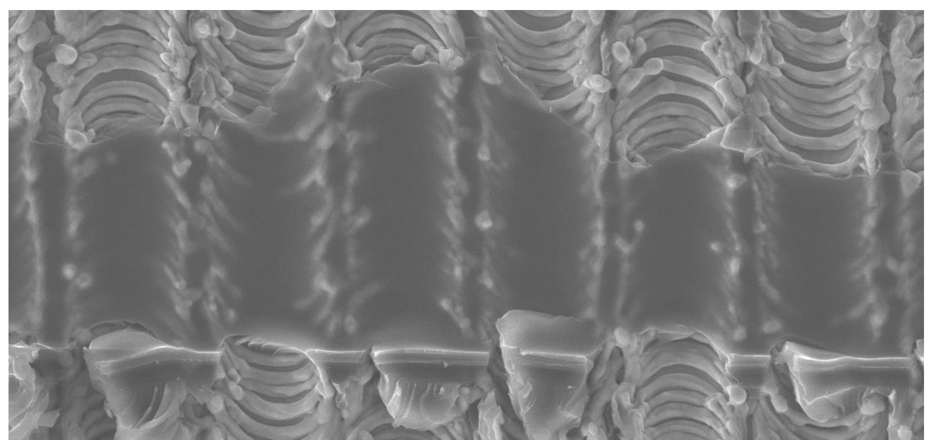
Fracture Surface of Laser Ablated Ti (1 hr Hydrolyzed)



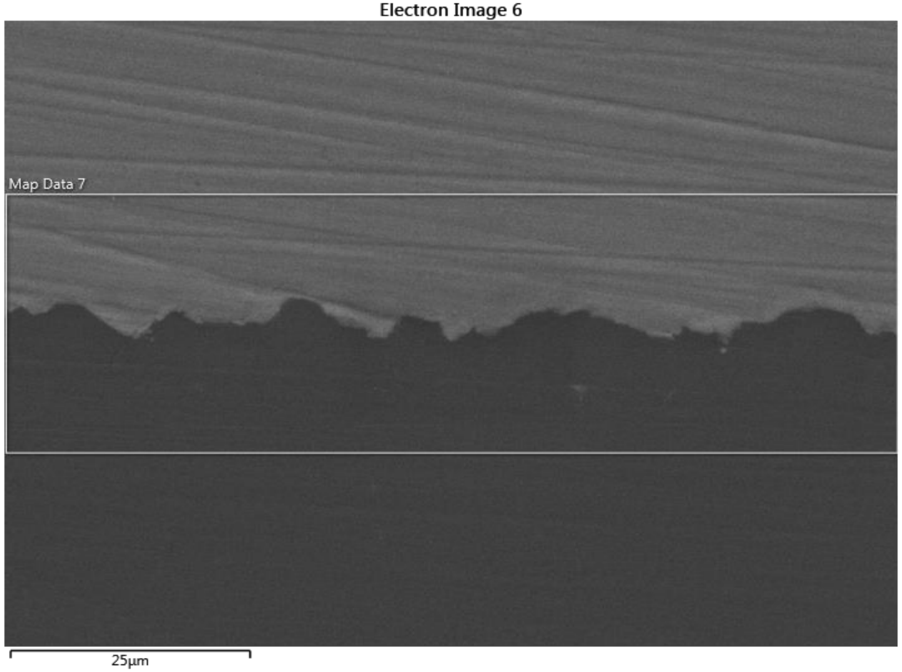


→ Crack growth direction

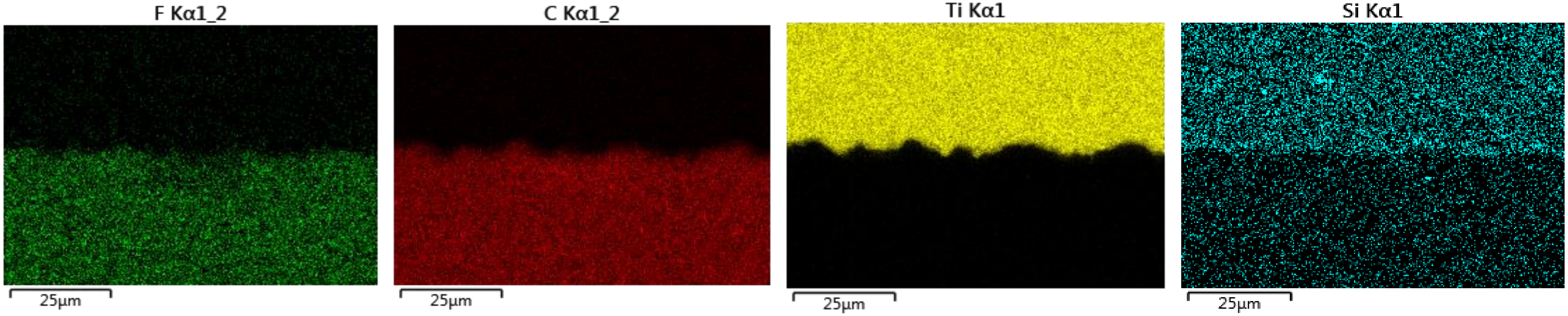
- Additional fracture mechanism due to stress concentration results in higher fracture toughness



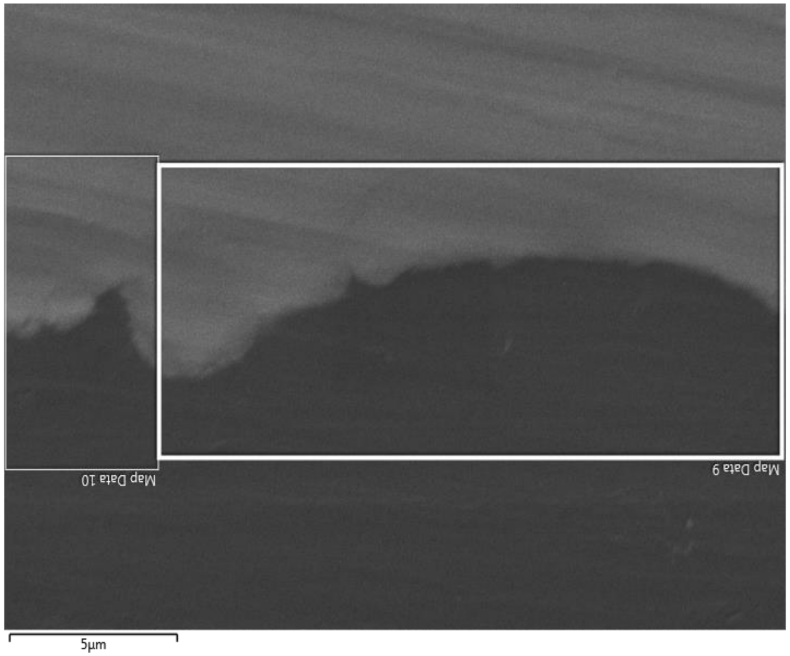
EDS on Cross-section of Ti-PMC Interface (Sol-gel Hydrolyzed ~16 hrs)



- Si, C, and F diffuse into the ablated Ti layer. This could be due to the porosity on the Ti foil surface caused by laser ablation.
- High Si concentration is observed in Ti layer. Notice that the sol-gel solution has low viscosity and was sprayed onto the laser ablated Ti foil. The time of panel processing at high temperature was long. This could be sufficient for the silane groups to diffuse into the Ti layer.

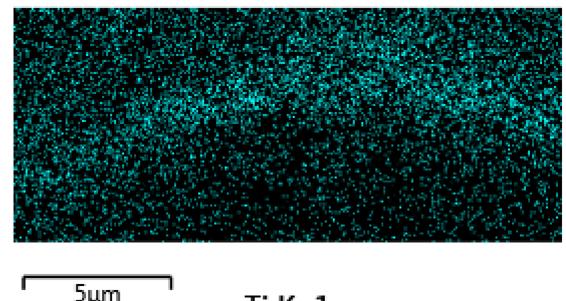


EDS on Cross-section of Ti-PMC Interface (Sol-gel Hydrolyzed ~16 hrs)

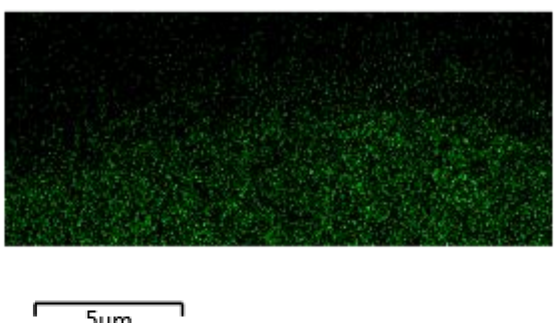


- Si, C, and F penetrate into ablated Ti surface
- Interphase region is ~2 μm indicated by the area where the high concentration of Si is observed

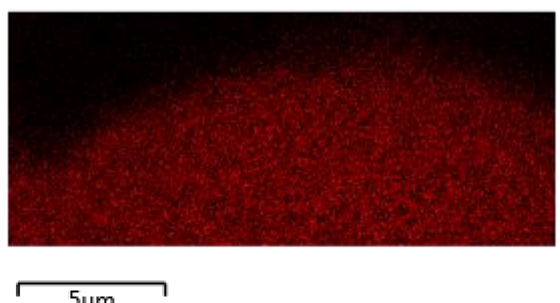
Si Kα1



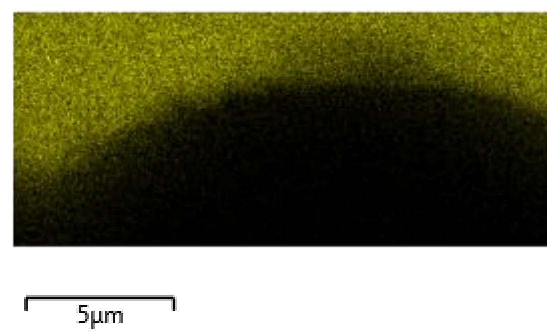
F Kα1_2

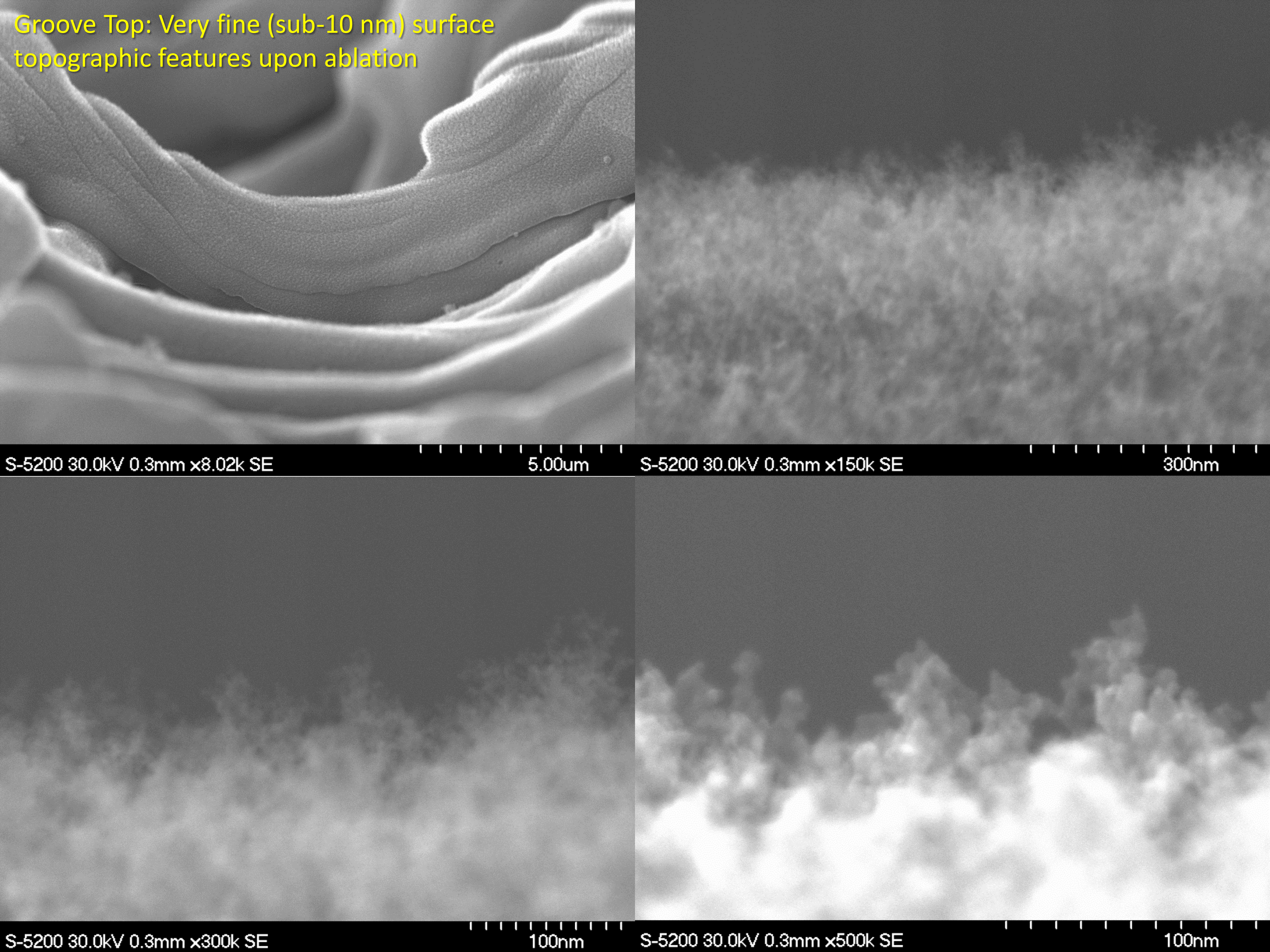


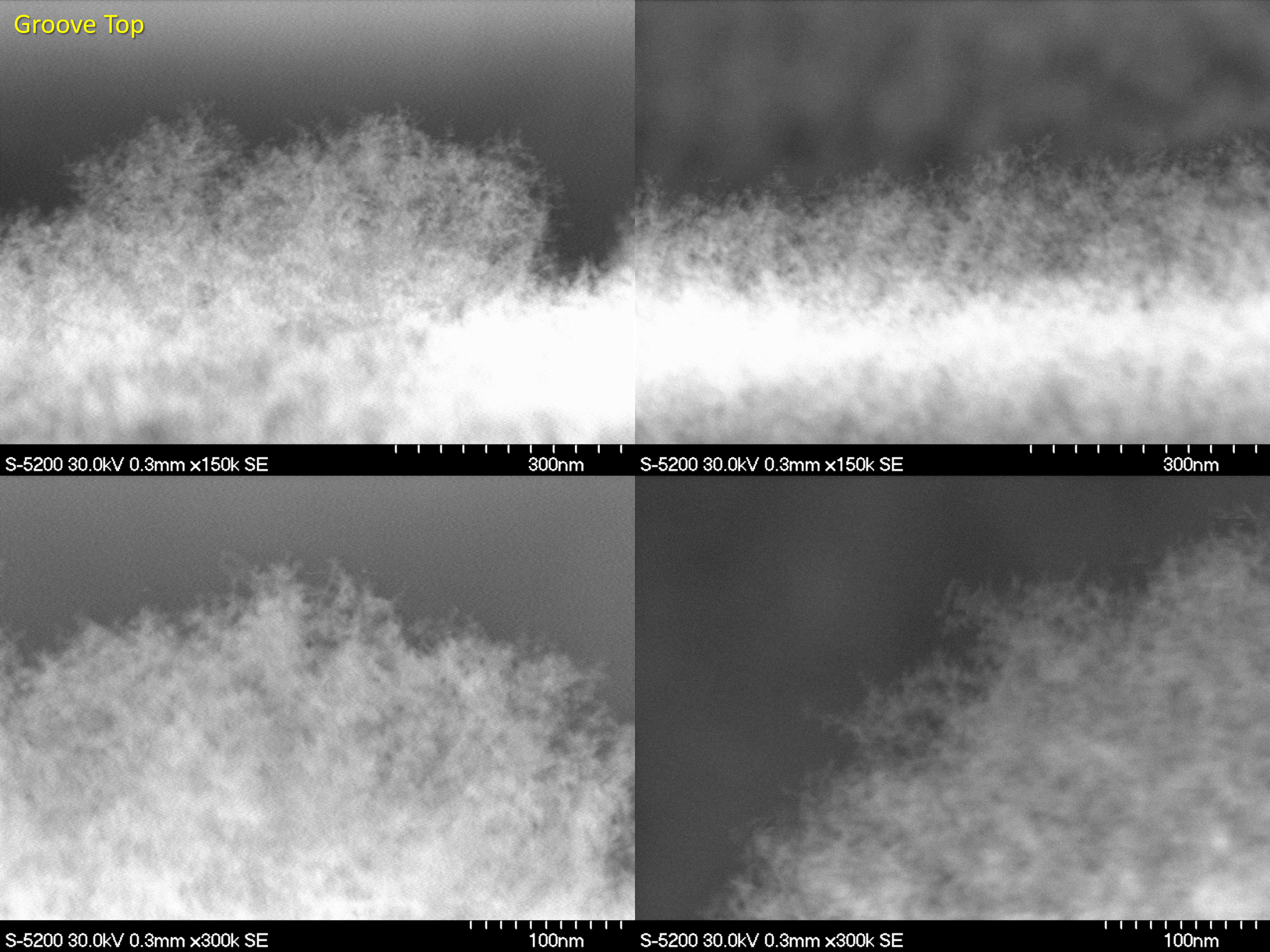
C Kα1_2



Ti Kα1




Groove Top: Very fine (sub-10 nm) surface topographic features upon ablation
S-5200 30.0kV 0.3mm x8.02k SE
5.00um
S-5200 30.0kV 0.3mm x150k SE
300nm
S-5200 30.0kV 0.3mm x300k SE
100nm
S-5200 30.0kV 0.3mm x500k SE
100nm


Groove Top
S-5200 30.0kV 0.3mm x150k SE
300nm
S-5200 30.0kV 0.3mm x150k SE
300nm
S-5200 30.0kV 0.3mm x300k SE
100nm
S-5200 30.0kV 0.3mm x300k SE
100nm

Conclusions

- Successfully created hybrid laminates where Ti foil was embedded in HTPMC. No adhesive was required.
- Laser ablation and imide-sol gel surface treatment resulted in a robust interface between Ti or Ni/Ti and PI PMC. Si clearly penetrated into the Ti surface.
- The time associated with sol-gel hydrolysis was important and the best results were achieved with 16 hrs, further optimization may be possible.
- Cohesion failures were observed up to 250^{o}C. Adhesion failures appeared at 315^{o}C.
- Different failure mechanisms were observed as test temperature increased.

Future Work

- Perform more detailed study on the influence of hydrolysis time on interfacial adhesion
- Study shape-memory alloy transformation during fracture process
- In-situ fracture toughness testing using DIC
  - Cohesive zone calibration for FEM
- Investigate different fabric architecture and matrix resin on fracture behavior

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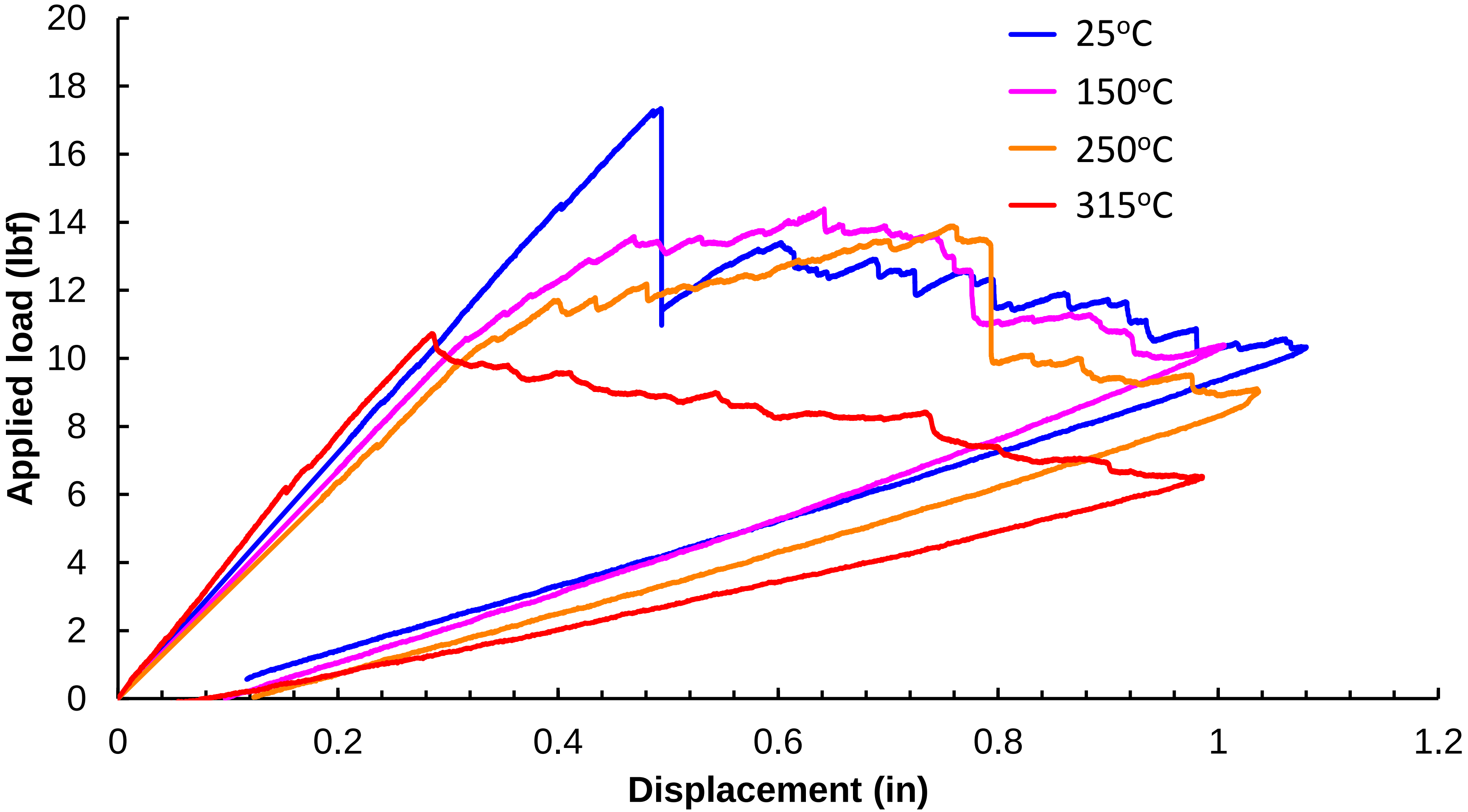
- Danny Lovaglio
- Jim Baughman
- Clay Claytor
- Crystal Chamberlin

Thank you for your attention!

Questions?

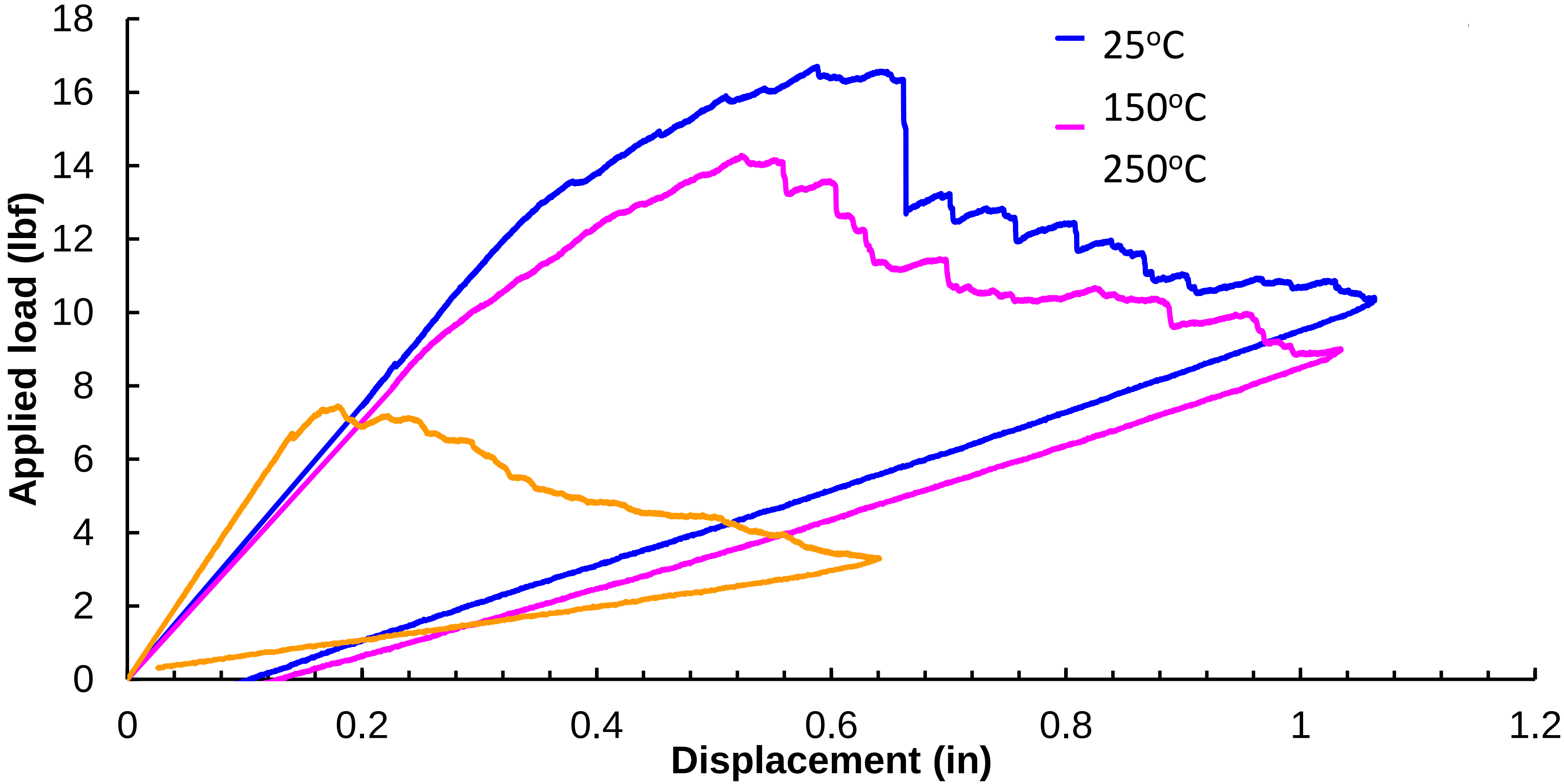
Back Up Charts

Load-displacement Curves (DCB w/ Ti foil Treated w/ Sol-gel Hydrolyzed 16 hours)



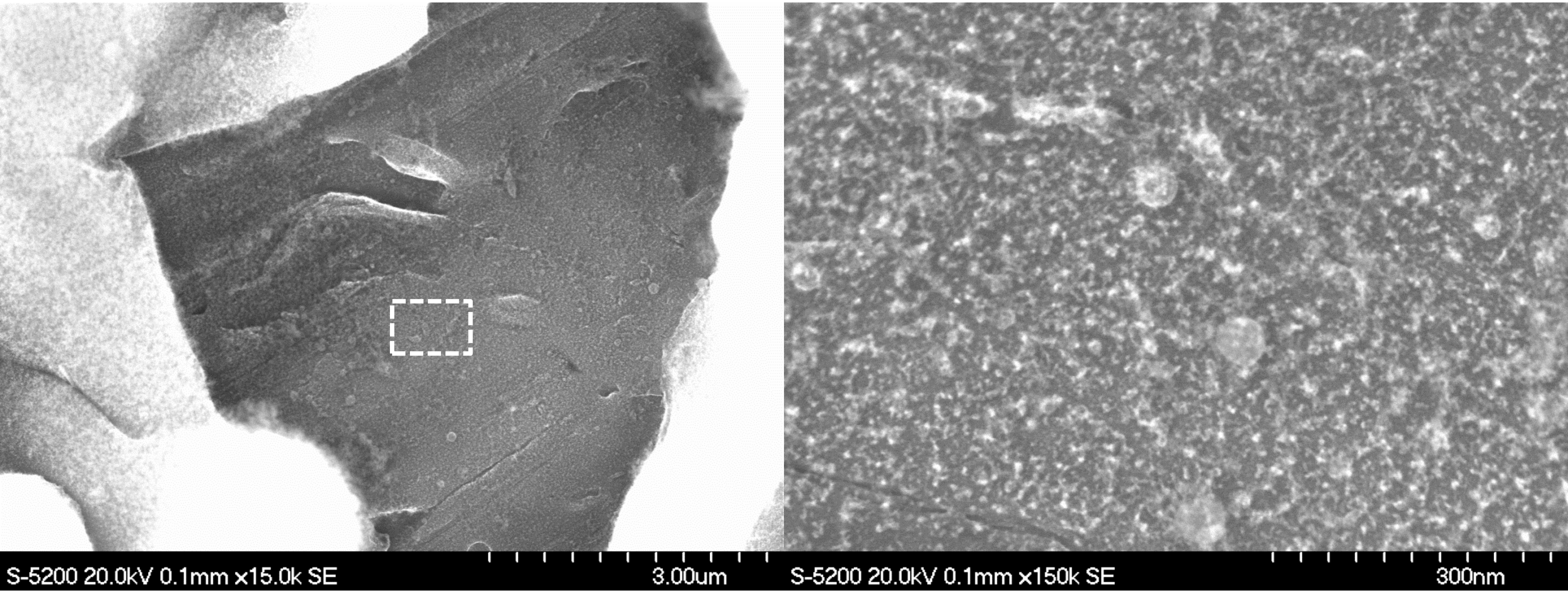
- Saw-tooth behavior is observed from tests at 25 °C, 150 °C and 250 °C, indicating cohesive failure.
- At 315 °C, the failure mode is adhesive.

Load-displacement Curves (DCB w/ Ti foil Treated w/ Sol-gel Hydrolyzed 1 Hour)

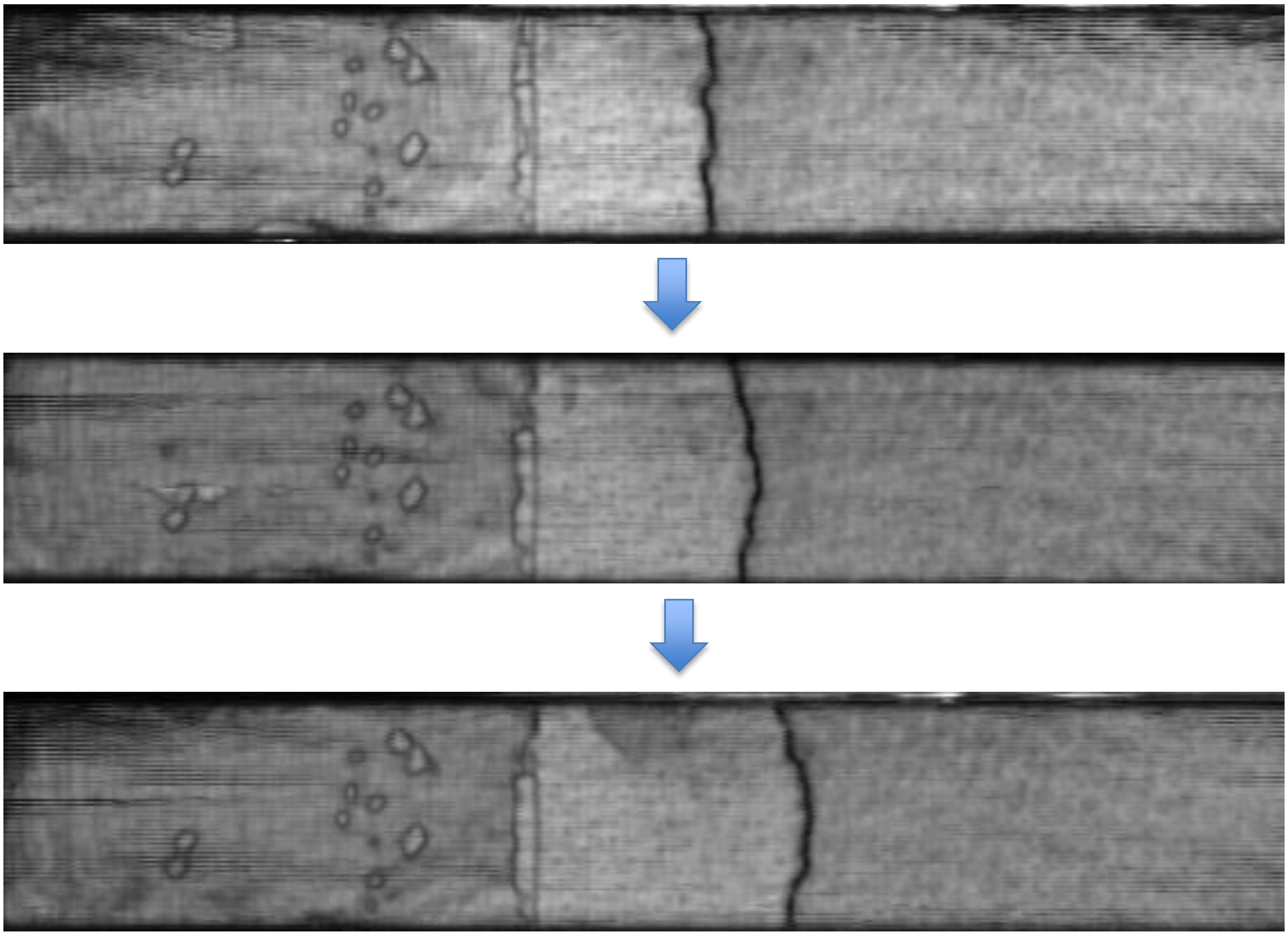


- Saw-tooth behavior is observed from tests at 25 °C and 150 °C. This shows unstable crack growth (growth-arrest) in the composite region, indicating cohesive failure at the interface.
- Smooth load-displacement curve is obtained from test at 250 °C. This shows stable crack growth near the Ti surface, indicating weak interface or adhesive failure.

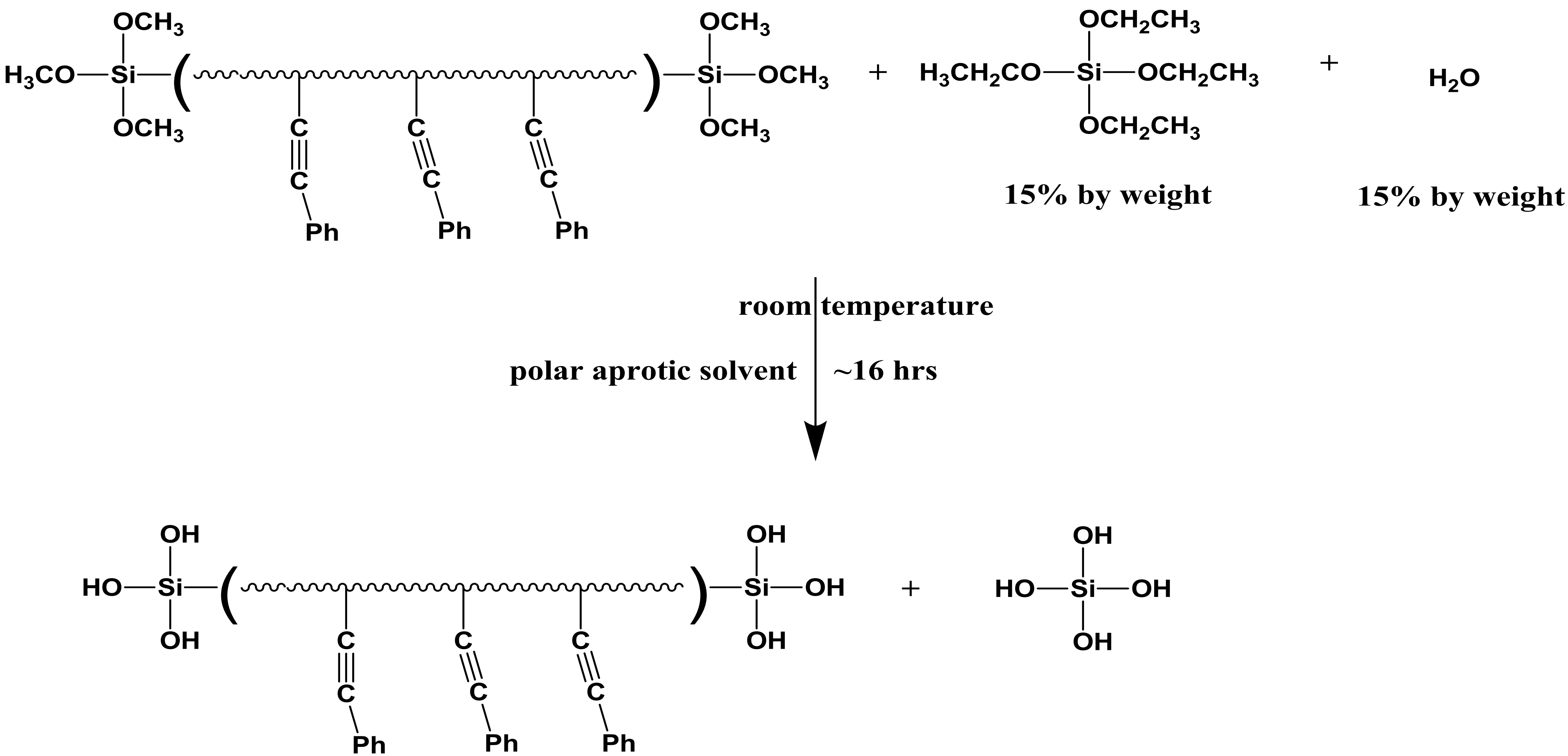
Non-ablated Ti Surface



Monitoring Crack Growth using UT Scan (1 hr hydrolyzed)



Synthesis of Amide Acid Sol-Gel Surface Treatment

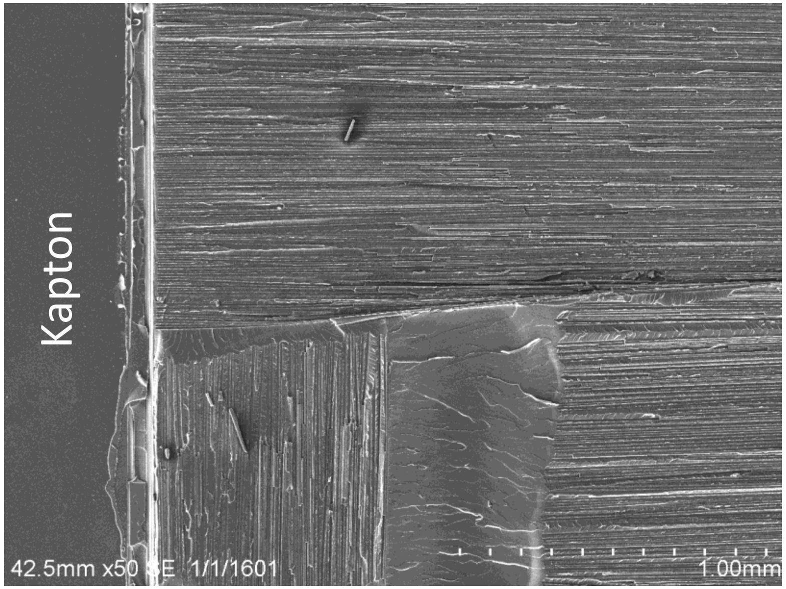


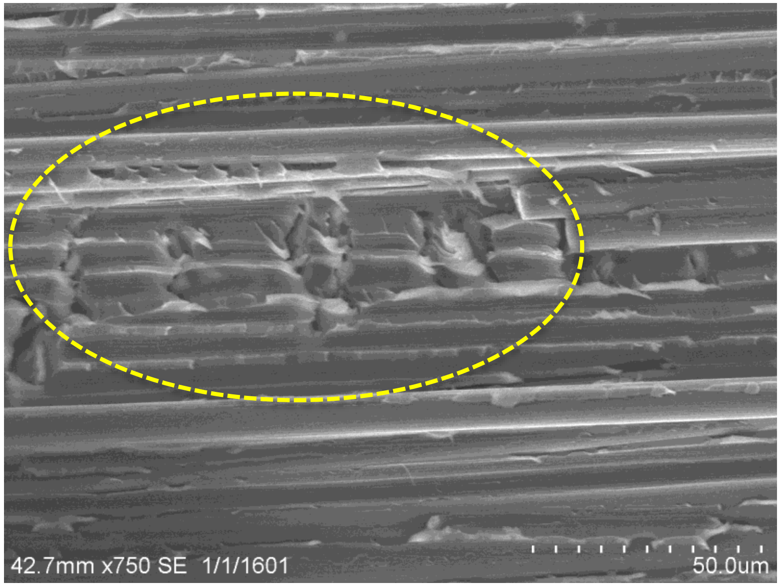
surface treatment solution

Park, C., Lowther, S.E., Smith, J.G., Connell, J.W., Hergenrother, P.M., and St. Clair, T.L.; "Polyimide-Silica Hybrids Using Novel Phenylethynyl Imide Silanes as Coupling Agents for Surface-Treated Alloy" International Journal of Adhesion and Adhesives, 20, 457-465 (2000).

SEM Fracture Surface (Ti side – RT and 150 ºC DCB with Sol-gel Hydrolyzed ~16 hr)

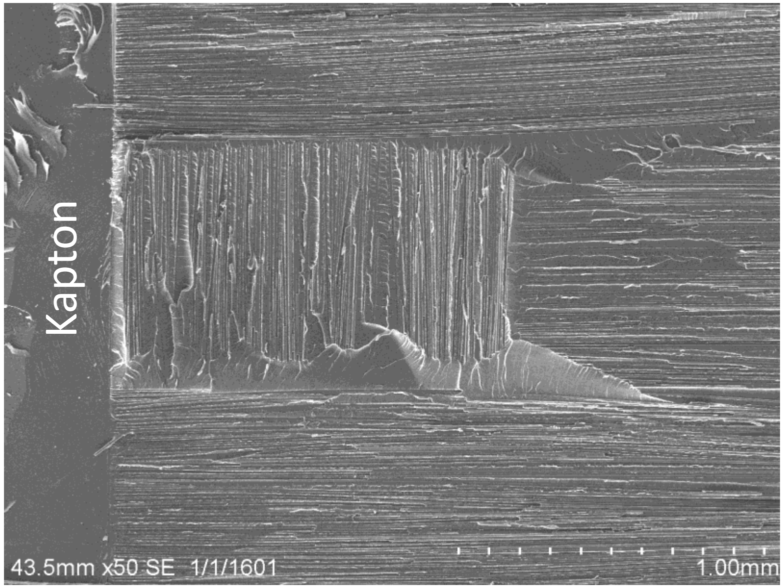
RT DCB

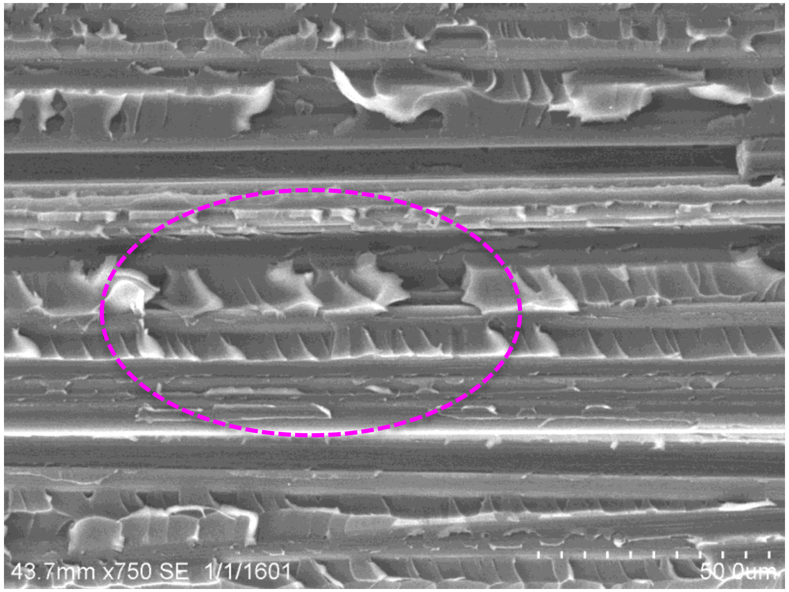




Micro-crack due to stress concentrat-ion at the troughs of laser ablated pattern on Ti surface

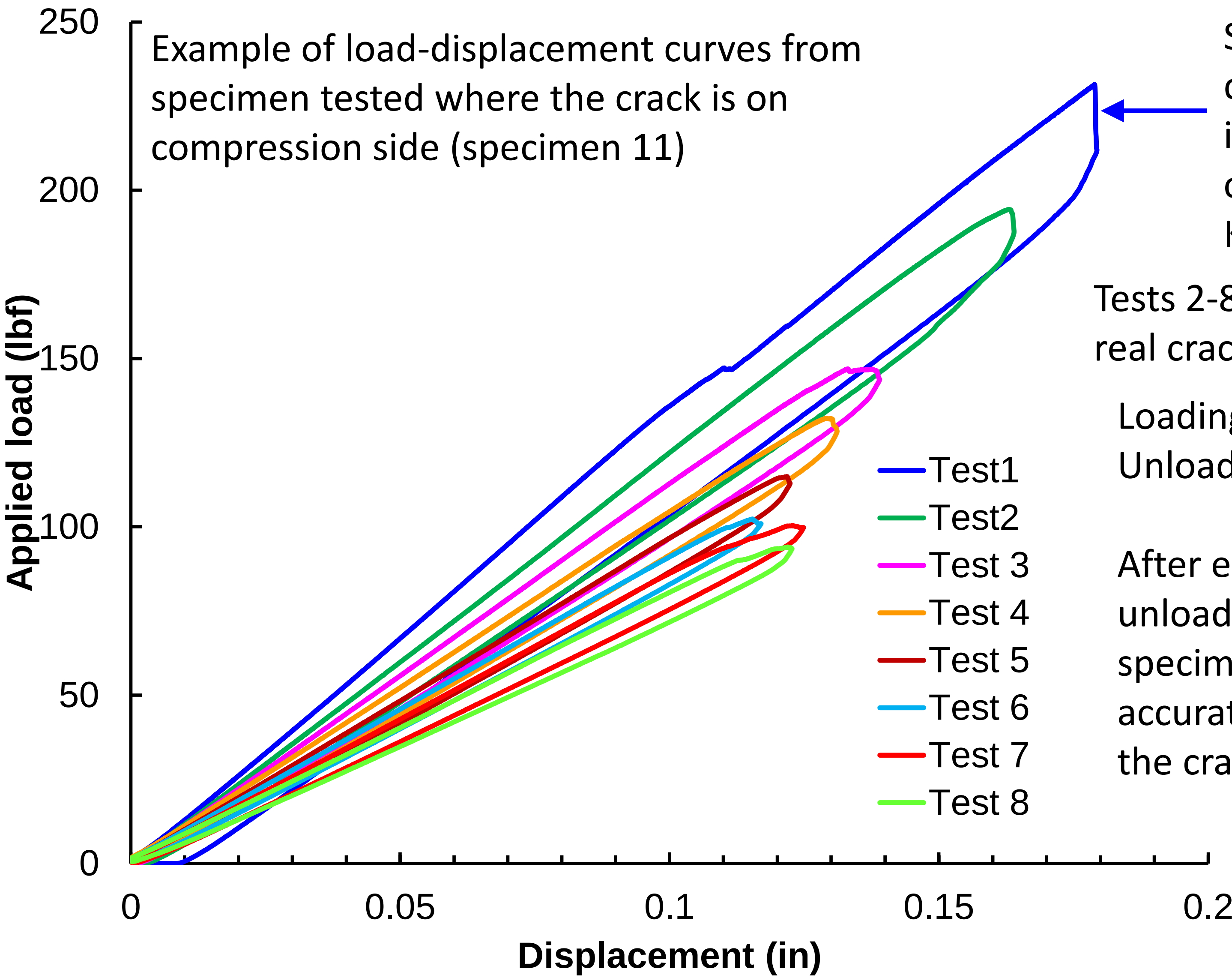
150ºC DCB





More upright cusps at HT due to matrix softening and shearing at fiber-matrix interface

Load-Displacement Plots



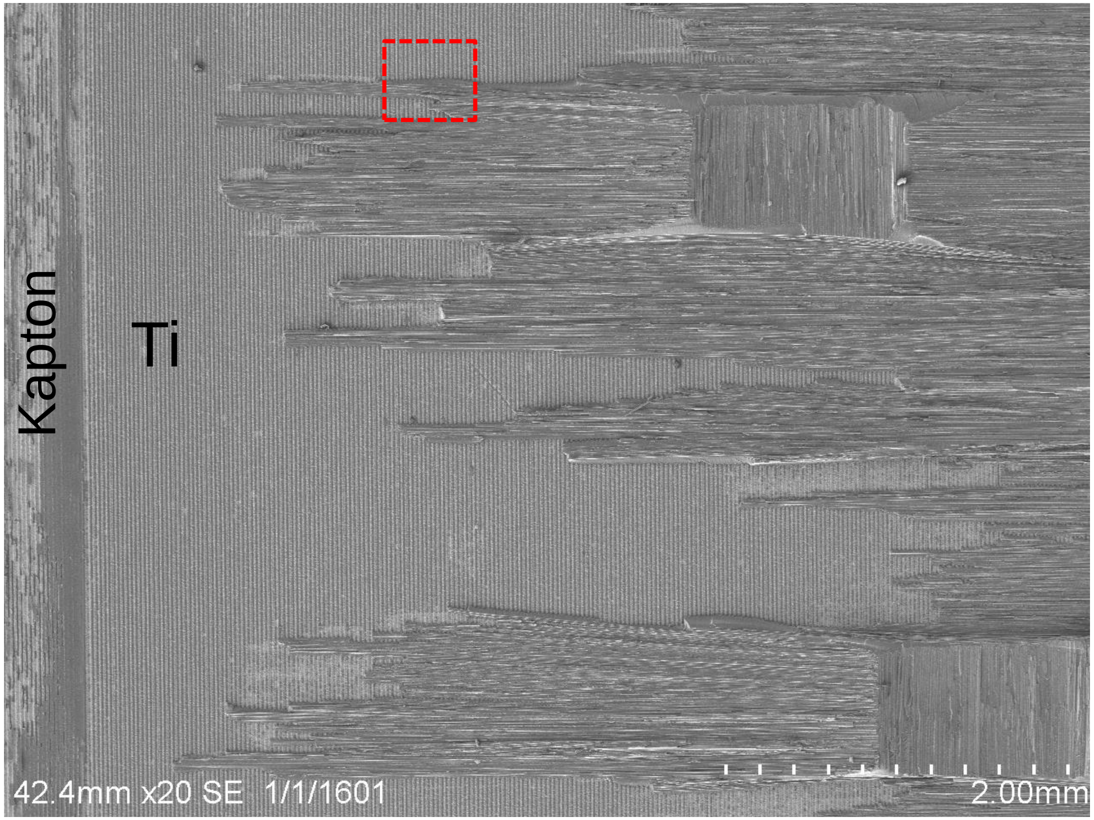
Sudden (sharp) load drop during Test 1 indicates unstable crack growth from Kapton insert.

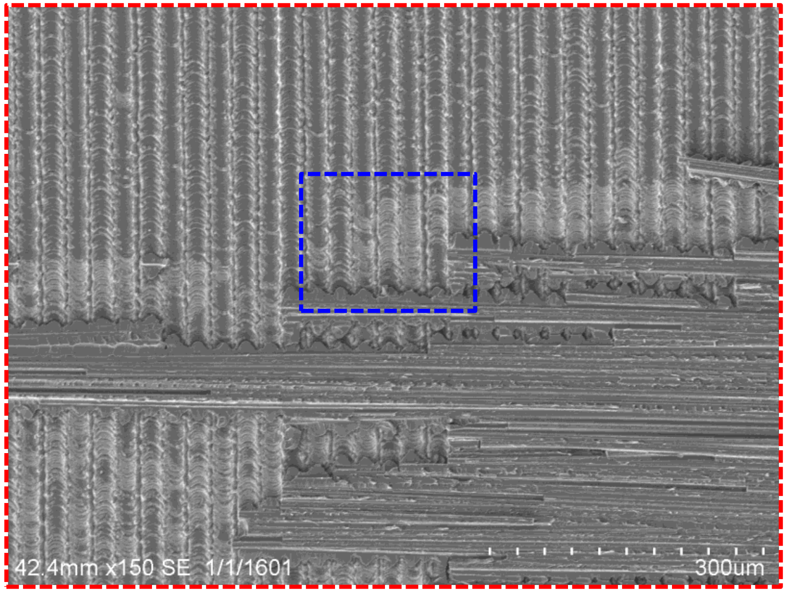
Tests 2-8: crack growth from real crack front is stable.

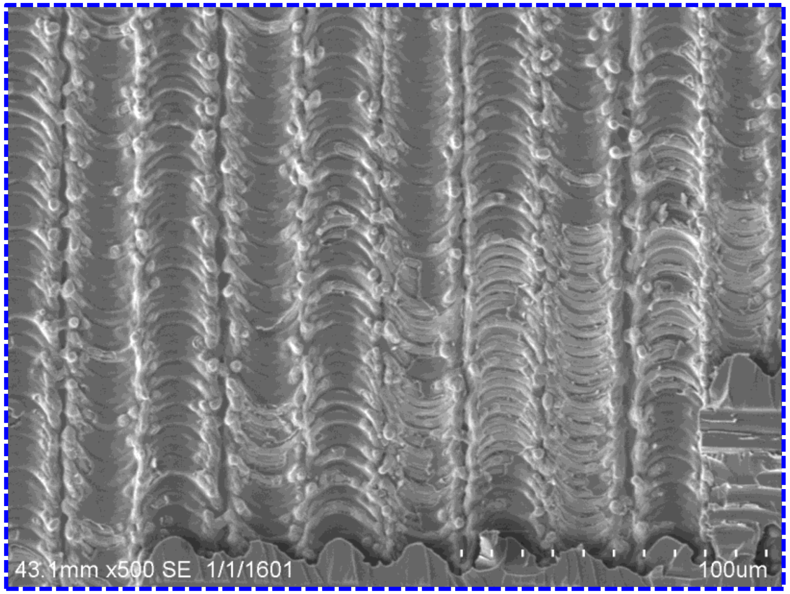
Loading rate: 0.02 in/min
Unloading rate: 0.1 in/min

After each test (1 loading-unloading cycle), the specimen is UT scanned for accurate measurement of the crack length.

SEM Fracture Surface
(Ti side, 1 hr Hydrolyzed, RT DCB)





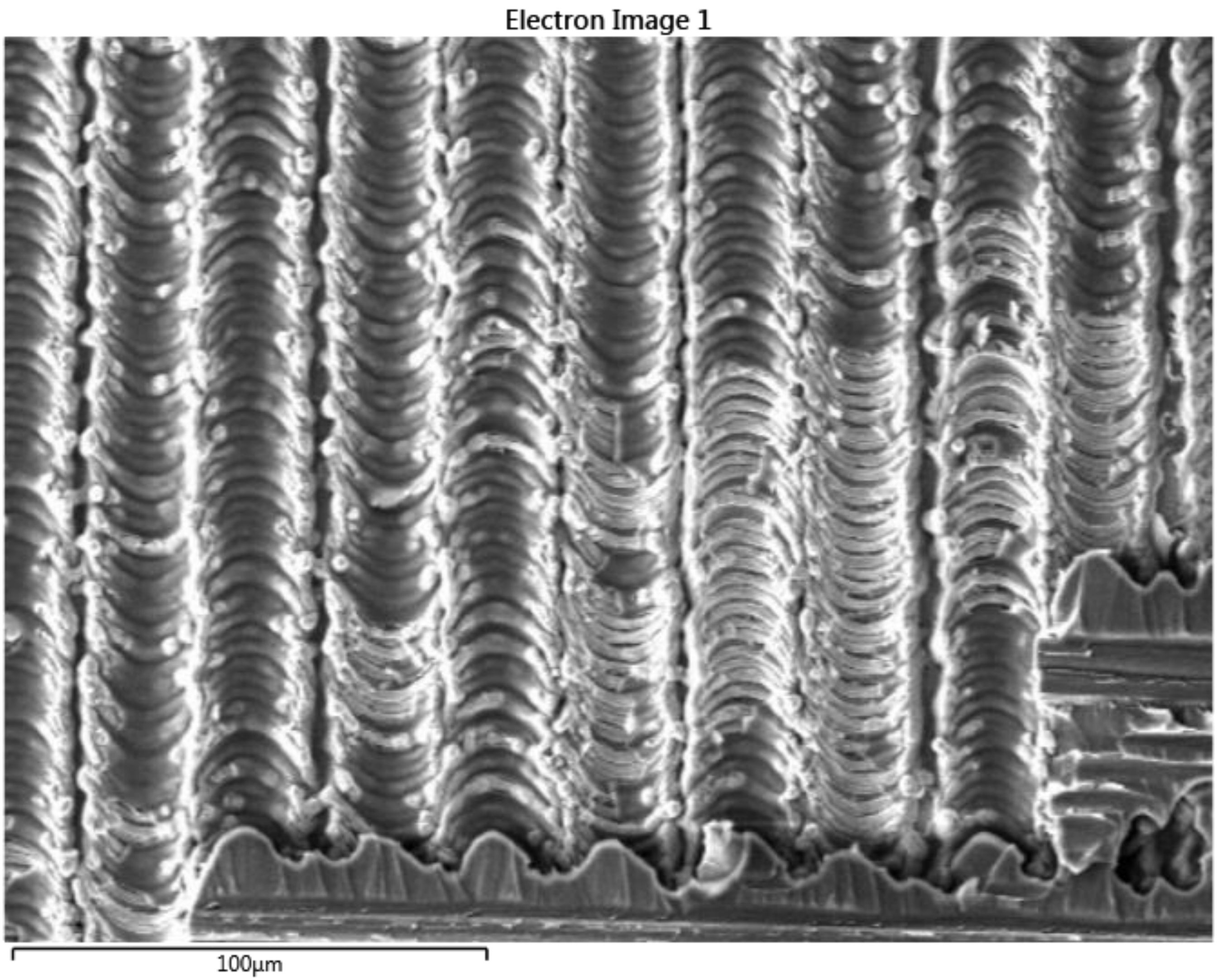


Crack growth direction

Area where elemental mapping obtained using EDS

EDS Fracture Surface (RT DCB specimen)

Sample with sol-gel hydrolyzed ~1 hr



EDS Elemental Mapping (RT 1 hr Hydrolyzed DCB Specimen)