American Society for Gravitational and Space Research
2014 Annual Meeting, Oct. 11-14, 2015
Alexandria, Virginia

Evaluation of Two Ionic Liquid-Based Epoxies from the MISSE-8 (Materials International Space Station Experiment-8) Sample Carrier

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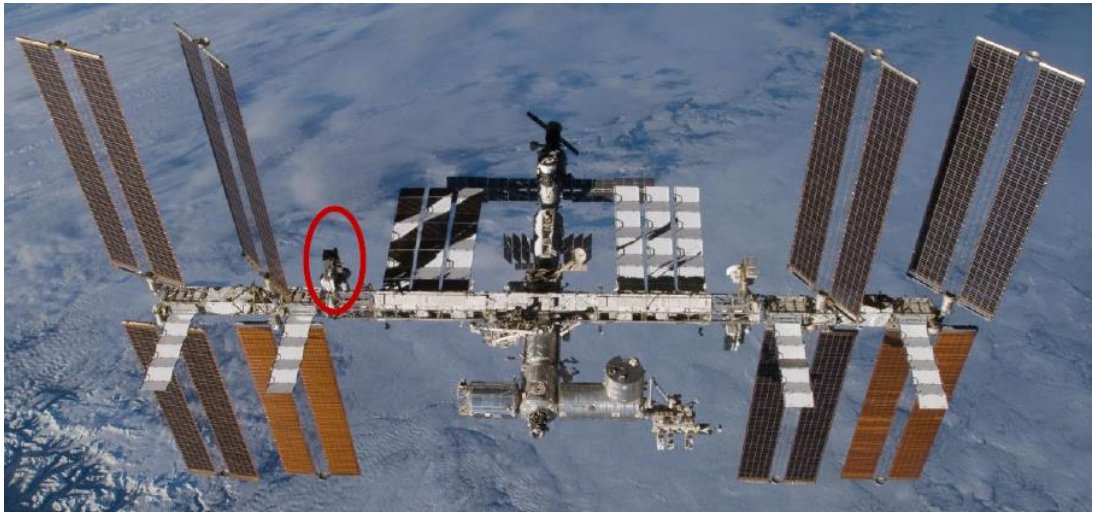
Materials International Space Station Experiment-8*

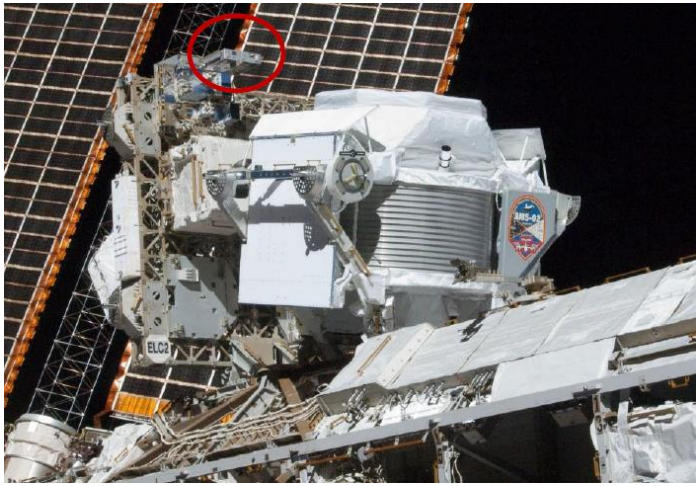
- Deployed May 20, 2011 on STS-134
- Located on Express Logistics Carrier-2 (ELC-2)
- Retrieved July 9, 2013
- Returned to Earth May 2014 on SpaceX Dragon CRS-3

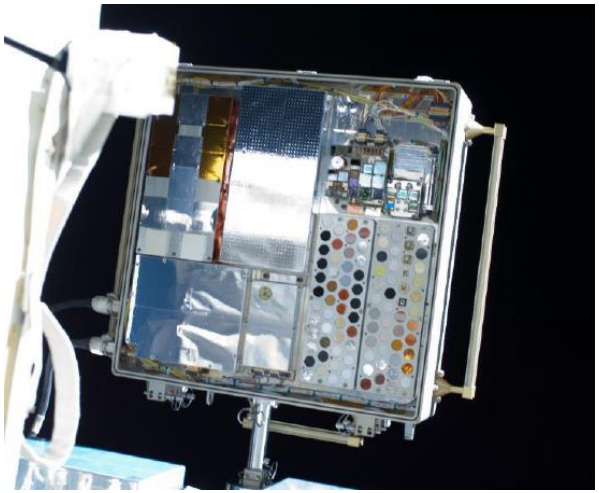
MSFC flew two passive sample trays on nadir side. Total of 96 samples.

*M.M. Finckenor: National Space & Missile Materials Symposium, Chantilly, VA, June 2015.

MISSE-8





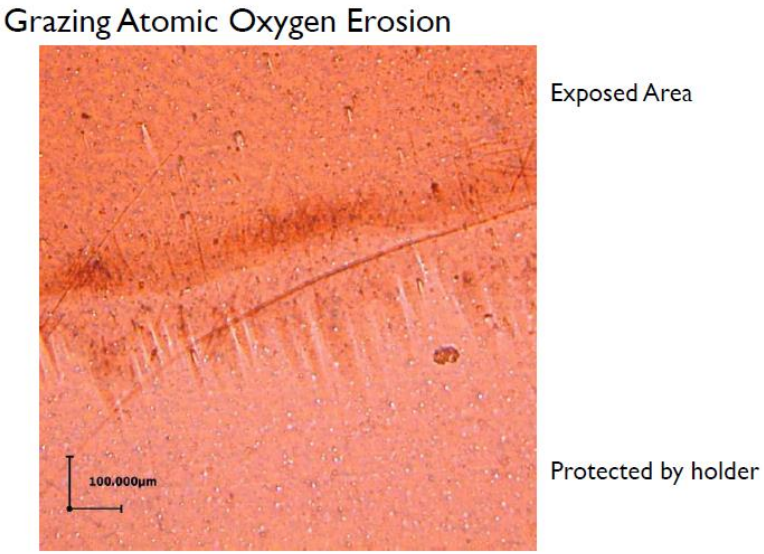


Atomic Oxygen Fluence

$3.6 \pm 0.1 \times 10^{19}$ atoms/cm^2

Determined by mass loss and thickness loss of Kapton HN

Very low fluence due to nadir location and ISS shielding



*M.M. Finckenor: National Space & Missile Materials Symposium, Chantilly, VA, June 2015.

Ultraviolet Radiation Exposure

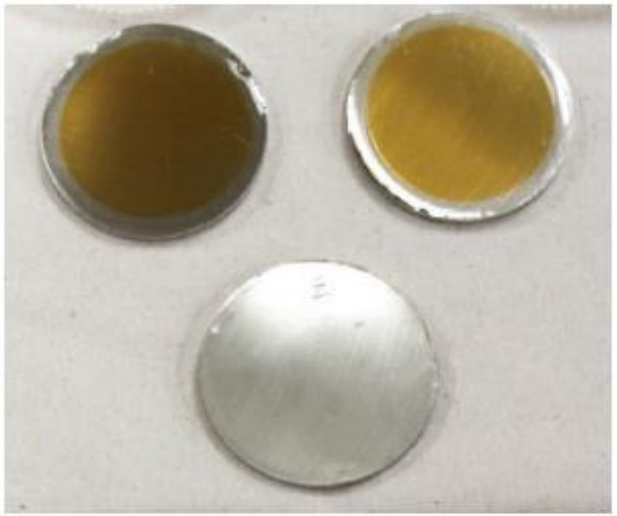
Exact dose unknown at this time

UV darkening observed on beta cloth, IL epoxy samples, others

This suggests a minimum of 500 equivalent sun hours (ESH).

Ionic fluid samples – Flight

Control



*M.M. Finckenor: National Space & Missile Materials Symposium, Chantilly, VA, June 2015.

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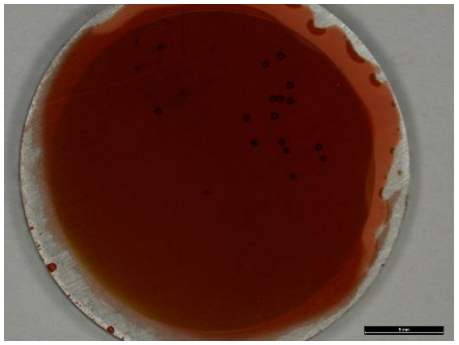
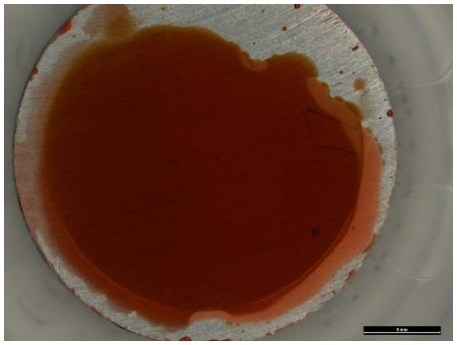
Pre-flight

Post-flight

Environmental Conditions

- 2 years and 2 months Nadir Exposure
- A O Fluence $3.6 \pm 0.1 \times 10^{19}$ atoms/cm^2
- 12,500 cycles between ~-40°C and +40°C
- High Vacuum Environment, Radiation





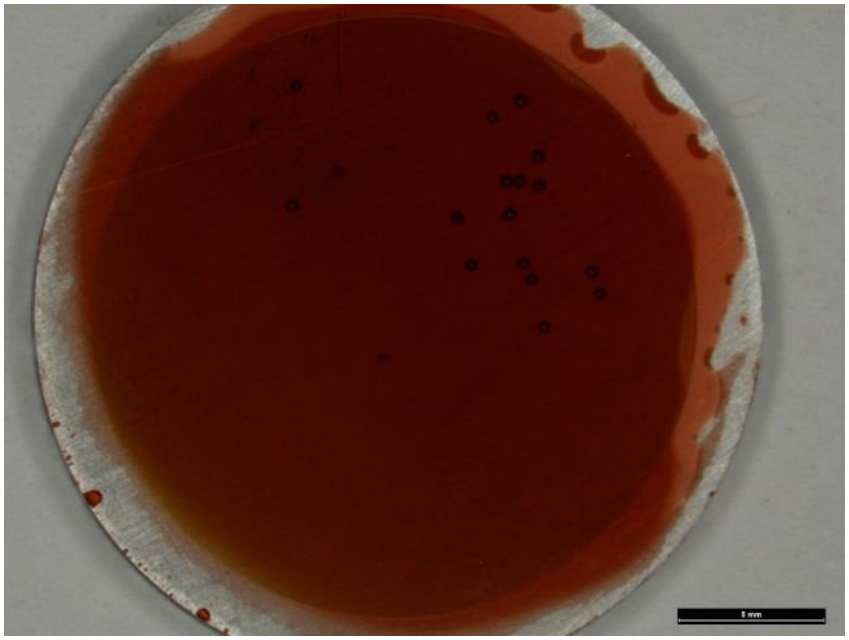
ILEP 15 ILEP 17

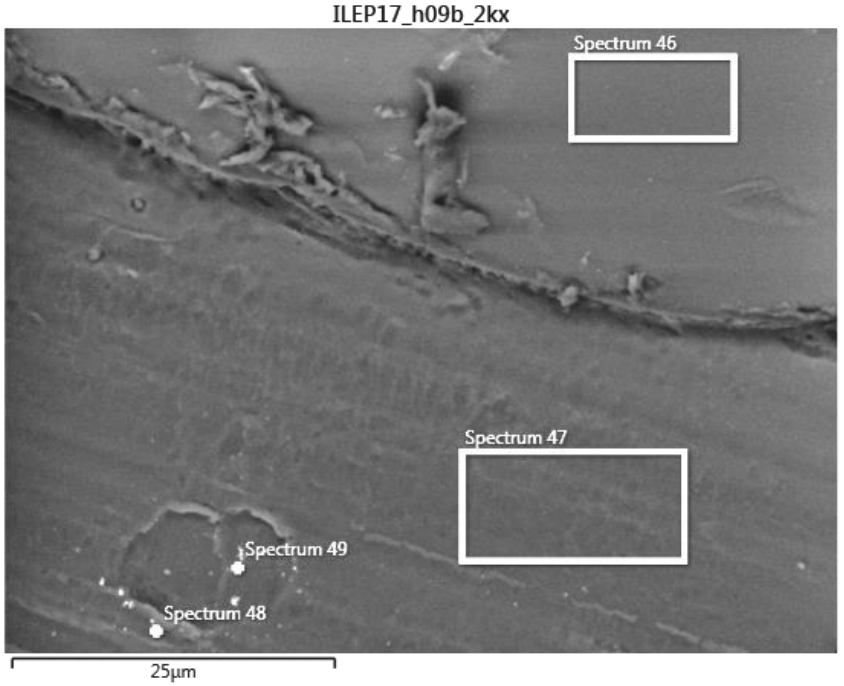
	Pre-flight weight (g)	Post-flight weight (g)	Delta
ILEP 15	2.35486	2.3547	-0.00016
ILEP 17	2.32939	2.32902	-0.00037

Initial Observations

- Negligible Weight Change
- Continued Strong Adherence

Continued Evaluation

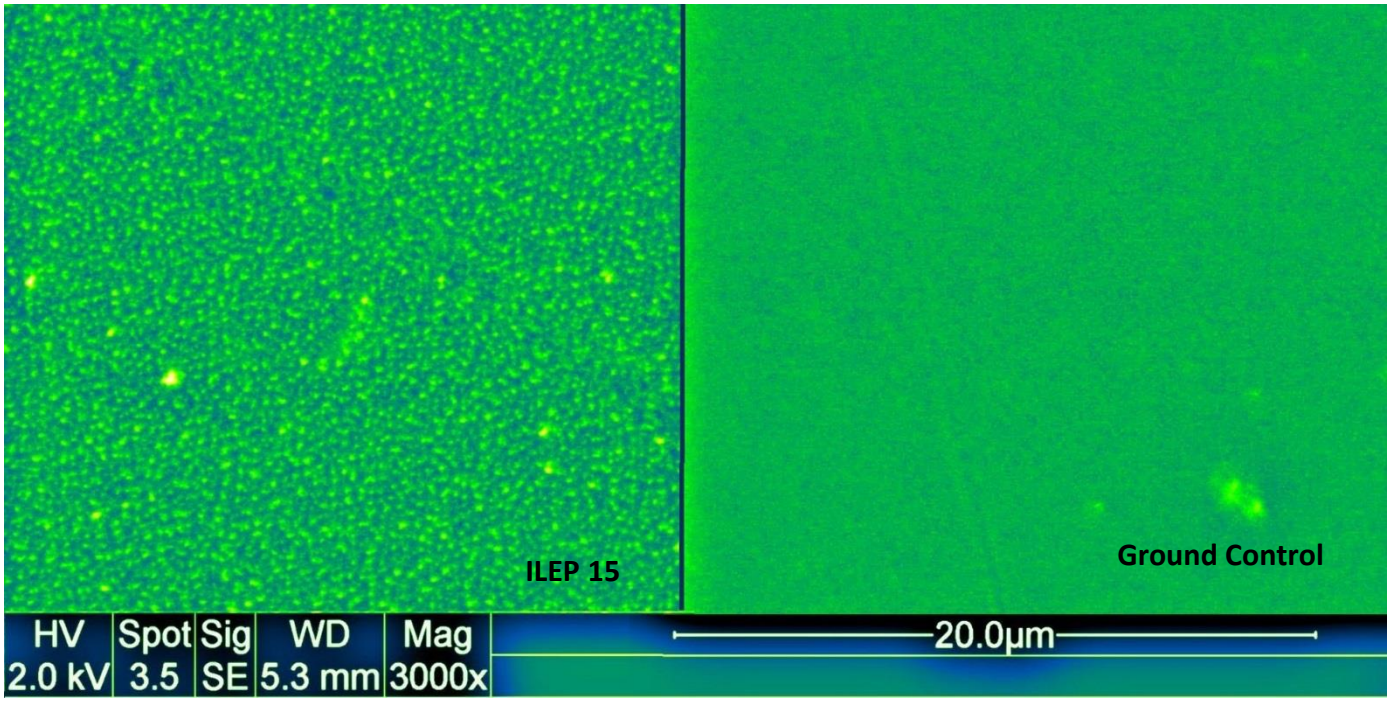




Label	C	O	F	Al	S	Cl
Spectrum 46	64.43	14.12	13.73	0.55	6.59	0.59
Spectrum 47	65.38	14.76	13.17	0.48	5.51	0.70

Color change likely a result of ultraviolet (UV) radiation

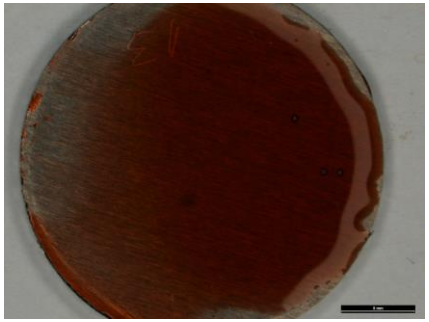
Nano-scale Dimpling



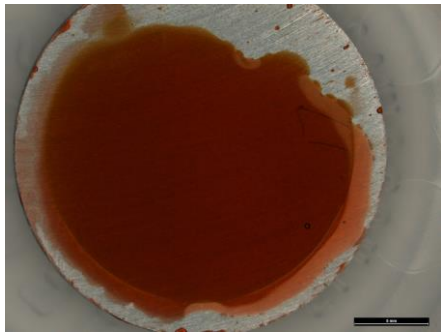
Referencing the MISSE Database for similar surface structures

Note: Air Curing Epoxy results in an oxidation layer, scales may be similar

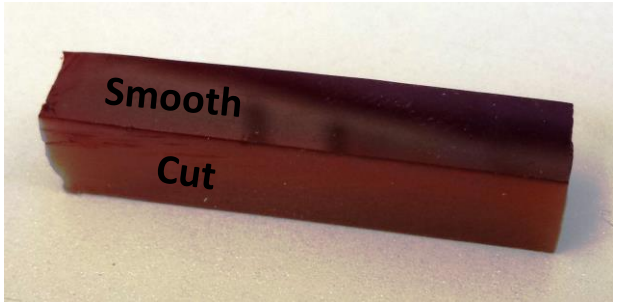
ESCA (Electron Spectroscopy for Chemical Analysis)



ILEP-13 Ground

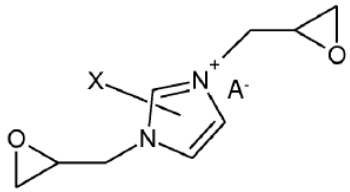


ILEP-17 ISS

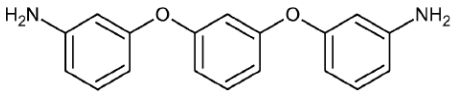


IL Epoxy Ground Test Samples
- Smooth – Exposed during cure
- Cut – Interior bulk material

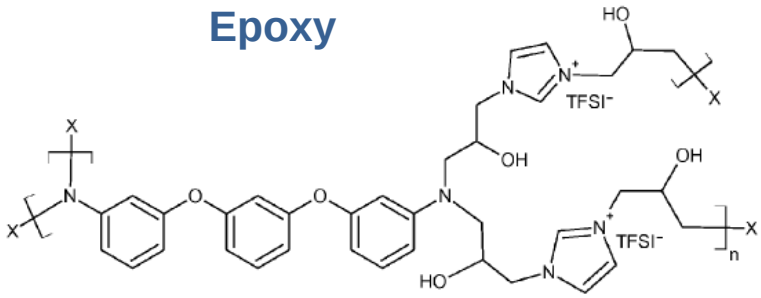
Epoxy Monomer + **Curing Agent** = **Epoxy**



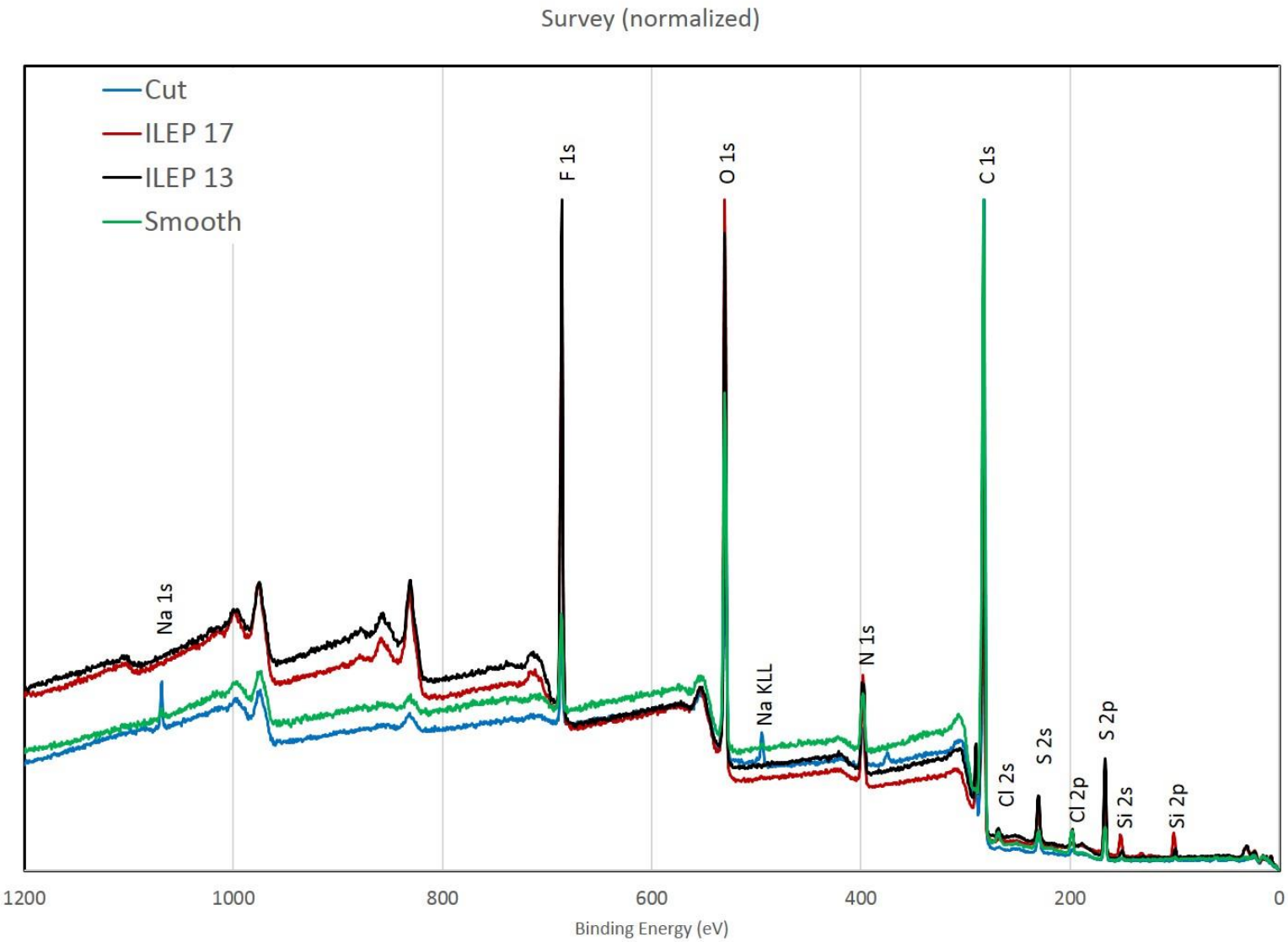
1,3-bisglycidylimidazolian

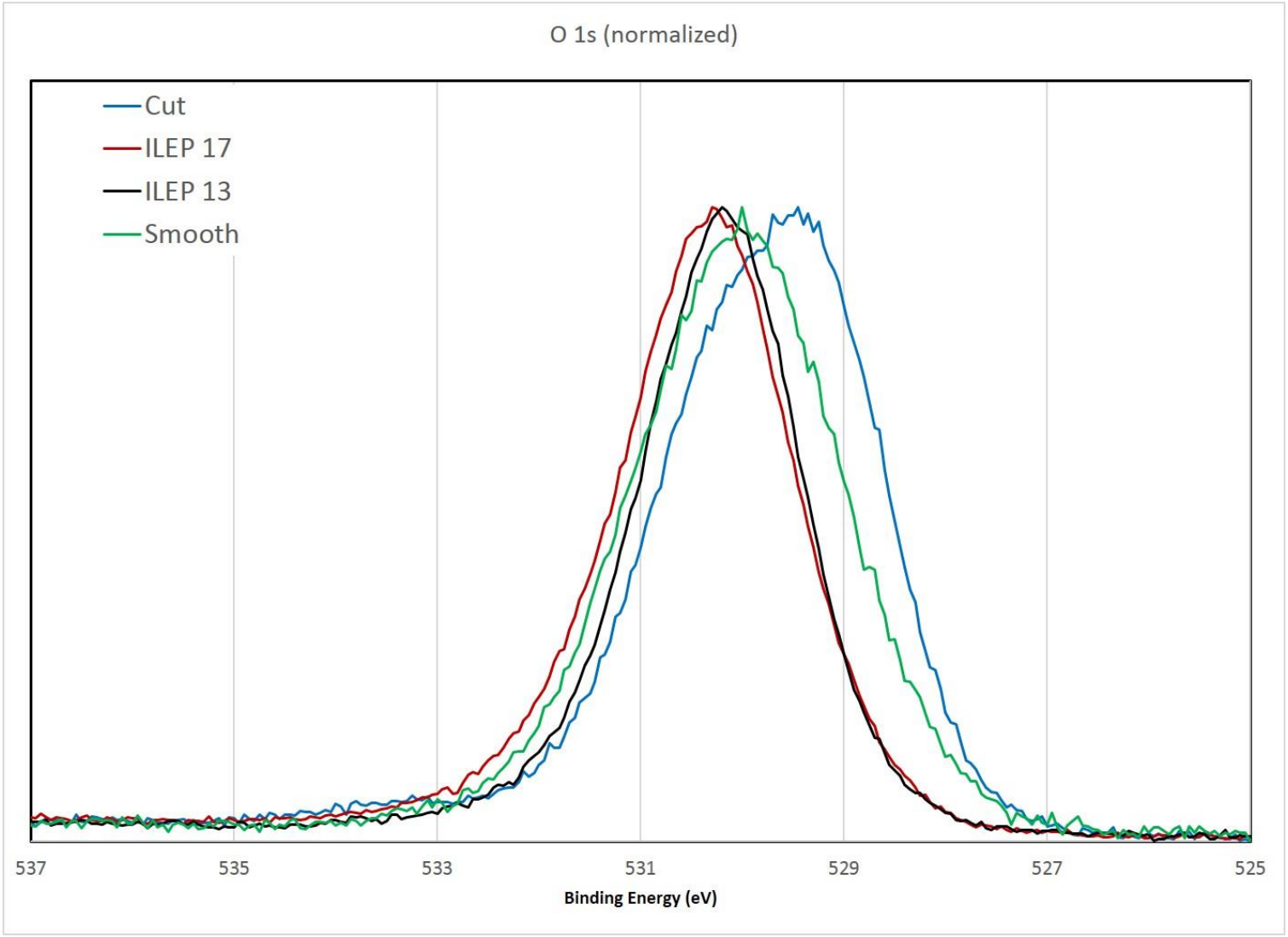


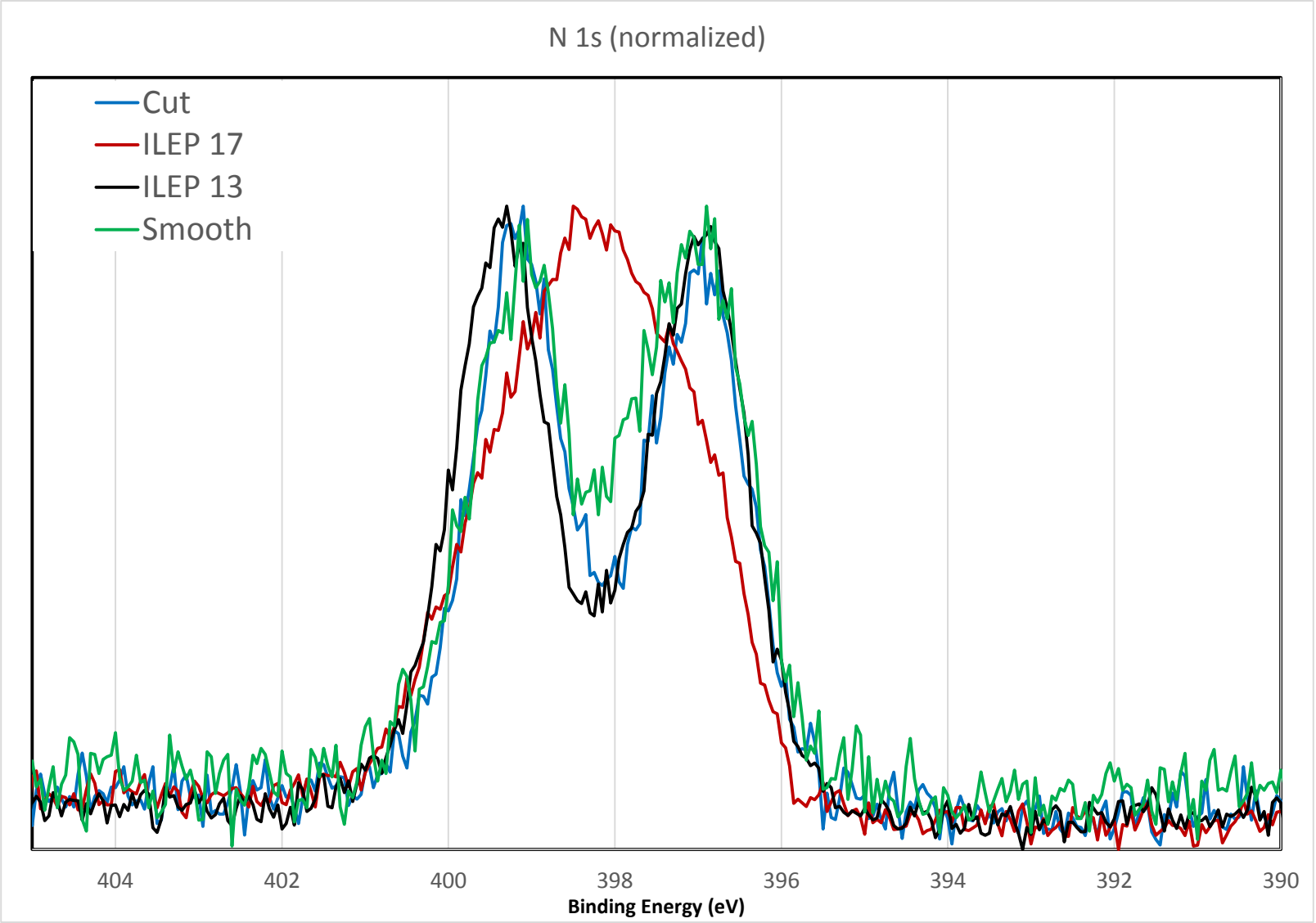
Bis-Aminophenoxybenzene (APB)

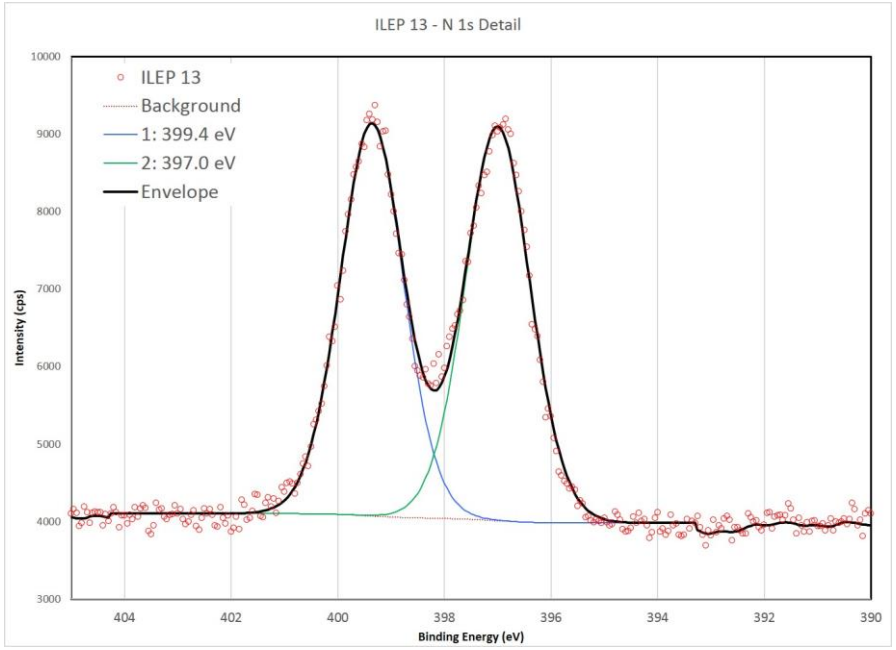


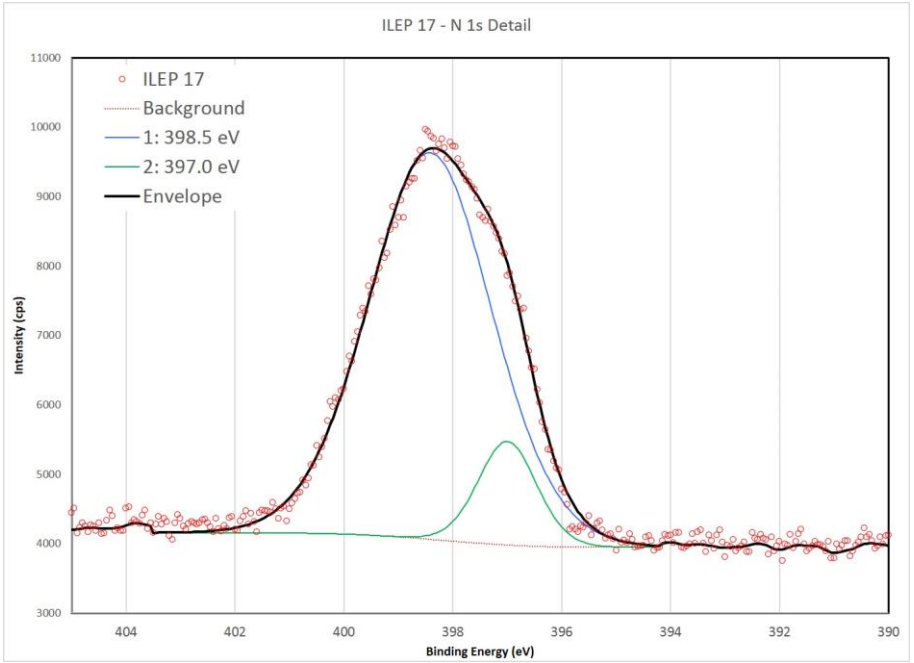
ILEP-2

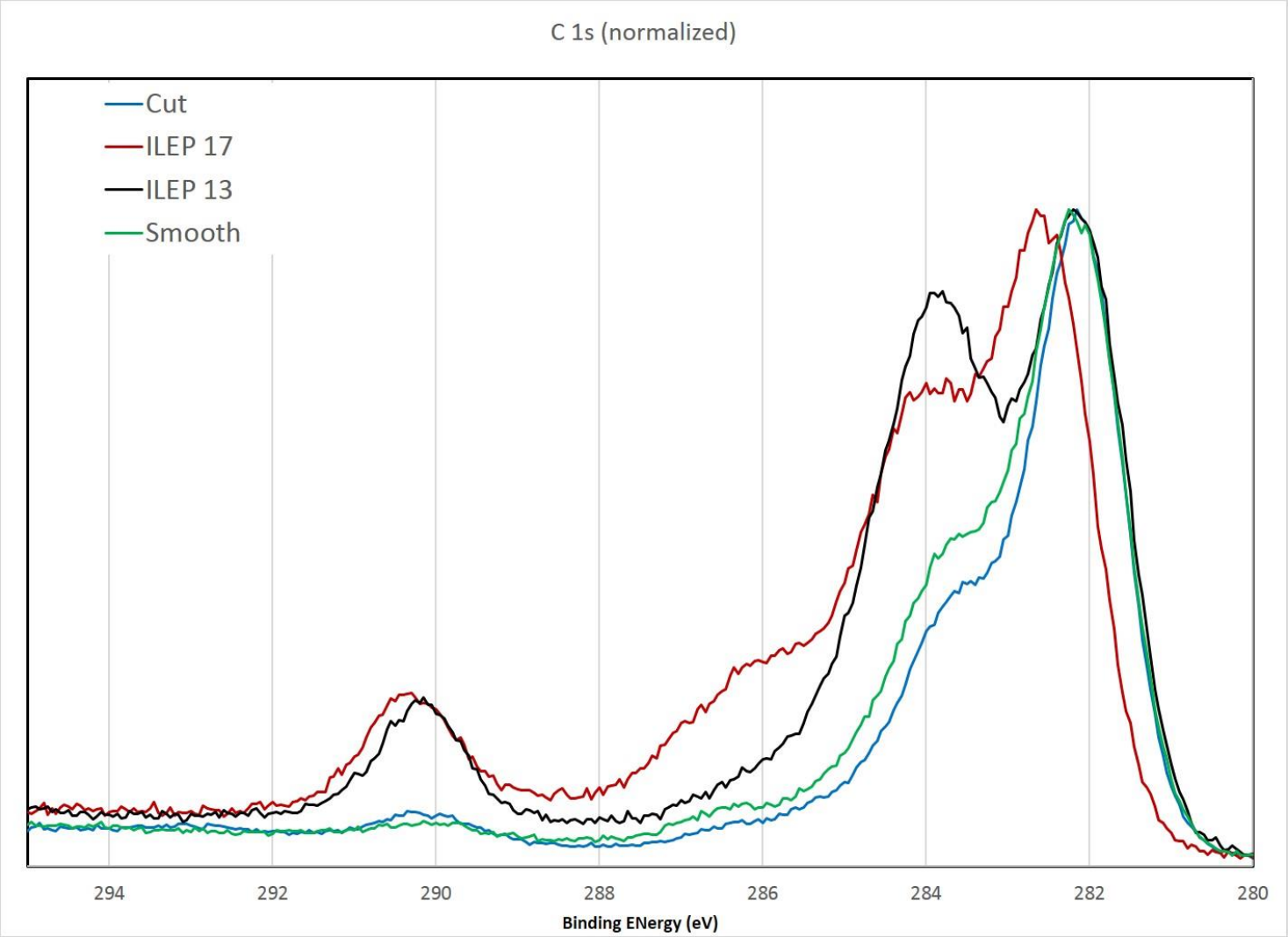

Survey (normalized)
Cut
ILEP 17
ILEP 13
Smooth
F 1s
O 1s
C 1s
Na 1s
Na KLL
N 1s
Cl 2s
S 2s
Cl 2p
S 2p
Si 2s
Si 2p
1200
1000
800
600
400
200
0
Binding Energy (eV)


O 1s (normalized)
Cut
ILEP 17
ILEP 13
Smooth
537
535
533
531
529
527
525
Binding Energy (eV)


N 1s (normalized)
Cut
ILEP 17
ILEP 13
Smooth
404
402
400
398
396
394
392
390
Binding Energy (eV)


ILEP 13 - N 1s Detail
ILEP 13
Background
1: 399.4 eV
2: 397.0 eV
Envelope
Intensity (cps)
Binding Energy (eV)
10000
9000
8000
7000
6000
5000
4000
3000
404
402
400
398
396
394
392
390


ILEP 17 - N 1s Detail
ILEP 17
Background
1: 398.5 eV
2: 397.0 eV
Envelope
Intensity (cps)
Binding Energy (eV)
11000
10000
9000
8000
7000
6000
5000
4000
3000
404
402
400
398
396
394
392
390


C 1s (normalized)
Cut
ILEP 17
ILEP 13
Smooth
294
292
290
288
286
284
282
280
Binding ENergy (eV)

Summary of IL Epoxy Exposure on MISSE-8

- No weight change
  - ♦ Extremely low vapor pressure
- Continued strong adherence to aluminum base
  - ♦ No cracking, de-bonding, or other observable deformations
- Nano-scale dimpling on surface
  - ♦ Not resolved
- ESCA results
  - ♦ Some bond breaking of the N molecules on the surface
  - ♦ No obvious O changes
  - ♦ C variance probably due to contamination
    - → Analysis ongoing

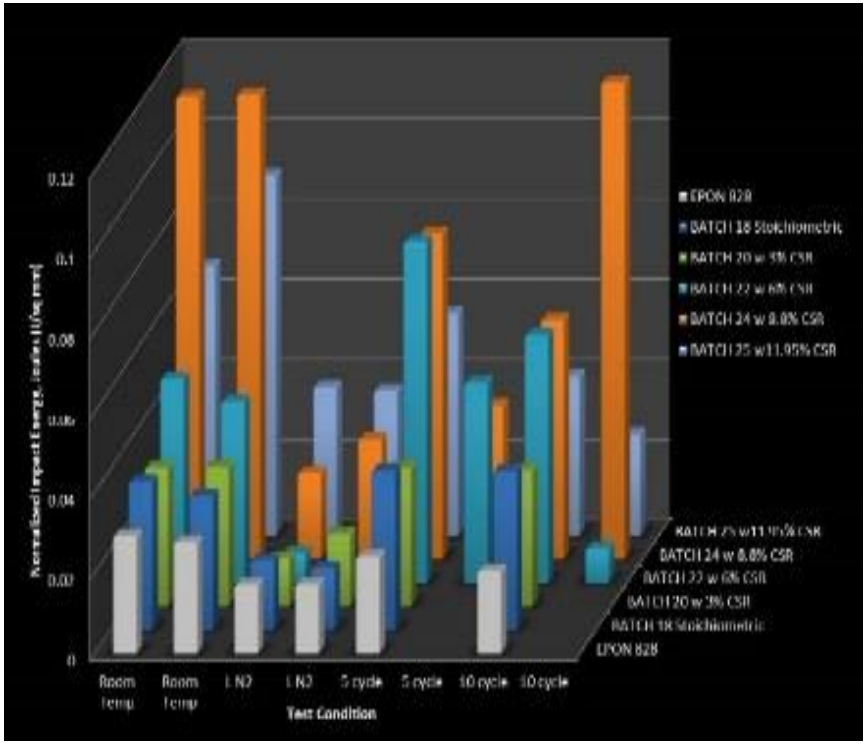
Appears to well tolerate the harsh environment of space

Other Ionic Liquid Epoxy Properties

- Strong ionic bonding
- Very small coefficient of thermal expansion
- Hydrophobic

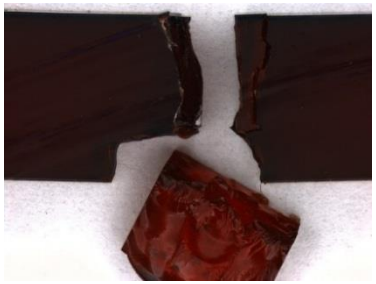
Applicable to Fabricating Carbon-fiber Composite Tanks for Cryogenic Liquid Containment

CSR: Impact test results

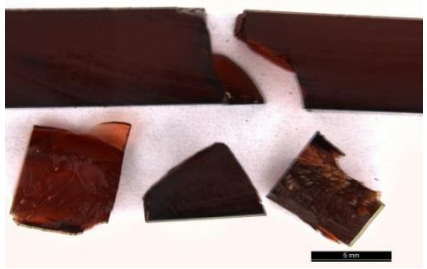


Plot of impact test results with increasing percentages of CSR for room and liquid nitrogen temperatures.

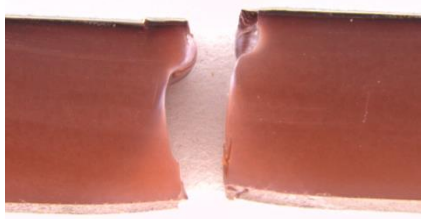
Comparable Improvement in Tensile Test Results



Batch 18
0% CSR RT



Batch 18
0% CSR
LN2



Batch 22
8.8% CSR RT

Batch 22
8.8% CSR LN2

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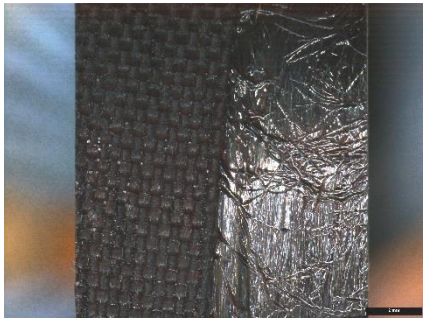
Cryogenic Testing in LOX and LH2

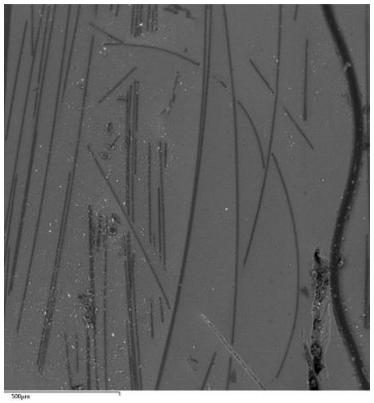
LOX: Potential Fuel Candidate, Much more Reactive than LN2
LH2: Potential Fuel Candidate, Much Colder (~20K) than LOX (~90K) or LN2 (~77K)

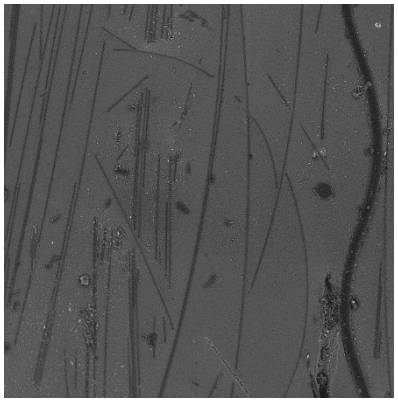
As-Fabricated Cylinder Section

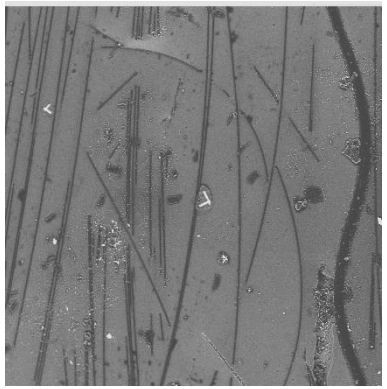
After two dunks in LOX



After LOX plus two 1 hour Soaks in LH2



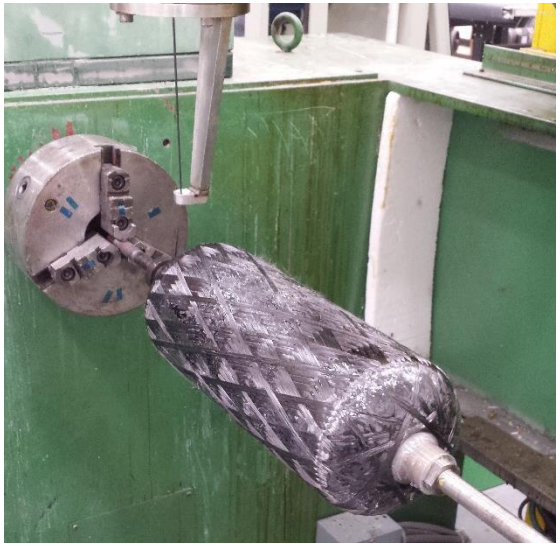




14 Samples Tested: High power microscopy and some fluorescent dye penetrant showed no degradation, cracking, or delamination

Epoxy is cured at 150°C (423K), 423K-20K (LH2) = ΔT = 403K!

Fabricate Composite Overwrap Pressure Vessels (COPV)



Wrapping

Curing

Epon 828 Resin with Huntsman T-403 Curing Agent

IL Resin (no CSR) with APB Curing Agent

Conclusions

- Ionic liquid-based epoxy well tolerates the space environment
- Other properties suggest application to fabricate carbon-fiber composite tanks for cryogenic liquid containment
- Testing/evaluation will continue

Acknowledgments

This work is supported by a MSFC Center Innovation Fund

Appreciation is expressed to Ms. Miria Finckenor for her contributions to the MISSE program and this presentation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