Spring 5-14-2012

In-situ imaging and strain determination during fracture in a SiC/SiC ceramic matrix composite

Joaquin Ramirez
Universidad de Sevilla

F. Stoltzenburg
Northwestern University

K.T. Faber
Northwestern University

J. Almer
Argonne National Laboratory

D. Singh
Argonne National Laboratory

See next page for additional authors

Authors
Joaquin Ramirez, F. Stoltzenburg, K.T. Faber, J. Almer, D. Singh, and J. Routbort

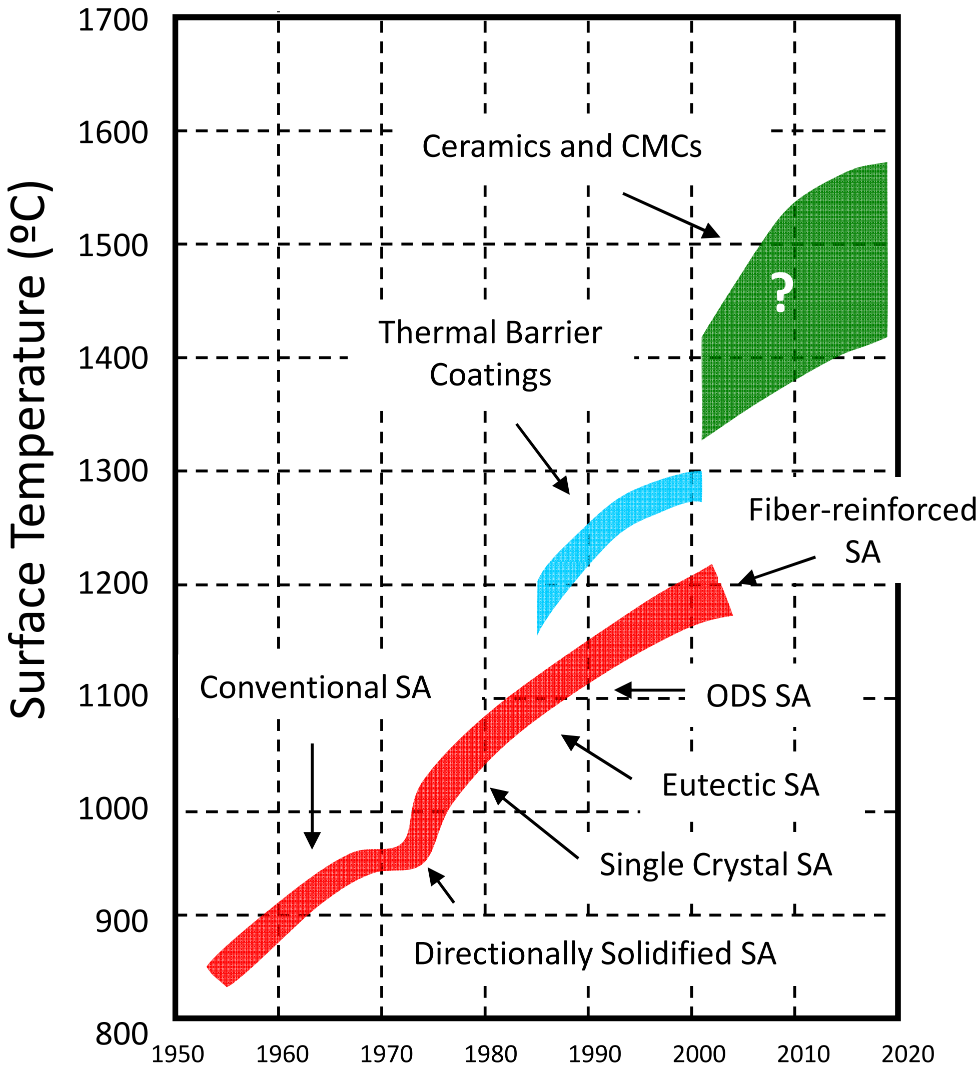
In-Situ Imaging and strain determination during fracture in a SiC/SiC Ceramic Matrix Composite

J. Ramirez-Rico[1], F. Stoltzenburg[2], K. T. Faber[2], J. Almer[3], D. Singh[3], J. Routbort[3]

[1] Universidad de Sevilla – CSIC, [2]Northwestern University, [3]Argonne National Laboratory

Introduction

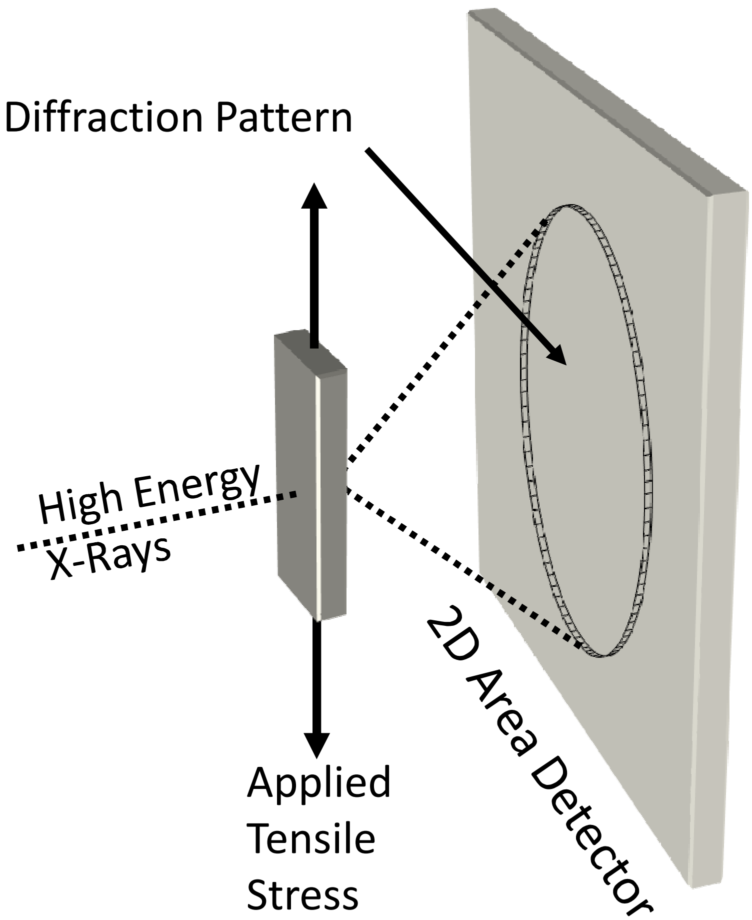
- Brittle failure is one the main shortcomings preventing wide scale deployment of structural ceramic components.
- Usual approach involves making fiber reinforced composites with enhanced toughness.
- Fracture mechanisms become very complex
  - Statistical fiber failure
  - Load transfer effects
  - Fiber sliding and pullout
- There is a need for *in-situ* techniques that allow for observation of fracture mechanisms under close to final application conditions.



H. Ohnabe et al. Proceedings of the Third International Symposium on Ultra High Temperature Materials, 1993

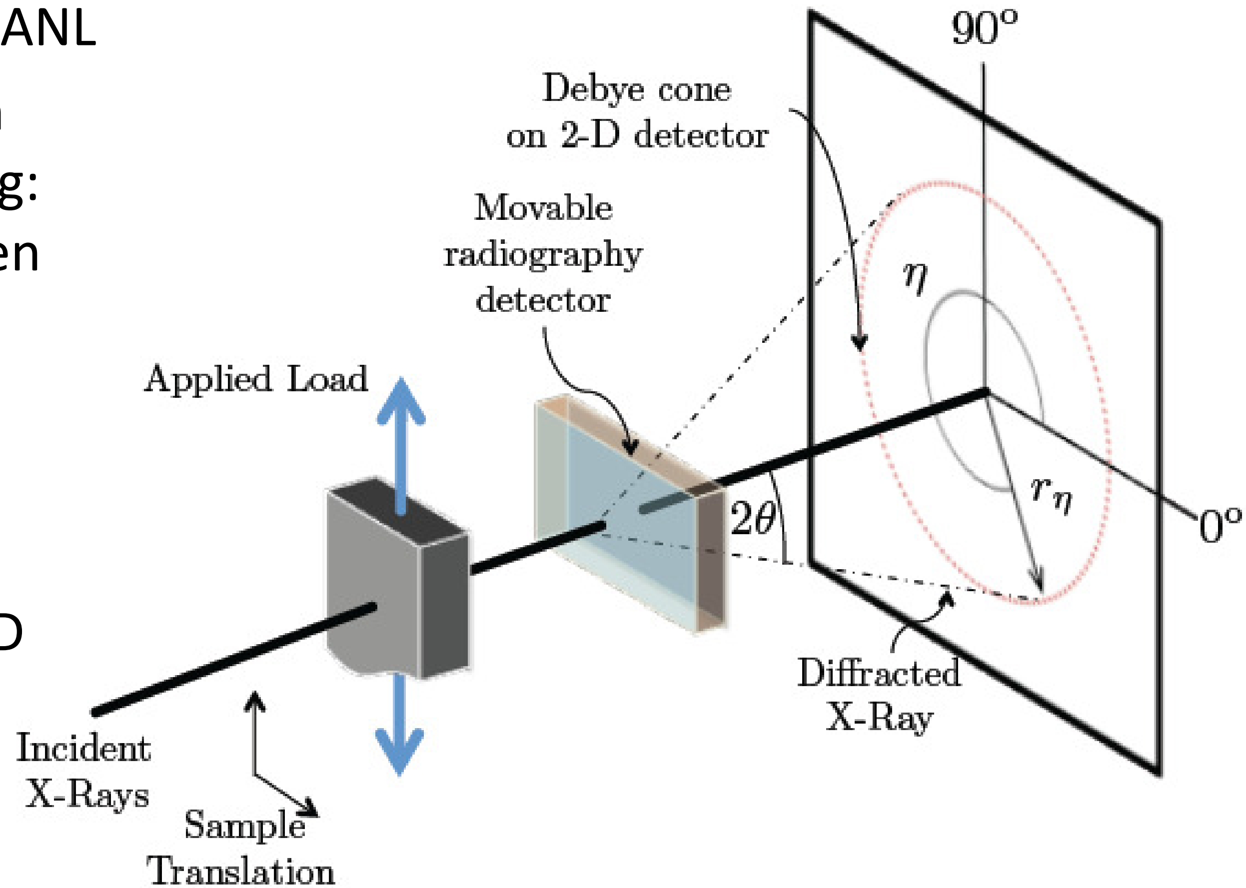
Objectives

- Develop a methodology to study fracture processes in real time using a synchrotron source
  - Observe crack propagation in real time: *Radiography.*
  - Determine strain/stress fields in the vicinity of the crack tip: *Microdiffraction.*
- Need high-energy/high brilliance X-rays to study materials in transmission at reasonable measurement times
- Need a dual imaging/diffraction setup that can quickly shift between the two modes



Experimental setup

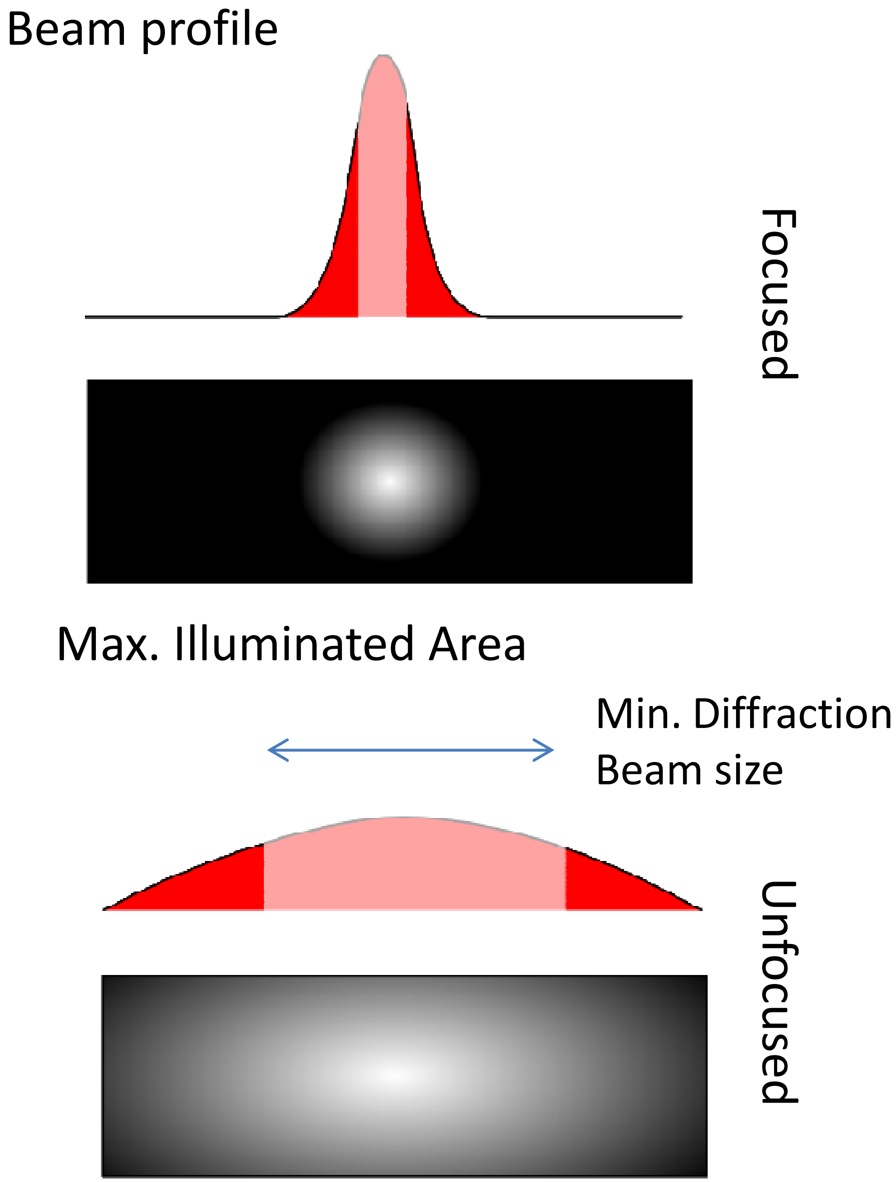
- Setup at 1-ID-C beamline, Advanced Photon Source, ANL
- Movable detector set with reproducible re-positioning: avoid recalibration between measurements.
- Beam size continuously modified by using a set of motorized W slits.
- LAG:Eu scintillator and CCD camera for radiography
- a-Si area detector for diffraction



E = 70 keV ; λ = 0.1771 Å
Microdiffraction resolution: (50x50) µm^2 - (200x200) µm^2
Radiography: (2.5 x 1) mm^2 - nominal resolution ~ 1.5 µm / **Room temperature**

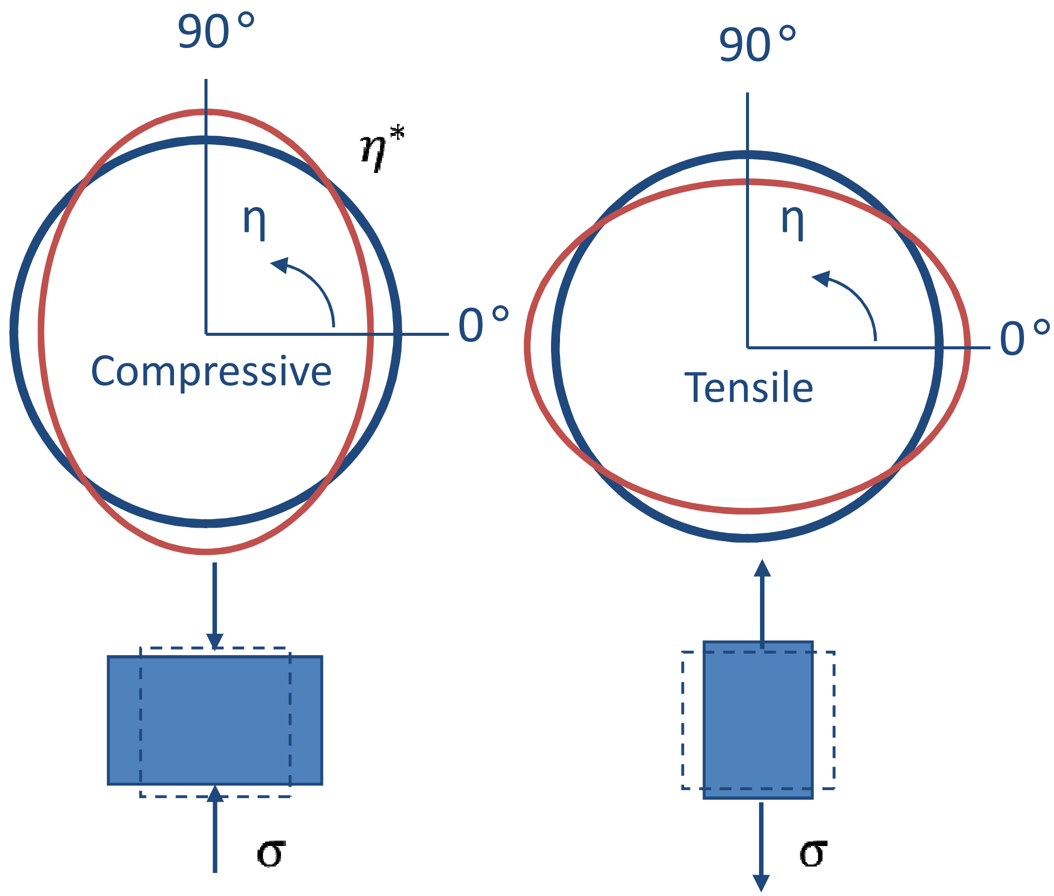
Beam size: microdiffraction vs. imaging

- A focused beam allows for very high resolution strain mapping (~1µm)
- Maximum illuminated area is then too small to image a large portion of the sample.
- Beam defocusing mitigates this, at the cost of losing spatial resolution (diffraction measurement too slow).
- A compromise needs to be made between illuminated area and spatial resolution.



Strain determination from 2D diffraction patterns

- Unstressed polycrystals will give perfectly circular Debye (diffraction) rings.
- The presence of stress elliptically deforms the rings
- The amount of ellipticity is related to the strain tensor (averaged)
- If the material's poisson ratio is known, individual strain components are resolved
- Otherwise strain amplitude can still be determined



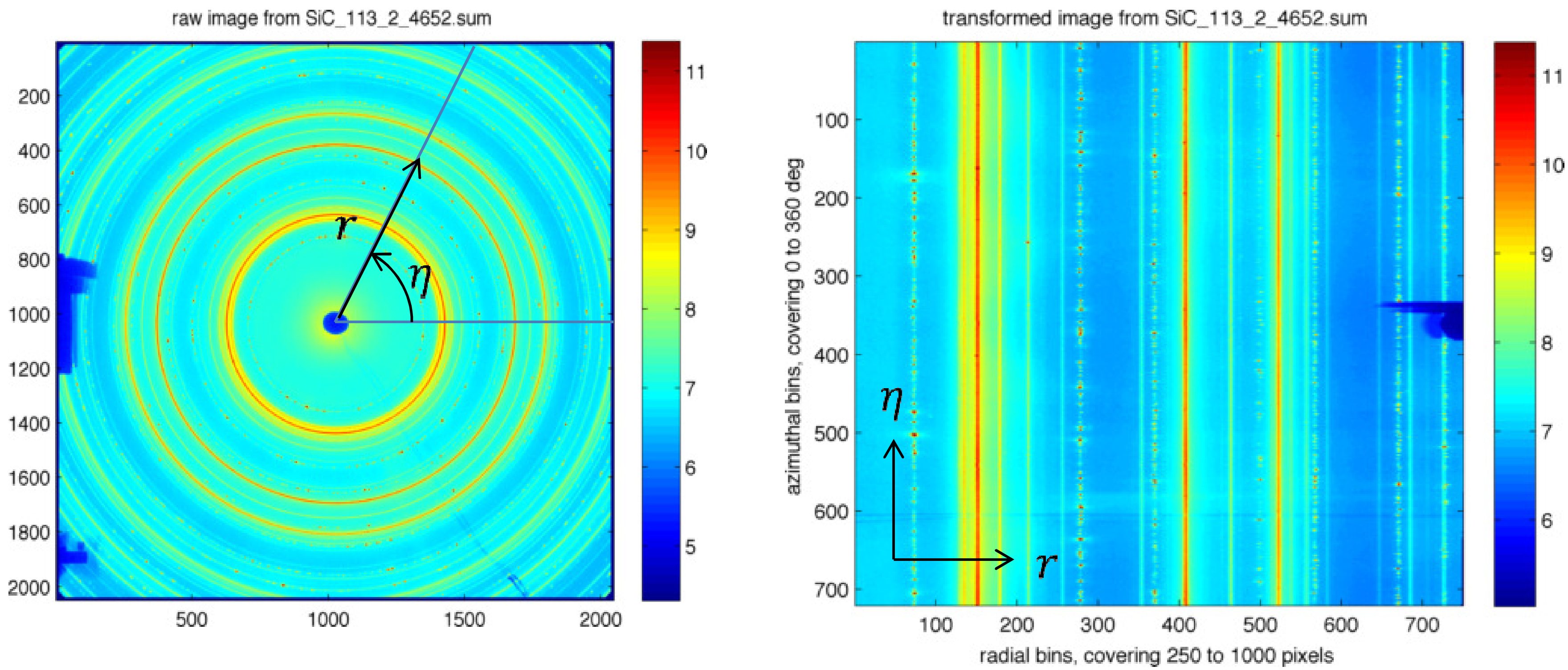
$$\epsilon_{11} = \frac{r_0 - r(\eta = 0^o)}{r_0}$$

$$\epsilon_{33} = \frac{r_0 - r(\eta = 90^o)}{r_0}$$

$$r_0 = r(\eta = \eta^*)$$

$$\nu = \frac{\sin^2 \eta^*}{(1 - \sin^2 \eta^*)}$$

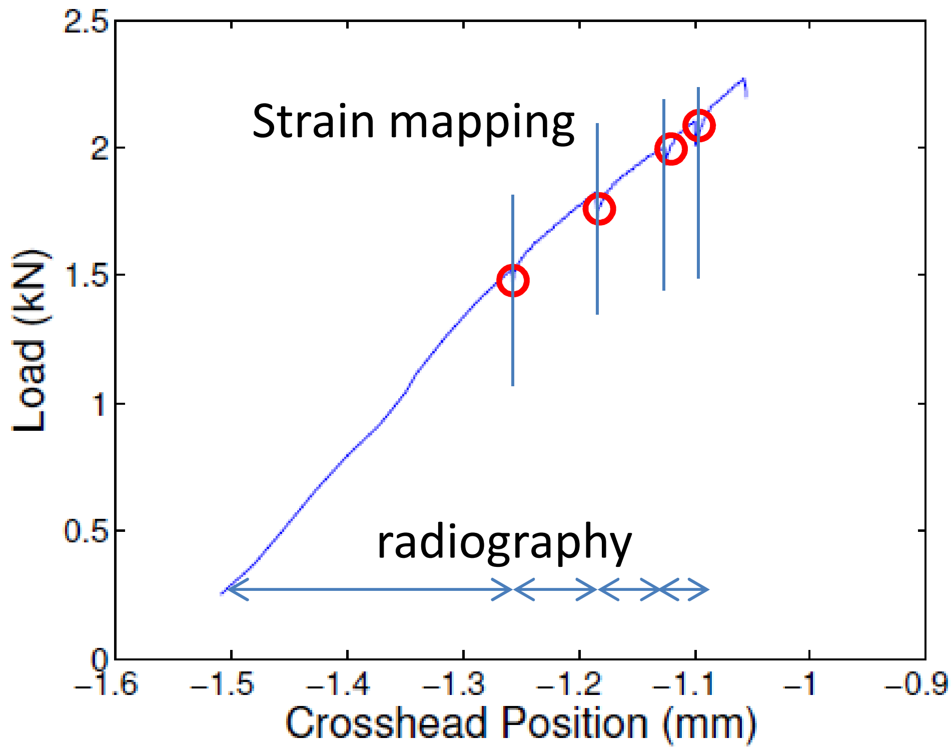
Strain determination from 2D diffraction patterns



- Reflections are fitted to pseudo-voigt peak functions to obtain peak positions as a function of azimuth $r(\eta)$.
- If $r(\eta^*)$ is known, the average 2D strain tensor can be determined from every point in the map
- Interaction (averaging) volume is equal to beam section x sample thickness

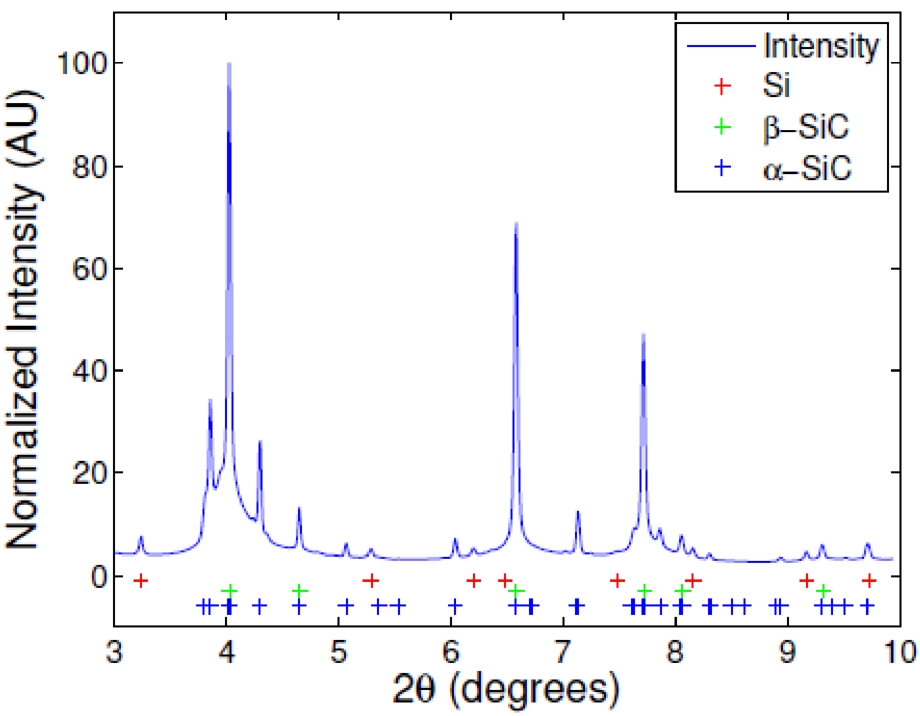
Measurement procedure

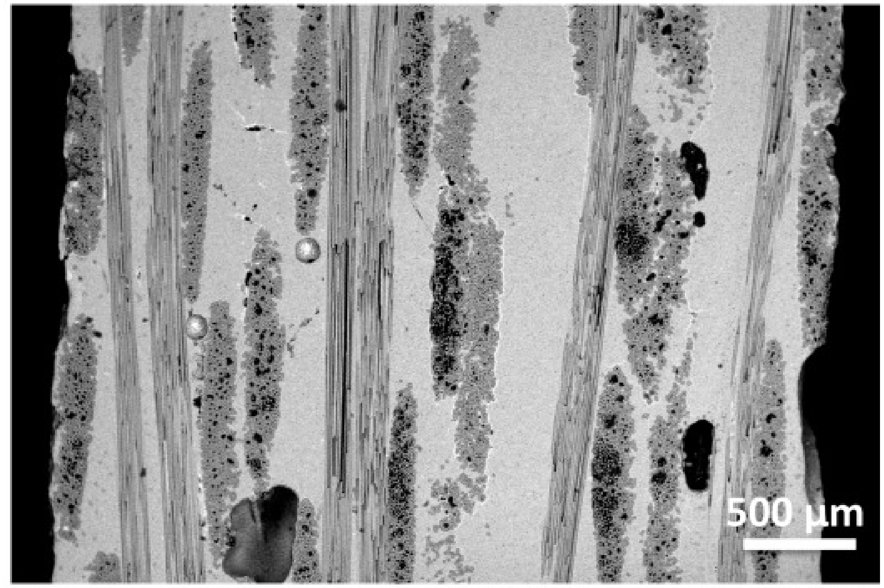
- Radiographic imaging is performed while loading
- Once a feature of interest is observed, the beam is made progressively narrower while imaging
- The desired area is scanned by obtaining diffraction patterns at equidistant points
- Diffraction patterns are then processed to determine thickness averaged strains
- Diffractometer is calibrated using a CeO_2 standard at the beggining and at the end of each run

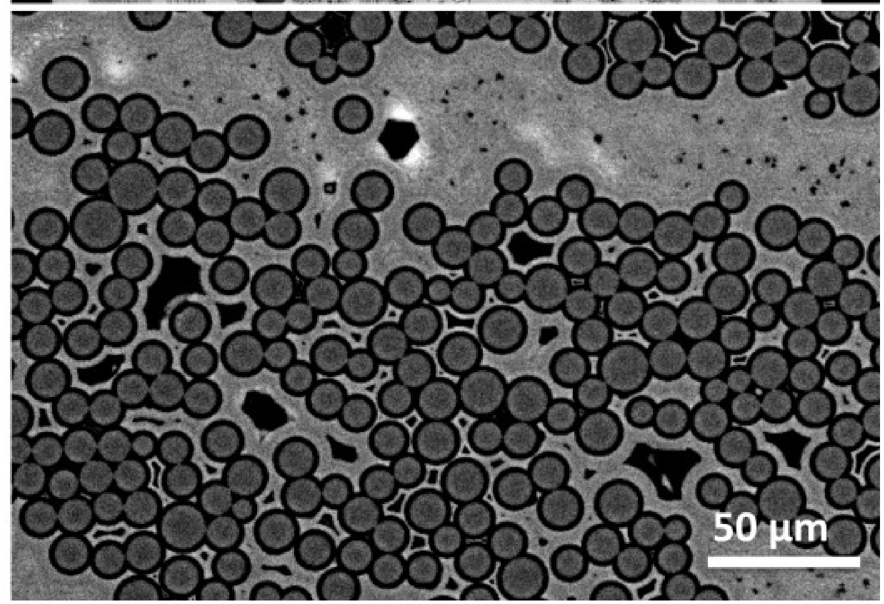


Studied material

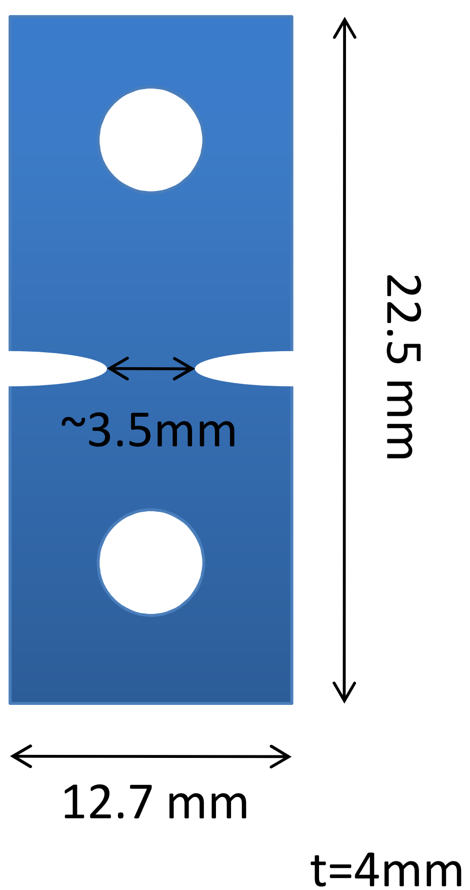
- 8-ply stacked 2D woven Sylramic(c)-iBN fiber
- CVI – SiC infiltrated.
- SiC/Si slurry melt infiltrated
- Provided by Dr. G. N. Morscher (Akron University)

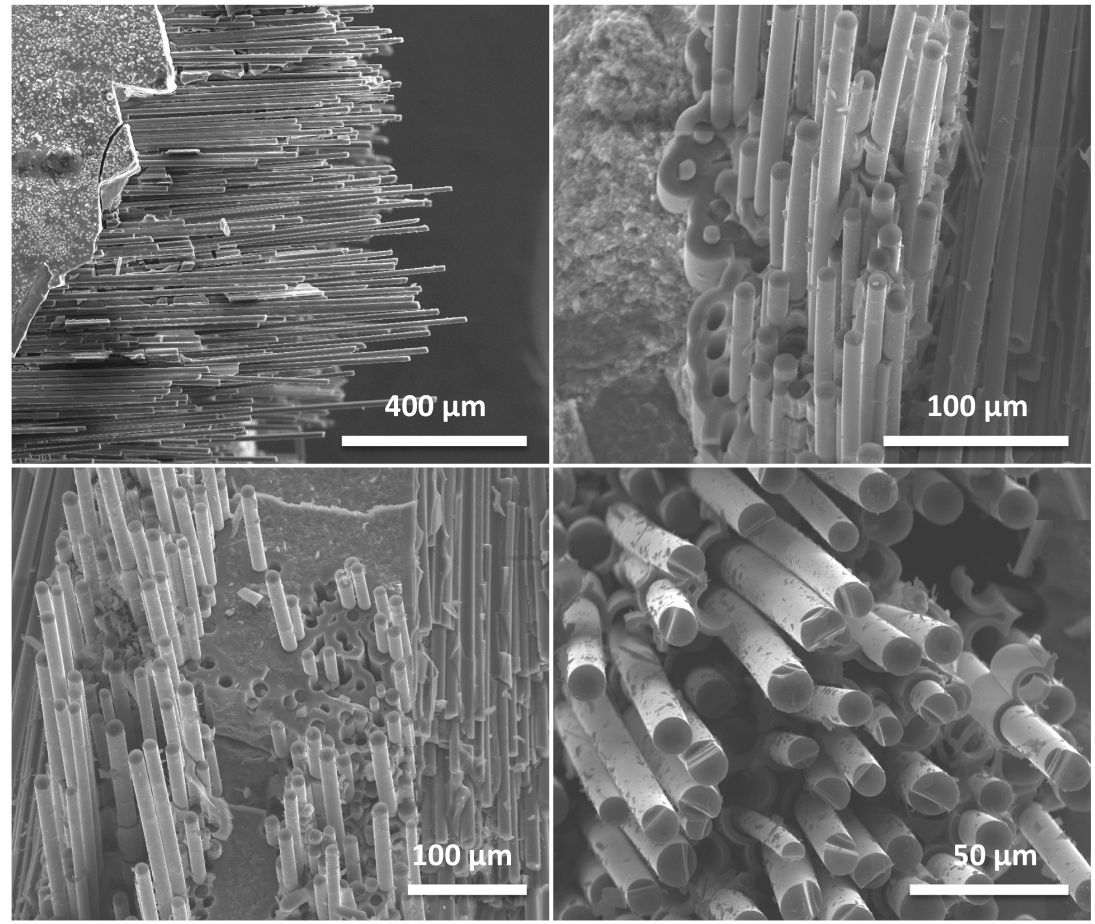




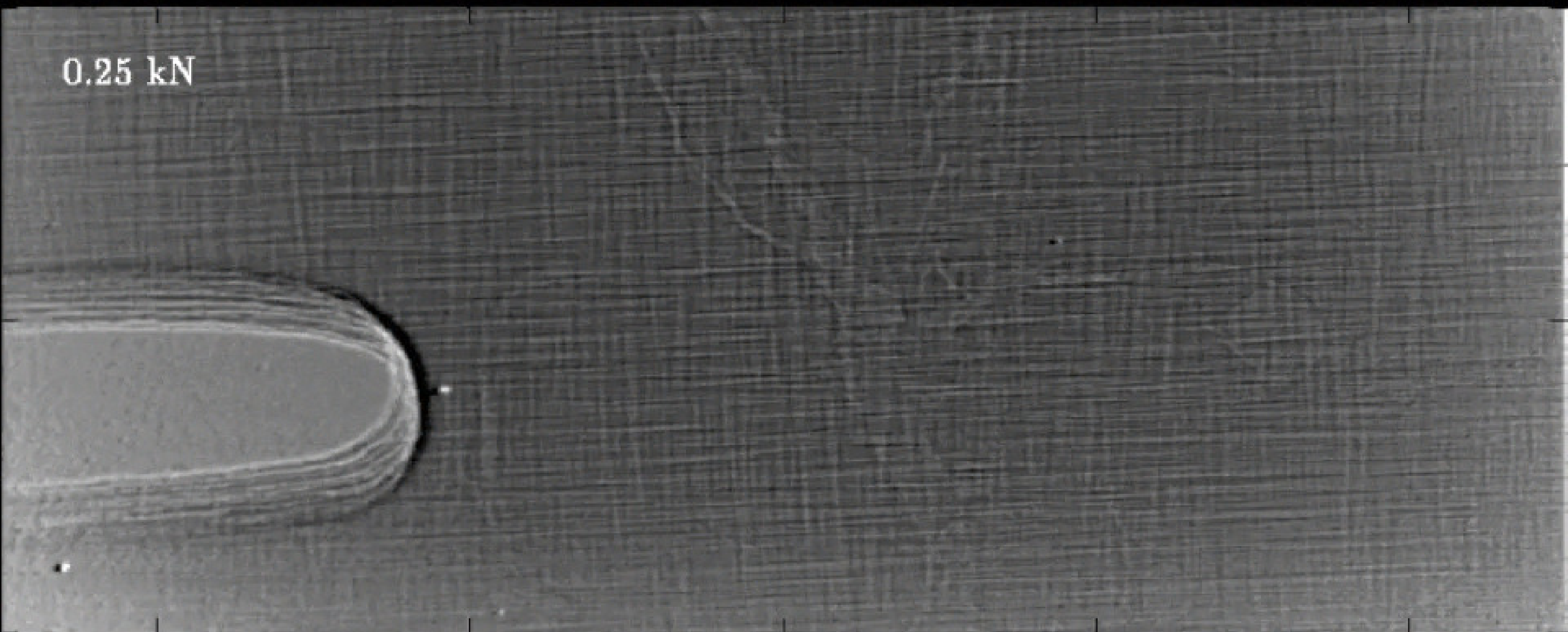


Sample geometry



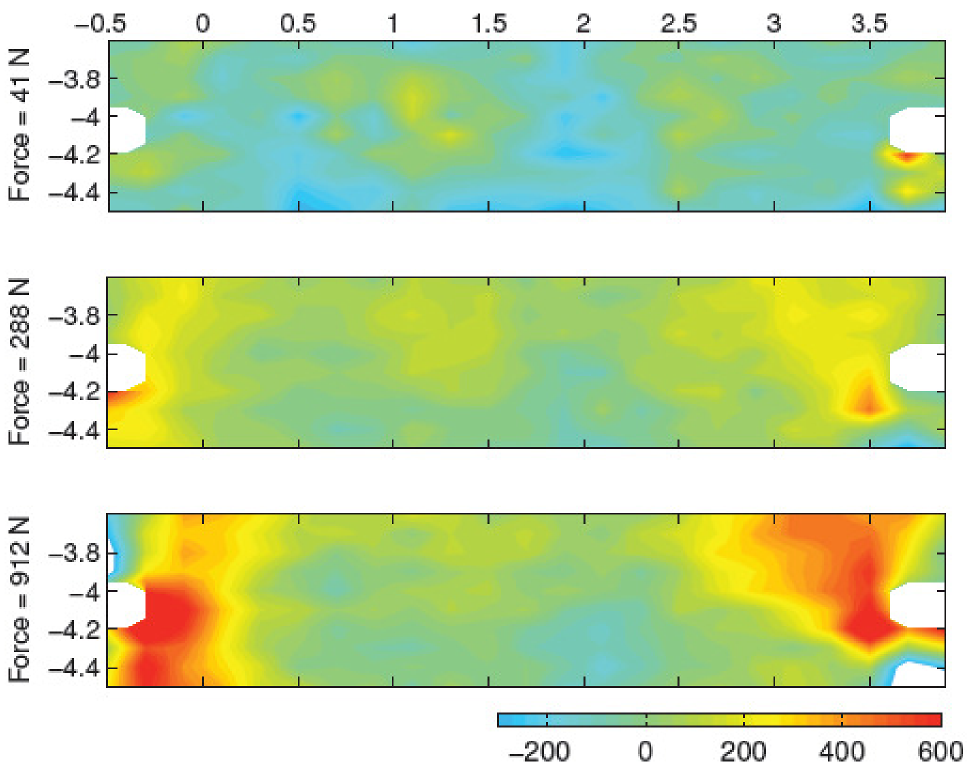


- Notched rectangular coupons were used, loaded using a pin-and-clevis mechanism.
- After testing samples were observed under SEM, extensive fiber pullout was observed

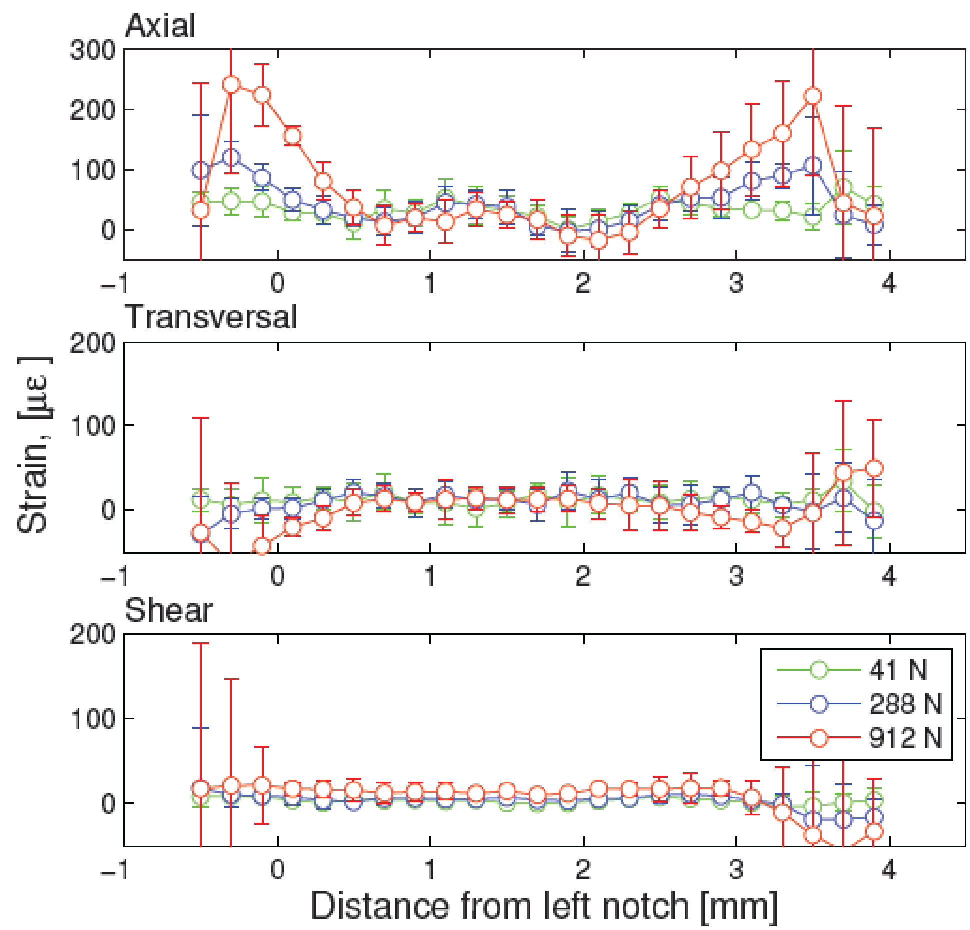

0.25 kN

Strain maps and profiles

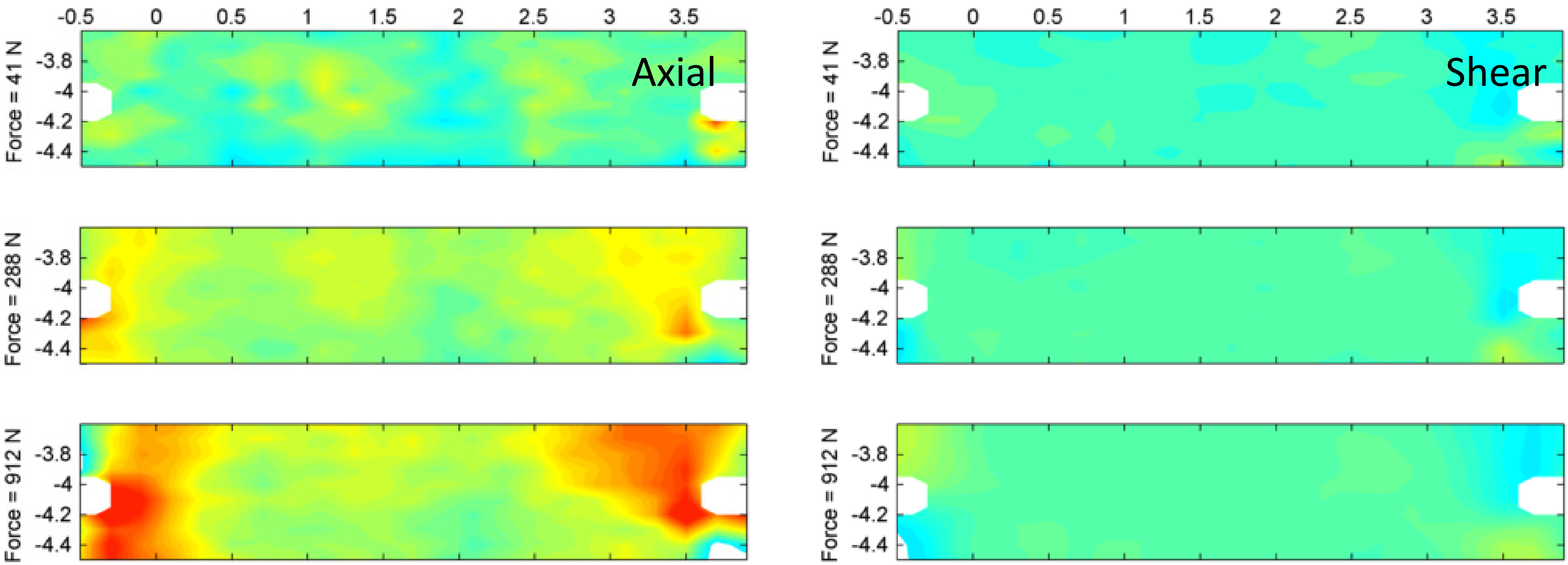
Fiber strain as a function of applied load

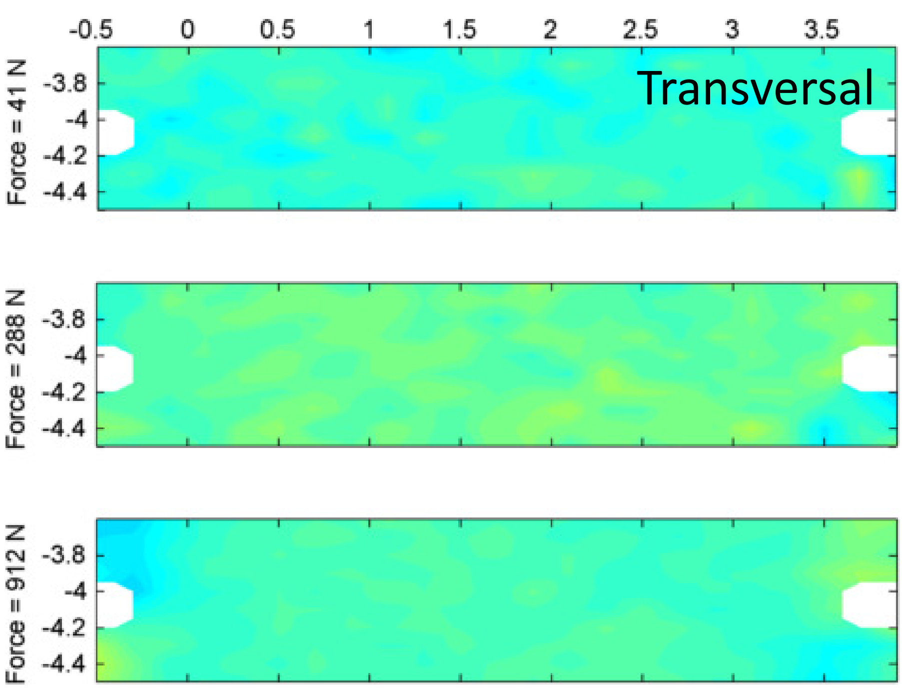


200 µm (horizontal) x 100 µm (vertical)



The strain field can be mapped to obtain spatially resolved information

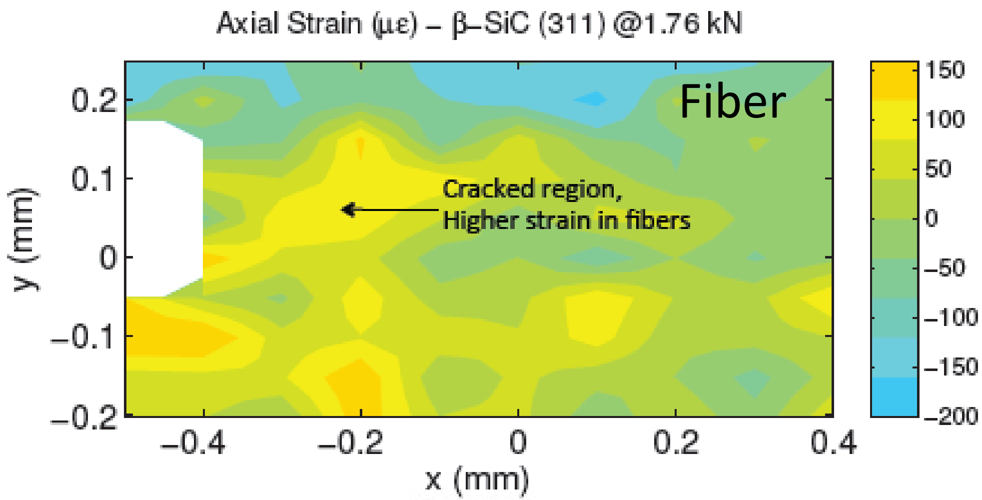


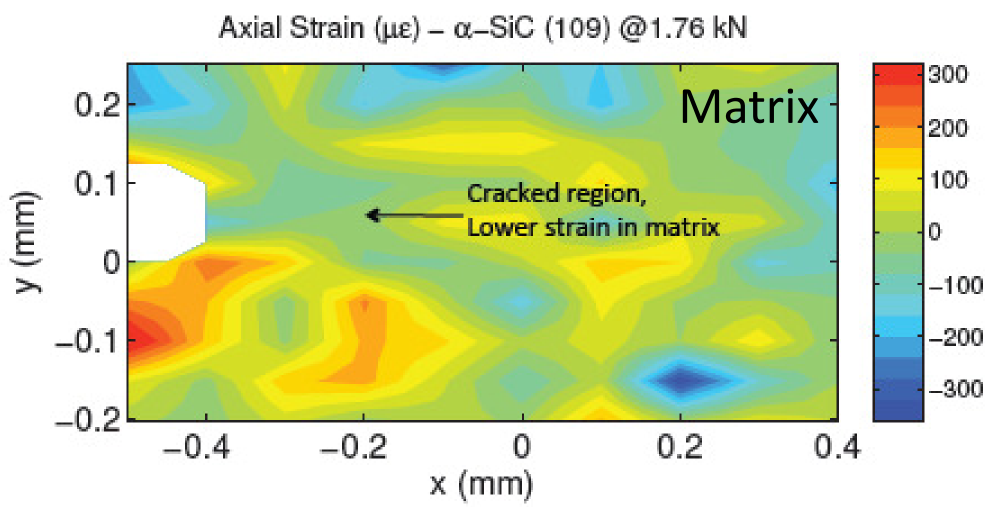


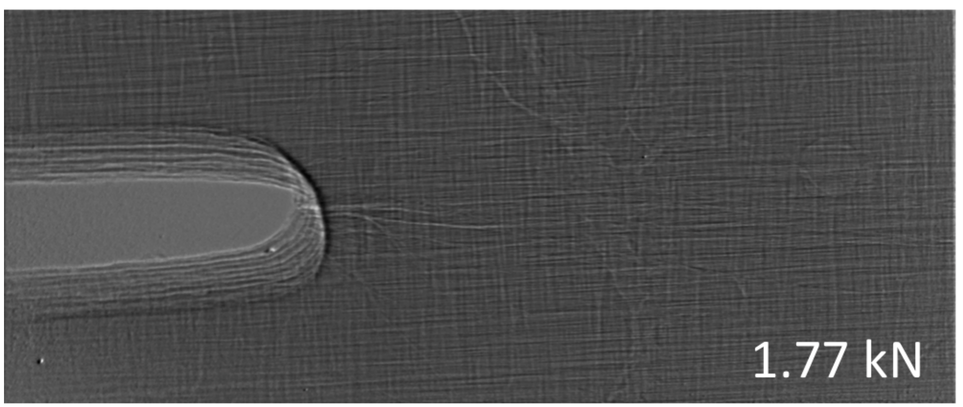
- The three components of the 2D (volume averaged) strain tensor can be determined from a single set of diffraction measurements

High resolution strain maps

- Qualitative evidence of load-transfer effects between fibers and matrix.
- Higher resolution is needed if these effects are to be quantitatively determined.







0.5 mm

100 μm (horizontal) x 50 μm (vertical)

Conclusions

- A combined imaging/microdiffraction technique was developed to study fracture processes *in-situ* in CMCs.
- Strain fields can be determined with a spatial resolution of 50 µm as a function of applied load.
- Real time imaging is possible using radiography with a spatial resolution around 2 µm.
- In the future, we hope to modify the experimental setup to allow for high-temperature measurement in different atmospheres

Thank you for your attention