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U.S. DEPARTMENT OF ENERGY

Mechanical Performance of Irradiated Adhesive Samples for ATLAS ITk

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Overview

- Why should we do more radiation testing?
- Historic data
- Testing paradigm
- Good results and suspect results
- Conclusion

Existing Radiation Testing

- The ATLAS Inner Pixel detector has targeted Dow SE4445 for attaching modules to local supports
  - CERN has irradiated and tested SE4445, but not to the radiation level we need (https://maxrad.web.cern.ch/maxrad/)
  - It was noted during testing that some results were suspect
    - Peel tests had anomalous reports
- We decided we still needed to test new samples to understand:
  - Various glue behavior up to 15MGy
  - Glass spheres (bond line control) don’t affect bond strength
  - Additional adhesives: Hysol EA 9396, neat and filled with graphite powder (even in CERN yellowbook these are not tested beyond 10MGy)
  - Some adhesives were eliminated due to handling difficulty

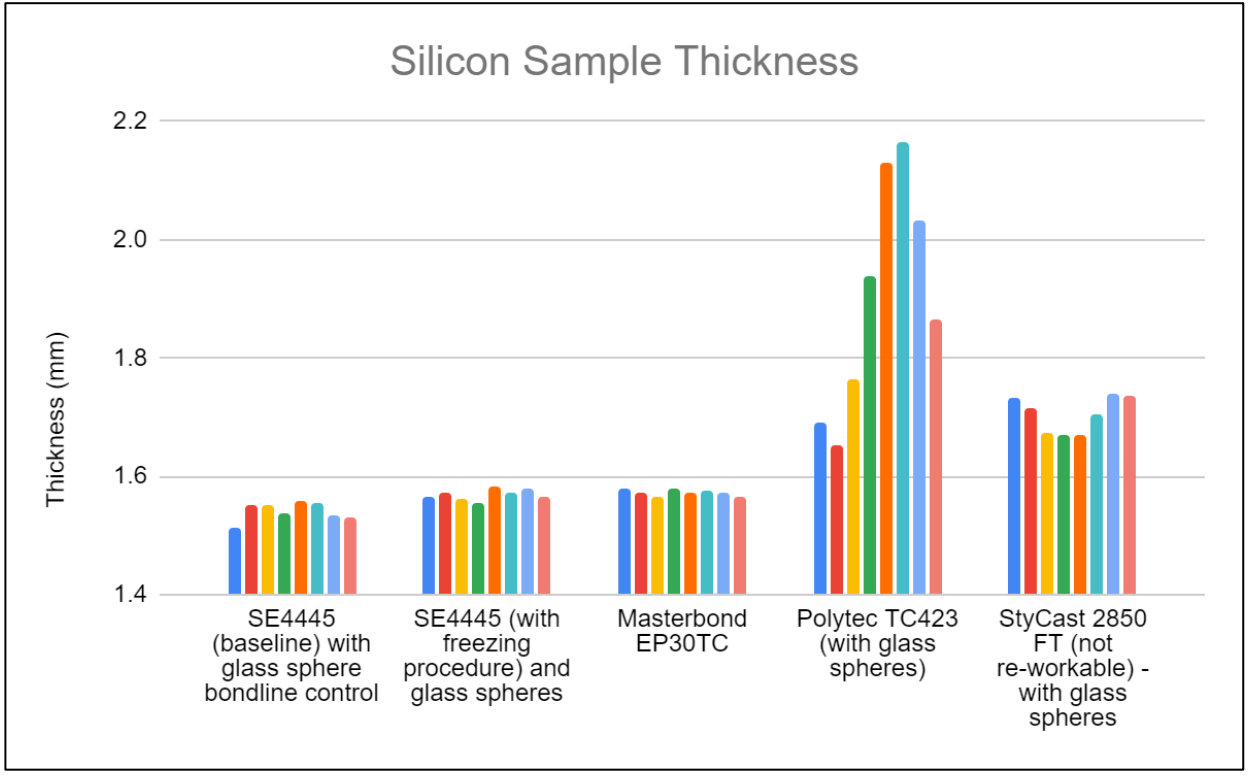
Table of Samples

Glue type		# samples available	0 MGy	5 MGy	10 MGy	15 MGy
SE4445 (baseline) with glass sphere bondline control	1	56	14	14	14	14
SE4445 (with freezing procedure) and glass spheres	1	56	14	14	14	14
Masterbond EP30TC (with glass spheres)	1	54	13	13	14	14
Polytec TC423 (with glass spheres)	1	56	14	14	14	14
StyCast 2850 FT (not re-workable) - with glass spheres	1	56	14	14	14	14
Hysol EA9396, unfilled with glass spheres	1	56	14	14	14	14
Hysol EA9396, graphite filled with glass spheres	1	56	14	14	14	14
Total number of test combinations	**7**	**390**	**97**	**97**	**98**	**98**

Polytec and Stycast were not usable due to high viscosity

Thermal compounds were bonded to Silicon, EA9396 bonded to CFRP (0, 90, 0 laminate as used in the detector)

Sample thickness measured before dicing in 8 positions around wafer

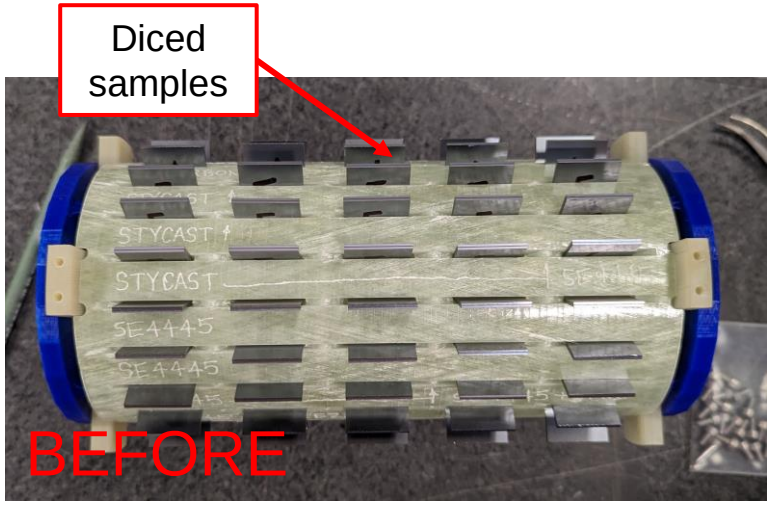


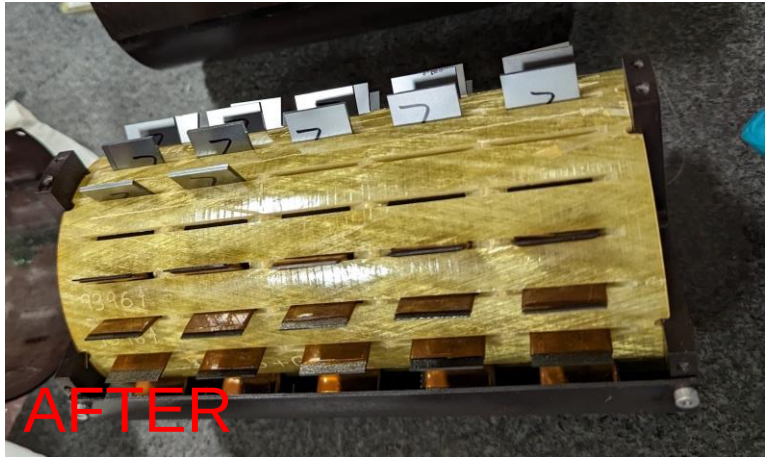
Sample preparation

- Original plan was to use pre-diced 2 x 2 cm Si pieces, but the fixturing during the gluing was very difficult
- Two silicon wafers were bonded together with sample adhesives
  - 104 um borosilicate glass spheres used as bond line control
- This yielded 57, 2cm square samples to be irradiated
- CFRP samples also bonded in large plate and cut into 2x2 sample size with diamond saw

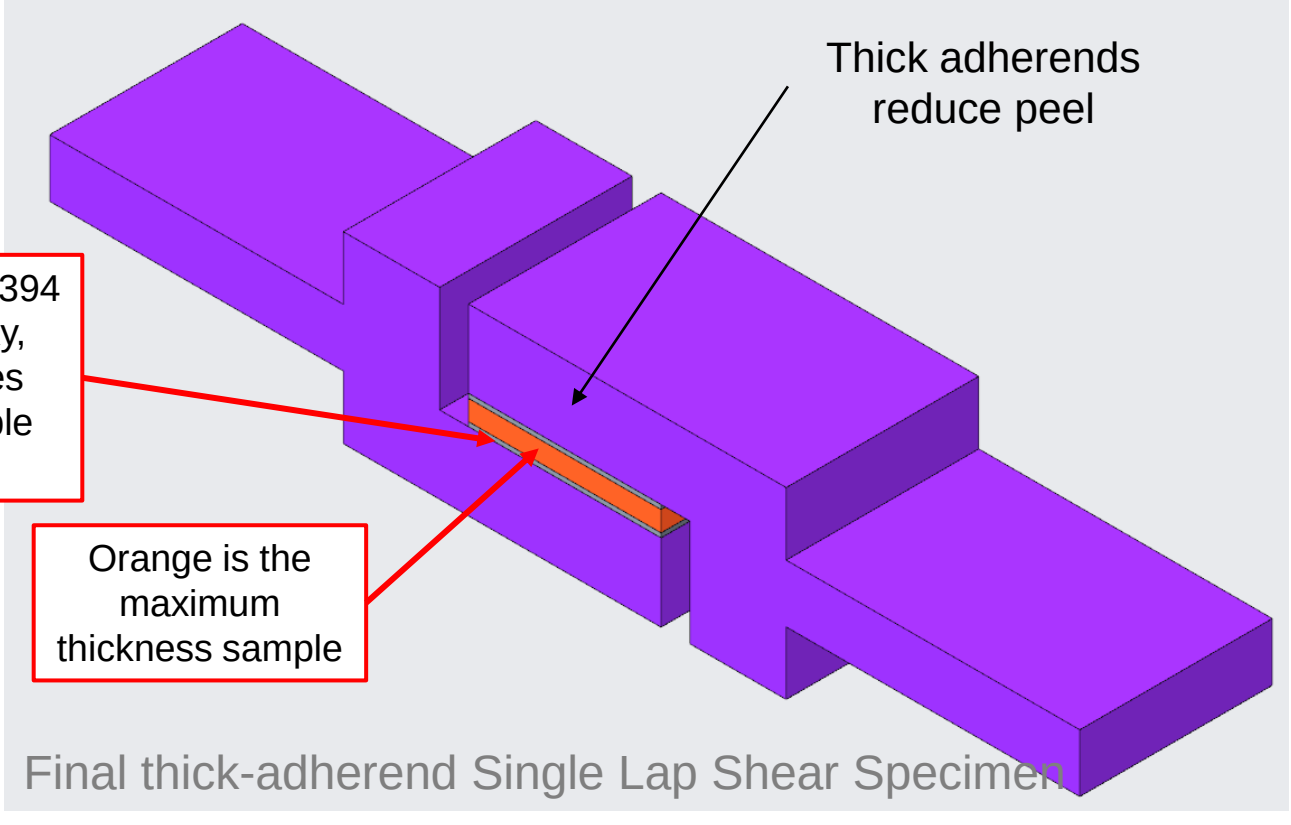
Irradiation Carrier

- Samples are delicate – to protect them and homogenize the does, they were loaded into specially made carriers for shipping and irradiation
- Fully fiberglass for transparency, is made to be compatible with ventilation, and holds up to 100 samples safely
- Carrier AFTER irradiation is at right bottom
- Fiberglass is NOT normally black and yellow… this is radiation damage!



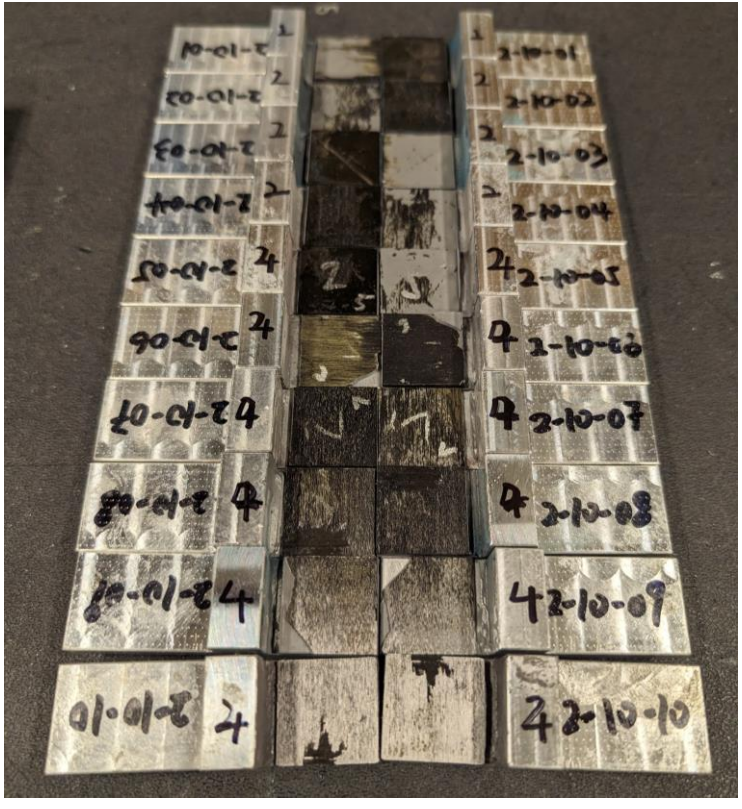


Thick Adherend Single Lap Joint



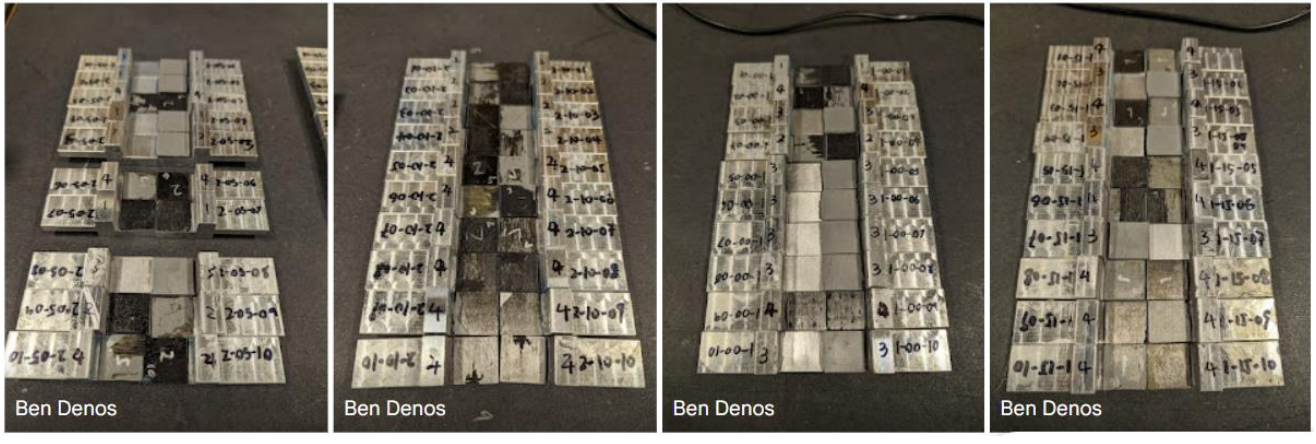
Prepared samples

Initial Results



- Some samples failed at the aluminum to EA9394 interface
  - This is a failed test, but the failure strength exceeds typical structural adhesives
- Some carbon fiber samples had interlaminar shear failures, adhesive strength is stronger than the laminates that are used in our local supports

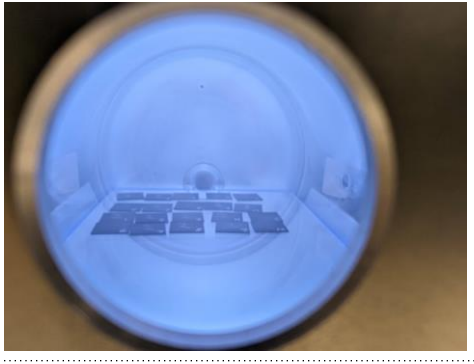
A Sample of the Test Samples

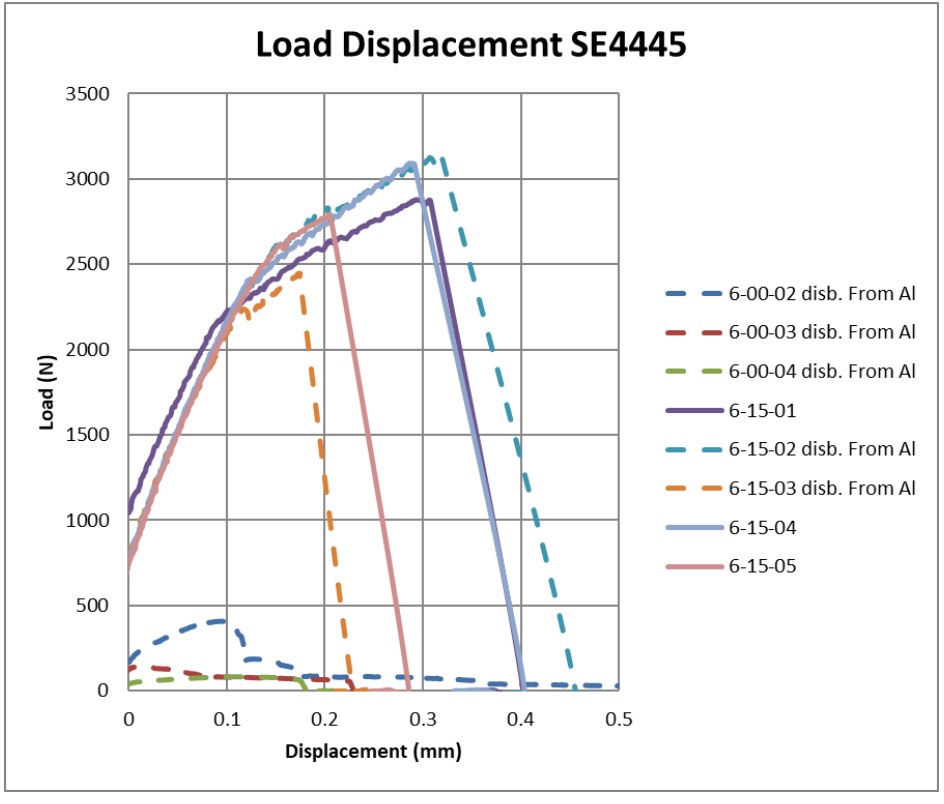


Approximately 40 of the ~120 samples tested

First Results for SE4445

- In first round of testing, All unirradiated bonds failed at the Aluminum to EA9394 interface
- Epoxy used was expired
- Aluminum adherends were only manually cleaned with IPA
- Later, test samples and adherends were plasma cleaned before bonding



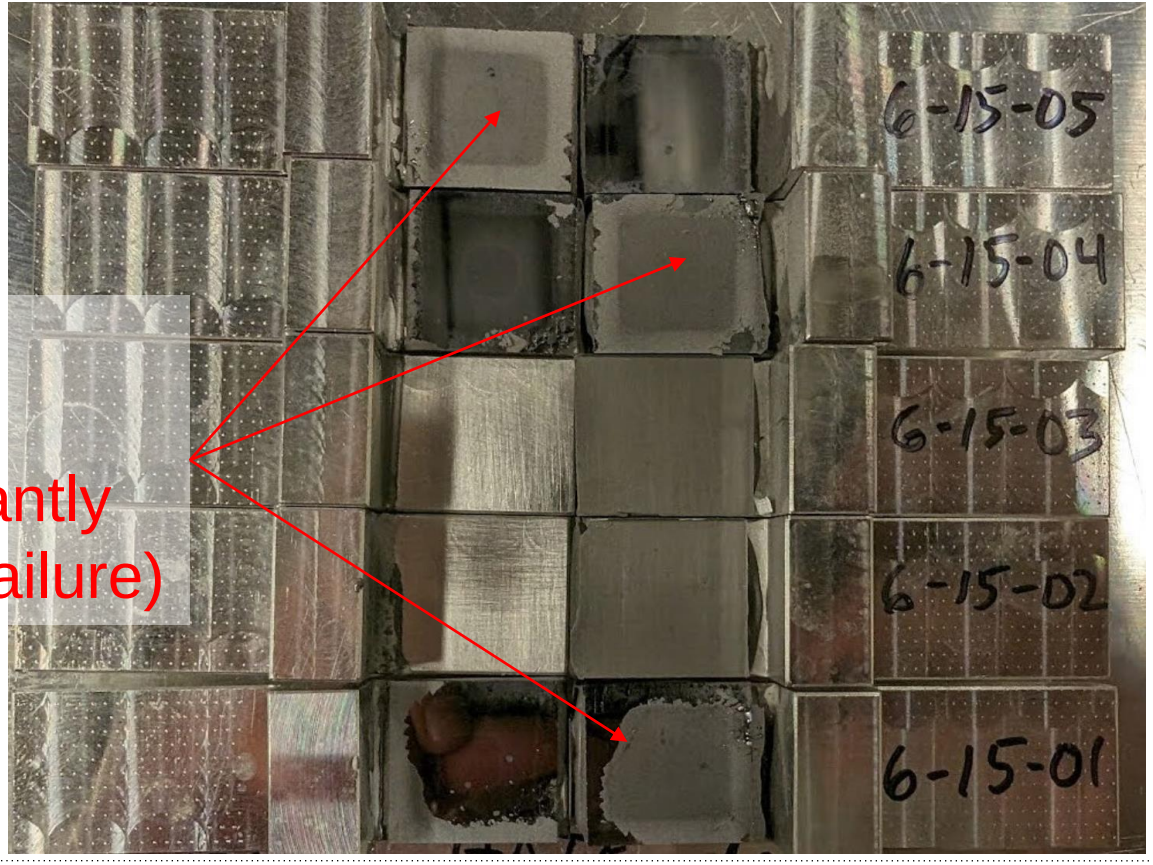


Second set of SE4445 tests

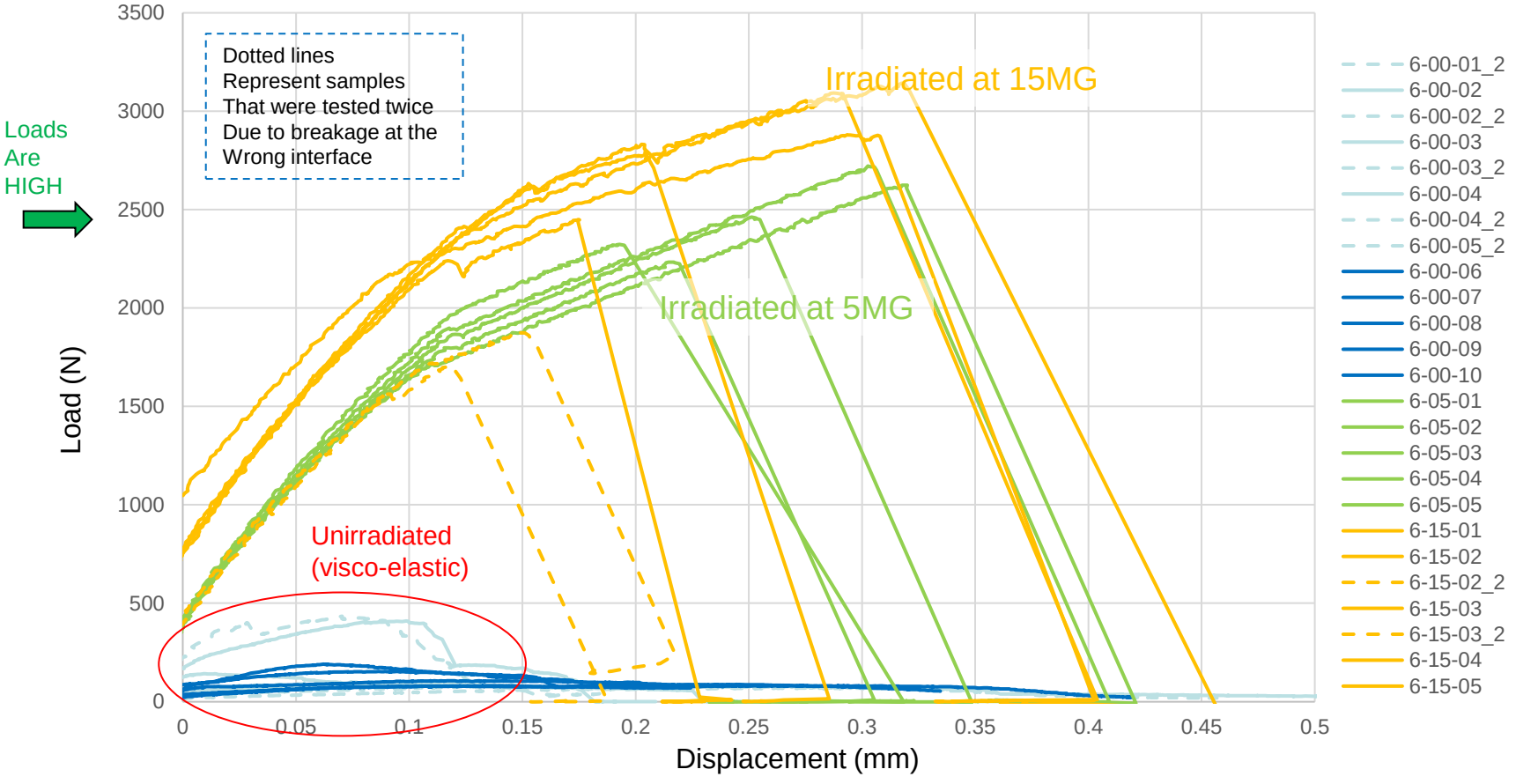
- Since these irradiated samples are very valuable (hard to produce), we decided to proceed cautiously and try to maximize the data produced
- Were able to recover some of the samples that broke at the Aluminum interface and re-bond
- Tested remaining 0 MGy samples
- Re-tested “bad” 15 MGy samples
- Added 5 MGy samples to see trend in strength with increasing irradiation

Failures at the SE4445 Interface

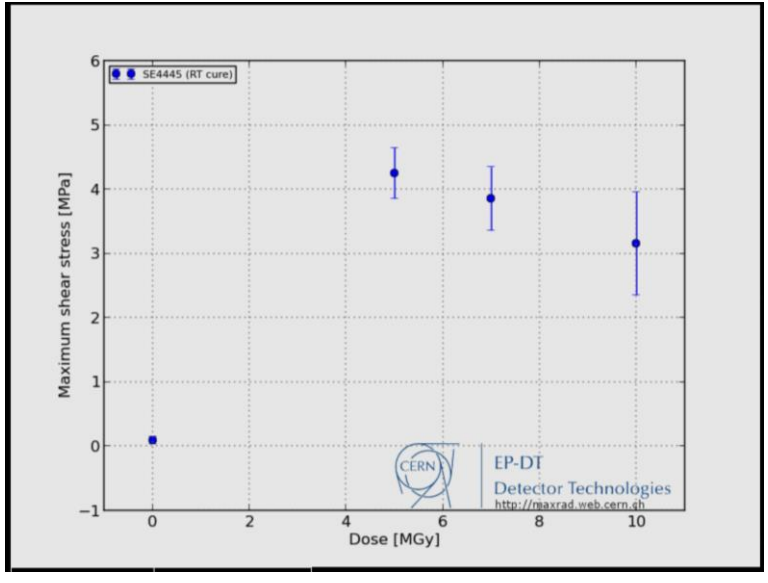
SE4445 Failures (predominantly Adhesive failure)

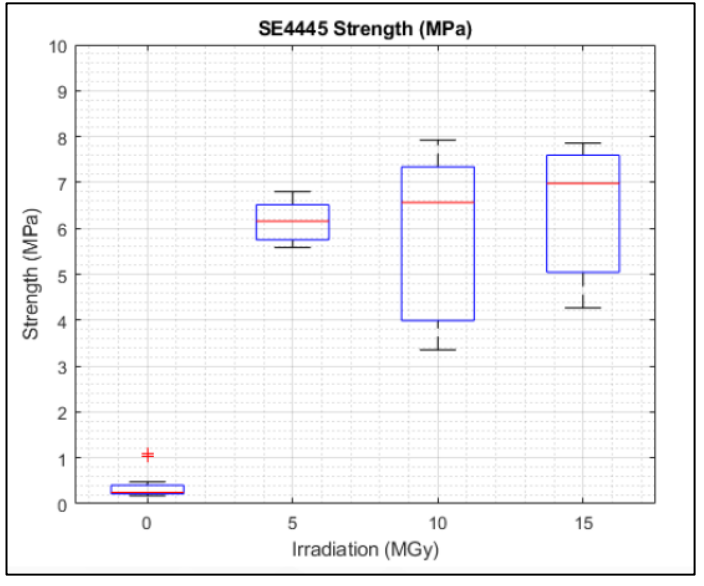


Force-Displacement Curves for SE4445



Comparison to historic data





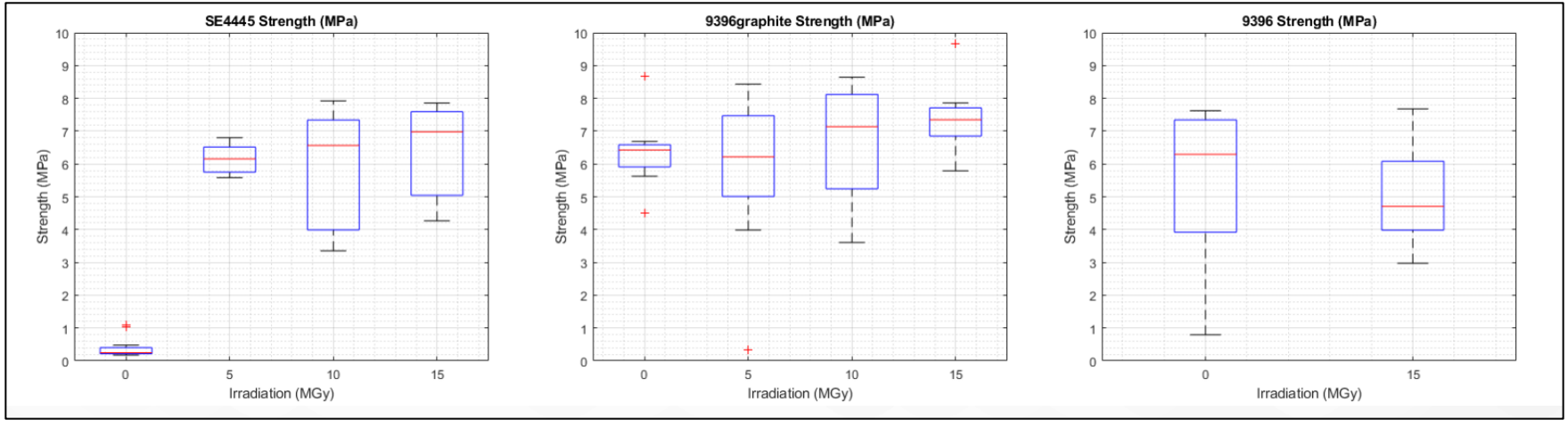
MaxRad tests to 10 MGy, consistent with current results (though we observed somewhat higher strength)

MaxRad ~5 samples at each dose, new data doubles statistics

SE4445 and EA9396 strength results

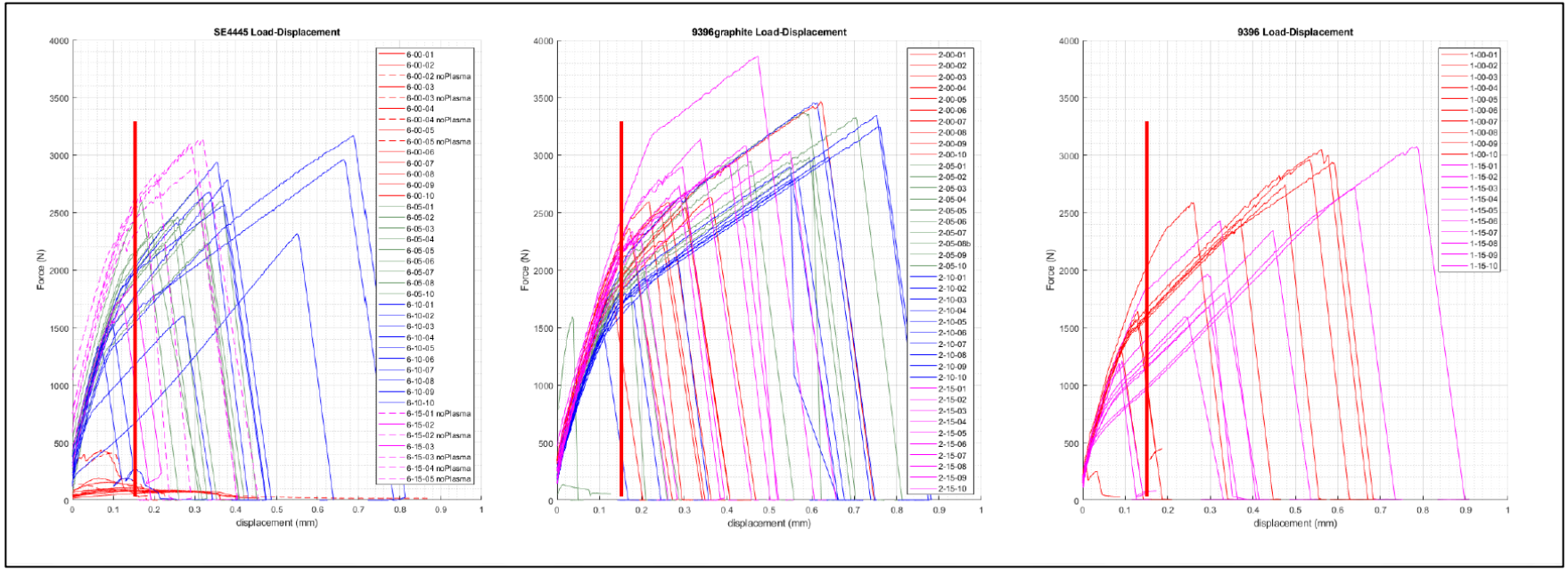
All samples fail around the same stress

This is good for SE4445, but seems low for epoxy



Force-Displacement curves for all adhesives

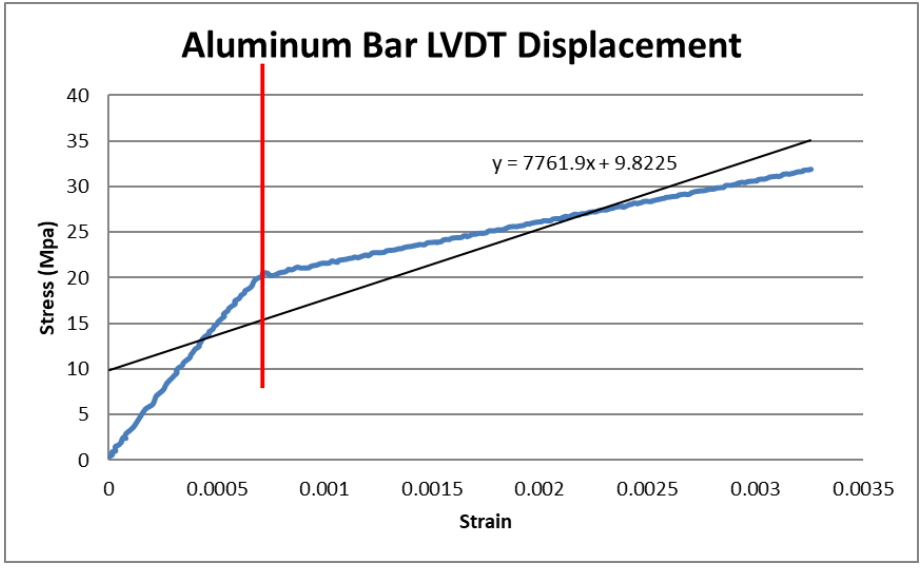
There is an anomalous knee in the data around 0.15 mm of extension

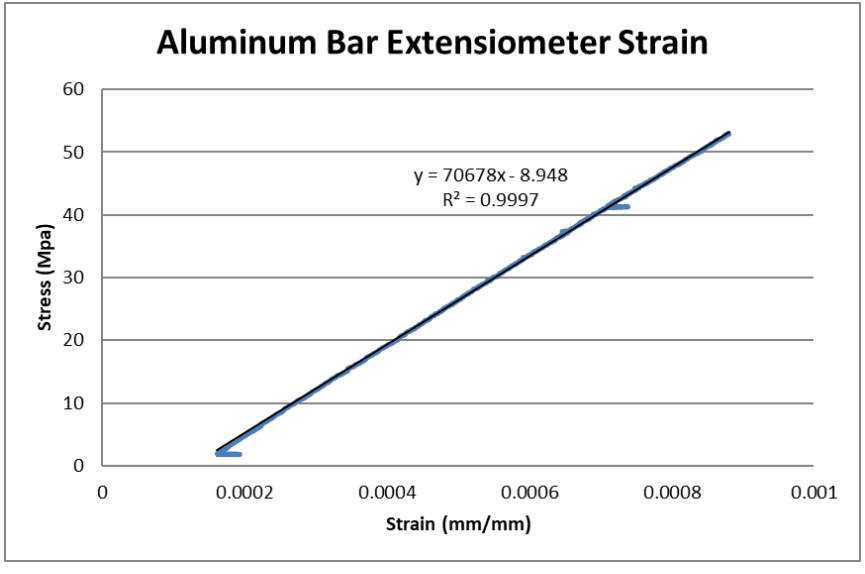


Measuring a solid Aluminum bar

Strain measurements need to be investigated (shows similar knee anomaly)

Strength data seems to be valid (no strange results from load cell)





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Conclusions

- SE4445 strength does not degrade after irradiation, in fact, it acts more like a true adhesive
  - Unirradiated SE4445 is qualitatively a different *type* of material
  - Strength increases by 10x or more under irradiation (not sure where this threshold lies, but it is less than 5 MGy)
- There are no worries that modules will de-bond from local supports throughout the lifetime of ATLAS ITk
- All structural adhesives used in the Inner System are good to beyond our expected dose
- Data is compatible with general trends seen in previous irradiation testing

Limitations

- Even with this promising strength data, there is a problem in the displacement measurements made
  - This problem makes it impossible to calculate shear modulus, which was also a prime interest in doing the study
- In addition, the strength shown with EA9396 is comparable to that shown with SE4445, even though it should be considerably stronger
- All displacement data shows a “knee” or a change in sensitivity at low deflection, which is clearly not a physical aspect of the materials

Further Work

- Investigating with the testing machine company to see what may be the cause of the suspect deflection data
  - Can it be corrected with a calibration curve?
  - Can it be truncated to the region of interest?
- Will produce in the near term an FEA model to check overall compliance and predicted deflections
- Will produce “neat” samples with only adhesive and adherends in order to check adhesive-only strengths and deflections
  - Will attempt to rapidly send some of these for additional irradiation
  - These will be instrumented with additional extensiometer during testing
- Will test (or fabricate and test) additional Carbon/EA9396 samples (no irradiation) in order to test on a second machine at an outside institute