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Investigating Irradiated Superconducting Magnet Insulation Materials for Particle Accelerators and other High-Dose Environments

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Superconducting Magnet Program Technical Meeting 12/13/2022

Introduction

The Superconducting Magnet Frontier and Epoxy Resin Systems

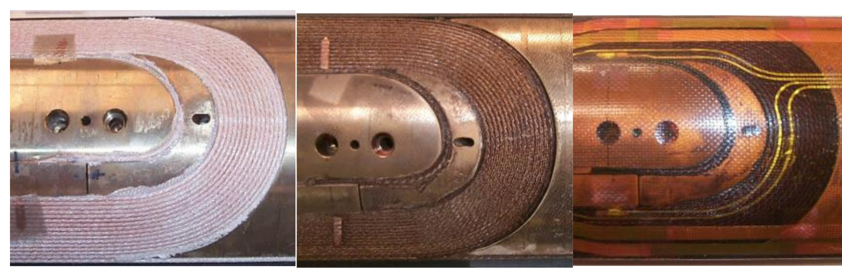


Introduction

LTS Magnets at the forefront of several fields of research rely on epoxy resins

- Low-Temperature Superconducting (LTS) magnets are crucial to a wide range of applications including Nuclear Magnetic Resonance (NMR), Magnetic Resonance Imaging (MRI), Energy and Power, Magnetic Confinement Fusion
- Typical wind-and-react, high-field Nb_3Sn magnets are impregnated in epoxy resin systems for structural support
- It is hypothesized that epoxy cracking and bonding failures during cooldown result in the release of stored energy as thermal dissipation, locally inducing quench
- A robust understanding of how these epoxies behave under in-service conditions is therefore paramount.

Nb_3Sn quadrupoles for HL-LHC



After winding/curing After reaction After impregnation

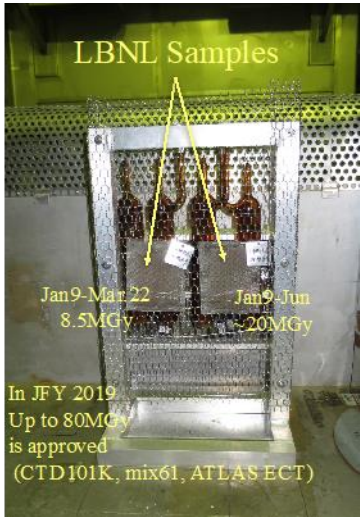
Experimental Details

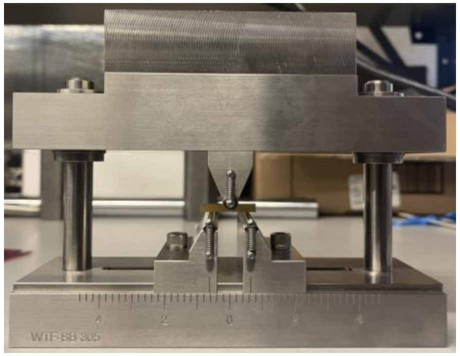
Meaningful analysis called for a diverse test matrix

Experimental Details

Gamma-radiation and liquid nitrogen broaden the gamut of results

- **Materials Used**
  - CTD-101K
  - NHMFL mix-61
  - ATLAS-ECT
- **Gamma-Doses**
  - 0, 25, 50, 75, 100 MGy
- **Temperatures**
  - 293, 77 Kelvin
- **Tests**
  - Physical Changes
  - Short-Beam Shear Strength
  - Flexural Properties
  - Failure Mode Analysis
  - Hardness Testing
  - Scanning Electron Microscopy

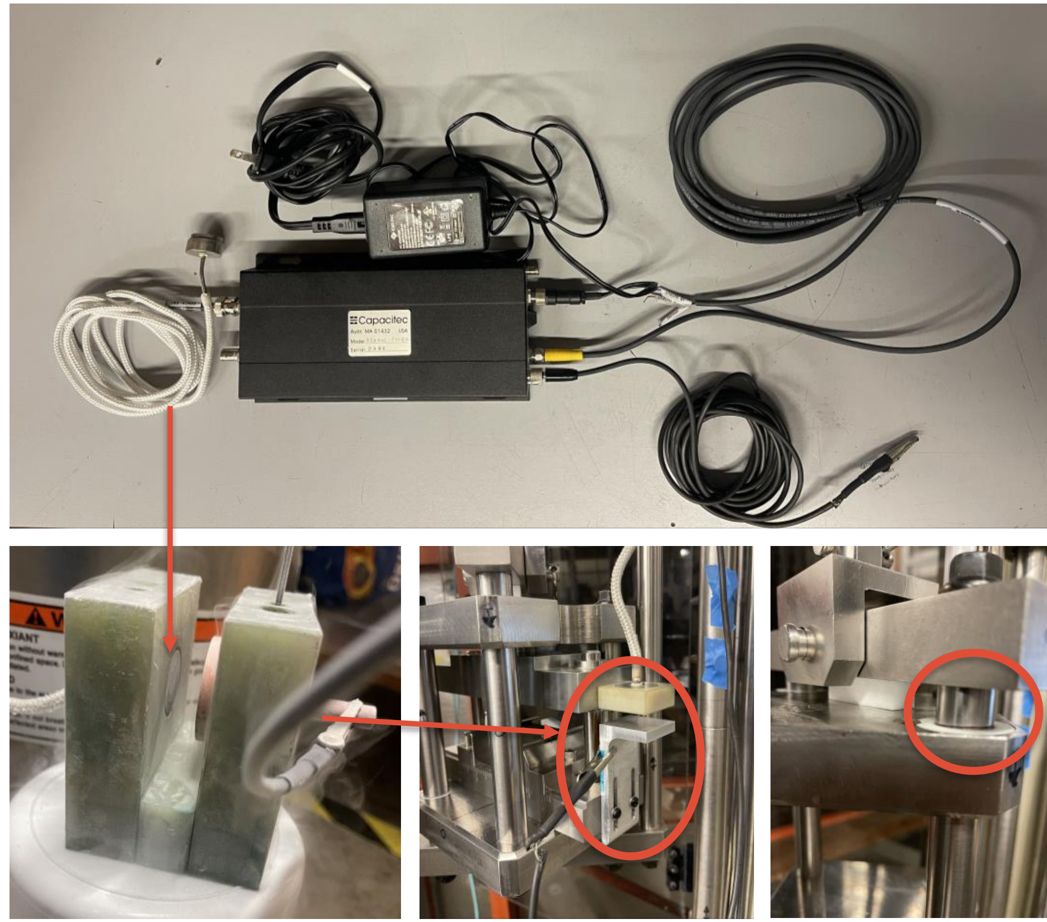




Experimental Details

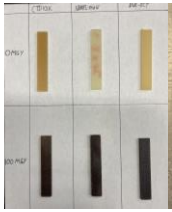
Liquid nitrogen testing required ingenuity

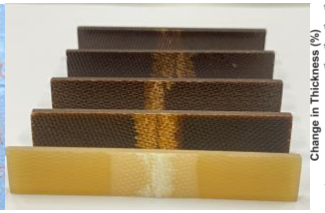
- LN test environment made displacement measurements difficult
- We tested the viability of adapting a Capacitec® displacement sensor for this task
- A G-10 block was fabricated to house the HPC-500A ½”-range cylindrical probe for recalibrating amplifier DC output
- Once the equipment was validated, the 3-point compression block was modified to house the probe/target
- Ultra-low friction Teflon sleeves were also added to combat friction

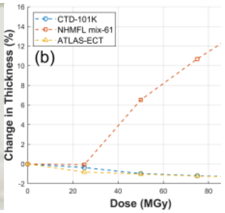


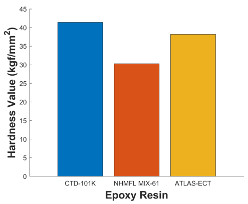
Results

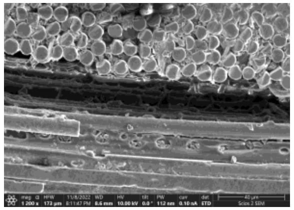
Mixed results from physical testing with interesting microstructural observations







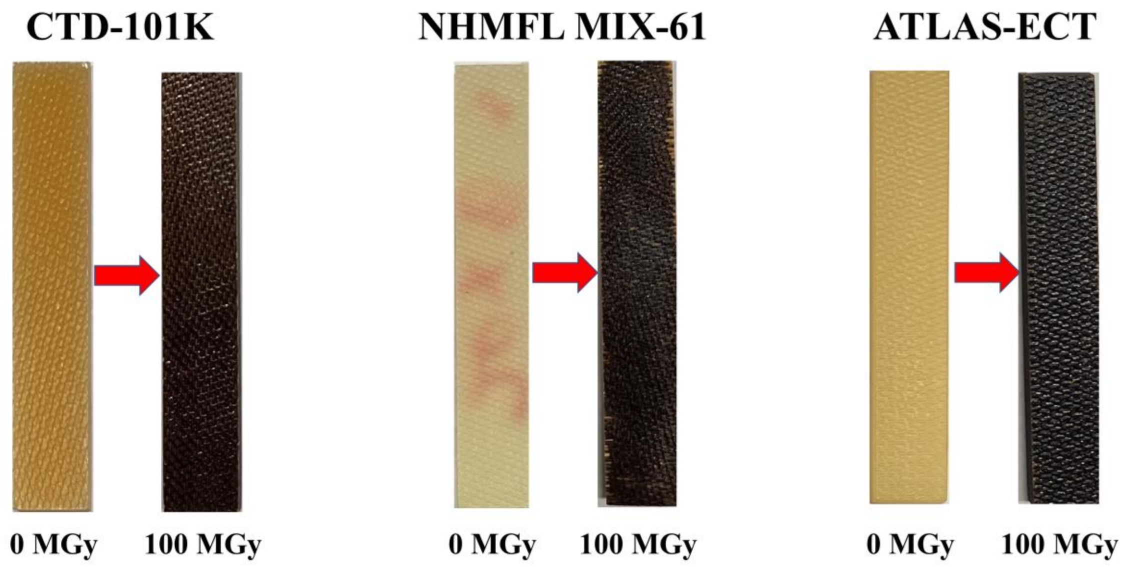




Appearance Changes

The appearance of the samples underwent darkening, dusting and minor flaking

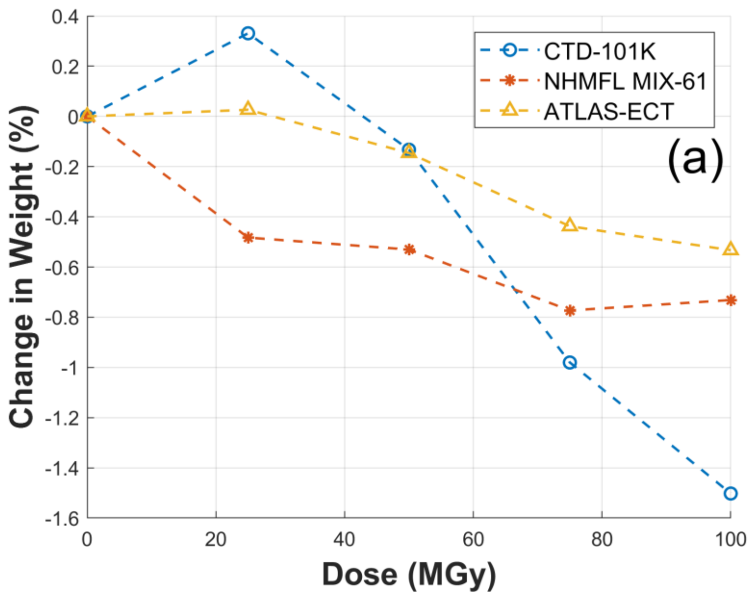
- There was a clear darkening of samples from gamma flux
- From brightest to darkest, pristine epoxies varied as NHMFL mix-61, ATLAS-ECT, CTD-101K
- At all other fluences as CTD-101K, NHMFL mix-61, ATLAS -ECT
- Relative darkness between doses was indistinguishable, suggesting darkening happened at doses much lower than 25 MGy.

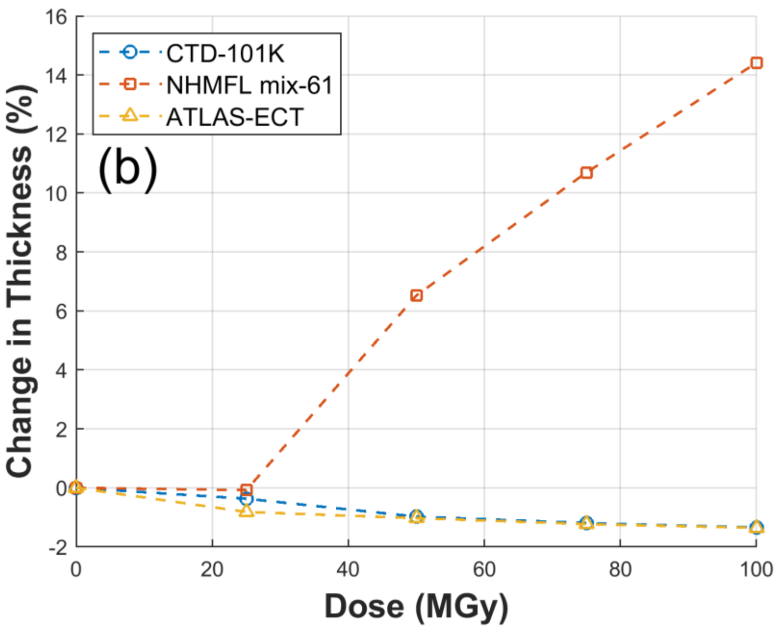


Color changes previously observed in epoxies irradiated to doses in the range of 1-50 kGy have been ascribed to the oxidation of the double benzene rings of the constituent mate-rials

Dimensional Changes

The geometry of the 101K, ECT was largely unchanged, but mix-61 clearly morphed





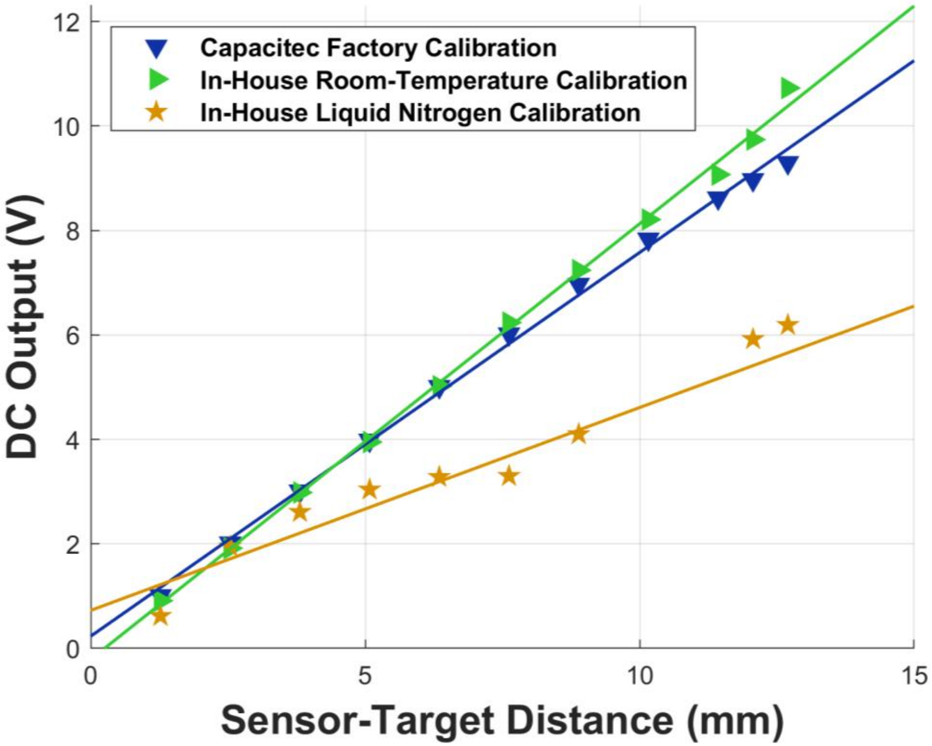
- All 3 materials saw a general trend of weight loss as a function of dose

- Thickness along with, length and width, saw negligible changes *except* for mix-61 which saw considerable swelling due to irradiation

Capacitec Calibrations

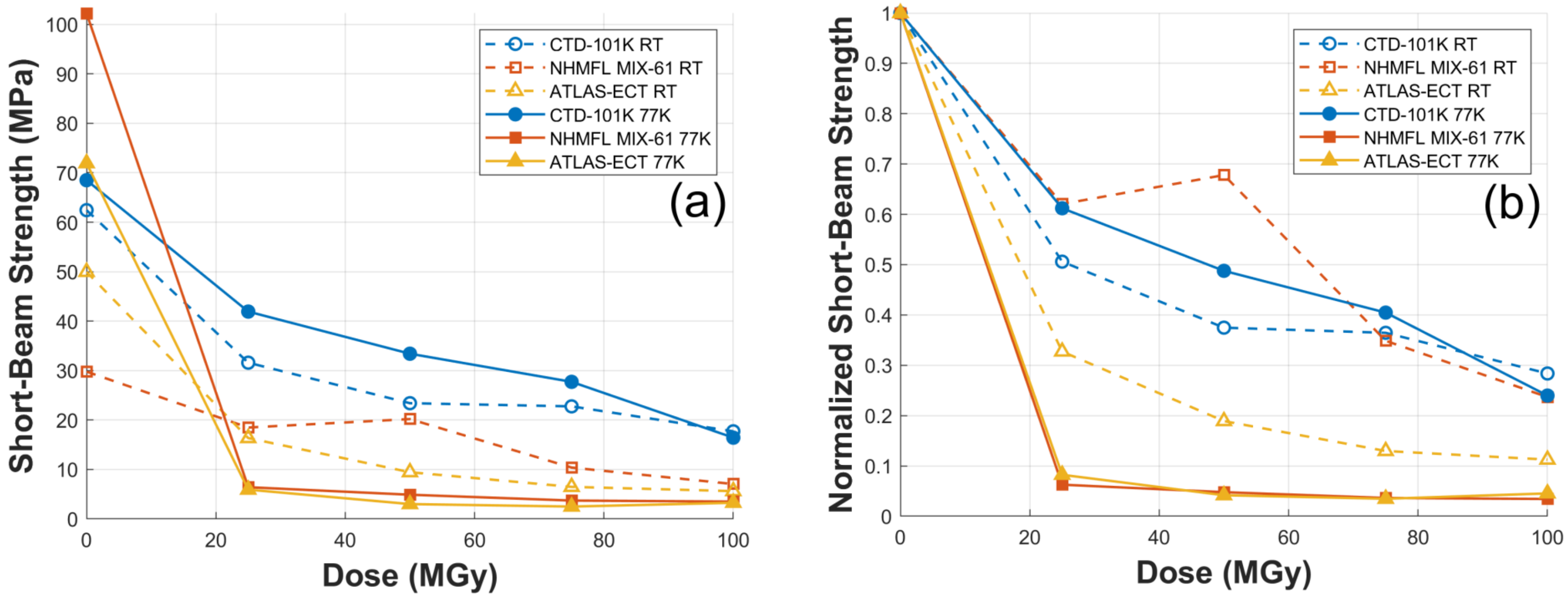
The Capacitec sensor worked well in LN and was successfully calibrated for this medium

- Probe proved extremely sensitive to conductive surfaces in its field of view
- Calibration was therefore done in-situ, to most accurately replicate test conditions, with gauge blocks used to set sensor-target distances
- In-House testing done in 46 closely replicated the factory calibration sent by Capacitec, especially at the lower end of the sensing range
- Liquid nitrogen calibration was done to facilitate cryo-displacement measurements
- The suppression of V/mm gradient was in good agreement with what is expected from fundamental physics being roughly equal to the inverse ratio of relative permittivities of the two media



Short-Beam Shear Strength

SBS tests show gains for mix-61, ECT but poor rad resistance compared to 101K

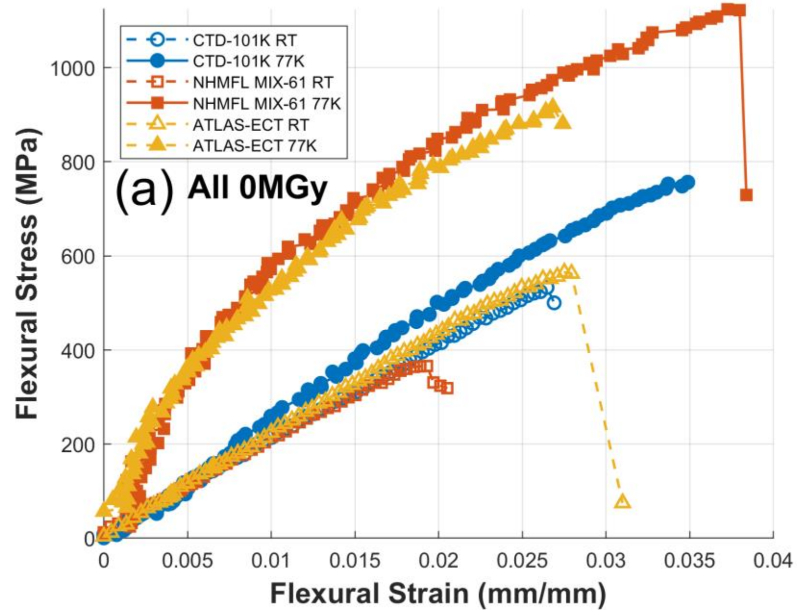


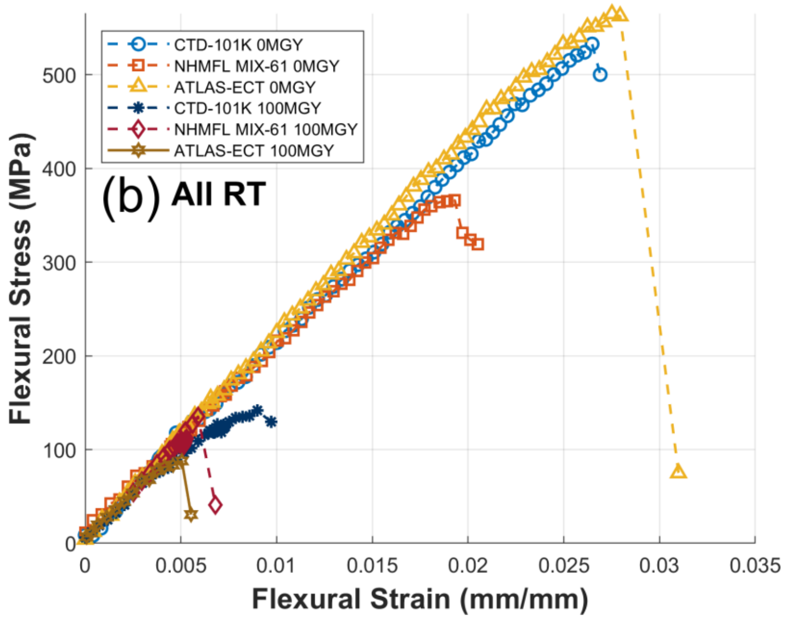
- At RT, 101K had the highest strength pristine, and at every gamma fluence
- At 77 K, major increases seen for mix-61, ECT samples surpassing 101K
- However, 101K's radiation resistance outmatched the other resins at highest doses at RT and vastly outperforming them at 77 K.
- Mix-61, ECT saw precipitous declines in SBS strength even at the lowest gamma fluence

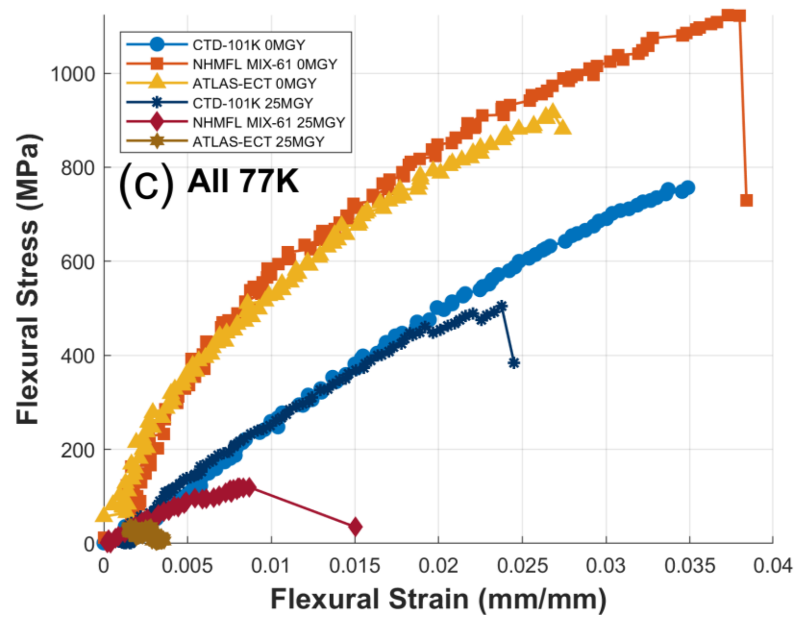
Flexural Properties

Flex properties echoes SBS findings

a) At RT, pristine 101K and ECT had similar toughness, while mix-61 trailed. At 77 K however, both amine-cured resin systems showed substantial gains in toughness
b) At RT, 100 MGy samples saw tremendous declines in toughness, of the three resin systems tested, 101K still lead at the highest fluence.
c) At 77, 101K again manages to retain the highest toughness post-irradiation, trailed remotely by mix-61. ECT's decline in toughness was immense, barely registering at 25 MGy of gamma fluence.

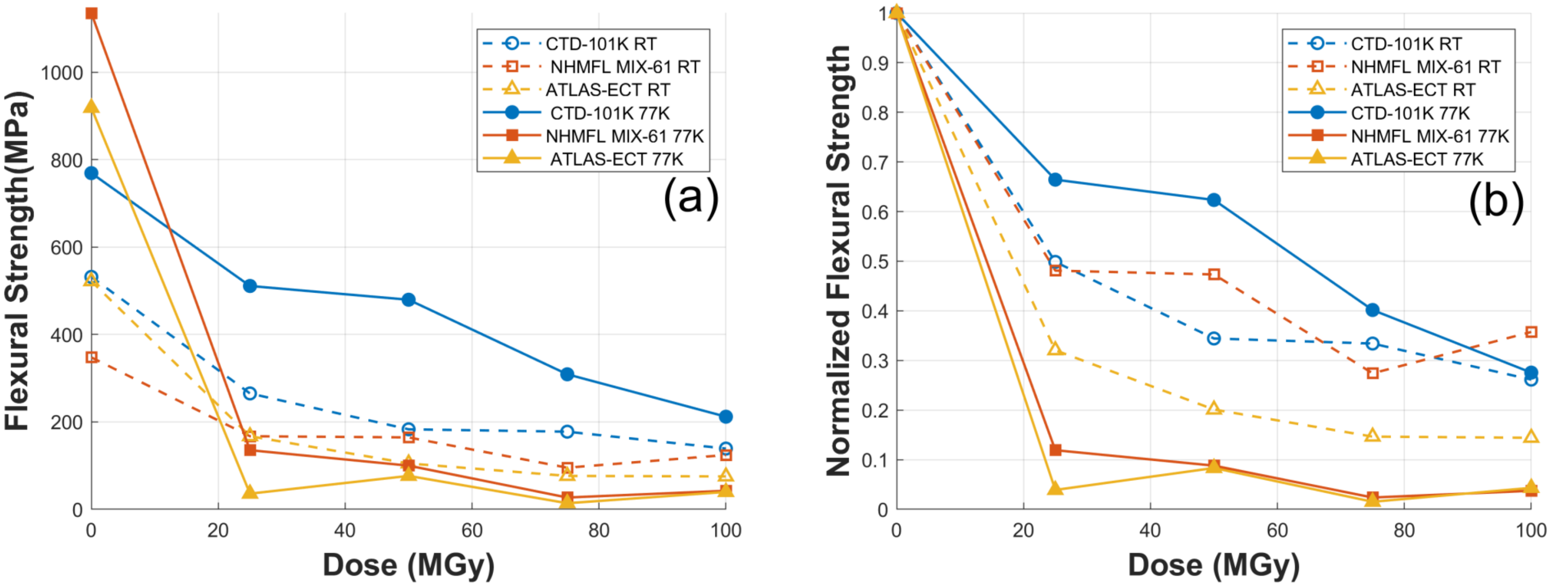






Flexural Properties

Mix-61, EC again show severe sensitivity to gamma-flux despite pristine superiority

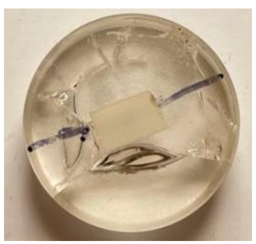


- At RT, 101K maintains highest strengths at all doses followed by ECT and mix-61
- At 77 K, mix-61 and ECT see staggering gains in strengths but after 25 MGy plummeted to strength levels less than half of 101K
- Again, in terms of relative radiation resistance CTD-101K is the outperformer
- Flexural data show remarkable symmetry with SBS equivalents, validating general findings.

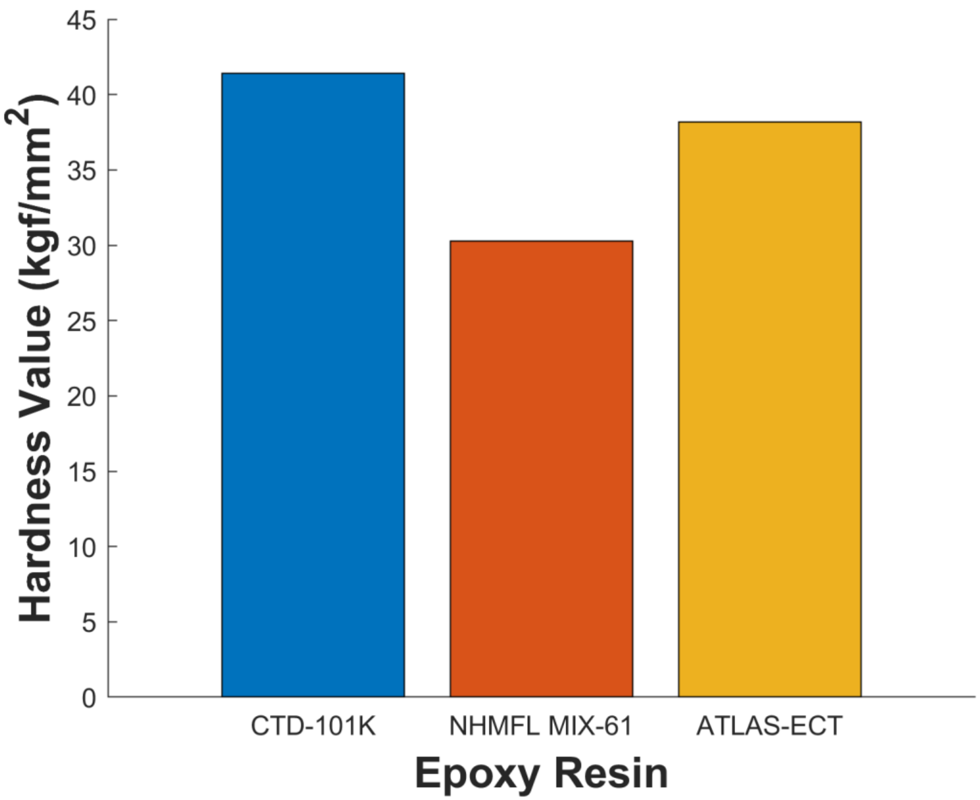
Hardness Testing

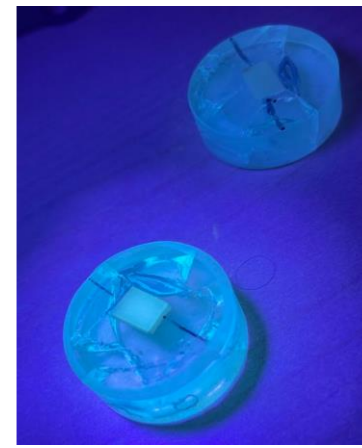
Experimenting with hardness testing was crucial in revealing microstructural features

CTD-101K



ATLAS-ECT





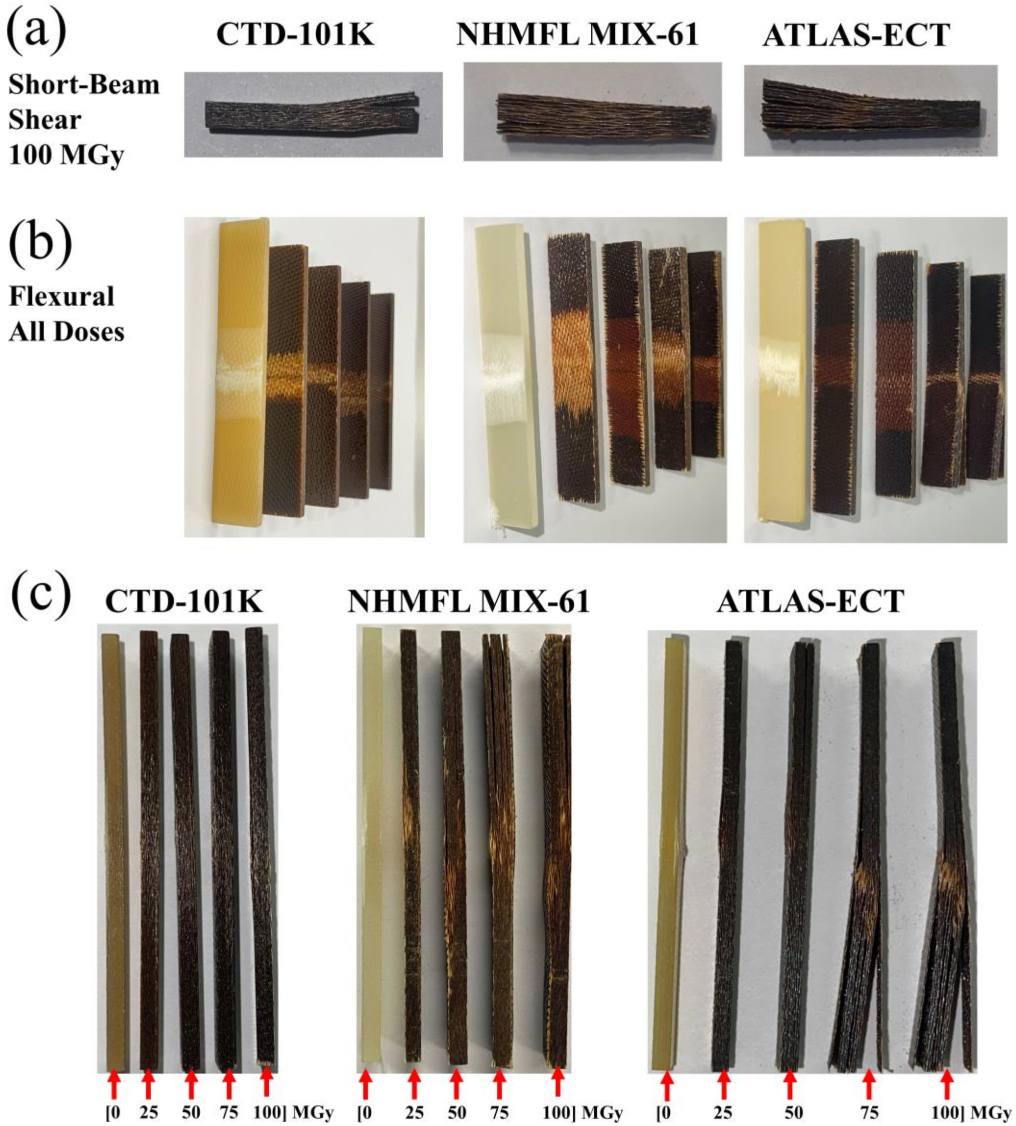
101K, ECT under blacklight

RT hardness testing revealed CTD-101K had the highest Vickers Pyramid Number. Tests at 77 K were impossible (visibility issues, thermal shock, etc.) and motivated microscopy

Failure-Mode Analysis

FMA consistent with SBS, Flex findings.
This and hardness pointed to microscopy

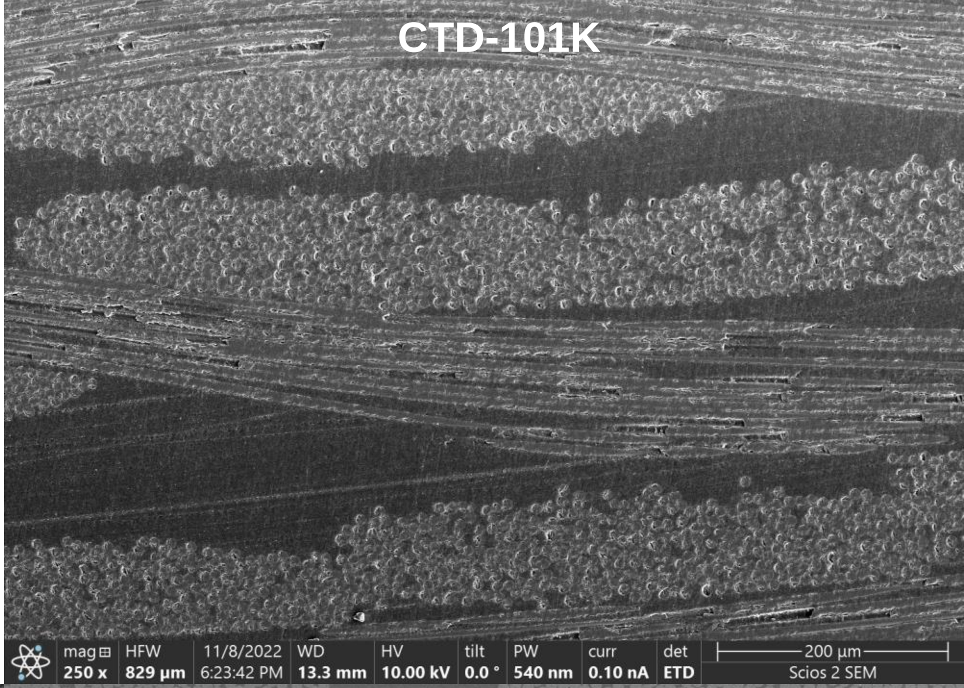
- Post-mortem failure analysis was done to better understand the nature of sample failure
- Interlaminar shear was the primary failure mode for both SBS and Flex
- SBS samples showed 101K maintaining best structural integrity while mix-61, ECT 'fanned out' more from the compressive loading
- Flexural samples echoed this, with 101K showing very little of this fanning due to interlaminar shear, whereas mix-61 saw some shearing through entire length. ECT was worst, also shearing through the length but with many more layers

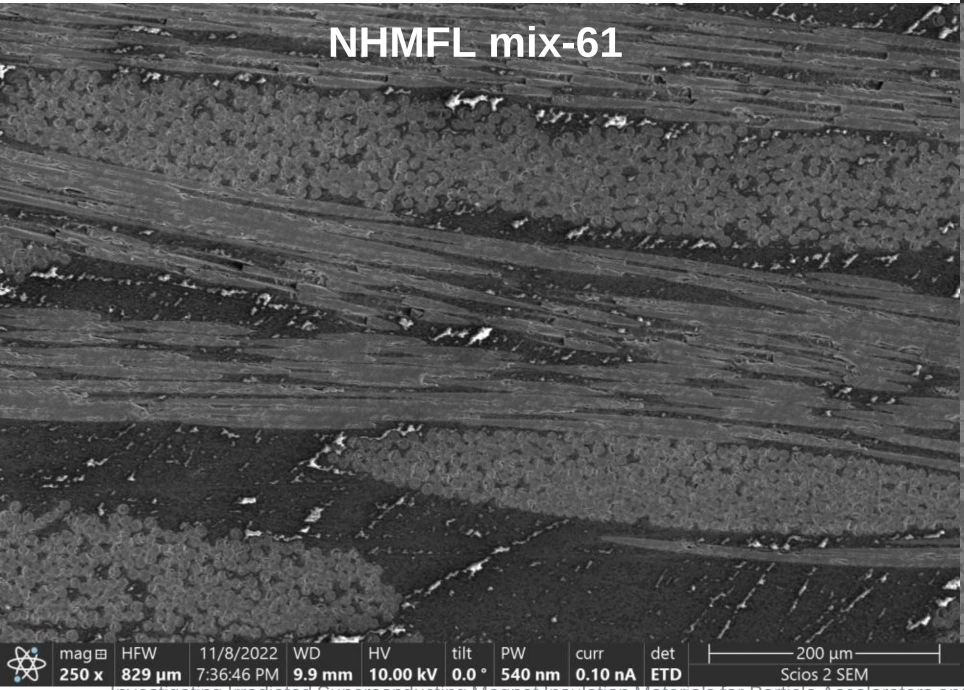


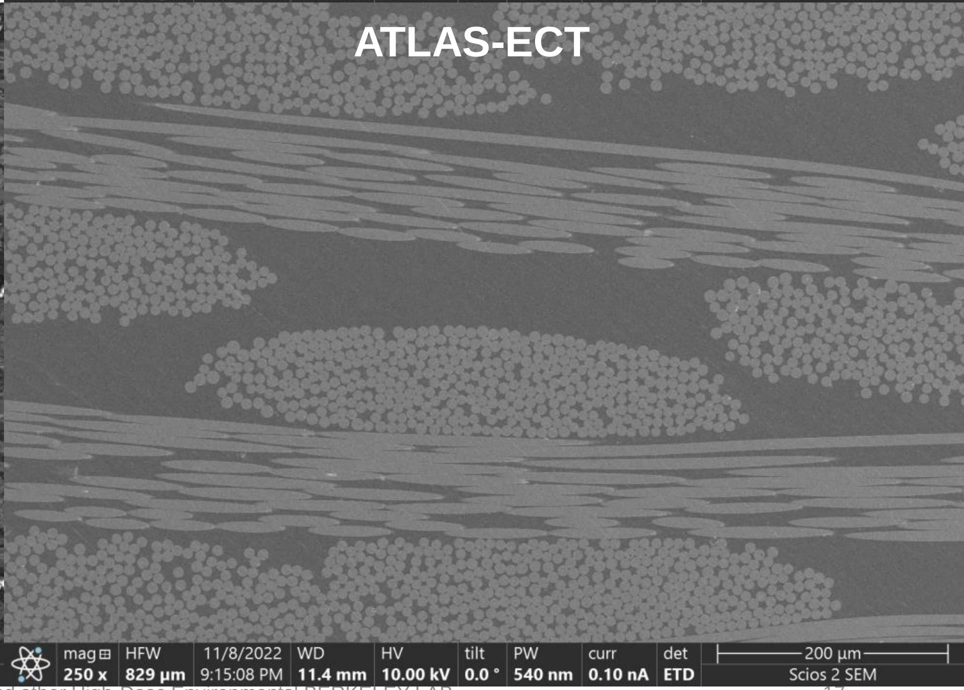
Microstructural Analysis

SEM revealed interesting details

Here are all three materials before irradiation. Interestingly, ECT seemed to have the finest microstructure before irradiation, with 101K and mix-61 roughly the same



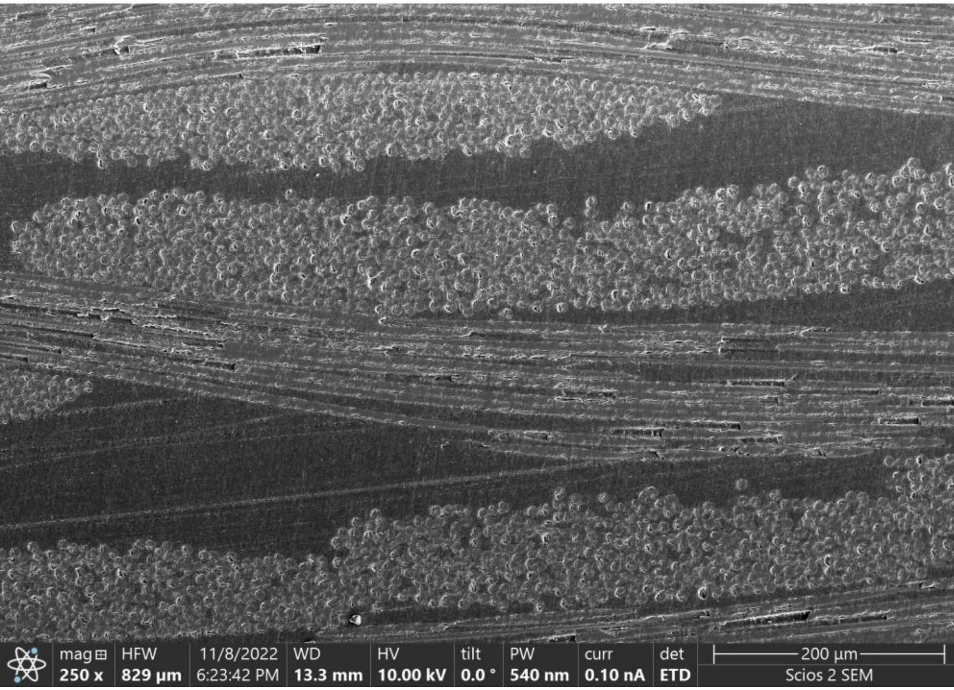




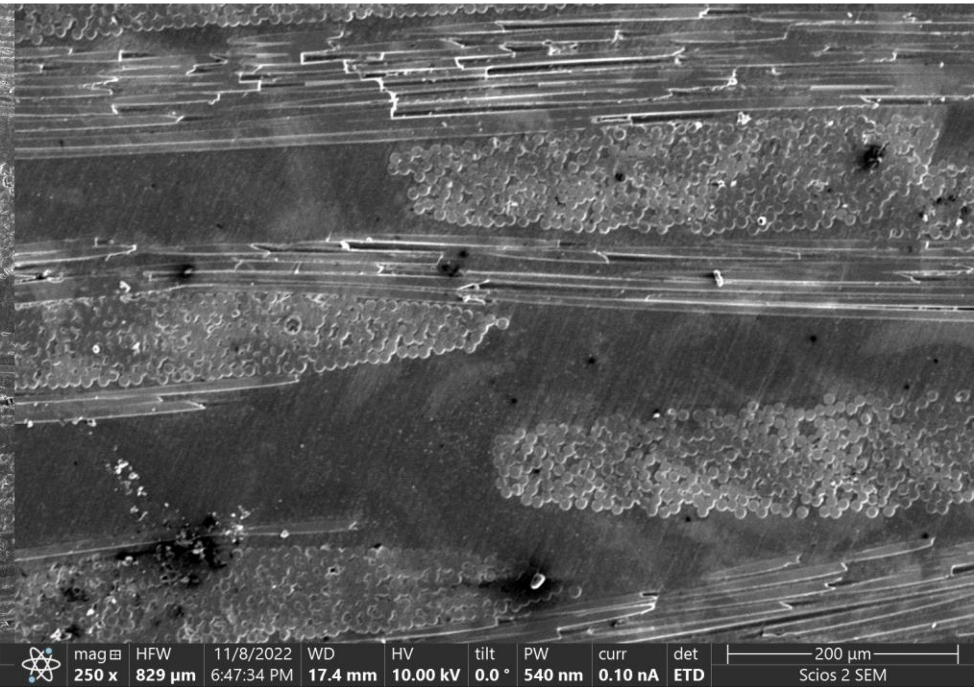
Microstructural Analysis

CTD-101K showed little microstructural change even at the highest dose

CTD-101K, 0 MGy

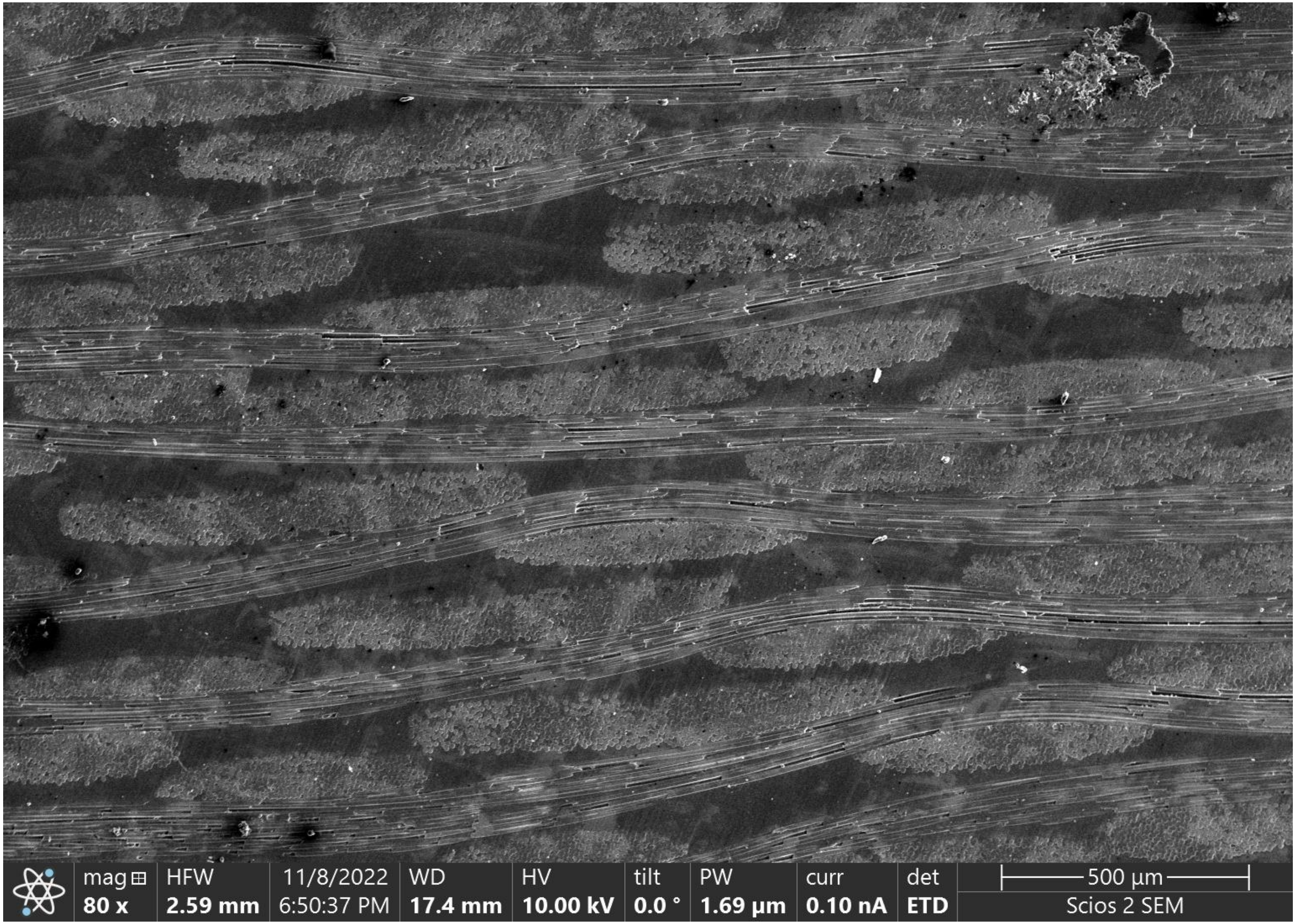


CTD-101K, 100 MGy



S-2 matrix fibers undergo embrittlement, flaking, and breaking out of place in some spots. Besides this, the microstructure appears to hold up very well, especially when compared to the changes seen in the other two epoxies

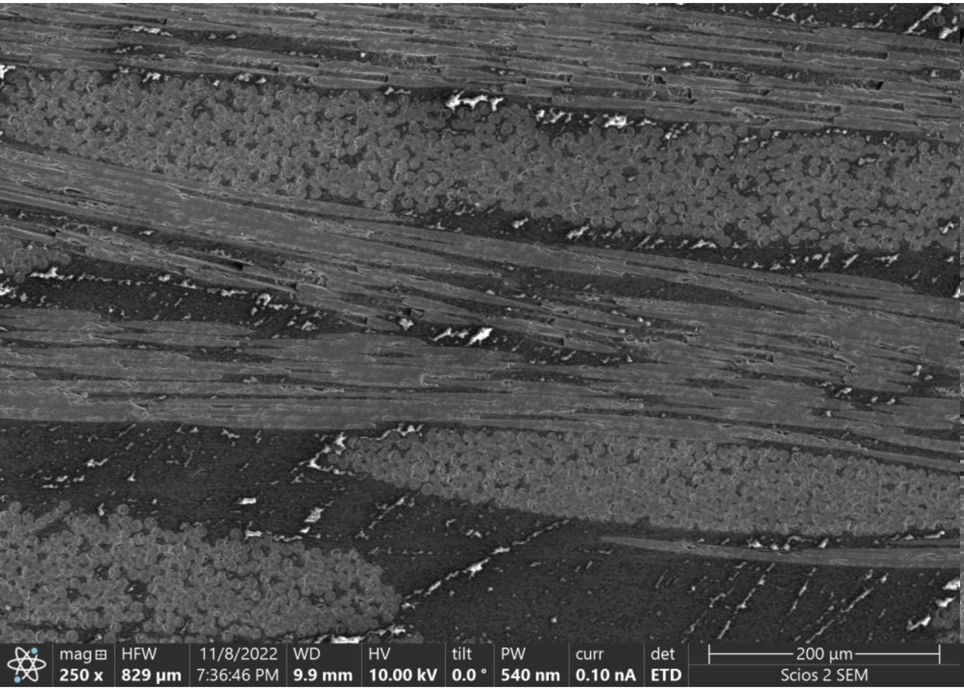
CTD-101K 100MGY



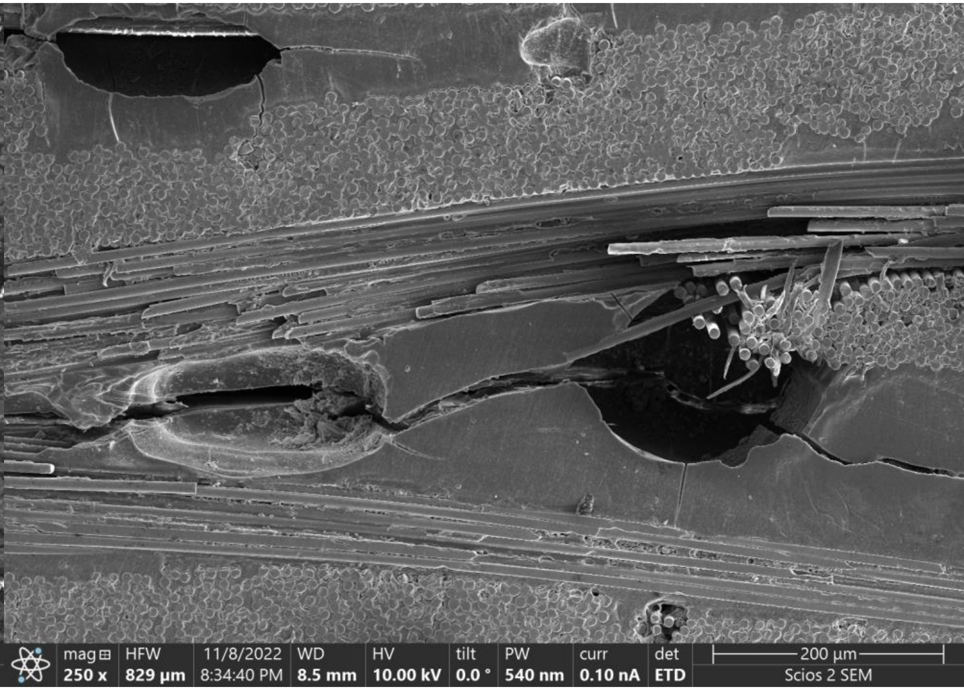
Microstructural Analysis

Mix-61 saw significant bubble production, prescient of macroscopic swelling and behavior observed

NHMFL mix-61, 0 MGy

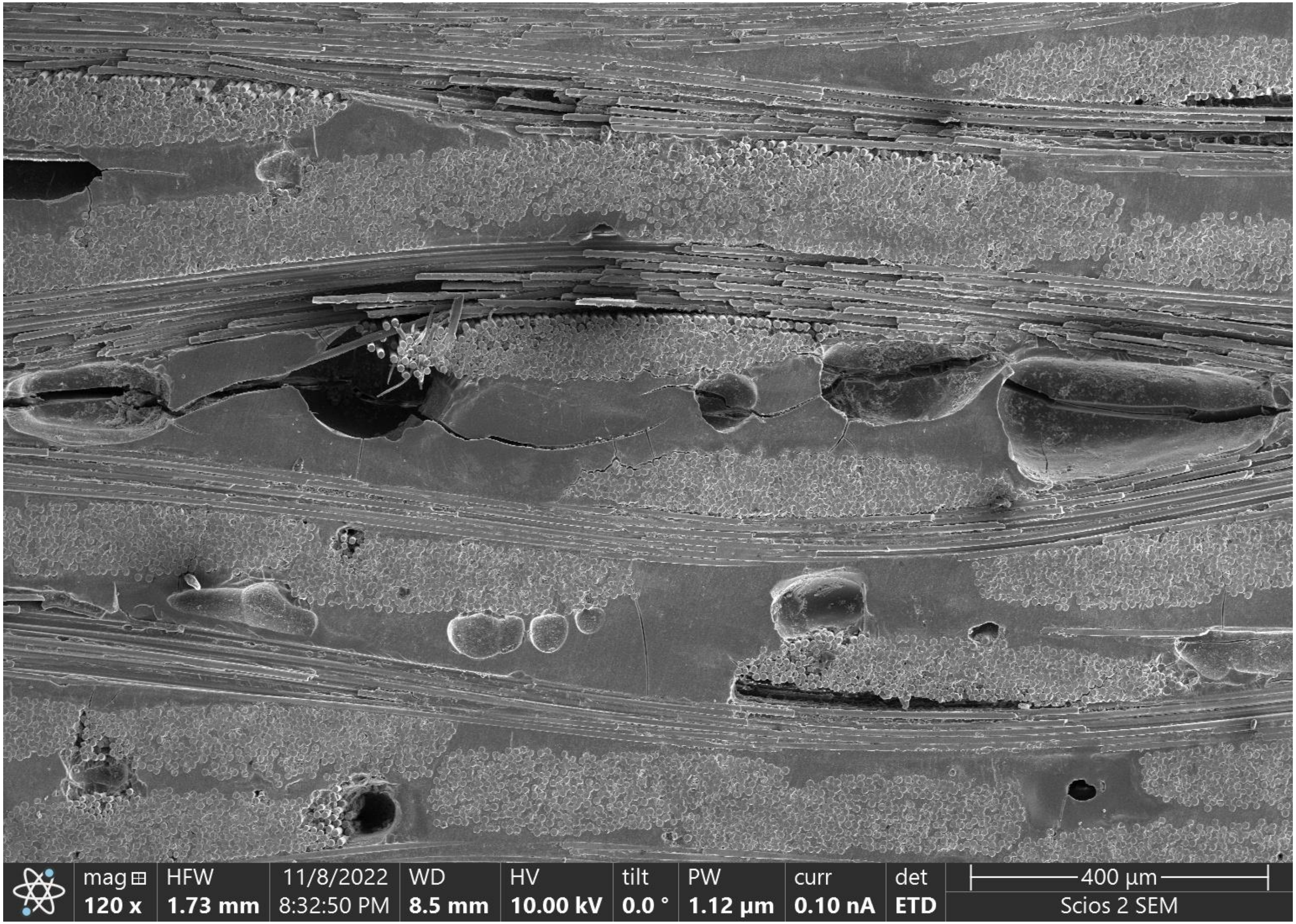


NHMFL mix-61, 100 MGy



Mix-61 exhibits widespread bubble production throughout its entire thickness. Gas evolution is a well-known consequence of irradiating organic materials, with bubbles accumulating when they cannot diffuse out fast enough. Moreover, we see cracks propagating along these bubbles, hinting at the physical mechanism underpinning the macroscopically observed failure mode

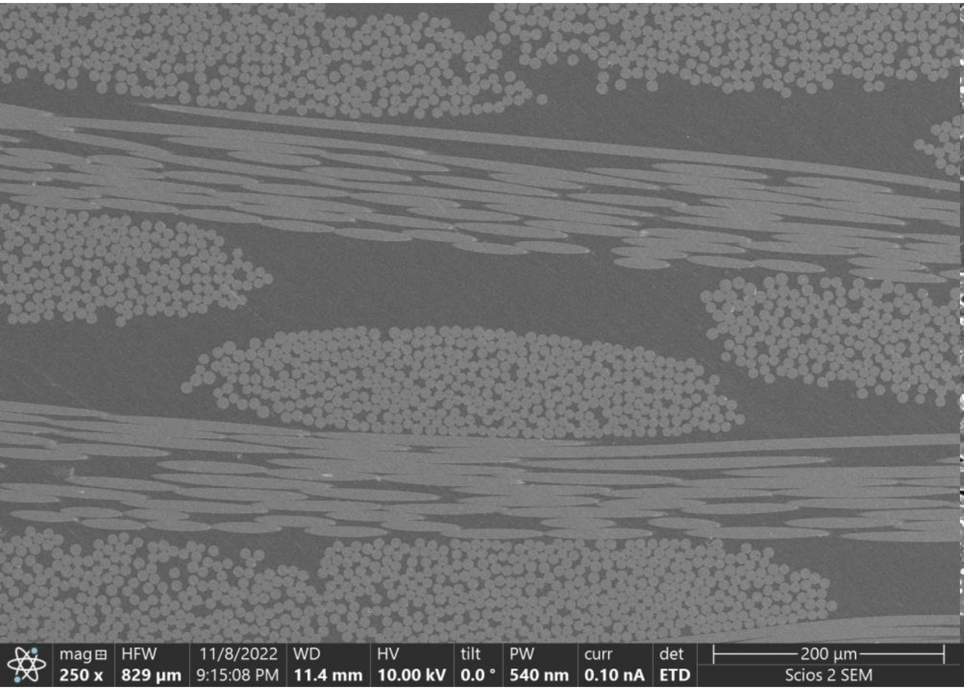
NHMFL mix-61 100MGY



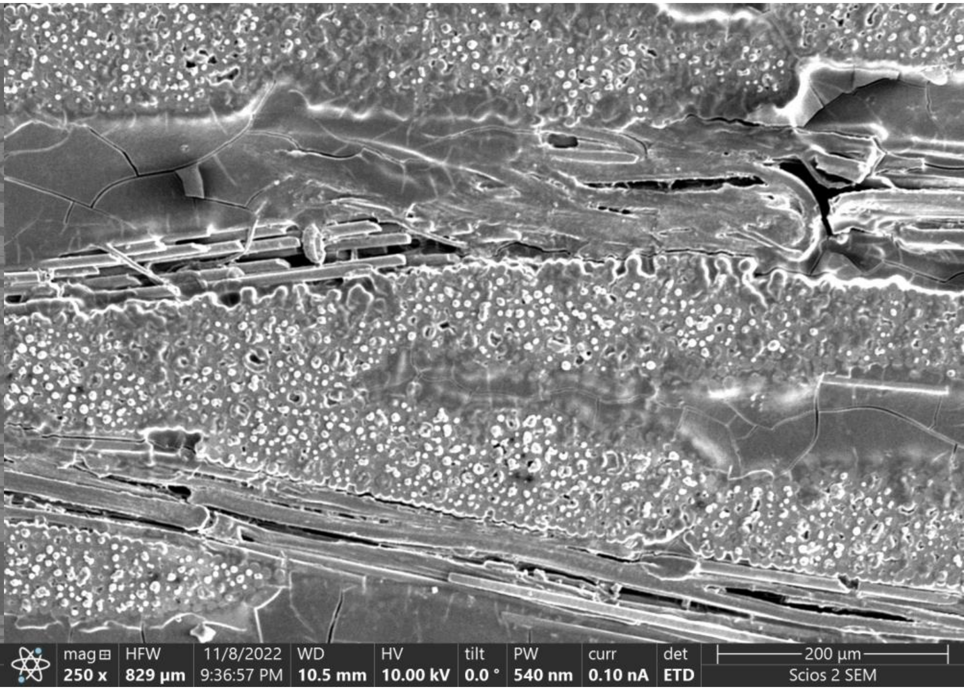
Microstructural Analysis

ATLAS-ECT saw major fiber breakdown, voids and widespread epoxy cracking

ATLAS-ECT, 0 MGy

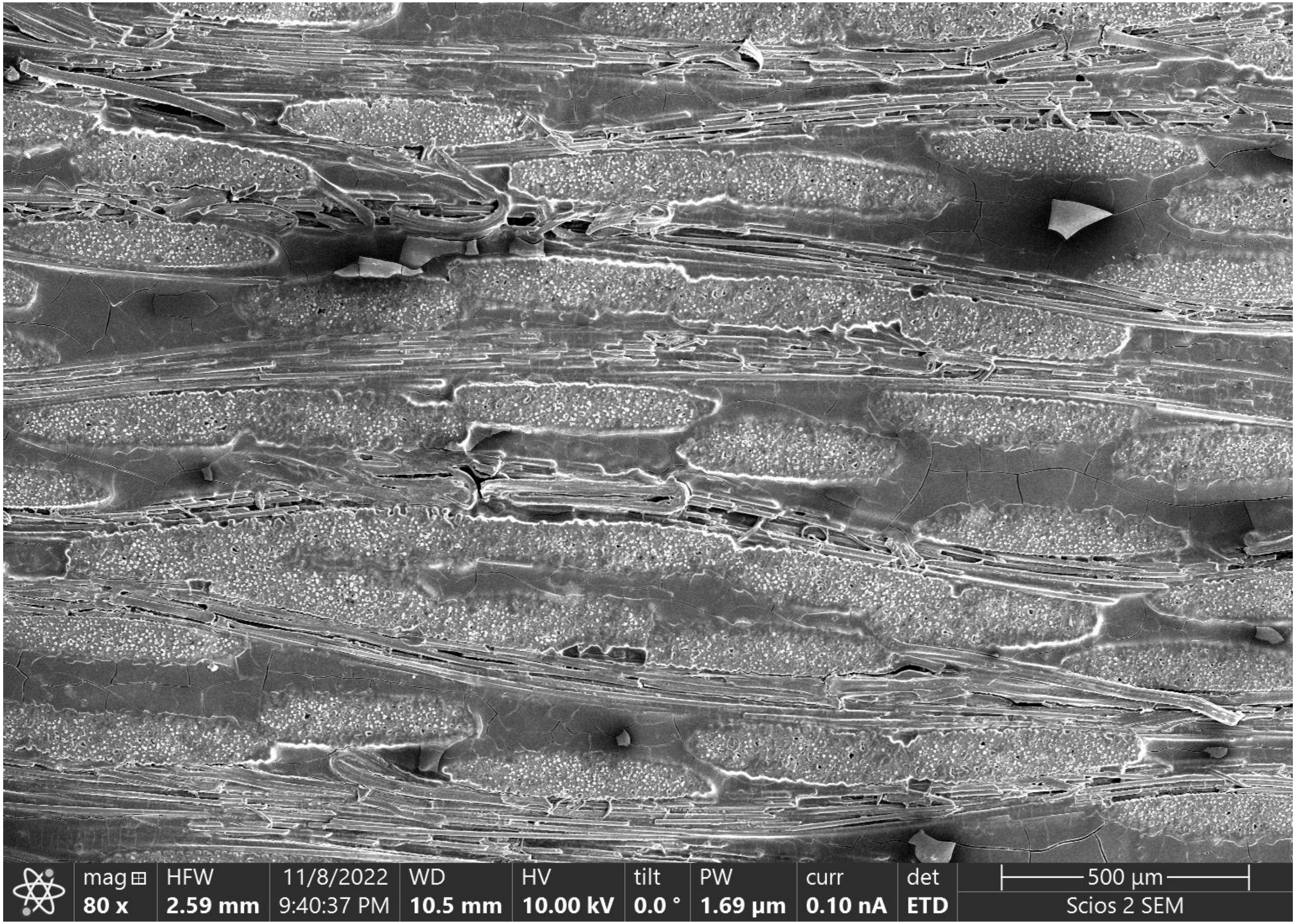


ATLAS-ECT, 100 MGy



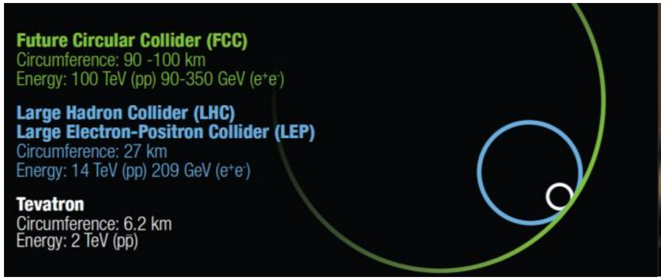
This material also undergoes a much more drastic change as a function of dose when compared to CTD, akin to yet somewhat different than mix-61. The S-2 fibers underwent a substantial deformation, resulting in voids within individual bundles. The resin itself also suffered much more pervasive cracking and became eroded near the glass-resin boundaries along which cracks clearly propagated. These again help elucidate the macroscopically observed failure mode

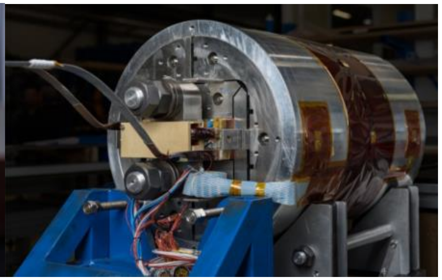
ATLAS-ECT 100MGY



Conclusions

Radiation levels crucial for selecting the right material

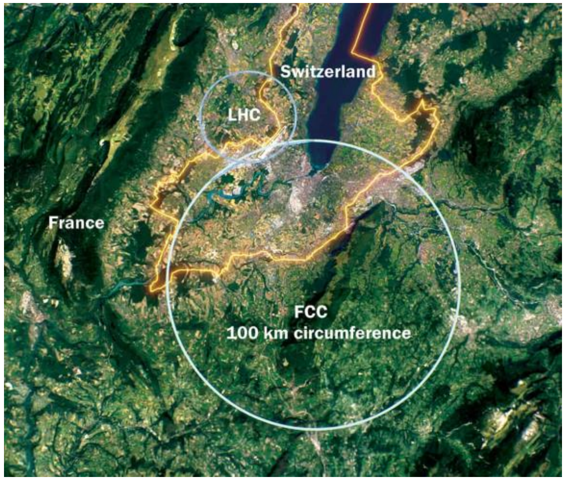


Conclusions

Radiation resistance a key factor in materials selection for future LTS magnets

- Sensitivity to radiation was perhaps the biggest takeaway from testing
- From the data, we recommend amine-based epoxies like NHMFL mix-61 and ATLAS-ECT for low-dose applications such as NMR due to clearly superior toughness
- For high-radiation environments like High-Energy Physics and Magnetic Confinement Fusion we recommend CTD-101K for its strength and unparalleled radiation resistance



Thank You!