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Round Robin Tests of Electron Irradiated Polymers via Pulsed Electroacoustic Measurements

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Chateaubriand Fellowship Program

Science, Technology, Engineering, Math & Health

MATERIALS PHYSICS GROUP

Laplace

UtahStateUniversity

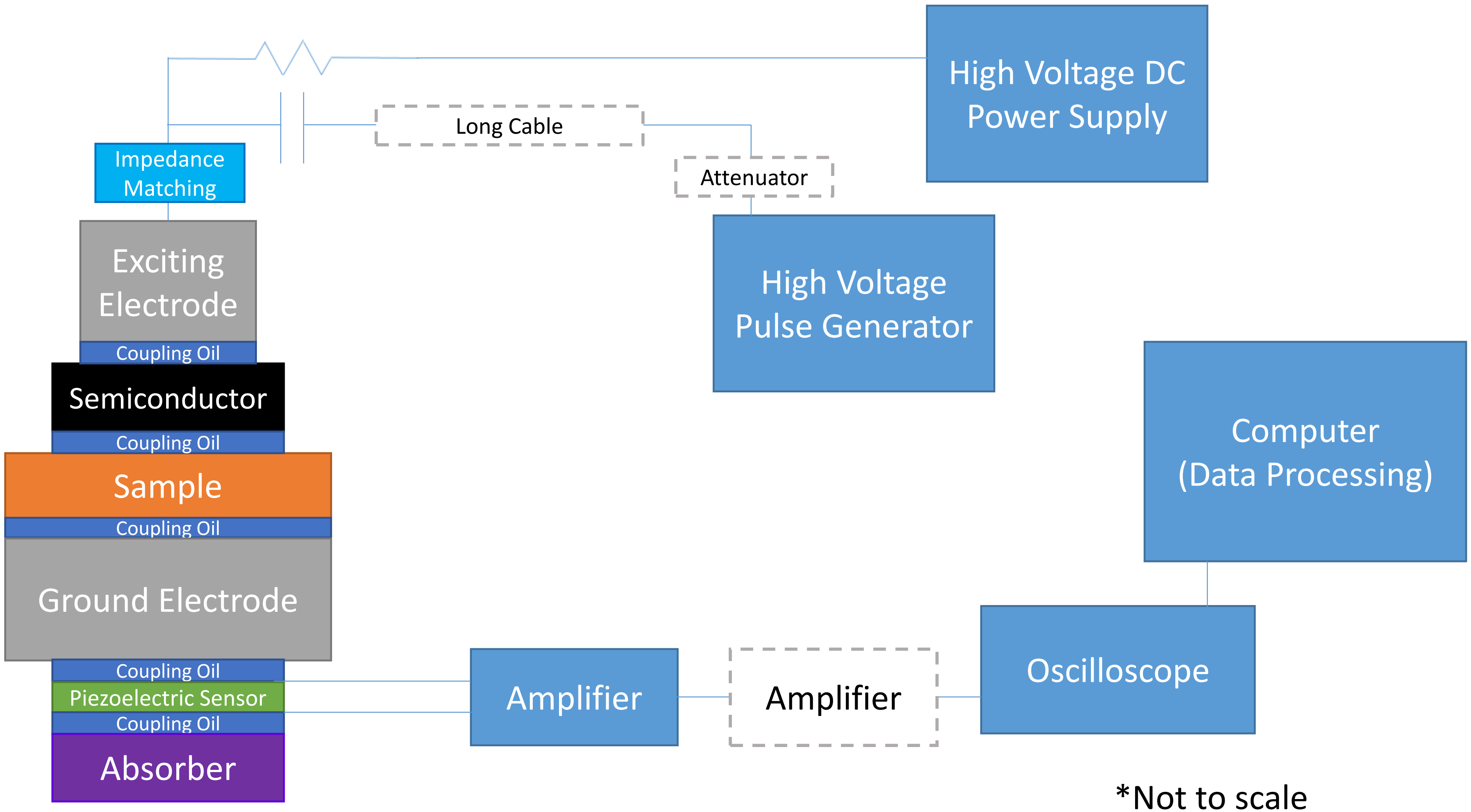
Overview

- Motivation
  - Validate Comparison of PEA Results
- Experiment
  - PEA System Comparison
  - Sample and Irradiation Details
- Results
  - Compare PEA Measurements
- Conclusions

Motivation – Validate Comparison of PEA

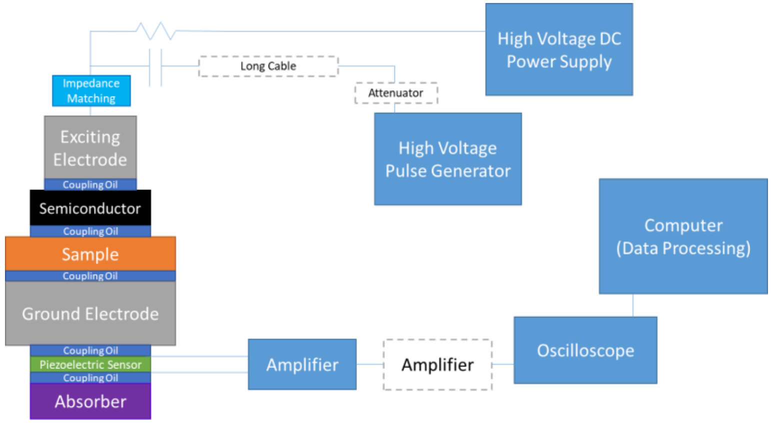
- PEA is a well established method for measuring internal charge distributions in dielectric materials
- There is a standard method of calibration
  - Apply known amount of charge and measure with PEA
  - Use as reference for calibration
- Calibration is a tough issue
  - Difficulties arise when calibrating samples with embedded charge or when using open PEA
  - Potential issues can result from applied pulsed voltage, electrode material, coupling media, semi-conducting layer, etc.

Overview of PEA System – Key Components



*Not to scale

Comparing PEA Systems



Utah State University PEA System

- Ambient, parallel plate capacitor
- ~5 ns, ~300 V exciting pulse
  - 1 kV pulse through 8 dB attenuator and short cable
- 50 Ω impedance match
- PVC semiconductor film
- Al electrodes
- 9 µm PVDF piezoelectric sensor
- 40 dB gain (1 amplifier)
- Light machine oil (coupling media)
- 5000 traces averaged/measurement
- Data is filtered with modified Gaussian
- Reference obtained with charged sample

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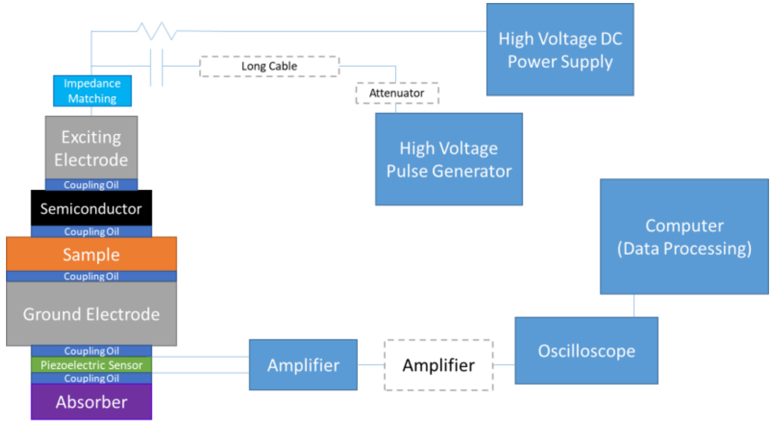

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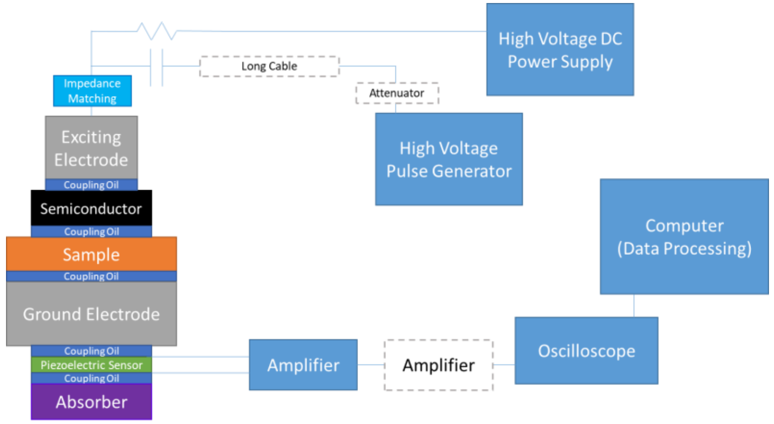

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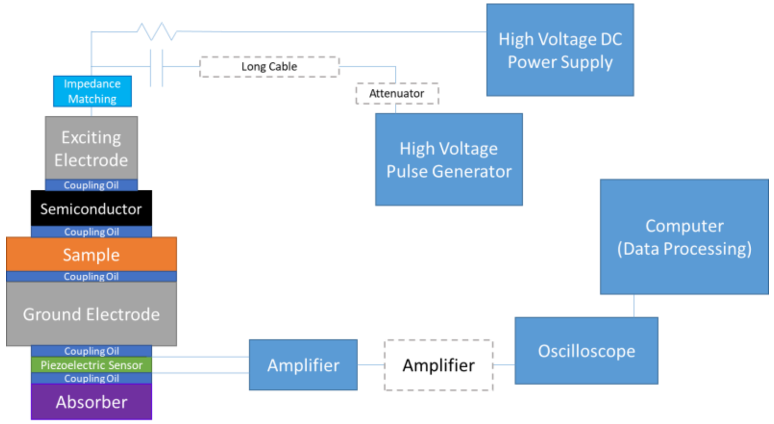

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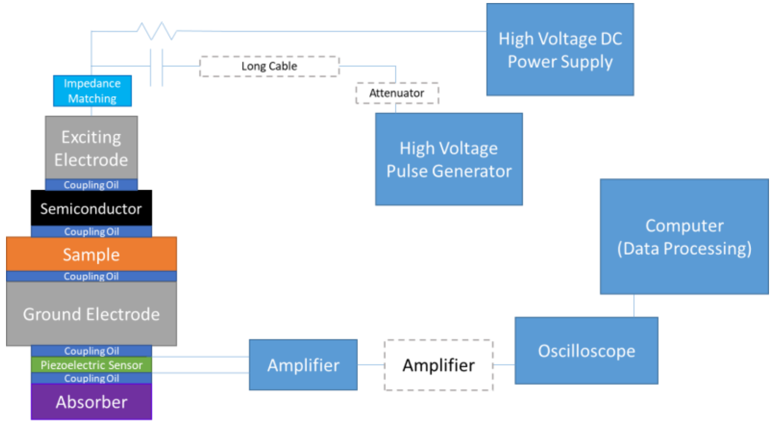

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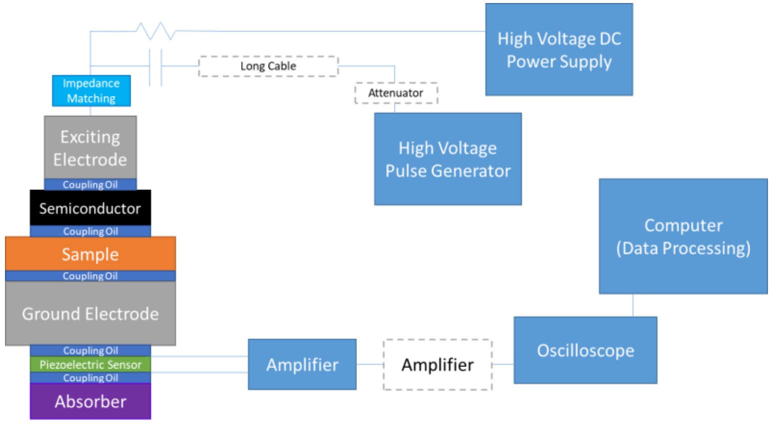

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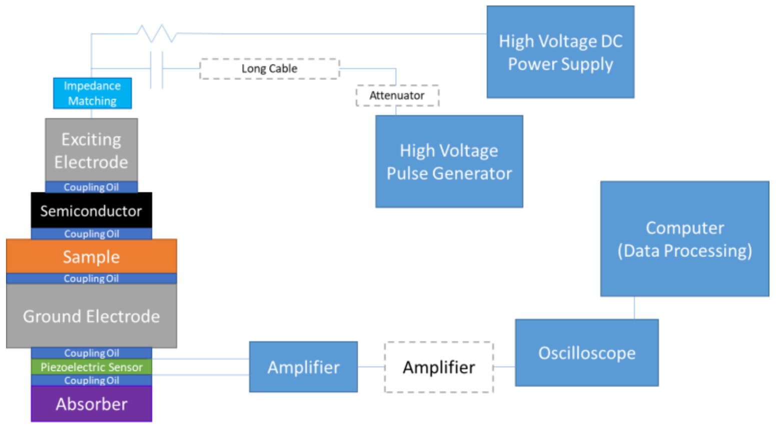

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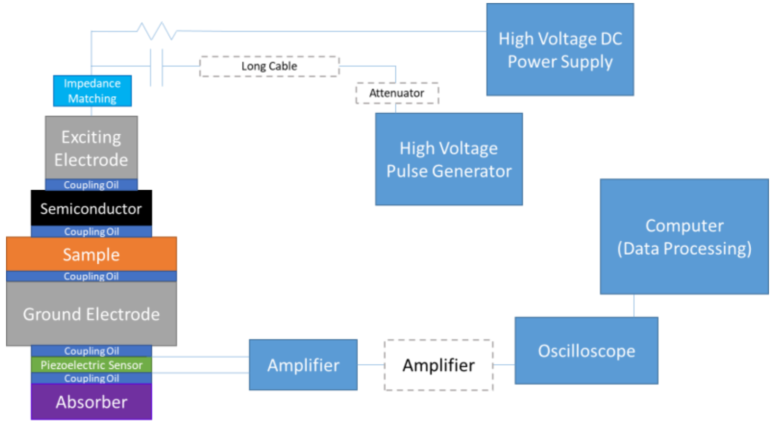

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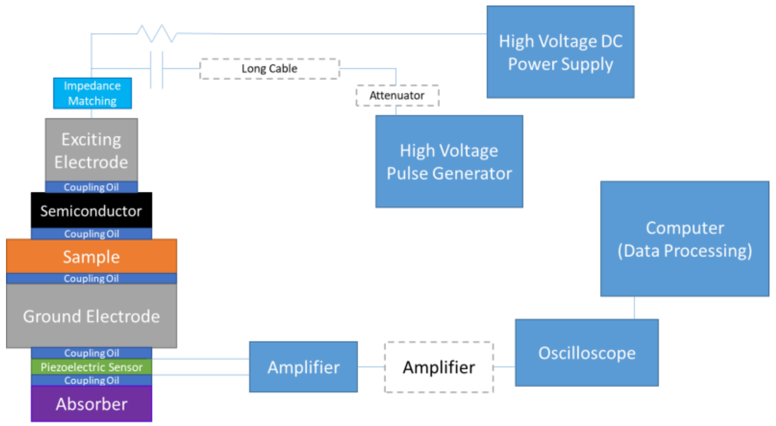

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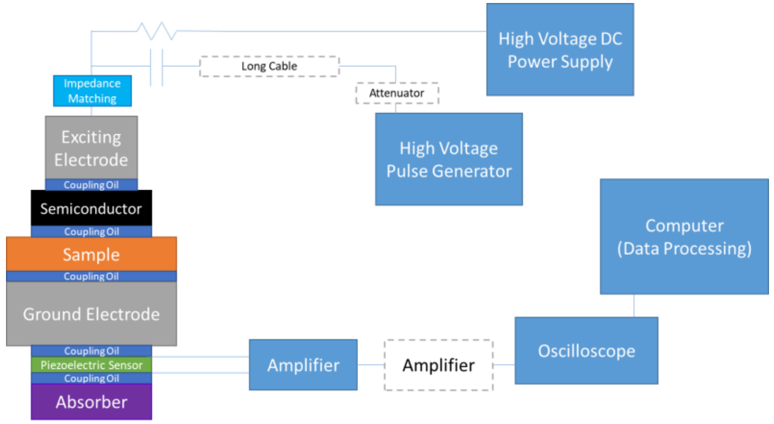

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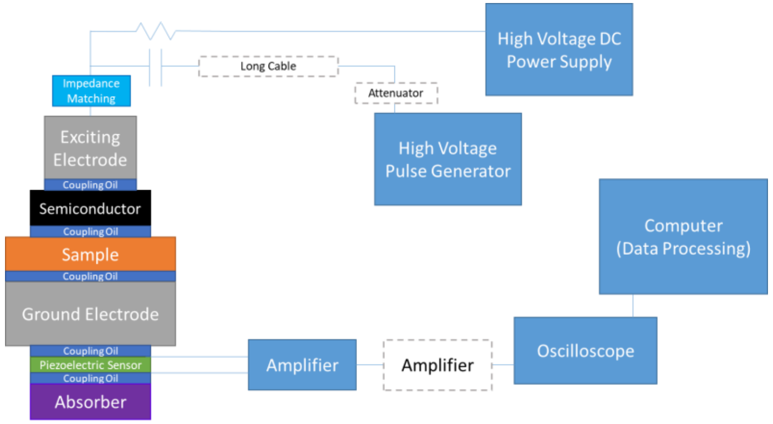

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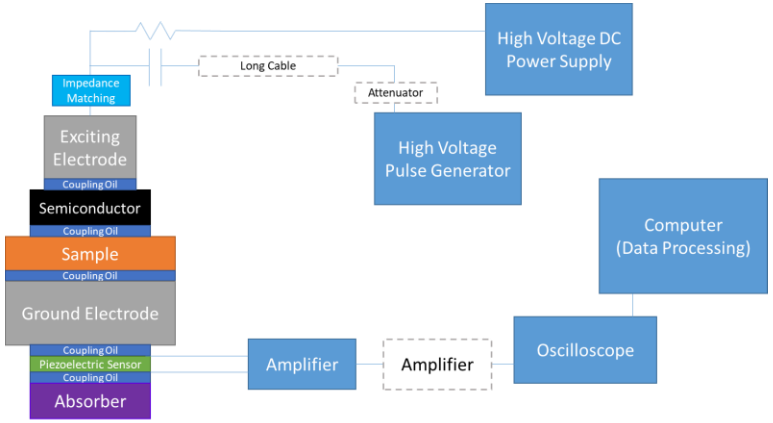

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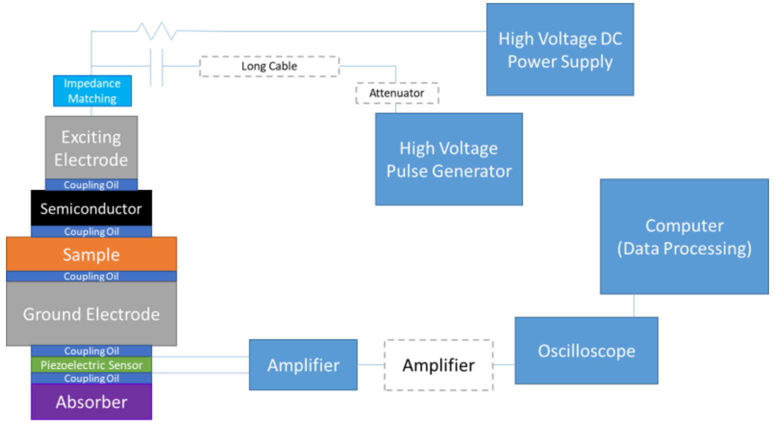

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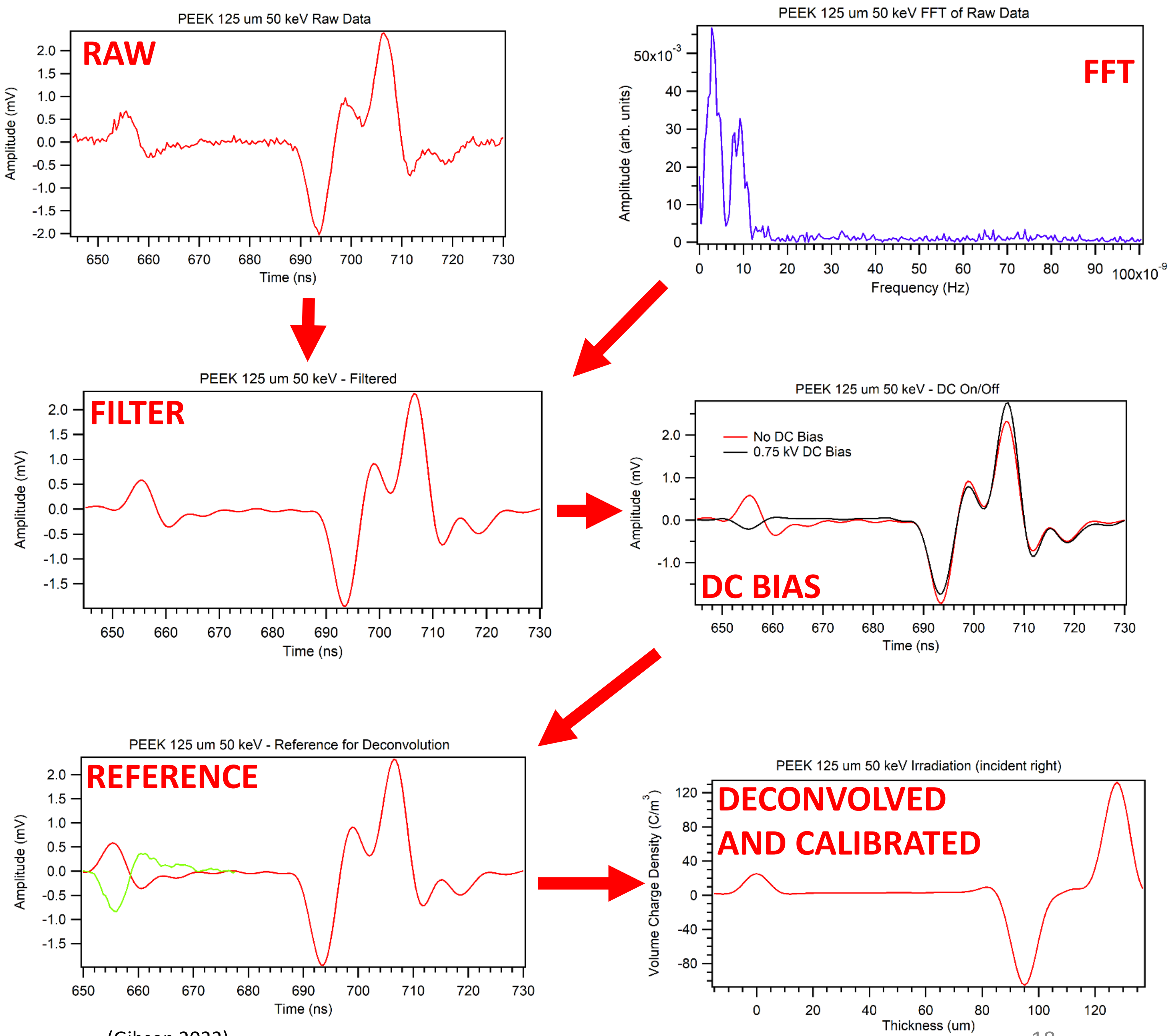
Signal Processing

Processing Steps:

- Average multiple measurements and compute statistics (not shown)
  - The rest of the processing is done on the averaged measurement
- Compute FFT to determine filter parameters
- Modified Gaussian filter used on data
- Take difference of DC on – DC off to obtain reference wave (Chen 2006)
- Use system response to perform deconvolution

Calibration

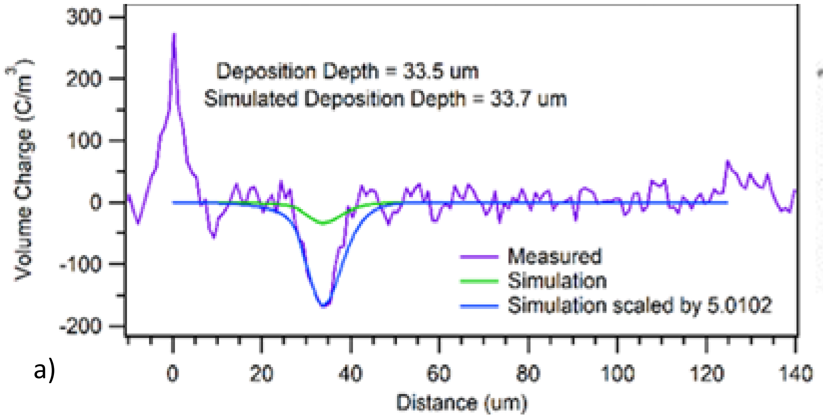
- Multiply by calibration factor
  - $Calibration\ Factor = \frac{\epsilon_r \epsilon_0 V_{DC}}{d \int V_{RefSignal} dx}$
- Calibrate x-axis to distance using the speed of sound calculated from the measured thickness and peak-to-peak time difference of the two interfaces



(Gibson 2022)

Signal Processing – Filtering Effects

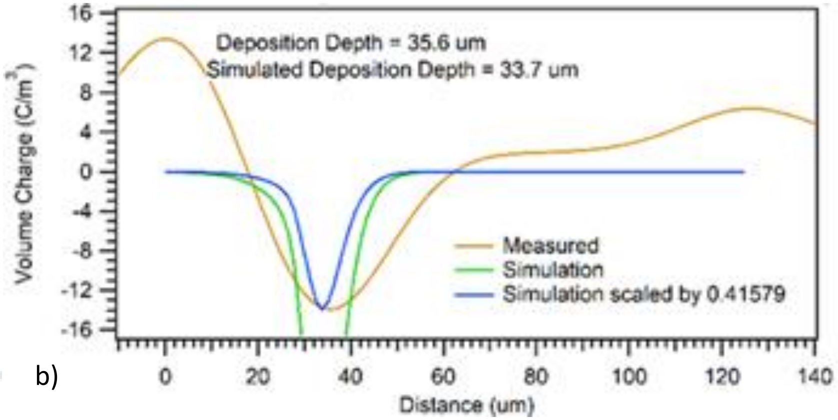
No Filter



a)

- Higher spatial resolution
- Very noisy
- Overestimate charge density

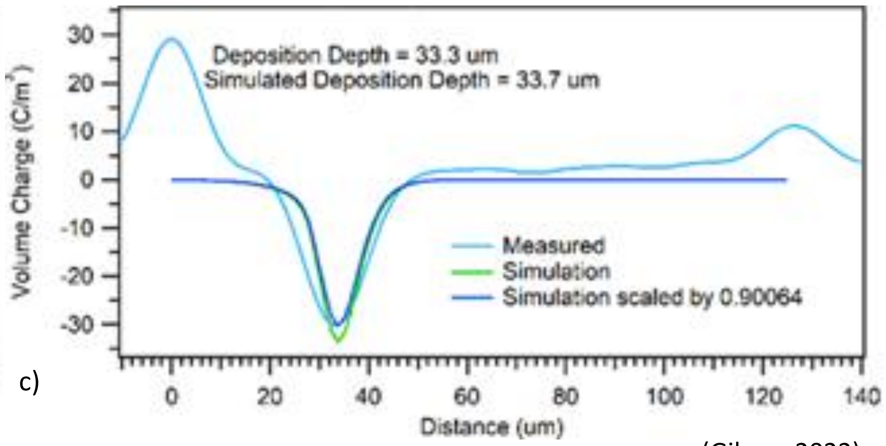
Too Much Filtering



b)

- Very low spatial resolution
- Underestimate charge density
- Low noise

Approximately Optimized Filtering



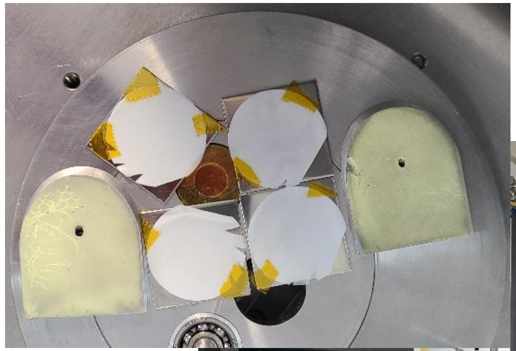
c)

(Gibson 2022)

- Optimal charge density
- Low noise
- Optimal spatial resolution

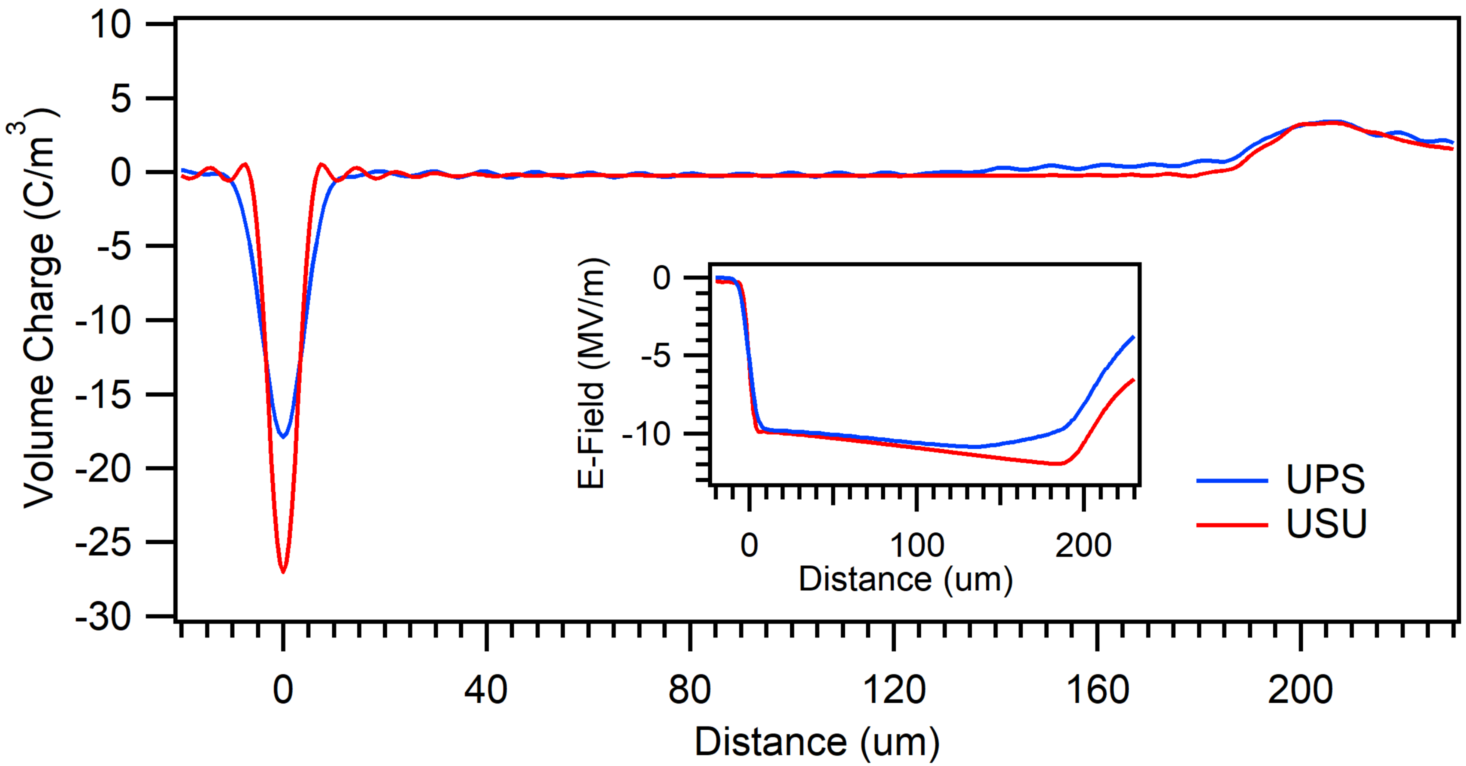
Experimental Details – The Experiment

- Experimental Overview:
  - Measure pristine sample with DC bias only
  - Measure samples with embedded charge
  - Compare PEA measurements in ambient conditions at Utah State University (Logan, UT) and Université Paul Sabatier (Toulouse, France)
- Sample details
  - Samples must be very resistive so charge will not migrate during transportation
  - Materials used are polytetrafluoroethylene (PTFE) and polyether-etherketone (PEEK) of 200-250 µm thickness
- Irradiation chamber details
  - Samples irradiated with 50, 60, or 70 keV electrons with a flux of ~1 nA/cm^2 for 10 min
  - Irradiation completed at UPS with MATSPACE chamber



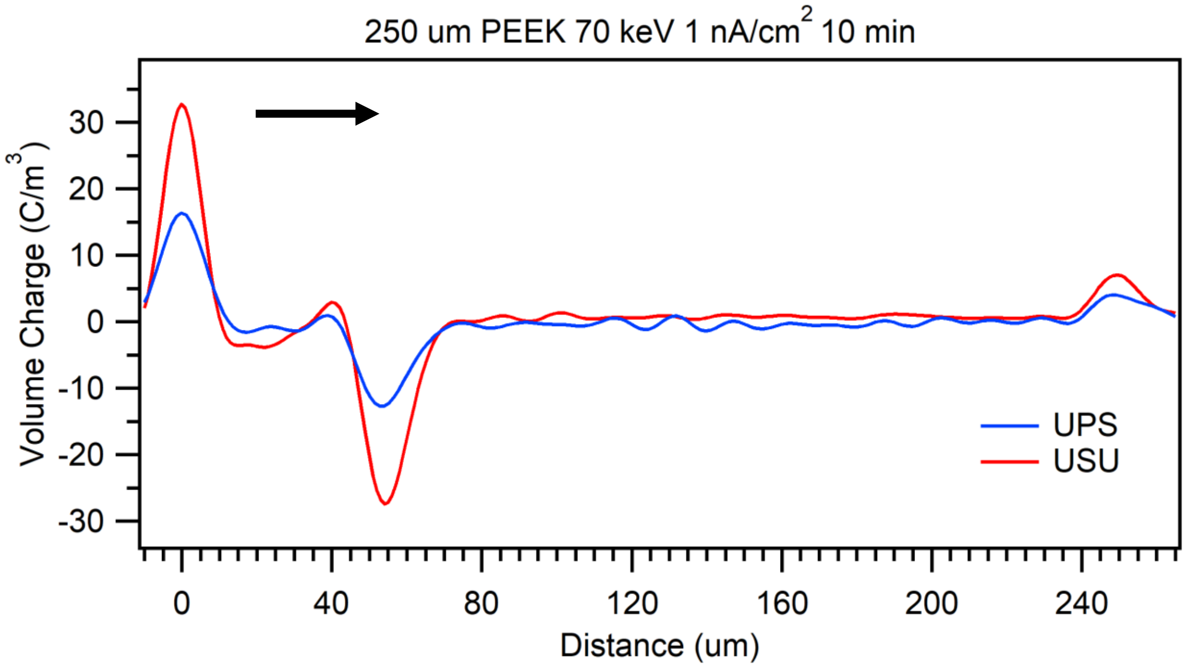
Comparing PEA – PTFE DC Bias

- Basically the same results
- The charge magnitude agrees *by definition*
- Slight difference in analysis/processing

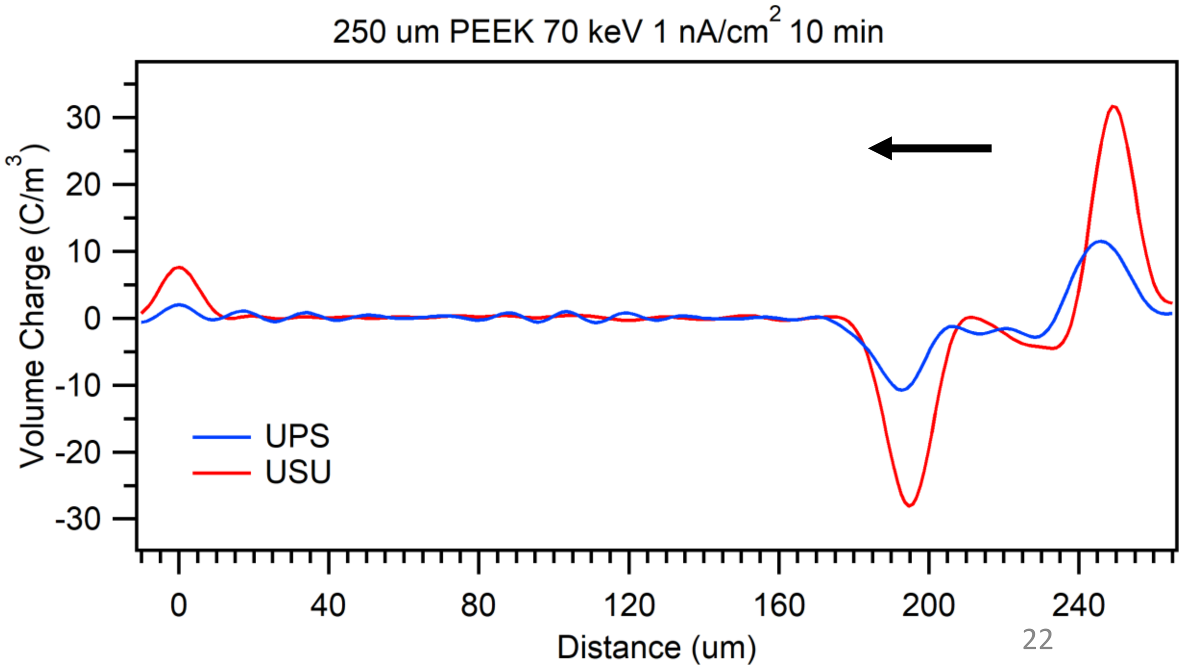


PEA measurements of 200 μm thick PTFE with 2 kV DC bias. Inset depicts internal electric field calculated from the measured charge.

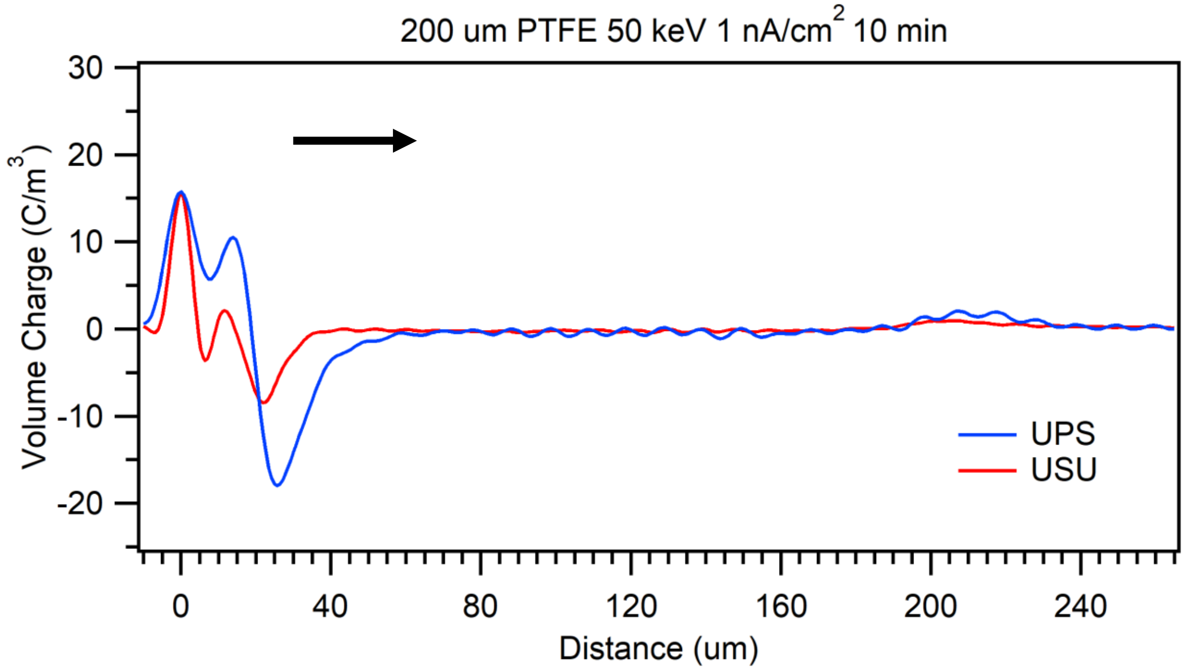
Comparing PEA – 70 keV PEEK



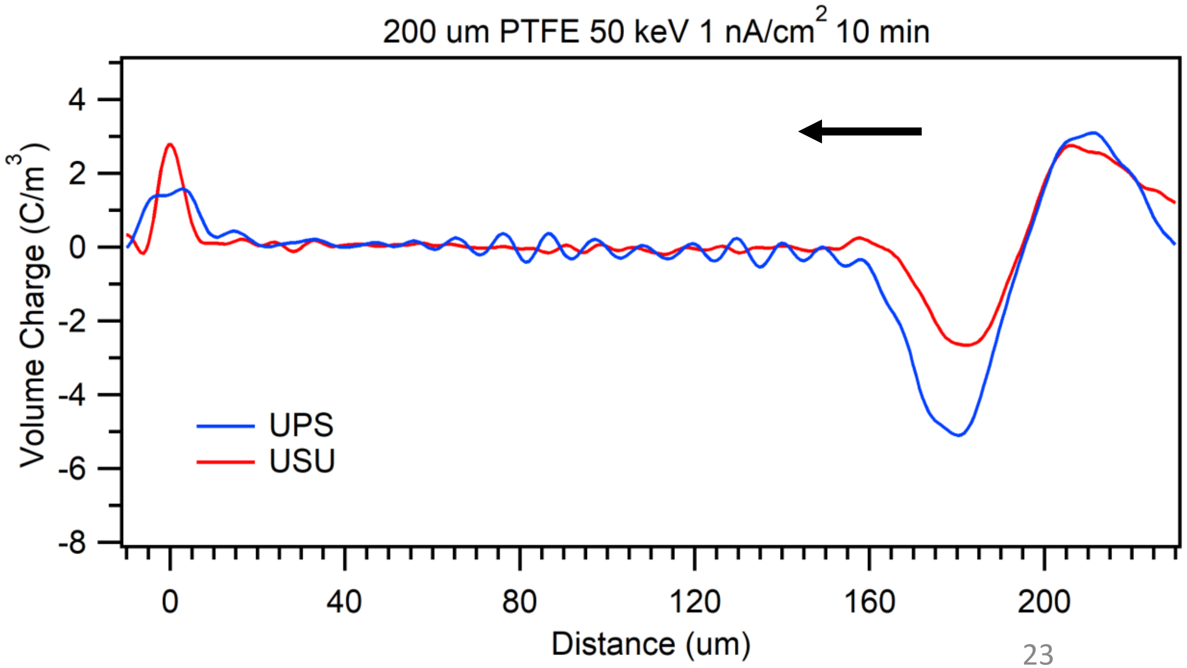
- Features are similar
- Charge magnitude greater for USU data



Comparing PEA – 50 keV PTFE

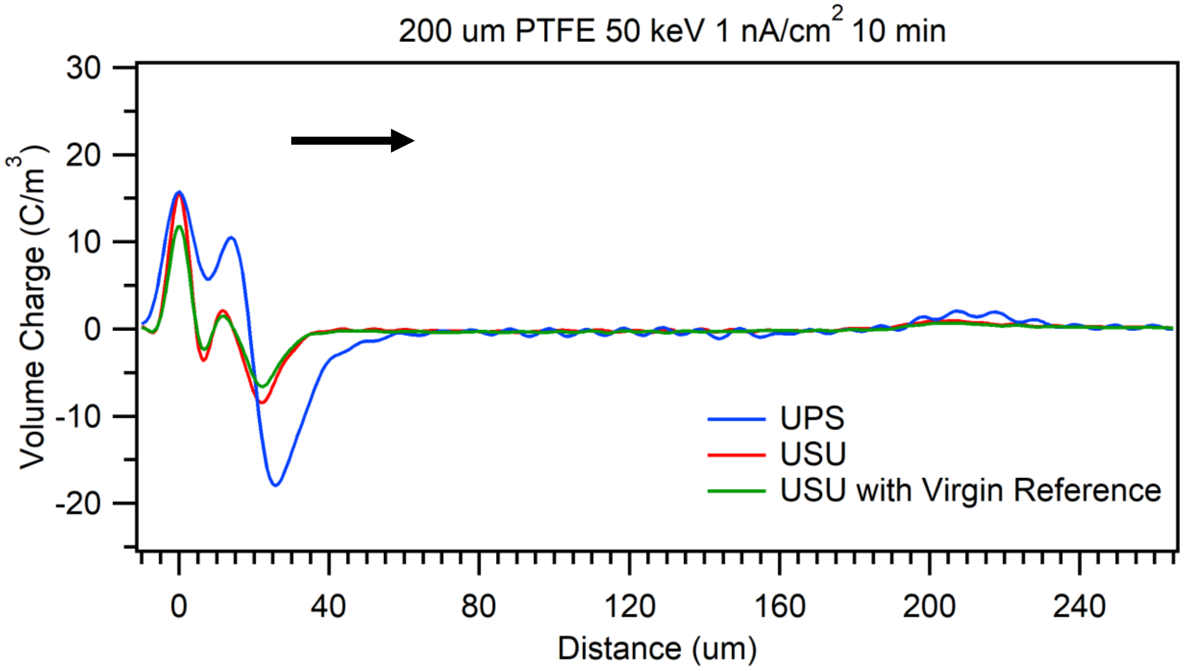


- Features are similar
- Charge magnitude greater for UPS data

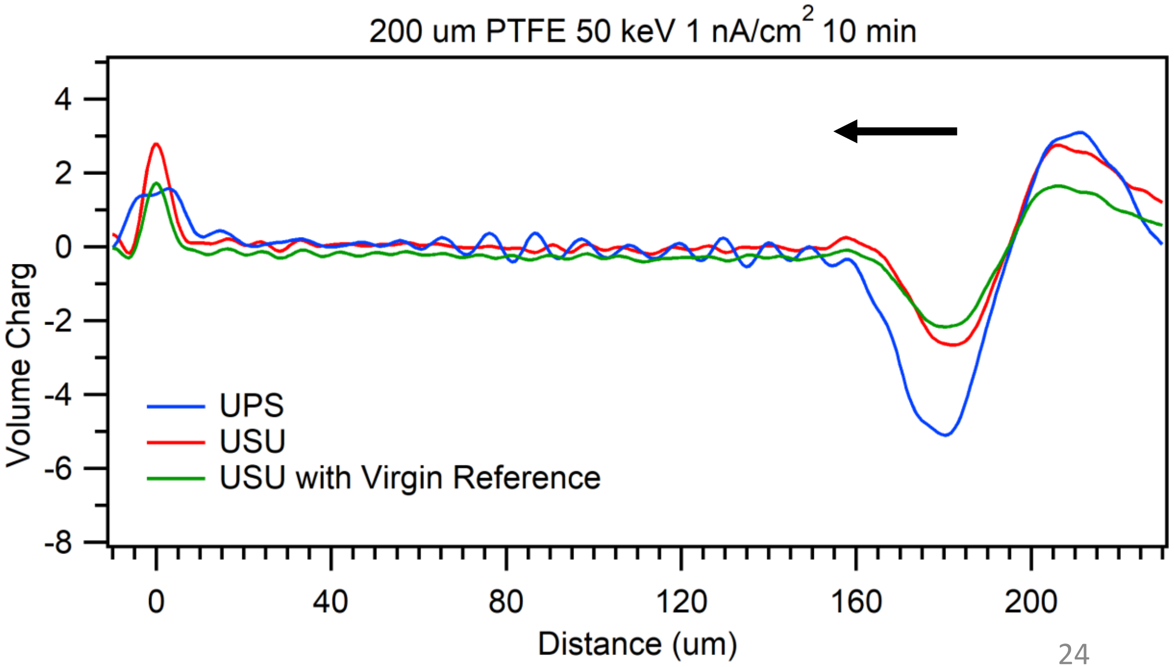


- Attenuation/dispersion is apparent in "incident right" plot for PTFE

Comparing PEA – 50 keV PTFE – Classic Calibration



- The USU data was also calibrated with “classic” method (pristine sample)



- Classic calibration only changes charge magnitude slightly
- Changes in the “wrong” direction (less agreement)

Comparing PEA – Other Results

- Similar results were obtained for the rest of the measurements
  - PEEK irradiated with 60 keV electrons
  - PTFE irradiated with 60 keV and 70 keV electrons

- It was attempted to calibrate USU data with pristine sample reference but this provided a minimal change, and in the “wrong” direction

- There is no obvious answer to the discrepancies in the charge magnitude of the measurements

Conclusions

- The **shape** of measurements are in **agreement**
- The **distance scale** is in **agreement**
- The **charge magnitude** is **not in agreement** for measurements with embedded charge distributions

- Possible issues:
  - Electrode materials, coupling media, data analysis/processing, pulse effects, temperature, clamping pressure, humidity, errors (applied voltage, etc.)
  - Calibration used is from *surface charge,* perhaps allowing surface effects to contribute to error

- Ideal calibration for comparing absolute charge magnitude:
  - Known charge magnitude and distribution in bulk of material used for calibration
    - This is much more difficult to achieve
    - Perhaps an irradiated sample of PEEK or PTFE with magnitude of charge verified by surface potential measurement

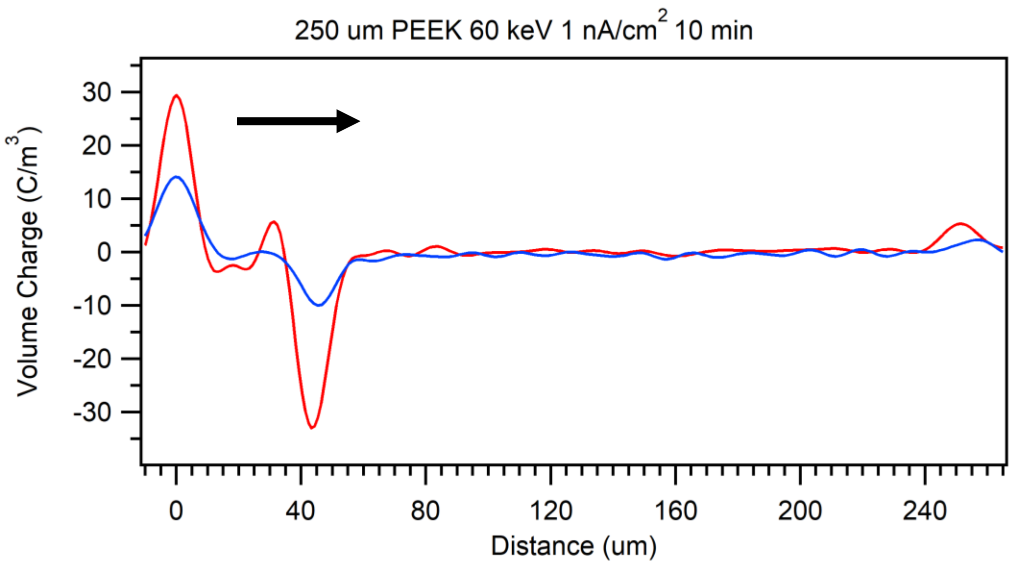
References

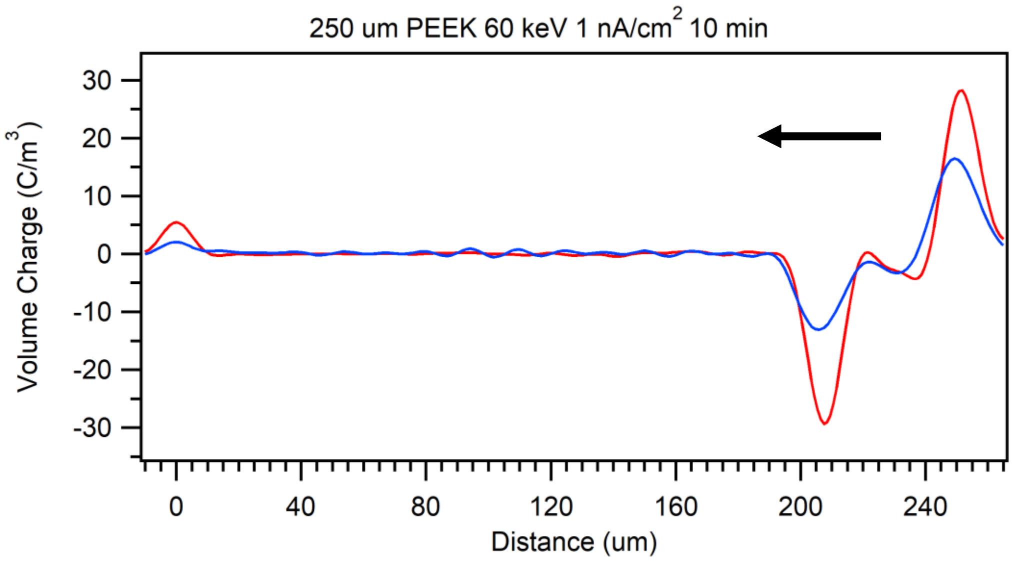
- Chen, G., Y. L. Chong, and M. Fu. "Calibration of the pulsed electroacoustic technique in the presence of trapped charge." *Measurement Science and Technology* 17.7 (2006): 1974.
- Gibson, Z., Dennison, J.R., Beecken, B., and Hoffmann, R., “Comparison of Pulsed Electroacoustic Measurements and AF-NUMIT3 Modeling of Polymers Irradiated with Monoenergetic Electrons,” *Journal of Spacecraft and Rockets* (2022) (under review)

Questions?

Backup slides

Other Results – PEEK





Other Results – PTFE