Precise Determination of Charge Distributions in Electron Irradiated Polymers Via Pulsed Electroacoustic Measurements With Applications to Spacecraft Charging

Zachary Gibson
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PRECISE DETERMINATION OF CHARGE DISTRIBUTIONS IN ELECTRON IRRADIATED POLYMERS VIA PULSED ELECTROACOUSTIC MEASUREMENTS WITH APPLICATIONS TO SPACECRAFT CHARGING

by

Zachary Gibson

A dissertation submitted in partial fulfillment of the requirements for the degree of DOCTOR OF PHILOSOPHY in Physics

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ABSTRACT

PRECISE DETERMINATION OF CHARGE DISTRIBUTIONS IN ELECTRON IRRADIATED POLYMERS VIA PULSED ELECTROACOUSTIC MEASUREMENTS WITH APPLICATIONS TO SPACECRAFT CHARGING

by

Zachary Gibson, Doctor of Philosophy
Utah State University, 2023

Major Professor: J.R. Dennison, Ph.D.
Department: Physics

Understanding charge accumulation and migration in dielectrics is paramount for myriad of applications from high voltage DC cable transmission to spacecraft charging. The leading cause of space environment induced anomalies of spacecraft are due to spacecraft charging. The most critical charging occurs due to electrons in the energy range of 10-50 keV, translating to electron penetration depths of 1’s to 10’s of microns. Arguably, the best method for measuring internal charge distributions is the pulsed electroacoustic (PEA) method. The PEA method allows for non-destructive determination of the spatial distribution of embedded charge in dielectric materials. PEA instrumentation, test methods, and analysis were extensively characterized and improved. The PEA method was tested as to its relevance to spacecraft charging applications and the applicability was extended by approaching spatial resolution from a new perspective and implementing a novel method for measuring shallowly deposited charge distributions in highly insulating materials. The PEA method is shown to be relevant and useful for understanding charge dynamics in the context of spacecraft charging.

(227 pages)
Understanding how charge builds up and moves around in materials that are highly insulating, such as dielectrics, is important for many applications from power transmission to spacecraft charging. The leading cause of issues in spacecraft due to interactions with the space environment is spacecraft charging. That is, the accumulation of charge on insulating materials leads to arcing and sparking aboard the spacecraft. The most critical charging occurs due to electrons in a particular energy range of 10-50 keV. Electrons with these energies can travel 1’s to 10’s of microns into relevant materials. To measure where the charge is embedded and how it moves, the pulsed electroacoustic (PEA) method can be used. The PEA method allows for non-destructive measurements of embedded charge distributions in these dielectric materials. PEA instrumentation, test methods, and analysis were extensively characterized and improved. In this work, the PEA method was tested as to its relevance to spacecraft charging applications and the applicability was extended with a novel method for measuring shallowly deposited charge distributions in highly insulating materials. The PEA method is shown to be relevant and useful for understanding how charge builds up and moves inside dielectric materials in the context of spacecraft charging.
ACKNOWLEDGMENTS

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Zachary Gibson
# CONTENTS

<table>
<thead>
<tr>
<th>Section</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>ABSTRACT</td>
<td>iii</td>
</tr>
<tr>
<td>PUBLIC ABSTRACT</td>
<td>iv</td>
</tr>
<tr>
<td>ACKNOWLEDGMENTS</td>
<td>v</td>
</tr>
<tr>
<td>LIST OF TABLES</td>
<td>ix</td>
</tr>
<tr>
<td>LIST OF FIGURES</td>
<td>x</td>
</tr>
<tr>
<td>NOMENCLATURE</td>
<td>xiv</td>
</tr>
<tr>
<td>LIST OF ABBREVIATIONS</td>
<td>xvi</td>
</tr>
<tr>
<td>1 INTRODUCTION</td>
<td>1</td>
</tr>
<tr>
<td>1.1 Spacecraft Charging</td>
<td>3</td>
</tr>
<tr>
<td>1.1.1 Typical Near-Earth Orbit Electron Environments</td>
<td>4</td>
</tr>
<tr>
<td>1.1.2 Typical Spacecraft Charging Conditions</td>
<td>4</td>
</tr>
<tr>
<td>1.1.3 Length Scales for Spacecraft Charging</td>
<td>5</td>
</tr>
<tr>
<td>1.2 PEA History and Other Measurement Methods</td>
<td>6</td>
</tr>
<tr>
<td>2 THEORY AND MODELING</td>
<td>9</td>
</tr>
<tr>
<td>2.1 Electron Transport in Crystalline Materials</td>
<td>9</td>
</tr>
<tr>
<td>2.2 Electron Transport in Amorphous Materials</td>
<td>10</td>
</tr>
<tr>
<td>2.2.1 Localization</td>
<td>11</td>
</tr>
<tr>
<td>2.2.2 Conduction Mechanisms</td>
<td>12</td>
</tr>
<tr>
<td>2.3 Modeling Charge Transport with AF-NUMIT3</td>
<td>16</td>
</tr>
<tr>
<td>3 EXPERIMENTAL METHODS</td>
<td>21</td>
</tr>
<tr>
<td>3.1 Pulsed Electroacoustic Method</td>
<td>21</td>
</tr>
<tr>
<td>3.1.1 Principles of PEA Method</td>
<td>21</td>
</tr>
<tr>
<td>3.1.2 USU PEA System</td>
<td>24</td>
</tr>
<tr>
<td>3.1.3 Spatial Resolution of PEA Measurements</td>
<td>30</td>
</tr>
<tr>
<td>3.2 Samples</td>
<td>33</td>
</tr>
<tr>
<td>3.2.1 Limitations</td>
<td>33</td>
</tr>
<tr>
<td>3.2.2 Preparation</td>
<td>34</td>
</tr>
<tr>
<td>3.2.3 Sample Characterization</td>
<td>35</td>
</tr>
<tr>
<td>3.3 Charge Deposition</td>
<td>35</td>
</tr>
</tbody>
</table>
### LIST OF TABLES

<table>
<thead>
<tr>
<th>Table</th>
<th>Description</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>5.1</td>
<td>Uncertainties of peak positions</td>
<td>52</td>
</tr>
<tr>
<td>5.2</td>
<td>Comparison of PEA systems</td>
<td>64</td>
</tr>
<tr>
<td>6.1</td>
<td>Sample IDs and Thicknesses</td>
<td>77</td>
</tr>
<tr>
<td>6.2</td>
<td>Properties of materials tested</td>
<td>78</td>
</tr>
<tr>
<td>6.3</td>
<td>PEEK irradiated with 50 keV electrons</td>
<td>83</td>
</tr>
<tr>
<td>6.4</td>
<td>PTFE irradiated with 50 keV electrons</td>
<td>84</td>
</tr>
<tr>
<td>6.5</td>
<td>PEEK irradiated with 80 keV electrons</td>
<td>85</td>
</tr>
<tr>
<td>6.6</td>
<td>PTFE irradiated with 80 keV electrons</td>
<td>87</td>
</tr>
<tr>
<td>6.7</td>
<td>Low energy electron irradiation details</td>
<td>97</td>
</tr>
<tr>
<td>A.1</td>
<td>Material properties of coupling media</td>
<td>175</td>
</tr>
<tr>
<td>A.2</td>
<td>Electrode surface roughness parameters</td>
<td>185</td>
</tr>
</tbody>
</table>
### LIST OF FIGURES

<table>
<thead>
<tr>
<th>Figure</th>
<th>Description</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>Figure 1.1</td>
<td>Representative electron environment fluxes versus electron energy</td>
<td>5</td>
</tr>
<tr>
<td>Figure 1.2</td>
<td>Electron range as a function of incident energy</td>
<td>7</td>
</tr>
<tr>
<td>Figure 2.1</td>
<td>Idealized band diagrams</td>
<td>11</td>
</tr>
<tr>
<td>Figure 2.2</td>
<td>2D representation of atoms</td>
<td>12</td>
</tr>
<tr>
<td>Figure 2.3</td>
<td>Schematic Diagram of the Mott transition</td>
<td>13</td>
</tr>
<tr>
<td>Figure 2.4</td>
<td>Schematic Diagram of the Anderson Transition</td>
<td>14</td>
</tr>
<tr>
<td>Figure 2.5</td>
<td>Schematic diagram of variable range hopping</td>
<td>15</td>
</tr>
<tr>
<td>Figure 2.6</td>
<td>Typical output plots from AF-NUMIT3</td>
<td>20</td>
</tr>
<tr>
<td>Figure 3.1</td>
<td>Representative schematic of the PEA system</td>
<td>22</td>
</tr>
<tr>
<td>Figure 3.2</td>
<td>PEA system schematic</td>
<td>27</td>
</tr>
<tr>
<td>Figure 3.3</td>
<td>Photos of the PEA system at USU</td>
<td>28</td>
</tr>
<tr>
<td>Figure 3.4</td>
<td>Representation of PEA sample stack</td>
<td>29</td>
</tr>
<tr>
<td>Figure 3.5</td>
<td>Pictures of the PEA sensor assembly</td>
<td>29</td>
</tr>
<tr>
<td>Figure 3.6</td>
<td>A representative PEA measurement</td>
<td>31</td>
</tr>
<tr>
<td>Figure 3.7</td>
<td>USU Electron Irradiation Chamber</td>
<td>36</td>
</tr>
<tr>
<td>Figure 3.8</td>
<td>Electrons transmitted through carbon foil</td>
<td>37</td>
</tr>
<tr>
<td>Figure 3.9</td>
<td>Peak transmitted electron energy</td>
<td>37</td>
</tr>
<tr>
<td>Figure 4.1</td>
<td>Outline of data processing for PEA</td>
<td>39</td>
</tr>
<tr>
<td>Figure 4.2</td>
<td>Filter plotted with FFT of PEA data</td>
<td>40</td>
</tr>
<tr>
<td>Figure 4.3</td>
<td>Effects of filtering on PEA results</td>
<td>41</td>
</tr>
<tr>
<td>Figure 5.1</td>
<td>Average flux incident on samples during the 50 keV irradiation</td>
<td>49</td>
</tr>
</tbody>
</table>
Figure A.5 Oscilloscope testing with 150 MHz signal ........................................ 148
Figure A.6 Oscilloscope testing with 1 MHz signal ........................................ 149
Figure A.7 Schematic of high voltage pulse generator test circuit ..................... 150
Figure A.8 Accuracy of FWHM settings ............................................................. 150
Figure A.9 Accuracy of amplitude settings ......................................................... 151
Figure A.10 HVPG bandwidth versus amplitude ............................................... 151
Figure A.11 2 kV example HVPG measurement ............................................... 152
Figure A.12 2 kV FFT example of HVPG measurement ..................................... 153
Figure A.13 Normalized 2 kV example HVPG measurement ............................. 153
Figure A.14 Normalized 2 kV FFT example of HVPG measurement ................. 154
Figure A.15 5 ns example HVPG measurement ............................................... 154
Figure A.16 5 ns FFT example of HVPG measurement ..................................... 155
Figure A.17 Normalized 5 ns example HVPG measurement ............................. 155
Figure A.18 Normalized 5 ns FFT example of HVPG measurement ................. 156
Figure A.19 12 V battery power supply noise ............................................... 157
Figure A.20 12 V AC-drive power supply noise ............................................ 158
Figure A.21 12 V power supply noise FFT ...................................................... 159
Figure A.22 High Voltage DC power supply testing ........................................ 161
Figure A.23 Frequency spectrum of high voltage DC power supply noise ........... 162
Figure A.24 RF amplifier test ................................................................. 163
Figure A.25 Amplifier gain ................................................................. 164
Figure A.26 Ultrasonic testing of PEA system .............................................. 166
Figure A.27 PEA system sensor thickness dependence ..................................... 168
Figure A.28 Impedance matching circuits ....................................................... 171
Figure A.29 Impedance matching and $n_{eff}(f)$ ........................................... 171
Figure A.30 Impedance matching PEA and pulse results . . . . . . . . . . . . . . 172
Figure A.31 Simplified drawing of the PEA sample stack with coupling media . . 175
Figure A.32 Various coupling layers for multilayer polyimide . . . . . . . . . . . . 177
Figure A.33 PEA measurements with copper backing . . . . . . . . . . . . . . 178
Figure A.34 Cable reflection effects for HPVG to sample stack . . . . . . . . . . 181
Figure A.35 Cable reflection effects for sensor to amplifier . . . . . . . . . . . . 182
Figure A.36 Cable reflection effects for amplifier to oscilloscope . . . . . . . . 183
Figure A.37 Confocal microscope measurements of polished Al electrode . . . . 186
Figure A.38 Confocal microscope measurements of unpolished Al electrode . . . 187
Figure A.39 Surface roughness of PTFE . . . . . . . . . . . . . . . . . . . . . . 188
Figure B.1 Amplitude of interfacial peaks as a function of DC bias . . . . . . . 191
Figure B.2 Calibration factor and speed of sound as a function of DC bias . . . 192
Figure B.3 Modified calibration factor with pulse effects . . . . . . . . . . . . 193
Figure B.4 Charging effects as observed through amplitude of interfacial peaks . 194
NOMENCLATURE

\( A \) = Area

\( A_i \) = Mass number of element \( i \)

\( A'_i \) = Atomic weight of element \( i \)

\( BW \) = Bandwidth

\( C \) = Constant

\( c \) = Speed of light

\( d \) = Thickness

\( \dot{D}(x) \) = Dose rate as a function of depth

\( E(x, t) \) = Electric field as a function of depth and time

\( e_p(t) \) = Electric field due to pulsed voltage

\( f \) = Frequency

\( f(x, t) \) = Maxwell stress

\( h(t) \) = System response function

\( H(\omega) \) = FFT of system response function

\( J(x, t) \) = Current as a function of position and time

\( J_0(x) \) = Initial current as a function of position

\( k \) = Wave vector

\( K \) = Calibration factor in units of \((C/m^2)(1/Vs)\)
\( K' \) = Calibration factor in units of \((C/m^3)(1/V)\)

\( k_B \) = Boltzmann constant

\( k_{RIC} \) = Coefficient of radiation induced conductivity

\( M \) = Molecular weight of compound

\( n_i \) = Number of atoms of element \( i \)

\( n(t) \) = Noise

\( N(\omega) \) = FFT of noise

\( n_{eff}(f) \) = Ratio of voltage across sample to applied voltage

\( p(x,t) \) = Pressure wave

\( P_{\text{incident}} \) = Incident power

\( P_{\text{reflected}} \) = Reflected power

\( Q \) = Charge

\( Q_{\text{noise}} \) = Noise parameter for deconvolution

\( r \) = Position vector

\( t \) = Time

\( t_{PK2PK} \) = Peak-to-peak time difference between interfacial peaks for a pulsed electroacoustic measurement

\( T_{\text{room}} \) = Room temperature

\( U \) = Energy depth of potential wells

\( u_k(r) \) = Periodic potential

\( V_{DC} \) = Direct current voltage
$V_{\text{meas}} = $ Measured voltage signal

$V_p(t) = $ Pulsed voltage

$v_s = $ Speed of sound

$W = $ Width between potential wells

$x = $ Distance

$x(t) = $ Embedded charge distribution signal

$X(\omega) = $ FFT of embedded charge distribution signal

$y(t) = $ Measurement

$Y(\omega) = $ FFT of measurement

$Z = $ Mean atomic number

$\epsilon_0 = $ Permittivity of free space

$\epsilon_r = $ Relative permittivity

$\rho(x) = $ Volume charge density

$\sigma(x) = $ Sheet charge

$\sigma_{\text{dark}} = $ Dark conductivity

$\sigma_{Pk2Pk} = $ Error in the peak-to-peak time difference of a pulsed electroacoustic measurement

$\sigma_{\text{speed}} = $ Error in the speed of sound of a material

$\sigma_{\text{Temp}} = $ Error in speed of sound due to temperature fluctuations

$\sigma_{\text{thickness}} = $ Error in the thickness of a sample

$\tau_{\text{decay}} = $ Charge decay time
\[ \tau_{\text{rise}} = \text{Rise time of pulse} \]
\[ \psi_k = \text{Wavefunction solutions for periodic potential} \]
LIST OF ABBREVIATIONS

<table>
<thead>
<tr>
<th>Abbreviation</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>AC</td>
<td>Alternating current</td>
</tr>
<tr>
<td>AF-NUMIT3</td>
<td>Air Force - NUMerical ITeration version 3</td>
</tr>
<tr>
<td>AFRL</td>
<td>Air Force Research Laboratory</td>
</tr>
<tr>
<td>ATS-6</td>
<td>Applications Technology Satellite-6</td>
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<tr>
<td>BEI</td>
<td>Box Elder Innovations</td>
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<tr>
<td>CASINO</td>
<td>monte CARlo SIMulation of electroN trajectory in sOlids</td>
</tr>
<tr>
<td>CEIDP</td>
<td>Conference on Electrical Insulation and Dielectric Phenomena</td>
</tr>
<tr>
<td>DC</td>
<td>Direct current</td>
</tr>
<tr>
<td>DICTAT</td>
<td>Dielectric Internal Charging Threat Assessment Tool</td>
</tr>
<tr>
<td>DRIC</td>
<td>Delayed radiation induced conductivity</td>
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<tr>
<td>EAR</td>
<td>electroacoustic reflectometry</td>
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<tr>
<td>EDEPOS</td>
<td>Energy DEPOSited</td>
</tr>
<tr>
<td>ESAW</td>
<td>Electrically stimulated acoustic wave</td>
</tr>
<tr>
<td>ESD</td>
<td>Electrostatic discharge</td>
</tr>
<tr>
<td>ESTAR</td>
<td>Electron STopping-power And Range</td>
</tr>
<tr>
<td>FFT</td>
<td>Fast Fourier Transform</td>
</tr>
<tr>
<td>FWHM</td>
<td>Full width at half maximum</td>
</tr>
<tr>
<td>GEANT4</td>
<td>GEometry ANd Tracking version 4</td>
</tr>
<tr>
<td>GEANT4-RIC</td>
<td>GEometry ANd Tracking version 4 with Radiation Induced Conductivity</td>
</tr>
<tr>
<td>HDIM</td>
<td>Highly disordered insulating material</td>
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<tr>
<td>HVDC</td>
<td>High voltage direct current</td>
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<td>HVPG</td>
<td>High voltage pulse generator</td>
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<tr>
<td>HV</td>
<td>High voltage</td>
</tr>
<tr>
<td>HWHM</td>
<td>Half width half maximum</td>
</tr>
</tbody>
</table>
IEC International Electrotechnical Commission
JPL Jet Propulsion Laboratory
LAPLACE Laboratoire Plasma et Conversion d’Energie
LDPE Low density polyethylene
LIMM Laser intensity modulation method
LSIP Linear subtraction of the interfacial peak
MCICT Monte Carlo Internal Charging Tool
MPG Materials Physics Group
MT Multiple trapping
NIST National Institute of Standards and Technology
NUMIT NUMerical ITeration
NUMIT 3D NUMerical ITeration in 3 dimensions
PEA Pulsed electroacoustic
PEEK Polyether-etherketone
PI Polyimide
PMMA Polymethyl methacrylate
PTFE Polytetrafluoroethylene
PVC Polyvinyl chloride
PVDF Polyvinylidene fluoride
PWP Pressure wave propagation
RIC Radiation induced conductivity
RIS Random interleaved sampling
RL Return loss
RPM Revolutions per minute
SCATHA Spacecraft Charging AT High Altitude
SiO2 Silicon glass
SMA SubMiniature version A
SNR Signal to noise ratio
<table>
<thead>
<tr>
<th>Abbreviation</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>SWR</td>
<td>Standing wave ratio</td>
</tr>
<tr>
<td>TAH</td>
<td>Thermally assisted hopping</td>
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<tr>
<td>THEMIS</td>
<td>Transport of Holes and electrons Model under Irradiation in Space</td>
</tr>
<tr>
<td>TID</td>
<td>Total ionizing dose</td>
</tr>
<tr>
<td>TSBC</td>
<td>Thermally stimulated discharge current</td>
</tr>
<tr>
<td>UK</td>
<td>United Kingdom</td>
</tr>
<tr>
<td>UPS</td>
<td>Université Paul Sabatier</td>
</tr>
<tr>
<td>US</td>
<td>United States</td>
</tr>
<tr>
<td>USU</td>
<td>Utah State University</td>
</tr>
<tr>
<td>VRH</td>
<td>Variable range hopping</td>
</tr>
<tr>
<td>1D</td>
<td>1 dimension</td>
</tr>
<tr>
<td>2D</td>
<td>2 dimension</td>
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<tr>
<td>3D</td>
<td>3 dimension</td>
</tr>
</tbody>
</table>
CHAPTER 1
INTRODUCTION

The focus of this dissertation is on electron transport in highly disordered insulating materials (HDIM) as explored by the use of pulsed electroacoustic (PEA) measurements. Knowledge of the spatial distribution and temporal evolution of embedded charge in HDIM is important in a variety of applications including semiconductor devices, high-power electronic devices, high voltage DC cabling, high energy physics facilities, plasma chambers, and spacecraft charging (Dennison and Pearson, 2013; Hussaini et al., 2015).

Electrostatic discharge (ESD) events are the leading cause of spacecraft failure due to the space environment and cause the majority of space environment induced anomalies (Bedingfield and Leach, 1996; Ferguson et al., 2015; Garrett and Whittlesey, 2000; Leach, 1995). As satellites orbit, they acquire excess charge by interacting with space plasma, solar wind, and trapped charges in the space environment. This is analogous to rubbing your shoes on the carpet to build up charge that can then be discharged by shocking a friend. Unlike you, the satellite has no friends and no place to ground and discharge the excess charge. When enough charge has accumulated in the insulating materials protecting the spacecraft, it can undergo electrostatic discharge (ESD). This often causes irreparable damage to the spacecraft. Charge dissipation (as opposed to electrostatic discharge) can occur due to radiation induced conductivity due to bombardment with radiation, field enhanced conductivity induced by the excess charge, and also bulk conductivity to adjacent materials and grounded surfaces (Purvis et al., 1984). To mitigate these effects, it is important to have a fundamental understanding of the charge accumulation, distribution, and migration in HDIM.

With PEA measurements you can essentially “pop the hood” and look at the charge embedded in the material. With a simple parallel plate sample configuration, a voltage pulse across the plates produces a force on the embedded charge effectively creating a pressure
wave that propagates through a test material to a piezoelectric sensor mounted on the back of an electrode. Given the speed of sound in the relevant materials, a simple time-of-flight analysis determines the internal charge distribution.

To assure successful space missions, spacecraft charging must be understood and mitigated. The most important factor for dielectric spacecraft materials is arguably the internal electric field, which is determined by the magnitude and spatial distribution of the internal charge distribution. There are many ways to attempt to determine what the electric field is inside a material. Measurements of material properties can help to inform through analytical formulas (Rao, 1966) or simulations such as AF-NUMIT3 (Beecken et al., 2015), JPL NUMIT (Jun et al., 2008), and DICTAT (Rodgers et al., 1998b). Necessary parameters for these calculations can include material properties that can be experimentally determined (NASA, 2011) such as the bulk conductivity/resistivity (Wood et al., 2018), breakdown field strength (Andersen, 2018), radiation induced conductivity (Boman et al., 2021; Gillespie, 2022), delayed radiation induced conductivity (Gillespie, 2013; Yang and Sessler, 1992), photovield, electron yield (Hoffmann et al., 2008; Thomson, 2005), ion yield, and permittivity (Lee et al., 2021). However, these measurements are indirect and only help to provide inferred values of the internal field when materials are exposed to the space environment. The most direct way to measure the details of the internal electric field is to determine the distribution and magnitude of the embedded charge. This is precisely the information pulsed electroacoustic measurements can directly provide.

Spacecraft charging issues result primarily from electron space plasma fluxes with 10’s of keV energies (Frooninckx and Sojka, 1992; Hastings and Garrett, 2004), which have penetration depths on the order of ∼1-100 μm (Wilson and Dennison, 2012). Typical resolutions of current state-of-the-art PEA systems are on the order of 10 μm (Arnaout et al., 2010; Boudou et al., 2004; Gallot-Lavallée et al., 2005; Griseri et al., 2006; Maeno and Fukunaga, 1996; Maeno et al., 1988), and the highest resolution systems reported in the literature include 1.6-2.5 μm (Kumaoka et al., 2014; Kumaoka et al., 2015; Sonoda et al., 2017) and 4.6 μm (Galloy et al., 2016). Spatial resolution is most commonly defined as the
full width at half maximum (FWHM) of the leading interfacial peak in a PEA waveform (Galloy et al., 2016; Gibson and Dennison, 2022b; International Electrotechnical Commission, 2012). Therefore, we have a need for higher spatial resolution to fully understand and address spacecraft charging issues. This will be discussed further in Section 1.1.

The aim of this dissertation is to address spacecraft charging issues with the PEA method. As such, an in-depth analysis of the uncertainty of the PEA method is performed, the improvement in spatial resolution is addressed, and exploration of other creative avenues for obtaining more information from PEA measurements relevant to spacecraft charging is explored.

The dissertation is organized as follows. The rest of this chapter will lay out the issues of spacecraft charging addressed here and the history of the PEA method. The background theory and modeling of charge transport in dielectric materials will be discussed in Chapter 2. The experimental methods, sample preparation, and charge deposition methods will be discussed in Chapter 3, followed by data processing procedures in Chapter 4. An assessment of the PEA method’s uncertainty (Gibson and Dennison, 2022b) and validation of the results (Gibson et al., 2022) will be discussed in Chapter 5. Then results of several experiments will be presented in Chapter 6 to illustrate the topics discussed above. These experiments aim to measure embedded charge to validate a deep dielectric charging code (Gibson et al., 2023), show the relevancy of PEA measurements to spacecraft charging (Gibson and Dennison, 2022a, 2023a) and provide a demonstration and justification of a novel method for measuring shallowly deposited charge distributions relevant to spacecraft charging (Gibson and Dennison, 2023b). Finally, Chapter 7 will provide a summary and conclusions of the dissertation, as well as propose future work.

1.1 Spacecraft Charging

The pulsed electroacoustic (PEA) method provides nondestructive measurements of charge distributions in dielectric materials (Dennison and Pearson, 2013; Maeno et al., 1988). Charge can be deposited into a material in the lab through high voltage DC bias across electrodes or via irradiation with a beam of charged particles.
In open PEA (or non-contact) systems, the excitation electrode is not in contact with the front surface of the sample (Griseri et al., 2003; Riffaud et al., 2016). With either short circuit or ambient/classic PEA systems, the excitation electrode is in direct contact with the front surface of the sample (Maeno et al., 1988). Note that for all PEA systems, the ground electrode is in direct contact with the back of the sample. There can be substantial differences in the results depending on the electrode configuration. For example, in an open PEA electrode configuration there is no signal from the surface of the sample near the excitation electrode due to the air gap (Perrin et al., 2008).

This section outlines representative electron space environments and typical spacecraft charging conditions. It concludes with identifying the relevant length scales in the regime of incident electron energies most relevant to spacecraft charging. The focus is on the electron environment, as electrons drive the bulk of the charging in the space environment (Dennison et al., 2007; Hastings and Garrett, 2004).

1.1.1 Typical Near-Earth Orbit Electron Environments

Figure 1.1 presents typical differential electron fluxes as a function of electron energy in near-Earth orbits, as well as for lunar and interplanetary environments. Note the log-log axes. Plotting differential fluxes as a function of electron energy is useful as the area under the curve corresponds to the total number of electrons in the energy range of interest. Space environments typically have higher flux at lower energies.

1.1.2 Typical Spacecraft Charging Conditions

Olsen (1983) suggests that there appears to be a threshold electron energy for charging of a spacecraft to occur. Data from ATS6 and SCATHA missions showing the spacecraft potential as a function of incident electron energy are plotted in Fig. 3 Olsen (1983). There is an onset or cut-off energy around 10 keV where charging appears to begin. Olsen (1983) discusses a “critical charging regime” for spacecraft that can vary from 10 keV to 80 keV: “...it appears that the spacecraft does not charge unless there are substantial (electron) fluxes between 10 and 20 keV.” Other literature offer similar conclusions (Frooninckx and
Figure 1.1: Representative electron environment fluxes versus electron energy. After (Dennison et al., 2014) and based on values from Minow, Sojka, 1992; Garrett, 1981; Garrett et al., 1980; Gussenhoven and Mullen, 1981; Reagan et al., 1981). This empirical result indicates that the electron energies of particular importance for spacecraft charging are in the region of 10 keV to 50 keV (highlighted in red in Fig. 1.1), and perhaps up to 80 keV or more.

The incident electron energy range of 10 keV to 50 keV is identified as a critical energy range from these observations for spacecraft charging. This range of incident energies needs to be correlated to a length scale to determine if embedded electron distributions resulting from electron fluxes in these energy ranges can be measured with PEA to address the relevance of PEA measurements to spacecraft charging studies.

1.1.3 Length Scales for Spacecraft Charging

It is helpful to consider two parameters to map the electron energy regime of interest into relevant length scales. These are the peak deposition depth and range of the incident
electrons. The peak deposition depth is the depth at which the maximum amount of charge is deposited. The range is the maximum depth even a single electron reaches into the material at a given incident electron energy. The peak deposition depth has been estimated as 2/3 of the range (Wilson, 2021). The range for numerous materials at energies, $\lesssim 10$ keV, have been modeled and tabulated; refer to ESTAR at NIST (Berger et al., 1999). The Material Physics Group (MPG) Electron Range Approximation Tool used here is in excellent agreement for the materials tabulated in ESTAR (Wilson and Dennison, 2012). The MPG tool provides the ability to predict the electron range for arbitrary materials down to lower energies $\leq 10$ eV using only the stoichiometry, density, and estimated bandgap (Wilson et al., 2013; Wilson et al., 2018). This capability was exploited to predict the range in several common spacecraft materials. A general trend is seen that materials with higher effective atomic numbers correspond to lower electron penetration depths.

The range for four ubiquitous insulating spacecraft materials, low density polyethylene (LDPE), polytetrafluoroethylene (PTFE), silicon glass (SiO2) and polyether-etherketone (PEEK), are plotted over the relevant energy range for spacecraft charging in Fig. 1.2. These calculations were produced with MPG Electron Range Approximation Tool v1.1 (Wilson and Dennison, 2012; Wilson et al., 2013; Wilson et al., 2018). Dashed lines indicate peak deposition depths as estimated as 2/3 of the range. The horizontal dashed black line is a distance of 15 $\mu$m. Note that the peak deposition depth for PEEK (yellow dashed line) is nearly identical to the range of PTFE (purple line).

Range depends on a number of factors, but largely on the mean atomic number, Z, of a material. LDPE and PTFE span the range from low Z to high Z, respectively (and hence low to high ranges at a given energy) for common spacecraft polymeric insulators. Silicon glass is a common spacecraft dielectric with higher Z and lower range. Throughout this dissertation, PEEK will often be the focus of studies as PEEK displays intermediate range values and advantageous acoustic properties for PEA measurements.

### 1.2 PEA History and Other Measurement Methods

The pulsed electroacoustic system is utilized worldwide by groups including those in
Figure 1.2: Electron range as a function of incident energy. The electron ranges for representative spacecraft materials as a function of incident electron energy.

Japan (Kumaoka et al., 2014; Kumaoka et al., 2015; Maeno, 1999, 2001; Maeno and Fukunaga, 1996; Maeno et al., 1988; Sonoda et al., 2017), France (Griseri et al., 2003; Griseri et al., 2004; Griseri et al., 2006; Griseri et al., 2009; Hole, 2008; Nguyen et al., 2011; Riffaud et al., 2016), United Kingdom (UK) (Chen et al., 2001), China (Huang et al., 2014), and Italy (Imburgia et al., 2016), but very few systems exist in the United States (US). It appears that the original pulsed electroacoustic system was developed by Maeno, Kushibe, Takada and Futami from Musashi Institute for Technology (now Tokyo City University) with Cooke from Massachusetts Institute for Technology (Maeno et al., 1988). One of Cooke’s PhD students developed a pulsed electroacoustic system but under the name of the electrically stimulated acoustic wave (ESAW) method (Bernstein, 1991, 1992). The system at Massachusetts Institute for Technology, as well as systems at University of Connecticut (Tefferi et al., 2019) and Rensselaer Polytechnic Institute (Nelson and Fothergill, 2004) are the only academic PEA systems that appear in the literature originating in the US. However, the latter seems to be unused as the professor, Keith Nelson, who used the system is now emeritus. Due to our collaboration with Box Elder Innovations (BEI) in developing the USU BEI PEA system, I am aware there are several other PEA systems that have recently been distributed throughout the US in private industry and national labs.
There are currently a few different methods for measuring charge distributions in thin HDIM. PEA measurements are arguably the most promising of several methods for making these measurements for many applications. The other two well-developed methods are Pressure Wave Propagation (PWP) and Laser Intensity Modulation Method (LIMM). A more recently developed method for measuring embedded charge distributions is called electroacoustic reflectometry (EAR) (Hamidouche et al., 2017). The advantages to the PEA method are nondestructive measurements, low cost, simplified modeling, and high resolution. For a more in-depth discussion refer to Dennison and Pearson (2013) and Imburgia et al. (2016).
CHAPTER 2
THEORY AND MODELING

A concrete understanding of the nature of electrical properties is elusive when it comes to disordered media. Insight has been gained through the help of dispersive transport models (Pfister and Griffiths, 1978; Scher and Montroll, 1975; Zallen, 1983). Transport models attempt to link the microscopic nature of the underlying physical processes to macroscopic properties such as measured conductivity and permittivity.

Charge transport in crystalline material is well understood through the use of extended state Bloch functions (Ashcroft and Mermin, 2022). This leads to the concept of band structure and band gaps, which helps to navigate the electrical properties of crystalline materials (Ashcroft and Mermin, 2022; Kittel, 1976). These methods depend upon the symmetries introduced through crystalline structure and must be almost entirely abandoned when there is a departure from crystalline structure, such as that introduced by defects or outright disordered materials with no long-range order. The conduction mechanisms in disordered materials have come to be understood through various mechanisms such as thermally assisted hopping (TAH), variable range hopping (VRH), multiple trapping (MT), and percolation theories. The common theme of these conduction mechanisms revolves around the idea of localization. These localized states appear within the band gap, fittingly referred to as the mobility gap for disordered media (refer to Fig. 2.1) (Mott and Davis, 2012; Wintle, 1983; Zallen, 1983). Sim (2013) dissertation provides a detailed review of the theory of charge transport in HDIM from the perspective of the USU MPG.

The theory behind charge transport in disordered insulating materials is summarized in this chapter, but first an overview is given for crystalline materials.

2.1 Electron Transport in Crystalline Materials

Electron transport in crystalline materials can be understood through the introduction
of a periodic potential in a lattice. It was discovered by F. Bloch that the Schrödinger
equation can be solved analytically for any infinite periodic potential. The solutions are
Bloch wavefunctions of the form,

$$\psi_k(t) = u_k(r)e^{ik\cdot r},$$  \hspace{1cm} (2.1)

where \( u_k(r) \) is the periodic potential with the same period of the crystal lattice, \( k \) is the
wave vector, and \( r \) is the position vector. This leads to the existence of band structures
(Ashcroft and Mermin, 2022; Kittel, 1976). This allows for the differentiation of electronic
properties according to the electron occupancy of the outermost valence bands. In other
words, the outermost band (in energy) is either partially filled (conductors) or exactly
filled (semiconductors and insulators) at low temperature. The difference between semicon-
ductors and insulators is the difference in energy between the filled valence band and the
next available conduction band. Semiconductors at room temperature \( T_{room} \) are somewhat
arbitrarily defined as having a bandgap of \( \sim 20-100 \) times \( k_B T_{room} \), and insulators above
that range, where \( k_B \) is the Boltzmann constant. In metals, electrons can move around
essentially freely in extended states in partially filled conduction band.

In disordered materials, there is no longer any long-range order (refer to Fig. 2.2). This
means that Bloch functions (Eq. 2.1) are no longer applicable due to a lack of a periodicity.
However, it turns out that something like band structures still exist in amorphous materials
as validated by experiments measuring a dispersion-like relation, relating the real electron
momentum, rather than the usual crystal momentum, to the energy (Cai et al., 1995;
Dennison and Ritter, 1996; Ritter et al., 1984; Vos et al., 1994). For disordered materials,
the effective band gap is referred to as the mobility gap and states within the mobility gap
are localized (refer to Fig. 2.1).

2.2 Electron Transport in Amorphous Materials

The theory of electron transport in amorphous materials is outlined in this section.
2.2.1 Localization

The understanding of materials through use of Bloch functions falls apart when disorder is introduced. The underlying assumption of periodicity can no longer be used. Some insight can still be gained through simple perturbations from periodicity of the energetic and spatial distributions of defect states. These two perturbations correspond to two transitions from metal to insulators described by Mott (1949, 1968) and Anderson (1958) by increasing spatial separation and adding energetic disorder, respectively. Figures 2.3 and 2.4 schematically show spatial and energy distributions of Mott and Anderson transitions,
Figure 2.2: 2D representation of atoms. Shown are representations for (a) crystalline material (b) glassy/disordered material (c) and gas. Crystals exhibit long range order. Glass can be seen to have short range order. Gas shows no order and the atoms do not have a steady state equilibrium point. After (Zallen, 1983).

respectively. If the width ‘W’ or energy ‘U’ is greater than the bandwidth ‘BW’, then a transition to insulator with localized states occurs. This is qualitatively simple to understand, since it seems intuitive that at some point the states will no longer interact with each other to form extended states if they are separated enough in either energy or space. The underlying mathematics are much more complex (Anderson, 1958; Mott, 1968, 1949; Mott and Davis, 2012; Sim, 2013; Zallen, 1983).

Often in crystalline materials, conduction via extended states dominate. In disordered materials where localized states are present, conduction can be dominated by hopping or multiple trapping with transport from one localized state to another adjacent localized state. This is the topic of discussion in the following section.

2.2.2 Conduction Mechanisms

Extended state conduction is still possible in disordered materials. However, sufficient energy must be given to an electron to excite it into the conduction band. This is more easily accessible for electrons in shallow traps near the mobility edge. Where “shallow” means small energies below the conduction band. Often, thermally assisted hopping (TAH) refers to the process of electrons excited due to thermal energy from shallow trap states up into the conduction band where they can conduct for a time before they fall back into a lower energy trap state, as seen in paths C and D in Fig. 2.5 (Zallen, 1983). This is more
Figure 2.3: Schematic diagram of the Mott transition. When the spatial separation is enough to decrease the electron bandwidth $BW$ to be sufficiently smaller than the electron-electron energy $U$, correlation-induced localization takes place leading to a conductor-insulator transition. After (Zallen, 1983)

accurately referred to as multiple trapping (MT). MT is a more fitting name as hopping seems to imply a quantum tunneling aspect which is not applicable in this description of TAH.

Variable range hopping (VRH) refers to hopping conduction such as depicted in paths A and B in Fig. 2.5 with energetic and spatial separations of $W$ and $R$, respectively (Sim, 2013). VRH dominates when the temperature is sufficiently low that MT is negligible and quantum effects take over. VRH is phonon assisted quantum tunneling (hopping). For a more in-depth and nuanced discussion of conduction mechanisms in HDIM refer to Sim (2013).

If excess energy is added through irradiation rather than thermal excitations then radiation induced conductivity (RIC) can occur (Fowler, 1956; Gillespie, 2022; Rose, 1951; Tyutnev et al., 2015). An electron in a deep trap state is excited into the conduction band in an extended state via irradiation (as opposed to a phonon assisted jump to a localized state within the mobility gap for VRH), where it will conduct for a time but will eventually
Figure 2.4: Schematic diagram of the Anderson transition. When the energetic disorder causes the width $W$ to be sufficiently larger than the bandwidth $BW$, disorder-induced localization takes place, leading to a conductor to insulator transition. After (Zallen, 1983)

fall back into a deep trap state. Schematically, this would correspond to a jump from a deeper trap to the conduction band. From there, the electron may continue to undergo MT or fall back into a deep trap state. Analogously, these same mechanisms can apply to hole transport if the electron vacancies are now considered hole occupancies. In other words, the valence band is essentially the conduction band for holes (Sim, 2013).

Another way to look at conduction in disordered materials is through percolation theories (Murayama and Mori, 1992; Pfister and Scher, 1978; Zallen, 1983). Zallen (1983) explains it simply as if a saboteur was trying to cut communications between two locations; how many links would need to be cut before connection was completely cut-off? This can be described as the fraction of links/bonds that need to be severed for a metal-insulator transition to occur. In other words, this critical percolation threshold defines an Anderson transition. This idea of blocked and unblocked sections of networks (or networks of resistors) can be extended to understand many different things. This will not be explored here, but the interested reader can refer to an in depth review of percolation and fractal theory
in the context of diffusion in disordered media by Havlin (Havlin and Ben-Avraham, 2002).

It has been shown by Pfister and Scher (1978) that any of these transport models (hopping, MT, and percolation) can lead to broad distributions of event times, and therefore dispersive transport. The dispersion parameter and charge transport in disorder materials are discussed extensively in references Gibson (2019) and Sim (2013).

While conceptual and analytic understanding of charge transport theory is important for building an intuition and understanding charge transport in dielectric materials, much of the work in many applications is often accomplished with models or simulations, which

**Figure 2.5:** Schematic diagram of variable range hopping. Energy is the vertical direction and position in the horizontal. After (Zallen, 1983).
are presented in the next section.

2.3 Modeling Charge Transport with AF-NUMIT3

Charging codes can simulate charging of materials much more quickly and easily than through experimentation. Simulation also allows for scenarios that may not be feasible through experimentation, thereby facilitating exploration of a broader range of parameters. However, it is of great importance to validate these codes experimentally to have the highest confidence in the results and an understanding of their limitations.

There are several charging models in use including MCICT (Lei et al., 2016; Pacaud et al., 2019), DICTAT (Rodgers et al., 1998b; Sorensen et al., 2000), THEMIS (Pacaud et al., 2019), GEANT4 (Agostinelli et al., 2003), GEANT4-RIC (Yu et al., 2016), and NUMIT (Beecken, 2014; Beecken et al., 2015; Jun et al., 2008; Kim et al., 2017). There has been a divergence of the NUMIT code since its original inception by the late A.R. Frederickson (Frederickson, 1974, 1975). One version has been adapted by the Jet Propulsion Laboratory (JPL) as NUMIT 2.1 (Jun et al., 2008), as well as a 3D version NUMIT 3D (Kim et al., 2017). This paper will focus on experimental validation of AF-NUMIT3, an adaptation of NUMIT developed at the Air Force Research Lab (AFRL) by Brian Beecken (2014) (Beecken et al., 2015, 2022).

As part of the work presented in this dissertation, PEA measurements were taken with the intent of validating AF-NUMIT3 (Gibson et al., 2023). As such, AF-NUMIT3 will be described in more detail in this section. Results of the validation of the charging code are presented in Chapter 6.

AF-NUMIT3 Overview

In the 1970s Frederickson developed a primitive computer model, dubbed NUMIT (NUMerical ITeration), designed to correlate experimental observations of charge deposition and transport in dielectric materials with the dynamic situation within the material (Frederickson, 1974, 1975). The 1D model estimated where within the dielectric the electric charge was deposited and the changes to the conductivity of the material resulting from
the concomitant deposition of energy (RIC). Then, internal electric fields were calculated, and the resulting movement of charge within the material was predicted. The simulation continued in an iterative fashion using user-defined short time intervals. Since Frederickson’s initial idea, numerous other investigators have pursued various improvements to his approach. These include the 1D code NUMIT 2.1 (Jun et al., 2008) and a 3D version NUMIT 3D (Kim et al., 2017) adapted by NASA JPL, and a NUMIT based code developed at Marshal Space Flight Center (Posey and Minow, 2013; Willis et al., 2013). A number of similar codes mentioned in the Introduction also trace much of their underlying physics principles to the original NUMIT work.

Results presented in this dissertation use AF-NUMIT3, a redesign and extension of the original NUMIT developed at AFRL by Brian Beecken, used primarily to simulate charging resulting from the space environment (Barton et al., 2015; Beecken, 2014; Beecken et al., 2015). Since the inception of NUMIT, significant further enhancements have been made. AF-NUMIT3 now allows additional phenomena to be simulated, such as a movable/removable front surface electrode, evolving dielectric surface potentials, reduction of incident electron energy due to changing surface potentials, photo-emission, and secondary electron emission (Beecken et al., 2022). In addition, changes in dark conductivity or RIC with time and/or temperature can be modeled. AF-NUMIT3 can simulate charging both in various lab settings (electron beam with normal incidence) and in an approximate space environment (isotropic flux of incident electrons). The code can handle monoenergetic or broadband sources. Further details of how AF-NUMIT3 works are described in (Beecken, 2014; Beecken et al., 2014; Beecken et al., 2015).

The following subsections outline the input parameters, computational methods, and outputs of the AF-NUMIT3 deep dielectric charging code.

**Input Parameters**

The material inputs to the AF-NUMIT3 can all be measured or calculated with tools available to the USU MPG lab. The input parameters are effective atomic number and mean atomic weight from the material composition and stoichiometry, density, relative per-
mittivity, dark (bulk) conductivity, and RIC coefficient. These parameters are all known or measured for the materials studied in this dissertation. The incident electron flux and energy (normal incidence or isotropic) are also input parameters; modeling in this dissertation was for monoenergetic normal incidence electron beams.

It is worth noting that the method used for determining the effective atomic number and atomic weight for mixtures or compounds is,

\[
Z_{eff} = \sum_i f_i Z_i, \quad A_{eff} = \frac{Z_{eff}}{\sum_i f_i A_i},
\]

(2.2)

where,

\[
f_i = \frac{n_i A_i'}{M} = \frac{n_i A_i'}{\sum_j n_j A_j'}.
\]

(2.3)

Here \(n_i\) is the number of atoms of element \(i\), \(A\) is the mass number, \(A'\) is the atomic weight, and \(M\) is the molecular weight of the compound. These equations are from Tabata and Ito (1974) and Rao (1966).

**Methods**

The method for determining charge dynamics utilized by AF-NUMIT3 is outlined in detail in Beecken and Wallin (2011) and Beecken et al. (2015), and is summarized below.

The AF-NUMIT3 code works by determining an energy deposition profile with EDEPOS (short for energy deposition), which was created by Tabata in 1974 (Tabata and Ito, 1974). An electron deposition profile is then determined using the output of EDEPOS. Once the energy and electron deposition profiles are determined, then the charge transport algorithm can be applied with AF-NUMIT3. The three major equations used are the continuity equation, differential form of Gauss’s Law, and a third equation representing the charge transport. The charge transport equation utilizes the Fowler Model for radiation induced conductivity.

\[
J(x, t) = J_0(x) + [\sigma_{dark} + k_{RIC} \dot{D}(x)] E(x, t),
\]

(2.4)
In Eq. 2.4, $J$ is the current density, $J_0$ is the initial current density, $\sigma_{\text{dark}}$ is the dark conductivity, $k_{\text{RIC}}$ is the coefficient of RIC, $\dot{D}$ is the dose rate, and $E$ is the electric field. The term in brackets multiplied by the electric field is essentially the effective conductivity as it is the sum of the dark conductivity and RIC.

The initial current is due to the incident electrons and is determined from the input flux and incident energy. The continuity equation then enables us to find the charge density a time increment later. Once the charge density is known, Gauss’s Law allows the determination of the electric field. In conjunction with the other input parameters, Eq. 2.4 can then be used to determine the charge transport. This process is then repeated for the designated duration of simulation time in time steps defined by the time increment. Note that a careful choice of the spatial and temporal increments must be made such that the code does not take too long to run and sufficient spatial and temporal accuracy is achieved.

**Outputs**

After running AF-NUMIT3, the output includes several plots and an Excel sheet. The Excel sheet will contain simulated data for the internal electric field, electron current profile, distribution of deposited charge, and distribution of deposited energy density (dose rate). This is automatically plotted by AF-NUMIT3 as well. An example output from AF-NUMIT3 is shown in Fig. 2.6; these include the (a) electric field, (b) electron current profile, (c) energy deposition (dose rate) profile, and (d) charge distribution as functions of depth at four sequential time steps. Different times during the irradiation are shown for increasing time in the order black, red, green, blue. In these graphs, the black lines represent the initial situation immediately after irradiation begins. Note the electron beam is incident from the left.
Figure 2.6: Typical output plots from AF-NUMIT3. Plots include the (a) internal electric field, (b) electron current profile, (c) distribution of deposited charge, and (d) distribution of deposited energy density (dose rate). (Gibson et al., 2023)
CHAPTER 3
EXPERIMENTAL METHODS

This chapter provides a detailed explanation of the PEA method and the experimental set up. First, the principles of the PEA method will be described. Then the USU PEA system will be described, as well as details of the data acquisition, and methods for sample preparation. The section will end with a discussion of the instrumentation used to deposit charge in samples, including high voltage DC bias and electron beam irradiation. Processing of the data will be presented in the Chapter 4.

3.1 Pulsed Electroacoustic Method

This section will give a description of the PEA method and the details of the ambient PEA system in the USU lab.

3.1.1 Principles of PEA Method

An overview of the PEA method is outlined in Fig. 3.1, as follows. An electric field is applied through a pulsed voltage signal across a dielectric sample via a high voltage pulse generator. This pulse in turn produces a force on any embedded charge in the sample, resulting in an acoustic pressure wave that propagates through the system and can then be detected by a piezoelectric transducer. The acoustic pressure wave is then dissipated by the absorber to mitigate multiple acoustic reflections. The piezoelectric sensor voltage signal is increased with an amplifier and recorded with a storage oscilloscope. Simple time-of-flight in conjunction with the speed of sound in the relevant materials determines the distribution of charges. In contemporary systems, the dielectric is typically clamped by the cathode and anode (Arnaout et al., 2010; Griseri et al., 2004). Note that there is often a semiconducting film between the cathode and the sample that is not shown in Fig. 3.1; this layer is used to improve acoustic coupling in the sample stack (International Electrotechnical...
A high value resistor is used between the sample stack and the high voltage power supply to mitigate the high voltage pulse from effecting the power supply. Similarly, a capacitor is used between the high voltage pulse generator and the sample stack to mitigate the high voltage reaching the pulse generator. Further details of the PEA system components are given in Section 3.1.2 and Appendix A.

The spatial resolution of the PEA system is defined by the full width at half maximum (FWHM) of the interfacial peak (Galloy et al., 2016; International Electrotechnical Commission, 2012), and is approximately 10 µm in the current system. However, the resolution can be affected by sample characteristics such as dispersion and attenuation, as well as the sensor thickness, applied voltage pulse amplitude and duration, and the coupling media used to improve acoustic coupling of materials in the PEA system (Gibson et al., 2018). This is discussed further in Section 3.1.3.

The mathematical underpinnings to the method can be found in many places throughout the literature, such as in Maeno (1999). An overview is presented below.

The thickness of the samples are typically on the order of 10’s to 100’s of µm, with diameters on the order of a few cm. This means the method is effectively 1D through the thickness of the film, as the sample with a diameter to thickness aspect ratio of $>10^3 - 10^4$ can be considered essentially infinitely wide due to the width being much greater than the thickness. For simplification, the problem is considered to be 1D, as the thickness is much
smaller than the width.

When a short-duration high voltage pulse \( e_p(t) \) is applied across the dielectric film, a Maxwell stress \( f(x, t) \) is imposed at the interfaces of the electrodes and dielectrics, where \( x \) is position and \( t \) is time. This is due to the difference in dielectric constant of the electrode and sample materials. The stress at the interface of the ground electrode and the sample \( f(0, t) \) is then

\[
f(0, t) = \epsilon_0 \epsilon_r [E(0) + e_p(t)]^2 / 2, \tag{3.1}\]

where \( \epsilon_0 \) is the permittivity of free space, \( \epsilon_r \) is the relative permittivity of the sample, \( E(0) \) is the initial electric field due to the applied DC voltage \( V_{DC} \) from the high voltage power supply, and \( e_p(t) \) is the electric field due to the pulsed voltage \( V_p \). Note that \( E(0) = V_{DC}/d \) and \( e_p(t) = V_p(t)/d \), where \( d \) is the thickness of the sample.

For a well calibrated measurement, \( E(0) \gg e_p \) such that the pressure wave originating from the ground electrode/sample interface is given by

\[
p(0, t) = C \epsilon_0 \epsilon_r E(0)e_p(t), \tag{3.2}\]

where \( C \) is a constant that can be determined from the materials acoustic properties. This shows that the polarity and magnitude of the pressure wave is proportional to the electric field \( E(0) \). Similarly, the pressure wave produced at the interface between the excitation electrode and the sample (at \( x = d \)) is proportional to \( E(d) \). These pressure waves can then be detected by the piezoelectric sensor which produce voltages proportional to displacement as voltage signals; thus, \( E(0) \) and \( E(d) \) can be measured.

Similarly, the force on any embedded charge is given by determining the Coulomb force on the charge. This can be done simply by considering a sheet charge and noting that this can be extended to a sum of discreet sheet charges. For a discrete sheet charge embedded in the bulk of a dielectric sample, the force \( f(x_b, t) \) and resulting pressure wave \( p(x_b, t) \) at the location \( x_b \) is given by

\[
f(x_b, t) = \epsilon_0 \epsilon_r E(x_b)e_p(t) \tag{3.3}\]
and

\[ p(x_b, t) = C\varepsilon_0\varepsilon_r E(x_b)e_p(t). \] (3.4)

This allows for any \( E(x) \) to be measured for any embedded charge.

The embedded charge at any location can then be determined as the acoustic displacement or pressure is proportional to the amount of charge with the equations

\[ E(0) = \sigma/\varepsilon_0\varepsilon_r \] (3.5)

and

\[ p(x, t) = C\sigma(x)e_p(t), \] (3.6)

where \( \sigma(x) \) is the areal sheet charge density at position \( x \).

The details of signal calibration and deconvolution to remove system response are presented in the Chapter 4. Details of the spatial resolution of measurements and how to improve it are discussed in Section 3.1.3.

### 3.1.2 USU PEA System

The USU PEA system is outlined in Fig. 3.1 and presented in more detail in Fig. 3.2. The current USU PEA system works in ambient temperature and atmospheric pressure conditions and is considered a classic PEA system. Photos of the system are shown in Figs. 3.3 and 3.5.

The pulse generator used is a FPG 2-10PM5L high voltage pulse generator capable of varying the pulse width between 0.5–5 ns and pulse amplitude from 1–2 kV. More information on the pulse shape (see Fig. A.11) and frequency spectrum (see Fig. A.12) are provided in Appendix A.

This pulse is carried through a coaxial cable (Coax Bracke RG142B/U) with N-type connectors on both ends. The cable attaches to an 8 dB attenuator (Barth Electronics, Inc. high voltage 8 dB attenuator 142-NMFP-8 B) that is connected to an N-type tee. The attenuator is employed to reduce reflections within the cable between the high voltage pulse
generator and the sample. See Appendix A) for detailed information on multiple reflections in cables and impedance matching. Note that a long cable could also be used but results in dispersion and attenuation of the pulsed voltage, giving lower spatial resolution. The tee is terminated with a 50 Ω terminator (Bracke BM11073) for electrical impedance matching of the pulse, and also connected to the top of the PEA top electrode assembly. Within the PEA top electrode assembly is a 1 nF capacitor (C in Fig. 3.3) connected to the top electrode; this keeps the DC high voltage from reaching the pulse generator. Also connected in parallel at this point is a 1 MΩ current limiting resistor (R in Fig. 3.3) connecting the high voltage DC power supply (Glassman High Voltage Inc., FJ10R12) to the excitation electrode (cathode) via a RG-8U high voltage cable, with the purpose of isolating the DC high voltage system from the high frequency voltage pulse. The high voltage power supply is capable of applying a DC bias from 0–10 kV. Further details of this cabling, the HVDC power supply, and the electrical impedance matching are given in Appendix A. Voltage polarity reversal is possible by hard re-wiring of the HV power supply (not switchable). Photos of the PEA system are shown in Fig. 3.3.

The PEA sample stack (see Fig. 3.4) consists of the top/excitation electrode (Al), semiconducting film (500 µm PVC film; McMaster-Carr 87875K17), the sample, and bottom/grounded electrode (Al). There is a thin layer of light machine oil (Singer all purpose machine oil) on each surface for improved acoustic impedance matching. The semiconducting film is for improved acoustic impedance matching to avoid acoustic reflections from the interface at the top of the sample. The bottom grounded electrode is affixed directly to the PEA box via several bolts. The top PEA electrode assembly is also affixed to the PEA box for stability and to apply uniform controlled pressure to the sample stack. Sufficient pressure is necessary for good alignment and for the acoustic wave to propagate efficiently. A circular rubber gasket is used for alignment of the top electrode. The excitation (top) electrode is 0.5” in diameter and the ground electrode is 1.33” in diameter. Refer to Fig. 3.4. Appendix A discusses the surface roughness of the electrodes, which appears to have negligible effects on the PEA results.
On the other side of the bottom electrode is the sensor assembly. The polyvinylidene fluoride (PVDF) sensor (9 µm thick metallized PVDF, Measurement Specialties, Inc. DESC: 9UM/60D/Metalized, 400A Cu/150A Ni, P/N: 1003702-7) is metallized and also oiled with the light machine oil. A thick PVDF puck (0.12”), housed in an Al shield, serves as a backing material to absorb the acoustic pressure waves after passing through the piezoelectric sensor and mitigates multiple reflections that result in artifacts in the measured signal. The thick PVDF is pressed against the PVDF sensor with an o-ring beneath the puck for alignment. PVDF is used to enable a low acoustic impedance mismatch with the sensor, increasing the transmission of acoustic signals at the interface, and the large thickness (as compared to sensor and sample thicknesses) allows for large attenuation of the signal and moves any reflected waves far outside of the measured signal in time. The electrical connection to the PVDF sensor is made with a small spring-loaded gold plated contact. This gold contact is adhered inside a small hole made in the PVDF puck. The contact was spot welded to a thermocouple wire. This wire terminates in an SMA connector. Details of the PEA sensor assembly are shown in Fig. 3.5.

A coaxial cable with SMA connections then connects the PVDF sensor to the low noise, 50 Ω input impedance, 40 dB gain amplifier (Wenteq ABL0300-00-4030) that is located inside the heavily shielded and grounded PEA box, near the sensor to minimize electrical noise. Refer to Fig. 3.3(e).

The output signal from the pre-amp is fed into a 50 Ω impedance input of a high speed digital oscilloscope (Lecroy HD4104 HD4096, 2.5 GS/s, 4 channels). The signals from the sensor are then averaged and recorded by the digital oscilloscope. Typically measurements are made in sets of 5 with 1000 waves averaged per measurement. The pulse repetition rate is typically 1 kHz. The measurements are then saved on a computer via a LabVIEW program developed by BEI.

An in-depth characterization of each component is found in Appendix A. Spatial resolution of PEA measurements is discussed in the next section.
Figure 3.2: PEA system schematic. Detailed schematic of PEA system with legend and power supply diagram.
Figure 3.3: Photos of the PEA system at USU.
**Figure 3.4:** Representation of PEA sample stack. Coupling oil is shown in green and spring loaded contact is shown in gold.

**Figure 3.5:** Pictures of the PEA sensor assembly. The piezoelectric sensor is placed on top of the sensor backing (white puck). When assembled, the sensor is clamped between the backing and the ground plate.
3.1.3 Spatial Resolution of PEA Measurements

This section discusses the definition of the spatial resolution of PEA measurements, what has been done to improve the resolution, and what could still be done.

Defining Spatial Resolution

The spatial resolution of PEA measurements are defined as the FWHM of the leading interfacial peak (Galloy et al., 2016; Hole, 2008; International Electrotechnical Commission, 2012). This is due to the assumption that the charge at the interface is at the surface of the electrode/sample. It also assumes that both the surface of the electrodes and the sample are smooth, the electrodes and the sample are in full contact, and that the thickness of any coupling layers are negligible. These are relatively good assumptions as the electrodes and sample surfaces are smooth to within a few \( \mu \text{m} \) (see Appendix A.12, the coupling layers are \( \leq 1 \mu \text{m} \), and the sample is in sufficient contact due to the clamping forces. These assumptions assure that the spatial width of the interfacial charge distributions are small compared to the spatial resolution of the PEA system. This surface charge can then be assumed to be a delta function or sheet charge. Note that as the spatial resolution nears \( \leq 1 \mu \text{m} \), these assumptions will need to be revisited.

The PEA system response can then be determined directly from the measurement of this delta function or sheet charge distribution. This can be seen in Fig. 3.6 displaying an example PEA measurement that has been processed and calibrated. Other moments of the PEA signal can also be used to gain insight, such as the peak position and asymmetry. These other moments will be explored in Chapters 5 and 6.

The spatial resolution is determined primarily by (i) the HV pulse width, (ii) the thickness of the piezoelectric sensor, and (iii) the speed of sound in the sensor (Bernstein, 1991, 1992; Hole, 2008; International Electrotechnical Commission, 2012; Maeno and Fukunaga, 1996).

Another way to think about spatial resolution is positioning and discerning resolution (Hole, 2008). As discussed in Hole (2008), positioning resolution is the resolution of the position of a charge distribution when comparing between different measurements. Discerning
Figure 3.6: A representative PEA measurement. Plotted is a representative PEA measurement with dashed vertical lines corresponding to a representative figure of a sample with embedded negative charge and induced positive charge at the electrode interfaces. The bottom portion of the figure depicts the electrons being embedded in the material before introduction to the PEA system.

resolution is the resolution of discerning two different charge distributions contained within a single measurement. The theoretical resolution for each of these limits are \( \frac{0.98\nu_s\tau_{\text{rise}}}{\text{SNR}} \) and \( \frac{1.68\nu_s\tau_{\text{rise}}}{\sqrt{\text{SNR}}} \) for positioning and discerning resolution respectively, where \( \nu_s \) is the speed of sound in the material, \( \tau_{\text{rise}} \) is the rise time of the pulse (from 10% to 90%) and SNR is the signal-to-noise ratio (Hole, 2008). For further details refer to Hole (2008). The SNR is the maximum amplitude of the signal divided by the root-mean-square amplitude of the noise in the measured signal. Therefore, the spatial resolution is dependent upon the speed of sound in the material, the inverse of the SNR, and the rise time of the excitation pulse.

This is complicated by the fact that the signal must be measured by a piezoelectric
sensor. The piezoelectric sensor acts as a high pass filter for the signal, but it also has a high frequency limit related to the thickness of the sensor (Sensors, 2008). This can then be a limiting factor in the spatial resolution of a PEA system. A thinner sensor results in a higher limit for the frequency response, and therefore a higher limit for spatial resolution. Refer to Fig. A.27 in Appendix A for data demonstrating the effects of sensor thickness on the measured PEA signal and the frequency content of the signal.

**Improving Spatial Resolution**

The spatial resolution is affected by a myriad of sources from data processing to sample characteristics. The data processing aspects will be discussed in Chapter 4.

To improve the spatial resolution, the parameters outlined in the previous section must be addressed. That is, the factors that can be improved are the SNR, speed of sound, rise time of the pulse, and the thickness of the sensor. These will each be addressed individually. However, though the speed of sound can not be changed for a given material, the rest of the parameters provide insight into improving the spatial resolution. Note that the speed of sound variations due to thermal variations/gradients and sample inhomogeneities or sample batch differences do have an effect on the uncertainties.

The SNR is improved simply by taking several measurements and averaging them. The SNR is proportional to the square root of the number of measurements being averaged for standard counting statistics (Taylor, 1997). Therefore, there is a diminishing return with increased number of waves averaged. Typically, 1000 traces are averaged per measurement and 5 sets of measurements are taken. These 5 measurements are then averaged during processing to result in 5000 total traces averaged per processed and calibrated measurement. Filtering of the signal can also reduce the noise and increase SNR, but can decrease the frequency content of the signal resulting in decreased spatial resolution. This thus requires a balance between the two to be struck. This will be discussed further in Chapters 4 and 5.

The rise time of the pulse can be improved by using a faster pulse. Currently in the USU system the high voltage pulse generator is capable of providing a pulse with full width at half maximum of 0.5 ns to 5 ns. Using the narrowest pulse, this provides a lower SNR.
and is not currently a limiting factor of the resolution of the USU PEA system. Details on the effects of the pulse width and pulse amplitude on the measured PEA signal are given in Appendix A.3.

As discussed, another potential limiting factor of spatial resolution is the thickness of the piezoelectric sensor in the PEA system. The piezoelectric sensor typically used in PEA systems (and used in the USU PEA system) is made of polyvinylidene fluoride (PVDF). Commercially available PVDF sensors are available down to a few micron thickness, but are reliably available down to 9 µm. Thinner sensors can be purchased but have been unreliable when used in our lab. It is also possible to use other sensors or to manufacturer your own piezoelectric sensors down to 1 or 2 µm thickness through techniques such as electrospinning (Kalimuldina et al., 2020). Decreasing the thickness of the PVDF sensor would increase the spatial resolution of the USU PEA system and appears to be the current limiting factor.

3.2 Samples

The limitations and preparation of samples are described in this section.

3.2.1 Limitations

The samples used in the PEA system must meet certain requirements to obtain robust results. The sample must be a dielectric, as charge moves too quickly in conductors or semiconductors for useful measurements to be obtained. The samples must also be at least as wide (as large a diameter) as the excitation electrode, but ideally are as wide as the ground electrode to mitigate any arcing and sparking if a high voltage is applied during measurement. This is not as necessary for samples measured without any applied voltage.

The thickness of the samples are limited by acoustic attenuation to ~1 mm at the thickest and are limited at the thinnest by the spatial resolution and system response function, as discussed in the prior section. Obviously, the maximum sample thickness that can be measured with PEA depends on the acoustic attenuation of the material; the maximum sample thickness that can be measured increases for lower attenuating materials. Currently, the system response function is the limiting factor in the thickness of samples.
that can be measured in the USU PEA system, as is often the case. The system response function is removed from measured signal traces via deconvolution, but the sample must be thick enough to obtain this response function without overlapping with another signal. This will be discussed further in the Chapter 4. Samples with >75 µm thickness can easily be measured in the USU PEA system. If limited by spatial resolution only, measurements of samples <50 µm thick could be obtained.

The PEA signal can be affected by multiple acoustic reflections within either the sample or the sensor. This is mitigated in the sensor by using an absorber with the same or similar acoustic impedance. Reflections of acoustic signals at interfaces are minimized by matching acoustic impedance, just as optical reflections are minimized by matching the index of refraction as modeled by the Fresnel equations. In this case, the material on each side of the sensor/absorber interface is the same (PVDF); refer to Figs. 3.4 and 3.5. Note that the absorber is unpoled PVDF. Multiple acoustic reflections are mitigated in the sample by using a semiconducting layer between the excitation electrode and the sample (International Electrotechnical Commission, 2012), as well as using a good coupling media between the sample and the electrodes (Gibson et al., 2018). Ideal coupling materials have nearly the same acoustic impedance as the samples. However, this cannot be achieved for a single coupling material for different sample materials.

3.2.2 Preparation

Sample preparation involves cutting samples either by hand or with an automated sticker cutter, typically in 1.33” diameter discs with thicknesses between 100 - 250 µm. The samples are then cleaned by wiping down with methanol and then baked out. That is, they are placed in a vacuum chamber (typically < 10^{-5} Torr) at an elevated temperature (typically 50°C to 105°C) for >72 hrs to remove any water or other volatile compounds (Gillespie, 2022). This is important as water content can have a large effect on electrical properties such as the dark conductivity, up to several orders of magnitude (Wood et al., 2018), or to a lesser extent electrostatic field strength (DeWaal and Dennison, 2022).
3.2.3 Sample Characterization

Each sample is characterized in the lab with several different measurements. All samples are measured with a digital micrometer (Mitutoyo IP65; ±0.5 µm resolution), as the thickness is a key parameter used in data calibration for PEA measurements. Often, permittivity (Lee et al., 2021) and conductivity (Wood et al., 2018) are measured for a given dielectric material. Other measured parameters include RIC (Boman et al., 2021; Gillespie, 2022) and breakdown field strength (Andersen, 2018). However, these parameters are not obtained on the actual sample being measured with the PEA system but on a similar, typically thinner, sample. If these experiments have not been completed for a given material, then a value from the literature is obtained.

3.3 Charge Deposition

Charge can be injected into samples in several ways. The two primary charging methods employed with the USU PEA system are electrode injection and electron irradiation. Electrode injection occurs when a high voltage DC bias is applied to the sample in the PEA system (Dekany et al., 2013; Wood et al., 2018). Electron irradiation is a bit more complicated and will be the focus of the rest of this section (Dennison et al., 2015).

Results in this dissertation were obtained for samples irradiated in several different facilities at the Air Force Research Lab in Albuquerque, NM (Cooper and Hoffmann, 2015), the LAPLACE lab at University of Paul Sabatier III in Toulouse, France (Griseri et al., 2010), and in the USU lab.

The USU electron irradiation chamber utilizes a high energy electron gun (Staib EH-80), capable of electron energies from 10-80 keV and flux up to ~1 µA/cm², see Fig. 3.7. Typical irradiation is performed at room temperature, but it is possible to cool the sample with a closed-cycle He cryostat down to ~40 K (Dekany et al., 2014; Lee et al., 2021). The beam size is on the order of millimeters, but is spread further with the use of a graphitic amorphous carbon scattering foil. The typical foil used is 595 µg/cm² (ACF - Metals). Using this foil, an electron beam of ~6.4 cm FWHM can be produced.

The use of a scattering foil results in a reduced and broadened energy distribution,
Figure 3.7: USU electron irradiation chamber.

with larger effects at lower initial electron energies. This is relevant for a specific experiment in Chapter 6. The transmitted electron energy was simulated with CASINO v2.51 (Drouin et al., 2007). The resulting electron energy distributions after passing through the 595 µg/cm² carbon foil are presented in Fig. 3.8. The peaks of the transmitted energy distributions are plotted as a function of initial energy and fit with a linear model; refer to Fig. 3.9. This linear fit peak transmitted energy can be estimated using the equation

\[ E_{\text{transmitted}} = (1.0547 \pm 0.006) \cdot E_{\text{initial}} - (6.78 \pm 0.3) \quad \text{[units in keV].} \quad (3.7) \]

Note that this equation to estimate the landing energy of electrons incident on the sample after passing through the C foil is only accurate when the electron gun is set to energies \( \gtrsim 30 \) keV, as the fit was only for simulated irradiation with initial electron energies \( >30 \) keV. There is a noticeable deviation from the fit for an initial electron energy of 20 keV, as seen in Fig. 3.9.
Figure 3.8: Electrons transmitted through carbon foil. Normalized distributions of electron energies after passing through the 595 \( \mu \text{g/cm}^2 \) carbon foil as determined by CASINO simulations (Drouin et al., 2007) are plotted.

Figure 3.9: Peak transmitted electron energy. Transmitted electron distribution peaks as a function of initial electron energy. The red line shows a linear fit to all but the 20 keV data points.
CHAPTER 4
DATA PROCESSING

This section discusses processing of the data and their effects on the PEA measurement results.

There are various artifacts that can be left in the raw PEA data including multiple reflections (in sample or sensor), effects from sensor/amplifier frequency response, and high frequency noise (Mulla et al., 2020). These can be removed through processing the data (Pearson et al., 2017). Many of these effects are discussed in further detail in Chapter 3 and Appendix A. An overview of the data processing flow is given in Fig. 4.1, and is explained in detail below.

4.1 Filtering

To increase the SNR, many repeated measurements are averaged to mitigate the high frequency noise. Typically an equivalent of 5000 traces are averaged, as discussed in Chapter 3. To further increase the SNR, filtering of the signal can be done. The filtering method used for the data presented in this dissertation is a band-pass filter. The exact filter used is a modified Gaussian filter, \( \text{Filter}(f) \), applied to the FFT of the averaged data and represented by the equation,

\[
\text{Filter}(f) = e^{-\frac{4 \ln(2)}{BW^2} (f-f_0)^2} \sin\left(\frac{\pi f}{2f_0}\right),
\]

where \( f \) is the frequency, \( f_0 \) is the center frequency and \( BW \) is the frequency full width at half maximum or band width of the filter (Pearson et al., 2017). The Gaussian filter must be modified to ensure that it goes to zero as the frequency goes to zero. This is accomplished with a sine function.

To decide on the parameters used for the filter, a Fast Fourier Transform (FFT) is taken of the raw data [Fig. 4.2] and is plotted with the filter. This gives a visual representation
Figure 4.1: Outline of data processing for PEA. The (a) raw data undergoes an (b) FFT and is then (c) filtered. (d) A measurement with a small DC bias applied is used to obtain (e) a reference signal. The reference signal is used to (f) deconvolve and calibrate the data.

to be sure that the filter is not removing important frequency components of the data, as the goal is to remove the high frequency noise; refer to Fig. 4.2. Once the filter parameters are decided, the filter [Fig. 4.2] is applied in frequency space and the data is then inverse transformed back into real space [Fig. 4.1(c)]. It has been found that filtering only once after deconvolution provides the best final result. Deconvolution is described in the following section.

The parameters chosen for filtering can have a large effect on the resulting final PEA measurement. This is shown in Fig. 4.3 and discussed further in the following sections.
Other methods of filtering can also be employed, such as other shapes of filters than Gaussian or split spectrum processing (Pearson et al., 2017). This will not be explored here.

4.2 Deconvolution

The raw PEA data contains not just random noise but also various systematic artifacts associated with the PEA system. These artifacts can be grouped together and referred to as the response function of the PEA system. To remove the system response and be left with a representation of the embedded charge in the material, the data must be deconvolved.

The deconvolution process is as follows (Pearson et al., 2017). A reference signal is obtained to determine the system response function $h(t)$ of the PEA system. This is convolved with the signal from the embedded charge $x(t)$. The resulting measurement $y(t)$ can then be represented by

$$y(t) = h(t) * x(t) + n(t),$$

(4.2)

where $n(t)$ is the noise and the notation $h(t) * x(t)$ represents the convolution of a temporal function $h(t)$ with function $x(t)$. This can be written in frequency domain using the convolution theorem as

$$Y(\omega) = H(\omega)X(\omega) + N(\omega),$$

(4.3)
Figure 4.3: Effects of filtering on PEA results. Examples for the effects of filtering on PEA results are shown for a sample irradiated with monoenergetic electrons with deconvolved calibrated data when there is (a) no filter, (b) too much filtering, (c) approximately optimized filtering, (d) split spectrum processing, and (e) compared all together. Superimposed on each plot is an AF-NUMIT3 simulation. Plots reproduced from (Gibson et al., 2023).

where each function is the Fourier transform of its time domain counterpart. The desired function is \( x(t) \), so to solve for \( X(\omega) \) a Wiener deconvolution (Honarvar et al., 2004) can be used. This is mathematically expressed as

\[
X(\omega) = \frac{Y(\omega)H^*(\omega)}{|H(\omega)|^2 + Q_{\text{noise}}^2 |H(\omega)|_{\text{max}}^2},
\]

(4.4)

where the second term in the denominator is a measure of the noise, \( Q_{\text{noise}} \) is an adjustable parameter controlling the magnitude of noise suppression in the deconvolution process, and \( H^*(\omega) \) is the complex conjugate function of \( H(\omega) \) (Pearson et al., 2017).
There are several ways to obtain a reference signal. All reference signals are obtained from the electrode sample interface signal, that is from the leading interfacial peak. The typical way, referred to here as the classic method, is to obtain a reference signal on a pristine sample (no embedded charge) by applying a DC bias. The DC bias induced a displacement charge on the excitation electrode/sample interface (see Section 3.1.2). Assuming that this induced charge layer has negligible width, that charge has not migrated into the sample under the influence of the DC bias, and that there are not other embedded charge layers in the dielectric near this surface, then this interfacial charge peak is a response to a delta function charge layer. In essence, this signal is then the response function reference signal. This can either be done on the same sample that charge will be embedded into or on a pristine sample similar to a sample with charge already embedded. It is best practice to use the same sample and to not remove the sample from the PEA system after obtaining a reference signal, however this is not always possible.

Another method is to obtain the reference signal directly from a sample with embedded charge (Chen et al., 2006). This is only possible if the charge is essentially stationary on the time scale of the measurement. This method is the one typically employed in the USU PEA system. The method is as follows. The sample with embedded charge is measured without any DC bias applied. A measurement is then obtained with a small DC bias applied so as to not disturb the charge or breakdown the sample. The difference between the no DC bias and small DC bias measurements are obtained and used as the reference signal. This resulting reference signal is the PEA response to the small DC bias with the effects of the embedded charge removed.

With both methods, care must be taken not to inject any charge with the applied DC bias used to obtain a reference signal. It is worth noting that the effects of the pulsed voltage are taken care of with the alternative method but not in the classic method. This is described in detail in (Chen et al., 2006).

4.3 Calibration

The distance scale and the magnitude of the charge must both be calibrated.
4.3.1 Distance Scale

There are two ways the calibration of the distance scale can be done. The two methods
differ by the starting information used. Either the thickness of the sample or the speed of
sound in the sample are measured independently of the PEA measurement.

If you know the thickness of the sample by independent measurements, distance can
be calibrated by identifying the two electrode/sample interfacial peaks (in time). The
distance calibration can use different reference locations on these interfacial signals such as
the location of the maximum of the peaks or the rising edges. Typically the peaks of the
signals are used, as the peaks are a better gauge of position, as will be demonstrated in
Chapter 6, see Fig. 6.9. The speed of sound is then simply the ratio of the thickness divided
by the time difference between the two peaks.

Once the speed of sound is known, one simply multiplies each time step by the speed
of sound to convert to distance/thickness.

For further discussion on distance calibration and the uncertainty refer to Section 6.1.1
and Appendix B.2.

4.3.2 Charge Magnitude

A derivation of the calibration factor used to calibrate the charge magnitude is given
in this section. I have derived this calibration factor independently and the result agrees
with the literature (Maeno, 1999).

Consider a dielectric sample in a parallel plate capacitor configuration in the PEA
system (see Fig. 3.4). Start with the basic definitions of parallel plate capacitance solved
for the charge per unit area or areal charge density,

\[
\sigma = \frac{Q}{A} = \frac{\epsilon_r \epsilon_0 V_{DC}}{d} .
\]  

Here \( Q \) is total charge, \( A \) is the area of the sample/electrode/capacitor, \( \epsilon_r \) is the relative
permittivity of the sample, \( \epsilon_0 \) is the vacuum permittivity, \( V_{DC} \) is the applied DC voltage,
and \( d \) is the thickness of the sample.
Assume one has a calibrated signal $\rho(x)$ (volume charge density). This could then be integrated over the sample thickness to obtain the areal charge density. This should be equivalent to integrating the (deconvolved but not calibrated) measured voltage signal as a function of time (for the reference wave), multiplied by a calibration factor. This calibration factor $K$ would have units of $(C/m^2)(1/V_s)$ to convert the measured piezo-sensor voltage signal to the areal charge density, or from units of $V_s$ to $C/m^2$. If we set these two expressions equal,

$$\sigma_1 = \int_{\text{Calibrated Signal}} \rho(x)dx = K' \int_{\text{Raw Data}} V_{\text{meas}}(t)dt,$$  \hspace{1cm} (4.6)

where the integration is done over the first interfacial peak (of the reference wave). $\sigma_1$ is the total charge per area of the first interfacial peak. Then, with use of differentials we can show,

$$t = \frac{x}{v_s} \rightarrow dt = \frac{1}{v_s} dx,$$  \hspace{1cm} (4.7)

where $t$ is time, $x$ is distance, and $v_s$ is speed of sound in the sample. This can be inserted into the integral 4.6, resulting in the following equation,

$$\rho(x) = \frac{K}{v_s} V_{\text{meas}} = K' V_{\text{meas}}(t).$$  \hspace{1cm} (4.8)

$K'$ now has units of $(C/m^3)(1/V)$ to convert from a measured voltage signal to a volume charge distribution. The $1/v_s$ factor is simply to convert from time to distance, multiply by the speed of sound. To calibrate data that has not had the distance scale calibrated, the calibration factor used would be $K'$

Now, if the calibration for distance is done for the measurement first (as is typically the case), then Eq. 4.6 simply becomes,

$$\sigma_1 = \int_{\text{Calibrated Signal}} \rho(x)dx = K \int_{\text{Distance Scaled Data}} V_{\text{meas}} dx \rightarrow \rho(x) = KV_{\text{meas}},$$  \hspace{1cm} (4.9)
resulting in the removal of the dependence on the speed of sound in the final calibration factor. That is, $K$ is the final calibration factor instead of $K'$. 

To obtain the value of $K$, set it equal to the original equation for surface charge on a capacitor 4.5,

$$\sigma = \frac{Q}{A} = \frac{\varepsilon_r \varepsilon_0 V_{DC}}{d} = K \int_{\text{Distance Scaled Data}} V_{\text{meas}}(x) dx$$

(4.10)

$$\rightarrow K = \frac{\varepsilon_r \varepsilon_0 V_{DC}}{d \int_{\text{Distance Scaled Data}} V_{\text{meas}}(x) dx}.$$

Now the measured sensor voltage can simply be multiplied by the calibration factor $K$ to get the calibrated volume charge distribution. Remember that $K' = K/\nu$. This calibration factor should be consistent for a given sample or perhaps even a given material for a set of measurements to convert from measured voltage signal to calibrated volume charge density. However, this is not necessarily accurate depending on various system parameters. That is, if the system response is nonlinear in some way then the calibration factor will differ for different amounts of charge. This is explored in Appendix B.

To summarize, the measured voltage trace data are calibrated to be charge density by simply multiplying the deconvolved data by either $K'$ (if the data has not been calibrated for distance scale) or $K$ (if it has). Typically the distance scale is calibrated first and the calibration factor used is $K$. The parameters needed for calibration are the permittivity and thickness of the sample, applied voltage, and the integral of the leading interfacial peak of the reference signal. Note that calibration should be done after deconvolution and filtering of the data. A discussion of the calibration factor uncertainty is given at the end of Appendix B.1.

4.4 Attenuation and Dispersion

Attenuation and dispersion are an issue as well, as will be seen for measurements of PTFE in later chapters (e.g. Fig. 5.5). Attenuation decreases the amplitude of the signal and dispersion essentially means the material is acting like a low-pass filter for the acoustic
signal propagating through it. The effects are readily apparent in materials such as PTFE. Although, it is quite difficult to quantitatively determine and correct for the attenuation and dispersion effects due to complications with acoustic impedance matching, transmission, and reflection effects. This has not been addressed in the data analysis pipeline for this work. However, it can be done. Refer to (Vazquez et al., 1999). This will be left to future work. It may also be possible to extract the attenuation and dispersion effects from data obtained with an ultrasonic transducer and the PEA system, refer to Appendix A.7.
CHAPTER 5
VALIDATION

This section contains validation of the PEA method in the form of statistical analysis of data to investigate reproducibility, as well as comparison of data obtained with the USU PEA system with results from another PEA system.

The background, methodology, and data analysis processes have been presented for the PEA method. This section addresses the validation of the USU PEA system. The PEA system was validated by studying the reproducibility, uncertainty, and resolution of the measurements. This leads into a discussion of alternative definitions of spatial resolution for PEA measurements. This section ends with measurements validating the PEA method in general by comparing measurements of pristine and electron irradiated polymers that were obtained with both the USU PEA system and a PEA system at University of Paul Sabatier in Toulouse, France.

5.1 Reproducibility and Uncertainty

One of the most difficult parts of the signal processing and reproducibility of data analysis is the deconvolution to remove the system response from the measured signal. Several effects are observed in the final PEA charge profiles when varying deconvolution parameters (Gibson et al., 2023; Gupta and Reddy, 2016; Pearson et al., 2017), as discussed in Chapter 4, as well as the details of signal treatment, such as averaging and filtering (Gibson et al., 2023). However, the focus here is on the uncertainty due to experimental methods.

Presented here are repeated measurements of polyether-etherketone (PEEK) films with charge embedded in them with an electron beam to study the reproducibility of acquired data and determine the experimental uncertainties inherent in the PEA system. Samples of PEEK irradiated with slightly different doses of 50 keV monoenergetic electrons are
then compared to show the small differences in the peak position of the embedded charge distributions to demonstrate the uncertainty. Finally, the slow migration of the charge distributions over several months is monitored for some of the 50 keV irradiated PEEK samples.

Some results of these experiments were originally published in (Gibson et al., 2023) with the goal of comparing PEA measurements to AF-NUMIT3 (Beecken and Wallin, 2011) simulations; these results are presented in Chapter 6. New measurements were acquired for these samples to determine the reproducibility of the data acquired with of the PEA system, as well as to measure the slow migration of the embedded charge distribution as a demonstration of the precision in measuring the peak position.

5.1.1 Experimental Details

The PEEK samples used for this experiment (Victrex PEEK Film Technology—APTIV 1000 Series) are nominally 125 µm (1000-125G) and 250 µm (1000-250G) thick. The 125 µm thick sample of PEEK used for reproducibility measurements (ID K1A, following the naming convention as presented in Chapter 6) was irradiated with 80 keV monoenergetic electrons. The samples irradiated with 50 keV electrons have ID’s K4A and K6A for the 125 µm samples, and K4B and K6B for the 250 µm samples. Detailed material properties and experimental details can be found in Chapter 6.

Samples were baked out at 100°C under a vacuum of $<10^{-4}$ hPa for >72 hours before being irradiated to remove absorbed water and other volatile compounds. They were irradiated at room temperature under a vacuum pressure of $8\times10^{-7}$ Torr in the Jumbo chamber at the Air Force Research Lab (AFRL) in New Mexico (Cooper and Hoffmann, 2015; Gibson et al., 2023). Samples were shielded from ambient light, using only red light, and stored in Ar purged containers before and after irradiation. Samples were mounted to an Al carousel that rotated them into and out of the monoenergetic electron beam path during irradiation in an attempt to ensure even exposure. The flux (measured with an in situ Faraday cup) for 50 keV irradiation is plotted as a function of time in Fig. 5.1. For the first \(~10\) s there is a substantially higher flux, followed by a few steps of lowering flux until
Figure 5.1: Average flux incident on samples during the 50 keV irradiation. The absolute current density was determined by scaling the beam profile to the measured Faraday cup current at the center of the sample carousel and averaging over the path through the beam. The red dashed line indicates the mean flux during full irradiation interval.

...it stabilizes for the rest of the irradiation. The time averaged flux during the irradiation was 840 pA/cm² (red dashed line, Fig. 5.1), including the higher initial steps of flux. However, the carousel rotates at only 2 rpm. As the duration of the higher flux is ∼10 s, this means that only some of the samples received this higher dose. It is thought that the K4A and K6A samples received the higher dose and K4B and K6B did not, resulting in the differing peak deposition depths. Higher fluxes can potentially have a substantial effect on charge dynamics during charge deposition through enhanced radiation induced conductivity.

5.1.2 Determining Uncertainty

The experimental uncertainty in the PEA system was determined by repeated measurements of a 127±0.8 µm thick PEEK sample (K1A) with charge embedded via an 80 keV monoenergetic electron beam (Gibson et al., 2023). The single sample was measured many times while changing different measurement parameters such as the number of measurements averaged, pulse width and amplitude, and whether or not the sample was removed and replaced in the PEA fixture between measurements. A total of 25 measurements were obtained for each set of these parameters. Shown in Fig. 5.2 are the results for measurements using typical acquisition settings for our PEA system averaging 1000 waveforms,
with a pulse width of 0.5 ns and amplitude of \(\sim 300\) V, with removing the sample between measurements. Results for the data set investigating the effects of sample removal and replacement are summarized in Table 5.1 as well. The results for the variation of other parameters (half width half maximum, skewness, etc.) should be studied as well, but the focus of this work is on the precision of the position (peak) of the embedded charge distributions.

Several peak statistics were determined for the data sets, using analysis methods detailed in (Taylor, 1997). The standard deviation and standard error in the measured waveforms at each time step are shown in Fig. 5.2, along with other statistics determined for the set of measurements. 5.2(a) displays the averaged PEA measurements (black) with point-wise standard deviation (grey) and standard error (red) with all 25 measurements superimposed in various colors. Figure 5.2(b) determined the peak positions for each measurement and calculated the average and standard deviation of the peak positions for the interfacial peaks and the deposited charge distribution. Figure 5.2(c) plots the error (±standard deviation) for each peak to visualize the uncertainty in the peak positions. Figure 5.2(d) depicts the reduced chi-square for each measurement as compared to the average of the measurements. Figure 5.2(e) depicts the point-by-point reduced chi-square as compared to each point of the average of the measurements. Figure 5.2(f) shows a box plot of the point-by-point deviation from the average with a Gaussian fit superimposed, allowing the visualization of the distribution of deviations from the mean. Figure 5.2(e) plots the percent error (red, left axis) as compared to the normalized average amplitude for each point (black, right axis).

The distributions of the deviations from the mean for all time steps for the 25 waveforms were normally distributed; this confirms that the estimated uncertainties (mean of the standard deviations is \(\sim 2.4\)% or mean of the standard errors is \(\sim 0.5\)% of the amplitude of the leading interfacial peak were driven by the randomly distributed uncertainty of the instrumentation and analysis methods and not any systematic errors inherent in the PEA system. The reduced chi-squared values averaged for each of the 25 measured waveforms.
Figure 5.2: Reproducibility and uncertainty plots. Plotted are statistics determined for a set of 25 measurements where the sample was removed and replaced in the PEA system between measurements. Shown are the: (a) averaged PEA measurements (black) with point-wise standard deviation (grey) and standard error (red) with all 25 measurements superimposed in various colors, (b) tracked peak positions for each measurement with average (avg) and standard deviation (sd) listed, (c) error in the peak positions, (d) reduced chi-square for each wave as compared to the average, (e) reduced chi-square for each point as compared to the average, (f) a box plot of the point-by-point deviation from the average with a Gaussian fit superimposed, and (e) the percent error (red, left axis) as compared to the normalized average amplitude for each point (black, right axis).
Table 5.1: Uncertainties of peak positions. Uncertainties listed for locations of interfacial and embedded charge peaks for reproducibility measurements.

<table>
<thead>
<tr>
<th>Uncertainty in Peak Position</th>
<th>Peaks</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Leading (Left) Interface</td>
</tr>
<tr>
<td>Without Sample Removal</td>
<td>59.4 ps (150 nm)</td>
</tr>
<tr>
<td>Sample Removal and Replacement</td>
<td>77.7 ps (197 nm)</td>
</tr>
</tbody>
</table>

was 0.97±0.09, confirming the use of the standard deviation or standard error of the 25 repeated waveforms at each time step as an appropriate measure of the precision.

The reproducibility of the leading interface (left), trailing interface (right), and embedded charge distribution peak times were also determined, as listed in Table 5.1. Note that the differences in the leading peak times are negligible when raw waveform time stamps are adjusted to align these peaks, as is typically done (no alignment of waveform data for Table 5.1 results). For data obtained without removing the sample from the PEA system, the variations (standard deviations) of the embedded charge, leading interface, and trailing interface peak times are <20% of the 0.4 ns time interval between waveform points set by the 2.5 GS/s capacity of the 1 GHz digital oscilloscope (Lecroy, Model HDO4104) used in the PEA system. Using the measured speed of sound in PEEK of 2531±17 m/s (±0.7%), this translates to a precision of ≤0.2 µm for the positions of each peak. This speed of sound was determined with a measured average of the K1A sample thickness (127±0.8 µm or ±0.6%) divided by the time separation of the leading and trailing interface peaks for the 25 waveforms, with uncertainties determined largely by ±0.6% uncertainties in the film thickness, with smaller contributions from the precision of peak position and temperature variations in the speed of sound totaling approximately 0.4%. The uncertainty of peak times increased slightly (still ≤20% of the 0.4 ns oscilloscope sample spacing) when the sample was removed and replaced between each of 25 measurements, as compared to not removing the sample from the PEA system for 25 measurements; refer to Table 5.1. This suggests that neither sample positioning, sample clamping, nor thickness of the interfacial coupling
layers had significant impact on the measured peak position results.

Note that the uncertainties as a function of time are not addressed. That is, the data compared were taken on the same day under the same conditions. It is possible that alignment of the data is necessary over longer time frames such as days, weeks, months, or years. This was found to be the case in the data presented for the slow migration of charge distributions presented in the following section. However, there were slight modifications to the PEA set up as well (different cables, electrode plates, etc.) that could have been the cause of the slight shifts of the PEA data in time which resulted in the necessity of aligning the data during analysis and comparison of the data. Differences in the pulse width and amplitude can also cause changes in the measurement time of the PEA data; refer to Appendix A. To be clear, what is being discussed is the effect on the timing of the measurement. Other changes in the timing of the measurement could also result in changes in the triggering settings. However, it should not be an issue to align measurements by the interfacial peaks during processing as the peak provides an accurate determination of the location of the interfaces and charge distributions. This is shown for the novel method presented in Chapter 6.

5.1.3 Testing Uncertainty: Differentiation of Charge Distributions

To test the uncertainty of the PEA system, measurements of 50 keV irradiated PEEK samples were obtained.

Charge profiles of each of the four PEEK samples irradiated with 50 keV monoenergetic electrons are compared in Fig. 5.3. Note that the interfacial peaks corresponding to the irradiated surface of the sample for each waveform were aligned to accurately compare deposited charge distributions. Embedded charge peak positions for both the 125 µm and 250 µm samples are shown for the electron irradiated interfaces at the PEA high voltage electrode [● in Fig. 5.3(b) inset] and the grounded PEA sensor electrode [► in inset], respectively. The embedded charge peak depth is 32.9±0.3 µm for the 125 µm thick samples and 29.4±0.2 µm for the 250 µm thick samples. The 3.5±0.4 µm shift in the embedded charge peak for different sample thicknesses is an order of magnitude larger than the spread
in peak positions at fixed thickness as well as the precision of the peak positions. However, this shift is only 35% of the ∼10 µm spatial resolution of the PEA system, traditionally defined as the FWHM of the leading interfacial peak. There are smaller differences in the embedded charge peak depths when comparing orientations of the electron deposition interface, on the order of the precision of the peak positions as well as the uncertainty in the measured sample thickness. Analysis of additional moments of these interfacial peaks and deposited charge distributions are presented in Chapter 6. Note that calibration and signal processing can potentially add uncertainty to the final signal processed PEA charge profiles, as has been discussed in Chapter 4.

5.1.4 Testing Uncertainty: Long Time Charge Migration

Deposited charge can migrate under the influence of self repulsion, occurring even without grounded or biased electrodes. However, for these PEEK samples, little to no charge migration was expected, given its very low dark current conductivity \( \sigma_{DC} = (7 \pm 3) \cdot 10^{-21} (\Omega \cdot \text{cm})^{-1} \) (Wood et al., 2018) and long decay time \( \tau_{\text{decay}} \equiv \frac{\epsilon_r}{\sigma_{DC}} \) of 440±200 days with \( \epsilon_r = 3.0 \pm 0.2 \) (Lee et al., 2021). The decay time roughly corresponds to the time it would take for the charge to dissipate if a sample surface was grounded. This was not the case during these tests as the samples were stored at room temperature without any biased or grounded electrodes; hence, much longer charge dissipation times are expected. PEA measurements of the charge profiles and charge peak depth for the K4A and K6A 125 µm samples were taken on July 7th, 2021, and again on February 22nd, 2022, a separation of 229 days. Figure 5.4 plots the results of the slow migration tests. The plots shown are raw data aside from being rescaled for distance using the measured thickness of the samples. Plots for measurements in both sample orientations in the PEA fixture (electron irradiated interfaces at the PEA high voltage electrode [▶ in Fig. 5.4(a) inset] and the grounded PEA sensor electrode [◀ in inset],) are presented to demonstrate close agreement for the two orientations.

The embedded charge peak positions were observed to have migrated slightly deeper into the samples. The distributions shifted by 0.8±0.2 µm for K4A and 0.65±0.2 µm for
Figure 5.3: PEA measurements at differing doses. PEA charge profiles of four PEEK samples irradiated with 50 keV monoenergetic electrons. Both the 125 µm (K4A and K6A) and 250 µm (K4B and K6B) samples are plotted. Plotted are measurements with the apparent incident electron direction (a) from the left and (b) from the right. Embedded charge peak positions for both the 125 µm and 250 µm samples are shown for the electron irradiated interfaces at the PEA high voltage electrode (incident right) [● in inset] and the grounded PEA sensor electrode (incident left) [▲ in inset], respectively. Measurements were aligned to the peak of the irradiated side of the sample to accurately compare the deposited charge distributions.
K6A, \( \sim 2\% \) of the deposition depth for the embedded charge peaks. These shifts in the embedded charge peaks are similar to the spread in peak positions at fixed thickness and a few times larger than the precision of the peak positions. However, once again these shifts are more than an order of magnitude smaller than the \( \sim 10 \mu m \) defined by the FWHM of the leading interfacial peak.

### 5.1.5 Conclusions and Overview of PEA Uncertainty

The experimental uncertainties were determined with the repeated measurements of sample K1A. There were only small variations in the amplitude when the sample was not removed between measurements. The experimental uncertainty was determined to be random by showing the distribution of the deviations about the mean was normally distributed and the average reduced chi-squared value is \( \sim 1 \) using the standard deviation at each time step as an estimate of the uncertainty. From these results, the error for repeated PEA waveform measurements is primarily random with some small systematic errors introduced by positioning, clamping, etc.

The position of the peaks in the PEA measurements were determined to have an uncertainty of \( \leq 0.2 \mu m \). These data show the high precision possible for PEA measurements of the positions of charge distribution peaks and peak migration. Note that the typical quoted spatial resolution defined by the FWHM of the leading interfacial peak is \( \sim 10 \mu m \) for this and similar PEA systems, a factor of \( \sim 50 \) larger than the precision of the peak position. The FWHM-based spatial resolution is appropriate as a measure of the separation required to resolve two charge distributions (i.e., two embedded charge distributions or an interfacial distribution and an embedded distribution). It is clear from these differences that it is imperative to consider what definition of spatial resolution is best suited for the specific question posed in each test set.

A first test of the high precision in peak position of the deposited charge distribution was obtained by analysis of measurements of PEEK samples with differing doses of 50 keV electrons. The difference between the two sets of samples was quite evident, with an appreciable and consistent difference of \( 3.5 \pm 0.4 \mu m \) between the peak depths of the
Figure 5.4: Slow migration of the charge distributions in samples K4A and K6A. Plotted are the raw PEA measurements (aside from distance scaling) with electrons (a) incident from the left and (b) incident from the right. An original measurement was taken on July 7th, 2021 and compared to a measurement taken on February 22nd, 2022. Measurements were aligned to the peak of the irradiated side of the sample to accurately compare the deposited charge distributions. An inset in (a) highlights the change in peak position with time. Note that the system response has not been removed and there is not a positive embedded charge distribution.

deposited charge distributions. This is well above the uncertainty in peak position of ±0.2 \( \mu \text{m} \).
A second test of the high precision in peak positions was obtained by measuring the slow migration of the charge distribution in very low conductivity PEEK samples K4A and K6A over several months. An apparent migration of the charge of $0.8\pm0.2\ \mu m$ and $0.65\pm0.2\ \mu m$ were observed for K4A and K6A, respectively. This is still above the experimental uncertainty in peak position, but near the present resolution limits.

However, there are still other factors to consider that can affect the uncertainty of the measurement such as alignment of the measurements (one 0.4 ns time step corresponds to $\sim1\ \mu m$ in PEEK). There is also the question of whether to align waveforms to the leading interfacial peak or if aligning to the rising edge is more accurate. Alignment is often only necessary for measurements not carried out on the same day. Further investigation of the uncertainties of the PEA system over time is also desired. It would be helpful to have more than two data sets for charge migration studies to confirm there is in fact a consistent evolution of embedded charge peak position. Further work is needed to investigate this issue.

Nonetheless, the presented PEA measurements demonstrate the experimental uncertainty in peak position of the system. This study shows that PEA measurements can be useful for differentiating positions of single peaked charge distributions between measurements with spatial resolution of peak positions on the order of $\leq1\ \mu m$ and indicate that better precision may be obtained with an oscilloscope with a faster data acquisition rate.

The appropriate choice of measures of the system spatial resolution have been clarified in the previous section. The results have highlighted the question as to whether the spatial resolution of PEA measurements should be defined solely in terms of the FWHM of the leading interfacial peak, as the peak positions can be determined with much greater precision. The relevant “spatial resolution” of PEA measurements change based on the context. For example, the relevant spatial resolution is clearly different for differentiating single peaked distributions of charge between different measurements, resolving multiple peaked embedded charge distributions, and resolving charge distributions near surface interfaces.
5.2 Alternative View of Spatial Resolution

As discussed in Chapter 4, the spatial resolution of PEA measurements are typically defined to be the full-width-at-half-maximum (FWHM) of the leading interfacial peak of the PEA waveform (Galloy et al., 2016), which for typical PEA systems are $\sim 10 \mu m$ (Dennison and Pearson, 2013). However, the FWHM is just one moment of the charge distribution. There are also the magnitude, peak position, and skewness of the charge distribution (Gibson et al., 2023). It is relatively well known that the charge distribution typically has quite a large uncertainty in the absolute determination of the magnitude, particularly for samples with charge already embedded. This is due to the difficulty in calibration of the charge magnitude. There has been much work to increase the accuracy of the calibration by taking into account such effects as those from the exciting pulse, coupling layers, interfaces, electrode materials and electrode morphology (Arnaout et al., 2011; Chen et al., 2006; Huang et al., 2014; Maeno, 1999; Maeno and Fukunaga, 1996; Mier-Escurra et al., 2018; Mulla et al., 2020).

The measured peak position can be determined to much higher precision than the spatial resolution, as defined by the FWHM. High precision determination of the peak position of the charge distribution is important for many applications such as studies of low-energy electron irradiation of polymers (important for spacecraft charging) (Gibson and Dennison, 2022a), radiation induced conductivity (and delayed radiation induced conductivity) (Boman et al., 2021), and slow migration of charge in highly insulating materials.

The results and discussion have also highlighted a few areas in need of further inquiry, such as the possible variation of charge distribution position measurements when taken at different times and proper alignment of waveform profiles for comparison and will be explored further in Chapter 6. Further work is needed to determine the extent of the uncertainty in the magnitude and other moments of the measured charge distributions, as well as to study the uncertainties introduced through signal processing.

5.3 Comparison of Data

To further validate the PEA measurements, an experiment was devised to compare
measurements using the USU PEA system with a results from a PEA system in Toulouse, France. Both PEA systems are classic PEA systems.

There are many papers in the literature outlining the PEA method, calibration, and data processing techniques (Anderson, 1958; Dennison and Pearson, 2013; Hussaini et al., 2015; Imburgia et al., 2016; Maeno, 1999; Maeno and Fukunaga, 1996; Pearson et al., 2017; Rizzo et al., 2019). There is a published standard for calibration of PEA measurements (International Electrotechnical Commission, 2012) that has provided a standard procedure for calibrating, processing, and analyzing PEA data, which provides representative results from several different groups for comparison (Anderson, 1958). However, the results compared in this standard are only for uncharged samples with a DC bias applied. While this is an important first step, a comparison of PEA measurements of samples with embedded charge is now needed. It should be noted that the International Electrotechnical Standard (IEC) International Electrotechnical Commission (2012) is not well known among the PEA community. There appear to be no other published attempts to directly compare PEA data of any kind between different groups. There is also a lack of comparison of PEA measurements in different systems of samples with embedded charge in the literature.

The main purpose of the study presented in this section is to validate results of the PEA method for dielectric materials with embedded charge by comparing results of two different PEA systems. The first PEA system is located at Utah State University (USU) in Logan, UT, USA. The other system is located at Université Paul Sabatier (UPS) in Toulouse, France. The samples chosen for this study are polytetrafluoroethylene (PTFE) and polyether-etherketone (PEEK), as their bulk conductivities are so low that the embedded charge can be considered stationary on the timescale of the experiment (Gillespie, 2022; Wood et al., 2018), even considering the prolonged travel time of the samples between the two laboratories located on different continents. The charge was embedded in these samples with a monoenergetic electron beam.
5.3.1 Experimental Details

The sample and irradiation details for PEEK and PTFE samples used in this study are described below.

Sample Details

The samples irradiated with 60 and 70 keV electrons used in this study are PEEK from APTIV Victrex PEEK Film Technology (1000-250G) and PTFE from McMaster-Carr (MCVS005X12X3). They are nominally 250 µm thick. The PTFE (GoodFellow product of DuPont) used for the DC bias measurements and irradiated with 50 keV electrons are nominally 200 µm thick. The speeds of sound were determined using respective PEA measurements as the ratio of the measured thickness and the time difference between the two interfacial peaks.

Electron Irradiation

Samples were irradiated with 50 keV, 60 keV, or 70 keV electrons with a flux of \( \sim 1 \text{ nA/cm}^2 \) for 10 min. The samples were irradiated at UPS in the MATSPACE chamber (Griseri et al., 2010) with a 100 keV electron gun (built at LAPLACE) at room temperature and a vacuum level of \(< 10^{-5}\) mbar. Irradiated PEEK samples were exposed only to darkness or red light to avoid any potential photo-induced charging/discharging. This is potentially important for PEEK samples as they have an optical bandgap in the visible range of \( \sim 3.1 \text{ eV} \) (Mackova et al., 2014).

5.3.2 Comparing PEA Systems

Although the USU PEA system has been described elsewhere in this dissertation (see Section 3.1.2), an overview is presented here to help provide a direct comparison to the UPS PEA system. A highlight of the key features with similarities and differences of the two PEA systems is given in Table 5.2.
USU PEA System

In this study, the USU PEA system used an excitation pulse of 5 ns width and \(~300\) V amplitude (FID GmbH Model FPG 2-10PM5L High Voltage Pulse Generator), representative pulse can be seen in Fig. A.11. The pulse amplitude is initially 1 kV, but is reduced to \(~300\) V with an 8 dB attenuator (Barth Electronics, Inc. High Voltage 8 dB Attenuator 142-NMFP-8 B). The pulse is electrically impedance matched to the exciting electrode with a 50 Ω to ground terminator, refer to schematic in Fig. 3.2. A semiconducting material (PVC McMaster-Carr 87875K17, 500 µm thick) is used to acoustically impedance match the sample to the excitation electrode. Both the excitation electrode and ground electrode are made of Al. The piezoelectric sensor used is a metallized PVDF sensor, nominally 9 µm thick (Measurement Specialties, Inc. DESC: 9UM/60DMetalized, 400A Cu/150A Ni, P/N: 1003702-7). A backing of PVDF was used to mitigate multiple reflections in the sensor. The signal is amplified with a low noise, 50 Ω impedance, broadband (9 kHz to 3 GHz), 40 dB gain amplifier (Wenteq ABL0300-00-4030,). The semiconducting layer, sample, and piezoelectric sensor were lightly oiled with light machine oil (Singer All Purpose Machine Oil) to improve acoustic impedance matching (Gibson et al., 2018).

For each measurement, 1000 traces were averaged and recorded five times with an oscilloscope (LeCroy HD4104 HD4096 2.5 GS/s) for each measurement and then averaged together for an effective total of 5000 averaged traces. The reference signal for deconvolution for charged samples was determined by applying a small 1 kV DC bias (Chen et al., 2006; Gibson et al., 2023). The signal was filtered with modified Gaussian filter to remove high frequency noise and parameters were adjusted manually to attempt an optimal final charge distribution measurement (Gibson et al., 2023; Gupta and Reddy, 2016; International Electrotechnical Commission, 2012; Pearson et al., 2017).

UPS PEA System

In this study, the UPS PEA system used an exciting pulse of \(~5\) ns width and \(~250\) V amplitude from a pulse generator (FID GmbH Model FPG 2-50NK). It should be noted that the pulse from this generator was less than ideal, with extraneous lower frequency
negative voltage features before and after the large positive fast pulsed voltage. The pulse
travelled along a 25 m long cable that caused attenuation and dispersion of the pulse before
being applied to the sample in the PEA system. The pulse is matched to the sample
with a 50 Ω resistor to ground. A semiconducting material (carbon black prepared from
granules of low density polyethylene loaded with carbon by hot pressing to a thickness of
~650 µm) was used to improve acoustic coupling between the exciting electrode and the
sample. The sample and semiconducting material were lightly oiled with silicone oil to
improve acoustic impedance matching. Both the pulsing and ground electrodes are made
of Al. The piezoelectric sensor used is a metallized PVDF sensor, nominally 9 µm thick
(Piezo Tech). A backing of PVDF was used to mitigate multiple reflections in the sensor.
The signal is amplified by two 20 dB, low noise, 50 Ω impedance, broadband (100 kHz - 1
GHz), amplifiers (Mini Circuits, ZFL-1000LN+), for a total of 40 dB amplification.

Measurements of 8000 traces were averaged and recorded with an oscilloscope (LeCroy
WaveRunner 204Xi, 10 GS/s). The reference signal for the deconvolution for the charged
sample measurements was determined by measuring a pristine sample of the same material
with an applied voltage of 2 kV DC bias. UPS data was filtered with a modified Gaussian
filter and parameters are adjusted manually to attempt an optimal final result, similar to
USU.

The PEA systems are largely the same with the primary differences being a different
method of obtaining calibration signals for samples with embedded charge and the excitation
pulse characteristics, see Table 5.2.

5.3.3 Comparing PEA Results

The results of uncharged PTFE with a 2 kV DC bias applied are presented first to show
agreement between the two systems at the most basic level. This is followed by results for
PTFE and PEEK samples irradiated with monoenergetic electrons.

Pristine Sample

These tests were on the exact same samples, transported between laboratories. The
Table 5.2: Comparison of PEA systems. Differences are color coded where green indicates similar/the same, yellow indicates a slight deviation, and red indicates a large difference in the systems.

<table>
<thead>
<tr>
<th>Utah State University PEA System</th>
<th>Université Paul Sabatier PEA System</th>
</tr>
</thead>
<tbody>
<tr>
<td>• Ambient, parallel plate capacitor</td>
<td>• Ambient, parallel plate capacitor</td>
</tr>
<tr>
<td>• ~5 ns, ~300 V exciting pulse</td>
<td>• ~5 ns, ~250 V exciting pulse</td>
</tr>
<tr>
<td>• 1 kV pulse through 8 dB attenuator and short cable</td>
<td>• Low frequency artifacts, pulse traveled along 25 m cable</td>
</tr>
<tr>
<td>• 50 Ω impedance match</td>
<td>• 50 Ω impedance match</td>
</tr>
<tr>
<td>• PVC semiconductor film</td>
<td>• Carbon black semiconductor film</td>
</tr>
<tr>
<td>• Al electrodes</td>
<td>• Al electrodes</td>
</tr>
<tr>
<td>• 9 μm PVDF piezoelectric sensor</td>
<td>• 9 μm PVDF piezoelectric sensor</td>
</tr>
<tr>
<td>• 40 dB gain (1 amplifier)</td>
<td>• 40 dB gain (2 amplifiers)</td>
</tr>
<tr>
<td>• Light machine oil (coupling media)</td>
<td>• Silicone oil (coupling media)</td>
</tr>
<tr>
<td>• 5000 traces averaged/measurement</td>
<td>• 8000 traces averaged/measurement</td>
</tr>
<tr>
<td>• Data is filtered with modified Gaussian</td>
<td>• Data is filtered with modified Gaussian</td>
</tr>
<tr>
<td>• Reference obtained with charged sample</td>
<td>• Reference obtained with pristine sample</td>
</tr>
</tbody>
</table>

results of the DC bias measurements from both the USU and UPS PEA systems are shown in Fig. 5.5. Qualitatively, the plots are very similar, with very little variation for the pulsing electrode interfacial peak. By definition, from calibration, the areas under the curve for the leading interfacial peaks are identical for both measurements and give the same internal electric field for both measurements (refer to Fig. 5.5 inset). The leading interfacial peak is slightly narrower for the USU data than for the UPS data due to the differing resolution of the two PEA systems as well as contributions from the differences in the signal processing. Note that the measurements could likely be forced to agree more qualitatively with modification of filtering parameters, such that the curves lie on top of each other, but no attempt to match the data was made. This is because the point of the study is to compare results from different PEA systems as they are produced in different laboratories. The small differences in the bulk appear to be due to signal processing and deconvolution. The interfacial peak from the excitation electrode (right) is in excellent agreement as the spatial resolution is limited by the dispersion of the acoustic signal in the PTFE material rather than the respective PEA system or data processing, providing evidence to the above argument that the measurements could be forced to agree more qualitatively with a different (matching) choice of filtering parameters.
Figure 5.5: PEA measurements of 200 µm thick PTFE with 2 kV DC bias. Inset depicts internal electric field calculated from the measured charge.

Samples With Embedded Charge

The results of PTFE irradiated with 50 keV, 60 keV, and 70 keV electrons are presented in different measurement orientations in Figs. 5.6, 5.7, and 5.9, respectively. Results for PEEK irradiated with 60 keV and 70 keV electrons are presented in Figs. 5.8 and 5.10, respectively.

The overall shape and features of the internal charge distributions are clearly in good agreement for all irradiated samples presented. However, the magnitude of charge measured, both at the interfaces and embedded in the material, are in disagreement between the two systems. This difference is particularly large for the 70 keV irradiated PEEK in Fig. 5.9, with approximately a factor of 2 difference in the measured amplitude of the charge magnitude. Recall that the USU PEA measurements are calibrated using a measurement of the charged sample with an applied voltage, whereas the UPS calibration is made with a measurement of a biased pristine sample prior to irradiation. The USU data for 50 keV irradiated PTFE was recalibrated with the 2 kV DC bias measurements taken on the pristine PTFE sample in an attempt to more accurately compare to the UPS data. This resulted in a difference of only ~5-10% in the measured amplitude of the charge magnitude for the two calibration methods applied to the USU data, implying this is not enough to account for the discrepancy between USU and UPS measurements.
There is also slight disagreement in the distance between the interfacial charge peaks. This is due to a difference in signal processing, where the UPS PEA measurements use a pristine sample of known thickness (or the same sample before being irradiated) as a reference for charge magnitude as well as for distance scaling (by measuring the speed of sound), whereas the USU PEA measurements assume the distance between the interfacial peaks of the irradiated samples are equal to the measured thickness of the sample in determining the speed of sound for the specific sample under test. Note that the discrepancy between measurements is on the order of a few percent, which is similar to the uncertainty in the speed of sound in the material, driven largely by uncertainties and inconsistencies in the sample thickness and in variations in the speed of sound due to uncertainties in the sample temperature (Gibson et al., 2023), see Section 5.1.2 for more details.

The discrepancies in charge magnitude for PTFE irradiated with 50 keV electrons (refer to Fig. 5.6) appear to be due to differences in spatial resolution of the USU and UPS systems, causing distributions of positive and negative charge to overlap. The PEA system can only measure net charge, so overlapping distributions appear to have counterbalanced charge (zero measured charge) if positive and negative charge distributions are too close together. While there appears to be better agreement seen in Fig. 5.6(b), this is due to the same effect for overlapping distributions. Because there are large effects from attenuation and dispersion in PTFE, the details of the underlying charge distributions are obfuscated and the resolution of the PEA measurements are no longer limited by system parameters and processing, but rather by the attenuation and dispersion in the material.

Discussion of Results

It seems clear that any DC bias measurements of uncharged samples compared with any PEA system will be in good agreement, as long as their data are calibrated the same way. This is because they are in agreement by definition for the conventional method of calibration, see Section 4.3.2 and the IEC standard International Electrotechnical Commission (2012). When comparisons of charged samples are made, it is clear from the results presented here that there are discrepancies that need to be investigated.
The differences in the qualitative shape of the PEA measurements are largely explained by differences in the spatial resolution of the respective PEA systems. For a direct comparison of measurements, it is best to have similar spatial resolution, or to filter the data so that the resulting measurement is of the same spatial resolution. This is demonstrated in Fig. 5.6(b) for 50 keV irradiated PTFE as the material itself acts as a low pass filter of the acoustic signal.

The differences in thickness or distance scaling of the material are due to the different methods of calibration. The signal is calibrated by multiplying the time of each data point by the speed of sound. There is an inherent uncertainty in the speed of sound from the uncertainties of the thickness and temperature of the sample, the clamping force of the electrodes, and the definition of the position of the interfaces in the data (e.g., peak position or rising edge). This uncertainty is on the order of a few percent of the thickness of the sample. Considering this uncertainty, the USU and UPS data are in excellent agreement. For best comparison, the distance scale should be determined with the same method or, at the very least, the irradiated interfaces should be aligned when differing data is plotted together and the difference in overall thickness should be considered in the uncertainty.

The discrepancy in charge magnitude is harder to interpret. The 50 keV irradiated PTFE data appears to be in good agreement, while the 70 keV irradiated PEEK is in much poorer agreement. The other measurements also show discrepancies in the charge magnitude of similar magnitudes. There does not appear to be a correlation between PEA system used or material measured and the discrepancy in charge magnitude. For example, it was not found that the USU PEA system always overestimated charge magnitude or PTFE always appeared to have less charge magnitude using the UPS PEA system. The differences are likely due to interfacial effects, as the signal used for calibration is from charge at the interface but the measured charge is in the bulk of the material. Differences in coupling oil, applied pressure, surface roughness, and electrode properties could all cause differences in the measured calibration signal. An ideal calibration for PEA measurements would involve measuring an embedded charge distribution with known charge magnitude density.
and position, using the same material being tested. The charge distribution would be in a relatively thick sample, so that measuring in two orientations could give you information about the attenuation and dispersion effects of the material. This could potentially be done with a reasonable amount of uncertainty by irradiating a sample with a known energy beam and measuring the surface potential to determine the amount of charge embedded. A relatively low flux, fluence, and incident energy would be best to attempt to obtain a charge distribution that is easy to measure and understand. Perhaps this could be aided with the use of modeling codes such as AF-NUMIT3 (Beecken et al., 2015) or other deep dielectric charging simulations (Agostinelli et al., 2003; Rodgers et al., 1998a).

To summarize this section, measurements with two different ambient PEA systems at USU and UPS were compared for PTFE with a DC bias and PEEK and PTFE with charge embedded from irradiation with monoenergetic electrons. The results show that the overall shape of the curves are in excellent agreement when the spatial resolution is considered. The distance scale is in agreement on the order of the uncertainty, but there is a discrepancy in the charge magnitude of the measurements. Some of this discrepancy can be understood through differences in the PEA resolution; as positive and negative charge distributions overlap, the PEA measurements only capture the net charge distributions. However, this is not sufficient to explain the large discrepancies observed in charge magnitude. It is likely that this is due to the method of calibration employed for PEA measurements. While the most frequently employed calibration method is adequate for relative measurements of charge magnitude, alternative methods of calibration should be explored to reduce the uncertainty in the absolute charge magnitude of the PEA measurements.

Future work is needed to investigate these discrepancies further and to explore alternative calibration methods.
Figure 5.6: PTFE irradiated with 50 keV monoenergetic electrons. PEA measurements of 200 µm thick PTFE are plotted with incident electron orientation from the (a) left and (b) right. Superimposed in green are USU data calibrated with the classic method in attempt to be more similar to the UPS data processing.
Figure 5.7: PTFE irradiated with 60 keV monoenergetic electrons. PEA measurements of 250 \(\mu\)m thick PTFE plotted with incident electron orientation from the (a) left and (b) right.
Figure 5.8: PEEK irradiated with 60 keV monoenergetic electrons. PEA measurements of 250 µm thick PEEK plotted with incident electron orientation from the (a) left and (b) right.
**Figure 5.9**: PTFE irradiated with 70 keV monoenergetic electrons. PEA measurements of 250 µm thick PTFE plotted with incident electron orientation from the (a) left and (b) right.
Figure 5.10: PEEK irradiated with 70 keV monoenergetic electrons. PEA measurements of 250 µm thick PEEK plotted with incident electron orientation from the (a) left and (b) right.
CHAPTER 6

RESULTS

Now that the context, background, and understanding of the methodology is understood, the results of various experiments are presented in this chapter. The first result is comparing charge distributions embedded via electron irradiation with a deep dielectric charging code AF-NUMIT3. Then, the utility of PEA measurements in the context of spacecraft charging is put to the test. Finally, results on pushing the limits of the PEA system to allow for characterization of shallowly deposited charge distributions relevant to spacecraft charging are presented.

6.1 Comparing to AF-NUMIT3

Successful spacecraft design and charging mitigation techniques require precise and accurate knowledge of charge deposition profiles. This study (Gibson et al., 2023) compares models of charge deposition and transport using a venerable deep dielectric charging code, AF-NUMIT3, with direct measurements of charge profiles via PEA measurements. Eight different simulations were performed for comparison to PEA experiments of samples irradiated by 50 keV or 80 keV monoenergetic electrons in vacuum and at room temperature. Two materials, polyether-etherketone (PEEK) and polytetrafluoroethylene (PTFE), were chosen for their very low electrical conductivities so that minimal charge migration would occur between irradiation times and PEA measurements. PEEK was found to have low acoustic attenuation, while PTFE has high acoustic attenuation through the samples, which had nominal thicknesses of 125 µm and 250 µm for each material. The measurements were directly compared to AF-NUMIT3 simulations to validate aspects of the code and to investigate the importance of various simulation options. These were also used to characterize the PEA instrumentation, measurement methods, and signal processing used. The measurement and simulation values for magnitude of charge deposition, penetration depth,
and charge deposition spatial profiles are largely in agreement, though spatial and temporal distributions in incident electron flux and effects of radiation induced conductivity (RIC) and delayed RIC during the deposition process complicate the analysis. This work provides an experimental validation of the AF-NUMIT3 deep dielectric charging code and insight into the accuracy and precision of the PEA method.

The key parameter in dielectric breakdown is the electric field. Internal electric fields can either be approximated indirectly through measurements such as probing surface potential (Hodges et al., 2013) or determined directly by measurements of the internal charge distribution via methods such as the pulsed electroacoustic (PEA) method (Dennison and Pearson, 2013; Griseri et al., 2006). The PEA method is a well-established method for non-destructive measurements of embedded charge distributions in dielectric materials. There has been much work done to experimentally validate charging models (Griseri et al., 2006; Hands et al., 2019; Pacaud et al., 2019; Rodgers et al., 1998b; Song et al., 2021). Most relevant to this study was an attempt to directly measure internal potentials of electron irradiated materials by layering materials and electrodes and comparing to JPL’s NUMIT 2.0 (Kim et al., 2010).

However, there appear to be no published attempts to validate any version of NUMIT with direct measurements of the charge distribution via the pulsed electroacoustic method, aside from one recent study (Beecken et al., 2022). PEA measurements are a more fitting and robust test of deep dielectric charging codes such as NUMIT, as NUMIT determines the internal charge distribution to calculate the internal electric field. Experimental validation is particularly important at lower incident electron energies, below 100 keV, where the models for electron penetration depth used in these codes are somewhat less accurate (Beecken et al., 2015; Gibaru et al., 2021; Wilson and Dennison, 2012), and can be the most relevant to spacecraft charging (Jun et al., 2008). Fluxes at these lower energies from 10–100 keV are much higher in typical space environments (Hastings and Garrett, 2004) and have been shown to lead to most spacecraft anomalies (Frooninckx and Sojka, 1992), as discussed in Chapter 1.
In this section, a comparison of measurements to simulations for polymers irradiated by mono-energetic electrons is presented. The measurements were made using the PEA method and simulations predicted by AF-NUMIT3. The samples chosen to study were polyether-etherketone (PEEK) and polytetrafluoroethylene (PTFE) as both have very high resistivity so that the embedded charge will be “stationary.” The PEA method utilizes propagating acoustic pressure waves through the sample for measurement. As such, this provides a comparison of results for samples with high acoustic attenuation (PTFE) and low acoustic attenuation (PEEK), as is evident in the results. The incident electron energies chosen were 50 keV and 80 keV with average beam fluxes of $\sim 840$ pA/cm$^2$ and $\sim 640$ pA/cm$^2$, respectively. The nominal thicknesses of the samples were 125 $\mu$m and 250 $\mu$m. Two samples were irradiated at each energy, flux, and thickness in identical conditions to test the reproducibility of the experiment, for a total of 16 samples. The section starts by giving a description of the samples and their properties followed by the irradiation details. The results of the experiment and simulations are presented and discussed. The section ends with conclusions drawn from comparison of the measurements with simulations and a discussion of future work.

### 6.1.1 Sample Details

PEEK samples from Aptiv Victrex PEEK Film Technology were nominally 125 $\mu$m (1000-125G) and 250 $\mu$m (1000-250G) thick. PTFE samples from McMaster-Carr were nominally 125 $\mu$m (MCVS005X12X3) and 250 $\mu$m (SF103V0010) thick. Four samples of each material type and thickness were cut as discs of 3.38 cm diameter. Table 6.1 lists the samples studied, along with their sample designation and average thicknesses as determined by several measurements with a digital micrometer (Mitutoyo IP65; $\pm 0.5$ $\mu$m resolution).

The speed of sound for each sample was determined by dividing the measured thickness by the measured peak-to-peak time difference for the two sample interfaces in the PEA measurements. Estimated aggregate uncertainties were $\sim 0.5\%$ for PEEK and $\sim 3\%$ for PTFE (see Table 6.2). The primary sources of uncertainties were from the measured sample thicknesses ($\pm 0.4\%$) and individual sample thickness variations ($\leq 1\%$). Uncertainties in
the speed of sound at room temperature also include effects from ±1.5 K temperature variation in the lab, estimated to be on the order of ∼0.2% (Ginzel and Ginzel, 2017; Rae and Brown, 2016; Wood et al., 2018). This uncertainty also depends on the precision and accuracy of the peak-to-peak distance between interfacial peaks in PEA measurements, which have experimental uncertainties on the order of ∼ 10^{-10} s or ≤0.1%. There may also be systematic errors resulting from using the peak location instead of the rising edge or an envelope function and from distortions in the peak shapes due to ultrasonic attenuation (especially for PTFE). Sound speeds in polymers are sensitive to chemistry/processing and should be measured for individual samples (Rae and Brown, 2016). The speeds of sound for PEEK and PTFE measured in these studies are in reasonable agreement with literature values (Rae and Brown, 2016).

Table 6.2 lists materials properties for PEEK and PTFE with associated uncertainties; most of these were measured for the specific samples tested at Utah State University (USU). Note that all listed parameters are direct inputs to AF-NUMIT3 except for the speed of sound and the radiation induced conductivity (RIC) power (assumed to be unity in AF-NUMIT3 (Beecken et al., 2015)). Mean atomic numbers and mean atomic weights were calculated as defined in Ref. (Tabata and Ito, 1974). The extremely low measured dark conductivities, \( \sigma_{DC} \), for PEEK (Wood et al., 2018) and PTFE (Gillespie, 2022) coupled with the measured relative permittivity, \( \epsilon_r \), result in decay times \( \tau_{DC} \equiv \epsilon_r \epsilon_0 / \sigma_{DC} \) (Lee et al., 2021) of >4 months, meaning that deposited charge will be approximately stationary.
over the timescale of the experiment. Even after several months the charge migration is on the order of <1 μm; refer to Ref. (Gibson and Dennison, 2022b). This was an important consideration, as the samples were irradiated at the AFRL facility then shipped to USU for PEA measurement. The samples were transported and stored in purged, sealed, and light tight containers backfilled with high purity Ar. Argon storage has been shown to greatly extend recovery times of irradiated polymeric samples, as opposed to exposure to ambient air with moisture or even oxygen or dry nitrogen (Plis et al., 2018). During transport, handling, and PEA measurements, cumulative sample exposure to ambient atmosphere was limited to less than ~1 hr. Likewise, these samples were only exposed to red light to avoid photoconductive charging and discharging, as PEEK has a bandgap at ~3.11 eV, just beyond the visible range (Mackova et al., 2014) and exposure to ambient visible or UV light may potentially charge/discharge PEEK samples. The same protocol was followed for PTFE samples, although no photoconductive charging effects were expected.

### 6.1.2 Irradiation Details

Samples were baked out twice prior to irradiation, once at 100°C for 72+ hrs at ~10^{-4} Torr at USU before transport to AFRL, and again at 60°C for 24 hrs at ~10^{-1} torr at AFRL. Samples were irradiated at room temperature with either 50 keV or 80 keV using

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#### Table 6.2: Properties of materials tested. (a) Based on definition in Ref. (Tabata and Ito, 1974) (b) Manufacturer’s specifications (c) Ref. (Dennison and Pearson, 2013; Wood et al., 2018) (d) Ref. (Lee et al., 2021) (e) Refs. (Gillespie, 2022; Wood et al., 2018) (f) Refs. (Boman et al., 2021; Gillespie, 2022)

<table>
<thead>
<tr>
<th>Material</th>
<th>Mean Atomic Number (a)</th>
<th>Mean Atomic Weight (a)</th>
<th>Density, (g-cm^{-3}) (b)</th>
<th>Relative Permittivity (c,d)</th>
<th>Dark Conductivity, (S-cm^{-1}) (c,e)</th>
<th>RIC Coefficient, (rad^{-1}·s·S·cm^{-1}) (c,f)</th>
<th>RIC Power Δ (c,f)</th>
<th>Speed of Sound, (m·s^{-1}) (c)</th>
</tr>
</thead>
<tbody>
<tr>
<td>PEEK</td>
<td>6.12</td>
<td>11.75</td>
<td>1.32±0.2</td>
<td>3.45±0.1</td>
<td>(7±3)×10^{21}</td>
<td>(2.0±0.4)×10^{16}</td>
<td>0.911±0.05</td>
<td>249.5±14</td>
</tr>
<tr>
<td>PTFE</td>
<td>8.28</td>
<td>17.25</td>
<td>2.15±0.2</td>
<td>2.1±0.1</td>
<td>(2±3)×10^{20}</td>
<td>(2.9±0.6)×10^{17}</td>
<td>0.981±0.05</td>
<td>144.9±27</td>
</tr>
</tbody>
</table>
a monoenergetic electron beam (Kimball Physics EGH-8105 100 keV electron flood gun) with normal incidence with a vacuum pressure of $8 \times 10^{-7}$ torr in the Jumbo chamber at AFRL (Cooper and Hoffmann, 2015). Samples were mounted to a 20 cm OD 1.3 cm thick Al disc with two small strips of copper tape [see Fig. 6.1(a)]. The samples rotated into and out of the beam during irradiation; refer to Fig. 6.1. In Fig. 6.1(b), the red circles show representative sample positions. The solid black line is the path of the centers of the samples. The dashed black lines outline the $\sim 12$ mm diameter active area of the sample to be measured by the PEA system. Beam current is monitored with a Faraday cup at the center of the carousel. The map topology is identical for 50 keV and 80 keV, differing only slightly in amplitude. Pictured in Fig. 6.1(b) is a beam map scaled by a flux of 170 pA/cm$^2$ at the Faraday cup. The carousel rotated at 2 RPM for 150 s with the beam on. This gave each sample 75 s cumulative exposure in the beam path in 15 s intervals, as the samples are in the beam path for $\sim 50\%$ of the time due to rotation of the sample stage. The average flux while in the beam path was $840 \pm 740$ pA/cm$^2$ and $640 \pm 150$ pA/cm$^2$ and total fluence was $(6.3 \pm 5.6) \cdot 10^{-4}$ C/m$^2$ and $(4.8 \pm 1.1) \cdot 10^{-4}$ C/m$^2$ for the 50 keV and 80 keV beams, respectively. Note that there is a large error in the average flux due to the period of higher dose rate near the beginning of the 50 keV irradiation as shown in Fig. 6.2.

### 6.1.3 Modeling Details

The details of the model are presented in Chapter 2.

It is worth noting that the simulations were done before experimentation. That is, there was no fitting to the experimental data in any way. The only “free parameters” in AF-NUMIT3 are spatial bin size, temporal step size, and a smoothing factor. As is standard practice in simulations, the spatial bin size and temporal step size were chosen so that they had negligible effects on the simulation results while minimizing required computation time. The simulations were all performed with effectively no electrode on the irradiated surface and a grounded electrode on the back surface.

Photoemission and secondary electron emission features were not used for this simulation, as they are only important with incident light and low energy ($\leq 20$ keV) incident
Figure 6.1: Sample irradiation at AFRL. (a) Photograph of one sample set mounted to rotating carousel at AFRL irradiation facilities. The PEEK samples are tan and PTFE samples are white. (b) Beam map showing beam current density and the path of the samples through the beam.
electrons (Davis and Mandell, 2014), respectively. In the AF-NUMIT3 simulations the incident beam was approximated by applying the average beam flux for 75 s. Note that this ignores the increased dose for the 50 keV irradiation and slightly decreased dosed for 80 keV during the first few seconds of irradiation, as shown in Fig. 6.2. Also neglected were any delayed or time dependent RIC effects from ramping up/down the beam flux as the sample moves into and out of the center of the beam. Future work should investigate these discrepancies for more accurate predictions.

6.1.4 Measured and Simulated Charge Distributions

To compare the PEA measurements and simulation results, five parameters to characterize the spatial charge profile were extracted from each dataset and compared to results of the simulations. The integrated area of the charge distribution (total charge deposited per area), the charge distribution peak amplitude, peak deposition depth, FWHM, and half width at half maximum (HWHM) for the two sides of the distribution were determined. These can be directly related to the first four moments about the mean of the embedded charge distribution; that is, the integrated area under the distribution, mean value, standard deviation, and skewness. For example, a symmetric Gaussian distribution, which models the distribution of a thin layer (estimated as ±1 µm) broadened due to random surface roughness or instrumental effects, is peaked at the mean value and is symmetric about the mean so the difference between the HWHM’s is zero or their ratio is unity (i.e., the first and third moments about the mean of a symmetric Gaussian distribution are zero).

Measured Charge Distributions

Measured and simulated charge profiles for PEEK and PTFE samples irradiated with monoenergetic electrons of 50 keV and 80 keV incident energies are plotted in Figs. 6.3-6.6, with summaries of the peak distribution parameters listed in Tables 6.3, 6.4, 6.5, and 6.6. To get an idea of the spatial resolution of each measurement, the FWHM of the leading (left) interfacial peak was determined for each measurement, regardless of the direction of incident charge. The average value of the FWHM is 10.6±1.1 µm and 7.6±1.5 µm for PEEK
and PTFE samples, respectively. This provides an estimate of the instrumental resolution or width of the response function.

Each sample was measured in two orientations as a consistency check, once with the irradiated surface of the sample facing the cathode (pulsing electrode) such that the apparent incident direction is from the right, and once by inverting the sample so that the irradiated surface is facing the anode and the apparent incident direction is from the left. The direction of the incident beam is indicated by the arrows in Figs. 6.3-6.6. These results should agree, as they are measurements of the same sample in different orientations. Measured curves are aligned to the interfacial peak of the irradiated side, so as to accurately compare deposited charge distributions. This is necessary as samples are of slightly different thicknesses. Samples of differing thickness should also agree, within a few percent, on the deposition depth though there will be some differences in the position of the interfacial peak corresponding to the unirradiated surface due to sample thickness variations.

The left/right incidence measurements are in relatively good agreement for the measurements of PEEK (see Fig. 6.3). However, this is not the case for PTFE (see Fig. 6.4). This is to be expected as there are large dispersion and attenuation effects in PTFE.

In order to obtain an acceptable deconvolution, results for the left incident measurements of 125 µm and 250 µm PTFE, reference curves from the right incident measurements were used. Measurements of 250 µm PTFE samples 1 and 3 were unable to be
Figure 6.3: PEEK irradiated with 50 keV electrons. PEA measurements for 125 µm thick PEEK samples irradiated with 50 keV electrons are displayed with the AF-NUMIT3 simulations.

Table 6.3: PEEK irradiated with 50 keV electrons. Characteristic parameters of charge distributions are listed.

<table>
<thead>
<tr>
<th>50 keV Energy</th>
<th>125 µm PEEK</th>
<th>250 µm PEEK</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample ID</td>
<td>Simulation</td>
<td>K4A</td>
</tr>
<tr>
<td>Incident Direction</td>
<td>Left</td>
<td>Left</td>
</tr>
<tr>
<td>Amplitude (C·m⁻³)</td>
<td>50.2</td>
<td>51.5</td>
</tr>
<tr>
<td>Deposited Charge (10⁻¹² C·m⁻³)</td>
<td>5.6</td>
<td>5.6</td>
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<td>Peak depth (µm)</td>
<td>34.7</td>
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<td>FWHM (µm)</td>
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<td>10.8</td>
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<td>Left HWHM (µm)</td>
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<td>Right HWHM (µm)</td>
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<td>5.0</td>
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<tr>
<td>HWHM Ratio</td>
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<td>1.2</td>
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</table>
(a) 50 keV irradiated 125 µm PTFE, left incidence

(b) 50 keV irradiated 125 µm PTFE, right incidence

(c) 50 keV irradiated 250 µm PTFE, left incidence

(d) 50 keV irradiated 250 µm PTFE, right incidence

**Figure 6.4**: PTFE irradiated with 50 keV electrons. PEA measurements for 125 µm and 250 µm thick PTFE samples irradiated with 50 keV electrons are displayed with the AF-NUMIT3 simulations.

**Table 6.4**: PTFE irradiated with 50 keV electrons. Characteristic parameters of charge distributions are listed.

<table>
<thead>
<tr>
<th>50 keV Energy</th>
<th>125 µm PTFE</th>
<th>250 µm PTFE</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample ID</td>
<td>Simulation</td>
<td>T4A</td>
</tr>
<tr>
<td>Incident Direction</td>
<td>Left</td>
<td>Left</td>
</tr>
<tr>
<td>Amplitude (C·m⁻³)</td>
<td>65.8</td>
<td>40.6</td>
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<td>Deposited Charge (10¹⁴ C·m⁻³)</td>
<td>5.3</td>
<td>6.1</td>
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<tr>
<td>Peak depth (µm)</td>
<td>20.6</td>
<td>20.1</td>
</tr>
<tr>
<td>FWHM (µm)</td>
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<td>Left HWHM (µm)</td>
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<td>5.1</td>
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<td>Right HWHM (µm)</td>
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<td>9.4</td>
</tr>
<tr>
<td>HWHM Ratio</td>
<td>1.3</td>
<td>0.5</td>
</tr>
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</table>
Figure 6.5: PEEK irradiated with 80 keV electrons. PEA measurements for 125 µm and 250 µm thick PEEK samples irradiated with 80 keV electrons are displayed with the AF-NUMIT3 simulations.

Table 6.5: PEEK irradiated with 80 keV electrons. Characteristic parameters of charge distributions are listed.

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>125 µm PEEK</th>
<th>250 µm PEEK</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Simulation</td>
<td>K1A</td>
</tr>
<tr>
<td>Incident Direction</td>
<td>Left</td>
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<tr>
<td>Amplitude (C/m²)</td>
<td>17.0</td>
<td>172.6</td>
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<tr>
<td>Deposited Charge (10⁴ C/m²)</td>
<td>4.3</td>
<td>24</td>
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<tr>
<td>Peak depth (µm)</td>
<td>77.3</td>
<td>74.4</td>
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<tr>
<td>FWHM (µm)</td>
<td>21.1</td>
<td>13.5</td>
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<td>Left HWHM (µm)</td>
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</tr>
<tr>
<td>HWHM Ratio</td>
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<td>1.1</td>
</tr>
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</table>
(a) 80 keV irradiated 125 µm PTFE, left incidence

(b) 80 keV irradiated 125 µm PTFE, right incidence

(c) 80 keV irradiated 250 µm PTFE, left incidence

(d) 80 keV irradiated 250 µm PTFE, right incidence

Figure 6.6: PTFE irradiated with 80 keV electrons. PEA measurements for 125 µm and 250 µm thick PTFE samples irradiated with 80 keV electrons are displayed with the AF-NUMIT3 simulations.
Table 6.6: PTFE irradiated with 80 keV electrons. Characteristic parameters of charge distributions are listed.

<table>
<thead>
<tr>
<th>80 keV Energy</th>
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<th>250 μm PTFE</th>
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<td>Left</td>
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<tr>
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<td>6.9</td>
</tr>
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</tr>
<tr>
<td>Right HWHM (μm)</td>
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<td>9.7</td>
</tr>
<tr>
<td>HWHM Ratio</td>
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</table>

<table>
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<tr>
<td>Sample ID</td>
<td>Simulation</td>
<td>T1B</td>
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<tr>
<td>Incident Direction</td>
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<td>Left</td>
</tr>
<tr>
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<td>0.5*</td>
</tr>
<tr>
<td>Deposited Charge (10⁴ C-m⁻²)</td>
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<td>0.05*</td>
</tr>
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<td>Left HWHM (μm)</td>
<td>11.1</td>
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<td>Right HWHM (μm)</td>
<td>7.3</td>
<td>8.4</td>
</tr>
<tr>
<td>HWHM Ratio</td>
<td>1.5</td>
<td>0.3</td>
</tr>
</tbody>
</table>
accurately calibrated for volume charge. A poor calibration was obtained for the right incident measurements, and the left incident measurements use the reference curves from the right incident measurements, so no charge magnitude calibration was attempted for the left incidence. Left incident measurements of 125 µm PTFE samples 4 and 6 were unable to be accurately calibrated for volume charge as well. The right incident measurement of 125 µm PTFE sample 6 had to be calibrated using the sample 4 reference curve. All other samples were calibrated in a typical manner as outlined in the Section 4.3.

The integrated area of the charge distributions can be used as a consistency check by comparing it to the total expected fluence. The units of the integrated charge distributions are C/m². That is, the units are charge per unit area. As the measurements are 1D in nature, it is reasonable to express charge density or fluence in terms of charge per unit area. The total fluences expected based on the experimental incident electron fluxes are approximately $6.4 \cdot 10^{-4}$ C/m² and $4.8 \cdot 10^{-4}$ C/m² for 50 keV and 80 keV irradiations, respectively. Note that ~10% backscatter is typical for these higher incident energy electrons and was assumed for comparison to measurements. The total fluence was determined for a PEA measurement by integrating over the deposited charge distribution (ignoring induced charge on electrodes).

For 50 keV irradiated PEEK samples the fractional differences between predicted and measured total fluence are 4% and 29% for the 125 µm and 250 µm samples, respectively. Ignoring the uncalibrated data for 50 keV irradiated PTFE, these fractional differences are 8% and 19% for 125 µm and 250 µm samples, respectively. Thus, both the PTFE and PEEK are in quite good agreement with predicted charge deposition, considering the potential error introduced with signal processing and uncertainties in actual charge deposited due to issues from the rotation of the sample carousel and inconsistent flux.

The 80 keV irradiated samples (see Figs. 6.5 and 6.6) exhibit much poorer agreement. The thinner PEEK sample data exhibit a significant discrepancy in total charge deposited. Several repeated measurements indicate this is not a calibration issue. It is unclear where this charge in excess of the prediction (AF-NUMIT3 simulation) came from. The thicker
PEEK samples (see Fig. 6.5) have a relatively smaller discrepancy in deposited charge of \( \sim 55\% \). The thicker PEEK samples also appears to exhibit a positive charge distribution between the deposited negative distribution and the irradiation surface; more investigation is required to confirm if this positive distribution is real or an artifact of signal processing.

Some of the most interesting results are obtained from the 80 keV irradiated PTFE samples (see Fig. 6.6) which exhibit multiple peaks in the measured deposited charge distributions. For all 80 keV PTFE samples, there is a negative peak near the irradiated surface and another negative peak much deeper in the sample. The 250 \( \mu \)m PTFE samples also show a possible positive charge peak between the two negative charge peaks which could be due to deconvolution artifacts or may be real and a sign of arcing and/or aging of the material. Ignoring the uncalibrated measurements, the fractional differences (as compared to the AF-NUMIT3 simulations) in the total charge (totaled for all distributions in a sample of 80 keV irradiated PTFE) is 43\% and 80\% for the 125 \( \mu \)m and 250 \( \mu \)m samples, respectively.

The uncertainties in the peak deposition depth for the deposited charge profiles measured with PEA for PEEK are \( \leq 0.5 \) \( \mu \)m. The PEA measurements for the different thickness samples are in good agreement for the 80 keV data, but not for the 50 keV data. The discrepancy in peak deposition depth in the 50 keV measurements between the two thicknesses is thought to be related to the 125 \( \mu \)m thick samples receiving a significantly higher dose rate during the beginning of the irradiation. This was presented in Section 5.1.2. Fig. 6.2 shows a \( \sim \) 20 s long period of higher flux at the beginning of the 50 keV irradiation that was only incident on some of the samples as the samples are rotated into and out of the beam; refer to Fig. 6.1.

The uncertainties in the peak deposition depth for the deposited charge profiles measured with PEA for PTFE are \( \leq 2 \) \( \mu \)m. This uncertainty could be reduced by decreasing the uncertainty in the speed of sound. However, this process is complicated by large differences when comparing the left/right incidence measurements, due mostly to the large acoustic attenuation and dispersion effects for PTFE. The deposition depths measured for
left incidence are more accurate, as the attenuation and dispersion effects are minimized because the signal travels through less of the highly attenuating sample material.

Note that the skewness, as gauged by the half-width-half-maximum ratios (see Tables 6.3, 6.4, 6.5, and 6.6), can also change with perturbation of processing parameters. The PEEK samples of both irradiation energies have essentially no asymmetry (a ratio of \( \sim \)1). PTFE samples show asymmetry in measurements that are not consistent between left/right incidence measurements. This could be due to the dispersion and attenuation effects. Further investigation is needed to more accurately characterize the asymmetry of PEA measurements through comparison of asymmetries of the interfacial peaks for raw and processed data.

**Simulated Charge Distributions**

The AF-NUMIT3 simulation results were also characterized by the deposited peak characteristics, as outlined in the previous section.

The total charge deposited in the sample for the simulations is less than the expected total fluence from experimental fluxes by \( \sim \)10\%. This is due to a backscatter term used in the simulation code; refer to Eq. (8) in (Beecken and Wallin, 2011). This same backscatter correction is used for all the simulations, and the total deposited charge is the same for different thicknesses in the various simulations.

The amplitude and position of the deposited charge distribution is predicted by the simulations to be slightly different for different thicknesses. The amplitudes and peak deposition depths are lower for the 250 \( \mu \)m thick samples than 125 \( \mu \)m thick samples. However, these effects are relatively small, differing by only a few percent. These effects are likely due to RIC in the irradiated region during irradiation which can persist for some time after irradiation due to delayed RIC. There is no electrode at the front surface and hence no potential difference or electric field between the deposited charge distribution and the front surface driving charge transport in the irradiated region. However, a mirror charge induced on the rear grounded surface of the sample carousel due to the deposited charge distribution causes an electric field proportional to the distance between the deposited
charge distribution and the rear electrode. Thus, there will be a stronger electric field for thinner samples with reduced distance to the rear grounded surface than for thicker counterparts. RIC effects increase the conductivity enough for the charge in the irradiated region to be somewhat mobile during irradiation. This will allow the electric field to drive the peak of the charge distribution deeper into the sample. The effect of this is seen as a deeper peak deposition depth approaching the range of the irradiating electron flux and a higher amplitude peak as the charge tries to accumulate as deep as the dose (and therefore RIC effects) penetrate.

The simulations also predict an asymmetry in the deposited charge distributions. The asymmetry differs slightly for differing thicknesses as well, which is also likely explained by RIC effects described above. For PEEK samples, the charge distribution is skewed such that the distribution is wider deeper into the dielectric (away from the incident surface). The opposite is true for the PTFE simulations; the charge distribution is skewed towards the incident surface. Such differences may also be a reflection of a more complex spatial distribution of charge carriers (perhaps both electrons and holes).

6.1.5 Comparison of Measurements and Simulation Results

The results of the PEA measurements and simulations have been summarized. This section compares the PEA measurements with the AF-NUMIT3 simulations and comments on their agreement based on the characteristics of the deposited charge distributions.

The amplitude of any given charge distribution is not a good measure of accuracy, as this will change with the resolution and processing of the data. Note that the total charge is also not a good comparison, as the PEA calibration methods and data processing have not been optimized. However, even with suboptimal processing, we expect the magnitude of the charge distribution and total deposited charge to agree between simulation and experiment to within a factor of a few or better.

The AF-NUMIT3 simulations agree well with the 50 keV irradiated PEEK samples. The peak deposition depth from simulation of the 125 µm thick PEEK were within 5% of the average PEA measurement. The results for the 250 µm thick sample were not as
good, but still fell within 12%. However, it seems likely that the 125 µm PEEK samples were irradiated with the higher dose at the beginning of irradiation, as seen in Fig. 6.1. That change in irradiation was not included in the AF-NUMIT3 simulation predictions. It is also possible that the difference in agreement is a thickness effect. As discussed in the previous section, RIC allows charge to be somewhat mobile during irradiation. Delayed RIC would continue operating between the irradiation periods, and the AF-NUMIT3 simulation prediction did not account for that. Further investigation is needed.

The 80 keV irradiated PEEK PEA results, however, do not seem to corroborate the argument that it could be due to differences in thickness. The simulation peaks are at a depth that falls within 5% of the average PEA measurement for the 125 µm samples and within 2% for the 250 µm samples. The measured deposition depth disagrees with the simulated deposition depth, by being 1 – 3 µm shallower, depending on the thickness of the sample (deeper for thinner sample). Taking into account the large errors with the amplitude and total charge deposited for the PEA measurements, the simulation results, by almost all parameters, are in relatively good agreement for PEEK at both 50 keV and 80 keV incident energies (aside from K1A and K3A, which have inexplicably large amounts of charge).

The simulations did not predict a significant asymmetry in the embedded charge distribution and no significant asymmetry was measured for the PEEK data, except for the thicker 80 keV irradiation which was predicted and measured. Note that the narrow distributions are likely due to RIC effects and the slight discrepancy in the degree of asymmetry in the thicker 80 keV PEEK samples may be due to not having taken into account delayed RIC in the simulation. Delayed RIC is the residual RIC which decays after the radiation has stopped. This could potentially drive the charge deeper into the sample because the conductivity is elevated due to delayed RIC. RIC is dose rate dependent, so charge will be unable to travel deeper than the range of the deposited energy (dose). This results in a narrower distribution that is peaked slightly deeper than the original deposited distribution. Delayed RIC is an option that can be modeled using AF-NUMIT3, but was not utilized here, as the incident beam was modeled as a continuous irradiation using a constant
The width of the distributions for the 80 keV irradiated PEEK samples, as determined by the FWHM, are also narrower than the simulated charge distributions. This supports the delayed RIC argument. However, the FWHM are essentially as predicted for the 50 keV irradiated PEEK samples. This could be due to spatial resolution of the PEA system. Therefore, the actual charge distribution may be narrower than can be determined by PEA measurements. Higher spatial resolution measurements would be necessary to investigate this. The delayed RIC portion of the AF-NUMIT3 code should be tested against such data in the future. Note that there also appears to be a slight positive charge peak in the 250 µm 80 keV irradiated PEEK measurements, which may be due to arcing and/or aging effects. Here we are focusing only on the large negative charge peak for comparison to AF-NUMIT3.

PTFE data is much harder to deal with and to compare to the AF-NUMIT3 simulations. Several measurements were unable to be calibrated for volume charge, so the magnitude of charge cannot be compared to simulations. The 50 keV irradiated PTFE measurements agree with the simulations quite well for the peak deposition depth with five of six measurements within \( \sim 1\mu \text{m} \). There is less agreement with the right incident 250 µm samples, likely due to the high attenuation and dispersion. The measured peaks are wider than the simulations by approximately a factor of 2. This is likely due to lower spatial resolution in PTFE samples. The asymmetry of the charge distributions are predicted to be broader towards the irradiated surface but the measurements appear to exhibit a broader distribution deeper into the sample.

For 80 keV irradiated PTFE there is poorer agreement between measurement and simulation. PTFE is very hard to keep uncharged, so it is possible that there was surface charge on the samples before irradiation began. The samples were stored and handled carefully, and underwent vacuum bake out in an attempt to minimize residual charge. It is also possible that material damage played a role, as the onset of damage in PTFE is a total ionizing dose (TID) of \( \sim 10^4 \) rad (Chipară and Chipară, 1992; Shulman and Ginell, 1970) and the TID received here is \( \sim 10^4 - 10^5 \) rad.
It should be noted that dual negative peaks have previously been measured in electron irradiated PTFE (Perrin et al., 2008), as well as in other USU lab unpublished data. In Ref. (Perrin et al., 2008), the dual negative peaks appeared when the charge distribution was measured using what they termed “short circuit PEA,” which is essentially equivalent to the approach in the present paper. That is, electrodes on either surface of the sample. The difference for the “short circuit PEA” is that the electrodes are vapor deposited and the sample is in vacuum such that measurements can be made concurrently with electron irradiation. The authors of Ref. (Perrin et al., 2008), however, also did the same experiment with an “open PEA” system. This system utilizes a ground electrode in contact with the sample but the excitation electrode is floating above the sample. As such, “open PEA” is also sometimes called “non-contact PEA.” Using the “open PEA” approach produced one negative peak that has been precisely replicated with AF-NUMIT3 in Ref. (Becken et al., 2022). Work needs to be done to determine how the different irradiation environments and/or PEA systems produce such different results.

As the deposited distributions differ substantially from the simulated charge distributions in a quantitative comparison, it makes little sense in comparing the rest of the details of the simulation. However, it is perhaps worth noting that the FWHM of the simulations are larger than the measured FHWM of the deposited peaks for almost all 80 keV 250 µm PTFE measurements. The one exception is the deepest deposited peak in sample T3B. The FWHM is closer to agreement for samples T1A and T3A, but still differs by several microns.

This study presented a detailed comparison of PEA measurements and AF-NUMIT3 simulations for PEEK and PTFE samples irradiated with 50 keV and 80 keV monoenergetic electrons. The overall agreement of the simulations with experiment is very encouraging. However, there are several areas that can be improved upon.

Improvement to PEA measurements can be accomplished through optimization of PEA calibration and data processing methods for a more accurate comparison. In particular, this includes optimizing the filtering and deconvolution of data and taking into account
the dispersion and attenuation effects of the sample. Better and more fully characterized
spatial resolution of PEA systems would also be advantageous.

A potential improvement to the simulations is to better model time varying incident
fluxes. The results described here are an excellent example of the need for such a more
refined model, as the incident electron beam varied over the course of irradiation and the
flux incident on the sample varied further as the samples moved in and out of the beam
during irradiation. Inclusion of delayed RIC effects are likely to become important under
such circumstances, as conductivity then becomes time dependent.

The agreement between simulations and measurements here is very promising. Future
work should include addressing the improvements mentioned above and comparing PEA
measurements to other models such as the JPL version of NUMIT, studying the effects of
the presence or absence of surface electrodes during irradiation for both experiments and
simulations, and pushing the PEA system to measure polymers irradiated with even lower
incident energies that are particularly relevant to spacecraft charging (this work is underway
(Gibson and Dennison, 2022a)). The latter is addressed in the next section.

6.2 Relevancy of PEA Measurements to Spacecraft Charging

As discussed in Chapter 1, the critical incident electron energy range for spacecraft
charging concerns is $\sim 10-50$ keV. This translates to a range and estimated peak deposition
depth of $\sim 2-45$ $\mu$m and $\sim 1-30$ $\mu$m, respectively for PEEK.

To study the relevance of PEA measurements for spacecraft charging, a study was
devised (Gibson and Dennison, 2023a). In particular, it addresses the question of what are
the lowest incident energy electrons that can produce charge distribution peaks that are
resolvable using a typical PEA system as a criterion for the utility of PEA measurements
for the incident electron energies most commonly encountered in spacecraft charging events.
This was done by irradiating a typical spacecraft material, polyether-etherketone (PEEK),
with an electron beam with energies in the energy regime typical for spacecraft charging.
That is, the incident electron energies were nominally 10 keV, 20 keV, and 30 keV. This is
also compared to the results for 50 keV and 80 keV incident electron energies as presented
in the previous section.

PEEK was chosen as the test material since its very low conductivity meant that deposited charge can be considered stationary for the timescale of the PEA experiments (Wood et al., 2018). The relatively low acoustic attenuation and dispersion of PEEK allows for more accurate PEA measurements of the internal charge distribution (Gibson et al., 2023).

### 6.2.1 Experimental Details

The samples of PEEK used in this experiment are from APTIV Victrex PEEK Film Technology and are nominally 125 \( \mu \)m thick (1000-125G), the same sample batch as presented in the last section. The samples were prepared and treated in the same manner as before, described in Section 6.1.1.

The samples were irradiated with \( \sim 65 \pm 20 \) pA/cm\(^2\) for a total of 4 minutes each. A pair of samples were irradiated at each incident electron gun energy of 10 keV, 20 keV, and 30 keV, using a high energy electron gun (Staib EH-80) at USU with a vacuum level of \( \leq 10^{-6} \) hPa at room temperature. As the samples were irradiated at USU, the beam passed through a C foil to broaden the beam, as discussed in Section 3.3.

### 6.2.2 PEA Results for 10-30 keV Electron Irradiation

Measurements of the irradiated samples were obtained in two orientations such that the apparent direction of incident electrons are from the left or from the right. Only the measurements with left incidence are presented as they are much easier to align and compare as opposed to the right incidence measurements, which is particularly important for the 10 keV, 20 keV, and 30 keV incident energies. It should be noted that in this orientation there are also less attenuation and dispersion effects for the measured charge distribution, though this is not a large issue for PEEK.

Summarizing the results from the previous section, the PEEK sample irradiated with 80 keV electrons has a charge distribution with a peak deposition depth of 74.4\( \pm \)0.5 \( \mu \)m and the sample irradiated with 50 keV electrons has a peak deposition depth of 32.9\( \pm \)0.3 \( \mu \)m.
(Gibson and Dennison, 2022b; Gibson et al., 2023). The peak deposition depth here is the peak-to-peak distance of the irradiated surface’s interfacial peak to the embedded charge peak. The results for the nominal incident electron energies of 10 keV, 20 keV, and 30 keV can be seen in Fig. 6.7. The measured peak deposition depths are presented in Table 6.7 and the average values are plotted in Fig. 6.7(b). The pairs of measurements for each incident energy differ by \( \leq 0.3 \) µm, which is small compared to the apparent shifts of deposition depth between incident energies of \( \sim 1 \) µm for 10 keV, 20 keV, and 30 keV incident energies. This suggests that these shifts are real and not just a result of the variations due to instrument or sample effects.

There is a counterintuitive trend apparent in Fig. 6.7(b) for the measured peak deposition depth to decrease with increasing incident energy below \( \sim 25 \) keV. Possible causes for this phenomena include new physics (unlikely), effects of deconvolution, and mischaracterization of deposition depth due to superimposed charge distributions. To study the latter possible cause of this phenomena, a simple model was devised which can offer a qualitative explanation of this observation. This is a simple model of a negative embedded charge distribution near the surface in superposition with a positive interfacial charge distribution.

**Table 6.7:** Low energy electron irradiation details. Electron irradiation and charge deposition details for incident electron energies from 10 to 80 keV.

<table>
<thead>
<tr>
<th>Energy (keV)</th>
<th>Range (µm)</th>
<th>Estimated Peak Deposition Depth (µm)</th>
<th>Measured Peak Deposition Depth (µm)**</th>
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<tr>
<td>10 (0*)</td>
<td>2 (0*)</td>
<td>1 (0*)</td>
<td>12.5/12.5</td>
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<tr>
<td>20 (9)</td>
<td>9 (2)</td>
<td>6 (1)</td>
<td>11.2/11.6</td>
</tr>
<tr>
<td>30 (24)</td>
<td>20 (13)</td>
<td>13 (9)</td>
<td>10.1/10.6</td>
</tr>
<tr>
<td>50</td>
<td>45</td>
<td>30</td>
<td>32.9</td>
</tr>
<tr>
<td>80</td>
<td>100</td>
<td>67</td>
<td>74.4</td>
</tr>
</tbody>
</table>

*CASINO simulations indicated electrons would not penetrate the C scattering foil so incident energy is unknown but \( \leq 10 \) keV.

**Multiple values shown for the duplicate samples measured (e.g. sample 1/sample 2). Values in () are estimated from the average incident energy of electrons passing through the scattering foil as determined by the CASINO simulations (refer to Chapter 2).
Figure 6.7: Low incident energy results. (a) PEA measurements of PEEK irradiated with 10 keV, 20 keV, and 30 keV electrons are plotted, with arrow indicating incident electron direction. Only the section of the data near the irradiated surface is shown, as the charge is deposited at shallow depths. (b) The measured peak deposition depth versus incident electron energy is plotted with the range determined from the MPG Electron Range Approximation Tool v1.1 (Wilson and Dennison, 2012; Wilson et al., 2013, 2018) and the deposition depth estimated as 2/3 of the range.

Consider two Gaussian peaks, one positive and one negative, which are added together with varying peak-to-peak separations. If the Gaussians are of equal magnitude then the intuitively expected trend is observed; that is, the larger the separation between the initial Gaussian peaks, the larger the modeled separation in the peak-to-peak distance of the superimposed distributions, depicted as ratio of 1 in Fig. 6.8(b). However, this does not agree with the observed PEA measurements.
Figure 6.8: Simulated low incident energy results. (a) Superposition of two Gaussian distributions, one positive and one negative, with varying separations of the maxima. The positive distribution is scaled to 0.8 the amplitude of the negative distribution. The inset shows the resulting apparent deposition depth (peak-to-peak distance from positive to negative peak). (b) Plotted is a family of curves with various ratios of the amplitude of the positive distribution, including the plotted inset of (a).

This model with equal magnitude peaks is not representative for the PEA measurements. There is a lower magnitude charge distribution at the incident interface, due to the exciting pulse of the PEA system and a small amount of mirror charge at the opposite electrode. The pulse has an effect on the measured interfacial charge distribution (Chen et al., 2006), particularly when there is only a small amount of charge being measured in the bulk, as is the case for the PEA measurements in this study.
When the first positive Gaussian is reduced in magnitude as compared to the negative distribution, then a different trend emerges. As the separation between the initial Gaussians is increased, there is a trend in the resulting distribution of decreasing peak-to-peak distance with increasing initial separations (at small initial separations), followed by a return to the trend for increasing peak-to-peak distance with increasing peak separations modeled for equal peak amplitudes. This is illustrated in Fig. 6.8(a), which uses Gaussians of 10 µm FWHM with separations of 1 µm to 20 µm with the positive interfacial distribution having 0.8 times less magnitude than the negative distribution. At low initial separations, there is a small decrease in the resulting peak-to-peak distances before this then increases again; refer to inset of Fig. 6.8(a).

Figure 6.8(b) reproduces this inset, along with a family of such curves with ratios of interfacial positive peak magnitude to embedded charge negative peak amplitude at 1.0, 0.8, 0.6, 0.4, and 0.2. The results show that the apparent deposition depth increases with decreasing initial separation more rapidly with a more dramatic difference in magnitude of superimposed Gaussians (ratio further from 1). The dashed line indicates the actual deposition depth (which is the initial peak-to-peak distance between the two Gaussians). At \( \sim 1.5x \) the spatial resolution (FWHM of the Gaussians), the difference between the apparent deposition depth and the actual deposition depth become essentially negligible.

**Discussion of Results**

The PEEK samples irradiated with 80 keV and 50 keV electrons resulted in embedded charge distributions that are easily measured with the PEA system (Gibson *et al.*, 2023). This clearly demonstrates the applicability of PEA measurements in the higher energy regime of energies of most interest for spacecraft charging. However, as the samples are irradiated with even lower energy electrons from 30 keV down to 10 keV, the limits of the PEA system become more apparent.

The measurement of a peak deposition depth of 10.4±0.5 µm for the nominal 30 keV irradiated PEEK sample may well be an accurate depiction of the internal charge distribution in the sample; refer to Fig. 1.2. However, this is close to the spatial resolution of
the system, which is 10.6±1.1 \( \mu \text{m} \) for PEEK (FWHM of leading interfacial peak) (Gibson et al., 2023). The apparent peak deposition depths of 11 \( \mu \text{m} \) and 12 \( \mu \text{m} \), for the nominal incident energies of 10 keV and 20 keV respectively, are also comparable to the instrumental FWHM. As the incident electron energy is decreased to nominal energies of 20 keV and 10 keV, there is a very small (\( \lesssim 2 \mu \text{m} \)) apparent shift of the charge distribution deeper into the material evident in Fig. 6.7. That is, it appears that the charge is deposited deeper into the sample with decreasing incident electron energy. This is non-physical, as charge is expected to be deposited less deep into the material with decreasing energy. This trend is likely due, at least in part, to the effects modeled in Fig. 6.8. That is, this is due to the superposition of the signal from the interfacial positive peak with the signal from the deposited negative peak in the PEA measurements.

This work indicates that the most accurate results, that is results minimally obfuscated by the signal from the interfacial peak(s), can be obtained when charge distributions are separated by \( \sim 1.5x \) the spatial resolution, as determined by the FWHM of the leading interfacial peak. For the current USU system, and other similar PEA systems, this optimal separation is on the order of \( \sim 15 \mu \text{m} \), which corresponds to the electron peak deposition depth estimated for a \( \sim 30 \text{ keV} \) electron beam incident on PEEK and from approximately 20 keV to 45 keV for low to high Z materials, respectively, shown in Fig. 1.2 from Chapter 1. A dashed line at 15 \( \mu \text{m} \) is plotted in Fig. 1.2 to indicate the region of materials and energies where a typical PEA system is likely to have accurate results. However, this is complicated due other factors. The spatial resolution can differ due to the speed of sound of the material measured. Materials with lower conductivity, such as LDPE, may not be easily measured in the same manner of this study as the charge may migrate or dissipate during transport of the sample out of the irradiation chamber to the ambient PEA system or during measurement in the PEA system.

This study has shown the relevancy of PEA measurements for measuring deposited charge distributions in the range of incident electron energies of importance for spacecraft charging. Charge distributions are easily resolved in PEEK for incident energies \( \gtrsim 30 \text{ keV} \)
with the current USU PEA system. Materials with lower Z should be able to be measured in this method with a typical PEA system when irradiated with energies $\gtrsim 20$ keV, as long as the conductivity is sufficiently low enough to keep the charge stationary on the timescale of the measurement.

The spatial resolution of the PEA method can be improved, through both experimental enhancements and data processing techniques, to push the resolution of the system to energies $\gtrsim 20$ keV. It should be noted that at least one PEA system does have spatial resolution on the order of a few microns (Kumaoka et al., 2014). PEA systems with this high spatial resolution should be capable of measuring deposited charge distributions from incident electron energies $\gtrsim 10$ keV; refer to Fig. 1.2 from Chapter 1.

Further work is needed to investigate what useful information can be extracted about embedded charge distributions near the surface given that the distributions are on the order of or narrower than the spatial resolution of the measurement. There are also issues with only considering the peak of the charge distributions. Consideration of the rising edge may give a more accurate depiction of deposition depths, but this would not provide any information about the shape of the distribution. Further work should be done to study the other moments of the measured charge distributions to see what useful information can be extracted.

Incident electron energies should be investigated in the range of 15 keV to 50 keV at smaller energy increments to better delineate the range of energies the current PEA system can accurately investigate and to increase confidence in the PEA measurements in this energy range, looking for the charge to be deposited deeper into the sample with increasing energy. The simple model of superimposed Gaussians could be improved to provide quantitative insight and should be explored further. The convolution of the PEA response function and an expected charge distribution as compared to a raw PEA signal could also provide further insight and intuition.

It is also possible to measure embedded charge distributions near the surface in open electrode configuration PEA systems (often called “open PEA” or “non-contact”) as the
excitation electrode is not in physical contact with the sample and therefore there is no measured mirror charge superimposed with the embedded charge signal (Arnaout et al., 2015; Arnaout et al., 2021; Griseri et al., 2003; Riffaud et al., 2016). This would also allow for higher conductivity materials to be easily measured without worry of charge migration during sample transport as is needed with an ambient PEA system. However, the difficulty then becomes determining the position of the surface of the sample to obtain low absolute error in the distance calibration. This would also provide a poor measurement for materials such as PTFE with high dispersion and attenuation if the sample is too thick.

Studies to investigate signal processing and data analysis techniques to improve uncertainties in key parameters of the measured charge distributions, such as the peak of the charge distribution, were presented in Chapter 5. The ability to resolve peak positions to $\sim 1$ $\mu$m (see Fig. 6.7 and 5) already suggest that such alternative measures of instrumental resolution may extend the validity of PEA measurements to lower energies relevant to spacecraft charging applications. Overall, PEA measurements are a promising tool to aid in the understanding and mitigation of spacecraft charging.

To improve the relevancy of PEA measurements to spacecraft charging, a novel method is explored employing signal processing to remove the effect of the interfacial charge in ambient PEA measurements so as to measure only the deposited charge distribution near the surface.

6.3 Measuring Shallow Charge Distributions

This section presents a proof-of-concept study for a simple method, referred to as the method of linear subtraction of the interfacial peak (LSIP), to remove the signal originating from the interfacial charge from the PEA measurement to more accurately observe shallow charge distributions (Gibson and Dennison, 2023b). The method proposed is to simply effectively subtract the signal from the induced mirror charge on the electrode near the deposited charge distribution from the measurement PEA profile. This is accomplished by using a pristine reference sample measured with varying applied voltages. This scaled reference can then be directly subtracted from the raw data of the original PEA measurement.
of the irradiated sample with the shallow charge distribution.

This section begins by outlining the experimental details and is followed by an explanation of the proposed method. It ends with a discussion of the results, conclusions drawn from the study, and suggested future work.

The experimental details are simple as the sample measured is one of the 30 keV electron irradiated PEEK samples presented in the previous Section 6.2 (Sample 1).

6.3.1 Proposed Method: Linear Subtraction of the Interfacial Peak

The proposed analysis method involves measuring two samples. The first sample is pristine and has no embedded charge. This sample was measured with varying applied DC bias from -258 V to +258 V to obtain a set of reference measurements to use for removing the signal originating from the electrode-sample interface; refer to Fig. 6.9. The second sample contains embedded charge near the surface. Typically a DC bias is not applied during measurement of the irradiated sample. However, this was done here to provide multiple test measurements to attempt the proposed method with varying amounts of charge on the electrode-sample interface in addition to the mirror charge, induced by the embedded charge, on this interface. The applied DC bias to the irradiated sample was 0 V to +387 V; refer to Fig. 6.10.

An example outlining this novel method is presented in Fig. 6.11. The measurement of the irradiated sample with no DC bias, the reference measurement used to remove the signal from the charge at the electrode sample interface, and the resulting determination of the deposited charge distribution are displayed in red, blue, and green respectively. The analysis can be done in two ways, either by adding a reference of negative DC bias or subtracting a reference of positive DC bias. The response of the PEA system should be linear and it should not matter which reference is used (and scaled). However, it is suggested that the same sign of charge is removed to avoid any potential nonlinearities in the measured PEA signal. That is, for the positive induced charge it is best to remove a negative DC bias reference signal, as this induces positive charge at the electrode sample interface near the embedded charge distribution. This also removes the effect of the excitation pulse as it is
Figure 6.9: PEA measurements of pristine PEEK with varying DC bias applied. Inset shows amplitude as a function of applied voltage (markers) with a linear fit for the left (red) and right (black) interfacial peaks.

Figure 6.10: PEEK irradiated with 24 keV electrons. PEA measurements obtained with varying DC bias applied.

included in the reference signal. In the other case where the opposite polarity is used, the effect of the excitation pulse is instead doubled.

For analysis, a reference measurement must be chosen to subtract from the irradiated sample measurement that is closest to the actual amount of charge that should be removed. It is unlikely that a reference measurement will contain exactly the right amount of charge
Figure 6.11: Example of shallow charge measurement process. Example of measurements used to find the actual deposited charge distribution without effects of surface charge.

to remove and therefore must be scaled accordingly, following the linear trend observed in the inset of Fig. 6.9. An iterative process of changing the linear scaling factor and removing the signal of the electrode interface charge distribution must be completed to find the ideal combination of reference signal and linear scaling factor. In this study, this was done “by eye.” That is, the linear scaling factor was adjusted by hand until a best fit was determined.

The best fit can be determined in the following way. Starting with the reference signal closest to the ideal amount of charge (peak amplitude) to be removed from the irradiated sample measurement. Currently, this is found by trial and error by subtracting various reference signals from the sample data. If there is a positive charge peak still apparent, the amount of charge to be removed by the reference measurement is adjusted by increasing the linear scaling factor. This iteration is continued until the apparent embedded charge distribution appears to increase in magnitude (rising edge moves closer to interface). Plotting a horizontal line at zero amplitude can help to aid the eye when adjusting the scaling factor until there is no positive charge peak and the embedded charge distribution does not appear to increase. Ideally, a protocol or optimization algorithm for obtaining a best fit should be produced to remove the “human error” from of the result.

This scaling of the reference signal is justified in the following ways. The amplitude
of the measured PEA signal for the pristine sample with applied voltage scales linearly, as shown by the inset in Fig. 6.9. The average FWHM of these reference signals is 21.5±0.8 µm, with no correlation with the measured amplitude or applied voltage. The interfacial peak position was determined to have an error of ±0.7 µm, similar to the error of the FWHM. Better uncertainties can be obtained with less noisy data. Typical PEA measurements use applied voltages in the kilovolt range, where the applied voltage in this experiment is only 100’s of volts. This is due to the very low amount of charge deposited in the sample, inducing a low potential at the sample-electrode interface. Due to the low amplitude signals, the signal-to-noise ratio is lower and there is higher uncertainty in the peak position. This could of course be mitigated with the averaging of more measurements or increasing the amount of charge deposited in the sample.

6.3.2 Demonstration of Shallow Charge Measurement

It is clear in the measurements of the irradiated sample in Fig. 6.10 that it is difficult to accurately distinguish the charge distributions from the induced mirror charge and the embedded charge. However, once the signal from the sample electrode interface is removed, the embedded charge distribution becomes clear, as is apparent in Fig. 6.12. All measurements of the irradiated sample with varying DC bias with the induced charge removed agree quite well, as depicted in Fig. 6.12 where the resulting charge distributions are superimposed. Note that no fitting or optimization was attempted to force the results to agree. The FWHM of the determined shallow charge distributions were 19.6±1.7 µm. This is lower than the FWHM for the reference signals, but is within uncertainty. The effective amount of charge removed by subtracting the reference signal was determined for each measurement as a test of the validity of the analysis method and this effective amount of charge was found to scale linearly with the applied DC bias. This is another justification that the method is valid.

As a further test of the measurements and method, a simulation of the deposited charge distribution was determined with AF-NUMIT3. This can be seen as the narrower green distribution superimposed on the plot in Fig. 6.12. The simulation is narrower as
Figure 6.12: Results from all measurements of irradiated sample. The corresponding interfacial charge has been removed. An output from AF-NUMIT3 simulation is superimposed and references the right axis. A reference signal is plotted in black to indicate the position of the sample surface.

the simulation does not take into account the broadening due to the spatial resolution of the PEA system. The peak deposition depth as determined by the measurements and the simulation are $8.2 \pm 1.3 \mu m$ and $7.3 \mu m$, respectively. This slight discrepancy of $\sim 1 \mu m$ is already in excellent agreement and within uncertainties. The results could perhaps be further in agreement if effects from delayed radiation induced conductivity were included in the simulation (AF-NUMIT3 has the capabilities to do this but it was not used to obtain the presented results). When a surface of a sample is grounded during irradiation (the back surface in this case), delayed radiation induced conductivity will cause a slight shift in the peak position of the deposited charge distribution towards the grounded surface driven by the electric field due to the induced mirror charge; refer to Section 6.1.5 for a discussion of the effect.

6.3.3 Conclusions

The results indicate that the method of linear subtraction of the interfacial peak is a valid method for obtaining measurements of charge distributions near the sample surface
using an ambient PEA system. The peak position of the signal shows the position of the charge and this does not change with magnitude (amount of charge), as demonstrated in Fig. 6.9 by varying the applied voltage. This means that subtraction of a suitable linearly scaled reference signal can be used to accurately determine the location of the surface of the sample to an accuracy of $\leq 1\mu m$ in a well characterized PEA system (Gibson and Dennison, 2022b). The black curve in Fig. 6.12 is a reference measurement indicating the position of the surface of the sample. This allows for determination of the peak deposition depth of the charge distribution to be determined with a high degree of accuracy.

There are improvements that should be made for the LSIP method. As mentioned, an optimization algorithm should be implemented to remove the “human error” of this method. The optimized average linear scaling factor (with uncertainty) can be determined from a slope of a plot such as the inset of Fig. 6.9. This optimized linear scaling factor could then be used in a linear interpolation routine based on reference curves above and below the interface amplitude measured in the sample profile. The optimized linear scaling factor may also be determined using peak area (total charge) rather than peak amplitude; this should be investigated.

The sample measured contained a relatively low magnitude of charge and resulted in relatively noisy measurements, as the charge magnitude was approaching the sensitivity limits of the PEA system. Further work should be done with a higher magnitude of charge embedded and studies should also be extended to lower energies. The demonstrated measurement of a charge distribution with a deposition depth of $\sim 8\mu m$ is impressive, but is near the spatial resolution of the system. This method should be pushed to explore the limits in resolution of deposition depth down to 10 keV incident energy, and perhaps lower.

It should be noted that the spatial resolution limits the amount of useful information that can be determined about the shallow charge distributions. The width of these charge distributions are clearly narrower than the spatial resolution of the PEA measurements. However, other parameters such as asymmetry and the charge magnitude can be obtained in addition to the peak deposition depth with further analysis and processing of the data.
It should also be noted that measurements can be obtained using “open PEA” methods where the electrode near the irradiated surface is not actually in contact with the sample and there is no signal measured from that interface. This allows for measurements of less resistive materials where this is not possible when using an ambient PEA system, as charge may migrate during transport from an irradiation chamber to the measurement system if the sample is not sufficiently resistive. However, it then becomes more difficult to determine the exact position of the surface of the sample.
CHAPTER 7
CONCLUSIONS

The aim of this dissertation has been to put the PEA method in context and improve upon its applications to spacecraft charging. This chapter will summarize its contents and discuss the conclusions drawn from this work. The chapter ends with proposed future work.

7.1 Summary of Dissertation

This dissertation has put into context PEA measurements of polymers irradiated with low energy electrons, in the range of 10-50 keV, and the relevance to spacecraft charging. This was accomplished by describing the typical spacecraft environment and identifying the length scales associated with the critical incident electron energies most relevant to spacecraft charging. It was argued that PEA measurements are a suitable method for understanding and aiding in mitigating spacecraft charging.

To understand charging of dielectrics, the theory and modeling of charge dynamics were laid out. This lead into the experimental methods that gave an in-depth description of the PEA method, the USU PEA system, spatial resolution of PEA measurements, sample preparation, and charge deposition. To understand the data obtained through these methods, the data processing was described in detail. The effects of data processing on the final result was also discussed.

The PEA system was then characterized thoroughly, with particular emphasis on the uncertainty for the peak position. The peak position provides for an alternative view of the spatial resolution of a PEA measurement. Some PEA measurements of pristine and irradiated polymers were then compared with measurements obtained in a PEA system at UPS in Toulouse, France to provide validation.

Once the background and method were established, characterized, and validated, the results of several projects were presented. These included the validation of a deep dielectric
charging code (AF-NUMIT3), testing the utility of PEA measurements for low energy electron irradiated polymers relevant to spacecraft charging, and pushing the limits of the PEA method by demonstrating a novel method (LSIP) to measure shallow charge distributions resulting from low energy electron irradiation.

The next section overviews the conclusions that can be drawn from this work.

7.2 Conclusions From Work

7.2.1 Working PEA System

A working PEA system has been built and implemented in the USU MPG lab. The PEA system was developed in cooperation with Box Elder Innovations (BEI) and funding from the Air Force Research Laboratory. This has been shown through careful characterization of the components and study of the experimental uncertainties of the measurements. Furthermore, the measurements made in comparison with the UPS PEA system validate the USU PEA system and the PEA method. However, these measurements shed light on the issue of charge magnitude calibration in PEA measurements. Although both systems clearly measured the same distributions with the same features, the measurements did not agree on the magnitude of the charge embedded, from a few percent to more than a factor of 2, depending upon the measurement. Some of the disagreement between the measurements was due to the spatial resolution, as determined by the FWHM of the leading interfacial peak.

7.2.2 Spatial Resolution

The most commonly used metric of PEA spatial resolution, the FWHM, is not sufficient for defining the spatial resolution of the PEA method. The FWHM is but one moment used to define a Gaussian like charge distribution. The other parameters related to the first four moments are the peak position, half width at half maximum, and magnitude. It has been demonstrated through careful measurements of slightly differing doses in PEEK and slow charge migration that the uncertainty of the peak position is more than an order of
magnitude better than the spatial resolution as defined by the FWHM. This indicates that increased precision of measurements and understanding of the embedded charge distributions can be gained by addressing more than the FWHM.

Other methods for improving the spatial resolution of the PEA system have been accomplished in other PEA systems and can be implemented and improved upon in the USU PEA system. The potential improvements include thinner sensors; improved pulse generators with better shaped pulses, shorter pulse duration, and faster rise times; and higher speed oscilloscopes. Thinner sensors can be purchased or manufactured, high speed oscilloscopes are commercially available, and better pulse generators are typically custom built.

7.2.3 Validation of AF-NUMIT3

Through comparison of PEA measurements of PTFE and PEEK irradiated with various incident electron energies, the deep dielectric charging code AF-NUMIT3 has been validated. This also brought to light a few key aspects to improve the code, such as careful consideration of RIC and DRIC effects.

For a better comparison and validation of AF-NUMIT3, simulations should be modeled to more accurately reflect the reality of the experiment. That is, use the actual measured time varying flux, rather than an average flux, and include effects of DRIC as the samples move into and out of the beam.

The overall agreement of experiment with AF-NUMIT3 simulations is encouraging. The key feature of the embedded charge distributions, the peak deposition depth, was in agreement to within a few microns for a majority of measurements. The determination of the absolute charge magnitude via PEA measurements proved to be less reliable, as can also been seen with the validation measurements in Section 5.3. One of the other major differences between PEA data and AF-NUMIT3 simulations is the spatial resolution, as well as the effects of attenuation and dispersion. AF-NUMIT3 spatial resolution is simply limited by the user-defined spacing of the nodes in the simulation, while the spatial resolution of the PEA system is much lower.
In comparing PEA measurements to AF-NUMIT3 simulations, it was discovered that PEA data could be improved further through optimization of PEA calibration and data processing for more accurate results. This includes optimizing the filtering and deconvolution of data, as well as taking into account acoustic dispersion and attenuation of the signal.

7.2.4 PEA Relevancy to Spacecraft Charging

PEA measurements of PEEK and PTFE irradiated with low energy incident electrons (10-50 keV) demonstrated the utility and limitations for the PEA method in the context of spacecraft charging. With classical PEA measurements, useful measurements are limited to $\gtrsim 30$ keV, depending on the material. This is still quite useful for a large portion of the most critical charging energy range of electrons between 10 keV to 80 keV. However, it was demonstrated that a simple method, LSIP, can allow for measurements of shallow charge distributions in highly insulating samples with use of reference measurements in pristine samples. This can potentially push the PEA method to allow for measurements at the lower limit of the critical energy range most relevant to spacecraft charging issues. The method was demonstrated for $\sim 24$ keV incident electrons in PEEK and needs to be studied further for lower incident energy electrons and other materials.

7.3 Future Work

This section is divided into two parts, improvements in the PEA method and system, and proposed experiments.

7.3.1 Proposed Improvements

Improving Spatial Resolution

Improvement of spatial resolution and understanding of PEA data can be achieved through study of each moment of the measured signals in the PEA traces (interfacial and embedded charge distributions). The FWHM and the peak position has been thoroughly
vetted in this dissertation but further work is needed to characterize and extract information about the asymmetry/HWHM and the uncertainty of those parameters. This would include careful determination of the HWHM for raw, processed, and deconvolved data (separately) as well as testing for consistency of asymmetry. The consistency could be tested by measuring the sample in two orientations (flip sample over in PEA) and comparing the direction of measured asymmetry. Data presented in Section 6.1 did not seem to show consistent measures of asymmetry upon changing the orientation of the samples, but further work is needed.

Physical improvements to the PEA system should also be explored. These include: further study of acoustic coupling to mitigate multiple reflections of the acoustic pressure waves, including investigating other coupling media and sensor backing materials and configurations; creating and/or purchasing thinner PVDF sensors (Galloy et al., 2016); exploring other options for sensors that may provide higher spatial resolution or work at more extreme temperatures (Sonoda et al., 2017; Xu et al., 2014); building or purchasing a pulse generator with better shaped pulses, shorter pulse duration, and faster rise times; obtaining an oscilloscope with a faster acquisition rate; improving the electrical impedance matching of the pulse generator and sample stack (refer to Appendix A.9); and improving the electrical impedance matching of the sensor, amplifier, and oscilloscope circuit.

**Addressing Calibration Issues**

The calibration of PEA data is straightforward for the distance scale, but it has been shown that calibration of the charge magnitude is more problematic. This was demonstrated when data obtained with the USU PEA system was compared to AF-NUMIT3 simulations in Section 6.1 and compared to UPS PEA data in Section 5.3, with disagreement from a few percent to more than a factor of two. To address this, alternative methods of calibration should be explored, such as measuring a known charge distribution in the bulk of a material. This could be important as currently the signal from an interface is typically used for calibration and surfaces/interfaces may behave significantly different than bulk material.

Further inspection of the calibration factor used should be carried out as well. This
should provide for a better determination of magnitude of voltage to apply for the calibration measurements. This may include thorough measurements of materials with varying DC bias applied to be sure charge is not injected during reference measurements, as well as taking into account the effects of the high voltage pulse on the resulting PEA signal (Chen et al., 2006).

**Development of Open PEA System**

An open PEA system (Griseri et al., 2008) should be developed and built in the USU MPG lab. Many of the parts have already been obtained and the same basic concept as the classic PEA system is employed.

The major differences going from ambient to vacuum will be: higher possible voltages (less arcing), top/excitation electrode will not be in contact with the sample (sample will be glued to the bottom electrode) (Gibson et al., 2018; Griseri et al., 2003), charge injection via electrode charging or high energy electron sources, potential for intermittent surface potential measurements via surface voltage probe, and simultaneous PEA measurements during irradiation.

**7.3.2 Proposed Experiments**

**Validation of Charging Codes**

An important application of PEA measurements is to further validate deep dielectric charging codes to inform and mitigate spacecraft charging issues. Further validation of AF-NUMIT3 should involve measurements varying the effects of RIC/DRIC. This can be accomplished in several ways. Irradiation can be done with different electrode configurations, as grounded surfaces provide the electric field necessary for charge dynamics through induced charge. The difference between continuous and pulsed irradiation schemes should be investigated. The effects of pulsed irradiation with irradiation duration above and below typical RIC rise and decay times (typically 10-100 s) should be tested (Gillespie, 2022; Tyutnev et al., 2019). The effect of sample temperature during irradiation should be in-
vestigated, noting that typically the PVDF sensor limits the temperature range a PEA system is capable of. Higher temperature has the typical effect of increasing dark conductivity and RIC effects. Comparison of measurement to the spectral irradiation capabilities should be carried out either with a spectral electron source (e.g. Sr$^{90}$ or multi-energy gun) or by sequentially irradiating with various electron energies (Riffaud, 2016). These types of measurements will serve to answer questions about material behaviors such as whether deposition/transport effects are linear with energy and are there synergistic effects such as with irradiation and temperature (Rival et al., 2020).

These results should be compared to other charging codes as well such as JPL NUMIT and DICTAT, as outlined in previous sections. Comparing to many different models may be more beneficial than focusing on one, as this may lead to insights due to differences in experimental data, simulations, and simulation approaches.

**Improving Shallow Charge Distribution Measurements**

Efforts should be made to improve upon the method shown in this dissertation for measuring shallow charge distributions with a classic PEA system. This would involve irradiating a sample with various energies of low energy electrons (<30 keV) with higher fluence than was used for the proof-of-concept measurements presented here. The incident electron energy limit would then appear to be governed by the resolution of the deposition depth. Referring to Fig. 1.2, with a precision in peak position on the order of $\lesssim 1$ µm, the lower limit for incident electron energy would be $\lesssim 10$ keV. The protocol for the measurements should be further optimized and justified as well.

It may also be possible to obtain shallow charge distribution measurements by gluing together samples to remove the response of the interface and to only measure the embedded charge. This would involve measuring a pristine sample to find the precise location of the sample surface, gluing the irradiated sample to the pristine sample, and then measuring the multi-layer sample. An example with pristine samples is given in Appendix A.10.
Varying Dose and Dose Rate: Charge Deposition

The dose and dose rate can have a large effect on the resulting charge distributions. The nuances of these effects should be further investigated.

The dose can eventually lead to damage in the material, changing the charge dynamics. The dose rate directly affects the conductivity via RIC. Consider a constant total fluence (constant amount of charge deposited). At one extreme, the charge could be deposited slow enough such that RIC is negligible. This would likely deposit charge closely following the deposition of energy as a function of depth. At the other extreme, with a very high dose rate, RIC will cause the conductivity of the irradiated volume of the sample to increase such that the charge will essentially be mobile. In this case, if there is a grounded surface at the rear of the sample (opposite the irradiated surface), the embedded charge will induce charge at the grounded surface creating an electric field within the material. This electric field will drive the charge towards the grounded surface. The charge migration will stop either when RIC/DRIC wears off or at a depth where RIC is negligible. In broad strokes, irradiating with a higher dose rate should drive the charge deeper into the sample, provided there is a grounded rear surface. The charge dynamics will differ if there is an electrode on the irradiated surface of the sample or if the geometries are more complex.

It should also be investigated what happens when samples are irradiated sequentially with different electron energies (Riffaud, 2016). In essence, this would be exploring the effects of DRIC on charge dynamics.

Varying Dose and Dose Rate: Aging

The effects of total ionizing dose, dose rate, and source of dose should be investigated on the aging effects in polymeric samples. Damage may occur at varying amounts of total ionizing dose (and dose rate). For example, damage to PTFE may begin to occur in the range of $\sim 10^3 \text{–} 10^5 \text{ rad}$, while for PEEK the range may be $\sim 10^6 \text{–} 10^7 \text{ rad}$ (Shulman and Ginell, 1970; Von White II et al., 2013).

The differing effects of irradiation by electrons, x-rays, and gamma rays is currently being explored by the MPG at USU in collaboration with UPS, but has not been presented
Further investigation of the dose rate appears to be of importance as differing effects appear to be apparent. That is, for a constant total ionizing dose, preliminary results seem to indicate that a higher dose rate appears to have a more substantial effect on the charge transport properties of a material.

**Constant Voltage Conductivity and $Q(t)$ Measurements**

The PEA method provides information about the embedded charge distributions. This information can be used in conjunction with complementary measurements such as the constant voltage conductivity method. To further understand constant voltage conductivity measurements, a measurement with PEA could be replicated. This could allow insight into the various features of the constant voltage conductivity current/conductivity versus time plots (Wood et al., 2018). The evolution of the charge distribution, as well as the equilibrium charge distribution reached for the dark conductivity measurement, could potentially be measured with the PEA method. Comparison of the constant voltage conductivity method to the “leaky capacitor” conductivity measurement method (Andersen et al., 2022) could potentially be explored as well.

A question of whether the PEA system is sensitive enough to measure the charge distributions must be addressed. To address this question, the limits of the charge resolution must be determined. From (Dennison and Pearson, 2013), the charge resolution of the PEA method is typically $\gtrsim 100$ nC/cm$^3$ ($\gtrsim 0.1$ C/m$^3$). For a $20 \mu$m thick charge layer, this is a limit of $10^{-5}$ C/m$^2$. Referring to the PEA data presented in Fig. 6.7, the samples irradiated with the nominal 10 keV incident electron energy were measured to have $\sim 10^{-5}$ C/m$^2$ (Gibson and Dennison, 2023b). This shows that the PEA method is able to measure a charge distribution on the order of this magnitude.

An estimate of the charge within a sample during a constant voltage conductivity measurement can be determined. Using data from (Wood et al., 2018) for PEEK, the average charge in the sample is determined at the transit time when all recorded currents are believed to be displacement currents (i.e. injected charge that has not left the sample). Using the measured conductivity (at the transit time), thickness, transit time, and applied
voltage from (Wood et al., 2018) [as well as electrode area from (Dekany et al., 2013)], the average charge within the PEEK sample was determined to be $\sim 0.12 \text{ C/m}^3$. This is very near the limit of the PEA charge resolution. However, it is possible the charge is much more localized, rather than equally distributed through the sample thickness. This is also a lower bound as the conductivity used for the calculation is at the transit time, which is $\sim 2\times 10^{-20} \Omega\text{cm}$. This quick calculation demonstrates that this idea should at least be given more thought and more thoroughly investigated. It may also be possible to implement a constant voltage conductivity type measurement concurrently with the PEA system, but this would require substantial further work and consideration.

Another method that has been used to concurrently to complement PEA measurements is the $Q(t)$ method (Ren et al., 2021). From (Ren et al., 2021), the $Q(t)$ method “…can obtain the whole charge information including the induced surface charge, the internal space charge, and the leakage charge of the insulation.” Further investigation to the utility and feasibility of implementing the $Q(t)$ method should be explored.

**Charge Dissipation**

Several interesting experiments could be carried out to study the effects of charge dissipation via introduction of environmental factors such as light sources, radiation, and increased temperature. For example, samples with embedded charge could be exposed to various light sources and light intensities to study photo-discharging and -charging of samples. Samples could also be heated to study charge dynamics at elevated temperatures and to be sure vacuum bake out procedures are sufficient for samples to dissipates any embedded charge.

**Long Time Charge Migration**

In dielectrics, charge moves very slowly. It would be quite interesting to study just how slow the charge moves. This could be accomplished by placing grounded electrodes on different surfaces of samples with embedded charge and simply leaving them there for long periods of time between PEA measurements. PEA measurements would need to be
obtained before and after, and perhaps periodically. This would also provide a measure of the conductivity and the evolution of the charge distribution across the dielectric with time, similar to the “leaky capacitor” method of measuring conductivity (Andersen et al., 2022).

**Critical Transitions**

There are critical transitions in the charge transport features. There is a dispersive to non-dispersive transition with increased temperature and/or applied electric field. This should be studied for materials such as LDPE and PTFE, which are well studied. It has been observed in the literature that a packet-like charge transport regime can occur at high fields or high temperature (Matsui et al., 2005; Zhang et al., 2017), signifying the transition from dispersive to non-dispersive transport. It would also be quite interesting to compare constant voltage conductivity and RIC measurements as a function of temperature to compare the dispersion parameter extracted from those experiments to the dispersion parameter measured in the PEA system. The dispersion parameter defines the transition of dispersive to non-dispersive transport. For a detailed discussion of the dispersion parameter and the relevance to several electrical characteristics of materials, refer to (Gibson, 2019).

**Partial Aging**

It would be interesting and perhaps beneficial to spacecraft charging applications to study the effects of partial aging of materials. It would also be interesting to create a nonlinear or periodic aging gradient in the sample as well, as non-uniform aging of materials in the space environment is likely due to the varying energetic and spatial distribution of incident radiation. Partial aging of a sample can occur when the ionizing radiation does not pass through the sample completely. This can occur via irradiation with lower energy electrons. To mitigate arcing and sparking of the sample during irradiation, a vapor deposited electrode could be introduced to the front surface of the sample and a voltage could be applied to remove the electrons after they are deposited in the sample. A high enough flux would be necessary to ensure that RIC is sufficient to mitigate charge from building up high enough to reach breakdown.
Partially aged samples could be tested by applying a DC bias in the classic PEA system to study charge dynamics effects at various levels of aging. It may be possible to create a gradient of aging in the sample. This could be accomplished by irradiating with various incident electron energies to give different depths of the sample different doses. Then the dosing of the sample could be controlled as a function of depth. If there was a gradient in the density of defect states caused by the partial aging, then it is possible that charge would preferentially migrate in the direction of higher defect density. That is, the charge may migrate towards the more damaged area of the sample. However, charge must be present in the sample to gauge the dynamics. This could be accomplished as the area of the sample near the irradiated surface would have the highest dose/most aging/highest density of defect states. It is suggested that the final incident electron energy would be a high energy such that it is deposited deep in the bulk of the sample. Then, perhaps over time one could see the charge migrate towards the surface. Some calculations need to be made to determine how fast things will move and the order of magnitude of the effect. It may become necessary to “freeze” the charge in place so that DRIC effects do not dissipate the charge too quickly. This may be accomplished by lowering the temperature of the sample, thereby reducing the conductivity. It may also be possible to irradiate with a lower flux such that RIC does not cause the charge to become mobile without the aid of increased temperature or applied DC bias, which may be applied in the PEA fixture.

**Multi-layer Samples**

PEA measurements of multi-layer materials should be further investigated as there are a plethora of applications. Interfacial phenomena can drive charge transport characteristics and electrical properties of materials, from nanoparticle/bulk to crystalline/amorphous region interfaces (Dang *et al.*, 2013; Tan, 2020; Upadhyay and Reddy, 2017). This is an active area of research where nanoparticles/nanofillers are being implemented in an attempt to tune and improve material properties.

To study the effects of interfaces, it may be beneficial to conduct a simple study. Select a common bulk material and nanoparticle. Measure the charge transport properties of the
bulk material and a bulk sample comprised of the same material as the nanoparticle. Then, create a multi-layer sample of the two bulk materials. Create samples that contain varying sizes of particles embedded in the bulk material, eventually creating the nanoparticle filled material, measuring the charge transport properties at each step. A systematic study like this may aid in understanding the role of interfaces on charge transport properties.
References


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APPENDIX A

PEA Characterization

Each component of the PEA system (refer to schematic diagram in Fig. 3.1) was tested and verified to eliminate possibilities that they introduced errors or resulted in failures. This includes testing connectors, cables, terminators, and attenuator with a vector network analyzer to determine return loss (power loss); testing electronic components of the PEA system including the oscilloscope, HVPG, HVDC power supply, amplifier, and amplifier power supply to be sure they are all working properly and to mitigate cross talk; testing the impedance matching network and cable lengths to determine effects on PEA output; testing of different coupling media; and determining effects of differing materials and material thicknesses, as well as differing sensor thicknesses.

A.1 Return Loss (Power Loss) in Cables and Connectors

The return loss was measured for various cables and components of the PEA system with an Agilent AT-N9914A FIELDFOX Handheld RF Analyzer, which was borrowed from the electrical and computer engineering department store at USU. The results are shown in Figs. A.1-A.4. The FieldFox has N-type connectors and is capable of measuring in the frequency range of 30 kHz to 6.5 GHz, although most measurements reported here were only obtained up to 2.5 GHz, as 6.5 GHz is far out of the frequency range of interest for the \( \geq 0.5 \) ns pulse widths and \( \geq 9\mu \text{m} \) sensor thickness. The PEA system measured frequency range is at the most \( \sim 1 \) GHz. However, the current system contains frequency information more typically only up to a few hundred MHz’s. That is, if you perform an FFT of a PEA measurement, the magnitude will become negligible above a few hundred MHz’s (ignoring high frequency noise). Note that higher frequency content (and therefore higher spatial resolution) is possible with improvements to the PEA system e.g. thinner sensors and shorter pulses. The FieldFox is calibrated with a standard calibration unit [Agilent 85515A...}
Figure A.1: Return loss for various PEA components. Plotted are return loss for (a) a perfect load (50 Ω wide band) and (b) the PEA system as a load. Return loss of the 8 dB attenuator, 50 Ω terminator, standard calibration load, and PEA cable were tested.

Cal Kit Type-N(f) 50 Ω DC to 9 GHz.

The return loss is defined as

$$RL(dB) = 10\log_{10}\frac{P_{\text{incident}}(\omega)}{P_{\text{reflected}}(\omega)},$$

(A.1)

where $RL$ is the return loss, $P_{\text{incident}}$ is the incident power, and $P_{\text{reflected}}$ is the reflected power. Generally, a high return loss is preferred and a lower return loss indicates there...
Figure A.2: Reflected power of PEA system and components. Plotted are reflected power with (a) an ideal load (50 Ω wide band) and (b) the PEA system as a load. The reflected power of the 8 dB attenuator (abbreviated attn. in legend), 50 Ω terminator, standard calibration load, and PEA cable are plotted.

will be reflections. Note that the return loss is directly related to the standing wave ratio (SWR). That is, the measurements are equivalent (like measuring inches instead of meters, sort of). The calibration standard is considered good if it has a return loss >35 dB. The standard calibration in our case was ∼40 dB.

In Fig. A.1, the return loss is shown for the Barth Electronics High Voltage 8 dB
attenuator [grey line in (a)], the Bracke BM11073 50 Ω high power terminator, and the PEA system [refer to (b)]. When testing the PEA system itself, the vector network analyzer was connected directly to the N-type connector at the top of the PEA excitation electrode assembly where the HVPG is usually connected. This is essentially testing the return loss for the electrical impedance matching network and the sample. For these tests, the PEA system had a 250 µm PMMA sample, T-pad impedance matching configuration (see Fig. A.28, and with or without a 50 Ω terminator at the connection to the PEA system. The “PEA Cable” refers to the 6 ft cable between the HVPG and the PEA electrode housing. All terminators, cable, and attenuator combinations (as indicated in the legends of Fig. A.2) tested were determined to have a $RL \gtrsim 35$ dB, which means they will not cause an issue. The 8 dB attenuator observed a 16 dB return loss [grey line in Fig. A.1(a)], but this makes sense as a reflected signal will pass through the attenuator twice to obtain a total loss of 16 dB.

The reflected power is much less than 1% for the PEA cable, 50 Ω terminator, and 8 dB attenuator, see Fig. A.2. Referring to Fig. A.2(b), the reflected power when measuring the PEA system as a load is less than ideal. Ideally, the PEA system would absorb all power and there would be a very high return loss. This is clearly not the case. The orange curve shows the PEA system in the T-Pad configuration (see Fig. A.28), with virtually all power reflected. When using a 50 Ω terminator this is improved but only for relatively low frequencies (however these low frequencies are in our range of interest). This is why it was opted to remove the T-pad circuit and use only a 50 Ω terminator for matching.

The reflected power was calculated from the return loss. The reflected power is related to the return loss in dB, to provide easier interpretation of these measurements, as

$$\text{Reflected Power} = (10^{-RL/10})^2.$$  \hspace{1cm} \text{(A.2)}$$

Recall that RL is the return loss. Note that the reflected power is equal to the reflection coefficient squared.

Figure A.2 displays the reflected power of each of the tested components. The PEA
Figure A.3: Reflected power of various PEA components and sections. The reflected power was measured for many different PEA components and are plotted versus frequency. HVTapoff refers to the 100:1 high voltage tap-off used to probe the high voltage pulse. CalCable is a calibration cable included with the FieldFox, as well as the CalStd which is the calibration standard. The rest of the measurements are various combinations of cables and connectors used in the PEA system.

Figure A.4: Reflected power for cables and terminators. Measurements of the reflected power for various terminators and cables are plotted versus frequency. A dashed line indicates the 1% reflection mark.
system had a 250 um PMMA sample, T-pad impedance matching configuration, and with or without a 50 Ω terminator at the connection to the PEA system. Reflections are much less than 1% for many of the components, but neared 100% reflection for the PEA system as a whole. This is due to poor electrical impedance matching of the HV pulse with the sample. This is difficult to do, especially in a broad frequency range, as the sample is a capacitive load, and therefore, the impedance is a function of frequency.

The reflected power for the majority of the rest of the PEA cables and connectors can be seen in Fig. A.3. Various combinations of the components as well as cables and connectors between different parts of the PEA system were tested. The overall performance is good, although the reflected power approaches 1% as frequencies increase to 1 GHz. The rest of the components [cables between battery supply/oscilloscope, and battery supply/PEA system (with and without SMA tee); various 50 Ω terminators; and a tapoff] can be seen in Fig. A.4. From these data, its evident that not all 50 Ω terminators are created equally. BNC 50 Ω terminators 4 (yellow curve) and 2 (brown curve) perform much worse than the other two. All other components had acceptable reflection, under 1%, in the frequency range of interest. Terminators 1 and 2 were then employed in the PEA system, discarding 2 and 4.

A.2 Oscilloscope

The oscilloscope used in the USU PEA system is a Lecroy HD4104 HD4096 Oscilloscope with 2.5 GS/s sampling rate and 4 channels. According to the Lecroy manual, the oscilloscope self calibrates at each start up, but can also be manually calibrated if desired. “Manual” calibration is still done internally in the oscilloscope and calibrations are good for ±5 K of the temperature during calibration. The temperature variation in the lab has been found to be approximately ±2 K (Boman et al., 2019; Wood et al., 2018). It is recommended that the oscilloscope is allowed to warm-up for 20 minutes prior to use.

The oscilloscope frequency response was also tested with a TENMA RF Generator. The results of the tests are plotted in Figs. A.5 and A.6. No issues were detected in the testing of the oscilloscope functioning.
Figure A.5: Oscilloscope testing with 150 MHz signal. Plotted is the Lecroy oscilloscope response to (a) 150 MHz sine wave (note the input signal is imperfect) and (b) FFT of the measurement.
Figure A.6: Oscilloscope testing with 1 MHz signal. Plotted is the oscilloscope response to a (a) 1 MHz sine wave and (b) FFT of the measurement.
Figure A.7: Schematic of high voltage pulse generator test circuit. Note that measurements were done with and without the attenuator (not shown) in place (between HVPG and tap off).

Figure A.8: Accuracy of FWHM settings. Comparison of the measured FHWM and the pulse width setting on the pulse generator.

A.3 High Voltage Pulse Generator

The pulse generator used for measurements in the PEA system is a FID GmBH FPG 2-10PM5L high voltage pulse generator (HVPG). The amplitude can be varied from 1 kV to 2 kV, but lower amplitude is achieved with an 8 dB attenuator. The pulse width is adjustable from 0.5 ns to 5 ns. The pulse repetition rate is also adjustable, but has been left at 20 Hz. Refer to Fig. A.7 for a schematic of the test set-up to probe the HVPG. Results of the testing are shown in Figs. A.10-A.18.

The HVPG was characterized with use of a 100:1 tap-off (Barth Electrics Inc. 245-NMFP-100, N-type) and a 50 Ω load. The 50 Ω load used was a set of 100 Ω resistors (Vishay Dale ROx-3/4N 100R0 FN 1746) in parallel, soldered to an N-type bulkhead (UG58 A/U).
Figure A.9: Accuracy of amplitude settings. Comparison of the measured amplitude and the amplitude setting on the pulse generator. Note that data was not corrected for attenuated data points at 1, 1.6, and 2 kV. Lower amplitude points correspond to shorter pulse widths, as amplitude decreases with decreasing pulse width.

Figure A.10: HVPG bandwidth versus amplitude. Comparison of the -3 dB bandwidth and the measured amplitude of the pulse. The color indicates the measured FWHM of the pulse.

Testing with other 50 Ω loads (such as Termaline Coaxial Resistor from Bird Electronics, Model 8401) were executed and in results with these alterations were negligible.

The oscilloscope LeCroy (Model HDO 3104) with 1 GHz and 2.5GS/s capabilities. The oscilloscope was used in RIS (random interleaved sampling) mode to get the most detailed/high resolution measurements of the pulses from the HVPG.
Measurements of output of the HVPG were measured for 1.0, 1.2, 1.4, 1.6, 1.8, and 2.0 kV and 0.5, 1.0, 1.5, 2.0, 2.5, 3.0, 3.5, 4.0, 4.5, and 5.0 ns pulse width, and every combination thereof. All these measurements were made without an attenuator. Measurements at 1.0, 1.6, and 2.0 kV, and 0.5, 2.5, and 5.0 ns were made with an 8 dB attenuator between the tap-off and the high voltage pulse generator. The FWHM and amplitude were determined for each pulse. These were compared to the expected pulse width and amplitude from the settings on the pulse generator; refer to Fig. A.8. The linear fit (black line) indicates a strong correlation between the pulse width and the measured FWHM, which is as expected. The amplitude measured was compared to the voltage setting as well; refer to Fig. A.9. A 1:1 black line was shown for the ideal output for reference. There is good correlation for both settings, aside from a lower output amplitude for the short pulse widths. The amplitude of the 0.5 ns pulse suffers the worst.

For each pulse measurement the FFT of the PEA voltage trace was determined. The bandwidth of the pulse was determined at the -3 dB level, that is, the point at which the FFT amplitude falls to half of the maximum value. The bandwidth is plotted against amplitude in Fig. A.10. There is a strong correlation between bandwidth and measured amplitude. It should be noted that the pulse width (which is directly proportional to the
Figure A.12: 2 kV FFT example of HVPG measurement. An example of the FFTs for the measured pulses. Shown is the frequency spectrum of the measured 2 kV pulses. The broadest spectrum is the 0.5 ns and the narrowest (lowest) is 5 ns.

Figure A.13: Normalized 2 kV example HVPG measurement. Shown is all measurements at 2 kV for every pulse width measured. The pulses have been aligned by their rising edges and amplitude normalized to one.

FWHM) is inversely proportional to the bandwidth. The shorter the pulse, the larger the bandwidth. This is to be expected and the reason why a shorter pulse width is necessary for higher spatial resolution PEA measurements. Note that the points in Fig. A.10 data points for less than 0.5 ns were not analyzed properly and should be ignored, as the parameters were extracted with a procedure (automated analysis code) in Igor Pro and did not process some lower amplitude/noisy data properly.
Figure A.14: Normalized 2 kV FFT example of HVPG measurement. An example of the FFTs for the measured pulses. Shown is the frequency spectrum of the measured 2 kV pulses. The FFTs have been normalized to one. The broadest spectrum is the 0.5 ns and the narrowest (lowest) is 5 ns.

Figure A.15: 5 ns example HVPG measurement. Shown is all measurements with 5 ns pulse width for every pulse amplitude measured. The pulses have been aligned by their rising edges.

Representative results of the measured pulse are shown in Fig. A.11 for 2 kV amplitude and various pulse width settings with corresponding FFTs plotted in Fig. A.12. See Figs. A.13 and A.14 for normalized plots of the 2 kV results and corresponding FFTs, respectively. Similar plots are shown for measured pulsed keeping the pulse width fixed at 5 ns and varying the amplitude instead. Figs. A.15 and A.16 plot the untreated data and Figs. A.17
Figure A.16: 5 ns FFT example of HVPG measurement. An example of the FFTs for the measured pulses. Shown is the frequency spectrum of the measured 5 ns pulses.

Figure A.17: Normalized 5 ns example HVPG measurement. Shown is all measurements with 5 ns pulse width for every pulse amplitude measured. The pulses have been aligned by their rising edges and amplitude normalized to one.

and A.18 plot normalized data. Note that the data were checked for reproducibility by measuring the same pulse before and after all of the measurements were taken and spot checking various measured pulses days after the original measurements. No substantial variation was observed.

The plots with fixed voltage (2 kV) show that there is a light decrease in amplitude as the pulse width is decreased. The attenuated data (indicated by abbreviation attn. in the
Figure A.18: Normalized 5 ns FFT example of HVPG measurement. An example of the FFTs for the measured pulses. Shown is the frequency spectrum of the measured 2 kV pulses. The FFTs have been normalized to one.

Legends of the figures) are of course lower amplitude, but when normalized do not show any signs of difference or degradation in signal quality. The FFTs reveal that narrower pulse widths result in higher frequency content, which is to be expected.

The plots with fixed pulse width (5 ns) demonstrate that there is very little variation in the quality and shape of the pulse with changing amplitude. The FFTs reveal that the frequency content is unchanged with amplitude, as is expected.

A.4 12 V Power Supply for Amplifier

The amplifier for the PEA system requires a 12 V DC power supply. Ripple in the power supply voltage can translate to significant ripple/noise in the PEA measurements if the ripple is too large. There are two different power supplies that have been considered and used. The first voltage source is a 12 V DC power supply (Mean Well MDR-10-12), powered by an AC voltage source (i.e. wall outlet). The other is a 12 V battery voltage supply. The USU-MPG has utilized batteries as a low noise/ripple voltage source in many sensitive electronic measurement apparatuses in the lab (Dekany et al., 2013). Measurements of the ripple and noise features are shown in Figs. A.19-A.21.

The input and output grounds of the AC-driven power supply are isolated. That is,
Figure A.19: 12 V battery power supply noise. The noise floor and a “noise pulse” are depicted for a (a) long duration and (b) zoomed-in on the noise pulse.

The ground that is part of input power to the power supply itself is isolated from the ground of the 12 V DC output the power supply provides. This isolation is good for reducing noise/ripple.

Considering the noise floor and ignoring the voltage pulses (which are random noise and can be averaged out during PEA measurements), the output DC voltages were measured to be 12.6636 ± 0.0010 V and 11.7987 ± 0.0017 V for the battery and AC-driven supplies, respectively. Note that the noise pulses plagued both power supplies equally. Both power supplies showed correlated noise as well. This was only seen when the PEA system was on. The spikes are measured at the beginning of a PEA excitation pulse and slowly damps out over approximately 1 µs, very likely due to induced signals from the HVPG. These large pulses are measured in the PEA voltage traces as well, but are seen at times before the desired PEA data. These larger voltage spikes are evident at a rate of 60 kHz, with frequency
Figure A.20: 12 V AC-drive power supply noise. The noise floor and a “noise pulse” are depicted for a (a) long duration and (b) zoomed-in on the noise pulse.

content of up to about 25 MHz. The amplitude of these voltage spikes are much larger than the random ripple, sometimes with amplitudes of several 100’s of millivolts. However, as the inclusion of a voltage spike in any given measurement is random, they should not have an appreciable effect on the PEA data as PEA measurements are an average of 1000 or more traces and not measured at a rate of 60 kHz. The voltage spike is essentially a modulated 25 MHz sine wave repeating at approximately 60 kHz. Apparently, this type of noise can sometimes come from Ethernet cables. The frequency 60 kHz is also a common switching speed. The actual source of this noise was never found.

Further inspection of the “noise pulses” showed that the frequency content was around 20 MHz and 90 MHz. Floor noise and noise pulse are plotted in Figs. A.19 and A.20, with corresponding FFT’s shown in Fig. A.21, for both the 12 V battery supply and the AC-driven supply.
Figure A.21: 12 V power supply noise FFT. The FFT for the measurements of the noise floor and a “noise pulse” are depicted as a function of frequency for the (a) battery supply (see Fig. A.19) and (b) AC-driven supply (see Fig. A.20).

In conclusion, although there is a decreased ripply using the battery power supply, either power supply should be sufficient for PEA measurements.
A.5 High Voltage DC Power Supply

The high voltage DC (HVDC) power supply (Glassman FJ10R12 DC source 0-10 kV) was tested for its precision and accuracy. The accuracy of the power supply was tested by measuring the applied voltage with a Fluke 80K-40 HV probe. The probe made connection with the PEA circuit between the excitation electrode and the coupling capacitor/current limiting resistor (refer to Fig. 3.1) to put the probe in parallel with the sample and effectively measure the voltage drop across the sample. However, the probe (1000 MΩ) is designed to work with a multimeter of 10 MΩ resistance. The oscilloscope was set to a 1 MΩ input resistance (with the other option being a 50 Ω input resistance). This requires a correction factor of 1.901, and since the probe is a 1000:1 voltage divider, the scaling factor for the measurement is 1901:1. Refer to the manual for the Fluke probe for more details (Fluke, 1997). This is indicated on the y-axis of Fig. A.22(a). The resistance of the sample in the PEA system during measurements [250 μm polymethyl-methylacrylate (PMMA)] was estimated to be $3 \times 10^{14} \, \Omega$. Measuring the voltage at 5 kV and 7 kV, the voltage was determined to be only 96% of the HVDC supply setting value, with a standard deviation of the measurements of 1.6 (0.03%) and 2.6 V (0.04%), respectively.

For a more accurate ripple measurement (precision), the oscilloscope was set in AC coupling mode. This showed the noise floor to be substantially lower at less than $\pm 1 \, \text{mV}$. There were occasional “noise pulses” with amplitudes of $\pm 8 \, \text{mV}$. This is a miniscule precision error considering the applied voltage is in the kV range. Plots showing the DC voltage, noise floor, and noise pulse for the HV DC supply are shown in Fig. A.22. A DC-coupled measurement showing the value of output of the HVDC power supply is shown in Fig. A.22(a). Figures A.22(b) and (c) show AC-coupled measurements depicting noise pulses for zoomed-in and zoomed-out perspectives, respectively. The frequency content of the noise in the HVDC power supply output is shown in Fig. A.23. Several different noise sources were determined such as Aggie Radio (89.5 FM), DaystarTV (207 MHz), band 71 "LTE" (600 MHz cell phone band), 700 MHz cell phone band, and 800 MHz cell phone band.
Figure A.22: High voltage DC power supply testing. Plotted are the (a) DC coupled measurements, (b) zoomed-in and (c) zoomed-out noise pulse measurements for the HVDC power supply.
Figure A.23: Frequency spectrum of high voltage DC power supply noise. Plotted are the FFT’s of various HVDC measurements to determine frequency content of the noise.

A.6 Amplifier

The amplifier in the PEA system (Wenteq ABL0300-00-4030) was tested. This was done by applying a high frequency signal of known amplitude to the amplifier and measuring the resulting output of the amplifier. The input signal was provided by an RF generator (Tenma RF Generator 72-585). Signals of 90 MHz and 150 MHz were used, as these are two key frequencies within the range of the frequency content of PEA measurements, refer to Fig. A.27(b).

The results of this test are shown in Fig. A.24, where the input signal, output signal, and their FFT’s are plotted. The distortion of the signal is very minimal and it should be noted that the input signal from the RF generator had a lot of jitter. Averaging of the signals (both the input and output of the amplifier) was necessary for good results.

The gain of the amplifier was also tested. This was done by dividing the output signal by the input signal. It was also done in frequency space by dividing the FFT of the output by the FFT of the input. These are shown in Fig. A.25. The gain as measured in real space was $59\pm110$ and $69\pm57$ for 90 MHz and 150 MHz signal measurements, respectively. The average gain from 0-1 GHz for the 90 MHz signal data was $55\pm22$ and for the 150 MHz signal data the gain was $62\pm12$. The high errors were due to spikes in the gain, as seen in Fig. A.25
Figure A.24: RF amplifier test. The input and amplified output signals of the amplifier are depicted for (a) 90 MHz and (b) 150 MHz. The corresponding FFT’s are shown in (c) and (d).
Figure A.25: Amplifier gain. The signal gain of the amplifier are shown for RF signals with peak frequencies of (a) 90 MHz and (b) 150 MHz. The respective gains as a function of frequency are shown in (c) and (d).
A.7 Ultrasonic Testing of PEA System

An experiment was devised to test the “sensor side” of the PEA system. This was designed to test everything from the sample to the oscilloscope as a whole. The test applied an ultrasonic signal from a 20 MHz transducer via an ultrasonic pulser/receiver system (UTEX Scientific Instruments Inc., Model UT340). The transducer was placed on the sample in the PEA system in place of the high voltage electrode. In this way, an ultrasonic measurement of the sample can be made simultaneously with the UT340 system and the PEA system, and then the results can be directly compared.

The sample used for this test was a nominally 127 µm thick film of polyimide (PI, specifically Kapton HN™). The measured signal at the UT340 and the signal measured by the PEA system are compared in Fig. A.26. In Fig. A.26(a), the blue curve is the signal measured by the UT340 and the red curve is that measured by the PEA system. There is a clear “trigger” signal near 0 s in the UT340 data, labeled as a blue 1. There is then a $\sim 4.27$ µs delay before the first signal from the transducer/sample interface is measured in the UT340, labeled as a blue 2. The corresponding first signal measured by the PEA system (red 1) occurs at $2.55 \mu s$ after the trigger (blue 1). The time it takes for the signal to be first measured by the PEA system is a sum of the time it takes the acoustic signal to traverse the delay line ($\sim 2.135 \mu s$), sample (thickness $\sim 127 \mu m$ and speed of sound $\sim 2140 m/s$, delay is $\sim 0.059 \mu s$), and sample plate (thickness $\sim 2 mm$ Al and speed of sound $\sim 6420 m/s$, delay is $\sim 0.31 \mu s$), as well as the time it takes the electrical signal to pass through the PEA system (two coaxial cables with total length $\sim 5 ft$, with signal traveling at 0.7 times the speed of light, delay is $\sim 0.0069 \mu s$), for a total of 2.51 µs. This estimate is remarkably close to the measured value of 2.55 µs.

Multiple ring-downs can be seen in both the PEA and UT340 data, separated by 4.27 µs, as the exciting signal bounces back and forth in the delay line. This is The start of each of these signals are indicated by red and blue 1’s and 2’s. Now that we have our wits about us with the data, we can take a closer look at the measurements. A similar effect can be seen due to multiple reflections in the Al electrode. Referring to Fig. A.26(a), the red 1,
Figure A.26: Ultrasonic testing of the PEA system. The results of an ultrasonic test of the PEA system is plotted. The response of the PEA system due to excitation by 20 MHz transducer is compared to a pulse-echo measurement. This is plotted for (a) an unaligned long time frame and (b) a shorter time window with data aligned.

1a, and 1b show ring downs due to multiple reflections in the Al ground electrode (\( \sim 0.6 \) µs between peaks, consistent with \( \sim 6420 \) m/s and \( \sim 2 \) mm thickness). Referring to Fig. A.26(b), the plot has aligned the first signals of the measurements (refer to red 1 and blue 2 in Fig. A.26) of the PEA and UT340 system for easy comparison. The traces were aligned by the center peak of the multiple reflections in the sample due to an interesting difference in signal for the initial measurement packet with the largest amplitude. Notice the opposite polarity of the UT340 data and an additional leading peak not shown in the PEA data. The
subsequent ring-downs (from the acoustic signal reflecting back and forth off the interfaces of the sample) are in much better agreement. Although there is some slight distortion between the signals from the two detectors, they are clearly very well correlated. Note that an attenuation coefficient could be attempted to be extracted for a material with data like this (or any PEA data with multiple reflections). However, it is complicated due to the fact that the reduction in amplitude of the reflected signals is not solely due to attenuation but also due to transmission/reflection at various interfaces.

This validation of the PEA system in Fig. A.26 shows that the “sensor side” of the PEA system is working properly together as a whole. It is unlikely that anything within this tested “sensor side” part of the system is causing any significant issues in the PEA measurements. This ultrasonic PEA testing approach could provide a useful tool for troubleshooting should there be damage or modifications to the PEA system.

### A.8 Sensor Thickness Effects

The effect of the sensor thickness on PEA traces, in particular peak width and shape, was investigated. Measurements of 250 µm thick PMMA were made in the PEA system using metallized PVDF sensors of nominally 9, 28, and 52 µm thicknesses. A 2 kV 2 ns pulse was used at a repetition rate of 20 Hz and 10 waves were averaged for each measurement. No DC bias was applied. Note that the thicknesses are specified from the manufacturer and are often measured to be different by several microns.

The PEA voltage trace measurements and FFT’s of these measurements are shown in Fig. A.27. PEA traces are adjusted so that the leading peaks of the ground/sample interface are aligned. It is evident in the PEA measurements that there appear to be artifacts/ringing that are dependent on the sensor thickness. The width of the leading peaks increase with increasing sensor thickness, as is expected, with \(\sim 5\) ns FWHM for the 9 µm sensor and \(\sim 8\) ns FWHM for both the 28 and 52 µm sensors. The ringing is likely due to multiple reflections within the sensor as indicated by the increasing of the period of the ringing with increased sensor thickness.
Figure A.27: PEA system sensor thickness dependence. (a) Measurements of PEA voltage traces versus time for 250 µm thick PMMA in the PEA system are shown for varying sensor thicknesses of 9 µm, 28 µm, and 52 µm. (b) The corresponding computed FFT’s are plotted versus frequency.

Looking to the FFT’s, the frequency content of each signal increases to higher frequencies with decreasing sensor thickness as expected. This is to be expected as the response of the piezoelectric sensor is proportional to the speed of sound and thickness of the sensor, therefore a thinner sensor will have a faster/narrower response signal, and therefore higher frequency content. The higher frequency content is indicative of the increased spatial resolution. Each sensor has a small peak in frequency around 50 MHz. The peaks in frequency are approximately 10, 20, and 40 MHz for each sensor in order of decreasing thickness, as
seen by the peaks in Fig. A.27(b).

If we refer back to the FFT of the HVPG in Fig. A.18, we can see that the limiting factor is the sensor frequency response and not the frequency content of the pulse. This is because the high frequency cut off in the HVPG FFTs are $\geq 200$ MHz, where even the 5 ns pulse, as seen in Fig. A.18, has higher frequency content than the 9 $\mu$m PVDF sensor, as seen in Fig. A.27(b). It should be kept in mind for any future attempts to improve the spatial resolution of the PEA system that the sensor thickness is a limiting factor on spatial resolution. As the highest spatial resolution is achieved with the thinnest sensor, the USU MPG PEA system typically uses the 9 $\mu$m thick PVDF sensor.

A.9 Impedance Matching: Electrical

Several calculations of electrical impedance matching were done for the PEA system. To accurately model the electrical impedance matching, a typical sample capacitance must be determined. The typical range of relative permittivity values is around 2-4 and thicknesses of 25-250 $\mu$m thick. The electrode size is 12.7 mm in diameter. Using the equation for parallel plate capacitance (refer to eqn. 4.5), this gives an approximate range of 20 – 200 pF. Our samples are typically on the lower end near 20 pF and we will use this in our calculations.

There are two types of impedance matching, passive and active. Passive uses only resistive elements and active can use capacitive or inductive elements in addition. It is an understatement to say that is difficult to achieve excellent impedance matching over a broad frequency range since even passive elements in impedance matching circuits are most often frequency dependent. It is quite easy to match impedance at a single frequency, particularly with active matching.

The first five set-ups tested were passive type impedance matching. They are (i) no matching, (ii) parallel, (iii) series and parallel, (iv) series, and (v) T-pad. These circuits are shown in Fig. A.28. After many pages of algebra, left up to the reader, equations were found for the impedance of the “load” (matching circuitry with 20 pF sample), and $n_{eff}(f)$ which is the ratio of the voltage across the sample over the total applied voltage as
a function of frequency. Ideally the impedance would be 50 Ωs and $n_{eff} = 1$. A comparison of the various matching circuits are shown in Fig. A.29.

It was found that the parallel circuit was the most robust, easiest, well-matched circuit among the group in the frequency range of interest. With the current USU-MPG PEA system, the frequency range is up to about 100 MHz, as seen in the FFT of PEA data in Fig. A.27. The parallel configuration offers the flattest response in that frequency range. Although the voltage drop is only about 50% across the sample, the response is flat and this is still a high enough voltage drop to obtain good PEA measurements.

Measurements of the applied voltage pulse and PEA voltage trace measurements were made to validate the theoretical predictions for the impedance matching circuits. Measurements of 250 µm thick PMMA with pulse voltages of 2 kV amplitude and pulse widths of 0.5, 2.5, and 5 ns were made with 0 kV and 5 kV DC bias. Representative measurements are shown in Fig. A.30 for 2.5 ns pulse width measurements. The measurements indicate that the choice of impedance matching circuitry did not have a particularly large effect on the quality of the signal. Focusing on the orange curve in Fig. A.29 (corresponding to the parallel configuration and the green curve in Fig. A.29), the amplitude is relatively high with comparatively minimal ringing in the signal. This is particularly apparent in the 5 kV DC bias measurements in Fig. A.30(b). This validates our choice for using the parallel impedance matching configuration over the other configurations considered.

Further electrical impedance matching to be explored includes trying active matching techniques using inductive and capacitive components. Although, active matching techniques are typically used to match at more specific frequencies rather than broad ranges. One could imagine a possible “sweeping” over the desired frequency space with several different impedance matching circuits and combining the results into an overall more accurate measurement. Could also have a few parallel matching terminals that could be swapped or switched between that are chosen to match more specifically to a particular sample/experiment. A modular circuit would allow impedance matching to be modified/swapped easily. This will be left to future work.
Figure A.28: Impedance matching circuits. Simplified impedance matching circuits are shown for each variation, (1) no matching, (2) parallel, (3) series and parallel, (4) series, and (5) T-pad. The matching part of the circuitry is circled in red.

Figure A.29: Impedance matching and $n_{eff}(f)$. (a) A plot of load impedance versus frequency, including both the sample and the matching circuitry, are shown for the various set-ups. (b) The voltage ratio parameter $n_{eff}(f)$ plotted as a function of frequency for each set-up.
Figure A.30: Impedance matching PEA and pulse results. Plotted are the PEA measurements of voltage traces versus time with (a) no DC bias (b) 5 kV DC bias, and (c) measurements of the HV pulse across the sample for the various impedance matching circuits as shown in Fig. A.28. Representative results are shown for HV pulse of 2 kV amplitude and 2.5 ns pulse width.
A.10 Impedance Matching: Acoustic

The choice of coupling media at the interfaces in the PEA system to achieve better acoustic impedance matching is nontrivial. There can be many adverse effects of the coupling media on the measured PEA waveform. If a coupling media has electrical conductivity comparable to the sample material, the electric field strength across the sample is diminished and harder to determine, thereby reducing the PEA signal strength and signal-to-noise ratio. Lower viscosity coupling layers can flow to unwanted areas, causing contamination and electric field issues. Low viscosity coupling layers most often also have higher vapor pressures which can be an issue for vacuum PEA systems, although it may be possible to use a vacuum grease such as Krytox™. Higher conductivity, low viscosity materials can cause parallel electrical paths around the sample resulting in a short circuit.

Regardless of conductivity, a thick or acoustically mismatched coupling layer will cause multiple acoustic reflections within the coupling media, thereby adding ringing to the resulting waveform. Non-uniform, poorly known thicknesses, and poor reproducibility of coupling layers can increase the spread in reflected signals (decrease PEA resolution) or add temporal offsets to the signals. Poor acoustic and electrical coupling causes enhanced reflection of the pulsed signals at interfaces and results in a diminished signal-to-noise ratio. This in turn affects the accuracy with which the pulse applied to the sample can be characterized. Other complications include polarization, relaxation effects, and incompatibility of outgassing of oils in vacuum systems.

A study was published on the effects of coupling media in the PEA system in the conference proceedings for the Conference on Electrical of Insulation and Dielectric Phenomena (CEIDP) (Gibson et al., 2018). For further details and analysis of the results please refer to the publication Gibson et al. (2018). A summary of the results are presented here. Several coupling media were tested including (i) no coupling media, (ii) light machine oil, (iii) silicone oil, and (iv) cyanoacrylate glue. Table A.1 gives an overview of the material properties for the relevant coupling media. A simplified diagram in Fig. A.31 shows where coupling media are used in the PEA system ”sample stack”.

Note that there is typically a PVC film
used to improve acoustic coupling between the HV excitation electrode and the sample, but this was introduced into the USU MPG PEA system some time after this study was completed. Note that the cyanoacrylate glue was only used on the ground electrode/sample interface as the PEA system would be glued shut if also applied to the high voltage/sample interface. The ground electrode is also easily removable and several were made to use as "sample plates". The same is not true of the HV electrode.

Results from this study allow specific conclusions to be drawn for each of the four coupling media. Note that the ratios between the amplitudes of the interfacial peaks are shifted closer to one with the addition of the PVC film at the excitation electrode/sample interface, which was not employed for this study.

No coupling media does not appear to be a viable option as there is little to no signal from the excitation electrode interface, indicating a poor acoustic coupling from the sample to the piezoelectric sensor. The reason the peak at the ground electrode can be seen is due to the fact that the charge at that interface is on the surface of the ground electrode itself as a result of the applied DC bias.

A similar amplitude of the ground electrode/sample interfacial peak was observed for the light machine oil, silicone oil, and no coupling media, agreeing to well within normal variation. Note, with systems requiring irradiation in vacuo the HV electrode is not placed directly in contact with the sample (Griseri et al., 2008), which is not currently an issue for the USU MPG ambient PEA system.

A spatial resolution, as determined by the FWHM of the leading interfacial peak, was determined to be approximately 11 µm and 9 µ for no coupling media, light machine oil, and cyanoacrylate, for the raw and deconvolved data respectively. The silicone oil resulted in the worst spatial resolution, with a FWHM that is larger by approximately 18%. However, silicone oil was the most efficient at propagating acoustic signals, seeming to provide the best acoustic coupling. It should be noted that there are problems with polarization of silicone oil under applied field that are not directly discussed or studied in this experiment (Gibson et al., 2018). Silicone oil should be avoided if possible.
Light machine oil appears to be the best viable option for ambient PEA systems out of the coupling medias tested. This is because the measured spatial resolution and peak amplitudes are better than silicone oil and comparable to the other coupling medias. It is also cheap and convenient to use. This is, however, not an option for \textit{in vacuo} systems.

Cyanoacrylate was observed to result in a slightly better spatial resolution than the other coupling medias after processing and deconvolution of the data, refer to Fig. 2 and Table 2 in Gibson \textit{et al.} (2018). Though the amplitude of the raw voltage trace data is lower than than the other coupling medias, the amplitude of the signal is comparable after deconvolution, as they are re-scaled. The signal-to-noise and raw data amplitude could potentially be increased with the addition of light machine oil applied to the PVDF sensor.
interfaces, which in our proposed custom vacuum PEA system is outside of vacuum and not routinely changed.

In an in vacuo PEA system, having a single glued interface is a viable and encouraged option, as it has the best resolution (11 µm raw, and 9 µm after signal processing), has good signal-to-noise properties, and is the only vacuum compatible coupling media tested.

In the USU MPG PEA system, light machine oil is typically used as it is much more convenient than using super glue (which limits removal of samples from sample mounting plates) and currently is an ambient system. Note that we have also found a commercially available 3 µm thick double-sided adhesive with a polyethylene terephthalate base (Ultra-thin PET-based 5 µm Double Sided Tape No.5600) that has yet to be investigated thoroughly and may prove to be much easier to use than the super glue.

Another investigation into the effects of the coupling layers was completed on multi-layer samples. Two 5 mil (50 µm) thick polyimide samples were adhered to each other and measured in the PEA system using all of the previously mentioned coupling media configurations. A 7 kV DC bias was applied across the samples, and the pulse used had a 0.4 ns rise time and 1 kV amplitude. Note that this was a previous version of the PEA system and used a different HVPG unit than is now employed in the current system. The results of the experiment are shown in Fig. A.32. Note that the coupling media at the electrode interfaces is always the light machine oil. It is clear that the two best coupling media are ultra thin cyanoacrylate glue (Bob Smith Industries, Super Thin Insta-Cure Cyanoacrylate Super Glue) and light machine oil.

The difference between thin and ultra thin cyanoacrylate glue are preparation procedures. To achieve ultra thin (on the order of a micron) thicknesses of cyanoacrylate glue, the sample must be clamped together with a micrometer. The fine thread on a micrometer allow a very tight clamping to be achieved on the sample to ensure a very thin layer of glue. The thickness of the ultra thin prepared cyanoacrylate super glue was measured with thin film interference and scanning electron microscope measurements to be on the order of about 1 µm. Polymeric samples aren’t glued very easily either and require a long time for
the glue to cure, despite the curing typically occurring in a matter of seconds. Aside from doing this for every sample (possibly damaging samples, particularly if they are charged) the best choice is to use the light machine oil. It should also be noted that the curing of the glue is an exothermic reaction. For example, if the cyanoacrylate glue is spilled and then attempted to clean up with a Kimwipe, the wipe will ignite and begin to smoke due to the large surface area of glue that is now allowed to cure all at once.

However, it is quite interesting that for the ultra thin cyanoacrylate glue the interface essentially disappears from the PEA voltage traces. It may be possible to take advantage of this effect to achieve “high resolution” PEA measurements, as the resolution of peak position of charge is very high in the PEA system, see Section 5.1.2 (Gibson and Dennison, 2022b). If charge were to be deposited in the sample near the surface, then adhered to another sample of the same material with ultra thin glue, this could potentially allow a much more accurate measurement of the deposited charge that is not convolved with the mirror charge signal of the electrode that would be there without the extra layer of material.

Another consideration to the acoustic coupling issue is the acoustic absorber added as the backing of the sensor. If a poor backing material and configuration is chosen then considerable ringing can be evident in the PEA data. Previously, a copper electrode was used as a sensor backing material as it allowed for easy connection to the piezoelectric sensor. However, this resulted in large acoustic reflections/artifacts in the PEA voltage traces measured, refer to Fig. A.33. To mitigate ringing, a backing of PVDF is now used.
Figure A.33: PEA measurements with copper backing. This measurement is of LDPE charged with an applied DC bias over several minutes. Large ringing artifacts are apparent in the measured voltage traces.

with a spring-loaded gold contact. This gold contact makes the electrical connection to the metallized PVDF sensor used in the PEA system, as discussed in Chapter 3. The PVDF sensor is metallized so that either surface of the sensor is conductive which allows for much easier electrical connection.

A.11 Reflections in Cables

Another issue related to the electrical impedance matching of the PEA system are electrical signals reflecting in the cables. Various cables were tested including those (i) between the HVPG and the load [sample and impedance matching circuitry (refer to Appendix A.9)], (ii) between the sensor and the amplifier, and (iii) between the amplifier and the oscilloscope. PEA measurements were done showing the reflections in the cable and in particular the delay times and amplitude attenuation due to these reflections. Electrical impedance matching, as shown in Appendix A.9, was implemented to mitigate these reflections between the sample/impedance matching network and the HVPG. However, some reflections are still evident, as shown in the measurements in Fig. A.34. To further mitigate these reflections, a high voltage fast-rise time 8 dB attenuator (Barth 142-NMFP-8 B) was
introduced between the HVPG and the impedance matching circuitry. In addition to the attenuator, it is found that the ideal pulse voltage is the lowest possible setting (in our case 1 kV before attenuation) so that further reflections are relatively small or completely undetected.

An experiment was devised to test the effect of varying cable lengths in three key locations: (i) between the HVPG and PEA stack, (ii) between sensor and amplifier, and (iii) between amplifier and oscilloscope. The resulting PEA measurements are shown in Figs. A.34, A.35, and A.36, respectively.

As seen in Fig. A.34, changing the cable length from 6 ft (1.83 m) to 16 ft (4.88 m) changes the resulting reflections from \(\sim 17\) ns to \(\sim 42\) ns. The blue curve is the resulting measurement with the 8 dB attenuator, effectively mitigating the artifacts of the reflections in the measurement. According to the manufacturer of the cable, the signal travels at 0.7c, where c is the speed of light. This translates to an estimated reflection time of 17 ns and 46 ns for the 6 ft and 16 ft cables, respectively. This correlates well with the data.

The cable between the sensor and the amplifier was exchanged between a 4 inch (10.2 cm) and 6 ft (1.83 m) cable. Results are shown in Fig. A.35. The 4 inch cable is typically used for standard sample measurements and the 6 ft was used cable to test for the effects of a longer cable. However, the test was a bit inconclusive. The longer cable was of subpar quality and did not allow for closure of the PEA system enclosure/box (no shielding of sensitive parts of the PEA system). As such, it appears the cable picked up noise with frequency content of about 177 MHz, not corresponding to the anticipated reflections frequency (17 ns or about 60 MHz). However, eliminating the 4 inch cable all together by connecting the amplifier directly to the sensor (directly to the SMA connector on the back of the sensor holder, see Fig. 3.3(e)) was also tested, yielding results that show there are no effects for the short 4 inch cable. Note that the connection to the metallized PVDF sensor is made with a gold spring contact that is spot welded to a few inch long piece of thermocouple wire that is attached to an SMA connector at the bottom of the sensor assembly, see Section 3.1.2.
The cable between the amplifier and the oscilloscope was tested as a 6 ft (1.83 m) cable and a 16 ft (4.88 m) cable, as shown in Fig. A.36. No difference was detected between the two measurements, indicating the length of this cable is not an issue.

In conclusion, the typical set-up of the PEA system works well as far as these cable lengths and types are concerned. The exception is for the cable between the HVPG and the PEA stack; however, these reflection issues were mitigated by introducing an 8 dB attenuator in the line.

Note that different cables may provide better results in the future. For example, low impedance cables may provide easier impedance matching to the sample stack but would require a new HVPG be built with a low output impedance.
Figure A.34: Cable reflection effects for HPVG to sample stack. Shown is (a) a schematic PEA diagram indicating the cable location tested and (b) the results of changing the cable length on the measured PEA voltage trace. Also shown is the introduction of an 8 dB attenuator (blue curve).
Figure A.35: Cable reflection effects for sensor to amplifier. Shown is (a) a schematic PEA diagram indicating the cable location tested, (b) an image of the PEA system with the 6 ft cable, and (c) the results of changing the cable length on the measured PEA voltage trace.
Figure A.36: Cable reflection effects for amplifier to oscilloscope. Shown is (a) a schematic PEA diagram indicating the cable location tested and (b) the results of changing the cable length on the measured PEA voltage trace. Also shown is the introduction of an 8 dB attenuator (blue curve).
A.12 Surface Roughness

Another potential factor in the resolution of the interfacial peaks of the PEA system is the surface roughness of the electrodes and samples. This could add some effective “width” to the surface charge and/or scatter the propagated acoustic wave signal from the sample.

Results for measurements of the Al polished and heavily used (unpolished) electrode surfaces with a confocal microscope are presented. This is followed by results of measuring a PTFE sample surface with a surface profiler. These are somewhat preliminary results and do not seem to affect the PEA results at this time. However, as spatial resolution is improved (as well as precision in peak position), a more careful analysis of the effects of surface roughness on the resolution and measurements of peak-to-peak distances should be conducted.

Electrodes

An attempt was made at quantifying the surface roughness of both a polished and heavily used electrode (unpolished) in the PEA system. This was done with a confocal microscope at the University of Utah. A summary of the results are given in Table A.2 and Figs. A.37 and A.38 for polished and unpolished electrodes, respectively. Table A.2 also provides a brief description of the parameters measured with the confocal microscope. The laser confocal microscope produces 3D imaging from optical sectioning by taking several images of the focal plane as seen Figs. A.37 and A.38. From these images, it is able to construct a 3D image of the sample surface.

Although there is clearly a difference in the surface of the polished and unpolished electrodes, PEA results were found to have a negligible difference using either electrode. That is, there was no discernible difference between measurements taken with either the polished or unpolished electrode. This is likely due to the coupling oil filling in the gaps caused by the roughness of the electrodes. The surface roughness does not appear to currently be limiting the spatial resolution of the PEA measurements at the current spatial resolution of approximately 10 μm.
**Table A.2**: Electrode surface roughness parameters. The parameters were measured with the confocal microscope for the polished and unpolished (rough) Al electrodes at 5x and 50x zoom factors. A brief description of each parameter is given as well. Values in units of µm.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Sq</th>
<th>Sk</th>
<th>Sku</th>
<th>Sp</th>
<th>Sv</th>
<th>Sdq</th>
<th>Sdr</th>
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<tbody>
<tr>
<td>Polished</td>
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<td>0.3</td>
<td>4.5</td>
<td>14.5</td>
<td>10.3</td>
<td>24.8</td>
<td>1.2</td>
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<tr>
<td>5x</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Polished</td>
<td>0</td>
<td>-3.5</td>
<td>166.8</td>
<td>2.5</td>
<td>2.1</td>
<td>4.6</td>
<td>0.7</td>
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<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Unpolished</td>
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<td>0.6</td>
<td>6.9</td>
<td>52.2</td>
<td>38.8</td>
<td>91</td>
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<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Unpolished</td>
<td>0.1</td>
<td>-1.3</td>
<td>5.1</td>
<td>0.6</td>
<td>1</td>
<td>1.6</td>
<td>0.1</td>
</tr>
<tr>
<td>50x</td>
<td></td>
<td></td>
<td></td>
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</tr>
</tbody>
</table>

**Samples**

The surface roughness of samples can also be an issue. An attempt was made to measure the surface roughness of a few 250 µm PTFE samples (McMaster Carr, SF103V0010) using a surface profiler (Keyence 3D Surface Profiler VK-X3000 Series) at Utah Valley University. The effects of sample surface roughness on PEA results has not been thoroughly investigated and is left to future work. The presented measurements serve to highlight that improved spatial resolution may require samples with smoother surfaces or an alternative calibration method. This can be seen as the surface roughness of the PTFE (sample 1) has features up to approximately 8 µm (highest peak to lowest valley), approximately on the order of the spatial resolution of the PEA system. On a more uniform sample (sample 2), the features are approximately 4 µm (highest peak to lowest valley). Representative images are shown in Fig. A.39.
Figure A.37: Confocal microscope measurements of polished Al electrode. An image of the polished electrode, representative surface image, and height histogram is shown for x5 and x50 zoom settings on the instrument.
Figure A.38: Confocal microscope measurements of unpolished Al electrode. An image of the unpolished electrode, representative surface image, and height histogram is shown for x5 and x50 zoom settings on the instrument.
Figure A.39: Surface roughness of PTFE. Representative images from two different PTFE samples are shown for (a) sample 1 and (b) sample 2.
APPENDIX B

Calibration

B.1 Calibrating Charge Magnitude

The charge calibration factor was defined and derived in Chapter 4. The charge calibration factor is determined by acquiring a PEA measurement with a known DC bias applied. However, the charge calibration factor should be the same for all measurements, particularly for all measurements within a given dataset, taken at the same time on the same day.

This is unfortunately not necessarily true. A set of measurements were devised to test the reproducibility of the calibration factor and the associated uncertainties. A 250 \( \mu \text{m} \) thick pristine PEEK sample was measured with various DC bias of 0.5 kV, 1 kV, 2 kV, and 5 kV. Then the charge calibration factor and the speed of sound were determined for all these measurements.

For an initial test of the linearity of the PEA system, tests were made to determine if the amplitude of the raw voltage trace data scaled linearly with the applied DC bias. This was also done for the calibrated PEA data. This is important because if the amplitude does not scale linearly then this could imply that the calibration factor is nonlinear. As expected, the amplitude of the PEA measurement scales linearly with the applied voltage, refer to Fig. B.1. This was also demonstrated in Appendix A, see Fig. figure:AmplAccuracy.

The charge calibration factor and speed of sound were then plotted as a function of applied DC bias, refer to Fig. B.2. The charge calibration factor is observed to be highly nonlinear at low DC bias. It appears that at \( \gtrsim 1 \text{ kV} \) DC bias the charge calibration factor is nearly a constant, although it does have a slight downward trend that is likely due to charging. This trend is still evident if the calibration factor is modified to remove the dependence on the speed of sound (see \( K \) versus \( K' \) in Chapter 4) and modified to take into
account the effects from the pulse voltage, as seen in Fig. B.4.

It is interesting the speed of sound, as determined by the peak-to-peak distance of the interfacial peaks in time divided by the measured thickness of the sample, also has a dependence on the applied DC bias. However, the speed of sound increases below $\sim 1\,\text{kV}$ as would be expected given the inverse dependence of $K$ or $K'$ on $v_s$ shown in Eq. 4.8.

It should be noted that with high applied DC bias, charge injection starts to occur as well, which can alter the signal observed from the ground electrode/sample interface by changing the area under the measured voltage trace and therefore alter the charge calibration factor. Charge injection is evident in Fig. B.3 at voltages $\gtrsim 2\,\text{kV}$. Refer to Chen et al. (2006) for more details about careful calibration, including careful measurement of charge injection and taking into account the pulsed voltage. A DC bias of approximately 1-2 kV should be applied to obtain a reference measurement to be sure of an accurate charge calibration factor that is approximately linear and avoids injecting charge into the sample. Further study should be completed into the non-uniformity of the calibration factor as a function of applied DC bias, particularly more careful measurements of the charge injection as a function of DC bias should be conducted. This would include more data points at smaller increments of applied DC bias, as well as studying other materials and thicknesses. It is likely to be useful to think in terms of applied electric field rather than applied DC bias.

The effect of the excitation pulsed voltage on the measured signal should also be considered more carefully. An attempt made to take the voltage pulse into account is shown in Fig. B.4. This plot shows how the modified charge calibration factor as a function of applied DC bias would change with various effective voltage effects from the excitation pulse. The maximum effective voltage is $\sim 300\,\text{V}$, as determined by the measured peak amplitude of the pulse across a 50 $\Omega$ load. The actual effective DC bias is likely less than 300 V. This is due to the transient nature of the pulse, as well as the potential for the full voltage not to be dropped across the sample. The voltage will be lower across the sample due to effects from electrical impedance matching (rather mismatching), and potential voltage drop across the
Figure B.1: Amplitude of interfacial peak as a function of DC bias. Plotted is the amplitude of the sample/ground electrode interfacial peak in the PEA measurements of pristine PEEK as a function of applied DC bias for (a) raw data and (b) deconvolved (but not calibrated) data. (c) The deconvolved PEA measurements of pristine PEEK are plotted.

semi-conducting layer and acoustic coupling layers. It is unclear how the transient effects of the pulsed voltage can be compared to an effective DC bias. The values shown in the legend of in the Fig. B.4 are the effective DC bias. There are definitely effects from the
pulsed voltage, as with a high voltage pulse on a pristine sample with no embedded charge or DC bias applied, a signal can be observed from the sample/ground electrode interface.

The uncertainties in the calibration factor are a bit difficult to determine. The relevant parameters are the permittivity, applied DC bias (with effects from the pulsed voltage), sample thickness, speed of sound, and area under the curve of the measured and deconvolved voltage trace, refer to Eq. 4.10. The uncertainty in the thickness and speed of sound will be covered in the following section. The error introduced from the uncertainty in the permittivity is approximately 2-5%. For example, the permittivity is 3.45±0.1 and 2.1±0.1 for PEEK and PTFE respectively (refer to Section 6.1.1 (Gibson et al., 2023)).

The area under the curve of the deconvolved voltage signal is what has been addressed above throughout this section. The voltage drop across the sample consists of the applied
Figure B.3: Modified calibration factor with pulse effects. The calibration factor is plotted as a function of DC bias with possible effects from the excitation pulse taken into account as well as the dependence of the speed of sound removed.

DC bias and the pulsed voltage. As discussed, the pulsed voltage is a bit tricky to find the effective voltage applied to the sample. For the DC bias applied, the voltage does not drop only across the sample, but the question is how much does?

To cause an appreciable drop across something other than the sample, either a parallel resistance (to the sample e.g. leakage current) would need to be on the order of $1 \text{M}\Omega$ or less (the value of the current limiting resistor), or the resistance would need to be on the order of the resistance of the sample or more, which is typically in excess of $10^{18}\Omega$. The voltage can drop across the semiconductor layer, in this case a 500 $\mu$m PVC film. The volume resistivity is approximately $10^{14}\Omega \text{ cm}$, resulting in a resistance of approximately $5 \times 10^{12}\Omega$ in series, which is much smaller than the typical sample resistance, and therefore negligible (this is estimated and should be measured in the future to be certain). The coupling oil adds both series resistance and has the potential for a parallel resistance leakage path. To make an estimate of the resistance of the coupling oil, a thickness of 1 $\mu$m and conductivity of $2 \times 10^{-9}\Omega \text{ cm}$ was used. An upper limit estimate for the series resistance is $4 \times 10^8\Omega$ and a lower limit estimate for the parallel resistance is $4 \times 10^{13}\Omega$, which are negligible for being too small and too large, respectively. To be certain, the effects of the coupling oil should be measured explicitly, as well as their impedance and permittivity to be sure of the effect the
Figure B.4: Charging effects as observed through amplitude of interfacial peaks. Charging of the sample due to the applied DC bias is evident around voltages 2 kV or greater. 

coupling layers have on the electrical impedance matching. With these estimations, it is likely that the largest error in the voltage applied across the sample is from the uncertainty from the HVDC power supply, refer to Section A.5.

The uncertainty in the calibration factor is difficult to determine but is likely $\geq 5 - 10\%$ relative uncertainty.

B.2 Calibrating Distance Scale

As discussed in Section 4.3.1, for calibrating the distance scale two pieces of information are needed. The needed information is the thickness of the sample and the speed of sound in the material. Either of these pieces of information can be obtained with the PEA measurement, but one must be known beforehand. The easiest method is to measure the thickness of the sample and to determine the speed of sound with the PEA measurement.

The actual calibration of the data is quite simple. With a known thickness and speed of sound, each time step in the measurement is simply multiplied by the speed of sound in the material. This transforms the time step to a distance step.
The uncertainty in the distance scale must still be determined. Through simple error propagation (Taylor, 1997), the error in the distance scale is represented as,

$$\sigma_{\text{distance}} = \sqrt{(t_{\text{step}}\sigma_{\text{speed}})^2 + (v_s\sigma_t)^2} \quad \text{(B.1)}$$

where $t_{\text{step}}$ is the time step as determined by the oscilloscope (0.4 ns in this case), $\sigma_{\text{speed}}$ is the error in the speed of sound, $v_s$ is the speed of sound, and $\sigma_t$ is the error in each time step. An example error for the error in the time step can be determined by adding the inherent error of the oscilloscope and the measured error of the peak position and adding in quadrature. Refer to Section 5.1.2 for a discussion of the error in peak position.

As for the error in the speed of sound, it is represented as,

$$\sigma_{\text{speed}} = \sqrt{\sigma_{\text{Pk2P}}^2 \left(\frac{d}{t_{\text{Pk2P}}^2}\right)^2 + \sigma_{\text{thickness}}^2 \left(\frac{1}{t_{\text{Pk2P}}^2}\right)^2 + \sigma_{\text{Temp}}^2} \quad \text{(B.2)}$$

where $\sigma_{\text{Pk2P}}$ is the error in the peak-to-peak time difference in the PEA measurement, $t_{\text{Pk2P}}$ is the peak-to-peak time difference, $\sigma_{\text{thickness}}$ is the error in the thickness, and $\sigma_{\text{Temp}}$ is the error in the speed of sound due to temperature fluctuations. To obtain the error in the speed of sound, the other error terms must be determined.

Typically the thickness is measured with a Mitutoyo IP65 digital micrometer, with a resolution of ±0.5 µm. Typically 10+ measurements are made in various positions on the sample. The average of these measurements gives the thickness and the standard deviation added in quadrature with the error of the measurement device gives the error in the measured thickness, $\sigma_{\text{thickness}}$.

The peak-to-peak time difference $t_{\text{Pk2P}}$ between interfacial peaks in a PEA measurement is simply determined by finding the peaks of the signals from the interfaces in a measurement. However, it should be noted that other features of the signals can be used to find the time difference such as the rising/falling edge and the envelope. The best parameter for accurate determination of the features in a PEA signal appears to be the peak position. This was demonstrated in Section 6.3, see Fig. 6.9. The error $\sigma_{\text{Pk2P}}$ is then determined by
the uncertainty in the peak positions. This is determined statistically for each measurement set as outlined in Section 5.1.2, see Fig. 5.2(b) and (c). Then $\sigma_{P_kP_k}$ is simply the error in the peak positions of the interfaces added in quadrature.

The final term $\sigma_{Temp}$ is a bit more difficult to determine. It could be measured by varying the temperature of the sample in a controlled way and measuring the change in $t_{P_kP_k}$ to add the error to the $\sigma_{P_kP_k}$ term. A similar scheme could be attempted to add the error to the $\sigma_{thickness}$ term. Currently, the error is estimated from the fluctuations in speed of sound as determined for similar materials in the literature, as discussed in Section 6.1.1.
Curriculum Vitae
Zachary Gibson
Phone: 801-830-7521 Email: zackgibson5@gmail.com
https://works.bepress.com/zachary-gibson/

Education

Utah State University (Fall 2016 – Summer 2023, 3.76 GPA)
◊ Doctor of Philosophy in Physics
◊ USU Presidential Doctoral Research Fellowship (Fall 2016 – Summer 2020)
◊ Chateaubriand Fellowship (Research Fellowship at Université Toulouse III Paul Sabatier in Toulouse, France from May – October 2022)

Utah Valley University (Fall 2012 – Spring 2016, 3.90 GPA)
◊ Bachelor of Science in Physics, Minor in Mathematics (Summa Cum Laude)
◊ Valedictorian for College of Science and Health (First Valedictorian from Physics Dept.)
◊ National Science Foundation Science Technology Engineering and Mathematics Scholarship awarded by Utah Valley University (Fall 2013 – Spring 2016)
◊ Exemplary Academic Merit Scholarship (Fall 2012 – Spring 2016)
◊ Dean’s list (Fall 2012 – Spring 2016)

Honors and Awards
◊ Nominated by Department of Physics for Utah State University’s Doctoral Researcher of the Year (December 2022)
◊ Outstanding Graduate Student Talk at American Physical Society Four Corners Region Conference (October 2022)
◊ 1st Place at USU Engineering Week STEM Short Film Contest (February 2022)
◊ Outstanding Graduate Student Talk at American Physical Society Four Corners Region Conference (October 2020)
◊ Best Graduate Student Poster in College of Science at Utah State University’s Student Research Symposium (April 2019)
◊ 2nd Place Oral Presentation at Utah Valley University Engaged Learning Week (March 2016)
◊ Outstanding Student Certificate from Utah Valley University’s Department of Physics in Recognition of Outstanding Accomplishments and Contributions (March 2015)

Peer-Reviewed Publications

Accepted

Published
◊ Zachary Gibson and J. R. Dennison, “Relevancy of Pulsed Electroacoustic Measurements for Investigating Spacecraft Charging,” IEEE Transactions on Plasma Science, 2023, doi:


Other Publications

Published


Brian Beecken, Zachary Gibson, and David Cooke “Toward Validation of the AF-NUMIT3 Dielectric Charging Simulation Model,” In Proc. Spacecraft Charging Technology Conference, 2022

Unpublished


Zachary Gibson, “The Physical Significance of $\alpha$ for Electron Transport,” Physics PhD Candidacy Exam, Utah State University, 2019


Presentations

Invited Talks

Zachary Gibson, “Application of the Pulsed Electroacoustic Method to Spacecraft Charging Issues,” Utah State University Physics Colloquium, Logan, UT, December 5, 2022

Polymers via Pulsed Electroacoustic Measurements,” Conference on Electrical Insulation and Dielectric Phenomena, Denver, CO, October 31, 2022


◊ Zachary Gibson, “Charge Transport in Dielectrics: Pulsed Electroacoustic Method,” Utah Valley University Physics Colloquium, Orem, UT, November 16, 2021

◊ Zachary Gibson, Megan Loveland, “Charge Transport in Disordered Materials and the Dispersion Parameter,” Utah State University Physics Colloquium, Logan, UT, October 2019 (talk)

◊ Zachary Gibson, “The Pulsed Electro-Acoustic Method”, Utah State University Physics Colloquium, Logan, UT, December 2017 (talk)

◊ Zachary Gibson, “Measuring Distances to Galaxies Using Infrared Surface Brightness Fluctuations”, Utah Valley University Physics Colloquium, Orem, UT, Spring 2016 (talk)

Award Winning Presentations

◊ Zachary Gibson, J. R. Dennison, and Virginie Griseri, “Effect of Fluence on Electron Deposited Charge Distributions in Polytetrafluoroethylene,” American Physical Society Four Corners Meeting, University of New Mexico, Albuquerque, NM, October 14-15, 2022, Awarded Outstanding Graduate Student Talk

◊ Zachary Gibson, Benjamin Bradshaw, J. R. Dennison, Elena Plis, Daniel Engelhart, and Ryan Hoffmann, “Energetic Distribution of States in Irradiated Low-Density Polyethylene from UV-Vis-NIR Spectroscopy,” American Physical Society Four Corners Meeting, University of New Mexico, Virtual Meeting, October 23-24, 2020, Awarded Outstanding Graduate Student Talk


International Conferences


◊ Zachary Gibson and J. R. Dennison, “The Relevancy of Pulsed Electroacoustic Measurements for Spacecraft Charging,” Spacecraft Charging Technology Conference, Virtual, 2022 (talk)

◊ Brian Beecken, Zachary Gibson, and David Cooke “Toward Validation of the AF-NUMIT3 Dielectric Charging Simulation Model,” Spacecraft Charging Technology Conference, Virtual, 2022 (talk presented by Brian Beecken)


National Conferences

◊ Zachary Gibson, J. R. Dennison, and Brian Beecken, “Comparison of Charge Deposition Profiles in Polymers Irradiated with Monoenergetic Electrons: Pulsed Electroacoustic Measurements and AF-NUMIT3 Modeling,” Applied Space Environments Conference 2021, Jet Propulsion Laboratory, Pasadena, CA (Virtual), November 1-5, 2021 (talk)


Regional Conferences

◊ Megan Loveland Dewaal, J. R. Dennison, Dallen DeWaal, and Zachary Gibson, “Effects of Exposure to Atmospheric Humidity on Breakdown Field Strength Measurements of Polymers,” Utah State University Fall Undergraduate Student Research Symposium, Logan, UT, December 8, 2022 (poster)


◊ Zachary Gibson, “Implementation of Peer Review for Lab Assignments in Conjunction with Automated Grading Via Open Source Python Code,” Empower Teaching Excellence Conference, Utah State University, Virtual Meeting, August 18, 2021 (talk)

◊ Zachary Gibson, Megan Loveland, and J. R. Dennison, “A Unified Description of the Electrical Properties of Low-Density Polyethylene via the Dispersion Parameter,” American Physical Society Four Corners Regional Meeting, Prescott, AZ, October 2019 (talk)

◊ Zachary Gibson, J. R. Dennison, Lee Pearson, Erick Griffiths, Anthony Pearson, “Charging Effects of High Voltage Probe Pulse on Pulsed Electroacoustic Measurements”, American Physical Society Four Corners Regional Meeting, Salt Lake City, UT, October 2018 (talk)


Other Presentations
◊ Zachary Gibson, J. R. Dennison, Lee Pearson, Erick Griffiths, Anthony Pearson, “Charge Distributions in Electron Beam Irradiated PTFE,” Utah State University Student Research Symposium, Logan, UT, April 2019 (poster, awarded Best Graduate Student Poster in College of Science)
◊ Zachary Gibson, J. R. Dennison, Lee Pearson, Erick Griffiths, Anthony Pearson, “Effects of Acoustic Coupling Layer Electrical Conductivity on Pulsed Electroacoustic Measurements”, Utah State University Student Research Symposium, Logan, UT, April 2018 (poster)

Undergraduate Researcher Mentees are underlined

Research

Experience

Material Physics Group (Fall 2016 – Spring 2023, Mentor: J.R Dennison, Utah State University)
Group emphasis on spacecraft charging mitigation through understanding of electrical properties of highly disordered insulating materials, typically polymers
-Pulsed Electro Acoustic System
System measures space charge distributions in thin (10’s – 100’s of microns thick) insulating materials
◊ Developed understanding, modeling, and verification of ambient pulsed electroacoustic system
◊ Improved signal quality through system modifications
◊ Achieved layer of glue on the order of one µm thick for sample stack adhesion and coupling
◊ Designing and developing in-situ pulsed electroacoustic system with ~1 µm resolution
◊ Gained experience with high energy electron gun and vacuum chambers
-Radiation Induced Conductivity
◊ Experience operating and trouble-shooting high/ultra-high vacuum systems and high energy (80 keV) electron gun
◊ Conducted sensitive measurements of currents on the order of $10^{-15}$ A in situ
-Constant Voltage Conductivity System
System measures DC conductivity of thin insulating materials
◊ Measured conductivity as a function of time and temperature
◊ Measurements made in ambient and vacuum conditions
◊ Temperatures as low as 240 K and currents as low as $10^{-16}$ A
-Other Experience in Group
◊ Observed sample morphology utilizing a Scanning Electron Microscope
◊ Utilized thin film interference reflectometry to measure sample thicknesses
 looseness permittivity of samples with impedance analyzer
◊ Measured surface resistivity of samples with low current ammeter
◊ Experience and training for radiation safety, high voltage, and high vacuum systems
◊ Irradiated/aged samples with x-ray and beta radiation sources
◊ Made connections in theory of radiation induced conductivity, DC conductivity, photoconductivity, and other electrical properties with use of dispersion parameter (Candidacy Exam Topic)

Sandia National Lab Internship (May – August 2023, Mentors: Leah Appelhans and Andrew Padgett, Sandia National Lab, Albuquerque, NM)
*Study of electrical properties of polymeric materials*
◊ Utilized and improved upon pulsed electroacoustic system obtained by Sandia National Lab
◊ Studied effects of aging epoxy composite materials on electrical properties
◊ Studied breakdown and flashover in epoxy materials

Chateaubriand Fellowship (May – October 2022, Mentors: Virginie Griseri and J. R. Dennison, Universite Paul Sabatier, Toulouse, France)
*Study of aging of polymeric samples based on the total ionizing dose (TID) and source of ionizing radiation*
◊ Irradiated polymeric samples with x-rays, electrons, and gamma rays with varying TID
◊ Deposited electrons in aged and virgin samples with high energy electron gun
◊ Measured deposited electrons with pulsed electroacoustic method

Air Force Research Lab Summer Scholar (Summer 2020, Mentor: Ryan Hoffman, Kirtland Air Force Base)
10 week remote internship (due to COVID-19), where research was done at Utah State University in collaboration with researchers at Kirtland Air Force Base in Albuquerque, NM
◊ Designed and implemented alterations to pulsed electroacoustic system to improve signal quality
◊ Determination of Urbach energy from UV/Vis data for neat and irradiated polymers

YUI Satellite Project (Spring 2019, Mentor: Toshihiro Kameda, University of Tsukuba)
*Short project involving attitude determination of small satellites*
◊ Implemented an opensource star tracking algorithm to determine attitude of satellites
◊ Code written in Python on a RaspberryPi

Ion Thruster Project (Spring 2019, Mentor: Junko Yamasaki, University of Tsukuba)
*Short project involving a search alternate fuel sources for hall thrusters*
◊ Read and discussed literature on hall thrusters
◊ Briefly aided in experimenting with alternative fuel sources, such as Ar/Xe mixtures, to replace pure Xe fuel typically used in ion thrusters

GAS Team High Altitude Balloon Project (Fall 2016, Mentor: J.R. Dennison, Get-Away-Special Team at Utah State University)
*High altitude balloon team that utilizes weather balloons to gather data while in flight*
◊ Analyzed data from high altitude balloon flights collected by students from University of Tsukuba
◊ Presented results at the American Physical Society Four Corners Conference in 2016
Infrared Surface Brightness Fluctuations Analysis and Distance Measurements of Galaxies (Spring 2014 – Spring 2016, Mentor: Joseph Jensen, Utah Valley University)

Cosmological distance scale research involving surface brightness fluctuations analysis and color measurements

◊ Acquired data from various telescopes such as Hubble Space Telescope and Gemini Observatory in Chile
◊ Data analysis process included data reduction, object masking, measuring point spread functions, determining colors, etc.
◊ Measured distances to various galaxies using surface brightness fluctuations analysis utilizing statistical methods and Fourier analysis
◊ Comparison of color measurements to models yielded stellar population characteristics within galaxies such as age and metallicity

Research Experience for Undergraduates at University of Utah: Ultra High Energy Cosmic Ray Research (Summer 2015, Mentors: John Belz and Rasha Abbasi)

◊ Investigated a new ultra high energy cosmic ray detection apparatus (Telescope Array RAdar project, TARA)
◊ Used C++/ROOT to time-match surface detector triggers at Telescope Array and data collected from TARA, and to analyze the waveform data from TARA in search for “chirps”
◊ Helped run multi-million dollar fluorescence detectors in Delta, UT to measure cosmic rays
◊ Presented the results at a symposium held at the University of Utah

Inertial Electrostatic Confinement Trap (Spring 2015 – Spring 2016, Mentor: Raymond Perkins)

◊ Researched and developed plan to build and assemble an inertial electrostatic confinement trap (fusor), including proposing a budget and gaining funding from department
◊ Ordered parts and started assembly

High Altitude Balloon - Project (HAB-P) (Spring 2014, Mentor: Kim Nielsen, Utah Valley University)

◊ Co-led a team to design and build the first payloads/experiments launched via a high altitude balloon by Utah Valley University to study characteristics of the atmosphere
◊ Gained experience working on a research team and programming arduinos/raspberry pi’s

Funding

Pulsed Electroacoustic Research

◊ Presidential Doctoral Research Fellowship awarded by Utah State University (Tuition + $24k/yr, 4 years)
◊ Chateaubriand Fellowship awarded by French Embassy in US (stipend for 5 months at Université Paul Sabatier)

Infrared Surface Brightness Fluctuation Research

◊ NASA Space Grant (Spring 2014, $1500)
◊ Summer Undergraduate Research Funding (SURF) (Summer 2014, $1500)
◊ Undergraduate Research Scholarly and Creative Activities grant (Spring 2015, $2000)
◊ NASA Space Grant (Fall 2015, $1500)
◊ Undergraduate Research Scholarly and Creative Activities grant (Spring 2016, $2000)

**High Altitude Balloon Project (HABP)**
◊ UVU Grant for Engaged Learning (GEL) (Spring 2014, ~$750)

**Inertial Electrostatic Confinement Trap**
◊ UVU Physics Dept. (Funding for project supplies ~$1000)

**Programming**
◊ Igor, utilized for data analysis in Material Physics Group
◊ Matlab, (1 cr. course, Fall 2014), utilized for data analysis at Sandia National Lab
◊ Mathcad, utilized for data analysis in Material Physics Group
◊ LabVIEW, earned Associate Developer Level 1 Certification (2 cr. course, Fall 2013) and utilized for data acquisition and analysis for Material Physics Group
◊ Python, utilized for attitude determination project (Spring 2019)
◊ Fortran, (3 cr. course, Spring 2018)
◊ Maple, (1 cr. course, Fall 2017)
◊ Scilab, (3 cr. course, Spring 2016)
◊ C++/ROOT, utilized for summer research project at University of Utah (Summer 2015)
◊ Unix, utilized for surface brightness fluctuation research (Spring 2014 – Spring 2016)
◊ Maxima, (3 cr. course, Spring 2014)

**Teaching**

**Adjunct Faculty Member** (Fall 2022 – Spring 2023, Southern Utah University)
◊ Taught Physics 1010 online asynchronous course remotely
◊ Improved course through increased interactions, student check-ins, and student feedback

**Lecturer of Record for Physics 1100 – Great Ideas in Physics** (Fall 2021, Utah State University)
◊ Prepared and presented lectures with active learning emphasized with use of discussions and in class group projects
◊ Implemented project for students to go above and beyond the in class material with checkpoints throughout the semester
◊ Actively checked in and made improvements during the course by utilizing student feedback and peer review by a professional educator

**Teaching Assistant for Physics 1100 – Great Ideas in Physics** (Spring 2022, Lecturer: Bela Fejer, Utah State University)

**Empowering Teaching Excellence – ETE 10 Teaching Scholar Certificates** (Fall 2020 – Spring 2022, Utah State University)
Master Teaching Scholar Certificate (Spring 2022)
◊ Earned an additional 10+ microcredentials beyond the teaching scholar certificate
◊ Microcredentials involved non-trivial amounts of time and effort and were in one of three categories: engage, implement, or contribute
◊ Experience included participating in research assignment workshops, learning about resources, learning circle book clubs, implementing and tracking outcomes of learned strategies in the classroom, and contributing to the campus teaching community through activities such as hosting book clubs on pedagogy and presenting at a teaching conference

Teaching Scholar Certificate – Foundations of College Teaching (Spring 2021)
◊ Earned an additional 10+ microcredentials beyond the teaching scholar certificate

Explore College Teaching Certificate (Spring 2020)
◊ Earned 5+ microcredentials to receive this graduate student oriented teaching certificate

Teaching Assistant for Physics 1800 – Conceptual Physics (Spring 2021, Lecturer: David Smith, Utah State University)
◊ Ran the recitation section of the class
  ○ This involved preparing my own lectures and example problems to work through during class time, as well as helping students to complete homework and study for tests
◊ Used my prior experience as a TA for this course to assist the lecturer in an easy transition to teaching this class for the first time
◊ Utilized a hybrid of in person and remote learning resources to aid all students to the best of my ability

Teaching Assistant for Physics 1040 – Introductory Astronomy (Fall 2020, Lecturer: David Smith, Utah State University)
◊ Helped design and implement interactive content and assignments to better deal with an online learning environment necessary due to the COVID-19 pandemic
◊ Improvements include using interactive software to view and learn about constellations and deep space objects, utilize crowdsourcing astronomy research as part of class, and developing hands-on astronomy projects for students to conduct remotely

Teaching Assistant for Physics 1800 – Conceptual Physics (Spring 2020, Lecturer: J. R. Dennison, Utah State University)
◊ Taught recitation, helped with lab/demos, and filled in as lecturer for class of 50 students
◊ Typical comments in student surveys include:
  ♦ “One of my favorite TA’s I have had, always helps.”
  ♦ “He’s the only reason I passed this Physics class. Zack breaks down the problem and explains it in a way that non-Physics majors can understand.”

Filled in as Lecturer for
PHYS 1800 in Spring 2020 (1 day, Lecturer: J. R. Dennison)
PHYS 1040 in Fall 2020 (1 ½ weeks, Lecturer: David Smith)
PHYS 1010 in Fall 2020 (high school distance learning course, 1 ½ weeks, Lecturer: David Smith)
◊ Prepared and delivered my own lectures as substitute for the classes
Utilized active learning techniques such as group discussions and break out groups 
Both in person and virtual teaching was employed

**Academic Tutoring** (August 2014 – January 2016, Utah Valley University)
- Provided tutoring for sciences (primarily for physics but also math, engineering, and chemistry), either in drop-in tutoring setting or group sessions for specific classes
- Earned College Reading and Learning Association Level 1 Certification

**Substitute Teacher** (Nebo School District, Spring 2015)
- Substitute taught 6th grade

**Service**

**American Physical Society Four Corners Region (APS 4CS) Executive Committee Student-Member-at-Large** (Fall 2021 – Fall 2022)
- Involved in outreach primarily through actively running social media accounts
- Met with committee monthly or more to help further goals of APS 4CS

**Judged the Harry Lustig Award Presentations at the American Physical Society’s Four Corners Region Conference as part of the Harry Lustig Award Committee** (Fall 2022)

**Empower Teaching Excellence Committee Member for Explore College Teaching Subcommittee** (Spring 2021 – Fall 2022, Utah State University)
- Provided insights and input at regular meetings
- Experience included performing outreach, organizing workshops, advocating for students and faculty with the graduate school, advocating for ETE programs and collaborating with other ETE subcommittees

**Diversity Equity and Inclusion Intern for Office of Equity** (Spring 2021, Utah State University, Mentor: Emmalee Fishburn)
- Involved adding new perspective as a STEM graduate student to improve the newly created diversity, equity, and inclusion training for search committees
- Helped develop and evolve a diversity, equity, and inclusion toolkit for use by search committees

**College of Science Graduate Student Council Physics Representative** (Summer 2018 – Summer 2021, Utah State University)
- Involves planning monthly events for physics graduate students, addressing student concerns, advocating for graduate students, meeting monthly with the College of Science Graduate Student Council, and meeting with Dept. Heads/Deans, etc.
- Successfully advocated for a physics graduate student lounge and improved mental health resources
- Successfully advocated for dental and vision insurance
- Successfully advocated for an Emergency Relief Fund for the College of Science, which provides students financial relief for medical crises
Reviewed Applications for Peak Summer Research Fellowship (Spring 2021)
◊ Involved reviewing complete application packets containing letters of recommendation, personal statement, CV, research proposal, and project timeline
◊ Provided feedback to applicants to aid future applications

Reviewed Abstracts for National Conference on Undergraduate Research (Fall 2020, Council on Undergraduate Research)
◊ Involved reviewing several abstracts submitted by undergraduate researchers for acceptance to present at the 2021 National Conference on Undergraduate Research

Reviewed Undergraduate Research and Creative Opportunities Proposals (Summer 2018, Spring 2019, Fall 2019, Spring 2020, Summer 2020, Fall 2020, Utah State University)
◊ Reviewed packets of letters of recommendation, 5-page project narratives, education plans, timelines, and CV’s for research proposals in STEM fields
◊ Utilized this experience to mentor undergraduates in my research group applying for funding

Judged Undergraduate Presentations at Utah State University’s Student Research Symposium (Spring 2019, Fall 2019, Spring 2020, Fall 2020, Spring 2023, Utah State University)
◊ Provided scoring and constructive feedback for undergraduate research in STEM fields

Reviewed IEEE Transactions on Plasma Science Paper (December 2018)
◊ Conducted a peer review of an IEEE Transactions on Plasma Science paper

President and Vice President of Distinguished Society of Physics Students Club (Fall 2015 – Spring 2016 and Fall 2014 – Summer 2015 respectively, Utah Valley University)
◊ Built physics demonstrations
◊ Conducted outreach that included visiting elementary school classrooms to showcase physics demonstrations to get young minds excited about science
◊ Helped plan and run physics club meetings

Volunteer for American Physical Society Four Corners Meeting (October 2014, Utah Valley University)
◊ Reviewed presentations, helped set-up and take-down, took pictures, asked presenters questions, etc.

**Memberships**

Council on Undergraduate Research (2020 – Present)
American Physical Society (2014 – Present)
Institute of Electrical and Electronics Engineers (2017 – Present)
Sigma Pi Sigma Honors Society (April 2019 – Present)