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

John Connell and Frank Palmieri
NASA Langley Research Center, Hampton, VA
Hieu Truong, Ozden Ochoa and Dimitris Lagoudas
Texas A&M University, College Station, TX

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Presentation Outline

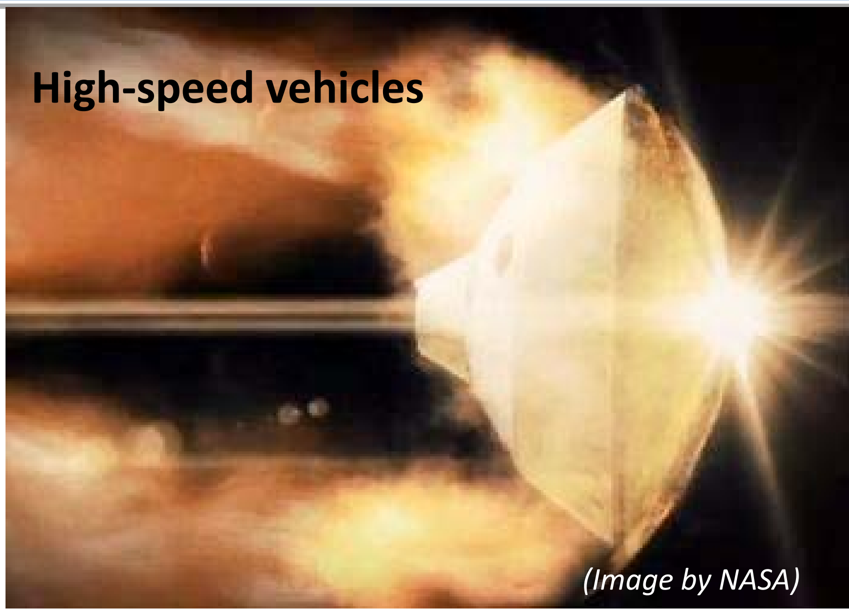
- Motivation
- Objectives
- Processing and Experimental Approach
  - Surface Treatment
  - Hybrid Laminate Fabrication
  - Fracture Toughness Tests with in-situ digital image correlation (DIC)
- Results
- On-going Work and Conclusion

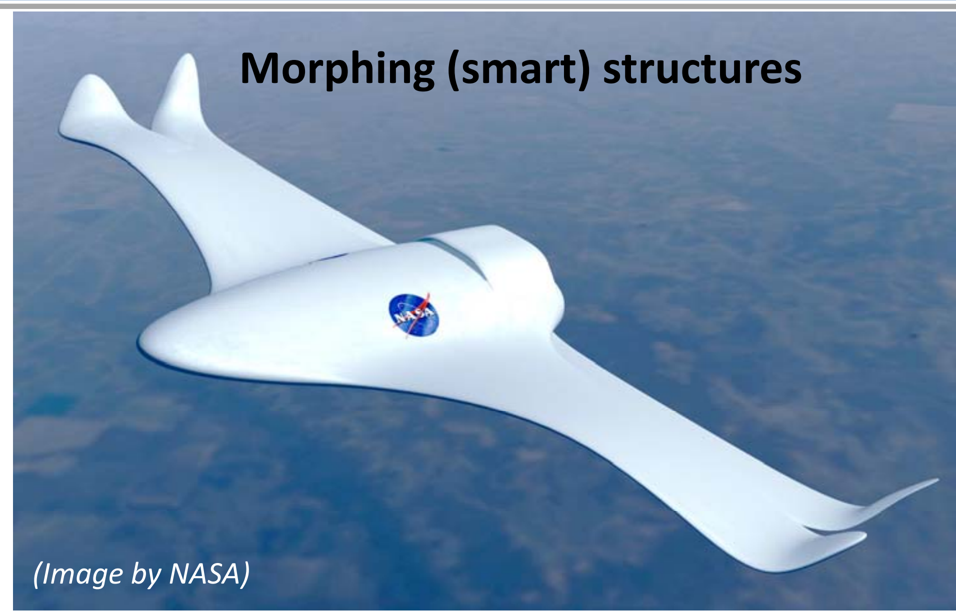
Outline

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

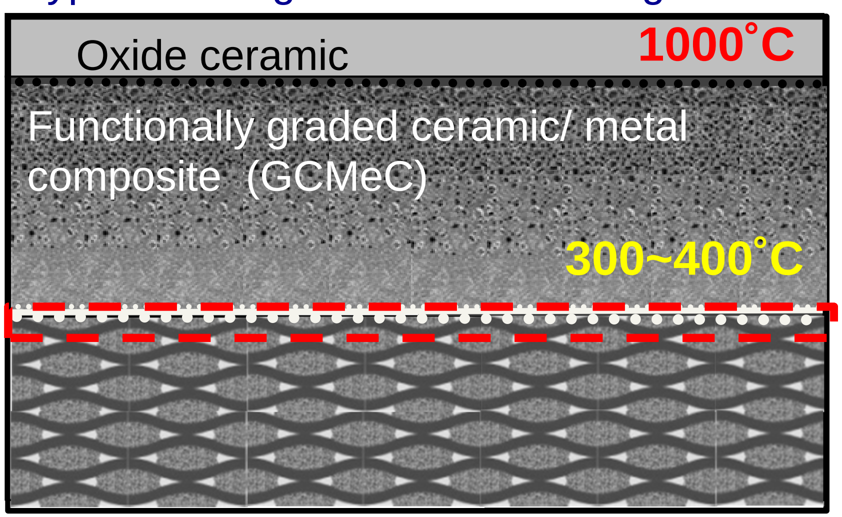
Approach: Fabricate double cantilever beam (DCB) and end notched flexure (ENF) specimens and conduct mechanical tests at elevated temperatures.

Motivation



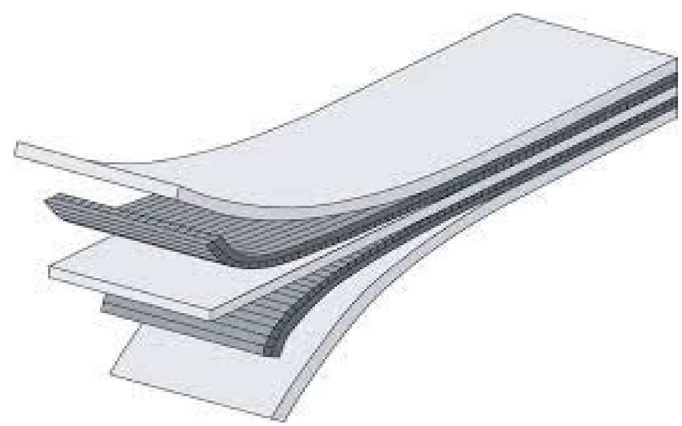


Hypersonic flight: frictional heating



(Courtesy of AFOSR-MURI18 -2009)

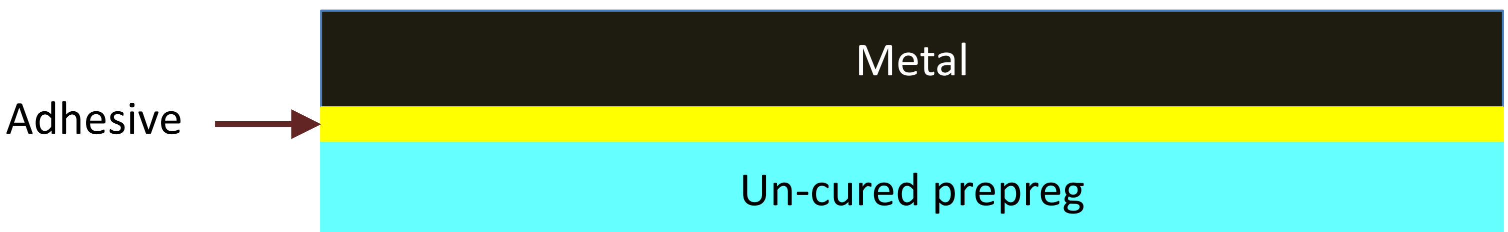
Fiber metal (hybrid) laminates



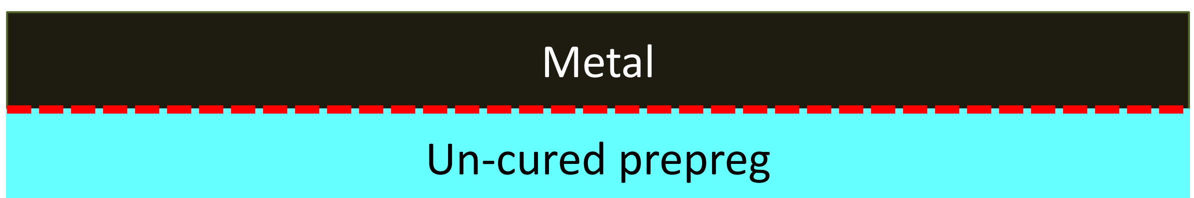
Composite Bonding Classifications

- 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
  - **NiTi (127 μm thick)**
  - ***Ti (127 μm thick)***
  - *Al (400 μm thick)*

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

Metallic surface treatment:

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

Current work: Layup and Sol-Gel Chemistry

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

PMC:

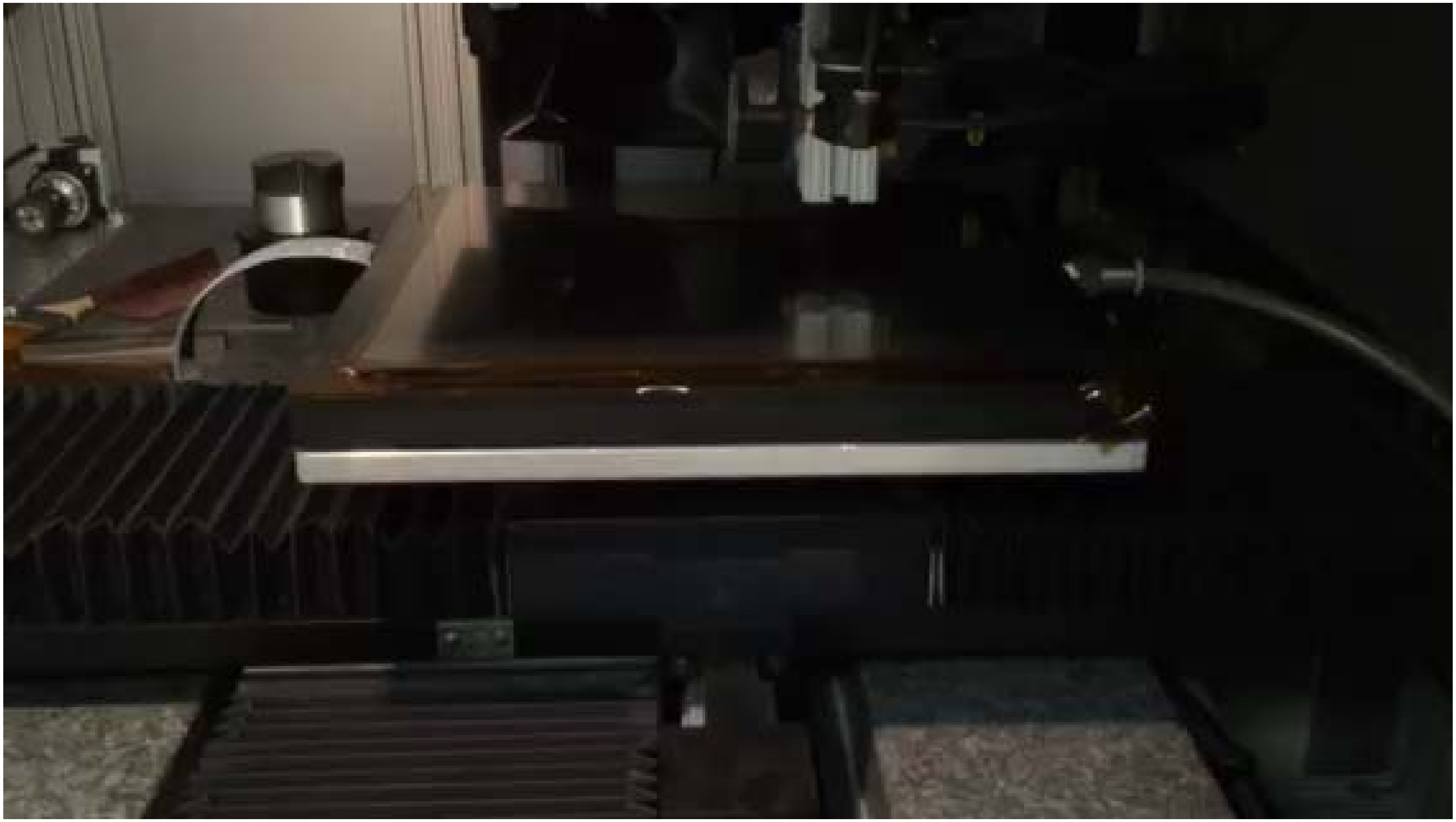
- Reinforcement: T650 carbon fiber, 8-harness satin weave
- Matrix: AFR-PE-4 polyimide resin

(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

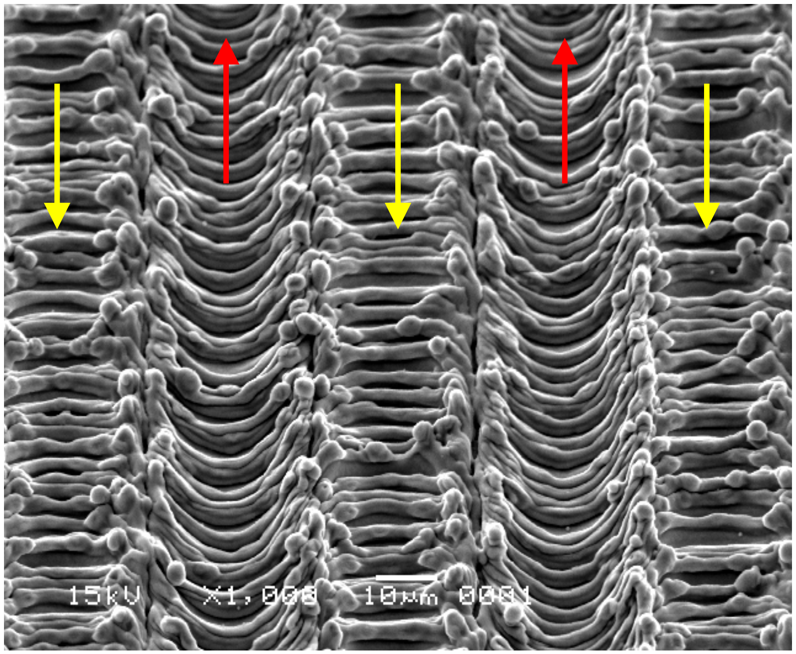
Laser ablation of Ti and NiTi foils



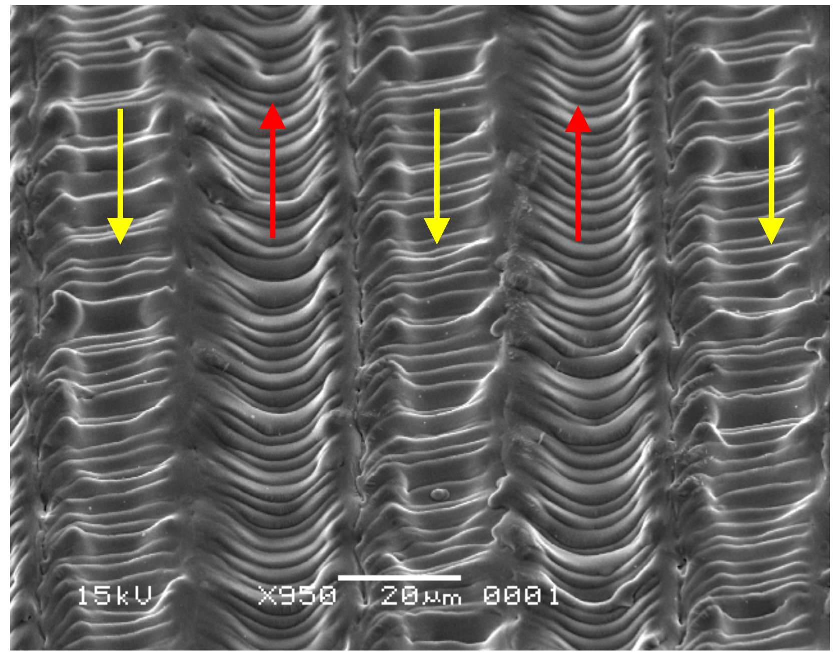
Selected laser ablation parameters: 80 kHz, 1.5W, 1 mil pitch

Surface of Ti and NiTi foil after laser ablation

Laser ablated Ti surface



Laser ablated NiTi surface

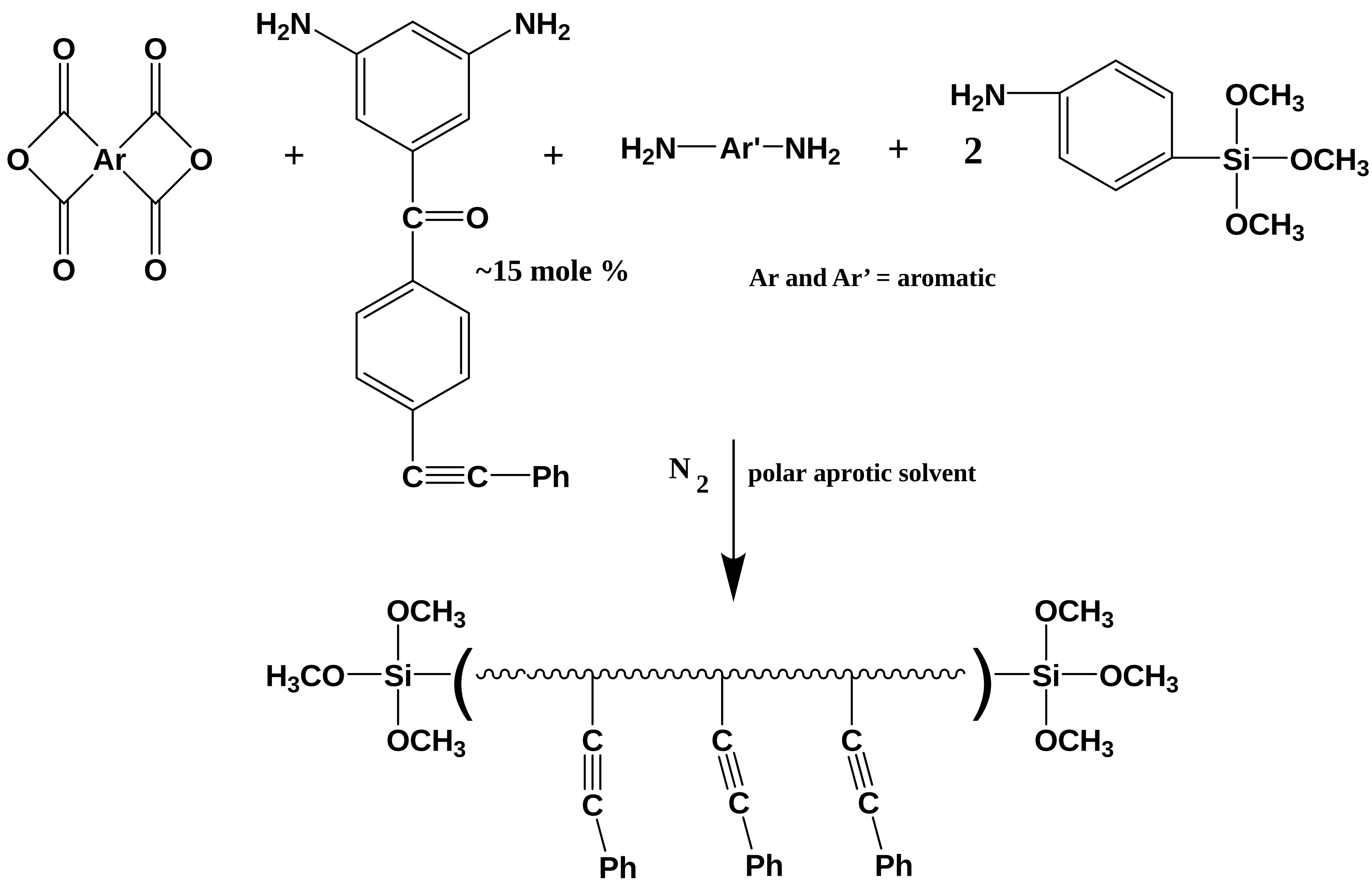


Arrows denote laser path direction

Laser ablation performed on Ti and NiTi surfaces:

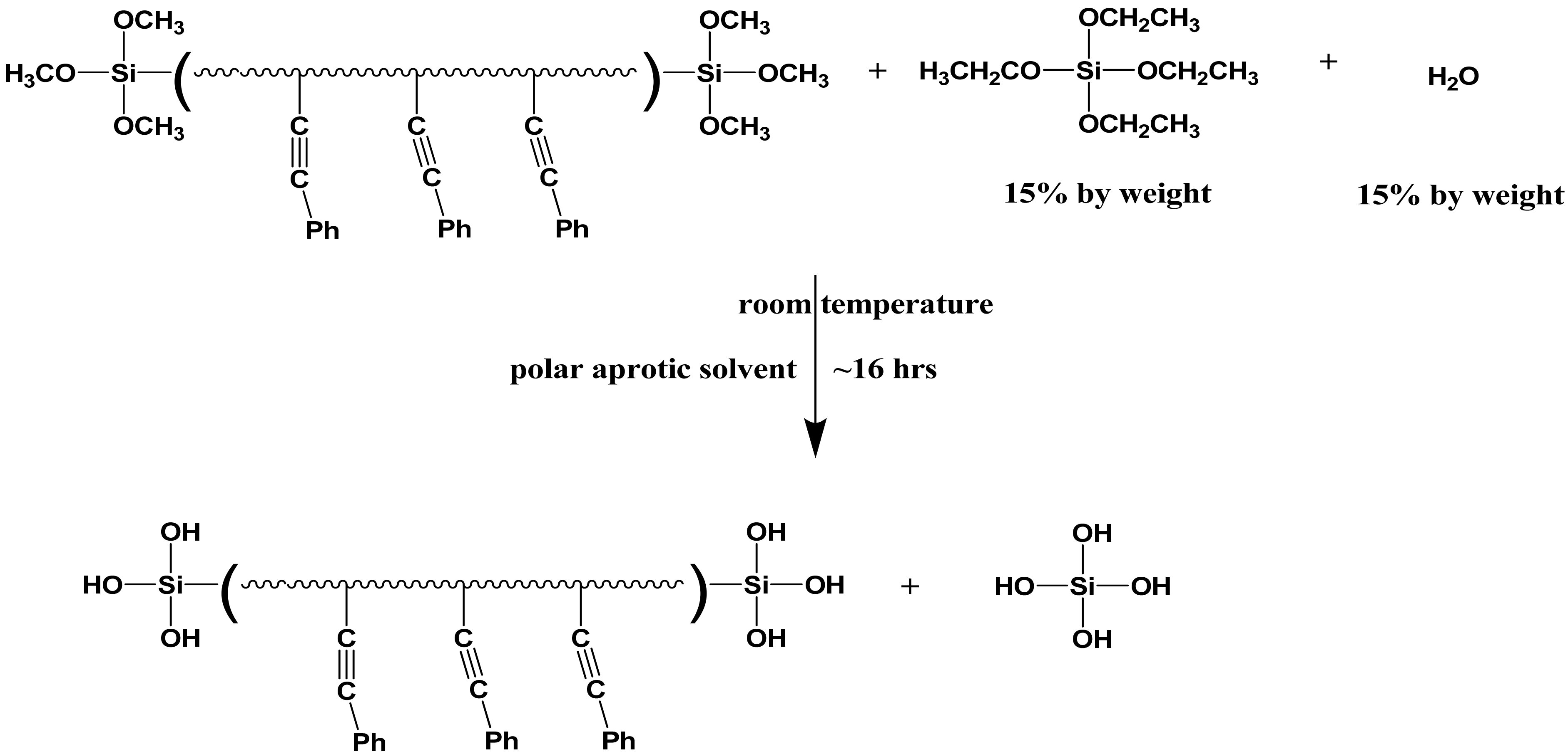
- Clean oxide layer on the metal surfaces was formed
- Created patterned micro-roughness on the metal surface
- Increased surface wettability
- No detrimental morphology or chemical species was formed

Synthesis of Amide Acid Sol-Gel Surface Treatment



Amide acid oligomer ~2500 g/mole

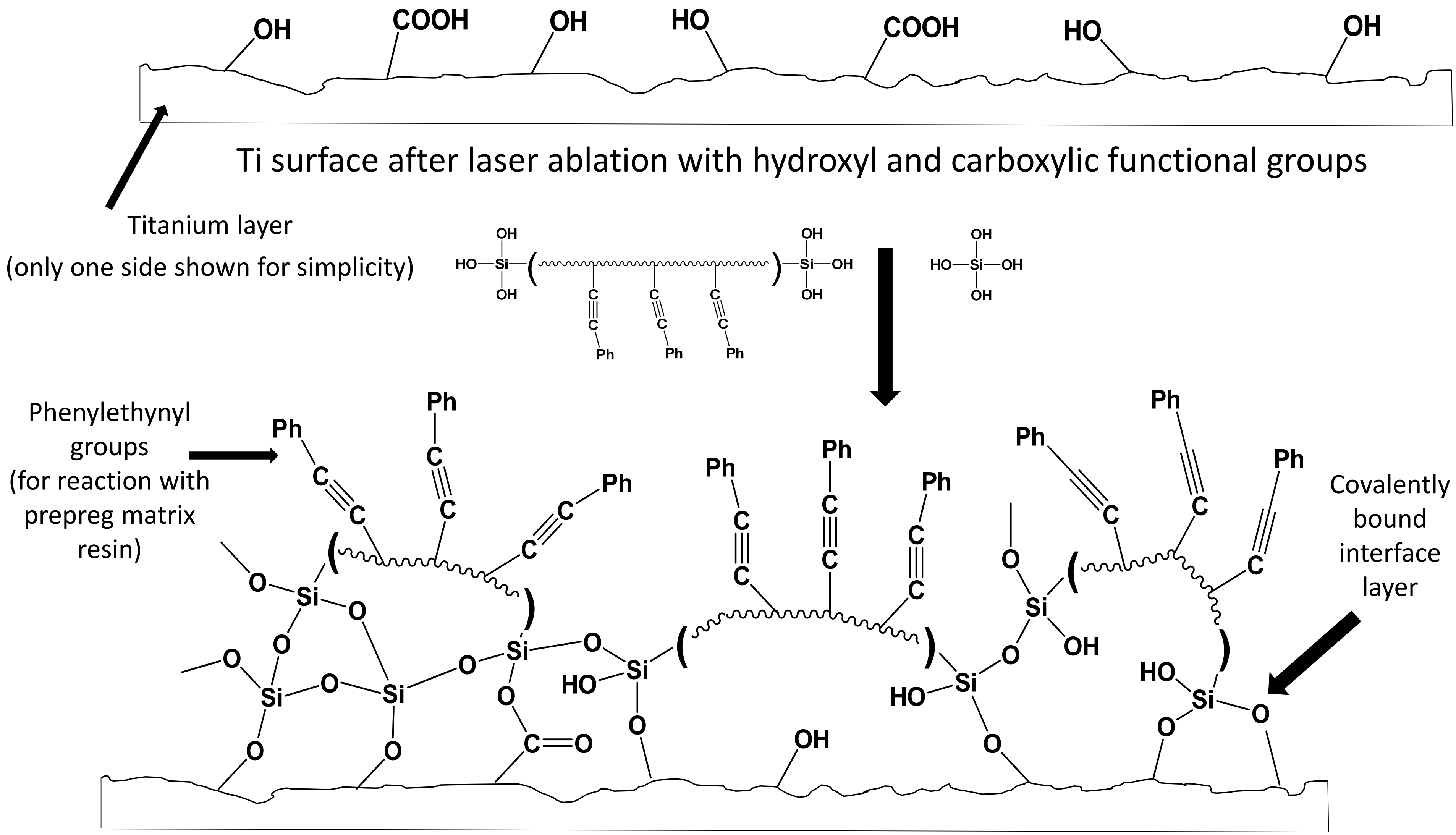
Synthesis of Amide Acid Sol-Gel Surface Treatment



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).

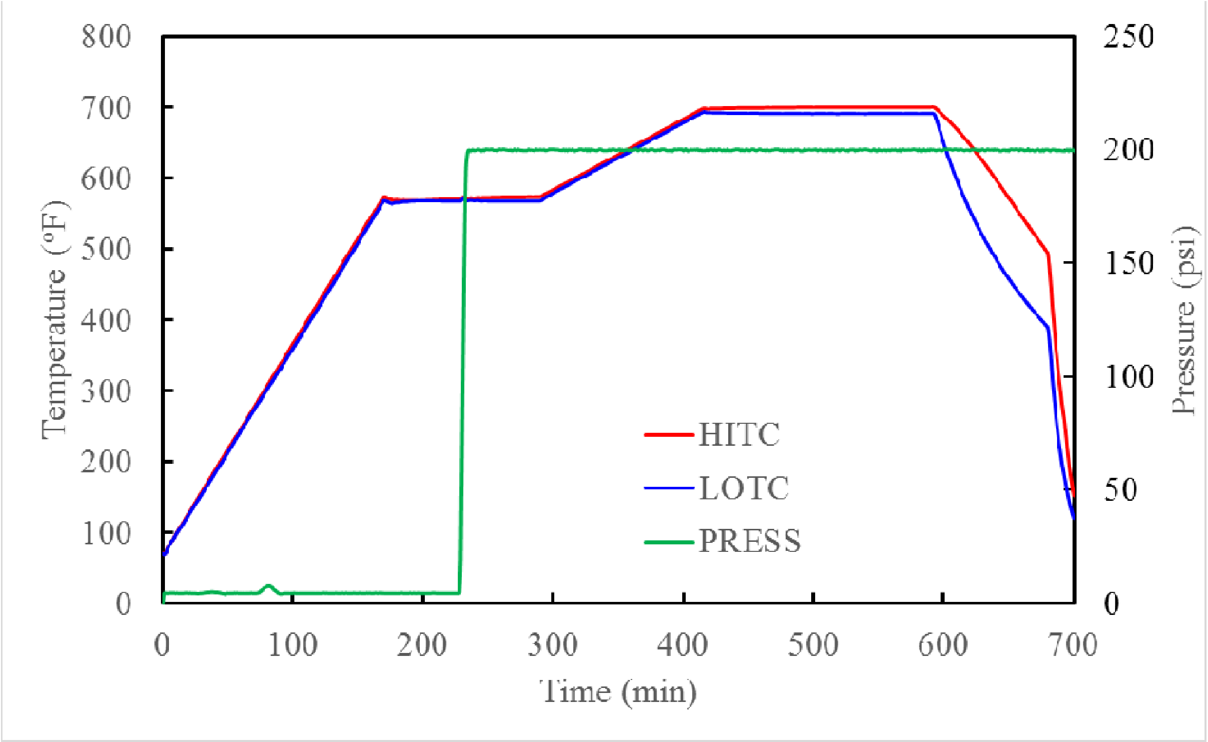
Representation of Ti Surface Treatment Chemistry



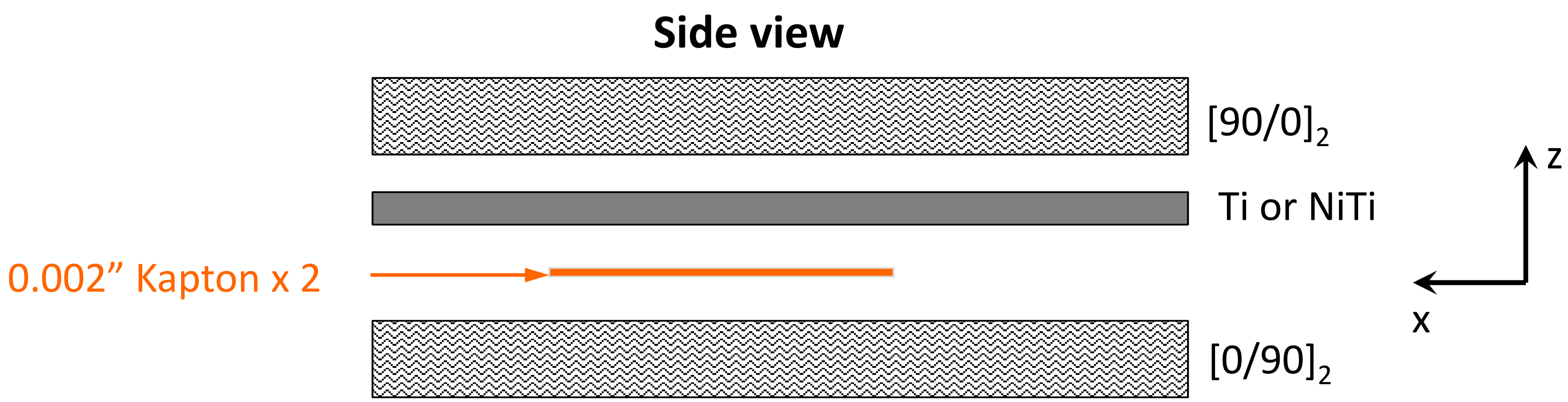
Frank L. Palmieri, Kent A. Watson, Guillermo Morales, Thomas Williams, Robert Hicks, Christopher J. Wohl, John W. Hopkins, and John W. Connell, "Laser Ablative Surface Treatment for Enhanced Bonding of Ti-6Al-4V Alloy", ACS Applied Materials and Interfaces, 5(4), 1254-1261, 2013.

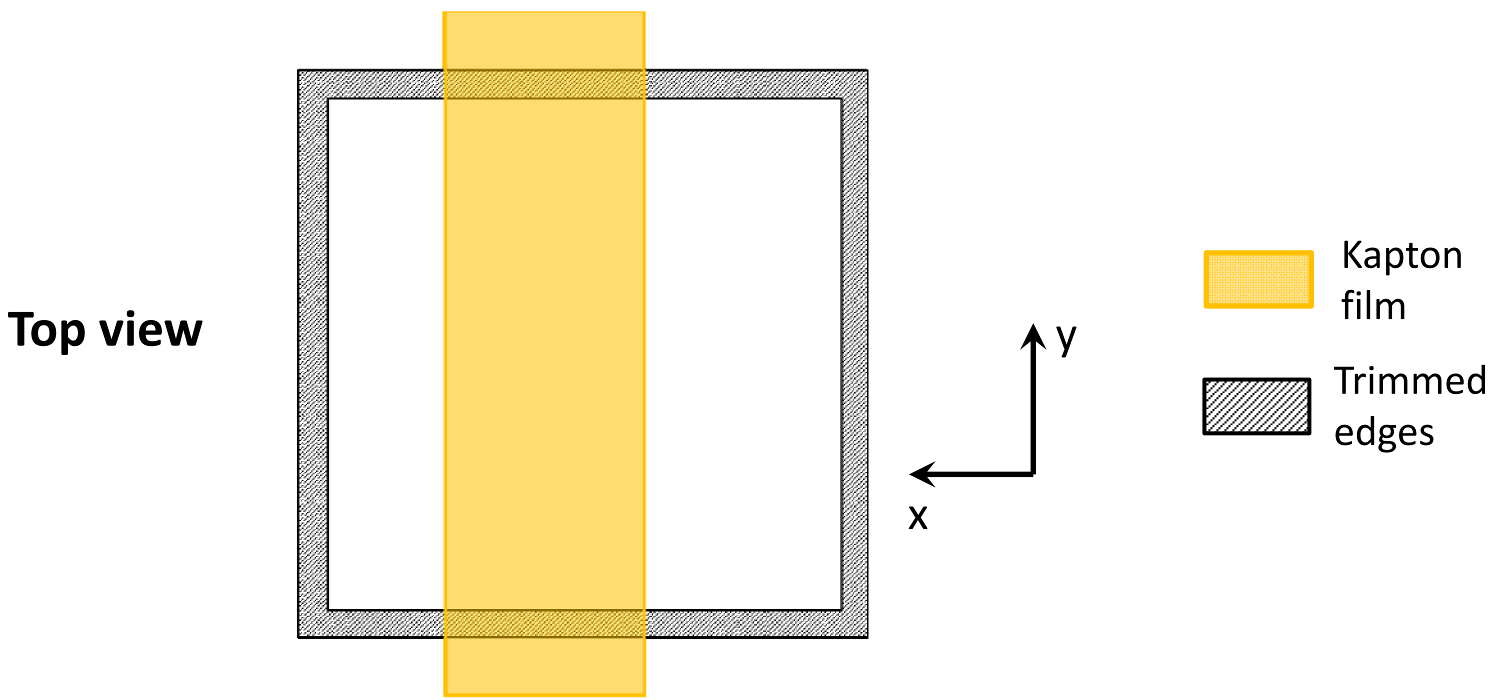
Fabrication Procedure

- Ti and NiTi foil (127 µm thick) were laser ablated and treated with sol-gel solution on both sides
- Panel preform was vacuum-bagged overnight
- Curing was done in a Wabash vacuum press (12 hr run)



Schematic of the hybrid panel

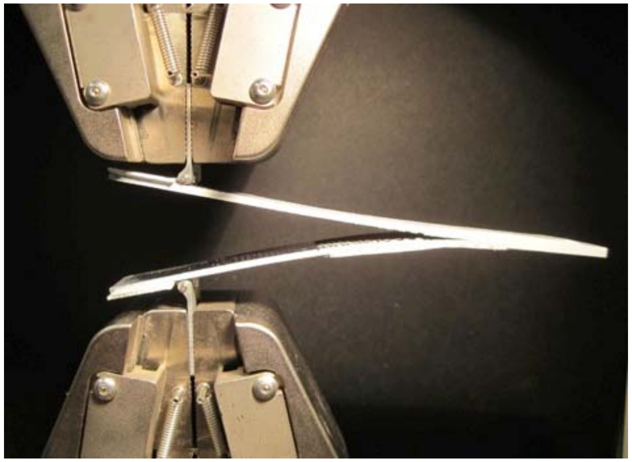




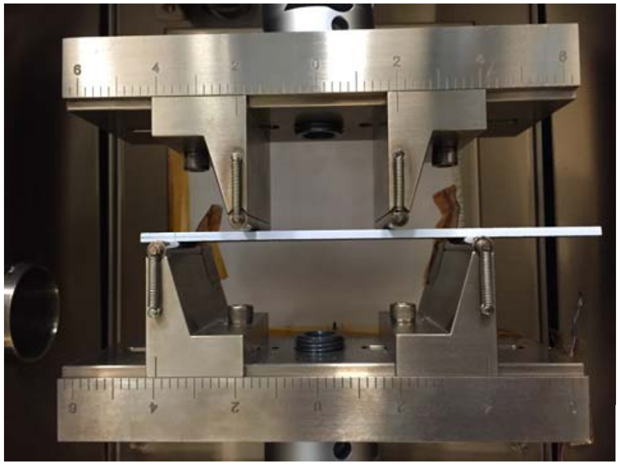
Characterization

Mode I and Mode II fracture toughness tests

DCB Test

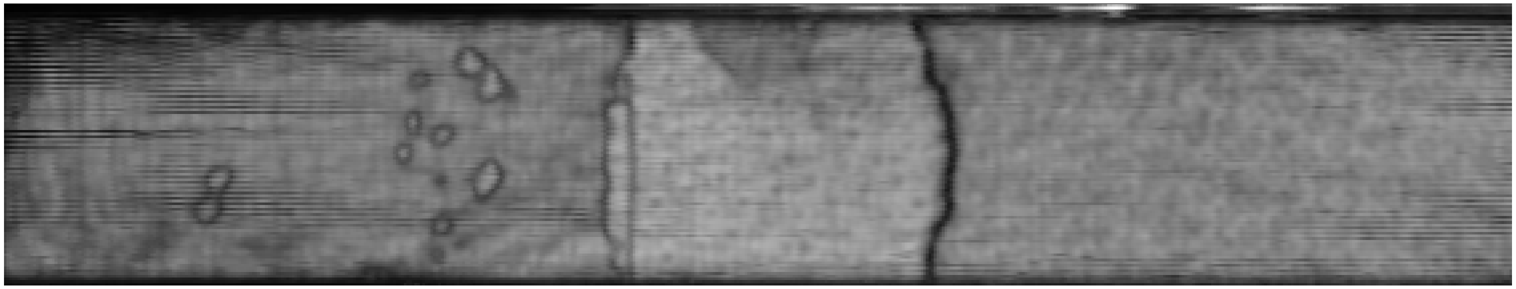


4-ENF Test



Fracture toughness tests carried out at RT, 150°C, 250°C and 315°C

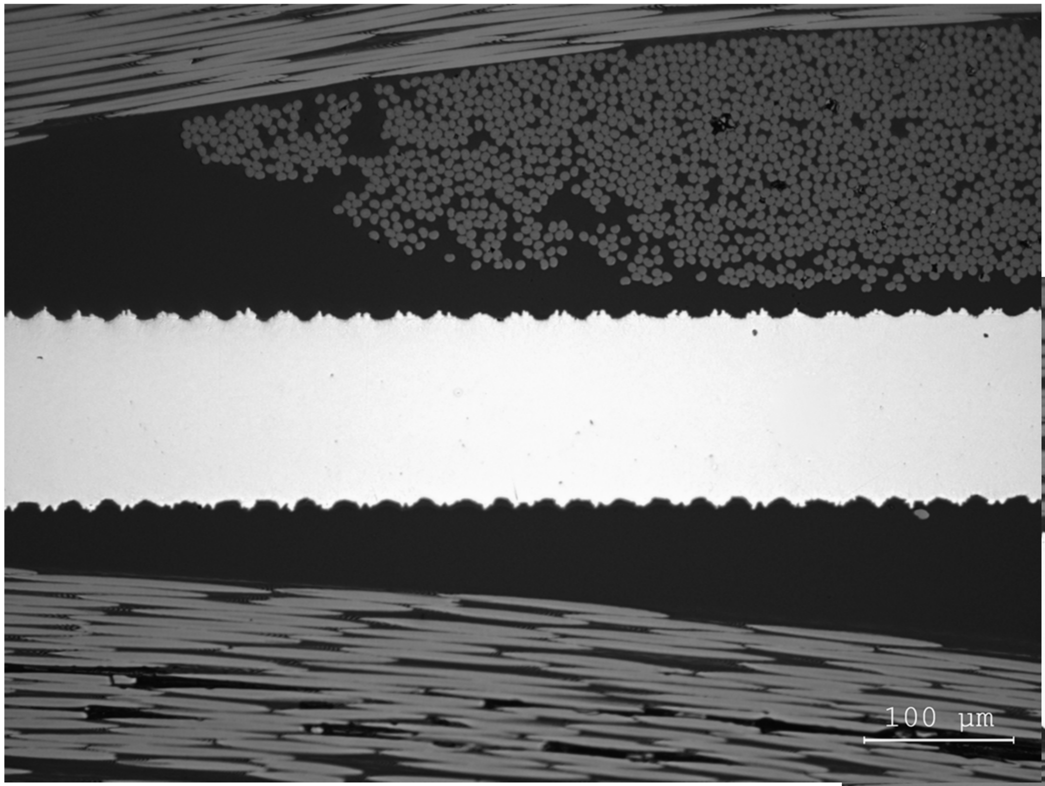
Monitor delamination growth with ultrasonic scanning



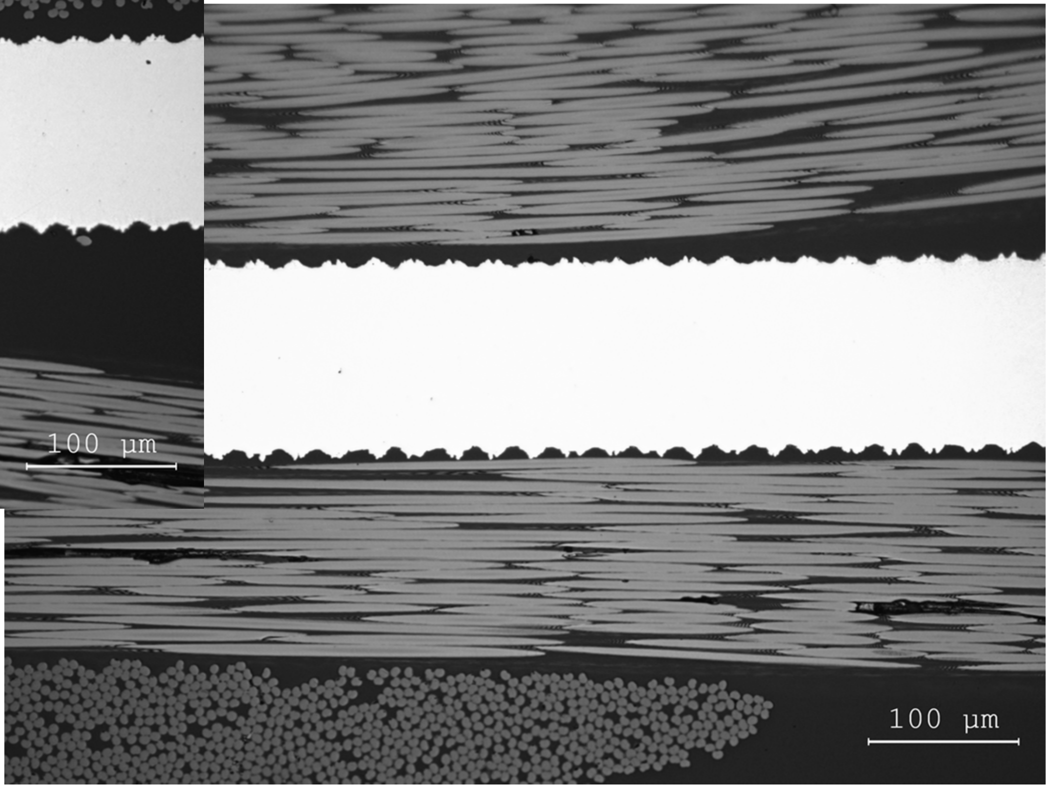
Other characterization techniques: TMA, DMA, FTIR, optical and electron microscopy

Cross-section of laser ablated Ti-PMC interface

Interfaces near resin-rich regions



Interfaces where fiber tows are adjacent to Ti surfaces

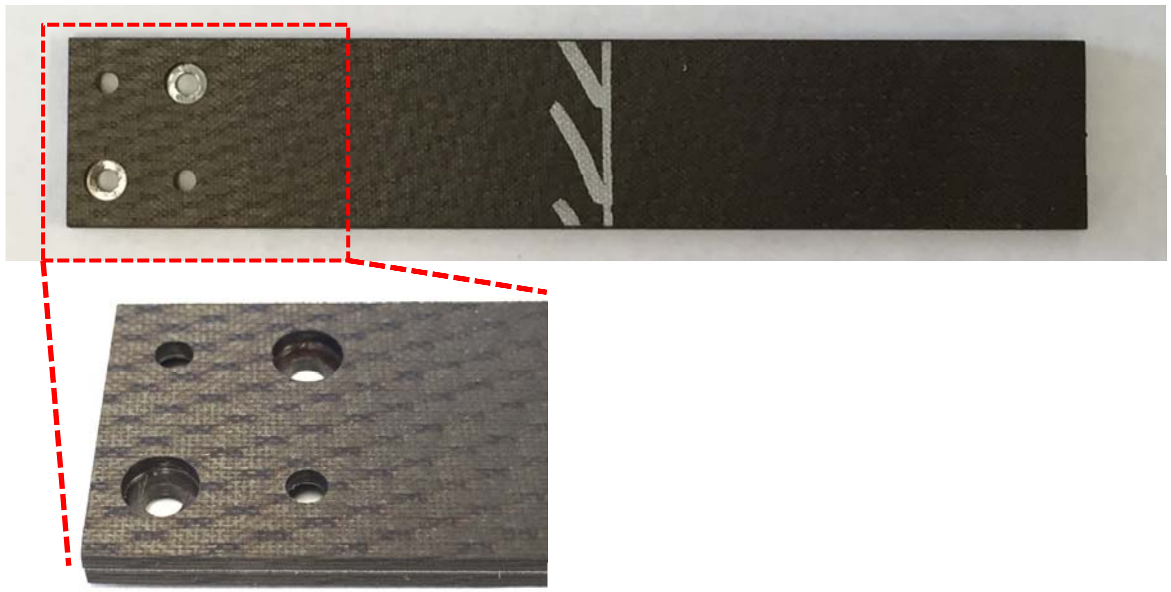


Mode I Fracture Toughness Test: Double Cantilever Beam test with in-situ digital image correlation (DIC)

DCB Specimens for High Temperature Tests

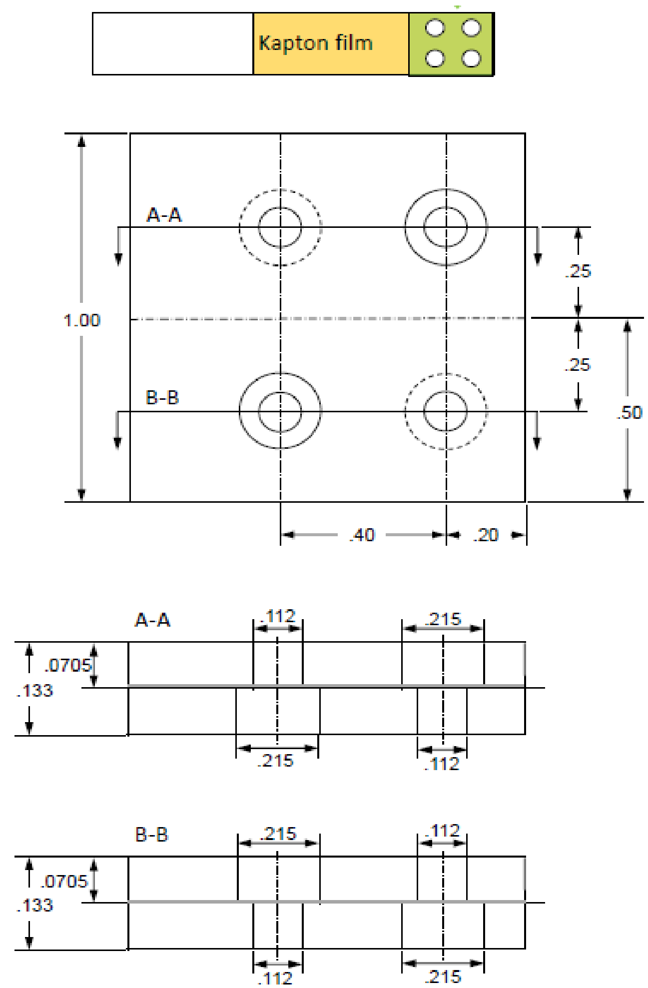
Hinges are mechanically attached to DCB using screws
to facilitate high temperature testing

Top view drilled DCB specimens



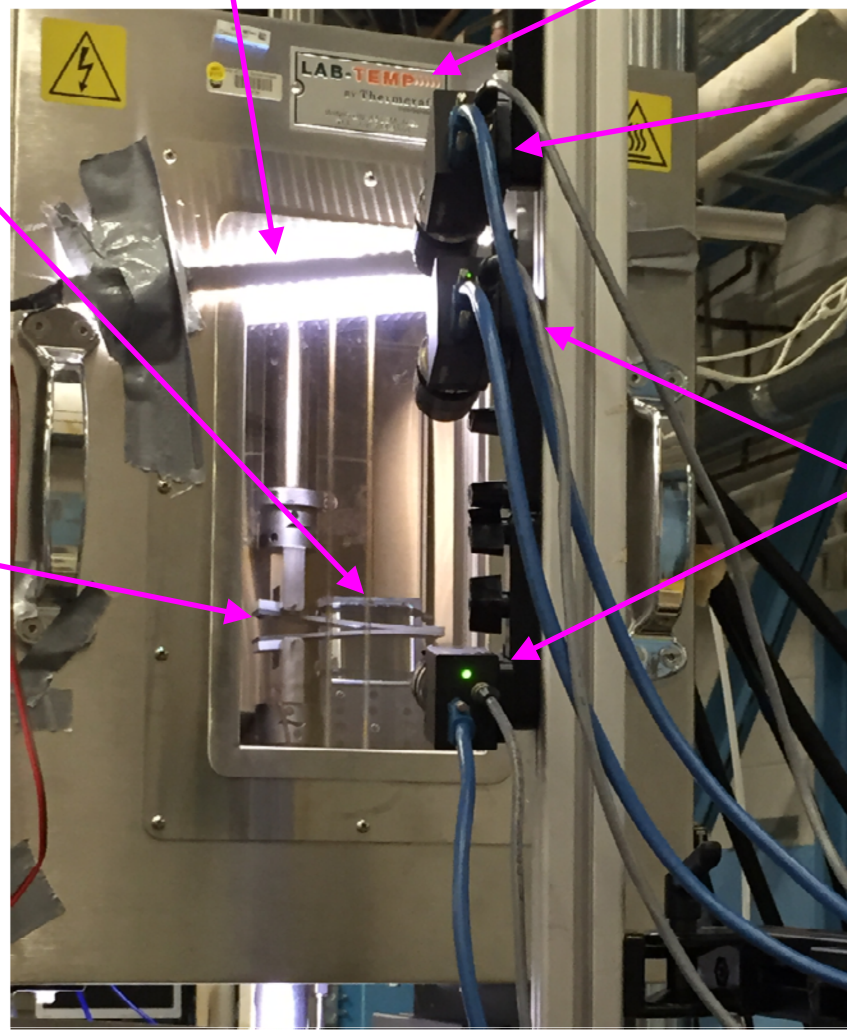
DCB with hinges attached

Sketch of for drilling of DCB specimens



Units are in inches

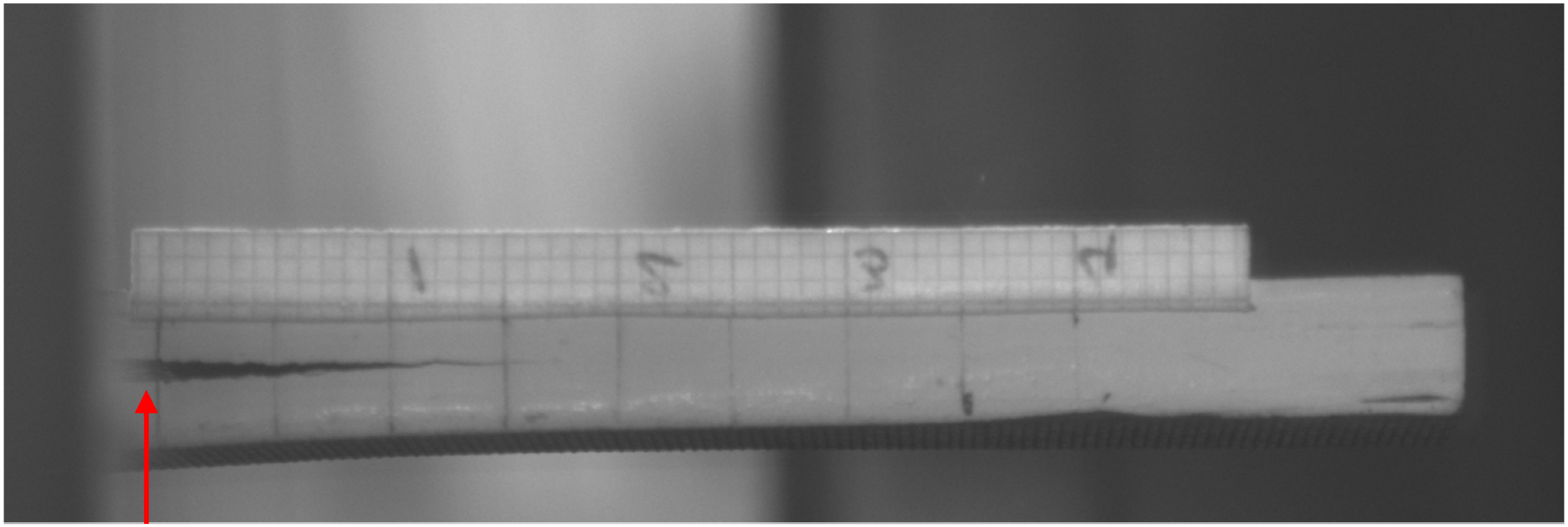
DCB Test Setup with in-situ DIC



Crack Growth Visual Monitoring

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

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 real specimen's back side.

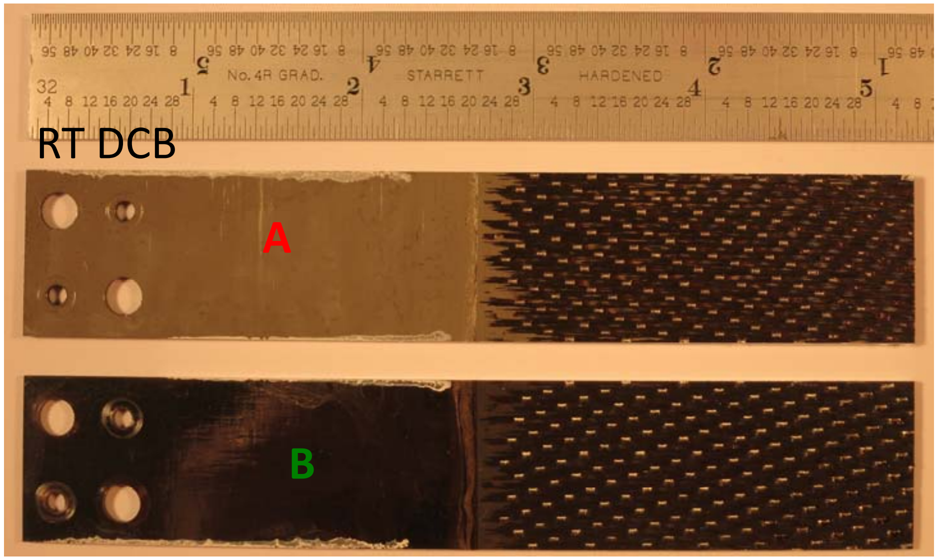


End of pre-crack location

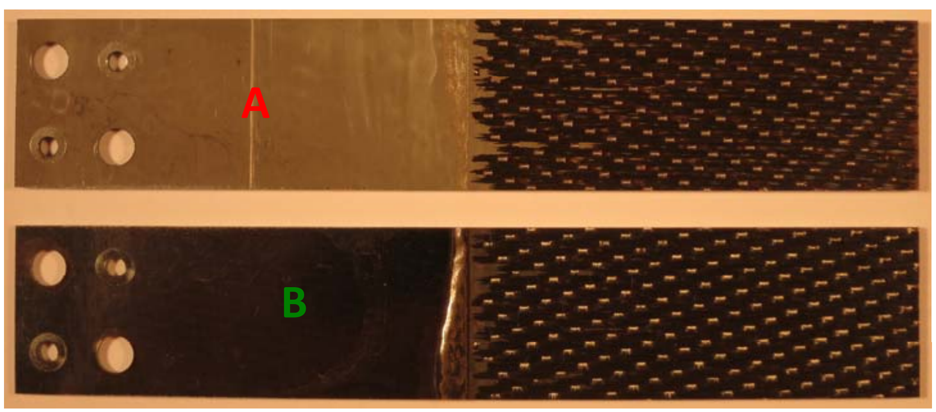
Fracture Surface (1 hr hydrolyzed)

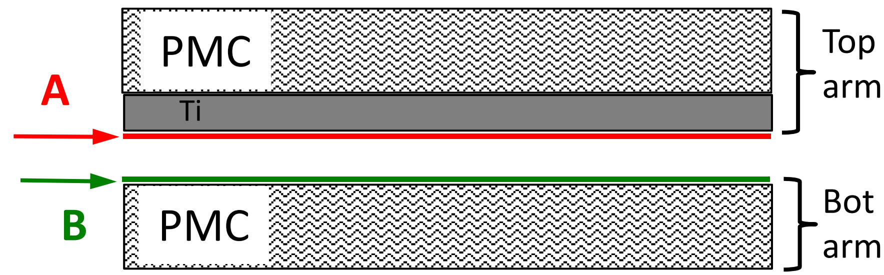
HT 09182015a

RT DCB

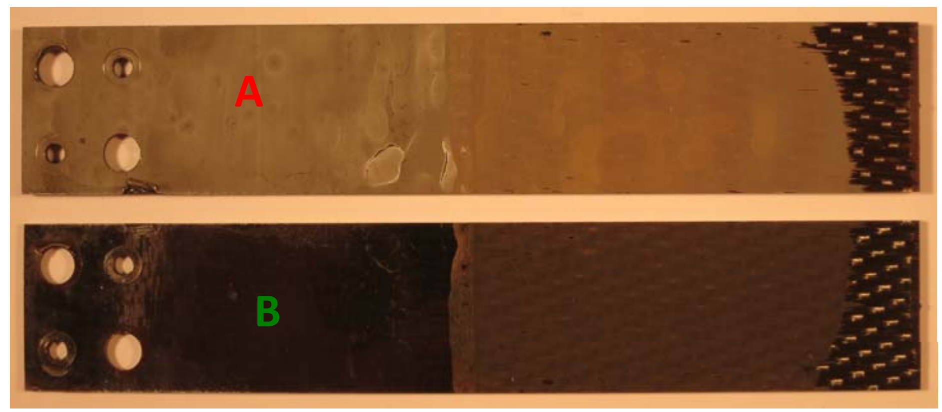


150ºC DCB



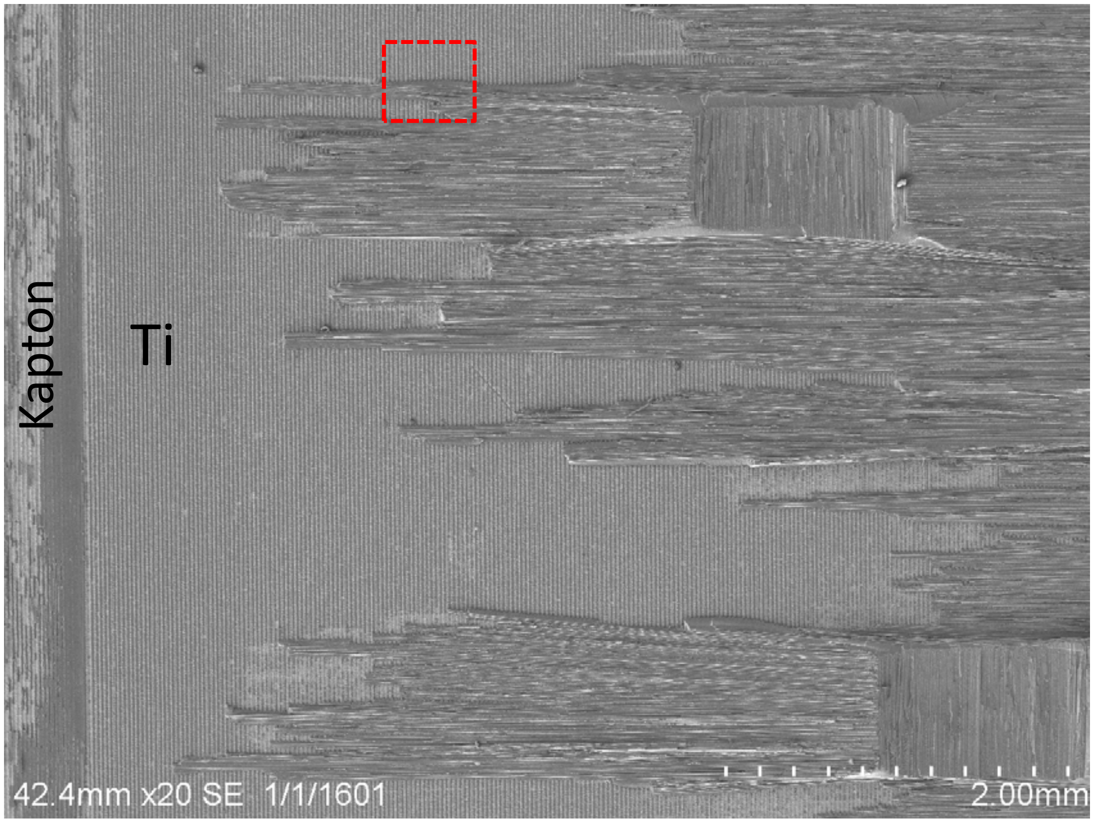


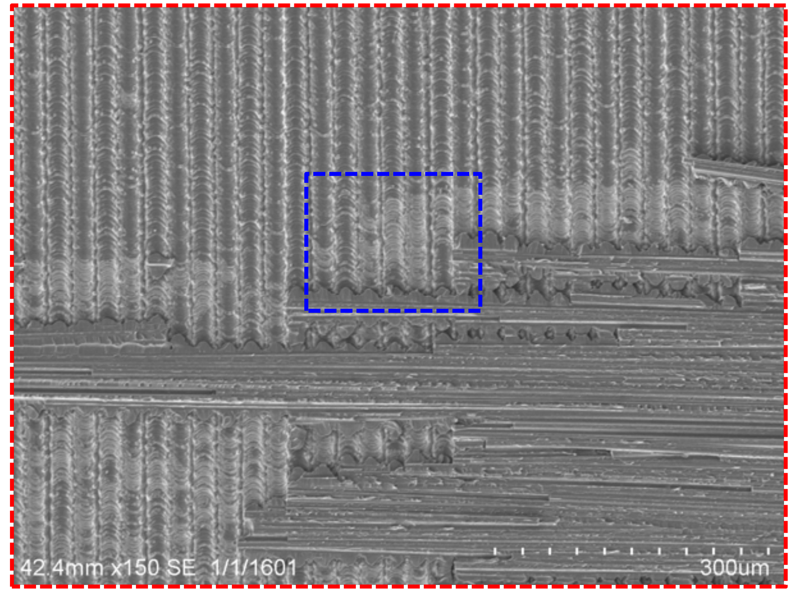
250ºC DCB



- Adhesion failure at 250ºC
- Cohesive failure at RT and 150ºC
    - Crack initiated at Ti surface then migrated to PMC

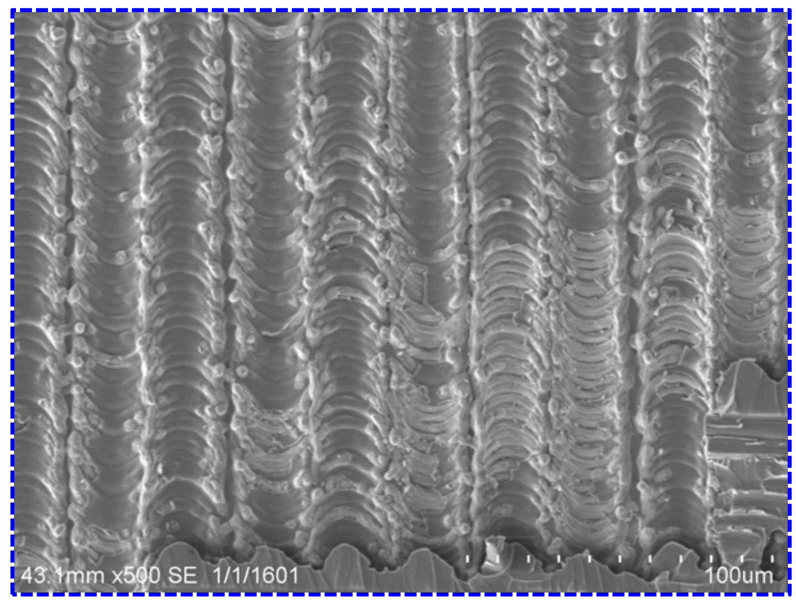
SEM Fracture Surface (Ti side, 1 hr hydrolyzed, RT DCB)





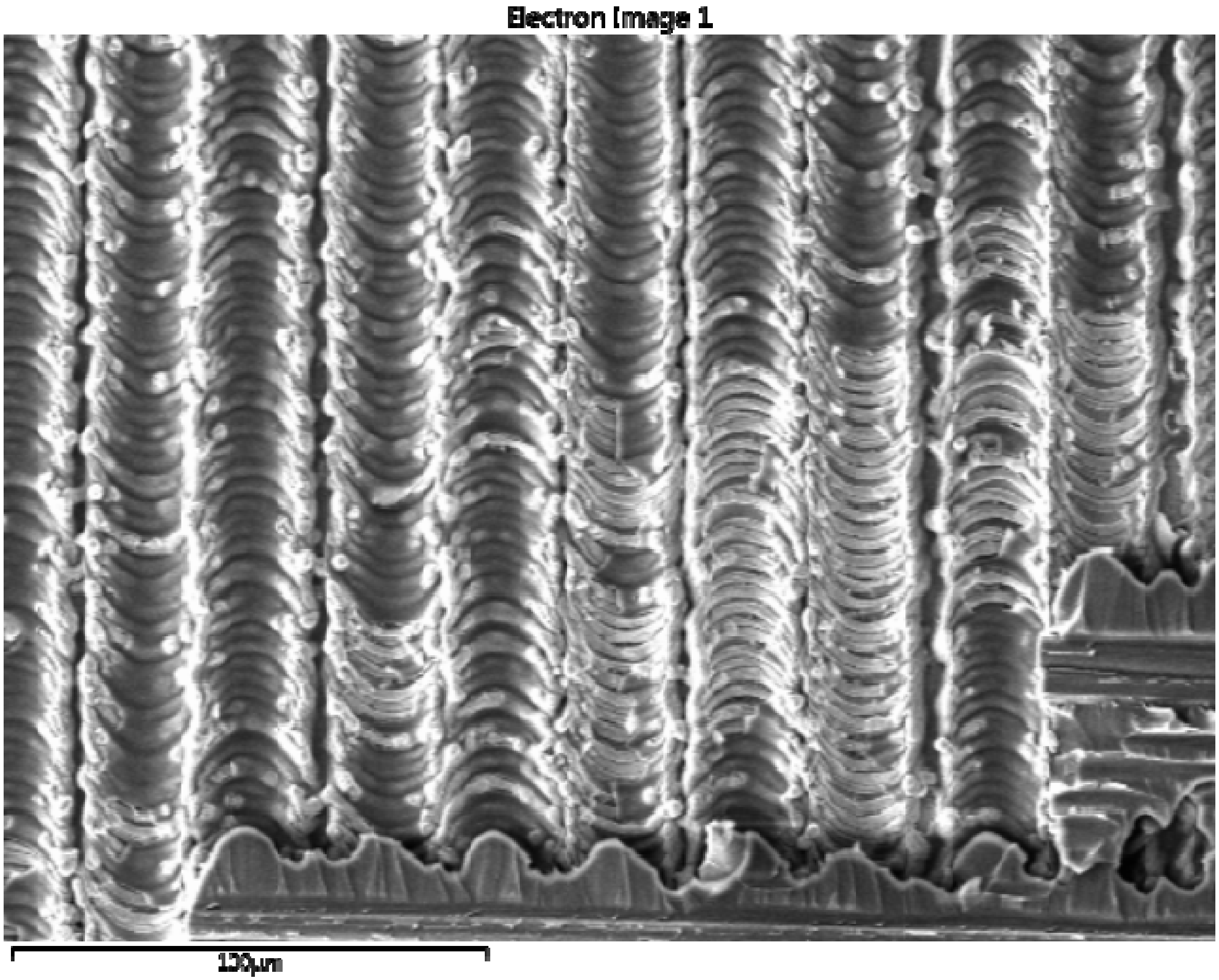
→ Crack growth direction

Area where elemental mapping obtained using EDS

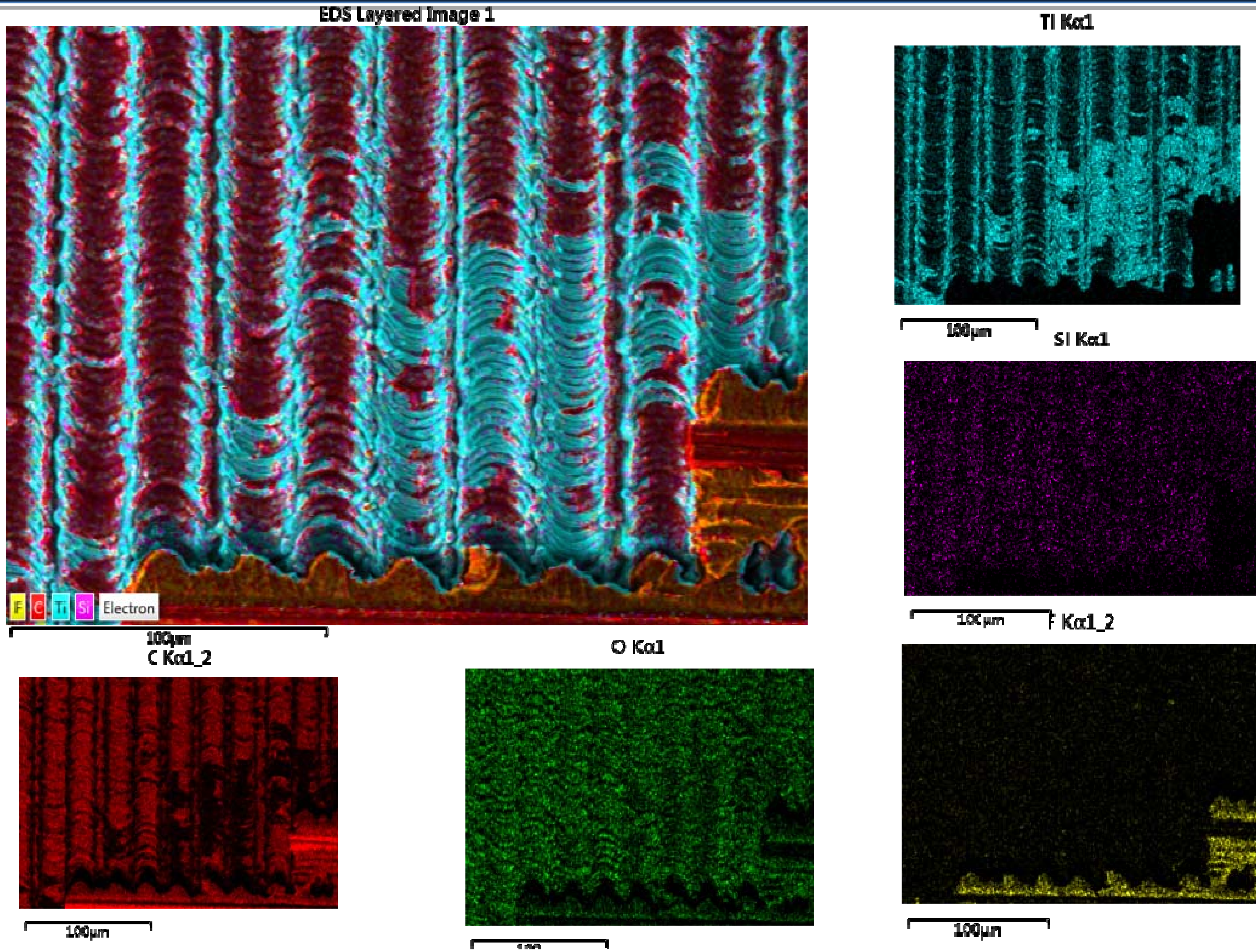


EDS Fracture Surface (RT DCB specimen)

Sample with sol-gel hydrolyzed ~1 hr

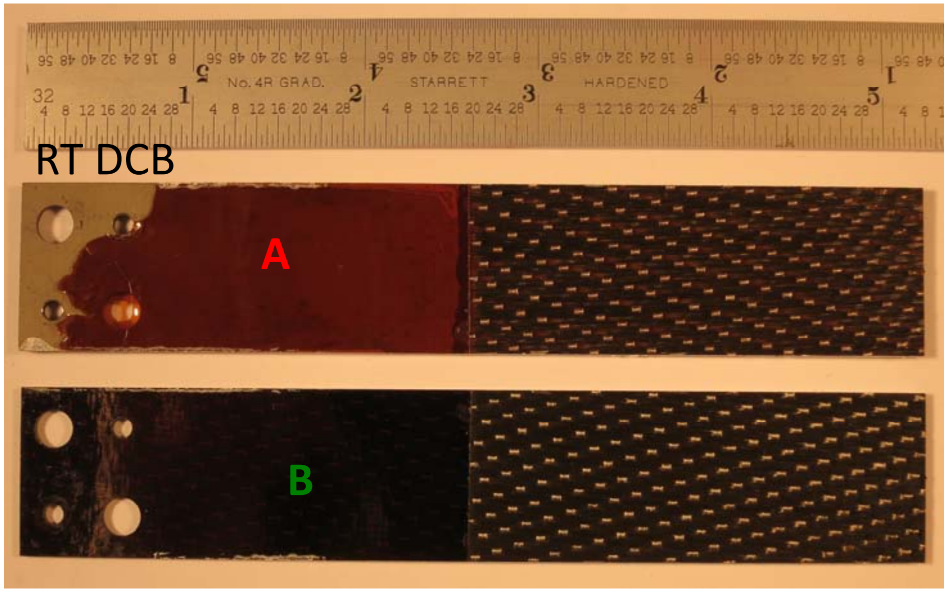


EDS Elemental Mapping (RT 1 hr hydrolyzed DCB specimen)

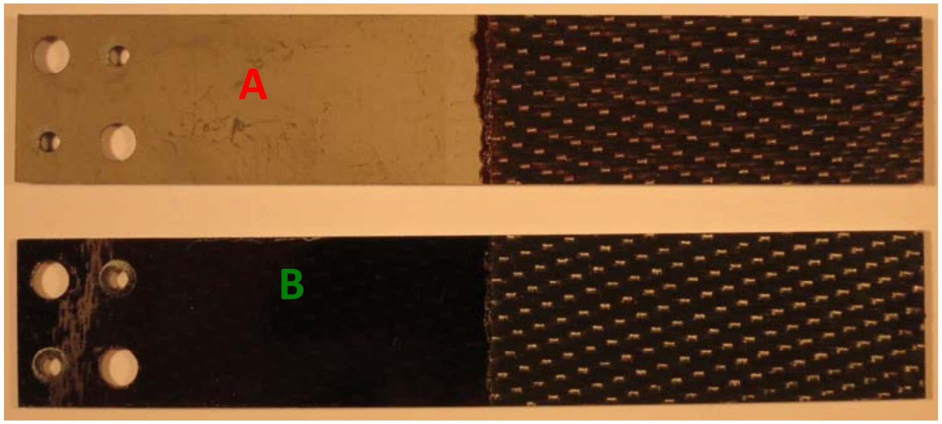


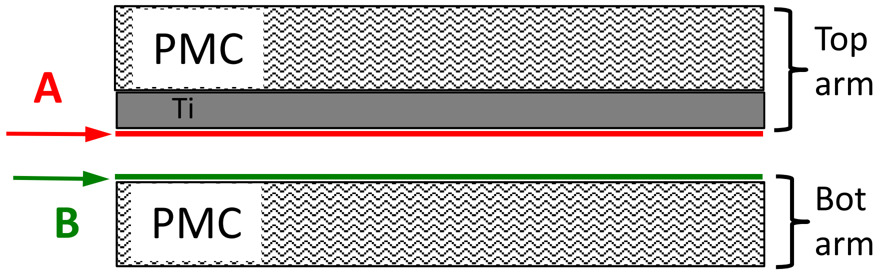
Fracture surface (16 hr hydrolyzed)

HT 10162015



150°C DCB

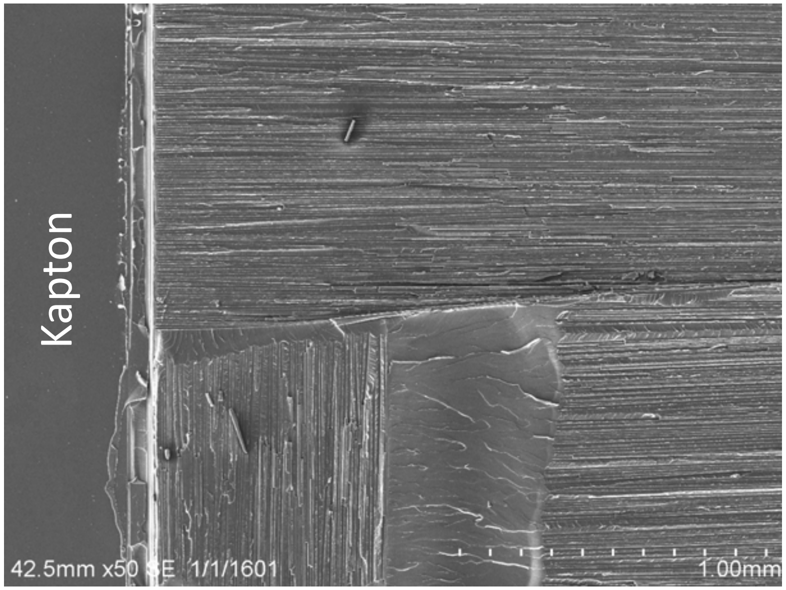


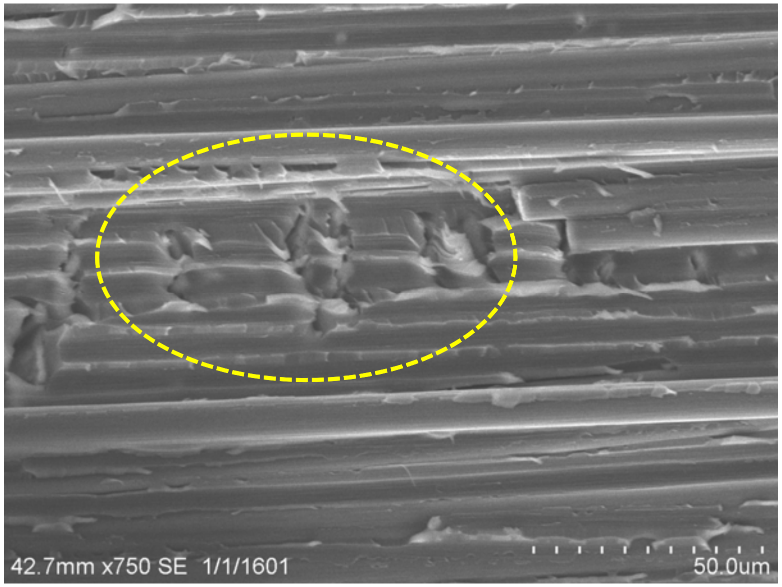


- Adhesive failure was observed at 315°C
- Cohesive failure at RT, 150°C and 250°C
  - Crack initiated in PMC side and remained in the same interface
  - No adhesive failure observed

SEM fracture surface (Ti side–RT and 150ºC DCB) (16 hr hydrolyzed)

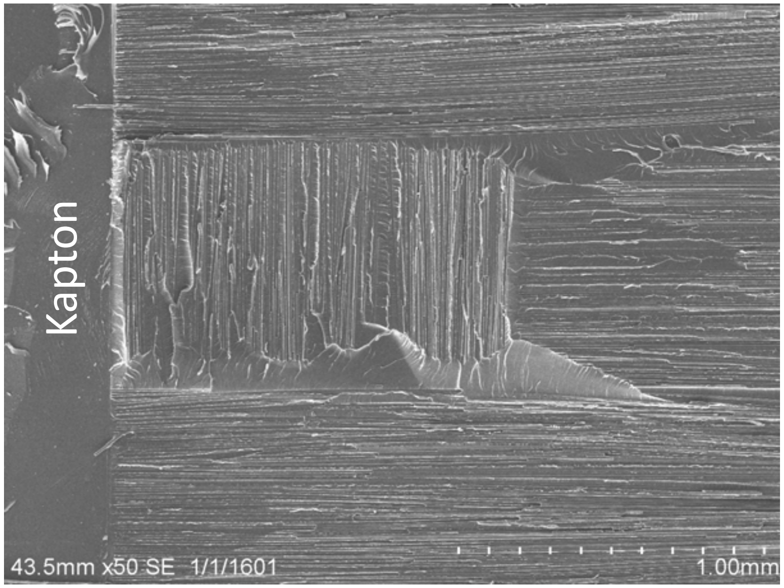
RT DCB

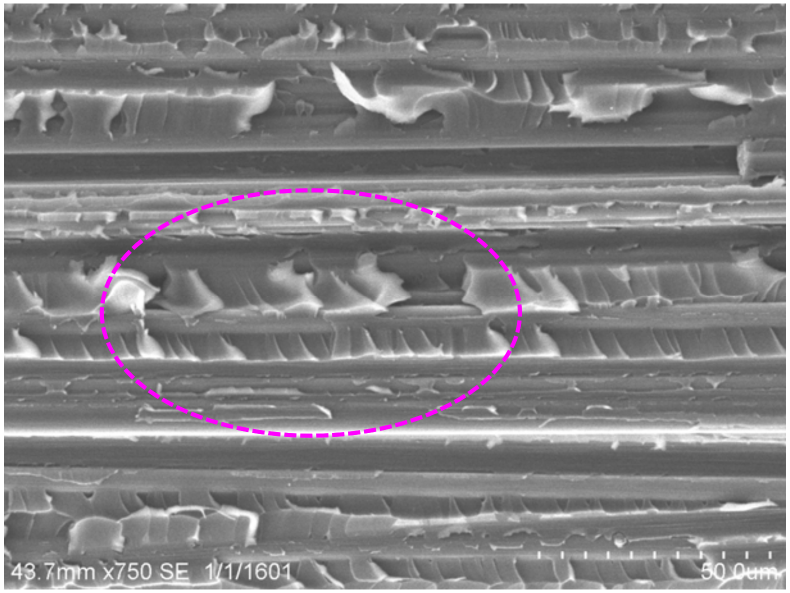




Micro-crack due to stress concentration at the troughs of laser ablated pattern on Ti surface

150ºC DCB



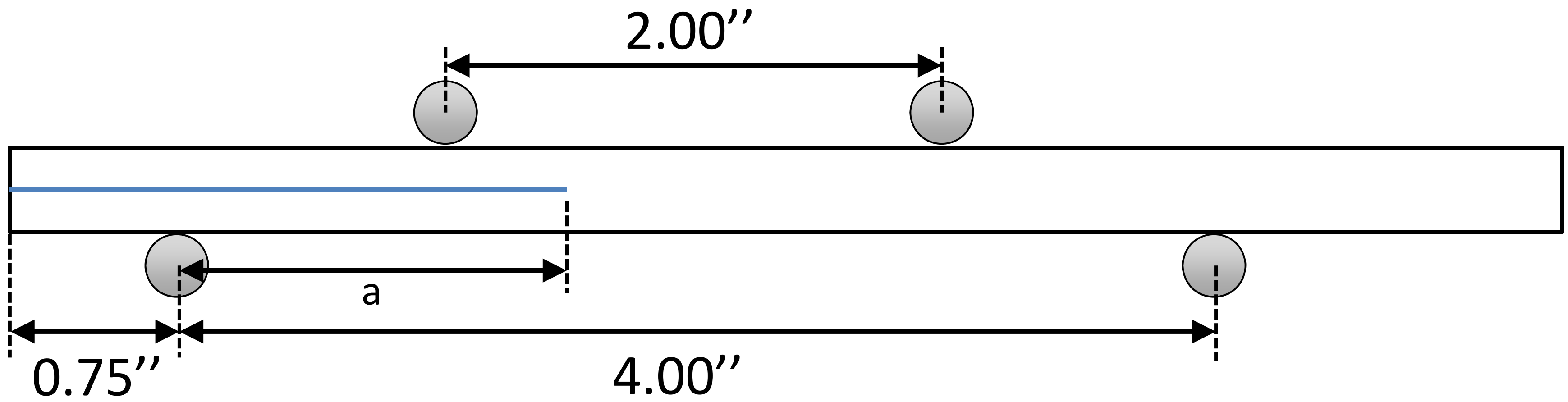


More upright cusps at HT due to matrix softening and shearing at fiber-matrix interface

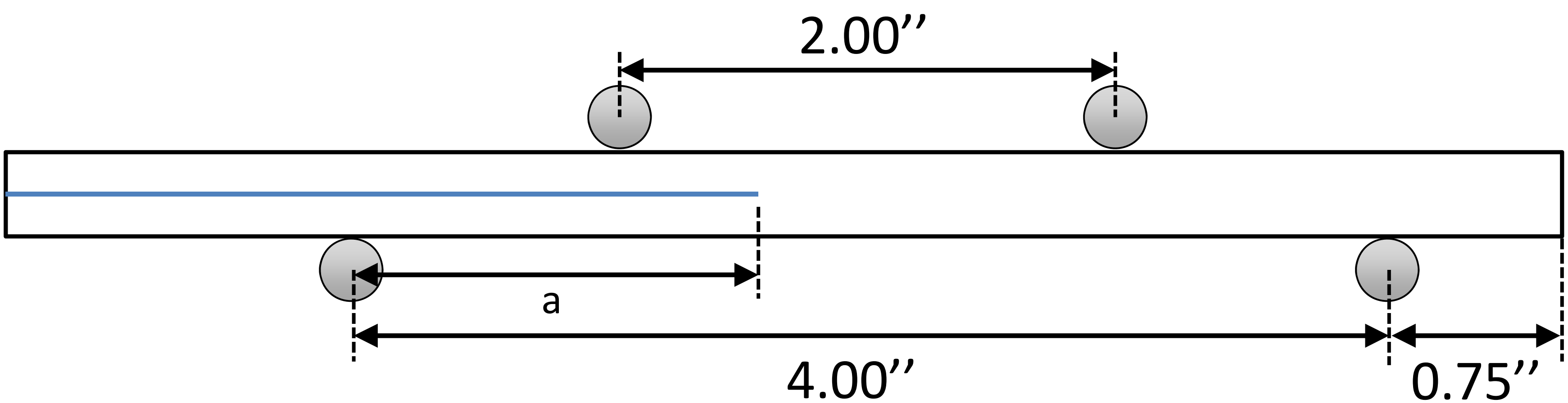
Mode II Fracture Toughness Test: 4-point End Notch Flexure Test

Schematic of 4-ENF Test

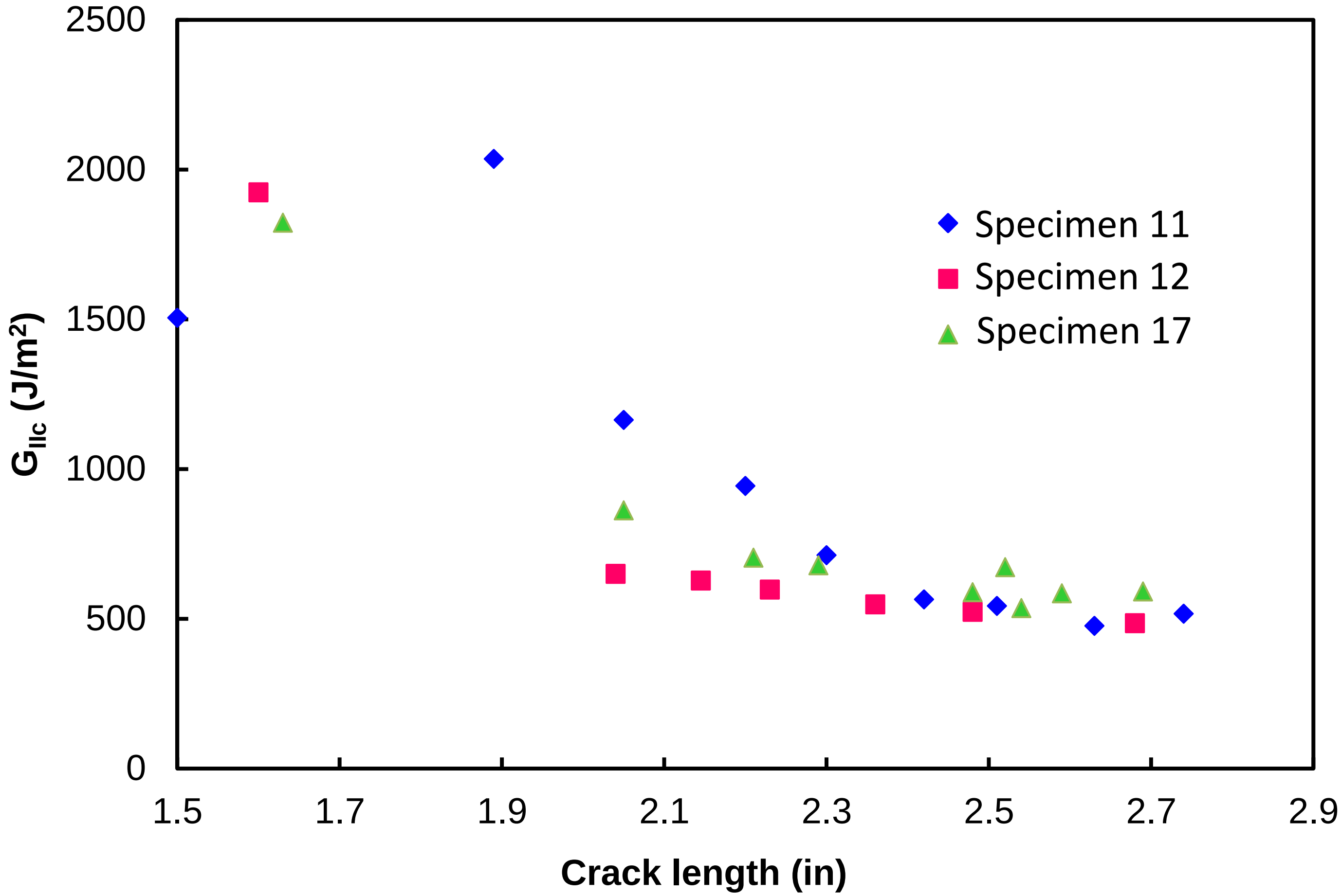
Loading location A: to grow crack from Kapton film insert



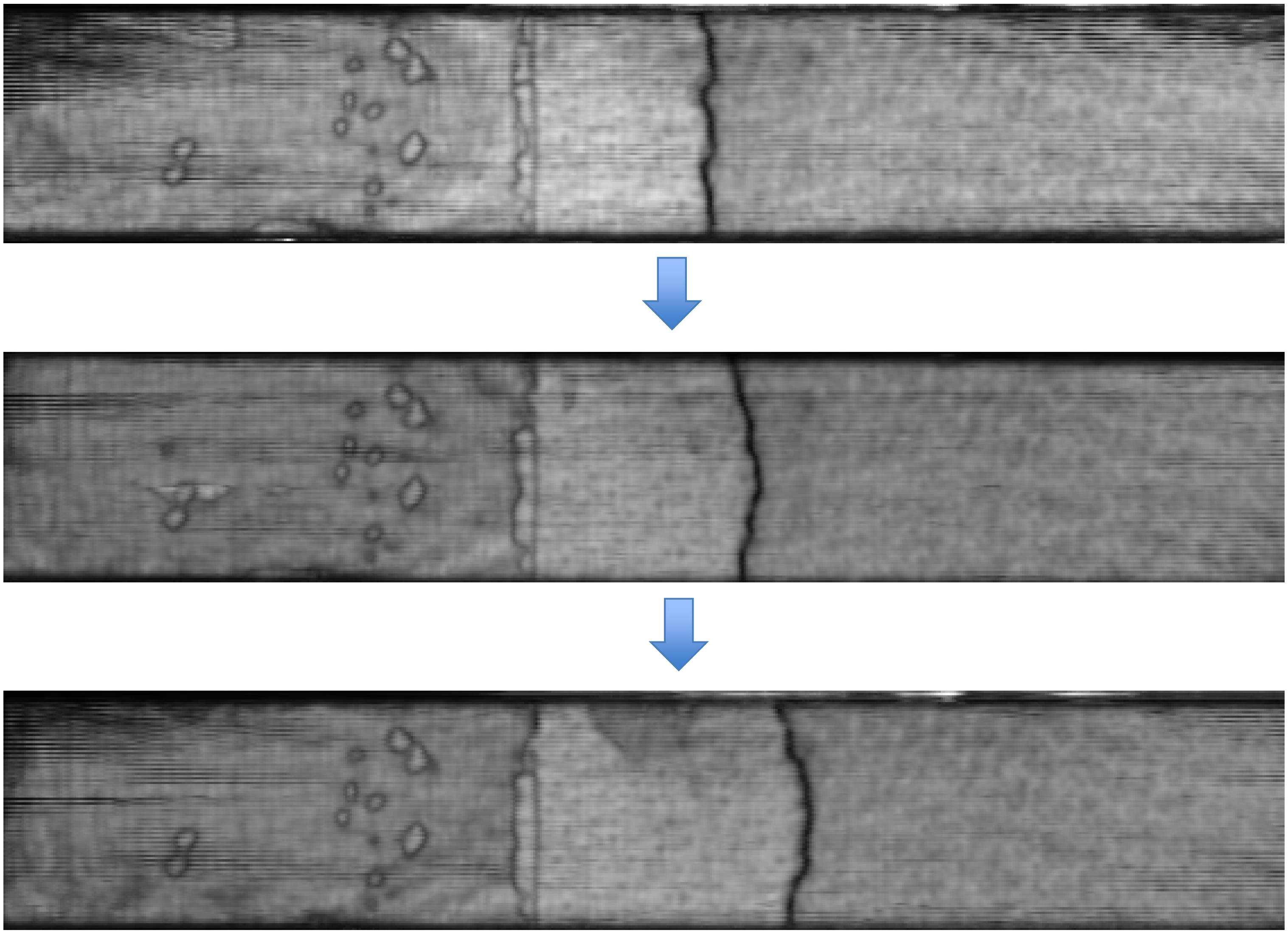
Loading location B: specimen is shifted to obtain additional fracture toughness values



RT G_{IIc} from (1 hr hydrolyzed)

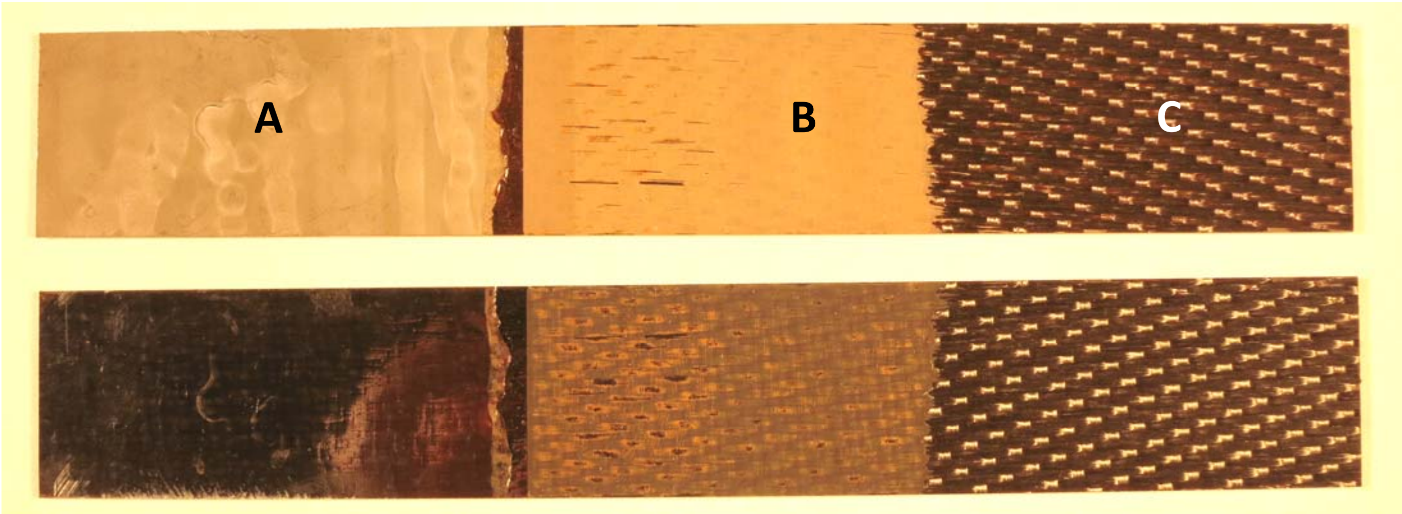


Monitoring Crack Growth using UT Scan (1 hr hydrolyzed)

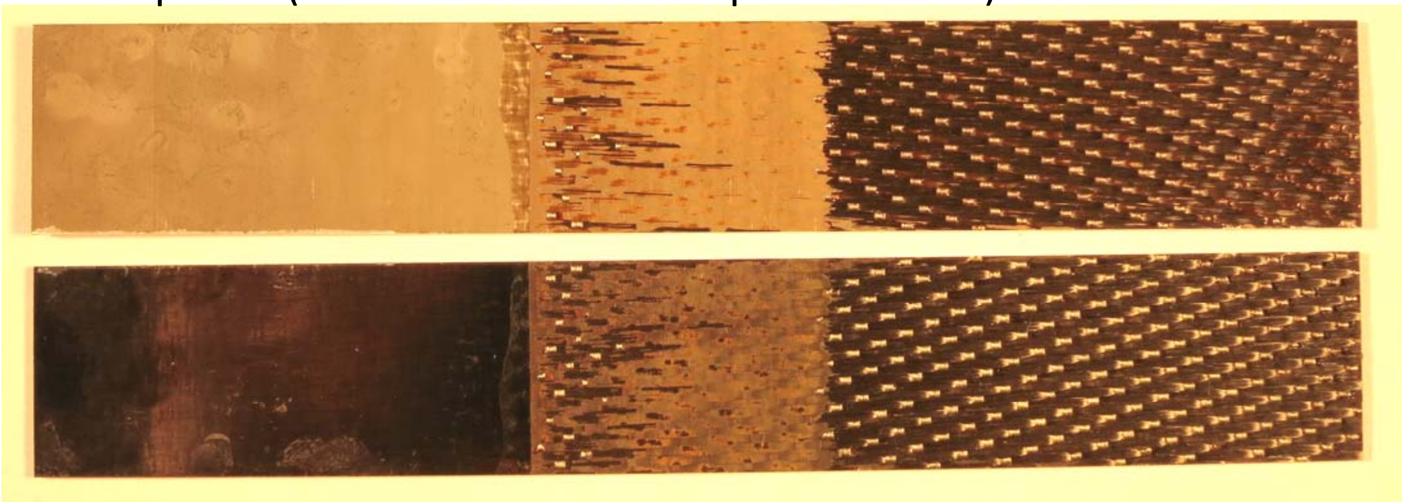


Fracture Surfaces after ENF Tests at RT (1 hr hydrolyzed)

Sample 17 (ENF test crack on tension side)

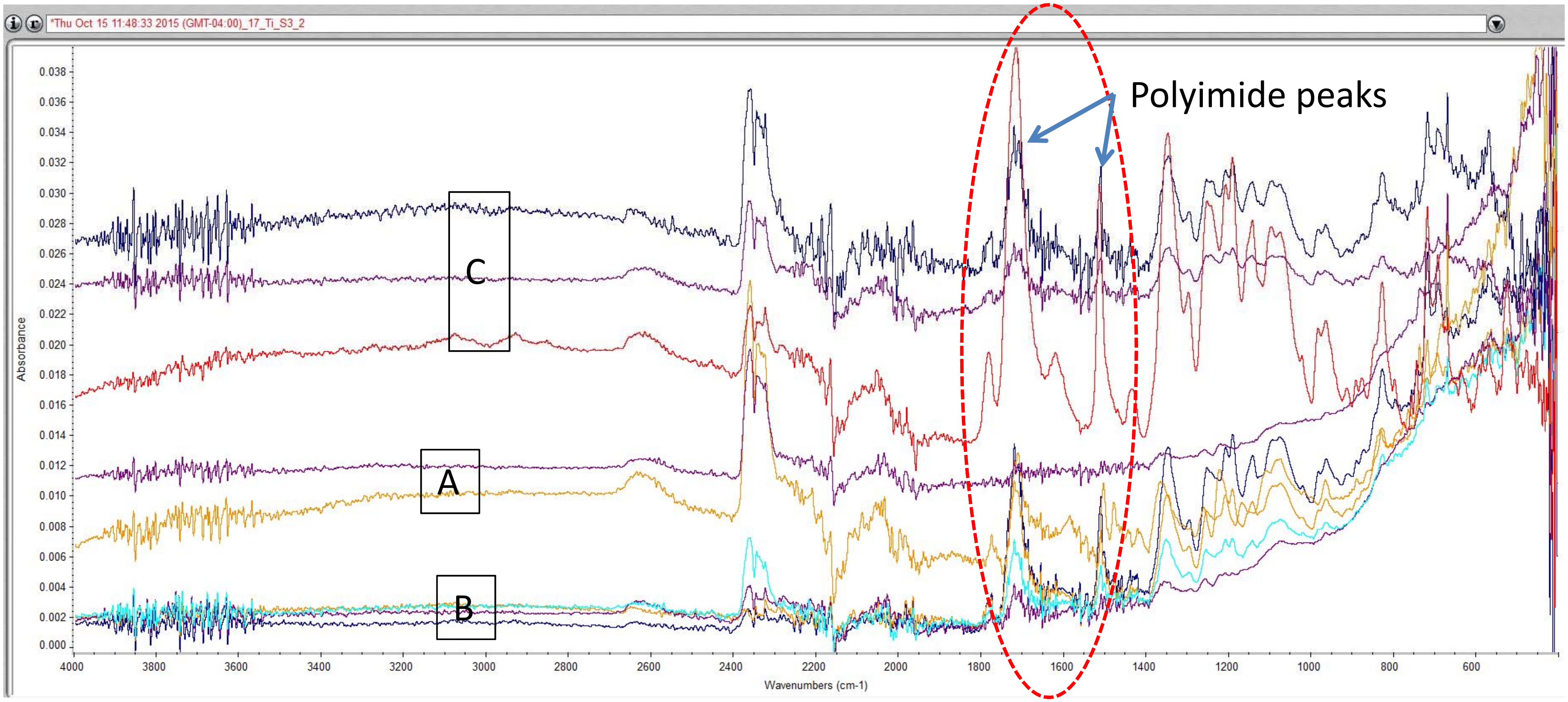


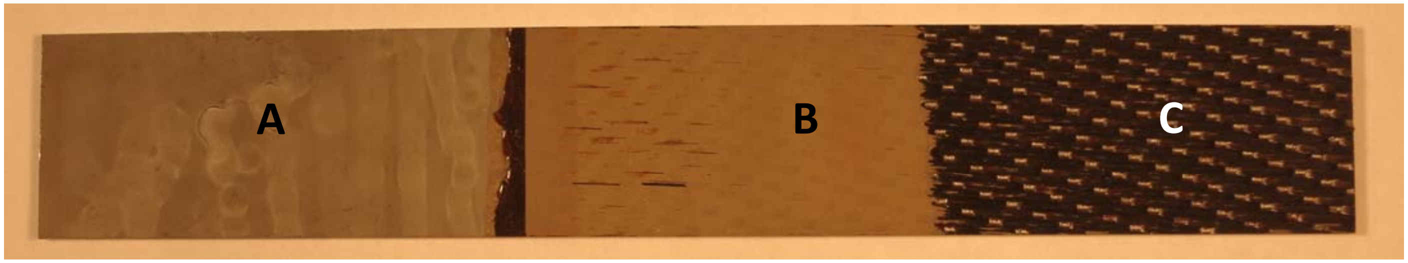
Sample 11 (ENF test crack on compression side)



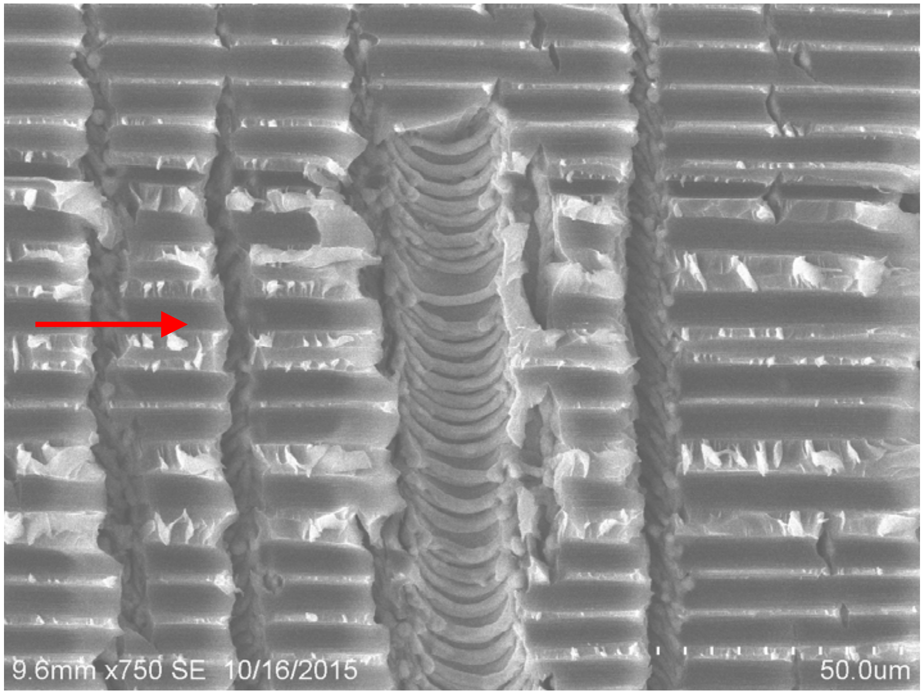
FTIR Spectra (1 hr hydrolyzed)

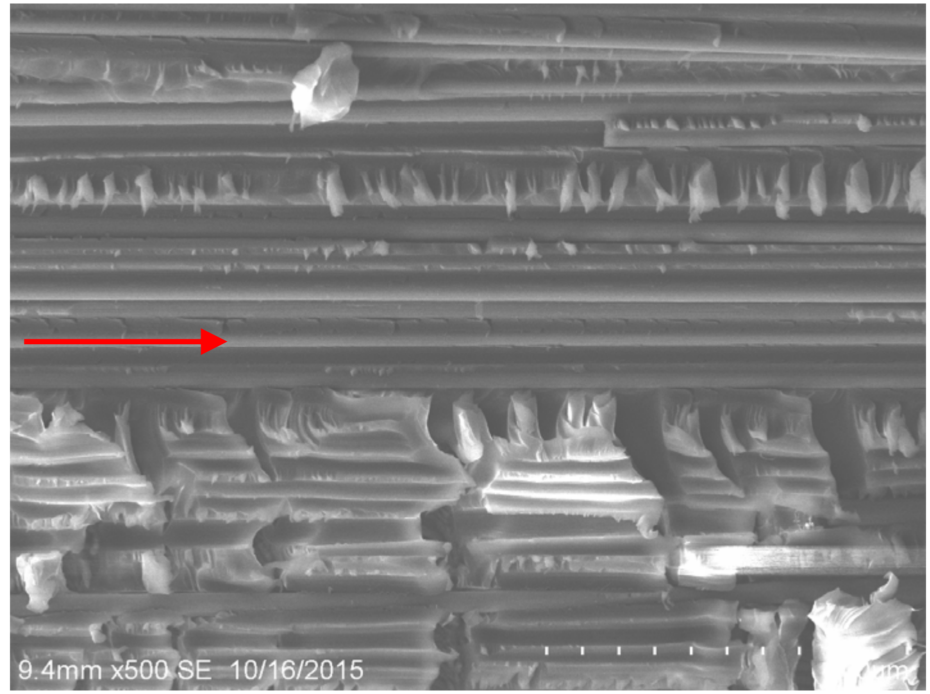
Ti side of Sample 17 (4-ENF test crack on tension side)





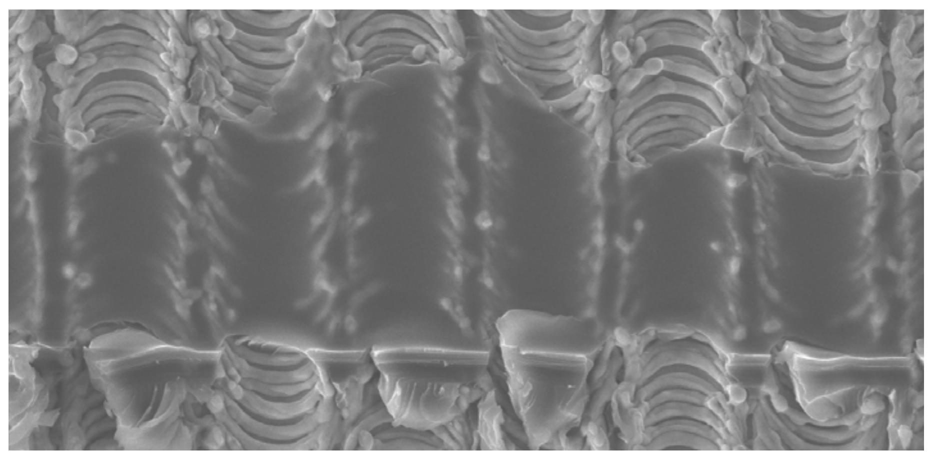
Fracture Surface of Laser Ablated TiI (1 hr hydrolyzed)





→ Crack growth direction

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



Conclusions

- Successfully created hybrid laminates where SMA/Metal foil is embedded in PMC. No adhesive was required.
- Laser ablation and imide-sol gel surface treatment resulted in a robust interface between Ti and Ni-Ti and PI PMC
- The time associated with sol-gel hydrolysis was important and the best results were achieved with 16 hrs, but this has not been sufficiently studied
- Cohesion failures were observed up to 250^{o}C
- Adhesion failures appeared at 315^{o}C
- Test configuration influenced failure mode
- 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

Acknowledgements

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- Clay Claytor
- Crystal Chamberlin

Thank you for your attention!

Questions?