Measurement of Angle-Resolved Secondary Electron Spectra

Robert Davies
Utah State University

Follow this and additional works at: https://digitalcommons.usu.edu/etd

Part of the Physics Commons

Recommended Citation
https://digitalcommons.usu.edu/etd/1698

This Dissertation is brought to you for free and open access by the Graduate Studies at DigitalCommons@USU. It has been accepted for inclusion in All Graduate Theses and Dissertations by an authorized administrator of DigitalCommons@USU. For more information, please contact digitalcommons@usu.edu.
MEASUREMENT OF ANGLE-RESOLVED SECONDARY ELECTRON SPECTRA

by

Robert E. Davies

A dissertation submitted in partial fulfillment of the requirements for the degree of

DOCTOR OF PHILOSOPHY

in

Physics

Approved:

R. Dennison
Major Professor

W. John Raitt
Committee Member

Charles G. Torre
Committee Member

D. Mark Riffe
Committee Member

Jan V. Sojka
Committee Member

Christine Hult
Committee Member

James P. Shaver
Dean of Graduate Studies

UTAH STATE UNIVERSITY
Logan, Utah

1999
ABSTRACT

Measurement of Angle-Resolved Secondary Electron Spectra

by

Robert E. Davies, Doctor of Philosophy

Utah State University, 1999

Major Professor: Dr. J. R. Dennison
Department: Physics

Theoretical formulations of secondary electron emission over the past 20 years have exceeded the confirming ability of available measurements. An instrument has been developed and tested for the purpose of obtaining simultaneous angle- and energy-resolved (AER) secondary and backscattered electron measurements for energetic electrons incident on conducting surfaces. The instrument is found to be in good working order and the data quality found to be excellent for nearly all angles and energies investigated. A representative set of AER measurements has been acquired for 1500 eV electrons normally incident on polycrystalline gold. The data have been used to construct angle-resolved (AR) spectra and energy-resolved (ER) angular distributions, which have been examined both as surface plots and cross sections. Analysis of the measurements strongly suggests that secondary electrons comprise the bulk of emitted electrons at energies much greater than the traditionally accepted maximum secondary electron energy of 50 eV. Additional evidence suggests the ability to investigate dominant secondary and backscattered electron production mechanisms in several energy domains.

(245 pages)
ACKNOWLEDGMENTS

My experience at Utah State University has been a rich mixture of formal coursework, research, teaching, and what can only be described as “physics banter” in the hallways, restaurants, bar (singular intentional) and coffee house (singular again intentional) of Logan. Many people have contributed to this experience that has filled the past eight years of my life, and it is my intention to acknowledge here as many as I may. There have been teachers, technicians, administrators, financiers, and friends.

Foremost, I am pleased to acknowledge the seemingly inexhaustible enthusiasm, optimism, and patience of my advisor, Dr. J.R. Dennison. His guidance throughout this project is greatly appreciated and I look forward to our continued association, both professional and personal.

I am also pleased to recognize the faculty and staff of the Department of Physics, whose enduring efforts have created an intensely supportive work and study environment in which I have been able to develop both as a physicist and personally. In particular, for continuing enthusiasm and patience in the classroom, and for always answering their doors when I knock, my sincere thanks to Professors Farrell Edwards, Charles Torre, and James Wheeler. They have made themselves available for what must seem at times an unending task of explanation. On all matters administrative, Barbara Phillips, Donna Eckburg, Deborah Reece, Marilyn Griggs, Shawna Johnson, and Melanie Oldroyd have kept beer and hotdog money in my pocket, supplies in my laboratory, and, most importantly, candy in their candy jars. Their support and friendship are warmly felt.

For their diligent review of this work and thoughtful inputs, I gratefully acknowledge the efforts of my committee, Drs. Mark Riffe, John Raitt, Jan Sojka, Charles Torre, and Christine Hult. For their invaluable technical assistance, I would like to acknowledge the contributions of god-level machinist (and fellow grad student basement denizen) Ali Sabbah; Chad Fish and Clint Thompson for their isolation circuit wizardry; Ted Lister for his guidance in gold sample cleaning; Creekside Technologies’ Tom Rush for his electron gun savoir faire (and the key to the double peak); Keithley’s Rose Lockhart for once again keeping my 616’s six-sixteenth; Graham Hunter for his excellent 3-d instrument illustration (p. 26); and Charlotte Fenno for her contribution of the REELS data. Data analysis was accomplished with the software IGOR for the MacIntosh, and the text of this document was prepared entirely on a MacIntosh.
computer using MS Word 98 for the Mac. In particular I would like to thank Apple Computers for a clean, sane alternative to the PC madness, and IGOR's makers, Wavemetrics, Inc., for their outstanding product and customer support: John and A.G., thanks for your phone time.

At this juncture in my formal education I feel it also appropriate to acknowledge important influences of past educators: they include Robert Daczewitz, Bob Haux, and Betty Trezona of Spearfish High School (math, physics, and literature/drama/creative writing, respectively), and Hans Graetzer and Warren Hein of South Datota State University's Department of Physics.

My studies have been made possible financially by a number of U.S. Government-sponsored programs. I gratefully acknowledge the support of NASA's Rocky Mountain Space Grant Consortium—and in particular my contact with this organization, Professor Jan Sojka, without whose efforts I would not have come to USU—the U.S. Air Force Office of Scientific Research Summer Researcher's Program (under the sponsorship of Dr. Charles Stein and the men and women of Phillips Laboratory's Space Environmental Effects Branch), and NASA's Graduate Student Researcher's Program (under the sponsorship of Dr. Dale Fergusson and NASA's Lewis Research Center). I am honored to have been a part of these programs, and deeply indebted to the people of the United States for their support of my experience at USU through our government.

For their invaluable support on the personal side, I reserve my deepest gratitude for the oldies, Doug, Karen, Tim, Paula, Dave, Susan, and Dana, and the Logan crowd, Janice, Anna, Sarita, Ludger, Rita, Paul, and Patti. And especially for Teresa, Balraj, Marc, and the Gooster... "you can all be replaced," and for Mom, whose love and encouragement have always made me believe in my dreams.

Robert E. Davies
## CONTENTS

<table>
<thead>
<tr>
<th>Section</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>ABSTRACT</td>
<td>iii</td>
</tr>
<tr>
<td>ACKNOWLEDGMENTS</td>
<td>iv</td>
</tr>
<tr>
<td>LIST OF TABLES</td>
<td>xi</td>
</tr>
<tr>
<td>LIST OF FIGURES</td>
<td>xii</td>
</tr>
<tr>
<td>ABBREVIATIONS, SYMBOLS, AND NOTATION</td>
<td>xvi</td>
</tr>
<tr>
<td>1. INTRODUCTORY</td>
<td>1</td>
</tr>
<tr>
<td>1.1 Motivation</td>
<td>1</td>
</tr>
<tr>
<td>1.2 Objective</td>
<td>2</td>
</tr>
<tr>
<td>1.3 Synopsis</td>
<td>3</td>
</tr>
<tr>
<td>2. SECONDARY ELECTRON EMISSION</td>
<td>5</td>
</tr>
<tr>
<td>2.1 Historical Perspective</td>
<td>5</td>
</tr>
<tr>
<td>2.2 General Description of SEE</td>
<td>6</td>
</tr>
<tr>
<td>2.3 Theory</td>
<td>9</td>
</tr>
<tr>
<td>2.3 (a) Semiempirical Formulation</td>
<td>9</td>
</tr>
<tr>
<td>2.3 (b) Quantum Mechanical Formulation</td>
<td>13</td>
</tr>
<tr>
<td>2.3 (c) Backscattering</td>
<td>16</td>
</tr>
<tr>
<td>2.3 (d) Discussion</td>
<td>16</td>
</tr>
<tr>
<td>2.4 Observation</td>
<td>17</td>
</tr>
<tr>
<td>2.5 Previous Investigations of Gold</td>
<td>21</td>
</tr>
<tr>
<td>3. INSTRUMENTATION AND METHODS</td>
<td>24</td>
</tr>
<tr>
<td>3.1 Instrument</td>
<td>24</td>
</tr>
<tr>
<td>3.1 (a) Vacuum Chamber</td>
<td>24</td>
</tr>
<tr>
<td>3.1 (b) Electron Gun</td>
<td>25</td>
</tr>
<tr>
<td>3.1 (c) Chamber Apparatus</td>
<td>25</td>
</tr>
<tr>
<td>3.1 (d) Measurement Circuits</td>
<td>30</td>
</tr>
<tr>
<td>3.1 (e) Data Acquisition</td>
<td>36</td>
</tr>
<tr>
<td>3.1 (f) Summary of Recent Modifications</td>
<td>37</td>
</tr>
<tr>
<td>3.2 Measured Quantities</td>
<td>38</td>
</tr>
<tr>
<td>3.2 (a) Notation</td>
<td>38</td>
</tr>
<tr>
<td>3.3 (b) Definitions</td>
<td>39</td>
</tr>
<tr>
<td>3.3 Measurement Technique</td>
<td>42</td>
</tr>
<tr>
<td>Section</td>
<td>Title</td>
</tr>
<tr>
<td>---------</td>
<td>----------------------------------------------------------------------</td>
</tr>
<tr>
<td>4.3 (a)</td>
<td>$\delta_{EE}$ Determination</td>
</tr>
<tr>
<td>4.3 (b)</td>
<td>$\delta_{ER}$ Determination</td>
</tr>
<tr>
<td>4.3 (c)</td>
<td>$\delta_{RR}$ Determination</td>
</tr>
<tr>
<td>4.3 (d)</td>
<td>Determination of the Total SE Yield</td>
</tr>
<tr>
<td>4.3 (e)</td>
<td>BSE Yield Determinations</td>
</tr>
<tr>
<td>4.1</td>
<td>Samples</td>
</tr>
<tr>
<td>4.1 (a)</td>
<td>Choice of Sample Material</td>
</tr>
<tr>
<td>4.1 (b)</td>
<td>Sample Preparation</td>
</tr>
<tr>
<td>4.2</td>
<td>Measurement Notes</td>
</tr>
<tr>
<td>4.3</td>
<td>Summary of Principle Findings</td>
</tr>
<tr>
<td>4.4</td>
<td>$\delta$ and $\eta$ as Functions of Surface</td>
</tr>
<tr>
<td>4.4 (a)</td>
<td>Description of Results</td>
</tr>
<tr>
<td>4.4 (b)</td>
<td>Analysis</td>
</tr>
<tr>
<td>4.4 (c)</td>
<td>Conclusions</td>
</tr>
<tr>
<td>4.5</td>
<td>$\delta$ and $\eta$ as Functions of Location</td>
</tr>
<tr>
<td>4.5 (a)</td>
<td>Description of Results</td>
</tr>
<tr>
<td>4.5 (b)</td>
<td>Analysis and Conclusions</td>
</tr>
<tr>
<td>4.6</td>
<td>$\delta$ and $\eta$ as Functions of Beam Energy</td>
</tr>
<tr>
<td>4.6 (a)</td>
<td>Description of Results</td>
</tr>
<tr>
<td>4.6 (b)</td>
<td>Analysis</td>
</tr>
<tr>
<td>4.6 (c)</td>
<td>Conclusions</td>
</tr>
<tr>
<td>4.7</td>
<td>$\delta$ and $\eta$ for a Negatively Biased Surface</td>
</tr>
<tr>
<td>4.7 (a)</td>
<td>Description of Results</td>
</tr>
<tr>
<td>4.7 (b)</td>
<td>Analysis</td>
</tr>
<tr>
<td>4.7 (c)</td>
<td>Conclusions</td>
</tr>
<tr>
<td>5.1</td>
<td>The Challenge of AR SE Measurements</td>
</tr>
<tr>
<td>5.2</td>
<td>Measurement Notes</td>
</tr>
<tr>
<td>5.3</td>
<td>Summary of Principal Findings</td>
</tr>
<tr>
<td>5.3 (a)</td>
<td>AR Yield Results</td>
</tr>
<tr>
<td>5.3 (b)</td>
<td>Instrument Performance</td>
</tr>
<tr>
<td>5.4</td>
<td>Measurement Procedure</td>
</tr>
<tr>
<td>5.5</td>
<td>Description of Results</td>
</tr>
<tr>
<td>5.5 (a)</td>
<td>Data Conditioning</td>
</tr>
<tr>
<td>5.5 (b)</td>
<td>Trends in the Data</td>
</tr>
</tbody>
</table>
5.6 AR BSE Yield Analysis
5.6 (a) BSE Angular Distribution Functions
5.6 (b) Incident Beam Alignment
5.6 (c) \( \eta(\alpha') \) Magnitudes
5.6 (d) Systematic Error in Total Yields: Estimate Refinement
5.7 AR SE Yield Analysis
5.7 (a) SE Angular Distribution Functions
5.7 (b) \( \delta(\alpha') \) Magnitudes

5.8 Conclusions

6. ANGLE-RESOLVED SPECTRA MEASUREMENTS
6.1 Measurement Notes
6.2 Summary of Principal Findings
6.3 Measurement Procedure
6.4 Description of Results
6.4 (a) AR Spectra Sets
6.4 (b) Repeatability Between Surfaces
6.4 (c) Fine Structure
6.4 (d) Discussion
6.5 High-Energy (>50 eV) Spectra Analysis
6.5 (a) General Comments
6.5 (b) Distinction Between the Low-Energy Tail and Inelastic Peak
6.5 (c) Elastic Peaks: Intensities, Structure, and Behavior as a Function of Emission Angle
6.5 (d) Elastic Peak Fine Structure
6.5 (e) Inelastic Peaks
6.5 (f) Low-Energy Tails
6.6 Low-Energy (<50 eV) Spectra Analysis
6.6 (a) Low-Energy AR Spectra Intensities
6.6 (b) AR SE Spectra Behavior as a Function of Emission Angle:
   The Low-Energy AER Distribution Function
6.6 (c) SE’s vs BSE’s: Where Does the SE Tail End?
6.6 (d) SE Peak Fine Structure
6.7 Discussion

7. SURFACE PLOTS AND ENERGY-RESOLVED ANGULAR DISTRIBUTIONS
7.1 AR Spectra Surface Plots
7.2 The Distinction Between AR Energy Distributions and ER Angular Distributions
7.3 The Value of ER Angular Distributions
7.4 Constructing ER Angular Distributions from AR Spectra
7.5 Results and Analysis ........................................................................................................... 149
  7.5 (a) Low-Energy ER Angular Distribution (0-450 eV) ...................................................... 152
  7.5 (b) High-Energy ER Angular Distribution (450-1500 eV) ............................................... 158
7.6 Discussion .............................................................................................................................. 164
7.7 Conclusions .......................................................................................................................... 166

8. CONCLUSION ............................................................................................................................ 169
  8.1 Principal Results—Summary of Instrument Performance and Experimental Findings.......... 169
    8.1 (a) Instrument Performance ............................................................................................... 169
    8.1 (b) Measurements and Analysis ......................................................................................... 170
  8.2 Suggested Instrument Modifications ....................................................................................... 171
  8.3 Recommendations for Further Research .................................................................................. 174
  8.4 Concluding Remarks ............................................................................................................... 175

REFERENCES .............................................................................................................................. 177
APPENDICES .............................................................................................................................. 182
APPENDIX A: CALCULATIONS ................................................................................................. 183
  A.1 Random Error in δ and η Determinations .............................................................................. 183
  A.2 Systematic Error in δ and η Determinations .......................................................................... 183
    A.2 (a) The Gamma Factor ........................................................................................................ 183
    A.2 (b) Estimating Gamma ........................................................................................................ 185
    A.2 (c) Changing Surface Conditions ....................................................................................... 187
  A.3 Total SE and BSE Yield Determinations for a Negatively Biased Sample.............................. 189
  A.4 SE and BSE Deflection Due to Ambient Electric and Magnetic Fields ................................. 190
APPENDIX B: SAMPLE SURFACE SUMMARY .......................................................................... 194
APPENDIX C: ANGLE-RESOLVED DIAGNOSTIC MEASUREMENTS ............................................... 198
  C.1 Rotatable Detector ................................................................................................................. 198
    C.1 (a) RD Alignment ................................................................................................................ 198
    C.1 (b) RD-Produced Secondaries ......................................................................................... 199
    C.1 (c) Energy Resolution ........................................................................................................ 199
    C.1 (d) Charging within the RD ............................................................................................... 202
  C.2 Sample Biasing ....................................................................................................................... 204
  C.3 Ambient Field Effects ............................................................................................................. 210
    C.3 (a) Spectra Repeatability (same surface) ............................................................................ 211
    C.3 (b) Spectra Repeatability Between Surfaces ...................................................................... 211
    C.3 (c) Spectra symmetry about the Beam Axis ...................................................................... 215
  C.4 Surface Contamination Effects .............................................................................................. 217
  C.5 Sample Current as a Measure of Beam Current .................................................................. 217
C.6 Diagnostic Summary ................................................................. 219

APPENDIX D: DATA REDUCTION ....................................................... 221
  D.1 Data Collection and Conditioning .................................................. 221
  D.2 Sample Current Reduction ............................................................ 221
  D.3 RD Current Reduction ................................................................. 223

CURRICULUM VITAE ............................................................................. 226
## TABLES |

<table>
<thead>
<tr>
<th>Table</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>4.1</td>
<td>54</td>
</tr>
<tr>
<td>4.2</td>
<td>58</td>
</tr>
<tr>
<td>4.3</td>
<td>65</td>
</tr>
<tr>
<td>4.4</td>
<td>74</td>
</tr>
<tr>
<td>5.1</td>
<td>95</td>
</tr>
<tr>
<td>5.2</td>
<td>97</td>
</tr>
<tr>
<td>5.3</td>
<td>99</td>
</tr>
<tr>
<td>6.1</td>
<td>115</td>
</tr>
<tr>
<td>6.2</td>
<td>116</td>
</tr>
<tr>
<td>6.3</td>
<td>116</td>
</tr>
<tr>
<td>6.4</td>
<td>124</td>
</tr>
<tr>
<td>A.1</td>
<td>184</td>
</tr>
<tr>
<td>A.2</td>
<td>184</td>
</tr>
<tr>
<td>A.3</td>
<td>188</td>
</tr>
<tr>
<td>C.1</td>
<td>217</td>
</tr>
<tr>
<td>D.1</td>
<td>222</td>
</tr>
<tr>
<td>D.2</td>
<td>222</td>
</tr>
<tr>
<td>Figure</td>
<td>Title</td>
</tr>
<tr>
<td>--------</td>
<td>----------------------------------------------------------------------</td>
</tr>
<tr>
<td>2-1</td>
<td>Schematic depicting electron emission from a surface resulting from</td>
</tr>
<tr>
<td></td>
<td>energetic electron bombardment</td>
</tr>
<tr>
<td>2-2</td>
<td>More detailed view of the physical processes resulting from energetic</td>
</tr>
<tr>
<td></td>
<td>electron bombardment of a surface</td>
</tr>
<tr>
<td>2-3</td>
<td>Comparison of cosine distribution with cosine distribution modified to</td>
</tr>
<tr>
<td></td>
<td>include surface refraction</td>
</tr>
<tr>
<td>2-4</td>
<td>Typical SE yield vs energy curve</td>
</tr>
<tr>
<td>2-5</td>
<td>Representation of a typical SE and BSE energy spectrum</td>
</tr>
<tr>
<td>2-6</td>
<td>Previous SEE measurements for Au, grouped by author</td>
</tr>
<tr>
<td>2-7</td>
<td>Previous SEE measurements of Au, grouped by type</td>
</tr>
<tr>
<td>3-1</td>
<td>Schematic depicting the chamber apparatus</td>
</tr>
<tr>
<td>3-2</td>
<td>Sample-beam-detector geometry</td>
</tr>
<tr>
<td>3-3</td>
<td>Schematic depicting the rotatable detector</td>
</tr>
<tr>
<td>3-4</td>
<td>Emission angle intercepted by the rotatable detector as a function of</td>
</tr>
<tr>
<td></td>
<td>detector position</td>
</tr>
<tr>
<td>3-5</td>
<td>Schematic depicting sample-current detection circuit</td>
</tr>
<tr>
<td>3-6</td>
<td>Schematic depicting connection scheme for primary and tertiary sample-</td>
</tr>
<tr>
<td></td>
<td>current detection circuits</td>
</tr>
<tr>
<td>3-7</td>
<td>Schematic depicting RD-current detection circuit</td>
</tr>
<tr>
<td>3-8</td>
<td>Schematic depicting connection scheme for RD-current detection circuit</td>
</tr>
<tr>
<td>3-9</td>
<td>Schematic depicting experiment geometry</td>
</tr>
<tr>
<td>4-1</td>
<td>Gold sample mounted on rhodium-coated blank, ready for vacuum insertion</td>
</tr>
<tr>
<td>4-2</td>
<td>$\delta$ and $\eta$ as functions of surface and location for samples</td>
</tr>
<tr>
<td></td>
<td>(a) $A$, (b) $B$, and (c) $D$, respectively</td>
</tr>
<tr>
<td>4-3</td>
<td>Scanning electron micrographs of polycrystalline Au before (top) and</td>
</tr>
<tr>
<td></td>
<td>after (bottom) argon sputtering</td>
</tr>
<tr>
<td>4-4</td>
<td>Energy dispersive x-ray analysis (EDX) spectra for samples of</td>
</tr>
<tr>
<td></td>
<td>polycrystalline Au before (top) and after (bottom) argon sputtering</td>
</tr>
<tr>
<td>4-5</td>
<td>Schematic depicting the five standard measurement locations on the Au</td>
</tr>
<tr>
<td></td>
<td>samples</td>
</tr>
</tbody>
</table>
4-6 $\delta$, $\eta$, and $\sigma$ as functions of incident electron energy: comparison of (a) surface A01 and (b) surface D9 measurements with previous experiment and empirical SEE models...................... 71

4-7 $\delta$ and $\eta$ as a function of negative sample bias for four different surfaces................................................. 75

4-8 Energy diagram depicting potential wells an escaping electron sees in its journey from an emitting surface to an absorbing surface when: (a) the emitter's work function is less than the absorber's, and (b) the emitter's work function is greater than the absorber's........................................... 77

4-9 Energy diagram depicting the change in potential, due to negative biasing of the emitter by an amount $\Phi_s$, seen by an electron leaving the emitting surface and going to an absorbing surface.............................................................................................. 78

4-10 Energy diagrams depicting the cases of (a) $\phi_{Au} = \phi_{tertiaries}$, (b) $\phi_{Au} < \phi_{tertiaries}$, and (c) $\phi_{Au} > \phi_{tertiaries}$, where the Fermi energies of the Au sample and two tertiary samples are tied together .......................................................... .................................................................. 81

5-1 Schematic depicting quality of angle-resolved measurements as a function of emission angle and energy................................................................................................................................................. 88

5-2 $\eta(\alpha')$'s for surfaces D2, D6, and D7. Data for each surface represents that for both 0 V and -1.50 V sample biases.............................................................................................................................. 91

5-3 $\delta(\alpha')$'s for surfaces D2, D6, and D7 at (a) 0 V and (b) -1.5 V sample bias......................................................... 92

6-1 Typical AR spectrum of this investigation. $E_B = 1.5$ keV, normal incidence......................................................... 104

6-2 Typical AR spectrum of this investigation plotted on a semi-log scale................................................................. 108

6-3 Surface D6 AR BSE spectra plotted on (a) linear and (b) semilog scales........................................................................ 111

6-4 Surface D6 AR SE spectra, adjusted for -1.5 V sample bias....................................................................................... 113

6-5 Surface D7 AR SE spectra, adjusted for -1.5 V sample bias....................................................................................... 114

6-6 Comparison of surface D6 and D7 AR SE spectra for selected emission angles................................................................. 117

6-7 Average AR SE spectra for three scattering angles on surface D7 depicting possible $\alpha$-dependent fine structure .................................................................................................................................................. 119

6-8 Log plot of surface D6 AR SE spectra set depicting evidence for $\alpha$-dependent fine structure in the low-energy peak tail.................................................................................................................................................. 120

6-9 Possible fine structure in the surface D6 AR elastic peak spectra ....................................................................................... 121

6-10 Comparison of partial AR BSE yields for the low-energy tails, inelastic peaks, and elastic peaks, as a function of emission angle (Surface D6).................................................................................. 125

6-11 Angular distribution of elastic yield as computed by Jablonski [1991] and measured in the present work (Surface D6).................................................................................................................................................. 129

6-12 Reflected electron energy loss (REELS) spectra for sample D, performed for ultrasonically cleaned surface (upper curves) and sputtered surfaces (lower curves)....................................................................................... 131
6-13 Comparison of partial AR yields for the low-energy (SE) peaks, low-energy tails, inelastic peaks, and elastic peaks, as a function of emission angle (Surface D6).................. 133

6-14 Fits of (a) Eq. (6.1) and (b) Eq. (6.2) to the surface D7, $\alpha = 17^\circ$, AR low-energy spectrum ....... 135

6-15 Comparison of surface D6 AR SE spectra to simple distribution functions................................. 137

6-16 Surface D6 low-energy AR spectra revealing trends in electron energy distributions ............. 139

6-17 Fits of Eq. (6.2) (solid lines) to surface D7 AR SE spectra (circles) demonstrating deviation of the fits from the data at $\sim 20$ eV for all angles .............................................................. 141

7-1 Surface plot of surface D6 AR spectra: (a) 0-1500 eV, and (b) 0-50 eV............................. 145

7-2 Surface plot of surface D7 AR spectra set ................................................................................. 147

7-3 Example of conversion from (a) AR spectra to (b) ER angular distributions (surface D6)......... 150

7-4 Example of normalized ER angular distributions (constructed from surface D6 AR spectra) .... 151

7-5 Normalized ER angular distributions for surface D6 (0-1500 eV) ......................................... 153

7-6 Normalized ER angular distributions for surface D7 (0-50 eV) ............................................. 154

7-7 Normalized ER angular distributions constructed from surface D6 AR spectra: (a) oblique view, showing relief in the surface and (b) head-on view, showing intensity falloff with energy ................................................................. 155

7-8 Normalized ER angular distributions constructed from surface D7 AR spectra: (a) oblique view, showing relief in the surface and (b) head-on view, showing intensity falloff with energy ................................................................. 156

7-9 Angular distributions for selected energy populations in the 0-50 eV range for (a) surface D6 and (b) surface D7 ...................................................................................... 157

7-10 Normalized ER angular distributions through 450 eV (surface D6): (a) oblique view, depicting relief and (b) head-on view, depicting intensity behavior with emission energy ................. 159

7-11 Angular distributions for selected energy populations in the 0-450 eV (surface D6) .............. 160

7-12 Normalized ER angular distributions through 1400 eV (surface D6): (a) oblique view, depicting relief and (b) head-on view, depicting intensity behavior with emission energy ................... 161

7-13 Angular distributions for selected energy populations in the 0-450 eV (surface D6) ......... 161

7-14 Angular distributions for selected energy populations in the elastic peak (surface D6) .......... 162

7-15 Schematic depicting five general categories of emitted electrons as a function of emission energy. (Surface D6, $\alpha = 17^\circ$) .......................................................... 167

7-16 Comparison between (a) theory and (b) measurement of the angular distributions of emitted SE’s and BSE’s as a function of emission energy ........................................ 168
8-1  Schematic depicting double-sphere design for measurement of angle-resolved SE spectra........ 173
A-1  SE's emitted into the shaded regions are subject to collection by the tertiary samples when the primary and tertiary samples are biased to +50 V .......................................................... 185
A-2  Currents to and from a sample under different conditions of sample bias ......................... 189
A-3  Minimum transverse electric and magnetic fields required to deflect an SE or BSE out of the first RD aperture as functions of electron energy .......................................................... 191
A-4  Deflection of an SE or BSE due to an ambient, transverse magnetic field......................... 192
C-1  (a) RD-produced secondaries and (b) comparison of RD- and sample-produced SE signals for surface D7........................................................................................................................................ 200
C-2  Angle-resolved spectrum depicting double BSE peak for surfaces D6 and B3 ................ 201
C-3  Diagnostic AR SE spectra identifying the presence of electrostatic charging within the RD .... 203
C-4  Angle-resolved SE and BSE yields as measured for surface D6 under varying conditions of negative sample bias, $\phi_3$ ................................................................................................................ 206
C-5  Diagnostic AR SE spectra as measured for surface B2 under varying conditions of negative sample bias for scattering angles of (a) $-19^\circ$, (b) $+53^\circ$, and (c) $+65^\circ$.................................................. 207
C-6  Diagnostic AR SE spectra, as measured for surface D1 under varying conditions of negative sample bias for scattering angles of $-17^\circ$ [(a) and (b)] and $-19^\circ$ [(c) and (d)]........................................... 208
C-7  Schematic depicting the effect on SE trajectories of an electric field due to negative sample biasing ...................................................................................................................... 209
C-8  Repeat AR SE spectra for three different scattering angles on surface D7 ....................... 212
C-9  Repeat AR SE spectra performed on surface D9 ................................................................. 213
C-10 Comparison of surface D6 and D7 AR SE spectra for selected emission angles .............. 214
C-11 Symmetry check of AR SE spectra for various angles on surfaces D6 and D7 ............... 216
C-12 Comparison of AR SE spectra for clean and contaminated surfaces ............................... 218
D-1 Integrated current and AR spectrum plots typical of this investigation .......................... 224
D-2 (a) RD current vs bias and (b) AR spectrum depicting the importance of scaling the RD current data with the sample current data ............................................................... 225
<table>
<thead>
<tr>
<th>Abbreviation</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>AR</td>
<td>angle-resolved</td>
</tr>
<tr>
<td>AI</td>
<td>angle-integrated</td>
</tr>
<tr>
<td>BSE</td>
<td>backscattered electron</td>
</tr>
<tr>
<td>CA</td>
<td>chamber apparatus</td>
</tr>
<tr>
<td>DAQ</td>
<td>data acquisition</td>
</tr>
<tr>
<td>EDX</td>
<td>energy-dispersive x-ray</td>
</tr>
<tr>
<td>ER</td>
<td>energy-resolved</td>
</tr>
<tr>
<td>ESA</td>
<td>electron-stimulated adsorption</td>
</tr>
<tr>
<td>ESD</td>
<td>electron-stimulated desorption</td>
</tr>
<tr>
<td>HV</td>
<td>high voltage</td>
</tr>
<tr>
<td>IP</td>
<td>inelastic peak</td>
</tr>
<tr>
<td>LET</td>
<td>low-energy tail</td>
</tr>
<tr>
<td>PE</td>
<td>primary electron</td>
</tr>
<tr>
<td>QM</td>
<td>quantum mechanical</td>
</tr>
<tr>
<td>RD</td>
<td>rotatable detector</td>
</tr>
<tr>
<td>RGA</td>
<td>residual gas analyzer</td>
</tr>
<tr>
<td>SE</td>
<td>secondary electron</td>
</tr>
<tr>
<td>SEE</td>
<td>secondary electron emission</td>
</tr>
<tr>
<td>SEM</td>
<td>scanning electron microscope</td>
</tr>
<tr>
<td>UHV</td>
<td>ultra-high vacuum</td>
</tr>
<tr>
<td>$\vec{E}$</td>
<td>ambient electric field</td>
</tr>
<tr>
<td>$\vec{B}$</td>
<td>ambient magnetic field</td>
</tr>
<tr>
<td>$\alpha$</td>
<td>emission angle</td>
</tr>
<tr>
<td>$E_B$</td>
<td>incident electron beam energy</td>
</tr>
<tr>
<td>$\Phi_{RD}$</td>
<td>sample bias</td>
</tr>
<tr>
<td>$d\Omega$</td>
<td>solid angle subtended by the RD</td>
</tr>
<tr>
<td>$n(x,E_0)$</td>
<td>average number of SE's produced per incident PE in a layer of thickness $dx$ at a depth $x$ below the surface</td>
</tr>
<tr>
<td>$f(x)$</td>
<td>probability for an SE to migrate to the surface from a depth $x$ and escape</td>
</tr>
<tr>
<td>$-\frac{dE}{dx}$</td>
<td>average PE energy loss per unit path length (stopping power)</td>
</tr>
<tr>
<td>$\delta_{max}$</td>
<td>maximum SE yield for a given material</td>
</tr>
<tr>
<td>$E_{max}$</td>
<td>incident energy at which $\delta_{max}$ occurs</td>
</tr>
<tr>
<td>$\varphi_{RD}$</td>
<td>detector position</td>
</tr>
<tr>
<td>$E_e$</td>
<td>emitted energy</td>
</tr>
<tr>
<td>$\delta$</td>
<td>total SE yield</td>
</tr>
<tr>
<td>$\delta_{MER}$</td>
<td>angle-energy-resolved SE yield</td>
</tr>
<tr>
<td>$\delta_{AR}$</td>
<td>angle-resolved SE yield</td>
</tr>
<tr>
<td>$\delta_{ER}$</td>
<td>energy-resolved SE yield</td>
</tr>
<tr>
<td>$\delta(\alpha')$</td>
<td>AR SE yield</td>
</tr>
<tr>
<td>$\delta(\alpha)$</td>
<td>SE angular distribution function</td>
</tr>
<tr>
<td>$\delta(E_e')$</td>
<td>energy-resolved SE yield</td>
</tr>
<tr>
<td>$\delta(E_e)$</td>
<td>SE energy distribution function</td>
</tr>
<tr>
<td>$\delta(\alpha',E_e')$</td>
<td>angle-energy-resolved SE yield</td>
</tr>
<tr>
<td>$\delta(\alpha,E_e)$</td>
<td>AR SE energy distribution function</td>
</tr>
<tr>
<td>$\delta(\alpha,E_e')$</td>
<td>energy-resolved SE angular distribution function</td>
</tr>
<tr>
<td>Symbol</td>
<td>Description</td>
</tr>
<tr>
<td>--------</td>
<td>-------------</td>
</tr>
<tr>
<td>$\delta(\alpha,E_e)$</td>
<td>SE angle and energy distribution function</td>
</tr>
<tr>
<td>$\eta$</td>
<td>total BSE yield</td>
</tr>
<tr>
<td>$\eta(\alpha')$</td>
<td>AR BSE yield</td>
</tr>
<tr>
<td>$\eta(\alpha)$</td>
<td>BSE angular distribution function</td>
</tr>
<tr>
<td>$\eta(E'_e)$</td>
<td>energy-resolved BSE yield</td>
</tr>
<tr>
<td>$\eta(E_e)$</td>
<td>BSE energy distribution function</td>
</tr>
<tr>
<td>$\eta(\alpha',E'_e)$</td>
<td>angle-energy-resolved BSE yield</td>
</tr>
<tr>
<td>$\eta(\alpha',E_e)$</td>
<td>AR BSE energy distribution function</td>
</tr>
<tr>
<td>$\eta(\alpha,E'_e)$</td>
<td>energy-resolved BSE angular distribution function</td>
</tr>
<tr>
<td>$\eta(\alpha,E_e)$</td>
<td>BSE angle and energy distribution function</td>
</tr>
<tr>
<td>$\sigma$</td>
<td>total emitted electron yield $(\delta+\eta)$</td>
</tr>
<tr>
<td>$\sigma(\alpha')$</td>
<td>AR total emitted yield</td>
</tr>
<tr>
<td>$\sigma(\alpha)$</td>
<td>emitted-electron angular distribution function</td>
</tr>
<tr>
<td>$\sigma(E'_e)$</td>
<td>energy-resolved emitted-electron yield</td>
</tr>
<tr>
<td>$\sigma(E_e)$</td>
<td>emitted-electron energy distribution function</td>
</tr>
<tr>
<td>$\sigma(\alpha',E'_e)$</td>
<td>angle-energy-resolved emitted-electron yield</td>
</tr>
<tr>
<td>$\sigma(\alpha',E_e)$</td>
<td>AR emitted-electron energy distribution function</td>
</tr>
<tr>
<td>$\sigma(\alpha,E'_e)$</td>
<td>energy-resolved emitted-electron angular distribution function</td>
</tr>
</tbody>
</table>
CHAPTER 1
INTRODUCTORY

“What is the path? There is no path. On, into the unknown...”
—Goethe, in Faust

At the mouth of Logan Canyon, water descending into Cache Valley is ushered through a hydroelectric station, its energy of descent harnessed to rotate large magnets and induce an electric current—a flow of electrons. In this manner the station supplies electrical power to the valley. Some of this power has been utilized in Room 21 in the basement of Utah State University’s Science, Engineering, and Research (SER) building—my laboratory. A tungsten filament has been connected to the power grid (the electron flow); a fountain of electrons erupts from its tip as a result and is formed, focused, and ushered from the filament to a near-atomically-clean gold surface. On striking the gold, tungsten electrons interact with gold electrons; electrons are excited, absorbed, emitted, and counted. Electron currents are measured. Via the motion of electrons, data is reduced on my computer. More filaments are heated and more electrons emitted, striking phosphor screens and displaying the results of my electron measurements on my Macintosh screen. This is a dissertation about electrons—specifically, about the phenomenon of secondary electron emission. But generally, it’s just about electrons.

1.1 Motivation

Secondary electron emission (SEE)—the ejection of electrons from a surface as a result of energetic electron, ion, or photon bombardment—is of interest from the perspectives of both practical application and fundamental physical theory. On a practical level, SEE plays a key role in myriad technologies (e.g., scanning and transmission electron microscopy, ultra-low pressure sensing, and photomultiplication) and underlies a number of other observed phenomena (e.g., the “snapover” of insulators in a plasma, some forms of electric discharge from surfaces, and the buildup of electrical charge on spacecraft surfaces). Fundamentally, SEE involves the exchange of energy between electrons. The
study of this phenomenon, therefore, is valuable to anyone interested in the details of how fundamental particles interact—that is, anyone who wants to know more about just what an electron "really" is.

Based on nearly a century of observation, a great deal is known regarding the general nature of SEE. How many secondary electrons (SE's) are produced, and with what energies, has been heavily investigated as a function of various sample and incident beam parameters. The results of these investigations are well understood qualitatively through a number of semiempirical theoretical formulations—essentially classical—which view SEE as the result of a simple momentum transfer between incident primary electrons (PE's) and electrons residing within the solid. But SEE is not, in fact, a single phenomenon; rather it is the result of multiple underlying mechanisms involving various flavors of electron-electron and electron-ion interactions. Details of these individual interactions—their relative importance, and their dependence on such factors as incident particle, incident energy, and material—are as yet poorly understood. Theories attempting to model SEE on this more detailed and fundamental level have been offered, each with its own assumptions and predictions; their level of detail, however, has exceeded the confirming ability of available data. This fact is the underlying motivation behind the work presented in this dissertation.

### 1.2 Objective

Pertaining to SEE observation, two prominent theoreticians in the field have stated that

"the maximum information about the SEE process can be obtained by measuring the number of SE's emitted per s from 1 cm² of the surface with energy E in the direction Ω"

[Rösler and Brauer, 1981a, p. 162, emphasis added]. In this spirit, the focus of the work presented here has been the development of a new tool for secondary electron emission investigation—an apparatus and method for acquiring simultaneous angle- and energy-resolved secondary electron (SE) measurements (i.e., angle-resolved SE spectra). Further, it has been desired to use this instrument to obtain new data—for
energetic electrons normally incident on conducting surfaces—and to apply these data to evaluation of fundamental SEE theory. Thus the objectives of this work have been threefold:

(i) development of an instrument and technique for the measurement of angle-resolved secondary electron spectra
(ii) acquisition of new data
(iii) application of the new data to theoretical evaluation.

1.3 Synopsis

Material presented in this report details an investigation of considerable length; presentation of this material is arranged in the following sequence:

Chapter 2. A substantial review of the SEE literature was accomplished as a necessary part of this investigation. Based on this review, a general description of SEE is presented, followed by a summary of SEE theory and observation. Particular attention is paid to the energy and angular distribution of SE's. A summary of electron backscattering is included. The chapter concludes with a summary of past SEE observation for energetic electrons incident on gold.

Chapter 3. Details of the experimental apparatus and method are discussed in this chapter and the quantities to be measured are precisely defined. Anyone of rapt enthusiasm for a nice, warm op amp and a DMM on a chilly November evening will relish this chapter.

Chapters 4-7. Experimental results and analysis of this investigation are divided into two general categories: total yield measurements, presented in Chap. 4, and angle-resolved measurements, presented in Chaps. 5-7. Among the angle-resolved measurements, yields, spectra, and angular distributions are discussed. Total and angle-resolved yields are considered in detail, with results applied primarily to an assessment of instrument and method. The angle-resolved spectra and energy-resolved angular distributions presented in Chaps. 6 and 7 are the principal results of this investigation.

Chapter 8. On completion of the detailed discussions of Chaps. 4-7, results are summarized in this chapter. Also, suggested improvements to the measurement apparatus and technique are detailed and thoughts are presented regarding the continuation of this research.
Information necessary to the arguments presented throughout the text, but distracting to the flow, is presented in four appendices at the end of the report. An index of abbreviations and symbols is provided just prior to Chap. 1. The notation [3.2 (a)] is used throughout the text to refer to Chap. 3, Sec. 3.2(a). As this document is to serve as a reference for students and professors attempting to build on this work, results contained in my laboratory notebooks are cited within the text from time to time in the form (LB II, p. 32y), meaning lab book II, page 32y.
CHAPTER 2
SECONDARY ELECTRON EMISSION

"You know, it would be sufficient to really understand the electron..."
—Albert Einstein

The bulk of the published work on SEE was accomplished in roughly the first half of this century; excellent reviews covering experiment and theory in this period have been given by Dekker [1958], Kollath [1956], Bruining [1954], and McKay [1948]. Each contains extensive bibliographies cataloging SEE literature through their respective dates of publication. Additionally, Bruining includes extensive discussions regarding experimental methods, apparatus, and practical application of SEE, and McKay is somewhat unique in his presentation, organizing results by material (metals, insulators, and composites). One final review of the time, that by Bunney [1964], concentrates primarily on the quantum mechanical formulation of the SEE problem. More recent reviews, though by no means comprehensive, are given by Schou [1988], primarily theory, and Reimer [1993], primarily experiment. This chapter introduces the topic of secondary electron emission (SEE) and provides a context for the work presented in Chaps. 2-6—it is not intended as a comprehensive review of the subject.

2.1 Historical Perspective

SEE—the ejection of electrons from a surface as a result of energetic electron bombardment—was first observed by Austin and Starke [1902]. Further observations were sparse through about 1930, when the potential for practical application was realized. An intense period of investigation followed, from roughly 1930 to 1960. A large number of measurements were made in this period, and theoretical formulations developed along two parallel tracks: the “semiempirical” theories, attempting only to describe experiment, and the quantum mechanical (QM) formulations, attempting to describe the fundamental physical processes underlying SEE. Ironically, interest in SEE observation began to wane just as the tools necessary for proper investigation of the phenomenon were becoming widely available—namely, ultra-high vacuum
(UHV) technology and vastly improved computing systems. However, the database and semiempirical formulation of the problem at the time was sufficient for most practical applications. Interest renewed somewhat in the 1970's. Improved computers and numerical methods made previously infeasible calculations possible, and as of the early 1980's there have existed exquisitely detailed formulations available for experimental evaluation [Rösler and Brauer, 1981a, 1981b; Schou, 1988; Ganachaud and Cailler, 1979a, 1979b; Pillon et al., 1976]. Though continuing, experiment has not kept pace with theory, and the field now finds itself in need of new, high-quality measurements.

2.2 General Description of SEE

Energetic electrons (termed primary electrons, or PE's) incident on a surface will induce, among other effects, electron emission from the surface (Fig. 2-1). Some of these emitted electrons have their origin in the incident (primary) population, while others originate from within the sample itself. Those emitted electrons originating as PE's are termed backscattered electrons (BSE's); they have penetrated the

![Diagram of electron emission from a surface resulting from energetic electron bombardment. Primary electrons (red) can be absorbed (pink), backscattered (red), or stimulate the ejection of secondary electrons (blue).](image)

**Figure 2-1.** Schematic depicting electron emission from a surface resulting from energetic electron bombardment. Primary electrons (red) can be absorbed (pink), backscattered (red), or stimulate the ejection of secondary electrons (blue).
Primary electrons (PE's) can produce backscattered electrons (BSE's), secondary electrons (SE), photons (γ), or Auger electrons (AE's), while BSE’s can themselves produce secondaries (SE2) and AE’s. Shaded regions represent volumes of electron trajectories, with trajectory densities increasing with darkness (adapted from Reimer [1993]).

surface, undergone one or more elastic or inelastic interactions within the solid, and been re-emitted from the surface. Those emitted electrons originating from within the solid itself are termed true secondary electrons (SE’s) (as distinguished from the defined SE’s, discussed in [2.4]); they have acquired excess energy through various interactions with the PE’s, resulting—for some—in their eventual ejection from the surface. On a more detailed level (though not quite as detailed as it gets), it is found that the BSE’s can also produce SE’s, and that Auger electrons (a special case of SEE) and photons are also produced as a result of electron bombardment of surfaces (Fig.2-2).

**Basic Features of SEE and Electron Backscattering**

Important SEE and backscattering behaviors, as they pertain to this investigation, are listed below. The terms SE yield (δ) and BSE yield (η) refer to, respectively, the numbers of SE’s and BSE’s emitted from a surface per incident PE, and are commonly used to describe the SEE and backscattering characteristics of a substance (these quantities are discussed in detail in [3.3 (b)]).
1. the vast majority of SE's have energies below 50 eV, with a peak in the energy distribution generally in the 2-5 eV range;

2. BSE's possess, on average, much greater energies than do SE's, and the BSE energy distribution includes a distinctive peak at the incident beam energy, often referred to as the "elastic peak";

3. SEE is primarily a surface phenomenon, occurring in the first ~50;

4. BSE's originate from much deeper in a sample, on average, than do SE's;

5. SEE is sensitive to such incident beam characteristics as energy and incident angle, and to such sample characteristics as material, crystal structure, and surface condition;

6. the BSE yield is far less sensitive to changes in surface condition than the SE yield, though other material parameters remain important;

7. the angular distribution of the emitted SE's is well-approximated by a cosine law, and only weakly sensitive to incident beam angle;

8. the angular distribution of the BSE's is generally much more sensitive to incident beam angle than that for SE's, and falls off much more rapidly than a cosine function with increased emission angle.

These characteristics have been noted for a broad range of materials, and for incident energies ranging from a few tens of eV to several MeV [Schou, 1988]. They can all be reasonably well understood via rudimentary classical arguments, summarized in [2.3(a)-(c)]. Throughout the remainder of this chapter it is important that the distinction between SE production and SE emission be made clear. Secondary electron production refers specifically to the excitation of electrons within the sample (usually conduction electrons in the case of metals, or valence electrons in the case of semiconductors [Schou, 1988; Woolridge, 1939]); secondary electron emission (SEE) refers to those secondaries which actually escape the surface—only a small fraction of those produced [Baroody, 1950]. SEE and backscattering are not exclusive phenomena, as it has been found that BSE's are in fact more efficient producers of secondaries than are PE's [Thomas and Pattinson, 1970; Kanter, 1961; Bennett and Roth, 1972]. Consequently, though the focus of this work is on SEE, backscattering is also discussed extensively.
2.3 Theory

Both the semiempirical and quantum mechanical approaches to SEE treat the phenomenon as a three-stage process: (i) production of SE’s within the solid, (ii) transport of SE’s to the surface, and (iii) emission of SE’s from the surface. The weakness of the semiempirical approach is that it does not directly address the mechanism(s) by which SE’s are produced and transported—and in fact any such attempt must be quantum mechanical. Until recently, the major difficulty with quantum theories has been their complexity: they can be formulated in a detailed manner, but prior to the advent of modern computing technology, could not be solved. This problem has now been overcome. Basic features of both approaches are now briefly discussed, with emphasis on the energy and angular distributions of SE’s and BSE’s.

2.3 (a) Semiempirical Formulations

Salow and Bruining first proposed what is now called the standard semiempirical theory of SEE [Dionne, 1973], with later improvements by Baroody [1950], Sternglass [1950, 1957], and a host of others [Barut, 1954; Lye and Dekker, 1957; Dionne, 1975; Suszczynsky and Borovsky, 1992]. The basic approach assumes that the SE yield $\delta$ as a function of incident energy $E_0$ may be written as

$$
\delta = \int n(x, E_0) f(x) dx
$$

for $n(x, E_0) dx$ the average number of SE’s produced per incident PE in a layer of thickness $dx$ at a depth $x$ below the surface, and $f(x)$ the probability for an SE to migrate to the surface from a depth $x$ and escape [Dekker, 1958]. Further, $n(x, E_0)$ is assumed proportional to $-dE/ dx$, the average PE energy loss per unit path length (sometimes called the “stopping power”), and $f(x)$ is taken as $f(x) = Be^{-Ax}$ for some absorption parameter, $A$ (the inverse mean-free-path of a secondary) and constant, $B$.

The distinguishing feature among most variations on this development is the treatment of the energy loss term, $-dE/ dx$. The simplest approximation assumes the rate of energy loss is inversely proportional to $E$ [Baroody, 1950]:

$$\frac{-dE}{dx} = \frac{a}{E} \quad (2.1)$$
which immediately leads to \( E^2 = E_0 - ax \). A more general version of this formulation assumes an inverse power law of the form

\[
\frac{-dE}{dx} = \frac{a}{E^{n-1}}
\]

(2.2)
giving \( E^n = E_0 - anx \) [Schou, 1988]. The parameter \( n \) can be fit to experimental data, allowing for an overall better mathematical model. Implicit in expressions (2.1) and (2.2) is that all primaries penetrate to the same depth, and that the energy loss per unit path length increases with decreasing energy. An improved formulation, based on experimental data indicating that the transmitted fraction of electrons decreases approximately linearly with the fractional range covered by the primary beam—i.e., that the scattering of primaries is important and that their endpoints are distributed throughout a surface layer of the sample—was introduced by Young [1956a, 1956b]. He proposed writing

\[
\frac{-dE}{dx} = \frac{E_0}{R}
\]

where \( R \) is the maximum primary penetration depth, and is found by setting the left-hand side of Eq. (2.2) to zero. Then \( R = E_0/an \) and we can write

\[
\frac{-dE}{dx} = an
\]

(2.3)

Finally, Sternglass [1950] attempted to incorporate the importance of atomic shell structure by using the Bethe expression for the energy loss term,

\[
\frac{-dE}{dx} = \frac{2\pi Ne^4}{E} \sum_{n,l} Z_{n,l} \log \left( \frac{2E}{I_{n,l}} \right)
\]

(2.4)

for \( N \) the number of atoms per unit volume, \( Z_{n,l} \) the number of electrons in the shell \( n,l \), and \( I_{n,l} \) the binding energy of electrons in the state \( n,l \).

Proceeding from this beginning, semiempirical SEE formulations successfully account for the general SEE features described in [2.2] above. Various expressions for \( \delta/\delta_{\text{max}} \) as a function of \( E/E_{\text{max}} \) are typically formulated from the relations (2.1)-(2.4) [Reimer, 1993], where \( \delta_{\text{max}} \) is the maximum SE yield for a given material, occurring for an incident energy of \( E_{\text{max}} \), and compared against experiment. (Distinctions between their predictions will prove relevant to the analysis of [4.6].) From a physicist's perspective, the
semiempirical formulation is ultimately unsatisfying; it replaces production interactions with a stopping power, and absorption interactions with a mean free path. To address fundamental physical processes, one must move to quantum mechanical formulations, such as those summarized in [2.3 (b)] below.

**SE Energy Distribution**

A relatively successful expression for the SE energy distribution of conductors has been developed by Chung and Everhart [1974]. Production and transport mechanisms are not explicit in the formulation. They start with a population of SE's that have already been excited within a surface and assume that (i) any electron-electron scattering within the surface results in absorption of the excited SE, and (ii) the solid is modeled by a degenerate free-electron gas (precluding ionic collisions). Then for an excited SE at depth \(x\), moving in a direction \(\alpha\) with respect to the normal, they write the probability of reaching the surface without being absorbed as

\[
P(E,\alpha,x) = \exp\left[-\frac{x}{\lambda(E) \cos \alpha}\right]
\]  

(2.5)

where \(\lambda(E)\) is the mean-free-path for electron-electron scattering, a function of \(E\). The \(1/\cos \alpha\) term arises simply because the distance an electron must travel to reach the surface increases with \(\alpha\) by this factor. Emission is modeled by demanding that once reaching the surface, an SE must have an energy \(E \geq E_F + \phi\) (measured from the bottom of the conduction band), where \(E_F\) is the Fermi energy and \(\phi\) is the work function of the metal. An expression for the energy distribution of the emitted SE's is derived as

\[
\frac{dN}{dE} = \frac{S(E)\lambda(E)}{4} \left[1 - \left(\frac{E_F + \phi}{E}\right)\right]
\]  

(2.6)

where \(S(E)\) is the number of SE's excited by the primaries per unit energy in the energy interval \(E+dE\). Expressions for \(S(E)\) and \(\lambda(E)\) are calculated by Baroody [1950] and Quinn [1962], respectively, and on substitution into Eq. (2.6) give the result

\[
\frac{dN}{dE} = \frac{k (E-E_F-\phi)}{E_F (E-E_F)^4}
\]  

(2.7)

where \(k\) is a material-dependent proportionality constant. Equation (2.7) is commonly rewritten in the form

\[
\frac{dN}{dE} = \frac{k E}{E_F (E+\phi)^4}
\]  

(2.8)
through a convenient redefinition of the zero-level energy [Reimer, 1993], and gives the simple result for the energy of maximum SE emission as \( E_{\text{max}} = \phi / 3 \).

Though Chung and Everhart do not explicitly introduce production or transport mechanisms, such features are implicit in the derivation of (2.8). Specifically, Baroody’s calculation of the production function \( S(E) \) assumes excitation of SE’s is due to electron-electron interactions based on Sommerfeld’s free-electron description of conductors. Further, electron-lattice interactions are included in a dual role: first, elastic electron-ion collisions are assumed to direct the SE’s from their excitation location to the surface, simultaneously producing an isotropic SE distribution from the highly anisotropic excitation process; second, absorption due to electron-lattice interactions is modeled with a finite mean free path for elastic scattering. Thus, the success of Eq. (2.8) does have its roots in some basic assumptions regarding real physical processes, however simplified.

**SE Angular Distribution**

Of particular interest in this investigation are the SE and BSE distributions with respect to emission angle, \( \alpha \), measured relative to the surface normal. [Note: the term “emission angle,” where \( \alpha = 0 \) is defined as the outgoing surface normal, is favored in this investigation over the term “scattering angle,” where \( \alpha = 0 \) is defined as the incoming surface normal.] The observed angular distribution of emitted SE’s is, in general, approximately a cosine law of the general form

\[
N_{\text{se}} = k \cos \alpha
\]

(2.9)

where \( k \) is a constant [Rössler and Brauer, 1981a]. More specifically, SE’s are predicted to follow a form of this distribution known as Lambert’s cosine law [Meyer-Arendt, 1984], where \( k \) is the number of SE’s emitted at \( \alpha = 0 \) (perpendicular to the surface):

\[
N_{\text{se}} = N_{\text{se}}(0) \cos \alpha
\]

(2.10)

and can be accounted for by assuming that (i) the SE’s are produced isotropically within the solid, or their internal angular distribution becomes isotropic as they diffuse to the surface, and (ii) the probability that an SE is emitted is inversely proportional to the distance it must travel to the surface [Chung and Everhart, 1974]. In this case, the path length is inversely proportional to the cosine of the emission angle, making the
probability of emission directly proportional to \( \cos \alpha \). In addition to accounting for the observed approximate cosine distribution of emitted SE’s, assumption of an isotropic distribution just prior to emission also correctly accounts for only a weak dependence of the emitted distribution on incident beam angle.

An alternate formulation of the emitted SE angular distribution has been offered by Baroody [1950], who includes surface refraction effects (i.e., conservation of energy and parallel momentum) as the electron leaves the surface. The result is a second-order correction to the Lambert cosine law (2.10):

\[
N_{se} = N_{se}(0) \cos \alpha (1 + 0.28 \sin^2 \alpha + 0.14 \sin^4 \alpha + \ldots)
\]

Equation (2.11) falls off slower with increasing \( \alpha \) than does the Lambert cosine law, as depicted in Fig. 2-3. None of the experimental results in the literature considered here reports confirmation of this modified cosine distribution.

2.3 (b) Quantum Mechanical Formulations

Quantum mechanical (QM) SEE formulations were first offered by Fröhlich [1932] and Woolridge [1939], and improved by Dekker and van der Zeil [1952] and van der Zeil [1953]. A host of investigators have since expanded and refined these formulations, culminating with theories such as that of Rösler and Brauer [1981a] and Ganachaud and Callier [1979a]. Such formulations seek to produce—via fundamental physical interactions—an initial angle and energy SE distribution, propagate this distribution
to the surface—including additional production and loss mechanisms experienced along the way—and then emit this transported angle-energy distribution to finally obtain an emitted angle-energy distribution.

Secondary electron production is attributed to (i) excitation of valence electrons, (ii) excitation of core electrons, and (iii) excitation due to plasmon decay (where the plasmons are initially excited by the PE's), and calculated from the general expression for the transition probability between Bloch states. Here the transition to transport theory is accomplished. Scattering probabilities and mean-free-paths are calculated for interactions between the SE’s and valence electrons, core electrons, and bulk and surface plasmons; the resulting distribution function is propagated to the surface via the Boltzmann transport equation. Applying this theory to aluminum, Rösler and Brauer [1981b] obtain an expression for the angle-energy distribution, $j(E, \Omega)$, of the emitted SE's:

$$j(E, \Omega) = \Theta(E' - W) \Theta(\cos \alpha - \cos \alpha_c) \left( 1 - \frac{W}{E'} \right) \sqrt{\frac{E' \cos^2 \alpha' - W}{E' - W}} \times \frac{l(E')}{l(E')} \sum_{\lambda} \psi_{\lambda}(E') P_{\lambda}(\cos \alpha)$$

for the Heavyside step function $\Theta$, interior and emitted energies ($E'$ and $E$) and angles ($\alpha_c$ and $\alpha'$), the composite mean free path $l(E')$ and excitation function $\psi(E')$, Legendre polynomials $P_{\lambda}(\cos \alpha)$, and $W$ the sum of the Fermi energy and work function, where $E' > W$. This expression represents a fundamental theoretical prediction for precisely the kind of measurement this investigation seeks to obtain.

Rösler and Brauer also develop expressions for the integrated energy distribution (integrated over all emission angles) and total SE yield (see [3.2] for detailed definitions of these quantities). In particular, fine structure is predicted in the energy spectrum due to plasmon decay, and compares well with experiment. However, they are unable to compare $j(E, \Omega)$ to experiment as there presently exist no measurements of the sort.

**Energy Distributions**

The SE energy distribution predicted by formulations such as that of Rösler and Brauer is the sum of distributions resulting from the various production and subsequent scattering processes considered. For
example, Rösler and Brauer calculate separate energy distributions for excitation due to core electrons, screened electron-electron scattering, and plasmon decay and combine them to form a single energy distribution for the internal SE population just after production. This distribution is then modified via inelastic and elastic scattering with the conduction electrons and ions, respectively, as the excited SE’s are propagated to the surface. Emission from the surface adds the additional effects of conservation of energy and conservation of parallel momentum [Rösler and Brauer, 1981a]:

\[ E' = E + E_{FE} + \phi \]

\[ p'_\parallel = p(E')\sin\alpha' = p(E)\sin\alpha \]

Generally, the energy distributions arising from the individual production and scattering processes are material dependent and behave quite differently from one another. Thus, the calculated composite energy distribution of the emitted SE’s exhibits noticeable fine structure and fundamental changes of behavior at energies where transitions between dominant production processes occur. Such predictions are ideal for comparison with experiment. As an example, Rösler and Brauer [1981b] have performed a detailed calculation for aluminum, predicting enhanced SE production at \( \sim 10 \) eV as a result of SE excitation due to plasmon decay. Careful observations by several sets of investigators [Krane, 1978; Everhart et al., 1975; Henrich, 1973; Powell and Woodruff, 1972] have supported this prediction.

**Angular Distributions**

Analogous to the case for energy distributions, the angular distributions predicted by detailed QM SEE formulations are the results of the angular behaviors of the individual production mechanisms and subsequent scattering processes. Again referring to Rösler and Brauer’s calculation for aluminum, they find that all of the production mechanisms considered (excitation of core electrons, screened electron-electron excitations, and excitation from plasmon decay) result in highly anisotropic angular distributions for the excited SE’s. However, elastic impacts with ions as the SE’s migrate to the surface transforms their initial, anisotropic angular distribution to an isotropic distribution, resulting in a predicted cosine distribution at all energies in the range 0-50 eV.
2.3 (c) Backscattering

Theoretical formulations for electron backscattering are abundant in the literature [Bishop, 1967; Jablonski et al., 1989, 1993; Jablonski, 1991]. A concise, representative discussion of the treatment is given by Reimer [1993]. Generally, elastic and inelastic processes are considered. Elastic scattering arises from the attractive Coulomb potential of the nucleus, screened by an electron cloud (termed screened Rutherford scattering). A simple expression for the screened Rutherford scattering cross section is given by Bishop [1967]:

$$\frac{d\sigma}{d\Omega} = k \frac{Z^2}{E^2 (1 + 2\beta - \cos \alpha)^2}$$

(2.13)

for constant $k$, atomic number $Z$, and screening factor $\beta$. (Quantum mechanically, this expression is obtained as the first Born approximation to the Yukawa potential [Sakurai, 1985, p. 388].) Eq. (2.13) is also known as a Mott cross section and can be written in the form

$$\frac{d\sigma_{\text{Mott}}}{d\Omega} = k r(\alpha) \frac{Z^2}{E^2 \sin^4 (\alpha / 2)}$$

(2.14)

where $r(\alpha)$ is the ratio between the Mott cross section and the unscreened Rutherford cross section [Reimer, 1993]. Inelastic scattering is due to intra- and inter-band transitions, surface and volume plasmon losses, single-electron excitations, and inner shell (core) ionizations [Reimer, 1993]. Detailed calculations utilizing this framework have been carried out by a number of authors and found to compare well with experiment [Bishop, 1967; Jablonski et al., 1989, 1993; Jablonski, 1991; Reimer, 1993]

2.3 (d) Discussion

Based on the preceding summaries of both semi-empirical and QM descriptions of SEE and electron backscattering, it will be useful to review the eight basic features of the phenomena listed in [2.2].

**SE Behaviors**

That most SE’s have very low energies is a consequence of the inelastic scattering processes leading to their excitation, which favor energy distributions peaked at low energies. That emitted SE’s are found to originate very near the sample surface is a consequence of their low energies: since they possess
relatively little excess energy, those produced deeper than a few tens of angstroms have a high probability of being reabsorbed before reaching the surface. In this context, dependence of the SE yield on various incident beam and sample parameters is readily understandable. The number of SE’s emerging from a sample (the SE yield) will depend on how many are produced very near the surface, in the SE “escape zone.” The number of SE’s produced near the surface depends on how much energy the PE’s deposit there, and how much energy the PE’s deposit near the surface depends on how deeply they penetrate into the sample (the deeper the penetration, the less energy deposited near the surface). For a given material, the energy and incident angle of the PE’s determines the PE’s penetration depth, and for a given beam energy and incident angle the penetration depth will depend on material properties such as density and crystal structure—hence the sensitivity of the SE yield to these incident beam and sample parameters. Finally, the observed angular behavior of the emitted SE’s, a Lambert cosine law, is a direct consequence of an isotropic distribution within the sample, due almost entirely to the angular behavior of elastic electron-ion collisions by which the excited SE’s migrate from their production depth to the surface.

**BSE Behavior**

For the BSE’s, a greater depth of origin is a result of smaller scattering cross sections (i.e., smaller probabilities) for near-field ionic collisions. Since such interactions are less likely to happen, PE’s must travel further in the solid, on average, before experiencing a collision that will transform them into BSE’s. Thus BSE’s originate (on average) deeper in the solid than do SE’s; that the BSE yield is less sensitive to surface condition is merely a reflection of this fact.

### 2.4 Observations

Experimentally, since it is impossible to determine directly which of the electrons emerging from a sample originated in the sample—the true SE’s—and which originated in the primary beam (the BSE’s), the SE’s are defined to be those electrons emitted with energies $E < 50$ eV. It is important to point out that this 50-eV boundary, though not unreasonable, is arbitrary; there is no physical reason preventing either the
SE’s or BSE’s from possessing any energy between 0 eV and the beam energy. Given the above assumptions, however, the likelihood of an SE possessing more than a few tens of eV, is small, as is the likelihood of a BSE possessing less than 50 eV. Still, if one wishes to investigate the fundamental nature of SE’s, the study of SE’s with energies greater than 50 eV may prove important.

**Categories of SEE Observation**

There are four basic flavors of SEE measurement: total yields, integrated energy spectra, angle-resolved yields, and angle-resolved spectra. Precise definitions of these quantities are presented in Chap. 3: for the present discussion, however, the following brief descriptions will be sufficient: *total yield* refers to the total number of SE’s emerging from a surface, without regard to emission angle or energy; *integrated energy spectrum* refers to the SE energy distribution, without regard to emission angle (i.e., integrated over angle); *angle-resolved yield* refers to the number of SE’s emerging from a surface as a function of emission angle; *angle-resolved spectrum* refers to the SE energy distribution as a function of emission angle. On reviewing the available literature, one notes the following key points regarding SEE measurement:

1. The vast majority of SEE measurements to date are total yields and integrated energy spectra;
2. There exist very few angle-resolved SEE measurements;
3. There is a persisting dearth of high-quality SEE measurements (see below);
4. There is a need to establish standardized reporting criteria for high-quality SEE measurements.

Total yield investigations generally examine δ and η as functions of incident energy, incident angle, and material. In particular, it is common to describe the SEE characteristics of various materials by plotting total SE yield vs incident energy (for normally incident electrons), as illustrated in Fig. 2-4. Similarly, integrated spectra are measured as functions of various incident beam and sample parameters; Fig. 2-5 depicts an energy spectrum typical of that found throughout the literature.
FIGURE 2-4. Typical SE yield vs energy curve (data is for electrons incident on aluminum).

FIGURE 2-5. Representation of a typical SE and BSE energy spectrum (adapted from Reimer [1993]).
The relative absence of AR SE measurements is generally attributed to the difficulty of performing them (discussed further in Chap. 5). AR yield measurements from only four authors were found in the literature [Gérard et al., 1992; Jahrreiss and Oppel, 1971; Jonker, 1951]; most notable was that of Jonker [1951]. Furthermore only one AR SE spectra investigation was found, Appelt’s study of Cu [Appelt, 1968].

The Issue of Measurement Quality

The vast majority of SEE measurements to date were performed on poorly characterized surfaces, prior to the introduction of ultra-high vacuum (UHV) technology [Schou, 1988]. Because of the extreme sensitivity of SEE to the presence of surface contaminants, most of these measurements should be regarded as unreliable. And while high-quality measurements have been performed in the intervening years, such investigations have been random, involving only a handful of materials and incident beam energies.

Referring to the need for systematic, high-quality studies, the following is taken from Schou’s 1988 review:

"Nevertheless, high-quality data from well-characterized surfaces, not only from nearly-free-electron metals, but also from the transition and Noble metals, are necessary to stimulate theoretical progress. In particular, yields $\delta$ and energy distributions $d\delta/dE$, in the energy range 2-20 keV are desirable...." (p. 628, emphasis added)

With the advent of UHV technology, measurement quality has improved; unfortunately, however, the need for adequate surface characterization, or even for reporting experimental parameters that would allow reasonable inference of surface condition, continues to elude many investigators. It is therefore suggested that the following experimental parameters need to be reported with all SEE measurement results, to aid in assessing their reliability:

1. Sample preparation and treatment prior to vacuum insertion
2. Base and operating pressures
3. Surface smoothness
4. Direct characterization of surface contamination when possible (e.g., Auger spectroscopy)
5. Sample treatment under vacuum (e.g., time under vacuum, sputtering, annealing, electron beam exposure, etc.)
6. Electron beam current density and time of sample exposure.

Backscattering Observations

Backscattering measurements in the literature generally take the same form as SEE measurements—primarily total yields and integrated spectra as a function of incident energy, incident angle, and material. As angle-resolved BSE measurements are easier to perform than their SE counterpart, the dearth of angle-resolved measurements seen in the SEE database is not as severe for the BSE’s [Reimer, 1993]. Also, since backscattering is far less sensitive to surface condition, measurement quality is not the issue it is for SE measurements.

2.5 Previous Investigations of Gold

As the prime objective of this investigation is to obtain new SEE measurements for electrons incident on Au—most notably, a full set of angle-resolved SE spectra—it is appropriate that previous SEE investigations for Au be reviewed. Figures 2-6 and 2-7 depict previous SEE measurements performed on gold—categorized by author, incident energy, and type of measurement. They show that while an appreciable number of total yield and integrated spectra have been measured, and over a considerable range of incident energies, only two measurements can be considered high-quality. Furthermore, no angle-resolved spectra measurements are found. Thus there appears to be considerable opportunity to add to the data base via angle-resolved investigation of polycrystalline gold.

Having reviewed both theory and measurement, we begin discussion of the present work with a summary of instrumentation, measurement goals, and measurement techniques.
FIGURE 2-6. Previous SEE measurements for Au, grouped by author.
FIGURE 2-7. Previous SEE measurements of Au, grouped by type.
CHAPTER 3

INSTRUMENTATION AND METHODS

"God curse the man who taught me my craft..."
—An ancient watchmaker

A thorough description of the apparatus and method employed in this investigation is given in Davies [1996]—a companion piece to this dissertation, describing instrumentation development and testing preliminary to the present work. While the bulk of the instrumentation described therein remains unchanged, a small—but important—number of improvements to both equipment and measurement technique have since been effected. For this reason, and for purposes of continuity, an abbreviated description of both apparatus and method is presented here.

3.1 Instrument

Primary components comprising the instrument utilized in this investigation are (i) an ultra-high vacuum (UHV) chamber, (ii) an electron gun, (iii) a sample and detector assembly mounted inside the UHV chamber, (iv) a series of measurement circuits, comprised of a number of power supplies, electrometers, and shielded cables, and (v) a data acquisition system. Summary descriptions of each of these components are presented below, along with a list summarizing instrument modifications accomplished subsequent to the detailed descriptions presented by Davies [1996].

3.1 (a) Vacuum Chamber

The UHV chamber is an ~25,000 cm$^3$ volume of standard stainless steel construction. Roughing is accomplished by a mechanically-backed turbomolecular pump, or by two cryosorption pumps cooled with liquid nitrogen; high-vacuum pumping is achieved with a magnetic ion pump, operated continuously and periodically supplemented by a water-cooled titanium sublimation pump (TSP). A 72-hour, 125 °C bakeout of the system routinely results in base pressures in the mid-10$^{-11}$ Torr range. UHV absolute pressure measurement is accomplished independently with both a nude UHV ion gauge and a quadrupole
mass spectrometer configured for total pressure measurement; these gauges typically agree to within less
(usually much less) than a factor of two.

3.1 (b) Electron Gun

The electron gun is a standard Pierce-type model with a tungsten filament and produces a beam in
the 1-3 keV energy range. As a result of a 5 V peak-to-peak ripple in the power supply's HV output, the
electron beam is actually bi-energetic; i.e., the beam is comprised of an electron population centered about
two energies—at approximately $E_B \pm 2.5$ eV. The measured energy spread $\Delta E$ in the two populations is
$\sim 2$ eV (FWHM). The beamspot at target distance is typically 1-2 mm in diameter and easily relocatable on
the sample via horizontal and vertical deflection controls. The beam current is controllable in the range
$\sim 0.1$ to $100$ μA, with a worst-case stability of $\sim 5\%$ over time scales on the order of 1 sec (stability
improves with increasing beam current, but worsens with increasing beam energy). For the beam currents
and energies typical of this investigation—200 nA and 1.5 keV, respectively—stability was approximately
2% over time scales of seconds. The gun is mounted to the chamber on a flange oriented 45° relative to the
horizontal and aligned on the chamber center.

3.1 (c) Chamber Apparatus

The assembly housing the samples and rotatable detector, termed the chamber apparatus (CA), is
depicted schematically in Fig. 3-1. The primary sample sits at the center of the chamber apparatus on a
sample holder, and is flanked on either side by two tertiary samples. It is the primary sample that is
bombarded by the incident electron beam; the role of the two tertiary samples—whose combined surface
area is equal to that of the primary—is to measure the current to the primary sample due to SE’s and BSE’s
that have scattered from the chamber interior and returned (termed the return current). Mounted inside the
primary sample holder are a small resistive heater and thermocouple used for active temperature control of
the primary sample ($27 ^\circ C < T < 400 ^\circ C$). Machined into the left center of the primary sample and holder
is a large Faraday cup (2 mm dia. x 20 mm deep) into which the incident electron beam can be deflected for
the purpose of beam current measurement. The additional presence of three smaller Faraday cups,
Primary Sample
(see Fig. 3-2 for more detail)

Attach to Feedthrough

Top Plate

Tertiary Samples

Sample Block

Rotating Plate

Rotatable Detector

Bottom Plate

Rotary Cable

FIGURE 3-1. Schematic depicting the chamber apparatus (minus the magnetic shield).
machined into the primary sample above, below, and to the right of center (Fig. 3-2), allows for precise positioning of the electron beam on the sample center: finely graduated horizontal and vertical deflection controls are used to move the beam about the sample and obtain vertical and horizontal coordinates for each of the four Faraday cups, allowing for calculation of the sample center coordinates (the sample center is located half-way between the left and right Faraday cups in the horizontal direction, and half-way between the top and bottom Faraday cups in the vertical direction). Such a capability is necessary for measurements utilizing the rotatable detector, which is permanently aligned on the sample center. The primary and tertiary samples and their respective holders are mounted in the sample block, depicted in Fig. 3-2, which orients their surfaces 45° relative to the horizontal—perpendicular to, and directly facing the incident electron beam.

The rotatable detector (RD), depicted schematically in Fig. 3-3, allows for angle-resolved measurements to be accomplished for a continuous range of emission angles relative to the sample normal; it consists of an aluminum Faraday cup mounted, via Teflon spacers, in a stainless steel housing. This assembly is mounted on a plate which can be rotated, by means of a flexible cable connection to a rotary motion feedthrough, 220° about the sample in the horizontal plane (the presence of the support rods,
FIGURE 3-3. Schematic depicting the rotatable detector.
depicted in Fig. 3-1, precludes an unrestricted, 360° rotation about the sample). Throughout rotation the RD remains aligned on the sample center at a distance of 74 mm to the Faraday cup (62 mm to the first aperture); at this distance, the solid angle subtended by the 1.5 mm aperture is \(~3.6 \times 10^{-4}\) sr, which translates to an angular resolution of \(~2°\). Because the sample is mounted at 45° relative to the horizontal and the RD is mounted at 59°, rotating about the sample in the horizontal plane, not all emission angles are accessible to the detector. Specifically, as the RD moves azimuthally about the sample, from a detector position of \(\varphi_{RD} = 15°\) to \(\varphi_{RD} = 180°\) (where \(\varphi_{RD} = 0°\) corresponds to the incident beam direction, as depicted in Fig. 3-2), the emission (polar) angle \(\alpha\) varies from \(\alpha = 16.6°\) to \(\alpha = 76°\). Two important consequences of this rather convoluted geometry should be noted: (i) the relationship between detector position and emission angle is highly nonlinear [Davies, 1996], as illustrated in Fig. 3-4, and (ii) the azimuthal plane of the sample, \(\varphi_s\), in which the detector is located changes along with the emission angle as it rotates about the sample. The first point simply means that equal changes in \(\varphi_{RD}\) do not result in equal changes in \(\alpha\) as the RD progresses about the sample. The second point is particularly important in that as a result, implicit in any meaningful comparison of measurements taken at varying \(\alpha\)'s is the assumption that the SE and BSE scattering cross sections are symmetric with respect to the azimuthal angle \(\varphi_s\). It is reasonable to assume

\[\text{FIGURE 3-4. Emission angle intercepted by the rotatable detector as a function of detector position.}\]
that indeed such is the case for polycrystalline targets under normally incident bombardment; for single-
crystal surfaces, however, or for nonnormal incidences, such an assumption is almost certainly flawed.

Enclosing the chamber apparatus is a cylindrical magnetic shield (not depicted in Fig. 3-1),
constructed of 0.020" CO-NETIC μ-metal, reducing the earth's ~500 mG ambient magnetic field, \( \mathbf{B} \), to
~4 mG in the region between the sample and the detector (measured with the chamber apparatus mounted
inside the vacuum chamber). Such a reduction in \( \mathbf{B} \) is necessary to the acquisition of low-energy
(0-20 eV), angle-resolved spectra. [Note: While the average \( \mathbf{B} \) in the 62-mm region between the sample
and detector is very low, the presence of the sample heater (with nickel housing), located just under the
primary sample, produces a magnetic field magnitude of ~30 mG at the sample surface; this field drops
below 10 mG after moving only a few millimeters away from the surface, and to only a few mG midway
between the sample and the detector.]

The entire chamber apparatus is mounted in the vacuum chamber via a 3-axis linear, 1-axis (z)
rotary motion feedthrough attached to the top plate, allowing for precise, remote positioning of the sample
within the chamber.

3.1 (d) Measurement Circuits

Three circuits are required for the current measurements necessary to this investigation: one each
for measuring the primary and tertiary sample currents, and one for RD current measurement. These
circuits are depicted schematically in Figs. 3-5 through 3-8. Note that these diagrams are not the same as
those presented in Davies [1996]. The primary and tertiary sample circuits are identical and can be treated
together. The RD circuit is somewhat different in that it must measure much smaller currents under
conditions of much larger biasing potentials, and is therefore discussed separately.

The object of the primary and tertiary sample circuits is to measure the net current reaching each
of the samples. The measurement technique employed in this investigation (see Sec. 3.2 below) requires
that these currents be measured under varying conditions of sample bias, where it is always the case that the
FIGURE 3-5. Schematic depicting sample-current detection circuit. Leakage current paths from the biased samples, through the ceramic spacers and coaxial insulators to the sample holders and coaxial shields, respectively, are evident.
FIGURE 3-6. Schematic depicting connection scheme for primary and tertiary sample-current detection circuits.
FIGURE 3-7. Schematic depicting RD-current detection circuit. Leakage current paths from the FC shield to the detector housing and coax shield, and from the inner triax shield to the outer triax shield, are evident. Also evident is the absence of a leakage current path from the Faraday cup to ground.
FIGURE 3-8. Schematic depicting connection scheme for RD-current detection circuit.
primary and tertiary samples be biased equally and simultaneously. Figure 3-5 depicts the measurement circuits schematically, while Fig. 3-6 illustrates the connection scheme by which these circuits are constructed. Two Keithley 616 electrometers— instruments well-suited for low-current detection—are used for sample current measurements (one for the primary sample, and one for the combined current of the two tertiary samples), and wires carrying the signal from the samples to the 616’s are fully shielded, low-noise cables. Simultaneous biasing of the primary and tertiary samples is accomplished by floating the primary and tertiary sample signal wires (and therefore the samples themselves) via connection of a single power supply to the signal return path of each of the 616’s (see Fig. 3-6). Under conditions typical of this investigation, the net current collected by the primary sample is \(- \pm 200 \) nA, while that collected jointly by the tertiary samples is smaller, varying from approximately \(-3 \) nA to \(-50 \) nA negative, depending on sample bias (discussed below). Testing of the circuits has revealed worst-case noise on the order of \(-3 \) pA (due to leakage current from the biased samples, through the ceramic spacers and coaxial insulators to the sample holders and coaxial shields, respectively—see Fig. 3-5); this translates to a worst-case signal-to-noise ratio of approximately \(1.7 \times 10^3 : 1\).

The object of the RD circuit is to measure the net current being collected by the detector under conditions of electrical bias varying from \(0 \) V to \(-3,000 \) V (again, see Sec. 3.2 below for details on measurement technique). Currents collected by the RD in this investigation were typically in the range \(-30 \) pA to \(-0.01 \) pA—more than three orders of magnitude smaller than those collected by the primary and tertiary samples. Nevertheless, the scheme employed for accurate RD current measurement is similar to that for used for the sample currents: the signal is carried via shielded cabling to a (third) Keithley 616 electrometer, and a high-voltage (HV) power supply connected in series with the 616’s signal return path is used to bias the detector. Implementation of this scheme requires a bit more effort than was necessary in the case of the sample current circuits; specifically, (i) since the maximum allowable potential difference between the 616’s signal low and case ground is \(\pm 1000 \) V, the entire electrometer must be floated if one wishes to bias the detector above this limit, and (ii) since the currents to be measured are so small, a more involved shielding scheme must be devised in order to eliminate the significant leakage currents (\(-1 \) nA)
which result from large detector biases. Figures 3-7 and 3-8 illustrate the resulting circuit design and implementation scheme. The electrometer is enclosed in an insulating box and powered by a 12 V battery, while an HV power supply is connected to both the electrometer ground and signal low for detector biasing. Leakage currents are eliminated through triaxial shielding of the signal wire, where the inner shield is biased along with the signal wire and the outer shield is grounded (to earth ground); any leakage current therefore occurs between the inner and outer shields rather than between the signal and inner shield. A Keithley 237 programmable power supply is used to effect detector bias magnitudes less than 1100 V negative (the upper limit of the 237); bias magnitudes greater than 1100 V negative utilize a Bertan 205A-05R supply, controlled by the Keithley 237. One drawback of this arrangement is a minimum step size in detector bias of 1 V for biases over 1100 V. Testing of the circuit to –3000 V has revealed no detectable leakage current nor system noise to $10^{-14}$ A, the detection limit of the Keithley 616.

3.1 (e) Data Acquisition

Automated data acquisition is accomplished via analog outputs from the three Keithley 616 electrometers: isolation circuits (necessary because of the various voltages to which the 616 signals are floated) transfer the 0-5 V analog output signals to a Data Translations DT2811-PGH I/O data card (12-bit resolution), mounted in a 486 PC, where they are read by LabTech data acquisition software (version 9.03 for Windows 3.1). Only two currents need be monitored for any given measurement. All signals are input differentially (as opposed to single-ended) through an RC low-pass (~4 Hz) filter (to minimize noise). Acquisition rates of the DT2811 as read by LabTech are limited to less than 1 kHz; in practice, 200 Hz is the maximum two-channel rate attainable, and most measurements in this investigation were accomplished at 100 Hz. The need for high-rate data acquisition arises from instabilities in the incident electron beam, since averaging a large number of current measurements dramatically reduces the random error introduced by the incident beam fluctuations. The heart of the RD isolation circuit is a Burr Brown ISO-121 isolation amplifier, capable of 3500 V rms signal isolation. The device is comprised of input and output sections galvanically isolated by matched 1 pF capacitors built into a ceramic barrier. The input is duty cycle
modulated and transmitted digitally across the barrier; the output section receives the modulated signal, converts it back to an analog voltage and removes the ripple component inherent in the demodulation (Burr-Brown tech. note PDS-820D, 1992). As configured for this investigation, one can choose output signal gains of 1, 2.5, and 10 (LB III, pp. 64-66). The sample current isolation circuits utilize a Burr Brown ISO-124 isolation chip similar to the ISO-121, but without the amplification capabilities and with a smaller maximum voltage isolation (1500 V rms) (Burr Brown tech. note PDS-1405A, 1997).

3.1 (f) Summary of Recent Modifications

The above descriptions of the instrument contain some modifications to those presented in Davies [1996]; these differences, along with a few that were not mentioned above, are summarized below:

1. The electron gun controller has been modified (through addition of a number of resistors) to enable beam currents as low as \(-100\) nA, thus eliminating previous difficulties with beam-induced sample surface modifications [Davies, 1996].

2. Separate high-voltage biasing of the RD shield has been added, along with full triaxial shielding and inner shield biasing of the signal wire, completely eliminating (to \(-10^{-14}\) A) all detector leakage current under biased conditions.

3. The original coaxial cable carrying the primary and tertiary sample signals has been replaced with low-noise cable [Belden RG-58/U], minimizing triboelectric effects.

4. Automated data acquisition capability for both RD and sample currents has been added, dramatically reducing the random error introduced by incident beam fluctuations.

5. The magnetic shield has been improved, reducing the ambient \(\vec{B}\) within the chamber apparatus to \(-4\) mG.

6. A thermocouple and heater have been added to the primary sample holder, enabling controlled sample heating in the range \(27 ^\circ C < T < \sim 400 ^\circ C\).

7. The mechanical design of the RD rotation scheme has been significantly improved, allowing for smoother RD rotation and more accurate RD placement about the sample.
3.2 Measured Quantities

Four distinct types of SE measurements are of interest in this investigation: (i) total yields, \( \delta \), (ii) angle-resolved yields, \( \delta(\alpha') \), (iii) energy-resolved yields, \( \delta(E'_e) \) and (iv) angle-energy-resolved yields, \( \delta(\alpha',E'_e) \)—together with their corresponding BSE measurements. Before proceeding with a description of precisely how the instrument described above is used to determine these quantities, a review of their definitions and explanation of notation are presented.

3.2 (a) Notation

Throughout the remainder of this work, variables followed by parameters in parentheses represent functions, while variables followed by primed parameters in parentheses represent functions evaluated at a specific value; thus, the presence of a prime denotes a specific (though arbitrary) parametric value. For example, \( \delta(\alpha) \) is a function describing the dependence of the angle-resolved (AR) SE yield on \( \alpha \) (i.e., the SE angular distribution function), and \( \delta(\alpha') \) is the value of that function evaluated at \( \alpha = \alpha' \) (i.e., the AR yield at \( \alpha = \alpha' \)): \( \delta(\alpha') \equiv \delta(\alpha) \bigg|_{\alpha = \alpha'} \). Also note that the presence of a primed parameter in a variable indicates that this variable is a little piece of a “parent” variable—i.e., a differential yield; for example, the angle-resolved SE yield \( \delta(\alpha') \) is a little piece of the total SE yield, \( \delta \), and the angle-energy-resolved yield \( \delta(\alpha',E'_e) \) is a little piece of the AR yield, \( \delta(\alpha') \), a little piece of the energy-resolved yield, \( \delta(E'_e) \), and a very little piece of \( \delta \). On occasion, a more suggestive notation with the presence of a “d” or “d” in the variable—e.g., \( d\delta(\alpha') \), \( d^2\delta(\alpha',E'_e) \), etc.—is used to emphasize the differential nature of a variable. As used in this work, the two notations are interchangeable:

\[
d\delta(\alpha') \Leftrightarrow \delta(\alpha')
\]

\[
d^2\delta(\alpha',E'_e) \Leftrightarrow \delta(\alpha',E'_e)
\]

etc.
3.2 (b) Definitions

[Note: Each of the definitions presented in this section has been developed in detail in Davies [1996], albeit with a slightly different notation; it is a summary of that work that is presented here.] Figure 3-9 depicts the physical situation under investigation: an incident beam of energetic electrons is normally incident on a conducting sample, producing SE's (with energies defined to be 0 to 50 eV) and BSE's (with energies defined to be 50 eV to $E_B^0$) which leave the sample surface in an electron spray; the distribution of this spray is assumed symmetric with respect to the azimuthal angle $\varphi_e$, and in some manner dependent on the emission angle, $\alpha'$ (presumably through some form of cosine law). With this picture in mind, the following definitions are presented:

1. Total SE Yield, $\delta$: the total number of SE's, $N_{se}$, produced per incident PE, at all energies (0 to 50 eV) and at all emission angles (0 to $\pi/2$ radians). Referring to Fig. 3-9, this would be all SE's passing through the entire hemispherical surface, divided by the total number $N_{pe}$ of incident PE's,

$$\delta = \frac{N_{se}}{N_{pe}}$$  \hspace{1cm} (3.1)

2. Angle-Resolved SE Yield, $\delta(\alpha')$: the number of SE's produced per incident PE, per unit solid angle (measured in steradians), about a given emission angle $\alpha'$. Referring to Fig. 3-9, define $dN_{se}(\alpha')$ to be the number of SE's passing through the area element $dA$ (centered on $\alpha'$ and subtending the solid angle $d\Omega$); $\delta(\alpha')$ can then be written

$$\delta_{AR} \equiv \delta(\alpha') = \frac{1}{N_{pe}} \frac{dN_{se}(\alpha')}{d\Omega}$$  \hspace{1cm} (3.2)

Implicit in Eq. (3.2) is the assumption that $\delta(\alpha')$ is independent of $\varphi_e$. Also, it is important to note that in this context, $dN_{se}(\alpha')/d\Omega$ does not represent a derivative in the traditional sense; rather, the form of Eq. (3.2) arises from the definition of a scattering cross section. Defining $\rho_{se}(\alpha')$ to be the number of SE's emitted per unit solid angle at $\alpha = \alpha'$, we have $dN_{se}(\alpha') = \rho_{se}(\alpha') d\Omega$ (where, again, we are assuming the SE emission to be independent of $\varphi_e$). Clearly $\rho_{se}(\alpha')$ will be proportional to the total number of incident electrons:

$$\rho_{se}(\alpha') = N_{pe} \delta(\alpha'), \text{ where } \delta(\alpha') \text{ represents the constant of proportionality for the case } \alpha = \alpha'.$$

Substituting, we can write

$$dN_{se}(\alpha') = N_{pe} \delta(\alpha') d\Omega$$  \hspace{1cm} (3.2a)
FIGURE 3-9. Schematic depicting experiment geometry.
which is just a rearrangement of Eq. (3.2), but with the desirable feature of being completely unambiguous mathematically.

3. **Energy-Resolved SE Yield, \( \delta(E'_e) \):** the number of SE’s produced per incident PE, per unit energy (measured in eV), about a given emission energy \( E'_e \). Referring to Fig. 3-9, define \( dN_{se}(E'_e) \) to be the number of SE’s passing through the entire hemispherical surface with energies in some small energy range \( E'_e \) to \( E'_e + dE_e \). We can then write

\[
\delta_{ER} \equiv \delta(E'_e) = \frac{1}{N_{pe}} \frac{dN_{se}(E'_e)}{dE_e}
\]  

(3.3)

or, as in the previous definition, the less mathematically ambiguous form

\[
dN_{se}(E'_e) = N_{pe} \delta(E'_e) dE_e
\]  

(3.3a)

4. **Angle-Energy-Resolved SE Yield, \( \delta(\alpha', E'_e) \):** the number of SE’s produced per incident PE, per unit solid angle (measured in steradians), per unit energy (measured in eV), about a given emission angle \( \alpha' \) and emission energy \( E'_e \). Referring to Fig. 3-9, define \( d^2N_{se}(\alpha', E'_e) \) to be the number of SE’s passing through the area element \( dA \), centered on \( \alpha' \), with energies in the range \( E'_e \) to \( E'_e + dE_e \); then we can write

\[
\delta_{AER} \equiv \delta(\alpha', E'_e) = \frac{1}{N_{pe}} \frac{d^2N_{se}(\alpha', E'_e)}{d\Omega dE_e}
\]  

(3.4)

or

\[
d^2N_{se}(\alpha', E'_e) = N_{pe} \delta(\alpha', E'_e) d\Omega dE_e
\]  

(3.4a)

Corresponding definitions for the backscattered yields \( \eta \), \( \eta(\alpha') \), \( \eta(E'_e) \), and \( \eta(\alpha', E'_e) \) are completely analogous to those presented above.

Finally, it is sometimes useful to speak of the **total electron yield, \( \sigma \)**, emitted from a surface, which is simply the sum of the SE and BSE yields. Thus, we have

\[
\sigma = \delta + \eta
\]

\[
\sigma(\alpha') = \delta(\alpha') + \eta(\alpha')
\]

\[
\sigma(E'_e) = \delta(E'_e) + \eta(E'_e)
\]

\[
\sigma(\alpha', E'_e) = \delta(\alpha', E'_e) + \eta(\alpha', E'_e)
\]
We now turn our attention to measurement of these quantities with the instrument described in [3.1].

### 3.3 Measurement Techniques

Determinations of all of the quantities described above are obtained via the measurement of net currents reaching various surfaces—under varying conditions of electrical bias—within the chamber apparatus. Net currents measured are those reaching the primary and tertiary samples at biases of 0 V and +50 V, and that reaching the Faraday cup inside the RD, with the Faraday cup biased anywhere between 0 V and −3000 V. Since the angle-energy-resolved yields are the most fundamental quantities in this investigation—from which all other quantities can, in principle, be calculated—the measurement technique for their determination is presented first. Specific procedures for acquiring data with these techniques are presented in Appendix D.

#### 3.3 (a) \( \delta_{\text{AER}} \) Determination

For a given emission angle \( \alpha' \), determination of \( \delta(\alpha', E'_e) \) is accomplished through RD current measurements performed at successively larger negative detector biases, \( \Phi_{RD} \), as follows: biasing the detector negatively serves to reject those electrons with energies \( E_e < e \Phi_{RD} \) (for \( e \) the electron charge, in Coulombs, and \( \Phi_{RD} \) the detector bias, in volts); one can then determine the current \( d^2 I_{12} \) due to secondaries with energies between \( E_1 \) and \( E_2 \) (centered on \( \alpha' \)) via the relation \( d^2 I_{12} = dI_{\phi 1} - dI_{\phi 2} \). If we define \( E_{e12} = (E_1 + E_2) / 2 \), we can then calculate \( \delta(\alpha', E'_e) \) via

\[
\delta(\alpha', E'_e) = \frac{1}{I_B} \frac{d^2 I_{12}}{d\Omega_{RD} dE_{e12}} \tag{3.5}
\]

for \( I_B \) the beam current, \( d\Omega_{RD} \) the solid angle subtended by the detector, and \( dE_{e12} = e(\Phi_{RD1} - \Phi_{RD2}) \) the energy interval \( E_1 - E_2 \). The necessary beam current measurement \( (I_B) \) is accomplished by monitoring the sample current while directing the electron beam into the large Faraday cup machined into the primary sample. One additional aspect of this technique should be mentioned: in order to facilitate angle-resolved
detection of the lowest-energy SE’s (0 to ~10 eV), it can be helpful to place some moderate (0 to 5 V) negative bias on the sample. Negative sample biasing has the effect of energizing the SE’s by an amount $e\Phi$, relative to the detector, where $\Phi$ is the sample bias, thereby reducing the effects of any small ambient magnetic or electrostatic fields, which tend to deflect the low-energy SE’s away from the detector. In order for this technique to be effective, however, the electric field between the sample and detector—resulting from the negative sample bias—must be very nearly radial; the question of whether or not such is the case for the sample-detector geometry specific to this investigation is addressed further in [5.3 (b)] and Appendix C.

We now note that measurement of $\delta(\alpha', E'_e)$ at a fixed $\alpha'$ for a sufficient number of energies in the range $0 \leq E'_e \leq 50$ eV, constitutes an angle-resolved SE energy spectrum, denoted $\delta(\alpha', E_e)$ [where the absence of the prime on $E_e$ is intended to differentiate the AR spectrum $\delta(\alpha', E_e)$—a function of $E_e$—from the differential yield $\delta(\alpha', E'_e)$, which is a number]. It is these spectra which constitute the most important results of this investigation, for given a sufficient number of these AR spectra one can in principle determine the simultaneous angle-energy SE distribution function $\delta(\alpha, E_e)$—the maximum amount of information one can know about the SE population.

3.3 (b) $\delta_{AR}$ Determination

For a given emission angle $\alpha'$, direct determination of $\delta(\alpha')$ is accomplished through RD current measurements performed at detector biases of 0 V and −50 V. Given these two currents, the SE current $dI_{se}(\alpha')$ due to SE’s emitted in the solid angle $d\Omega_{RD}$ is just $dI_{se}(\alpha') = dI_{rd0}(\alpha') - dI_{rd50}(\alpha')$; we can then compute $\delta(\alpha')$ via

$$\delta(\alpha')_{meas} = \frac{1}{I_B} \frac{dI_{se}(\alpha')}{d\Omega_{RD}}$$

(3.6)

where $I_B$, once again, must also be measured (as described above). The suffix “meas” has been added to $\delta(\alpha')$ in Eq. (3.6) to distinguish this quantity from that which may be obtained in a separate fashion; namely, in addition to what can be regarded as the above direct measurement of the angle-resolved yield, $\delta(\alpha')$ can also be calculated from the angle-resolved spectrum via
\[ \delta(\alpha')_{\text{calc}} = \int_{0}^{50eV} \sigma(\alpha', E_e) \, dE_e \] (3.7)

where the integration can be performed analytically if a sufficiently accurate fit to the spectrum is found, or numerically by computing the area under the \( \delta(\alpha', E_e) \) vs \( E_e \) curve. It is important to emphasize that Eqs. (3.6) and (3.7) represent two distinct (though not quite independent) methods of obtaining \( \delta(\alpha') \); the two results can therefore be compared and their level of agreement used as one means of gauging the consistency of measurements obtained with the RD.

We now note that calculation of \( \delta(\alpha') \) for a sufficient number of angles allows one to construct an angular distribution function, \( \delta(\alpha) \). This distribution function can also be determined via the angle-energy distribution function by integrating over the energy range 0-50 eV:

\[ \delta(\alpha) = \int_{0}^{50eV} \delta(\alpha, E_e) \, dE_e \] (3.7a)

3.3 (c) \( \delta_{E'\theta} \) Determination

Unlike the other quantities under discussion, \( \delta(E'_e) \) is unique in that it cannot be directly measured with this instrument; in principle, however, it can be calculated from angle-energy-resolved yields via

\[ \delta(E'_e) = \frac{\pi}{2} \int_{0}^{\pi/2} \delta(\alpha, E'_e) \, sin\alpha \, d\alpha, \] (3.8)

given \( \delta(\alpha', E'_e) \) for a sufficient number of emission angles. The integral can then be calculated analytically if a suitable expression for the function \( \delta(\alpha, E'_e) \) can be found, or numerically by computing the area under the \( \delta(\alpha', E'_e) \) vs \( \alpha \) curve.

As above, note that calculation of \( \delta(E'_e) \) for a sufficient number of energies in the range \( 0 \leq E'_e \leq 50 \) eV constitutes an integrated SE spectrum, \( \delta(E_e) \)—i.e., integrated over all emission angles—or one might prefer to call it an energy distribution function. This angle-integrated spectrum could also be calculated via the angle-energy distribution function by integrating over all emission angles:
\[ \delta(E_c) = 2\pi \int_0^{\pi/2} \delta(\alpha, E_c) \sin \alpha \, d\alpha \]  

(3.8a)

(where we have assumed the distribution symmetric with respect to the azimuthal angle \( \varphi \)).

3.3 (d) Determination of the Total SE Yield

Direct determination of \( \delta \) is accomplished by means of primary and tertiary sample current measurements performed at sample (both primary and tertiary) biases of 0 V and +50 V, as follows: biasing the primary sample to +50 V prevents the SE’s, nearly all of whose energies are less than 50 eV, from escaping, while monitoring the tertiary sample current tells us how much of the primary sample current is return current [defined in Sec. 3.1(c)]; the total SE current \( I_{se} \) is then given by

\[ I_{se} = \left[ I_{S(50)} - I_{r(50)} \right] - \left[ I_{S(0)} - I_{r(0)} \right], \]

for \( I_{S(50)} \) and \( I_{S(0)} \) the primary sample currents at 0 V and +50 V, respectively, and \( I_{r(50)} \) and \( I_{r(0)} \) the return (tertiary sample) currents at 0 V and +50 V, respectively. Given \( I_{se} \), along with the beam current, \( I_B \), we can calculate \( \delta \) via

\[ \delta_{\text{meas}} = \frac{\left[ I_{S(50)} - I_{r(50)} \right] - \left[ I_{S(0)} - I_{r(0)} \right]}{I_B} \]  

(3.9)

Once again note the addition of the “meas” suffix. In addition to the direct measurement of \( \delta \) described by Eq. (3.9), the total SE yield can be calculated from the angle-resolved yields via

\[ \delta_{\text{calc1}} = 2\pi \int_0^{\pi/2} \delta(\alpha) \sin \alpha \, d\alpha \]  

(3.10)

given \( \delta(\alpha') \) for a sufficient number of angles, or from the angle-resolved spectra via

\[ \delta_{\text{calc2}} = 2\pi \int_0^{50eV} \int_0^{\pi/2} \sigma(\alpha', E_c) \sin \alpha \, d\alpha dE \]  

(3.11)

given \( \sigma(\alpha', E_c') \) for a sufficient number of angles and energies. Here it is important to note the method for determining \( \delta \) described by Eq. (3.9) is entirely independent of that described by Eqs. (3.10) and (3.11): \( \delta_{\text{meas}} \) is obtained with primary and tertiary sample current measurements, while \( \delta_{\text{calc1}} \) and \( \delta_{\text{calc2}} \) are derived via data obtained with the RD.
Unfortunately there exists a potentially significant source of systematic error in the total yield measurement technique described above; it is as follows. With the primary sample biased to +50 V, the notion is that all SE’s being emitted from the primary sample will return to the primary sample (where it is emphasized that biasing the sample to +50 V does not prevent the SE’s from being emitted; rather, they are indeed emitted, though they must ultimately return as their energy is insufficient to escape the sample’s potential well). However, given the geometry of the primary-tertiary sample arrangement (Fig. 3-2) and the fact that the tertiary samples are biased along with the primary, it is reasonable to assume that at least some of the SE’s leaving the sample are being collected by the tertiary samples when both primary and tertiaries are biased to +50 V. Looking again at Eqs. (3.9) and (3.16), this fact implies systematic errors in the currents $I_{S(50)}$ and $I_{r(50)}$, resulting in systematic errors in $\delta$ and $\eta$; specifically, the measured values of $I_{S(50)}$ will be low and the measured values of $I_{r(50)}$ will be high. Precisely how this affects $\delta$ and $\eta$ must be determined; data enabling an estimation of this systematic error are presented in [4.4 (b)] and the calculation is carried out in detail in Appendix A.

3.3 (e) BSE Yield Determinations

Determination of the four BSE yields $\eta$, $\eta(\alpha')$, $\eta(E_e')$, and $\eta(\alpha', E_e')$, as well as the spectra $\eta(\alpha', E_e)$ and $\eta(E_e)$, is accomplished completely analogously to the corresponding SE yields and spectra discussed above (with the modification that $E'_e$ lies in the range $50 \text{ eV} < E'_e \leq E_b$). Since further discussion regarding these quantities would only be repetitive, the results are simply stated:

$$\eta(\alpha', E_{s12}) = \frac{1}{I_B} \frac{d^2 I_{12}}{d\Omega_{RD} dE_{s12}}$$

(3.12)

$$\eta(\alpha')_{\text{meas}} = \frac{1}{I_B} \frac{dl_{rd(-50)}(\alpha') - dl_{rd(\phi)}(\alpha')}{d\Omega_{RD}} = \frac{1}{I_B} \frac{dl_{bse}(\alpha')}{d\Omega_{RD}}$$

(3.13)

$$\eta(\alpha')_{\text{calc}} = \int_{50 \text{ eV}}^{E_B} \sigma(\alpha', E_e) dE_e$$

(3.14)
This concludes our review of the instrument and method utilized in this investigation; results of their application to polycrystalline gold are now presented.
CHAPTER 4
TOTAL YIELD MEASUREMENTS

Measurements accomplished in this investigation represent an essentially first-round evaluation of the instrument’s angle-resolved capabilities, and continued testing and verification of the instrument’s total SE and BSE yield capabilities. While the primary focus of this investigation has been the measurement of angle-resolved secondary electron spectra, considerable effort has been expended toward total SE and BSE yield determination as well. Specifically, δ and η measurements were accomplished for: (i) different surfaces at a given sample location (e.g., the sample center), (ii) different locations across a given surface, (iii) different incident beam energies (surfaces A0i and D9 only), and (iv) negatively biased samples. The primary purpose of these measurements is to assess the reliability and accuracy of the instrument and method; nevertheless, some measure of new discovery is present as well.

4.1 Samples

4.1 (a) Choice of Sample Material

The selection of a sample material for this investigation was based on the following: (i) the need to further verify and evaluate the instrument and techniques; (ii) the absence of in situ surface characterization capabilities; (iii) the desire to perform new—i.e., never-before-accomplished—SEE measurements; and (iv) the desire to accomplish some level of theoretical evaluation based on our experimental results. With respect to these considerations, polycrystalline gold offers a number of advantages. First, previous δ and η determinations, performed by other investigators, are readily available in the literature and in reasonable agreement with one another [Thomas and Pattinson, 1970; Kanter, 1957; Holliday and Sternglass, 1957; Reimer and Drescher, 1977]. Comparison of total SE and BSE yields obtained in the present work with results in the literature are therefore an important means of instrument verification. Second, gold forms no stable oxides; this fact means that it is relatively easy to clean and to keep clean, allowing some measure of confidence in surface condition—an important consideration given our present inability to perform in situ
surface characterization. Third, gold remains essentially uncharacterized with respect to angle-resolved SE spectra, presenting an opportunity for contribution of new measurements to the field. Finally, there exist a number of theoretical SEE and backscattering formulations, applicable to gold, in need of experimental evaluation [Chung and Everhart, 1974; Rösler and Brauer, 1981a; Gannachaud and Callier, 1979a; Jablonski et al., 1989, Jablonski, 1991; Jablonski et al., 1993].

4.1 (b) Sample Preparation

Data for this investigation were collected from 30 different surfaces on three separate samples of polycrystalline gold. All three samples were approximately 0.5 x 0.5 cm, cut from the same piece of 0.127-mm thick, 4N purity Au foil (obtained commercially from the Aldrich Co.). To remove surface contaminants prior to vacuum insertion, the samples were chemically cleaned in consecutive baths of toluene (2-4 min), methylene chloride (2-4 min), acetone (5-7 min), isopropyl alcohol (2-4 min), methanol (3-5 min), distilled water (rinse), warm nitric acid (~30 min), and distilled water (~10 min) (LB III, p. 30w). Upon completion of this sequence of solvents, the samples were passed through an oxidizing flame and then, in order to minimize exposure of the surface to atmospheric contaminants, stored in distilled, deionized water until vacuum chamber insertion. Just prior to chamber insertion, a given sample was affixed to a primary sample blank (a 25 x 25-mm piece of rhodium-coated OFHC copper) with a small amount of silver paint—a conducting, UHV-compatible adhesive (LB III, p. 30w). Figure 4-1 is a photograph of a typical sample and mount, ready for chamber insertion. While difficult to quantify, each

![Figure 4-1](image_url)
sample surface was generally smooth, though small scratches in the surfaces were visible (as seen in the figure). Throughout the course of the measurements, the surface on a given sample was considered to be "new" (different) whenever the sample was subjected to any process with a reasonable chance of altering its SEE characteristics—e.g., ion sputtering, annealing, and prolonged energetic electron bombardment. A detailed summary of the processes leading to each new sample surface is presented in Appendix B.

4.2 Measurement Notes

Unless otherwise specified, all measurements were accomplished under the following conditions:

- **Base Pressure:** $< 1 \times 10^{-10}$ Torr
- **Operating Pressure:** $< 5 \times 10^{-10}$ Torr
- **Sample Temperature:** $27 \, ^\circ C \pm 2 \, ^\circ C$ (room temp.)
- **Incident Beam Energy:** 1500 eV ± 1 eV
- **Incident Beam-Current Density:** $\sim 10^{-5}$ A/cm$^2$ ($I_B \sim 200$ nA, spread over a 1.5-mm beamspot)

Beam currents in this investigation were chosen in order to minimize the rate of beam-induced surface modifications—specifically, the well-known effect of carbon contamination through electron-stimulated adsorption (ESA) [Hillier, 1948; Davies and Dennison, 1997]. It was found that the ESA of carbon did not occur at a problematic rate for beam-current densities $\leq \sim 10^{-4}$ A/cm$^2$. The incident beam energy of $E_B = 1.5$ keV was chosen primarily as a result of performance issues relating to the electron gun (e.g., stability, beamspot size, etc.). Each pair of $\delta, \eta$ determinations required approximately 15 sec of beam time on the sample surface.

4.3 Summary of Principal Findings

A considerable volume of data and analysis are presented throughout the remainder of this chapter. Before viewing the data in detail, what are considered to be the principal findings of the total yield measurements are summarized. It is hoped that this approach will better enable the reader to assimilate the
more detailed presentation to follow. Pertaining to the present investigation, then, evidence to the following is presented throughout the remainder of the chapter:

1. Surfaces which most closely resembles pure gold are sputtered, baked-out surfaces;
2. Argon sputtering likely resulted in surfaces contaminated with deeply embedded argon, affecting both $\delta$ and $\eta$ determinations;
3. Gold samples were generally smooth at the micron level;
4. SEE and backscattering characteristics were generally uniform across the sample surfaces;
5. Systematic error in the total yield determinations is in the range $-6\%$ to $-20\%$ for $\delta$, and $+15\%$ to $+51\%$ for $\eta$.
6. Total yield vs negative sample bias measurements indicate differences in work functions between the sample and other surfaces in the vacuum chamber; for total yield measurements performed at zero sample bias only (in this investigation), there exists the possibility of obtaining erroneously low $\delta$ values do to these work function differences;
7. Work function differences between the primary and tertiary samples may be producing a sample-surface-dependence in the systematic error.

Details of the observations leading to the above conclusions are now presented.

**4.4 $\delta$ and $\eta$ as Functions of Surface**

The gold samples utilized in this investigation were subject to a number of potentially modifying processes with respect to SEE and backscattering. Specifically, these processes were:

(i) annealing
(ii) ion sputtering
(iii) electron bombardment.

The potential effects of these processes are summarized as follows:

1. *Annealing.* In general, heating a sample can lead to the diffusion of bulk contaminants from within the sample to the surface, the evolution of embedded contaminants from the surface, the desorption of weakly-bound (physisorbed) surface contaminants, the formation of more tightly bound (chemisorbed) surface contaminants through heat-induced (or enhanced) chemical reactions
at the surface, and actual changes in the crystalline structure of the sample. In this investigation, annealing was accomplished to only moderate temperatures (150 °C–300 °C), for periods varying in duration from a few hours to several days. More specifically, three general profiles were used: 150 °C for a period of 72–96 hr (associated with the vacuum chamber bakeout); ~200 °C for periods of 2–20 hr; and ~300 °C for periods of 10–30 hr. The melting point of Au is ~900 K, meaning anneals in this investigation were carried out in the range of ~45% - 65% of the melting point, significantly less than the ~80% required to effect significant structural rearrangement.

2. Ion-sputtering. Sputtering is generally employed as a tool for producing atomically clean surfaces via the removal of surface contaminants; it is an atomic-scale process akin to sandblasting the paint off of a car. In addition to the removal of surface contaminants, sputtering can also result in morphological changes in the surface (e.g., smoothing or roughening of the surface), and the embedding of contaminants—including the sputtering ions themselves—into the surface. Species employed are usually noble gases, in order to prevent chemical interactions with the sample. In addition to species, important sputtering parameters include incident energy, incident angle, and beam current density. Sputtering in this investigation was accomplished almost exclusively with argon (except for surface D9, which was sputtered with neon), at incident energies of either 500 eV or ~2.6 keV, at near-normal angles of incidence (~75°–90°), and with beam current densities in the range 0.1–1 μA/cm² for periods of 15 min to 15 hr (depending on beam current density).

3. Energetic electron bombardment. There are two well-known surface effects associated with energetic electron bombardment: electron-stimulated desorption (ESD) [Ramsier and Yates, 1990; van Oostrum, 1979] and electron-stimulated adsorption (ESA) [Hillier, 1947; Smith, 1976]. ESD involves the removal of surface contaminants as a result of energy deposited by the incident electrons, while ESA involves the deposition of surface contaminants—often carbon—as a result electron-beam induced chemical reactions at the surface. Important parameters in these processes are the incident electron energy and angle, and the beam current density. In this investigation, electron bombardment was accomplished almost exclusively at 1.5 keV and normal incidence, with beam current densities of ~10 μA/cm² (light) or 1000 μA/cm² (heavy).

Total SE and BSE yields for three basic surface types, corresponding to each of the above processes, were examined for three separate gold samples (see Appendix B for details regarding individual surfaces). By examining the relative effects of these processes on the SE and BSE yields, one hopefully gleans information regarding the relative natures of the surfaces—i.e., clean or contaminated, rough or smooth, etc. By comparing results between separate samples, one hopes to assess the level of measurement
repeatability, or precision. It is emphasized that these data do not point to the *accuracy* of the total yield
determinations; that question is addressed in Appendix A.

4.4 (a) Description of Results

Total SE and BSE yields for each surface examined in this investigation are presented in Table 4.1
and depicted graphically in Figs. 4-2 (a)–(c). From these data one notes the general result that annealing,
sputtering, and electron bombarding the samples do indeed produce distinct, measurable, and repeatable
changes in $\delta$ and $\eta$. Generally, it is observed that (i) light electron beam bombardment and short-duration
annealing result in small (~10% or less) but repeatable changes in $\delta$ and $\eta$, (ii) heavy electron beam
bombardment and longer-duration annealing, such as a 72-hour system bakeout, induce larger (~20%–50%)
changes in $\delta$ and $\eta$, and (iii) ion sputtering, regardless of the surface being sputtered, consistently produces
the same values for both $\delta$ and $\eta$. More specifically, it is observed that:

1. $\delta$ is a maximum and $\eta$ is a minimum for surfaces which have been annealed in the system
   bakeout, regardless of past sample history—i.e., sputtered or pristine (*e.g.*, A1a, B1, D3);
2. Sputtering produces consistent, repeatable values for both $\delta$ and $\eta$ (*e.g.*, A2, B3, D1);
3. Sputtering an atmosphere-exposed and/or baked-out surface always drives $\delta$ and $\eta$ in
   opposite directions; specifically, $\delta$ decreases and $\eta$ increases; further, the sputtering-
   induced changes are always nearly perfectly offsetting with respect to the total electron
   yield, $\sigma$—i.e., while $\delta$ and $\eta$ each undergo significant change individually as a result of
   sputtering, their sum, $\sigma$, remains nearly constant (*e.g.*, A1a→A2, B1→B2, D3→D4,
   D10→D11);
4. Strong enough annealing tends to raise $\delta$ and lower $\eta$ (*e.g.*, A7, A9, A10);
5. Heavy electron bombardment of any surface results in immediate, significant lowering of
   both $\delta$ and $\eta$ (*e.g.*, A4);
6. Light electron bombardment of a 200 °C-annealed surface for up to 45 hr has no
   significant effect on either $\delta$ or $\eta$ (*e.g.*, A3b);
7. Light electron bombardment of a sputtered surface after an extended (~1 month) UHV
   exposure has no significant effect on either $\delta$ or $\eta$ (*e.g.*, A1, D9);
TABLE 4.1. $\delta$, $\eta$, and $\sigma$ as a function of surface. Surfaces exposed to some measure of extended electron beam bombardment are listed in purple. Non-UHV surfaces are in blue. $E_b = 1.5$ keV.

<table>
<thead>
<tr>
<th>Surface</th>
<th>$\delta$ (±0.01)</th>
<th>$\eta$ (±0.005)</th>
<th>$\sigma$ (±0.02)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A0i</td>
<td>1.06</td>
<td>0.56</td>
<td>1.62</td>
</tr>
<tr>
<td>A0ii</td>
<td>0.99</td>
<td>0.55</td>
<td>1.54</td>
</tr>
<tr>
<td>A0iii</td>
<td>1.08</td>
<td>0.63</td>
<td>1.71</td>
</tr>
<tr>
<td>A0iv</td>
<td>1.07</td>
<td>0.64</td>
<td>1.71</td>
</tr>
<tr>
<td>A1</td>
<td>1.09</td>
<td>0.64</td>
<td>1.73</td>
</tr>
<tr>
<td>A1a</td>
<td>1.08</td>
<td>0.59</td>
<td>1.67</td>
</tr>
<tr>
<td>A2</td>
<td>1.00</td>
<td>0.68</td>
<td>1.68</td>
</tr>
<tr>
<td>A3</td>
<td>0.99</td>
<td>0.66</td>
<td>1.65</td>
</tr>
<tr>
<td>A3a</td>
<td>0.99</td>
<td>0.67</td>
<td>1.66</td>
</tr>
<tr>
<td>A3b</td>
<td>0.98</td>
<td>0.64</td>
<td>1.62</td>
</tr>
<tr>
<td>A4</td>
<td>0.50</td>
<td>0.48</td>
<td>0.98</td>
</tr>
<tr>
<td>A4a</td>
<td>0.51</td>
<td>0.48</td>
<td>0.99</td>
</tr>
<tr>
<td>A5</td>
<td>0.57</td>
<td>0.47</td>
<td>1.04</td>
</tr>
<tr>
<td>A6</td>
<td>0.98</td>
<td>0.69</td>
<td>1.67</td>
</tr>
<tr>
<td>A7</td>
<td>1.08</td>
<td>0.60</td>
<td>1.68</td>
</tr>
<tr>
<td>A7a</td>
<td>0.97</td>
<td>0.60</td>
<td>1.57</td>
</tr>
<tr>
<td>A7b</td>
<td>0.91</td>
<td>0.60</td>
<td>1.51</td>
</tr>
<tr>
<td>A8</td>
<td>0.96</td>
<td>0.70</td>
<td>1.66</td>
</tr>
<tr>
<td>A8a</td>
<td>0.97</td>
<td>0.69</td>
<td>1.66</td>
</tr>
<tr>
<td>A9</td>
<td>1.08</td>
<td>0.63</td>
<td>1.71</td>
</tr>
<tr>
<td>A9a</td>
<td>0.98</td>
<td>0.63</td>
<td>1.61</td>
</tr>
<tr>
<td>A10</td>
<td>1.03</td>
<td>0.59</td>
<td>1.62</td>
</tr>
<tr>
<td>A10a</td>
<td>0.91</td>
<td>0.58</td>
<td>1.49</td>
</tr>
<tr>
<td>B1</td>
<td>1.12</td>
<td>0.56</td>
<td>1.68</td>
</tr>
<tr>
<td>B2</td>
<td>0.95</td>
<td>0.68</td>
<td>1.63</td>
</tr>
<tr>
<td>B2a</td>
<td>0.99</td>
<td>0.67</td>
<td>1.66</td>
</tr>
<tr>
<td>B3</td>
<td>0.94</td>
<td>0.69</td>
<td>1.63</td>
</tr>
<tr>
<td>B3a</td>
<td>0.97</td>
<td>0.66</td>
<td>1.63</td>
</tr>
<tr>
<td>D1</td>
<td>0.98</td>
<td>0.68</td>
<td>1.66</td>
</tr>
<tr>
<td>D1a</td>
<td>1.03</td>
<td>0.63</td>
<td>1.66</td>
</tr>
<tr>
<td>D2</td>
<td>1.02</td>
<td>0.67</td>
<td>1.69</td>
</tr>
<tr>
<td>D3</td>
<td>1.17</td>
<td>0.54</td>
<td>1.71</td>
</tr>
<tr>
<td>D4</td>
<td>1.00</td>
<td>0.65</td>
<td>1.65</td>
</tr>
<tr>
<td>D5</td>
<td>0.97</td>
<td>0.65</td>
<td>1.62</td>
</tr>
<tr>
<td>D5a</td>
<td>1.03</td>
<td>0.64</td>
<td>1.67</td>
</tr>
<tr>
<td>D6</td>
<td>0.97</td>
<td>0.66</td>
<td>1.63</td>
</tr>
<tr>
<td>D6a</td>
<td>0.86</td>
<td>0.58</td>
<td>1.44</td>
</tr>
<tr>
<td>D7</td>
<td>0.95</td>
<td>0.68</td>
<td>1.63</td>
</tr>
<tr>
<td>D7a</td>
<td>0.89</td>
<td>0.63</td>
<td>1.52</td>
</tr>
<tr>
<td>D8</td>
<td>0.95</td>
<td>0.70</td>
<td>1.65</td>
</tr>
<tr>
<td>D9</td>
<td>0.95</td>
<td>0.69</td>
<td>1.64</td>
</tr>
<tr>
<td>D10</td>
<td>1.01</td>
<td>0.59</td>
<td>1.60</td>
</tr>
<tr>
<td>D11</td>
<td>0.93</td>
<td>0.71</td>
<td>1.64</td>
</tr>
<tr>
<td>D12</td>
<td>0.93</td>
<td>0.67</td>
<td>1.60</td>
</tr>
</tbody>
</table>
Figure 4-2 (a). $\delta$ and $\eta$ as functions of surface and sample location, sample $A$. $E_{ir}=1.5$ keV. Error bars are considerably smaller than the measurement markers displayed.
FIGURES 4-2 (cont'd). \( \delta \) and \( \eta \) as functions of surface and location for samples (b) B and (c) D, respectively. \( E_B = 1.5 \text{ keV} \). Error bars are considerably smaller than the measurement markers displayed.
8. Even light electron bombardment of a 300 °C-annealed surface results in an immediate reduction of δ, with no effect on η (e.g., A7a, A9a, A10a).

These trends are summarized by surface in Table 4.2. Standard error analysis accomplished for given surfaces and locations indicate standard deviations for both δ and η determinations are on the order of 0.5–1.0% (see Appendix A).

Surface Condition

Figure 4-3 depicts scanning electron microscope (SEM) images of a “pristine”, chemically cleaned and flamed surface, and an argon-sputtered surface (surface A11), respectively. Figure 4-4 depicts energy-dispersive x-ray (EDX) spectra for the same surfaces, accomplished concurrently with the SEM images. Both samples were exposed to atmosphere for ~24 hr prior to the imaging. The SEM images indicate that both the pristine and post-sputter surfaces are relatively smooth at the 1–10 micron level, the pristine surface is littered with micron-size contaminant particles, and the sputtered surface is clearly polycrystalline, dominated by grain boundaries, and devoid of visible contaminant particles. The EDX spectra, though not particularly surface sensitive (the penetration depth of the 10 keV beam into the surface is on the order of microns [Davies, 1996]), do indicate the relative absence of contaminant species—particularly carbon and oxygen—to the detection limit of the instrument (~0.1% concentration (Nabil Youssef, private communication, 1996)).

4.4 (b) Analysis

Sputtering Effects

Most notable of the above observations are the substantial changes in δ and η brought about by sputtering the initial, chemically cleaned and flamed, baked out surface. In particular, it is the significant (~30%) increase in η—the backscattering yield—which is most interesting. A significant change in δ as a result of sputtering is not surprising, as SEE is an extremely surface sensitive phenomenon; η, however, should not be particularly sensitive to changes in the first 50 Å or so in the sample surface. A significant change in η, therefore, may indicate sputter-induced effects occurring much deeper in the sample, perhaps
TABLE 4.2 Trends in $\delta$ and $\eta$ as a function of surface type.

<table>
<thead>
<tr>
<th>Surface Type</th>
<th>$\delta, \eta$ Characteristics</th>
<th>Example Surfaces</th>
</tr>
</thead>
<tbody>
<tr>
<td>Baked (150 °C, &gt;72 hr)</td>
<td>a. maximum $\delta$'s relative to other surfaces</td>
<td>A0i, A1a, B1, D3</td>
</tr>
<tr>
<td></td>
<td>b. minimum $\eta$'s relative to other surfaces</td>
<td>same</td>
</tr>
<tr>
<td>Annealed</td>
<td>a. low-temp (to ~200°C): no noticeable effect on either $\delta$ or $\eta$.</td>
<td>A0iv</td>
</tr>
<tr>
<td></td>
<td>b. moderate temp (to ~300°C): raises $\delta$ and lowers $\eta$.</td>
<td>A7, A9, A10</td>
</tr>
<tr>
<td>Sputtered</td>
<td>a. consistently sets $\delta$ to same value (~0.98, 0.97, and 0.96 for samples A, B, and D, respectively).</td>
<td>A2, A6, A8, B2, B3, D1, D4, D6, D7, D8, D9</td>
</tr>
<tr>
<td></td>
<td>b. consistently sets $\eta$ to same value (~0.69, for all samples).</td>
<td>same</td>
</tr>
<tr>
<td></td>
<td>c. sputtering after system bakeout or exposure to atmosphere significantly lowers $\delta$ and raises $\eta$, while leaving $\sigma (\delta+\eta)$ constant.</td>
<td>A0iii, B2, D4, D11</td>
</tr>
<tr>
<td>Electron Beam Bombarded</td>
<td>a. light (~10 $\mu$A/cm$^2$) bombardment of sputtered surface: $\delta$ increased initially (over ~first 10 hr), then decreased; no significant change in $\delta$ or $\eta$ for up to 5 hr</td>
<td>A3b, A8a, B2a, D1a, D5a, D6a, D7a</td>
</tr>
<tr>
<td></td>
<td>b. light bombardment of 200°C-annealed surface: no significant effect on either $\delta$ or $\eta$.</td>
<td>A3b</td>
</tr>
<tr>
<td></td>
<td>c. light bombardment of sputtered surface after extended (~1 month) vacuum exposure: no significant effect on either $\delta$ or $\eta$.</td>
<td>A1, D9</td>
</tr>
<tr>
<td></td>
<td>d. light bombardment of 300°C-annealed surface: immediate, continued lowering of $\delta$, no measurable effect on $\eta$.</td>
<td>A7a, A7c, A9a, A10a</td>
</tr>
<tr>
<td></td>
<td>e. heavy (~1000 $\mu$A/cm$^2$) bombardment of any surface: immediate, significant lowering of both $\delta$ and $\eta$.</td>
<td>A4</td>
</tr>
<tr>
<td></td>
<td>f. off beamspot locations: significantly increased $\delta$, no effect on $\eta$.</td>
<td>A4, D6a</td>
</tr>
</tbody>
</table>
Figure 4-3. Scanning electron micrographs of polycrystalline Au before (top) and after (bottom) argon sputtering.
FIGURE 4-4. Energy dispersive x-ray analysis (EDX) spectra for samples of polycrystalline Au before (top) and after (bottom) argon sputtering.
contamination which the sputtering removes, thus altering $\eta$ from that of the contaminant layer to that of gold, or the sputtering modifies the sample to a depth on the order of 500–1000 Å. Evidence and reasoning do not support the notion of a thick contaminant layer at the sample surface. First, the formation of a 1000-Å thick contaminant layer on gold, solely as the result of surface adsorption, seems unlikely; the chemical cleaning and flaming process described in (4.2) is known to produce a very clean gold surface (Greg Swain, Utah State University, Department of Chemistry, personal communication, 1998) and 1000 Å of contamination as a result of the short (~1 hr) exposure of the sample to atmosphere during vacuum chamber insertion is not reasonable. Second, the sputtering rates and durations used in this investigation were sufficient to remove, at most, ~100 Å from the surface. Third, the energy dispersive x-ray (EDX) spectra depicted in Fig. 4-4 show no evidence of a relatively thick contaminant layer—for either the pristine, chemically cleaned and flamed surface, or for the sputtered surface (Figs. 4-3). Finally, one might argue that the contaminant layer is not the result of adsorption, but due to the migration of bulk contaminants within the sample to the surface—perhaps due to the bakeout, which is essentially a 72-hour, 150 °C anneal. But if this were the case, the contaminant level at 1000 Å should be no different from that at, say, 10,000 Å, and sputtering should have no effect on $\eta$. Since this line of reasoning would seem to rule out the removal of thick contaminant layers as an explanation for the sputter-induced elevation of $\eta$, we are left with sputter-induced surface modification as the only apparent explanation.

Sputter-induced surface modification can take the form of morphology changes, the embedding of contaminants—likely argon—deep into the surface, or a combination of the two. Morphologically, it is not uncommon for argon sputtering of gold to result in significant roughening of the surface at the micron level [Barber et al., 1973], and Figs. 4-3(a) and 4-3(b) suggest that such may have been the case here. Figure 4-3(a) shows surface roughness on a scale of ~0.2 μm, while Fig. 4-3(b) shows surface roughness on a scale of ~2 μm (the distance between the horizontal striations visible in the second image). It appears, however, that the increased roughness was not so much caused by the sputtering, as uncovered by it. That is, the nature of the roughness—the parallel striations—would seem more plausibly a result of the sample fabrication (rolling, perhaps) than sputtering. Regardless, it is conceivable that the effect could be an
overall reduction in $\delta$, as is typical for rougher surfaces (though why $\eta$ should be affected is unclear).

Other evidence, however, casts doubt on this explanation: surface D2 is presumably very similar to A11 morphologically [the surface depicted in Fig. 4-3(b)], though its $\delta$ and $\eta$ values are nearly identical to those of surfaces A0/ and B1—surfaces that have never been sputtered, and therefore are presumably similar to Fig. 4-3 (a) morphologically. Thus it would seem that the case for morphology-induced changes in $\delta$ and $\eta$ is tenuous at best.

Evidence supporting deep argon embedding as the mechanism underlying the sputter-induced change in $\eta$ is stronger, though not entirely satisfying. Residual gas analyzer (RGA) measurements of the contaminant spectra during the lengthy, 300 °C anneals producing surfaces A7, A9, and A10 indicated the evolution of a considerable volume of argon from the sample, indicating argon had been embedded in the surface. Furthermore, it is observed that the influence of these anneals (and presumably the associated argon evolution from the sample surface) on $\delta$ and $\eta$ was to push them in the directions of their pre-sputter values. It is noted, however, that if the observed changes are a result of deep argon embedding, the effect is not particularly sensitive to species: measurements performed on surface D9, which was Ne$^+$ sputtered, revealed the same behavior. Thus, it would seem that the data presented thus far do not support an unequivocal solution to the mystery of increased BSE yield with sputtering: the removal of a thick layer of surface contamination can be ruled out, but evidence concerning morphology changes and argon embedding remains ambiguous. One additional clue, however, remains to be discussed.

Reviewing Table 4.1, it is observed that while sputtering atmosphere-exposed or baked-out surfaces consistently results in a 10%-15% drop in $\delta$ and a 15%-20% increase in $\eta$, the total electron yield, $\sigma$, remains nearly constant (to within ~3%)! In other words, it appears that while the numbers of SE's and BSE's are changing individually as a result of sputtering, the total number of electrons leaving the surface, $\delta + \eta$, is not changing (where it is again emphasized that these are the defined SE’s and BSE’s). This fact may simply prove to be coincidence, though to have the number of BSE’s increase by precisely the same number as the SE’s decrease seems a bit coincidental. The implication is that the effect is a matter of bookkeeping—i.e., for some reason electrons that were being counted as SE’s before sputtering are being
counted as BSE's after sputtering. One possible explanation is that sputtering is somehow affecting the energy distribution of the emitted electrons; that is, some of the electrons with energies below 50 eV prior to sputtering are being emitted with energies above 50 eV afterwards. While such a notion seems plausible at first glance, it does not hold up well under closer scrutiny. SE and BSE spectral evidence presented in Chap. 5 makes it clear that for such an effect to account for the δ, η, σ mystery quantitatively, SE's with energies as low as 20 eV would need to be energized above 50 eV as a result of sputtering, which is simply not reasonable. Another option is that some kind of instrument effect is at work—a systematic error—and in fact there is strong evidence to support this notion. Evidence confirming a systematic error in the instrument is presented at the end of this section, and a possible mechanism linking this error and the δ, η, σ observations is discussed in [4.7 (b)].

Annealing Effects

The relatively low annealing temperatures used in this investigation (150 °C–300 °C) were not sufficient to induce chemical reactions at the sample surface (Greg Swain, personal communication, 1998), nor changes in crystalline structure (which generally require temperatures on the order of 80% of a materials melting temperature—about 720 K in the case of Au, whose melting temperature is ~900 K). Thus changes in δ and η as a result of sample heating were likely due to the diffusion of bulk contaminants to the surface, the evolution of these contaminants from the surface, and the desorption of physisorbed surface contaminants. The results of surfaces D2 and D3 suggest that the 150 °C, 72-hr anneal in association with the vacuum chamber bakeout was sufficient for the removal of any contaminant layer formed as a result of atmosphere exposure during chamber insertion, and for the nearly complete evolution of any contaminant species embedded in the surface as a result of sputtering. The immediate change (lowering) in δ resulting from even light electron bombardment following the 300 °C anneals suggests a substantial presence of carbon on the sample surface following these anneals (where the ESA of carbon is responsible for the lower δ's). The carbon could have migrated to the surface and been concentrated there as a result of the anneal, or it could have evolved into the UHV atmosphere and adsorbed on the sample
surface. There is evidence to support the latter; RGA measurements of the contaminant species present in
the chamber during these anneals revealed a substantially increased presence of CO and CO$_2$, most likely
coming from the sample heater. Furthermore, it was observed that extended exposure of the surface to
vacuum (~1 month at ~1x10^{-10} Torr) appeared to reduce the carbon presence on the sample surface, as
evidenced by unchanging δ’s over indefinite exposure to light electron bombardment.

**Electron Bombardment Effects**

Changes in δ and η as a result of energetic electron bombardment were completely consistent with
the known effects of ESA and ESD. Specifically, the dramatic drop in both δ and η with extended
exposure to heavy electron bombardment (surface A4) is characteristic of the ESA of a thick layer of
carbon (~200 Å); furthermore, the simultaneous increase in δ for off-center sample locations can be
attributed to the desorption of embedded argon (and/or other surface contaminants), due to local heating
causd by the nearby electron beam.

**Comparison with Previous Measurement**

Based on the preceding discussion, it would seem that those surfaces which had just undergone a
bakeout are the closest to atomically clean gold (e.g., A0i, A1α, B1, and D3), while just-sputtered surfaces
appear to be gold modified by embedded argon. This hypothesis can be tested further by examining the
results of previous investigators—specifically, those of Thomas and Pattinson [1970]. These
measurements were chosen for comparison with the present work based on the comparability of the
samples and the quality of the measurement apparatus and technique [Thomas and Pattinson, 1969, 1970].
Specifically, their samples were high purity, polycrystalline Au, vapor deposited *in situ*, measurements
were performed under UHV and required only a few seconds to complete (giving high confidence that the
samples were very close to atomically clean, though *in situ* surface characterization was not performed),
and incident energies included $E_B = 1500$ eV—the energy used in this investigation. On careful
TABLE 4.3. Comparison of representative total yields for the present work, with those of Thomas and Pattinson [1970] for gold at $E_d = 1.5$ keV.

<table>
<thead>
<tr>
<th>Surface</th>
<th>$\delta$ (±.01)</th>
<th>$\eta$ (±.005)</th>
<th>$\sigma$ (±.02)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Thomas &amp; Pattinson (unknown precision)</td>
<td>1.21</td>
<td>0.460</td>
<td>1.67</td>
</tr>
<tr>
<td>Baked out</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>A0i</td>
<td>1.06</td>
<td>0.560</td>
<td>1.62</td>
</tr>
<tr>
<td>A1a</td>
<td>1.09</td>
<td>0.590</td>
<td>1.68</td>
</tr>
<tr>
<td>B1</td>
<td>1.12</td>
<td>0.560</td>
<td>1.68</td>
</tr>
<tr>
<td>D3</td>
<td>1.17</td>
<td>0.540</td>
<td>1.71</td>
</tr>
<tr>
<td>Sputtered</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>A2</td>
<td>1.00</td>
<td>0.680</td>
<td>1.68</td>
</tr>
<tr>
<td>B3</td>
<td>0.94</td>
<td>0.690</td>
<td>1.63</td>
</tr>
<tr>
<td>D6</td>
<td>0.97</td>
<td>0.670</td>
<td>1.64</td>
</tr>
<tr>
<td>D7</td>
<td>0.95</td>
<td>0.680</td>
<td>1.63</td>
</tr>
<tr>
<td>D8</td>
<td>0.95</td>
<td>0.690</td>
<td>1.64</td>
</tr>
</tbody>
</table>

examination of the reported details of their apparatus and technique, it is reasonable that the SE and BSE yields for a clean surface in the present investigation should be comparable to their results.

Table 4.3 shows that the total electron yield $\sigma$ obtained by Thomas and Pattinson is in excellent agreement with those observed in the present investigation, differing by less than 3% from the results obtained for all surfaces. However the comparison also reveals significant disagreement among the SE and BSE yields; specifically, the $\delta$'s of the present work are 10–30% low, while the $\eta$'s are 20–50% high (though one does note significantly less discrepancy for the baked out surfaces than for the sputtered surfaces—the smallest occurring for surface D3, which had been sputtered prior to its bakeout, and whose bakeout was the longest of all at ~96 hr—24 hr longer than the others). This result is taken as further evidence—albeit tenuous—supporting the notion that the baked-out surfaces in this investigation are essentially clean, while the sputtered surfaces are contaminated with embedded argon.

The discrepancies between the $\delta$'s and $\eta$'s of surface D3 and the results of Thomas and Pattinson (T&P) may be due to (i) actual physical differences between the samples, or (ii) systematic error in the
instrument and method of the present work. Regarding physical differences between the samples, surface contamination and surface morphology—namely roughness—are potential sources of discrepancy between the $\delta$'s. In particular, it is easily conceivable—even likely—that the surfaces of this work are "dirtier" than those of T&P, and it is easy to imagine that the mechanically formed sample of the present investigation, while appearing relatively smooth, is rougher than the vapor deposited sample of Thomas and Pattinson. Effects of both of these conditions could account for the observed lower $\delta$'s in the present work [Thomas and Pattinson, 1969; Reimer, 1993]. But while such conditions might readily account for discrepancies in the SE yields, it is hard to believe that either surface contamination or surface roughness could be the cause of disagreement between $\eta$'s—a parameter much less sensitive to surface condition. In fact, there is every reason to believe that the BSE yields of this investigation and those of T&P should agree to within a few percent, as there is every reason to believe that the bulk properties of the samples in the two investigations are nearly identical (i.e., high-purity, polycrystalline gold). Thus one is left with good reason to suspect some level of systematic error in the present work.

**Systematic Error**

The potential flaw in the total yield measurement technique noted in [3.3 (d)]—the collection of some SE’s by the tertiary samples (rather than the primary sample) when both primary and tertiary samples are biased to $+50$ V—remains the most glaring candidate for systematic error in the $\delta$ and $\eta$ measurements. If we assume that at least one of the surfaces of this investigation has the same BSE yield as that of T&P, and that any discrepancy between that surface's $\eta$ and that of T&P can be attributed to systematic error of this origin, upper and lower bounds on the magnitude of this error can be estimated with data from the surfaces with the largest and smallest calculated $\eta$'s. This calculation is done in detail in Appendix A [A.2], where only sample D surfaces were considered. The results indicate an upper bound on the systematic errors in $\delta$ and $\eta$ of $-20\%$ and $+51\%$, respectively (using surface D6 data), and lower bounds of $-6\%$ and $+15\%$ (using surface D3 data). Data allowing for the further refinement of these estimates are
presented in [5.2 (c)]; specifically, it is found that the systematic errors in the \( \delta \) and \( \eta \) determinations are about \(-18\%\) and \(+47\%\), respectively.

Finally, one additional check can be made on the viability of SE collection by the tertiaries as the source of systematic error. We note that if the tertiary samples are indeed collecting SE's erroneously at \(+50\) V, the result would be to increase \( I_{r(50)} \) and decrease \( I_{s(50)} \). Examining Eqs. (3.9) and (3.16), it is easy to see that in such a case the result would be a measured \( \delta \) that is too low and a measured \( \eta \) that is too high—consistent with what we are observing (if the T&P measurements are taken as correct).

4.4 (c) Conclusions

To review, the following general observations were made at the beginning of [4.4 (a)] regarding \( \delta \) and \( \eta \) as a function of surface: (i) light electron beam bombardment and short-duration annealing result in small (\(-10\%\) or less) but repeatable changes in \( \delta \) and \( \eta \); (ii) heavy electron beam bombardment and longer-duration annealing, such as a 72-hour system bakeout, induce larger (\(-20\%–50\%\)) changes in \( \delta \) and \( \eta \); and (iii) ion sputtering, regardless of the surface being sputtered (or sputtering species), produces consistent values for both \( \delta \) and \( \eta \). Pertaining to these observations, evidence to the following has been presented:

1. Ion sputtering results in a smooth gold surface, possibly contaminated with embedded argon to a depth of 500–1000 Å;
2. Hotter annealing (~200–300 °C) initially contaminates the sample surface with physisorbed carbon as a result of evolving CO and CO\(_2\) from the heater into the chamber atmosphere; the carbon will eventually desorb of its own accord if the sample is allowed to sit under UHV vacuum;
3. Heavy electron beam bombardment immediately results in characteristic ESA of carbon;
4. Light electron beam bombardment initially desorbs physisorbed contaminants and evolves embedded argon at the beam location through ESD; depending on the annealing history of the sample, noticeable ESD of carbon can be immediate, or may not occur at all (up to at least 45 hr of beam exposure);
5. The long-duration anneals (system bakeouts) desorb physisorbed surface contaminants and evolve embedded argon from the surfaces of previously sputtered samples; thus, these surfaces are the closest in this investigation to clean, polycrystalline gold;

6. Surface D3 is likely the best surface against which to compare the results of Thomas and Pattinson [1970]; it is assumed this surface is very similar to A11, for which the SEM image [Fig. 4-3 (b)] and EDX spectrum [Fig. 4-4 (b)] show no sign of surface damage or of carbon or oxygen contamination;

7. There is a systematic error present in the total yield measurement technique, and this error has been estimated; the results are upper bounds of −20% and +59% for δ and η, respectively, and lower bounds of −8% and +18%;

8. That δ decreases, η increases, and σ remains constant when baked-out or atmosphere-exposed surfaces are sputtered is possibly related to the systematic error in the total yield measurement technique.

4.5 δ and η as Functions of Location

To aid in assessing both surface uniformity and the precision of our δ and η determinations, measurements were performed for as many as five standard locations, depicted schematically in Fig. 4-5, on each sample surface. Results and analysis are presented below.

4.5 (a) Description of Results

Figures 4-2 (a) and (c) depict the results of δ and η measurements made for various sample locations on samples A and D, respectively. (Relatively few off-center measurements were performed on sample B.) Note that comparisons between the center location and off-center locations for purposes of assessing variability are not meaningful for cases in which the locations were not subjected to the same treatment, as in the case of surface A4 regarding electron beam exposure. These data indicate:

1. quantitative variability in δ and η across the sample surface of no more than 6% and 4%, respectively, (for a given surface);
2. the $\delta$ values obtained for the center location, where angle-resolved measurements were obtained, fall very near the average of the other locations (where comparative data are available);

3. a consistency among the relative values of the various sample locations as a function of surface (i.e., one notes that $\delta$ for the top center (TC) location is always greater than for any other location, while $\delta$ for the right center (RC) location is nearly always the least).

4.5 (b) Analysis and Conclusions

The ~5% agreement between measurements across the surface indicates a relatively uniform surface from the point of view of SEE and backscattering characteristics. Regarding measurement precision, standard error analysis accomplished on surfaces A0i, A3a, A6, D2, and D3—surfaces for which multiple measurements were accomplished over multiple locations—indicates relative uncertainties in $\delta$ and $\eta$ determinations of about 1% and 0.5%, respectively (see Appendix A). This is an acceptable level of precision.
4.6 \( \delta \) and \( \eta \) as Functions of Beam Energy

Total yield measurements for a number of incident beam energies in the range 1–3 keV were accomplished on surfaces \( A0i \) and D9. Comparison of these results with those of previous investigators aids in assessing the accuracy of the present yield determinations and serves to further assess the reliability of the instrument and method employed in this investigation. Additionally, there are a number of empirically derived relations for \( \delta \) vs \( E_B \) that are available for comparison with the present results.

4.6 (a) Description of Results

Figure 4-6 depicts \( \delta, \eta, \) and \( \sigma \) vs \( E_B \) for surfaces \( A0i \) and D9. Two sets of \( \delta \)'s and \( \eta \)'s are presented for each surface: values as measured (i.e., uncorrected for systematic error), and values corrected for the systematic error described in 4.4 (b). The corrected values are located midway between the upper and lower bounds determined by the systematic error estimates (calculated in Appendix A), while the error bars depict the upper and lower bounds themselves. Also presented in the figure are the results of three other investigators, Thomas and Pattinson [1970], Holliday and Sternglass [1957], and Reimer [1993], along with three semiempirical models describing \( \delta \) vs \( E_B \). Evident in the figure and data are the following:

(i) The shape of the \( \delta \) vs \( E_B \) curves obtained for both surfaces in this investigation closely match those of Thomas and Pattinson and the NASCAP model

(ii) Quantitative agreement with the Thomas and Pattinson results is better for surface \( A0i \) than for surface D9.

More specifically, \( \delta \)'s and \( \eta \)'s for surface \( A0i \) (corrected for systematic error) are nearly identical with those of Thomas and Pattinson. For surface D9, \( \eta \)'s are in excellent agreement with T&P, but the \( \delta \)'s are \( \sim 12\% \) low. For both surfaces, the total electron yield, \( \sigma \), is \( \sim 5\% \) lower than that of Thomas and Pattinson.
SE and BSE Yields vs. Incident Energy
Comparison with Previous Experiment

Figure 4-6. $\delta$, $\eta$, and $\sigma$ as functions of incident electron energy: comparison of (a) surface A0i and (b) surface D9 measurements with previous experiment and empirical SEE models. "Corrected" values are corrected systematic error, where the error bars represent uncertainty in the systematic error. Random errors for present experiment values are significantly smaller than the markers displayed. Experimental error for previous measurements were not reported by the authors.
4.6 (b) Analysis

**Comparison with Previous Experiment**

These results are encouraging; they indicate the expected trend of $\delta$ as a function of beam energy, and differences in magnitude can be attributed to surface contamination and systematic error in $\delta$ and $\eta$ determination. That surface $\text{A0}/i$ shows better agreement with the Thomas and Pattinson measurements than does $\text{D9}$ is generally consistent with the conclusions of [4.4(b)]—that $\text{A0}/i$ is unaffected by embedded argon (or neon, in the case of D9).

**Comparison with Theory**

The three models displayed in Fig. 4-6 are given by:

- **NASCAP:**
  \[
  \delta(E) = 1.114 \left( \frac{E}{E_{\text{max}}} \right)^{0.35} \left( 1 - \exp \left[ -2.28 \left( \frac{E}{E_{\text{max}}} \right)^{1.35} \right] \right) \delta_{\text{max}}
  \]

- **Reimer:**
  \[
  \delta(E) = 1.31 \left( \frac{E}{E_{\text{max}}} \right)^{0.8} \left( 1 - \exp \left[ -1.45 \left( \frac{E}{E_{\text{max}}} \right)^{1.8} \right] \right) \delta_{\text{max}}
  \]

- **Sternglass:**
  \[
  \delta(E) = \frac{E}{E_{\text{max}}} \exp \left[ 2 - 2 \left( \frac{E}{E_{\text{max}}} \right)^{0.5} \right] \delta_{\text{max}}
  \]

where $\delta_{\text{max}}$ and $E_{\text{max}}$ are 1.4 and 800 eV, respectively, as taken from the CRC [Weast, 1979, p. E-349]. These formulations correspond to two of the basic semiempirical formulations described in [2.3(a)]. Specifically, the NASCAP and Reimer models are based on a formulation by Dionne [1975] which assumes the constant-loss power law of Eq. (2.3), with $n = 1.35$ and $n = 1.8$, respectively. The Sternglass model, by contrast, is based on the Bethe relation of Eq. (2.4). Good agreement between the data of this investigation and the NASCAP model reaffirms the demonstrated success of this formulation for energies less than $-4 E_{\text{max}}$, and the previously observed failure of the Sternglass formulations for energies less than $-8 E_{\text{max}}$. 
4.6 (c) Conclusions

Results of the yield vs energy measurements lend additional support to the systematic error analysis of [4.4 (b)], as well as the notion that sputtering—for some reason—possibly argon embedding—is impacting $\delta$ and $\eta$ determinations in such a manner as to make them less gold-like (if the Thomas and Pattinson measurements are to be believed).

4.7 $\delta$ and $\eta$ for a Negatively Biased Surface

Since the angle-resolved yield and spectra measurements of this investigation were accomplished under varying conditions of negative sample bias, it is important to determine whether or not negative biasing alters a surface’s SEE characteristics—specifically the SE yield. The scheme used to determine $\delta$ and $\eta$ for an unbiased sample (described in [3.3 (d)]) was adapted for use in the case of a negatively biased sample (see [A.3] in Appendix A for details), and determinations were made for four different surfaces in the range 0 V to −20 V. As it turns out, these measurements bring an important and subtle aspect of SEE measurement into specific relief—namely, the importance of work functions.

4.7 (a) Description of Results

Total yield measurements ($E_b = 1.5$ keV) for surfaces D1, D10, D11, and D12 under varying conditions of negative sample bias are listed in Table 4.4 and depicted graphically in Fig. 4-7. The measurements have not been corrected for the systematic error discussed in [4.4 (b)] (the trends of the yields with increasingly negative sample bias are of interest, not their absolute magnitudes). Of the four surfaces, only measurements for D1 were performed under UHV; measurements for the other three surfaces were performed at $\sim 5 \times 10^{-9}$ Torr, where the major system contaminants were H$_2$O, CO, and CO$_2$ (see Appendix B for individual surface details). Examination of the data reveals the following:

1. Effects on UHV surface D1. Negative sample biasing has no measurable effect on the number of SE’s or BSE’s emitted from the UHV surface, D1 (to within precisions of 1% for both $\delta$ and $\eta$).
2. Effects on non-UHV surfaces $D_{10}$, $D_{11}$, and $D_{12}$. The non-UHV surfaces all exhibit significant (~10%-30%) increases in $\delta$ with increasingly negative sample bias; the values plateau in the bias range $-2 \text{ V}$ to $-4 \text{ V}$, beyond which they remain elevated but constant. In conjunction with their increasing SE yields, these surfaces also exhibit a small (~2%), simultaneous drop in their $\eta$'s; the effect is at the limit of the measurement precision, but is detected for all three surfaces.

4.7 (b) Analysis

Results for Surface D1—that negative sample bias (through $-20 \text{ V}$) has no discernable effect on $\delta$ or $\eta$—require little analysis; this result was expected. At $\Phi_s = -20 \text{ V}$, the electric field at the sample surface should be no greater than $\sim 500 \text{ V/m}$ (an order of magnitude estimate based on the geometry of the Chamber Apparatus); this is far less than the $\sim 10^5 \text{ V/m}$ generally required to effect field emission [Reimer, 1985] (p. 17), and no other obvious mechanism for affecting the total yields presents itself. Results for $D_{10}$, $D_{11}$, and $D_{12}$, however, are unexpected; the significant increase in the $\delta$'s of these surfaces with negative sample bias are not likely the result of field emission, and the fact that the increase occurs in the first few volts of sample biasing, leveling off afterward, is initially puzzling. But if field emission cannot be invoked to account for the observations, then what is the cause? A possible explanation is found in the

<table>
<thead>
<tr>
<th>Sample Bias (V)</th>
<th>$\delta$</th>
<th>$\eta$</th>
<th>$\delta$</th>
<th>$\eta$</th>
<th>$\delta$</th>
<th>$\eta$</th>
<th>$\delta$</th>
<th>$\eta$</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>1.01</td>
<td>0.63</td>
<td>0.88</td>
<td>0.62</td>
<td>0.94</td>
<td>0.71</td>
<td>0.93</td>
<td>0.67</td>
</tr>
<tr>
<td>-1</td>
<td>1.02</td>
<td>0.64</td>
<td>0.98</td>
<td>0.61</td>
<td>0.98</td>
<td>0.70</td>
<td>1.00</td>
<td>0.66</td>
</tr>
<tr>
<td>-2</td>
<td>1.02</td>
<td>0.63</td>
<td>1.08</td>
<td>0.61</td>
<td>1.01</td>
<td>0.70</td>
<td>1.02</td>
<td>0.66</td>
</tr>
<tr>
<td>-3</td>
<td>1.03</td>
<td>0.63</td>
<td>1.11</td>
<td>0.61</td>
<td>1.02</td>
<td>0.70</td>
<td>1.02</td>
<td>0.66</td>
</tr>
<tr>
<td>-4</td>
<td>1.03</td>
<td>0.63</td>
<td>1.13</td>
<td>0.61</td>
<td>1.04</td>
<td>0.70</td>
<td>1.01</td>
<td>0.66</td>
</tr>
<tr>
<td>-5</td>
<td>1.02</td>
<td>0.63</td>
<td>1.13</td>
<td>0.61</td>
<td>1.03</td>
<td>0.71</td>
<td>1.02</td>
<td>0.66</td>
</tr>
<tr>
<td>-10</td>
<td>1.03</td>
<td>0.63</td>
<td>1.11</td>
<td>0.62</td>
<td>1.04</td>
<td>0.71</td>
<td>1.02</td>
<td>0.66</td>
</tr>
<tr>
<td>-20</td>
<td>1.11</td>
<td>0.62</td>
<td>1.05</td>
<td>0.70</td>
<td>1.02</td>
<td>0.66</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Figure 4. \( \delta \) and \( \eta \) as functions of negative sample bias for four different surfaces. Measurements on non-UHV surfaces were accomplished at \(-5 \times 10^{-9}\) Torr. \( E_b = 1.5\) keV.

Importance of Work Functions

The work function, \( \phi \), for a uniform metal surface is defined to be the difference in potential energy between its vacuum and Fermi levels [Kittel, 1996]: \( \phi \equiv E_{\text{vac}} - E_F \). The Fermi level, \( E_F \), is the electrochemical energy of the electrons in the metal, which is the top of the conduction band (in this discussion it is assumed that all emitted electrons originate at the Fermi level). The vacuum level, \( E_{\text{vac}} \), is the minimum energy required to escape the surface—i.e., it is the lowest unbound energy state for an electron with respect to the metal. When one says that a sample is unbiased, one must specify with respect to what. In this investigation, we mean that the reference energy level—the Fermi level—is the same for the emitter (the Au sample) as for the absorbing surfaces (e.g., the vacuum chamber, detector, etc.). That is, we have tied (grounded) the Fermi levels of all of these objects together.

In order for an electron to go from its location in the conduction band of the emitter to a location in the conduction band in the absorber, it must overcome a potential barrier to leave the emitter (the
emitter's work function, \( \phi_e \), and overcome another potential barrier to enter the absorber emitter (the absorber's work function, \( \phi_a \)). There are two possible scenarios for the unbiased case: (i) the work function of the emitter is less than that of the absorber, or (ii) vice-versa. These scenarios are depicted schematically in Fig. 4-8. In Fig. 4-8 (a), where \( \phi_e < \phi_a \), it is easy to see that an electron may indeed escape the emitter and yet not possess enough energy to reach the absorber; in this case, the electron is forced to return either to the emitter, or to another surface with \( \phi \leq \phi_e \). (Since we are speaking of electrical potentials, the difference between the two vacuum levels results in an electric field between the emitter and absorber and, therefore, a force on the electron—returning it to the emitter.) In Fig. 4-8 (b), \( \phi_e > \phi_a \) and it is clear that any electron escaping the emitter automatically possesses enough energy to reach the absorber.

Negatively biasing a conductor amounts to relocating its Fermi level relative to some reference level—usually earth ground or, as in our case, the Fermi level of some other surface (the absorber). This scenario is depicted in Fig. 4-9; we have taken the case where \( \phi_e < \phi_a \) [Fig. 4-8 (a)] and biased the emitter negatively by an amount \( \Phi_s \)—where \( e\Phi_s \) is greater than the difference in the work functions of the emitter and absorber, \( \Delta\phi \). The result of this negative biasing is to raise both the Fermi and vacuum levels of the emitter above those of their respective counterparts in the absorber, producing a situation similar to that depicted in Fig. 4-8 (b). Thus, through negative sample biasing, it is possible to move from a situation in which not all of the emitted electrons are able to ultimately escape the sample (as a result of an absorber with a higher work function) to one in which all emitted electrons are absorbed by other surfaces; such a possibility brings up the notions of actual yields and effective yields.

**Actual Yield vs Effective Yield**

It has now become important to distinguish between the number of SE's initially leaving a surface with sufficient energy to escape—the *actual yield*—and the number of SE's which ultimately do escape—the *effective yield*. Clearly, given the situation depicted in Fig. 4-8(a), total SE yield measurements in this investigation will result in an effective yield determination; for the situation of Fig. 4-8(b), an actual yield will be obtained (neglecting the systematic error described in [4.4 (b)]. In this
FIGURE 4-8. Energy diagram depicting potential wells an escaping electron sees in its journey from an emitting surface to an absorbing surface when: (a) the emitter’s work function is less than the absorber’s, and (b) the emitter’s work function is greater than the absorber’s.
FIGURE 4-9. Energy diagram depicting the change in potential, due to negative biasing of the emitter by an amount $\Phi_s$, seen by an electron leaving the emitting surface and going to an absorbing surface. The gray line represents the potential of the system (as seen by the escaping electron) prior to negatively biasing the emitter, while the black line represents the potential after biasing.

Investigation (as in all SEE investigations, presumably) our purpose has been measurement of actual SE yields; however, given the scenario presented above it is clear that if total yield measurements are performed at zero sample bias only, there exists the possibility of mistaking an effective yield for an actual yield. Specifically, if the work function of the gold is less than those of most of the absorbing surfaces, then such will be the case. It is hypothesized that just this situation may be responsible for the yield-vs-bias measurements on surfaces D10, D11, and D12.

**Explaining the Data**

In the case of $\phi_e < \phi_a$, one can ask what would be the signature SE yield vs sample bias curve. Since work function differences among conducting surfaces are not likely to be more than a few eV, one would expect to see an increasing $\delta$ through the first few volts of negative biasing—as the vacuum levels of the emitter and absorber draw even—becoming constant with continued increase in negative sample bias (as the emitter's vacuum level surpasses that of the absorber). Also, the effect should be surface dependent, being more pronounced for emitters with lower work functions. Looking at Fig. 4-7, this is precisely the
signature exhibited for surfaces D10, D11, and D12; $\delta$'s increase with negative sample bias through $-2 \, \text{V}$ to $-4 \, \text{V}$, plateau, and remain constant, and the effect is notably more pronounced for surface D10—the unsputtered surface. Thus the observed yield vs. bias curves are generally consistent with the expected signature for $\phi_e < \phi_a$. But in order for this explanation to be correct, it must be that the surface D10, D11, and D12 work functions are lower than those of the absorbing surfaces; is it reasonable that this would be so?

Given the lack of in situ surface characterization capabilities of this instrument and no means of directly measuring the work functions of the surfaces, one can at best say that $\phi_e < \phi_a$ is plausible. At 5.3 eV, the work function for atomically clean gold is relatively high—higher than that for aluminum, aluminum oxide, or stainless steel [Hölzl and Schulte, 1979]. Surfaces D10, D11, and D12, however, are not atomically clean gold; D10 had been exposed to atmosphere and pumped down to $-5 \times 10^{-9} \, \text{Torr}$, with no bakeout. RGA measurements showed H$_2$O, CO, and CO$_2$ at $-1 \times 10^{-9} \, \text{Torr}$ in the system; thus these contaminant species were likely present on D10. Further, it is known that the presence of water vapor on a gold surface will lower its work function by $-0.5 \, \text{eV}$ (W. Hansen, personal communication, 1999); the presence of other contaminants could lower it even further. If this were the case, D10 should have the lowest work function of the four surfaces, followed by D11 (sputtered) and D12 (annealed); surface D1, having been baked out, further annealed, and finally sputtered should be the cleanest and should therefore have the highest work function. The data are consistent with this scenario: D10 shows the lowest effective yield at zero sample bias, followed by D11 and D12 (almost identical), and finally D1 with the highest. That D11 and D12 are not as high as D1 can be attributed to the fact that, although these surfaces were sputtered (D11) and annealed (D12), the system still had not been baked; contaminant levels, therefore, remained high. Finally, the behavior of the $\eta$'s is also consistent with the premise of differing work functions. Since BSE's do not originate in the sample, they should not be affected by an arbitrary setting of the sample’s Fermi level via biasing, which is essentially what is observed. (The slight drop in the $\eta$’s with negative sample bias may be accounted for by the role of the tertiary samples—see below.)
The δ, η, σ Mystery

From [4.4 (d)] recall that the δ, η, σ mystery is the observation that sputtering an atmosphere-exposed surface results in a decrease in δ and an increase in η in just such a way as to keep σ constant; this is a mystery because though it is expected that sputtering will change δ, it should not affect η. Differing work functions between the Au sample and tertiary samples, coupled with the systematic error discussed in [4.4 (b)], might account for this puzzling increase in η.

Recall that in order to affect an η determination in this investigation, the measured currents \( I_s(50) \) and/or \( I_r(50) \) must be affected [see Eq. (3.16)]. In [3.3 (d)] it was suggested that with both primary and tertiary samples biased to +50 V the tertiary samples might collect some of the SE’s emitted from the primary. Such an affect would indeed change \( I_s(50) \) and \( I_r(50) \), resulting in a systematic under counting of SE’s and over counting of BSE’s, and in [4.4 (b)] evidence that this is actually happening was presented. But closer examination of the process [Appendix A, Sec. A.2 (c)] showed that SE collection by the tertiaries could not account for the observed change in η with surface; without the systematic error η should be unchanged by sputtering, and with the systematic error, as it turns out, the sign of the change in η should be the same as for the change in δ—in conflict with the observation. However, it was also found that if the fraction of SE’s being collected by the tertiaries (\( γ \)) changes as well as the number of SE’s being produced (\( δ_{true} \)), the observations can be accounted for. Specifically, if an increase in γ accompanies a decrease in \( δ_{true} \), an increase in η will accompany a decrease in δ (see [A.2 (c)] in Appendix A). But what could be happening to increase γ?

Imagine the situation depicted in Fig. 4-10 (a), where the work functions for the Au and the tertiaries are the same; in this case, neither the primary or tertiary samples are favored energetically, and at +50 V bias some SE’s will be collected by each, depending on exit energy and trajectory. Contrasting this situation are the alternatives depicted in Figs. 4-10 (b) and (c): in these scenarios, either the primary or tertiary samples are energetically favored as a result of differing work functions. In Fig. 4-10 (b), where \( \phi_{Au} < \phi_{tertiary} \), the tertiaries are apt to collect fewer SE’s than in the case of equal work functions, lowering γ, and in Fig. 4-10 (c), where \( \phi_{Au} > \phi_{tertiary} \), the tertiaries are likely to collect more SE’s, increasing γ.
FIGURE 4-10. Energy diagrams depicting the cases of (a) $\phi_{Au} = \phi_{tertiaries}$, (b) $\phi_{Au} < \phi_{tertiaries}$, and (c) $\phi_{Au} > \phi_{tertiaries}$, where the Fermi energies of the Au sample and two tertiary samples are tied together.
And, in fact, it is not even necessary that $\phi_{Au}$ start out below or end up above $\phi_{\text{tertiary}}$, only that the pre-sputter $\phi_{Au}$ be less than the post sputter $\phi_{Au}$. Thus, if sputtering the Au surfaces acts to raise their work functions—as would appear to be the case, since $\delta$ is being lowered—then the $\delta, \eta, \sigma$ mystery can be accounted for.

4.7 (c) Conclusions

The $\delta$-vs-$\Phi_0$ data have turned out to be extremely valuable, lending considerable insight into the subtleties of SE measurement. Based on the data and analysis presented, the following conclusions are offered:

1. Yield-vs-bias curves can be adequately accounted for by invoking work function arguments—specifically, by considering differing work functions between the emitters and absorbers;

2. If total yield measurements are performed at zero sample bias only, there exists the possibility of mistaking an effective yield for an actual yield (for the measurement technique of this investigation);

3. Through negative sample biasing, one can obtain information regarding relative shifts of work function as a function of surface (e.g., sputtered, annealed, or atmosphere exposed);

4. If sputtering raises the work function of the sample, differing work functions between primary and tertiary samples—coupled with SE collection by the tertiaries under positive bias—can account for the significant changes observed in $\eta$ due to sputtering.

Having reviewed the principal findings pertaining to total SE and BSE yields, the focus of this report now turns to angle-resolved measurements.
CHAPTER 5
ANGLE-RESOLVED YIELD
MEASUREMENTS

Angle-resolved (AR) SE and BSE yield and spectra measurement has been the goal of this work since its inception nearly seven years ago (R. Davies, progress report to Phillips Laboratory, unpublished, 1992). Using the rotatable detector (RD) and measurement method described in Chap. 3, such measurements have now been performed for 1.5 keV electrons normally incident on polycrystalline gold. Results of the AR yield measurements are reported in this chapter; results of the AER yield measurements are presented in Chaps. 6 and 7. Diagnostic observations, discussed in detail in Appendix C, are summarized in [5.3 (b)].

5.1 The Challenge of AR SE Measurements

As charged particles, the trajectories of secondary and backscattered electrons are susceptible to ambient electric and magnetic fields once they have left the sample. Since the object of angle-resolved (AR) measurements is to characterize the SE and BSE populations as a function of emission angle, any deviation from this angle during the journey from the sample to the detector is detrimental: electrons that should be collected are deflected away from the detector, and electrons not meant for collection are deflected in. Accurate AR SE measurements are particularly difficult, as the magnitude of the deflection increases with decreasing electron energy. The extent to which an electron’s trajectory is affected depends on (i) the strength of the ambient fields, $\mathbf{E}$ and $\mathbf{B}$, (ii) the energy of the electron, $E_e$, and (iii) the distance over which the fields act on the electron—i.e., the distance between the sample and the detector, $d$. For a given sample-detector distance, then, the key to successful AR SE and BSE measurements is to minimize the strength of the ambient fields. Quantitative analysis of the field situation for this instrument is presented in Appendix A [A.4].
Reduction of ambient \( \mathbf{B} \) is straightforward; magnetic shielding surrounds the sample-detector assembly, and magnetic materials are excluded from the shielded region [3.1(c)]. From the analysis performed in [A.4], it is found that a transverse \( \mathbf{B} \) of \(-26\) mG is required to effect significant deflection of a 1 eV electron over the sample-detector distance, and \(-80\) mG is required for a 10 eV electron. (A deflection is considered significant if it is comparable to the size of the detector aperture—about 1.5 mm.) As mentioned in [3.1(c)], the ambient \( \mathbf{B} \) in the shielded region between the sample and detector was found to be \(-4\) mG and is therefore not expected to affect our measurements.

Reduction of ambient \( \mathbf{E} \) is more difficult; stray electric fields can result from differences in work functions between various components of the chamber apparatus [4.7 (b)], or from the differential accumulation of stray SE’s, BSE’s, and primary electrons on various surfaces within the chamber. Of particular concern is the charging of insulating oxide layers, likely present on a number of surfaces within the chamber apparatus—especially those made of aluminum. Again referring to the analysis in [A.4], it is found that a transverse \( \mathbf{E} \) of \(-1.6\) V/m is required to effect significant deflection of a 1-eV electron, while \(-16\) V/m is required for a 10-eV electron. Reviewing the geometry of the chamber apparatus, one finds that a potential difference of only one-tenth of a volt between some surfaces could produce an ambient \( \mathbf{E} \) with magnitude on the order of 2 V/m. Since it is difficult to assess the electric field environment inside the chamber apparatus while measurements are being performed, a number of diagnostic measurements were accomplished as a means of inferring the reliability of the AR observations (see Appendix C and [5.3 (b)] below). Based on these measurements, ambient electric fields due to charging are expected to affect SE’s with energies below \(-10\) eV.

5.2 Measurement Notes

Ideally, the purpose of AR yield measurements is to count SE’s and BSE’s as a function of emission angle. Inherent in these measurements as performed in this investigation, however, is the arbitrary definition of SE’s as those emitted electrons with energies less than 50 eV [3.3 (b)]. Thus, it is important to be clear about the terms AR SE yield and AR BSE yield as they are applied in this discussion:
as in Chap. 4, it is emphasized that these terms refer not to the angle-resolved SE and BSE populations, per say, but to the 0-50 eV and greater-than-50 eV populations, respectively. With the aid of AR spectra, we would like to get away from these definitions; still, there is something to be gained by defining and making the AR yield measurements in this manner. First, this is the manner in which angle-resolved measurements have been made by previous investigators [Reimer, 1993; Jonker, 1951]; comparisons can therefore be made between our results and those of others. Second, 0-50 eV yield measurements can serve as a check on the low-energy portion of the AR spectra measurements; that is, integrating the AR spectra through 50 eV should give the same results as the AR SE (0-50 eV) yield measurements [3.3 (b)].

AR yield measurements were accomplished for surfaces D2, D6, and D7; specific conditions under which the measurements were performed are summarized as follows:

1. **Basic operating parameters.** Unless otherwise specified, all measurements were accomplished under the following conditions:
   - **Base Pressure:** $< 7 \times 10^{-11}$ Torr
   - **Operating Pressure:** $\leq 1 \times 10^{-10}$ Torr
   - **Sample Temperature:** $27 \pm 2$ °C (room temp.)
   - **Incident Beam Energy:** 1500 eV $\pm$ 1 eV
   - **Incident Beam-Current Density:** $\sim 10^{-5}$ A/cm$^2$ ($I_B \sim 200$ nA, spread over a 1.5-mm beamspot)
   - **Incident Beam Angle:** Normal incidence

2. **Sample location.** Unlike the total yield measurements, all angle-resolved measurements were accomplished for a single location on the sample surface—the sample center—a consequence of instrument design [3.1(c)].

3. **Sample bias.** Two sets of AR yield measurements were performed on each surface, one at 0 V sample bias and one at −1.5 V sample bias. These biases were chosen as a balance between two competing considerations: (i) the desire to energize the very slowest SE's (in order to reduce their susceptibility to the deflecting effects of ambient fields), and (ii) the need to minimize the focusing effect of sample biasing (see [C.2] in Appendix C). Advantages and drawbacks of this technique are discussed in the analysis sections below. AR spectra measurements were performed for a sample bias of −1.5 V only.
5.3 Summary of Principal Findings

As with the total yield measurements in Chap. 4, a considerable volume of data and analysis are presented in this chapter (and in Appendix C). To better enable the reader to follow and interpret the data and detailed analysis below, principal results are now summarized. The summary is broken into two parts: AR yields, and instrument performance (diagnostics).

5.3 (a) AR Yield Results

Analyses of both the angle-resolved SE and BSE yields confirm that the instrument is performing well and that the sample is behaving as expected with respect to its SEE and backscattering characteristics. Specifically, it is found that

1. With respect to AR BSE yields:
   a. The chamber apparatus is properly aligned on the beam axis;
   b. The magnitudes and distributions of the $\eta(\alpha')$'s are consistent among the three surfaces, and unaffected by small negative sample biases;
   c. The magnitudes and distributions of the $\eta(\alpha')$'s are in excellent agreement with previous investigations; specifically, the total BSE yield calculated from the $\eta(\alpha')$'s is in complete agreement with that measured by Thomas and Pattinson, and the angular distribution is well-described by a screened Rutherford cross section;
   d. Quantitative analysis of the $\eta(\alpha')$'s has allowed for refinement in the systematic error estimates for $\delta$ and $\eta$.

2. With respect to AR SE yields:
   a. The angular distributions of the 0-50 eV populations as a whole are reasonably well described by a Lambert cosine law;
   b. The data do not exhibit the signature of a surface refraction effect, as suggested by Baroody [1950];
   c. Elevated $\delta(\alpha')$'s when the sample is biased to $-1.5$ V are the result of both energizing the SE's and focusing them toward the surface normal;
d. With the sample at 0 V, work function differences between the gold and other surfaces in the chamber are likely producing an electric field normal to the sample surface, focusing some SE's toward the surface normal.

None of the observed AR yield behaviors is surprising or unexpected, and there are no features of the data which are not reasonably well accounted for. Detailed analysis by which the above conclusions were reached is presented in [5.4] below.

5.3 (b) Instrument Performance

A detailed series of diagnostic measurements was performed concurrent with the AR yield and spectra measurements; they reveal important information regarding instrument performance and data quality in the various regions of the angle-energy measurement domain. Data and analysis are presented in detail in Appendix C; for convenience, a summary of the results is presented here.

1. Rotatable detector performance. SE production within the RD was detected, but is not significant relative to the sample signal; the RD's energy resolution was experimentally determined to be 0.5 eV ±0.0003E_B; surface charging was detected inside the RD, resulting in systematic errors in AR yield determinations on the order of 6-10% [C.3].

2. Sample biasing effects. Negative sample biasing has the desired effect of energizing the SE's (reducing the deflecting effects of ambient electric and magnetic fields), but the undesired effect of focusing SE's toward the surface normal—resulting in anomalously high SE collection at low \( \alpha \). For negative \( \Phi_s \) in the range 0-5 V, this focusing is significant for electron energies below \(-15\) eV; electrons with energies above \(-20\) eV are essentially unaffected.

3. Ambient field effects. Surfaces within the chamber apparatus are becoming electrostatically charged, stemming from the spray of SE's and BSE's within the CA when the beam is on the sample surface. The resulting electric field is found to be consistent between measurements for a given sample surface, and asymmetric about the beam axis. Electrons with energies below \(-10\) eV are being significantly affected, reducing SE collection at a given angle on the order of 10% (where the magnitude of the effect is greatest for \( \alpha > -55^\circ \)). BSE collection is unaffected by ambient fields.
4. Surface contamination effects. Surface contaminants affect all SE energies approximately equally, distinguishing the influence of surface contamination on SEE from that of ambient fields, which preferentially affect lower energies.

5. Validity of sample current as a measure of beam current. Changes in the sample current are an excellent indicator of proportionate changes in the beam current—a key concern, as the AR spectra measurement technique requires that this be so.

Synthesizing these results, we can make the following general statements: (i) there appear to be no significant adverse affects on the collection of electrons with energies $E_e > 15$ eV, and (ii) collection of electrons with energies $E_e < 15$ eV is adversely affected at some level by, charging within the RD, ambient electric fields, and negative sample biasing, with the strongest effects for electron energies below $\sim 5$ eV.

Based on these generalizations, the quality of measurements is found to be excellent in all but a very small region of the angle-energy domain investigated (see Fig. 5-1); specifically, measurements of the very lowest energy electrons at the very highest emission angles present a continuing challenge.

![Figure 5-1](image)

**Figure 5-1.** Schematic depicting quality of angle-resolved measurements as a function of emission angle and energy.
5.4 Measurement Procedure

Early in the instrument testing it was discovered that the 0 V RD signal was at a maximum upon placing the beam on sample center, and would gradually decrease over the next ~2 min, stabilizing at a value 6–10% below the initial reading (an effect attributed to charging within the chamber apparatus—see [C.3] in Appendix C). This effect resulted in a 10–15% difference between the initial $\delta(\alpha')$ measurement and the stable, 2-minute value; there was no effect on $\eta(\alpha')$ determinations. As a result, each AR yield is actually two sets of measurements—one performed as quickly as possible on placing the beam on the sample center, and one performed approximately two minutes later, when the RD signal had stabilized.

The specific procedure for accomplishing the AR yield measurements was:

1. **sample bias** – set as desired (generally 0 V, or −1.50 V negative)
2. **e-beam** – placed in large sample FC and beam current recorded
3. **e-beam** – moved to sample center and 0 V and −50 V RD currents immediately recorded
4. **e-beam** – left on sample center for ~ 2 min, after which 0 V and −50 V RD currents again recorded
5. **e-beam** – returned to large sample FC and beam current again recorded
6. **AR yields** – calculated as described in [3.2(b)].

All measurements in the above process were recorded manually. On resetting the RD bias from 0 V to −50 V, the RD current required about 10 sec to stabilize: each current measurement therefore required 10-15 sec to complete. Total beam time on the sample is a little over 2 min for each complete set of measurements (initial and 2-min yields). For nonzero sample bias, RD current measurements were not taken for RD biases of 0 V and −50 V, but rather for $\Phi_S$ and −50 V + $\Phi_S$ to compensate for the sample-biased-induced energizing of the SE’s. That is, we want to count the SE’s *emitted* with energies of 0-50 eV; for a negative sample bias, however, these electrons arrive at the RD with an energy $e\Phi_S$ additional to their emitted energy. Thus, for a sample bias of −1.5 V, for example, RD currents were recorded for RD biases of −1.50 V and −51.5 V in order to count the number of electrons with emitted energies of 0-50 eV.
5.5 Description of Results

Results for AR BSE yield measurements are presented in Fig. 5-2, while AR SE yield results are presented in Figs. 5-3 (a) and (b). The data were taken for surfaces D2, D6, and D7 at sample biases of 0 V and -1.5 V. Also included in all three figures are fits of the data to several angular distribution functions—to be discussed in the analysis below—and the results of Reimer, who has performed a Monte Carlo simulation specifically for backscattering of 1.5 keV electrons normally incident on gold [Reimer, 1993].

5.5 (a) Data Conditioning

For surfaces D6 and D7, the complete AR yield data sets were taken concurrently with the AR spectra sets presented in Chap. 6 (no spectra measurements were made for surface D2). As a result, each successive data set was taken on a surface with progressively more beam-exposure time—through a maximum of about 23 hr. Total yield measurements—taken before and after each AR data set—revealed a gradual reduction in the SE yield of about 10%, presumably due to surface contamination, over the full course of measurements for each surface; thus, each successive AR SE yield measurement is artificially diminished relative to the previous measurements. This poses a potential problem when comparing the results for a full range of angles. Examination of AR spectra for clean and contaminated surfaces, however, reveals that surface contamination of this nature reduces SE emission approximately equally for all emission energies through 50 eV (see [C.4] in Appendix C). As a result, the effect of AR yield reduction due to surface contamination can be approximately corrected for by scaling the AR SE yields to their respective total yields. For example, a δ(α') measurement taken at a time when δ is 5% lower than the initial δ is rescaled (up) by 5%. It is these scaled AR SE yield values which are plotted in Fig. 5-3.

5.5 (b) Trends in the Data

From the figures, a number of general characteristics regarding the AR yield data are evident:

1. Regarding the AR BSE yields at 0 V and -1.5 V sample bias:
   a. there is no significant difference between the $\Phi_S = 0$ V and $\Phi_S = -1.5$ V data;
Figure 5-2. $\eta(\alpha')$'s for surfaces D2, D6, and D7. Data for each surface represents that for both 0 V and -1.50 V sample biases (results did not vary significantly for the two cases). Random errors are no greater than the size of the data markers. Solid lines are fits to a screened Rutherford distribution [Eq. (5.1)] and dotted lines are fits to a fourth-order cosine distribution [Eq. (5.2)]. $E_a = 1.5$ keV, normal incidence. Angular accuracy is $\pm 1.5^\circ$. 
**Angle-Resolved SE Yields**

**Figure 5-3.** $\delta(\alpha')$'s for surfaces D2, D6, and D7 at (a) 0 V and (b) -1.5 V sample bias. Random errors are no greater than the size of the data markers; angular accuracy is $\pm 1.5^\circ$. Dotted lines are fits to (a) a cosine distribution [Eq. (5.3)] and (b) an offset cosine distribution [Eq. (5.4)]. A Baroody distribution [Eq. (5.5)] is presented in both figs. (dashed line). $E_B = 1.5$ keV, normal incidence.
b. there is excellent agreement (better than 5%) between the $\eta(\alpha')$'s of all three surfaces at all angles;

c. the $\eta(\alpha')$'s for $\alpha = \pm 17^\circ$ are in complete agreement (important in assessing beam alignment, discussed below);

d. qualitative agreement between the experimental results of this investigation and Reimer's Monte Carlo simulation is excellent;

e. quantitative agreement between the experimental data and the Monte Carlo simulation is not particularly good, with the experimental $\eta(\alpha')$'s anywhere from 10-40% higher than those of the model at most emission angles.

2. Regarding the AR SE yields at 0 V sample bias:

a. agreement between $\delta(\alpha')$'s for the three surfaces at the lower emission angles is relatively poor (~25%), improving significantly for $\alpha > -40^\circ$, (to ~10%);

b. the $\delta(\alpha')$'s for $\alpha = \pm 17^\circ$ are in relatively good agreement (~5%) for surfaces D2 and D7, noticeably less so (19%) for D6.

3. Regarding the AR SE yields at -1.5 V sample bias:

a. for lower emission angles, $\delta(\alpha')$'s for all three surfaces are significantly elevated (20-40%) as compared to the 0-V values; for $\alpha > -50^\circ$, however, $\delta(\alpha')$'s are essentially the same (to within ~5%) as the 0-V values;

b. agreement between $\delta(\alpha')$'s for surfaces D2 and D6 is excellent (within 5%) at all angles; $\delta(\alpha')$'s for surface D7 are significantly lower (~15%) than D2 and D6 at the lowest emission angles, but are in excellent agreement (less than 6%) with the other two surfaces for $\alpha > -20^\circ$;

c. the $\delta(\alpha')$'s for $\alpha = \pm 17^\circ$ are in good agreement (less than 5%) for all three surfaces.

Significance of the above results is now discussed. Since, when taken as single groups, the SE and BSE (0-50 eV and greater-than-50 eV) populations exhibit distinctly different behaviors, examination of the AR SE and BSE yield data will be accomplished separately.
5.6 AR BSE Yield Analysis

Two general aspects of the AR BSE yield data presented above should be examined: (i) the shape of the $\eta(\alpha')$ vs $\alpha$ curve for each surface—i.e., the angular distribution function $\eta(\alpha)$—and (ii) the magnitudes. Point (i) is addressed by fitting the data to specific functions and comparing these fits to theoretical predictions; point (ii) is addressed by integrating the fits over the full range of emission angles to obtain calculated values for the total yield and comparing these values to those measured directly.

5.6 (a) BSE Angular Distribution Functions

Displayed in Fig. 5-2, along with the $\eta(\alpha')$ data of this investigation and Reimer’s Monte Carlo simulation, are fits to two different distribution functions:

$$\eta(\alpha') = \frac{C}{(1 + 2\beta - \cos \alpha')^2}$$  \hspace{1cm} (5.1)

$$\eta(\alpha') = c_1 \cos^4 \alpha' + c_2 \cos^3 \alpha' + c_3 \cos^2 \alpha' + c_4 \cos \alpha' + c_5$$  \hspace{1cm} (5.2)

Eq. (5.1) is the screened Rutherford cross section discussed in [2.3 (c)] and Eq. (5.2) is simply a fourth-order cosine function. Brief examination of Fig. 5-2 confirms that both functions fit the data quite well; relative chi-squared values displayed in Table 5.1 show the Eq. (5.2) result to be the best (not surprising, given its five fitting parameters, compared to only two for Eq. (5.1)!). The obvious agreement of the data with the screened Rutherford distribution is particularly encouraging, as this theoretical formulation is intended to describe elastic scattering of electrons by solids and is often used to model electron backscattering measurements [Bishop, 1967; Reimer, 1993]. This being the case, that the fit is so good suggests that (i) the instrument is working properly, (ii) nothing overtly odd is occurring with the sample with respect to backscattering, and (iii) even though the $\eta(\alpha')$’s include electrons with energies as low as 50 eV, the bulk of the greater-than-50 eV population consists of higher energy, elastically scattered electrons. It is also important to note that the faster-than-cosine fall-off of the $\eta(\alpha')$’s is specifically predicted for gold—as compared with, say, aluminum—at low incident energies (1-5 keV) [Reimer, 1993]; this is a consequence of gold’s larger screening factor, $\beta$ (also, recall the constant $C$ in Eq. (5.1) includes the atomic number, $Z$).
TABLE 5.1. Results of AR BSE yield measurements for surfaces D2, D6, and D7 (results for all three surfaces were nearly identical) fit to two different angular distributions. Reduced $\chi^2$ analysis assumed $\sigma=0.05\eta(\alpha)$. Best fits and agreements with total yield measurements are highlighted in red. $E_B=1.5$ keV, normal incidence.

<table>
<thead>
<tr>
<th>Ang. Distr.</th>
<th>Parameters</th>
<th>$red\chi^2$</th>
<th>$\eta_{calc}$</th>
<th>$\eta_{meas}$</th>
<th>$\eta_{meas,(corr)}$</th>
<th>% Difference</th>
</tr>
</thead>
<tbody>
<tr>
<td>Screened</td>
<td>$k=0.057$</td>
<td>0.81</td>
<td>0.47</td>
<td></td>
<td></td>
<td>9%</td>
</tr>
<tr>
<td>Rutherford</td>
<td>$\beta=0.251$</td>
<td></td>
<td></td>
<td>0.68</td>
<td>0.43</td>
<td></td>
</tr>
<tr>
<td>[Eq. (5.1)]</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Fourth-order cosine</td>
<td>$c_1=0.884$</td>
<td>0.18</td>
<td>0.46</td>
<td></td>
<td></td>
<td>7%</td>
</tr>
<tr>
<td>[Eq. (5.2)]</td>
<td>$c_2=-1.17$</td>
<td></td>
<td></td>
<td>0.097</td>
<td>0.18</td>
<td></td>
</tr>
<tr>
<td></td>
<td>$c_3=0.420$</td>
<td></td>
<td></td>
<td>0.097</td>
<td>0.18</td>
<td></td>
</tr>
<tr>
<td></td>
<td>$c_4=0.097$</td>
<td></td>
<td></td>
<td>0.097</td>
<td>0.18</td>
<td></td>
</tr>
<tr>
<td></td>
<td>$c_5=0.0001$</td>
<td></td>
<td></td>
<td>0.097</td>
<td>0.18</td>
<td></td>
</tr>
</tbody>
</table>

*Corrected for systematic error as discussed in [5.4 (c)].

That the fourth-order cosine distribution fits the data so well is fortuitous; it was found somewhat intuitively and rather quickly, and has no physical basis that I am aware of for describing this system. It is discussed here only because it serves as a slightly more accurate $\eta(\alpha)$ for the purposes of integration a la Eq. (3.17).

5.6 (b) Incident Beam Alignment

That the $\eta(\alpha')$'s at $\alpha=\pm17^\circ$ are in such good agreement is significant from the point of view of incident beam alignment. The angular distributions of BSE's are much more sensitive to incident angle and energy than those of SE's [Reimer, 1993; Darlinski, 1981; Tesar and Eckertora, 1973; Gruzin et al., 1972]; specifically, significant deviation ($>\sim10^\circ$) of the incident beam from the surface normal should result in a detectable asymmetry in the AR BSE yields about the beam axis. The absence of any asymmetry in the observed yields is thus a good indicator that the incident beam in this investigation was properly aligned on the surface normal—a concern, as alignment of the chamber apparatus with the defined zero (the beam axis) was done visually during instrument assembly. If the chamber apparatus had been aligned, say, $5^\circ$ off, then all of the RD positions ($\varphi_{RD}$) would have been off by $5^\circ$, skewing the measured angular distribution—a fact that may prove important in interpreting the AR SE yield data.
5.6 (c) \( \eta(\alpha') \) Magnitudes

Examination of the \( \eta(\alpha') \)'s with respect to magnitude is based on (i) comparison with Reimer's Monte Carlo simulation, and (ii) the total yield obtained via integration of the AR yields. As noted in [5.4 (b)] above, quantitative agreement with Reimer's simulation is poor. Thus, the question is whether the disagreement is due to some systematic error in the measurements, or due to a failing of the model used in Reimer's calculation. Integration of the AR data for comparison with the measured total BSE yields can help with this question. This is accomplished via Eq. (3.17):

\[
\eta_{calc} = \int_{0}^{\frac{\pi}{2}} \eta(\alpha) \sin \alpha d\alpha = 2\pi \int_{0}^{\frac{\pi}{2}} \eta(\alpha) \sin \alpha d\alpha \tag{3.17}
\]

where \( \eta(\alpha) \) is given by the fit to Eq. (5.2), presented in Table 5.1. The result is

\[ \eta_{calc} = 0.46 \]

—in poor agreement (~30% low) with the average measured value of 0.67 for surfaces D2, D6, and D7, but in perfect agreement with the value of 0.46 obtained by Thomas and Pattinson [4.4 (b)]. Performing the same calculation for the fit to Reimer's AR data gives

\[ \eta_{calc}^{(Reimer)} = 0.36 \]

—in much worse agreement (~22% low) with the Thomas and Pattinson measurement. These results are summarized in Table 5.2.

Two pieces of evidence support the notion that \( \eta_{calc} = 0.46 \) is an accurate calculation and in fact the actual BSE yield of the gold surfaces under investigation. First, the AR diagnostic measurements detailed in Appendix C give every reason to believe that any systematic error in the \( \eta(\alpha') \)'s is small, due only to a slight misalignment of the RD. Second, agreement between \( \eta_{calc} \) and the Thomas and Pattinson measurement is excellent—where reasons for confidence in the Thomas and Pattinson results have already been addressed [4.4 (b)]. Additionally, such strong agreement between this investigation and that of Thomas and Pattinson lends credibility to both, suggesting that the Reimer calculation, while qualitatively successful, systematically underestimates the \( \eta(\alpha') \)'s. Reimer's model calculates screened Rutherford (Mott) crossections for both elastic and inelastic scattering processes, and then calculates a mean free
TABLE 5.2. Comparison of measured and calculated AR BSE yields.

<table>
<thead>
<tr>
<th>Present Work</th>
<th>Reimer</th>
<th>Thomas and Pattinson</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\eta_{\text{meas}}$: 0.68</td>
<td>$\eta_{\text{calc}}$: 0.46</td>
<td>$\eta_{\text{meas}}$: 0.46</td>
</tr>
<tr>
<td>$\eta_{\text{calc}}$: 0.46</td>
<td>$\eta_{\text{calc}}$: 0.36</td>
<td>$\eta_{\text{meas}}$: 0.46</td>
</tr>
</tbody>
</table>

path between scattering processes. FORTRAN programs based on these processes have been published by Reimer and Stelter [1986] and Joy [1987]. An examination of the source of the error in Reimer’s model would be an interesting and appropriate topic for future investigation.

5.6 (d) Systematic Error in Total Yields: Estimate Refinement

Agreement of $\eta_{\text{calc}}$ with the Thomas and Pattinson results allows for refinement of the systematic error estimates presented in [4.4 (b)]. Specifically, since AR measurements do not rely on tertiary biasing, SE collection by the tertiaries does not affect the $\eta(\alpha')$ measurements; the $\eta_{\text{calc}}$ calculation, therefore, avoids this particular systematic error [3.3]. Given the above strong evidence that the actual total BSE yield ($\eta_{\text{true}}$) for surfaces D2, D6, and D7 is $\sim$0.46, we can use the analysis of [A.1] in Appendix A to refine the earlier estimates of systematic errors in the total yield measurements. Using total yield data set D6.01a—typical of the surface D2, D6, and D7 total yield sets—the results are $-18\%$ and $+47\%$ for $\delta_{\text{meas}}$ and $\eta_{\text{meas}}$, respectively.

5.7 AR SE Yield Analysis

Paralleling the above $\eta(\alpha')$ analysis, we wish to examine both the distribution (shape) and magnitudes of the AR SE yields. The data are fit to specific distribution functions, examined with respect to theoretical predictions, and integrated over the full range of emission angles to obtain values for $\delta_{\text{calc}}$. Analyzing the $\delta(\alpha')$ data, we have the benefit of the $\eta(\alpha')$ analysis; namely, the knowledge that the Chamber Apparatus is aligned properly on the beam axis, and that the high-energy AR yields are making
sense (distribution and magnitudes). The challenge in the $\delta(\alpha')$ analysis is that the lower-energy SE’s are being significantly affected by ambient fields [5.3 (b)].

5.7 (a) SE Angular Distribution Functions

Recall from that theoretical SE formulations have predicted the angular distribution for true SE’s to be a simple Lambert cosine law [2.3 (a)], or perhaps a Lambert cosine law modified to account for refraction at the surface. Also recall that past measurements for 0-50 eV populations, though not numerous, have found only minor deviations from a Lambert cosine law [Jahrreiss and Oppel, 1971; Oppel and Jahrreiss, 1972; Jonker, 1951]. In this investigation there are actually three different angular distribution functions that are of interest:

\[
\begin{align*}
\delta(\alpha) &= \delta(0) \cos \alpha \\
\delta(\alpha) &= \delta(0) \cos(\alpha + \alpha_0) \\
\delta(\alpha) &= \delta(0) \cos \alpha[1 + 0.28 \sin^2 \alpha + 0.14 \sin^4 \alpha]
\end{align*}
\]

where Eq. (5.3) is the Lambert cosine law and Eq. (5.5) is an approximation of Baroody’s law [Eq. (2.20)], accounting for surface refraction. We will term Eq. (5.4) an offset cosine distribution, allowing for the cosine distribution to be centered about some angle $\alpha_0$ offset from the normal.

Both the $\Phi_N = 0$ V and $\Phi_N = -1.5$ V $\delta(\alpha')$ data have been fit to all three of the above distributions; results are presented in Table 5.3. [Note: the manner in which the $\chi^2$ values were calculated does not allow for meaningful comparison between the $\Phi_N = 0$ V and $\Phi_N = -1.5$ V trials for each surface.]

In agreement with the findings of previous investigators, the data are well described by the cosine distributions (5.3) and (5.4). The best fits are depicted along with the data in Fig. 5-3; the Baroody fit [Eq. (5.5)] to surface D2 is also shown, to illustrate its relatively poor success as compared to the other two.

That the $\Phi_N = -1.5$ V data are best described by the offset cosine distribution may be related to asymmetric focusing of the lower-energy SE’s toward the surface normal, induced by the negative sample bias. The relatively poor agreement between the $\pm 17^\circ$ $\delta(\alpha')$’s for surface D6 at $\Phi_N = 0$ V and for surface D2 at $\Phi_N = -1.5$ V may also result from asymmetric fields: that similar variability was not observed in the $\eta(\alpha')$
TABLE 5.3 Results of AR SE yield measurements for surfaces D2, D6, and D7 fit to three different angular distributions. Reduced $\chi^2$ analysis assumed $\sigma=0.18\sigma(\alpha)$. Best fits and agreements with total yield measurements are highlighted in red. $E_{\text{Ar}}=1.5$ keV, normal incidence.

<table>
<thead>
<tr>
<th>Surface</th>
<th>Angular Distribution</th>
<th>Parameters</th>
<th>$\text{red } \chi^2$</th>
<th>$\delta_{\text{calc}}$</th>
<th>$\delta_{\text{meas}}$</th>
<th>$\delta_{\text{meas (corrected)}}$</th>
<th>% Difference</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>$\delta(0)=0.272$</td>
<td>0.36</td>
<td>0.85</td>
<td>1.02</td>
<td>1.24</td>
<td>31%</td>
</tr>
<tr>
<td></td>
<td></td>
<td>$\delta(0)=0.278; \alpha_0=2.2^\circ$</td>
<td>0.21</td>
<td>0.87</td>
<td>0.77</td>
<td>0.77</td>
<td>30%</td>
</tr>
<tr>
<td></td>
<td></td>
<td>$\delta(0)=0.246$</td>
<td>3.27</td>
<td></td>
<td></td>
<td></td>
<td>38%</td>
</tr>
<tr>
<td>D2</td>
<td>cosine [Eq. (5.3)]</td>
<td>$\delta(0)=0.244$</td>
<td>1.5</td>
<td>0.77</td>
<td>0.78</td>
<td>0.99</td>
<td>36%</td>
</tr>
<tr>
<td></td>
<td>offset cosine [Eq. (5.4)]</td>
<td>$\delta(0)=0.248; \alpha_0=1.1^\circ$</td>
<td>0.90</td>
<td>0.78</td>
<td>0.68</td>
<td>0.68</td>
<td>36%</td>
</tr>
<tr>
<td></td>
<td>Baroody [Eq. (5.5)]</td>
<td>$\delta(0)=0.217$</td>
<td>4.8</td>
<td></td>
<td></td>
<td></td>
<td>44%</td>
</tr>
<tr>
<td></td>
<td></td>
<td>$\delta(0)=0.233$</td>
<td>0.18</td>
<td>0.73</td>
<td>0.73</td>
<td>0.96</td>
<td>38%</td>
</tr>
<tr>
<td></td>
<td></td>
<td>$\delta(0)=0.233; \alpha_0=-0.4^\circ$</td>
<td>0.2</td>
<td>0.73</td>
<td>0.66</td>
<td>0.66</td>
<td>44%</td>
</tr>
<tr>
<td></td>
<td></td>
<td>$\delta(0)=0.209$</td>
<td>0.8</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>D6</td>
<td>cosine [Eq. (5.3)]</td>
<td>$\delta(0)=0.315$</td>
<td>3.4</td>
<td>0.99</td>
<td>1.02</td>
<td>1.24</td>
<td>20%</td>
</tr>
<tr>
<td></td>
<td>offset cosine [Eq. (5.4)]</td>
<td>$\delta(0)=0.338; \alpha_0=6.2^\circ$</td>
<td>0.67</td>
<td>1.06</td>
<td>0.89</td>
<td>0.99</td>
<td>15%</td>
</tr>
<tr>
<td></td>
<td>Baroody [Eq. (5.5)]</td>
<td>$\delta(0)=0.283$</td>
<td>9.3</td>
<td></td>
<td></td>
<td></td>
<td>28%</td>
</tr>
<tr>
<td></td>
<td></td>
<td>$\delta(0)=0.311$</td>
<td>7.6</td>
<td>0.98</td>
<td>0.98</td>
<td>0.98</td>
<td>19%</td>
</tr>
<tr>
<td></td>
<td></td>
<td>$\delta(0)=0.352; \alpha_0=8.6^\circ$</td>
<td>1.7</td>
<td>1.11</td>
<td>0.85</td>
<td>0.99</td>
<td>8%</td>
</tr>
<tr>
<td></td>
<td></td>
<td>$\delta(0)=0.272$</td>
<td>14</td>
<td></td>
<td></td>
<td></td>
<td>30%</td>
</tr>
<tr>
<td>D7</td>
<td>cosine [Eq. (5.3)]</td>
<td>$\delta(0)=0.289$</td>
<td>0.64</td>
<td>0.91</td>
<td>0.96</td>
<td>0.96</td>
<td>22%</td>
</tr>
<tr>
<td></td>
<td>offset cosine [Eq. (5.4)]</td>
<td>$\delta(0)=0.304; \alpha_0=4.5^\circ$</td>
<td>0.30</td>
<td>0.96</td>
<td>0.96</td>
<td>0.96</td>
<td>18%</td>
</tr>
<tr>
<td></td>
<td>Baroody [Eq. (5.5)]</td>
<td>$\delta(0)=0.261$</td>
<td>2.3</td>
<td></td>
<td></td>
<td></td>
<td>30%</td>
</tr>
</tbody>
</table>
data simultaneously suggests that it is not a matter of improper beam alignment, and it is an effect specific
to lower energy electrons. Asymmetric fields, then, become a primary suspect. Azimuthally asymmetric
SE production is also a possibility, though as mentioned in [3.1 (c)] there is good reason to suspect that
such is not the case with the samples of this investigation.

5.7 (b) δ(α') Magnitudes

Examination of the δ(α')’s with respect to magnitude is based on (i) comparison of δ\textsubscript{calc} with
δ\textsubscript{meas} for all three surfaces, (ii) comparison of the individual δ(α')’s for the two different cases of sample
bias, and (iii) comparison of the δ(α')’s between the three surfaces.

δ\textsubscript{calc} vs. δ\textsubscript{meas}.

Regarding (i), calculated total yields are obtained via Eq. (3.10):

\[
\delta_{\text{calc}} = \int_{\text{half-sphere}} \delta(\alpha') d\Omega = 2\pi \int_{0}^{\pi/2} \delta(\alpha') \sin \alpha d\alpha
\]

where \(\delta(\alpha)\) is the distribution function best describing the data, as determined in the previous section.

Results δ\textsubscript{calc} determinations for both the \(\Phi_s = 0\ V\) and \(\Phi_s = -1.5\ V\) distributions are presented in Table 5.3,
along with comparisons to the measured total yields for all three surfaces. [Note: both the actual δ\textsubscript{meas} and
that corrected for the systematic error discussed in [5.4 (c)], δ\textsubscript{meas(corrected)}, are listed in the table; it is
the corrected value for which comparison against δ\textsubscript{calc} should be meaningful.] At 0 V sample bias, it is
seen that δ\textsubscript{calc} for each of the three surfaces is consistently 30-40% below the measured, corrected SE
yield. At -1.5 V sample bias, the disparity between δ\textsubscript{calc} and δ\textsubscript{meas(corrected)} drops to 10-20%. These
results are readily understandable as a consequence of ambient field effects on the trajectories of low
energy SE’s: a significant number of SE’s that would otherwise be collected by the RD are being deflected
away from the aperture, resulting in an undercounting at each emission angle. Better agreement between
δ\textsubscript{calc} and δ\textsubscript{meas(corrected)} when \(\Phi_s = -1.5\ V\) is likely the result of two effects: the slight energizing of the
SE’s, making them less susceptible to deflection, and focusing of the SE’s toward the normal.
\[ \delta(\alpha') \text{ as a Function of Sample Bias} \]

Comparing the AR SE yields of each surface for the two separate cases of sample bias, we noted in [5.4 (b)] that \( \delta(\alpha') \)'s for \( \Phi_3 = -1.5 \) V are elevated at lower \( \alpha' \)'s, while for the mid- to high \( \alpha' \)'s the values remain essentially unchanged from their \( \Phi_3 = 0 \) V readings. This effect is also at least partially understood through negative-sample-bias induced focusing, as such focusing will increase SE collection at lower emission angles. But collection at higher angles should also decrease, and this is not observed. It is possible, however, that decreased collection at higher \( \alpha' \)'s is being offset by an increased collection due to the energizing of the SE’s by the negative bias.

\[ \delta(\alpha') \text{ as a Function of Surface} \]

Looking at the relative magnitudes of the \( \delta(\alpha') \)'s between the three surfaces, we would like to understand why there is substantial disagreement for \( \alpha < \sim 40^\circ \), while the values for \( \alpha > \sim 40^\circ \) are in much better accord. First it is noted that the low-angle disagreement follows the trend of the measured total yields; \( i.e. \), surface D2 has the highest \( \delta \) [4.4 (b)] and the highest \( \delta(\alpha') \)'s, while surface D7 has the lowest \( \delta \) and the lowest \( \delta(\alpha') \)'s (for \( \alpha < 40^\circ \)). This pattern is generally consistent with the notion of work function differences introduced in [4.7 (b)]; if the work function of the gold is higher than all other surfaces, it effectively gives the gold a negative bias relative to those surfaces. In this case one might expect the total SE yield to be somewhat elevated, and the resulting electric field may produce some focusing toward the surface normal.

5.8 Conclusions

It is noted that considerably more analysis could be accomplished on the AR yield data than has thus far been completed; the \( \eta(\alpha') \) data in particular lends itself to further comparison with theory. However, the primary purpose of the AR yield examination in this investigation has been to provide a backdrop for the AR spectra measurements. The above data and analysis are intended to establish the general good behavior of the instrument and sample, and to simultaneously punctuate the shortcomings of this type of data; this goal has been met. It appears that both the \( \eta(\alpha') \) and \( \delta(\alpha') \) data are well accounted
for both qualitatively and quantitatively, and are generally consistent with past observations. Specifically, angular distributions of the > 50 eV populations are well described by screened Rutherford crossections, the 0-50 eV populations are in reasonable agreement with a Lambert cosine law, the magnitudes of the $\eta(\alpha')$'s are consistent with the total yield measurement of Thomas and Pattinson, and the behavior of the $\delta(\alpha')$ magnitudes are in reasonable agreement with the total SE yield measurements. Behaviors of the $\delta(\alpha')$'s with changing sample bias can be understood based on diagnostic measurements characterizing the perturbing presence of an ambient electric field.

It is also clear that the data, while of good quality, are ill-suited to address even slightly more detailed questions regarding the nature of SEE and electron backscattering. For example, there are two theoretical formulations describing the angular distribution of SE's [2.3 (a)]: one predicts a Lambert cosine law exactly, while the other [Baroody, 1950] predicts a cosine law modified in a specific manner to account for refraction at the surface. Not a single set of AR SE yield data to date has confirmed either of these predictions; at least minor deviations from the cosine law are always noted. However, the theories are for true SE's, and AR SE yield measurements are always defined, as in this investigation, as the yields due to the < 50 eV population—a population not necessarily comprised entirely of true SE's. In attempting to evaluate the two theories, it clearly would be helpful to know if the angular distribution of the 0-10 eV population differs significantly from, say, the 40-50 eV population. Similarly, we also note that although the screened Rutherford crossection describes the > 50 eV population quite well, the description is not exact—nor should we expect it to be. Rather, one should reasonably expect 55 eV electrons to have undergone a considerably different experience than 1500 eV electrons, and this difference will likely be reflected in their angular distributions. But given only AR BSE yields (i.e., an integrated angular distribution) for the > 50 eV population, one will never observe these differences. Thus, while valuable, AR yield data as previously obtained will never be sufficient for detailed evaluation of fundamental SEE and backscattering theories. Sets of AR spectra, by contrast, are much better suited for this task.
CHAPTER 6

ANGLE-RESOLVED SPECTRA
MEASUREMENTS

A complete set of angle-energy-resolved yield measurements—i.e., AR spectra—represents the maximum amount of information one can obtain for a given SE and BSE population leaving a surface [Rössler and Brauer, 1981a] (neglecting such properties as spin, etc.). While the AR spectra sets described in this chapter are by no means complete, they represent the most information collected to date for a given population of SE's leaving a gold surface. Following a summary of the principal findings and measurement procedure, the data are described in detail. To facilitate a more organized discussion of this considerable volume of data, analysis is accomplished separately for the high- and low-energy portions of the spectra. Generally, they are examined for intensity, shape, behavior as a function of emission angle, and fine structure. Additional analysis—construction and examination of energy-resolved angular distributions from the AR spectra—is presented in Chap. 7.

6.1 Measurement Notes

Measurement of simultaneous angle- and energy-resolved (AER) yields for a large number of energies at a given emission angle constitutes an AR spectrum [3.3 (a)]. Figure 6-1 depicts a full AR spectrum (0–1500 eV) typical of this investigation. From the plot one gains a sense of scale: by far the single most numerous population of electrons emanating from the sample are low energy SE’s, while elastically scattered BSE’s comprise a distant second. [Note: In the discussion that follows, the terms “SE spectrum” and “BSE spectrum” are sometimes used to refer to the 0-50 eV and greater-than-50 eV portions of the spectra, respectively. It is once again emphasized that these terms are simply convenient definitions; in general, the emitted electron spectra should be considered to be comprised of both SE’s and BSE’s at all energies.] AR SE spectra were measured for surfaces D6 (17° < α < 76°) and D7 (17° < α < 55°); AR BSE spectra were measured for surface D6 only, for the scattering angles 17° < α < 55°. All of the
Angle-Resolved Energy Spectrum
Surface D6
$\alpha = 17^\circ$

**Figure 6-1.** Typical AR spectrum of this investigation. $E_0 = 1.5$ keV, normal incidence.
measurements detailed in this chapter are for 1500 eV electrons normally incident on polycrystalline gold and were performed concurrently with the AR yield measurements described in the previous chapter. All spectra were acquired at a sample bias of $-1.50 \, \text{V} \pm 0.001 \, \text{V}$. Basic operating parameters were as described in [5.2].

6.2 Summary of Principal Findings

Following presentation of the AR spectral data [6.4], analysis begun in this chapter is continued in Chap. 7. In keeping with the format of Chaps. 4 and 5, the following summary of results is presented; these results represent the principal findings of this investigation to date:

1. AR spectra measurements clearly indicate that the definitions of SE’s and SSE’s as those electrons with energies $E_e < 50 \, \text{eV}$ and $E_e > 50 \, \text{eV}$, respectively, are problematic: if one wishes to investigate SEE on a fundamental level, it appears necessary that these definitions be abandoned;

2. Also indicated in the AR spectra are various subpopulations among the SE’s and SSE’s, generally distinguished by the slopes of the spectra in different energy domains: this observation is interpreted as evidence that different mechanisms dominate SE and BSE production at different energies;

3. Analysis of energy-resolved angular distributions, constructed from the AR spectra, reveals that the angular distribution of the SE and BSE populations is far from uniform: transitions from one angular behavior to another occur at energies in general agreement with those identified as transition energies in the AR spectra, supporting the notion that different production mechanisms are responsible for the electrons in different energy domains.

6.3 Measurement Procedure

As accomplished in this investigation, obtaining an AR spectrum requires the measurement of integrated RD current vs detector bias [3.2(b)]: the RD is set to a negative bias, the current is recorded, the bias is stepped up to a new bias, the current is again recorded, and so on until data for the desired energy range has been collected. As comparisons are desired between total- and AR-yield values obtained via
direct measurement and those calculated from the AR spectra [3.2 (b)], direct $\delta$, $\eta$, $\delta(\alpha')$, and $\eta(\alpha')$ determinations are accomplished just prior to each AR spectrum. Since acquisition of a complete spectrum presently requires extended exposure of the sample to the incident beam (~90–180 min), total yield measurements are also made at the end of each spectrum for comparison to the pre-spectrum values (assessing the presence of any beam-induced surface modification). For the data presented in this dissertation, specific procedure for acquiring AR spectra was as follows:

1. *sample bias* – set as desired
2. *total yield measurements* – accomplished for the sample center and at least one other location
3. *AR yield measurements* – accomplished for $\Phi_z = 0$ V and $-1.5$ V
4. *RD bias* – stepped through a predetermined biasing profile while RD and sample current measurements were recorded for each RD bias
5. *data reduction accomplished*
6. *AR spectrum* – extracted via differentiation of the RD current vs bias curve
7. *total yield measurements* – again performed for the same two locations as those made just prior to the spectra measurement.

Detector biasing and data acquisition (DAQ) for AR spectra measurements are automated: the Keithley 237 power supply steps the RD bias through a preprogrammed profile while RD- and sample-current measurements are recorded by the computer [3.1(e)]. The standard RD biasing profile is presented in Appendix D (Table D.1); it employs 84 points for an AR SE spectrum, 0–50 eV, and 154 points for a complete AR SE and BSE spectrum, 0–1500 eV. This profile was chosen as a balance between maximizing resolution near the low-energy (SE) and elastic (BSE) peaks, and minimizing the time required to complete a spectrum. For each RD bias, the RD and sample currents are each sampled at 100 Hz over 50 sec—for a total of 5000 measurements per RD bias. Between RD biases data acquisition is suspended for 15 sec to allow the RD current to stabilize; thus, 65 sec are required for each RD bias. An AR SE spectrum therefore requires 91 min to complete, while a full AR spectrum (0–1500 eV) requires 167 min. Data
reduction primarily involves scaling the RD current measurements to the changing beam current, as
determined through sample current measurement; details of this procedure are presented in Appendix D.

6.4 Description of Results

Figure 6-2 depicts the same spectrum shown in Fig. 6-1, this time plotted on a logarithmic scale. Contrasting Fig. 6-1, the broad region between the low-energy and elastic peak does not appear featureless in Fig. 6-2; rather, two prominent features become evident—a long, low energy tail extending from \( \sim 50 \text{ eV} \) to \( 450 \text{ eV} \), and a broad, high-energy peak beginning at \( \sim 450 \text{ eV} \) and reaching a maximum at about \( 100 \text{ eV} \) below the elastic peak. Based on this preliminary inspection of the logarithmic plot, each AR spectrum can be conveniently broken into four principal features: (i) a low-energy peak in the \( 0-50 \text{ eV} \) range, generally regarded as the SE peak, (ii) a long, shallow, low-energy tail (LET) extending from the low-energy peak to \( \sim 450 \text{ eV} \), (iii) a broad, high-energy peak located to the left of the elastic peak, which we shall term the inelastic peak (IP), and (iv) an elastic peak centered on the incident beam energy. For the present it will be convenient to build our description and analysis of the AR spectra around the framework of these features. It is emphasized, however, that partitioning of the spectra in this manner is temporary; on completion of our analysis at the end of Chap. 7, a more detailed classification of the emitted electrons, based on emission energy, is offered.

In our description and analysis of the above four spectra features, a number of characteristic parameters will be useful:

- \( E_{\text{onset}} \): the energy at which a given peak begins its monotonic increase;
- \( E_{\text{peak}} \): the energy at which a peak’s maximum AER yield is reached;
- \( N_{\text{peak}} \): the maximum AER yield for the peak (per incident primary);
- \( W_{\text{FWHM}} \): the full-width at half-maximum, characterizing the width of the entire peak;
- \( W_{\text{lead}} \): the “half-width” at half-maximum—calculated from \( E_{\text{peak}} \) to one-half \( E_{\text{peak}} \) on the leading edge of the peak—characterizing the width of the peak’s leading edge;
- \( W_{\text{tail}} \): the “half-width” at half-maximum—calculated from \( E_{\text{peak}} \) to one-half \( E_{\text{peak}} \) on the trailing edge of the peak—characterizing the width of the peak’s tail.
Angle-Resolved Energy Spectrum
Surface D6
\( \alpha = 17^\circ \)
(Semi-Log Plot)

\textbf{AR Low-Energy Peak}

\textbf{AR Inelastic Peak}

\textbf{AR Elastic Peak}

\textbf{Figure 6-2.} Typical AR spectrum of this investigation, plotted on a semi-log scale. \( E_\theta = 1.5 \text{ keV}, \) normal incidence.
Results of the AR spectra measurements are now presented, organized into three categories: AR spectra sets, repeatability between surfaces, and fine structure.

6.4 (a) AR Spectra Sets

Figures 6-3 and 6-4 depict a set of ten AR spectra—measured for 1.5 keV electrons normally incident on surface D6—broken into high-energy (Fig. 6-3) and low-energy (Fig. 6-4) portions. This sequence of measurements required ~23 hr of beam time on the sample. Total yield measurements performed after each spectrum revealed some evolution of δ and η as a result of the extended beam exposure; specifically, δ decreased monotonically from an initial value of 0.97 to a final value of 0.86 (uncorrected), and η decreased monotonically from 0.66 to 0.58 (uncorrected). Analogous to the D6 AR SE spectra set, Fig. 6-5 depicts a similar sequence of measurements for surface D7. Unlike the D6 set, however, concurrent AR BSE spectra were not acquired, and observations were made for more scattering angles over a smaller range (eleven spectra over the range 17° < α < 53). This sequence of measurements required ~15 hr of beam time on the sample; total yield measurements accomplished at the beginning and end of the set revealed a monotonic decrease in δ from 0.95 to 0.89 (uncorrected), and a monotonic decrease in η from 0.68 to 0.63 (uncorrected). As displayed in the figures, the AR SE spectra have been shifted to the left by ~1.5 V to correct for the energizing effect of the sample bias. Distinguishing parameters of the low-energy peaks, inelastic peaks, and elastic peaks of the individual spectra are presented in Tables 6.1, 6.2, and 6.3, respectively. From the figures and tables one observes the following general characteristics of the AR spectra:

1. Regarding the low-energy peaks:
   a. AEI yield magnitudes (i.e., AR spectra intensities) decrease with increasing α for all energies, with the exception of α = 19° and 25° for $E_e < ~5$ eV on surface D7;
   b. $E_{\text{onset}}$ increases with increasing α;
   c. $E_{\text{peak}}$ increases with increasing α;
   d. there are no discernible trends in the peak widths $W_{\text{FWHM}}$, $W_{\text{lead}}$, or $W_{\text{tail}}$ as a function of α.
2. Regarding the inelastic peaks:
   a. AER yield magnitudes decrease with increasing $\alpha$ for all energies;
   b. the drop in the AER yields with increasing $\alpha$ becomes notably more pronounced for $E_e > \sim 1000$ eV;
   c. the onset energy is 450 eV ($\pm$50 eV) for all angles;
   d. $E_{\text{peak}}$ is $\sim 1275$ eV ($\pm$50 eV) for all angles;
   e. for $\alpha < 35^\circ$, there is a dip in the BSE spectra between 1460 eV and 1490 eV in which the AER yields are negative.

3. Regarding the elastic peaks:
   a. there is a double elastic peak with $\sim 5$ eV between the two peaks;
   b. AER yield magnitudes do not decrease with increasing $\alpha$ for all energies; rather, the $\eta(\alpha', E_e')$'s for the 53° peak are actually greater than those for the 46° peak;
   c. the onset energy is 1494 eV at all angles;
   d. peak widths for the elastic peaks are nearly identical at all angles at $\sim 2.2$ eV

6.4 (b) Repeatability Between Surfaces

AR BSE spectra for surface D7 were not acquired, thus a comparison of spectra between surfaces D6 and D7 for $E_e > 50$ eV will not be possible. However, common AR SE spectra were acquired for five of the emission angles investigated; data for these common angles are shown in the shaded regions in Table 6.1. Examination of this data leaves the impression that the common spectra are not in particularly good agreement. Inspection of Fig. 6-6, however, gives a much clearer picture; the figure shows that for $E_e > \sim 5$ eV, agreement between the two spectra sets is excellent at all emission angles. For $E_e < \sim 5$ eV, agreement is also excellent for $\alpha \geq 46^\circ$; for $\alpha \leq 35^\circ$, however, there is some disagreement regarding the peak locations and magnitudes. Specifically, $E_{\text{peak}}$ is 0.5-1.0 eV lower for surface D6, and $N_{\text{peak}}$ is $\sim 20$-30% higher. These data have been particularly useful as a diagnostic tool assessing instrument performance; analysis to this effect is presented in detail in Appendix C. Here, the comparisons will be used to assess low-energy curve fits presented in [6.6 (b)].
Angle-Resolved High-Energy Spectra
Surface D6 (Linear Plot)

FIGURE 6-3. Surface D6 AR BSE spectra plotted on (a) linear and (b) semilog scales. $E_B = 1.5$ keV, normal incidence.
FIGURE 6-3. Continued. Point A indicates the onset of a noticeable divergence of the 17 and 25 degree spectra from the other three.
Figure 6-4. Surface D6 AR low-energy spectra, adjusted for -1.5 V sample bias. $E_B = 1.5$ keV, normal incidence.
Angle-Resolved Low-Energy Spectra
Surface D7

Scattering Angles
- +17 deg
- +19 deg
- +25 deg
- +30 deg
- +35 deg
- +38 deg
- +41 deg
- +46 deg
- +50 deg
- +53 deg
- +55 deg

**FIGURE 6-5.** Surface D7 AR low-energy spectra, adjusted for -1.5 V sample bias. $E_B = 1.5$ keV, normal incidence.
TABLE 6.1. Summary of low-energy peak parameters for the AR SE spectra of surfaces D6 and D7. Angles measured for both surfaces are shaded. $E_{\text{el}} = 1.5$ keV, normal incidence. Uncertainties are ±0.1 eV.

**Surface D6**

<table>
<thead>
<tr>
<th>$\alpha$</th>
<th>$E_{\text{onset}}$ [eV]</th>
<th>$E_{\text{peak}}$ [eV]</th>
<th>$N_{\text{peak}}$ [eV$^{-1}$sr$^{-1} \times 10^{-2}$]</th>
<th>$W_{\text{FWHM}}$ [eV]</th>
<th>$W_{\text{lead}}$ [eV]</th>
<th>$W_{\text{tail}}$ [eV]</th>
</tr>
</thead>
<tbody>
<tr>
<td>17°</td>
<td>1.0</td>
<td>2.25</td>
<td>3.20</td>
<td>6.0</td>
<td>1.1</td>
<td>4.9</td>
</tr>
<tr>
<td>25°</td>
<td>0.9</td>
<td>2.75</td>
<td>2.96</td>
<td>6.5</td>
<td>1.7</td>
<td>4.8</td>
</tr>
<tr>
<td>35°</td>
<td>1.2</td>
<td>2.75</td>
<td>2.78</td>
<td>5.9</td>
<td>1.5</td>
<td>4.3</td>
</tr>
<tr>
<td>46°</td>
<td>1.4</td>
<td>3.50</td>
<td>1.97</td>
<td>6.0</td>
<td>1.8</td>
<td>4.2</td>
</tr>
<tr>
<td>53°</td>
<td>1.7</td>
<td>3.75</td>
<td>1.52</td>
<td>6.0</td>
<td>1.7</td>
<td>4.3</td>
</tr>
<tr>
<td>59°</td>
<td>2.1</td>
<td>3.75</td>
<td>1.14</td>
<td>5.5</td>
<td>1.3</td>
<td>4.3</td>
</tr>
<tr>
<td>65°</td>
<td>2.1</td>
<td>4.25</td>
<td>0.705</td>
<td>6.2</td>
<td>1.2</td>
<td>5.0</td>
</tr>
<tr>
<td>73°</td>
<td>2.6</td>
<td>5.75</td>
<td>0.347</td>
<td>6.9</td>
<td>1.9</td>
<td>5.0</td>
</tr>
<tr>
<td>75°</td>
<td>2.5</td>
<td>6.50</td>
<td>0.342</td>
<td>7.1</td>
<td>2.6</td>
<td>4.5</td>
</tr>
<tr>
<td>76°</td>
<td>2.5</td>
<td>6.10</td>
<td>0.413</td>
<td>5.8</td>
<td>2.1</td>
<td>3.7</td>
</tr>
</tbody>
</table>

**Surface D7**

<table>
<thead>
<tr>
<th>$\alpha$</th>
<th>$E_{\text{onset}}$ [eV]</th>
<th>$E_{\text{peak}}$ [eV]</th>
<th>$N_{\text{peak}}$ [eV$^{-1}$sr$^{-1} \times 10^{-2}$]</th>
<th>$W_{\text{FWHM}}$ [eV]</th>
<th>$W_{\text{lead}}$ [eV]</th>
<th>$W_{\text{tail}}$ [eV]</th>
</tr>
</thead>
<tbody>
<tr>
<td>17°</td>
<td>1.1</td>
<td>2.75</td>
<td>2.30</td>
<td>8.6</td>
<td>1.5</td>
<td>7.1</td>
</tr>
<tr>
<td>19°</td>
<td>1.1</td>
<td>3.75</td>
<td>2.28</td>
<td>8.4</td>
<td>2.4</td>
<td>6.0</td>
</tr>
<tr>
<td>25°</td>
<td>1.1</td>
<td>3.25</td>
<td>2.44</td>
<td>7.8</td>
<td>2.0</td>
<td>6.2</td>
</tr>
<tr>
<td>30°</td>
<td>0.9</td>
<td>3.00</td>
<td>2.40</td>
<td>7.3</td>
<td>1.5</td>
<td>5.7</td>
</tr>
<tr>
<td>35°</td>
<td>1.1</td>
<td>2.75</td>
<td>2.22</td>
<td>7.3</td>
<td>1.3</td>
<td>6.0</td>
</tr>
<tr>
<td>38°</td>
<td>1.2</td>
<td>3.00</td>
<td>2.07</td>
<td>7.2</td>
<td>1.4</td>
<td>5.8</td>
</tr>
<tr>
<td>41°</td>
<td>1.2</td>
<td>2.75</td>
<td>2.08</td>
<td>7.0</td>
<td>1.1</td>
<td>5.8</td>
</tr>
<tr>
<td>46°</td>
<td>1.3</td>
<td>3.00</td>
<td>1.97</td>
<td>6.3</td>
<td>1.2</td>
<td>5.1</td>
</tr>
<tr>
<td>50°</td>
<td>1.5</td>
<td>3.75</td>
<td>1.54</td>
<td>6.5</td>
<td>1.8</td>
<td>4.7</td>
</tr>
<tr>
<td>53°</td>
<td>1.6</td>
<td>3.50</td>
<td>1.46</td>
<td>6.3</td>
<td>1.4</td>
<td>4.9</td>
</tr>
<tr>
<td>55°</td>
<td>1.9</td>
<td>3.75</td>
<td>1.25</td>
<td>6.3</td>
<td>1.5</td>
<td>4.8</td>
</tr>
</tbody>
</table>
TABLE 6.2. Summary of inelastic peak parameters for the surface D6 AR BSE spectra. $E_{B}=1.5$ keV, normal incidence.

<table>
<thead>
<tr>
<th>$\alpha$</th>
<th>$E_{\text{onset}}$ [eV]</th>
<th>$E_{\text{peak}}$ [eV]</th>
<th>$N_{\text{peak}}$ [eV$^{-1}$sr$^{-1}$]</th>
</tr>
</thead>
<tbody>
<tr>
<td>17$^\circ$</td>
<td>450</td>
<td>1330</td>
<td>$4.00 \times 10^{-4}$</td>
</tr>
<tr>
<td>25$^\circ$</td>
<td>450</td>
<td>1330</td>
<td>$2.68 \times 10^{-4}$</td>
</tr>
<tr>
<td>35$^\circ$</td>
<td>450</td>
<td>1300</td>
<td>$1.40 \times 10^{-4}$</td>
</tr>
<tr>
<td>46$^\circ$</td>
<td>450</td>
<td>1225</td>
<td>$1.06 \times 10^{-4}$</td>
</tr>
<tr>
<td>53$^\circ$</td>
<td>450</td>
<td>1300</td>
<td>$9.73 \times 10^{-5}$</td>
</tr>
</tbody>
</table>

TABLE 6.3. Summary of elastic peak parameters for the surface D6 AR BSE spectra. $E_{B}=1.5$ keV, normal incidence.

<table>
<thead>
<tr>
<th>$\alpha$</th>
<th>$E_{\text{onset}}$ [eV]</th>
<th>$E_{\text{peak}}$ [eV]</th>
<th>$N_{\text{peak}}$ [eV$^{-1}$sr$^{-1}$]</th>
<th>$W_{\text{FWHM}}$ [eV]</th>
</tr>
</thead>
<tbody>
<tr>
<td>17$^\circ$</td>
<td>1494</td>
<td>1497</td>
<td>$1.83 \times 10^{-3}$</td>
<td>2.3</td>
</tr>
<tr>
<td>25$^\circ$</td>
<td>1494</td>
<td>1497</td>
<td>$1.04 \times 10^{-3}$</td>
<td>2.3</td>
</tr>
<tr>
<td>35$^\circ$</td>
<td>1494</td>
<td>1497</td>
<td>$4.92 \times 10^{-4}$</td>
<td>2.1</td>
</tr>
<tr>
<td>46$^\circ$</td>
<td>1494</td>
<td>1497</td>
<td>$2.65 \times 10^{-4}$</td>
<td>2.2</td>
</tr>
<tr>
<td>53$^\circ$</td>
<td>1494</td>
<td>1498</td>
<td>$1.80 \times 10^{-4}$</td>
<td>2.1</td>
</tr>
</tbody>
</table>
Repeatability Between Surfaces

FIGURE 6-6. Comparison of surface D6 and D7 AR SE spectra for selected emission angles. The data have been adjusted to compensate for the -1.5 V sample bias. $E_B = 1.5$ keV
6.4 (c) Fine Structure

Another of the principle uses for AR SE and BSE spectra is identification of angle-dependent fine structure. Specifically, the identification of (i) enhanced SE production—possibly as a result of bulk and surface plasmon decay—and (ii) plasmon loss peaks in the BSE spectra are of interest. The quality of the data obtained in this investigation is at the threshold of that required for fine-structure identification. Energy resolution is sufficient at all energies, but uncertainty in the data below ~10 eV on the low-energy side (due to ambient fields) and the complicating presence of a double peak on the high-energy side (due to a bi-energetic incident beam) are difficulties yet to be overcome. Nevertheless, indications of $\alpha$-dependent fine structure within the AR spectra are common, if not definitive.

Data suggesting the presence of fine structure embedded in the low-energy peaks, low-energy tails, and elastic peaks are depicted in Figs. 6-7, 6-8, and 6-9, respectively. In Fig. 6-7, all three of the spectra are actually three-spectra averages, where the error bars are $\pm 1$ standard deviations calculated from the averages. They show evidence for an angle-dependent shoulder at ~4.5 eV (after correcting for the −1.5 V sample bias). Figure 6-8 depicts evidence for additional $\alpha$-dependent fine structure in the surface D6 low-energy tails; specifically, there is a feature that develops at 30-40 eV in the range $53^\circ < \alpha < 65^\circ$, another feature at ~45 eV in the range $46^\circ < \alpha < 65^\circ$, and another at 80-100 eV for $17^\circ < \alpha < 46^\circ$. These features were not observed in the surface D7 spectra. On the high-energy side, Fig. 6-9 depicts possible energy loss peaks just below the surface D6 elastic peaks, consistently present at 1493 eV for all five angles observed.

6.4 (d) Discussion

Overall quality of the AR spectra is good: results are highly repeatable for the same surface and between surfaces, and the energy resolution in particular is excellent (see Appendix C). Reductions in both $d$ and $\eta$ indicate some degree of surface modification over the course of the spectra set measurements. As a result, intensities have been artificially reduced somewhat for successive spectra (increasing $\alpha$). The contrasting pictures of the spectra presented by Figs. 6-1 and 6-2 are particularly interesting. Specifically, the linear plot of Fig. 6-1 suggests a natural break in the low-energy portion of the spectrum in the 50-100
AR SE Spectra Fine Structure

**FIGURE 6-7.** Average AR SE spectra for three scattering angles on surface D7, depicting possible $\alpha$-dependent fine structure (adjusted for $-1.5$ V sample bias). Error bars represent random error only ($E_B = 1.5$ keV, normal incidence).
Figure 6-8. Log plot of surface D6 AR SE spectra set depicting evidence for $\alpha$-dependent fine structure in the low-energy peak tail (points A, B, and C). $E_B = 1.5$ keV, normal incidence; $\Phi_s = -1.5$ V
Elastic Peak Fine Structure
(Surface D7)

FIGURE 6-9. Possible fine structure in the surface D6 AR elastic peak spectra. Angles are the same as those in depicted in Fig. 6-3. $E_B = 1.5$ keV, $\Phi = -1.50$ V.
eV range, while the log plot of Fig. 6-2 shows a more natural transition between low energy and high energy at \( \sim 450 \) eV. These two views are explored in greater detail below, along with the other observed behaviors noted in this section. Since one generally expects fundamentally different behavior between SE's and elastically and inelastically scattered BSE's, the low-energy and high-energy portions of the spectra are examined separately.

### 6.5 High-Energy (> 50 eV) Spectra Analysis

Similar to the AR yield analysis, we wish to analyze the above AER yield data from the perspectives of the shapes of the AR spectra (i.e., the AER yield distributions) and their intensities. Additionally, the spectra are examined with respect to their behavior with changing \( \alpha \). We begin with some general analysis of the high-energy spectra as a whole, continue with the distinction between the low-energy tail and inelastic peak, and conclude with individual discussions on the elastic peaks, inelastic peaks, low-energy tails, and fine structure.

#### 6.5 (a) General Comments

**Distribution**

The shapes of the > 50 eV portions of the AR spectra are in excellent qualitative agreement with the results of previous investigators for angle-integrated (AI) spectra—as evidenced by a quick comparison of Fig. 6-2 with Fig. 2-5 in Chap. 2. That angle-resolved spectra are qualitatively similar to angle-integrated spectra should be expected, as theoretically one should be able to take a set of AR spectra, construct an angle-energy distribution function, and integrate over all emission angles to obtain an AI spectrum \( a la \) Eq. (3.8a).

**Intensity**

Quantitatively, it is more difficult to analyze the spectra intensities at every energy—i.e., the individual AER yield magnitudes; diagnostic measurements, however, give every reason to believe that the quality of the high-energy (> 50 eV) spectra is excellent at all energies (with the exception of the small
range where some of the AER yields are negative—see Fig. 6-3(a) and Appendix C); thus confidence in the quantitative accuracy of the spectra is high. As a simple check, the AR BSE spectra can be integrated over energy to obtain values for \( \eta(\alpha')_{\text{calc}} \) for comparison against the appropriate measured AR BSE yields:

\[
\eta(\alpha')_{\text{calc}} = \int_{50 \, \text{eV}}^{E_g} \sigma(\alpha', E_e) \, dE_e
\]

(3.14)

Results are presented in Table 6.4; they reveal excellent agreement (~4%) between the \( \eta(\alpha')_{\text{calc}} \) and \( \eta(\alpha')_{\text{meas}} \) values for all angles investigated—essentially confirming that the RD is behaving consistently.

A more interesting analysis of the AR spectra intensities is to calculate partial AR yields for the low-energy tail (50-450 eV), inelastic peaks (450-1450 eV), and elastic peaks (1487-1506 eV). This has been done, and the results used to calculate the percentage of the total AR BSE yield (50-1506 eV) comprised by each of the three subpopulations. These results are depicted in Fig. 6-10. The figure reveals two interesting facts: first, the elastic peak electrons comprise only a very small portion of the \( > 50 \, \text{eV} \) population; second, though weak trends are evident in each of the three populations with increasing \( \alpha \), the fractions they comprise are relatively uniform for all emission angles (through 53°): ~66-45% for the inelastic peak electrons, ~20-30% for the low-energy tail electrons, and ~1-5% for the elastic peak electrons. More than anything else, these results give us a picture of elastic backscattering as a relatively rare event and inelastic scattering as the dominant flavor of PE-surface interaction.

6.5 (b) Distinction Between the Low-Energy Tail and Inelastic Peak

Underlying the distinction between the low-energy tail and inelastic peak electrons are the differing behaviors of the two populations with increasing emission energy. Specifically, the LET intensity decreases monotonically with increasing \( E_e \), while the IP intensity increases monotonically. This behavior suggests that there is something fundamentally different about the two populations. It is hypothesized that this difference is primarily one of origin—namely, that the LET electrons are chiefly comprised of secondaries, while the IP electrons are (mostly) inelastically scattered primaries.
TABLE 6.4. Comparison of measured and calculated AR yields for surfaces D6 and D7. $E_0 = 1.5$ keV, normal incidence.

<table>
<thead>
<tr>
<th>Surface D6</th>
<th></th>
<th>Surface D7</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>$\alpha$ (°)</td>
<td>$\delta(\alpha)$ (eV$^{-1}$ sr$^{-1}$)</td>
<td>$\eta(\alpha)$ (eV$^{-1}$ sr$^{-1}$)</td>
<td>$\delta(\alpha)$ (eV$^{-1}$ sr$^{-1}$)</td>
</tr>
<tr>
<td>17°</td>
<td>0.325</td>
<td>0.301</td>
<td>7</td>
</tr>
<tr>
<td>25°</td>
<td>0.307</td>
<td>0.290</td>
<td>6</td>
</tr>
<tr>
<td>35°</td>
<td>0.266</td>
<td>0.251</td>
<td>6</td>
</tr>
<tr>
<td>46°</td>
<td>0.199</td>
<td>0.186</td>
<td>7</td>
</tr>
<tr>
<td>53°</td>
<td>0.150</td>
<td>0.143</td>
<td>5</td>
</tr>
<tr>
<td>59°</td>
<td>0.113</td>
<td>0.105</td>
<td>7</td>
</tr>
<tr>
<td>65°</td>
<td>0.078</td>
<td>0.072</td>
<td>8</td>
</tr>
<tr>
<td>73°</td>
<td>0.045</td>
<td>0.041</td>
<td>9</td>
</tr>
<tr>
<td>75°</td>
<td>0.042</td>
<td>0.039</td>
<td>7</td>
</tr>
<tr>
<td>76°</td>
<td>0.044</td>
<td>0.041</td>
<td>7</td>
</tr>
<tr>
<td>76°</td>
<td>0.044</td>
<td>0.041</td>
<td>7</td>
</tr>
</tbody>
</table>
SE's vs BSE's: Comparison with Theory

The question of precisely which emitted electrons are the true SE’s and which are the true BSE’s remains one of the most interesting in SEE investigations. Historically, the dividing line has been drawn at 50 eV [2.2 (a)]; there is little evidence in Fig. 6-2, however, that suggests a shift in the origin of the emitted population as one moves from $E_e < 50$ eV to $E_e > 50$ eV. Rather, a fundamental change in the spectrum’s behavior is not observed until 450 eV, where the falling intensity begins to rise. If one assumes that all of the electrons down to 50 eV are BSE’s, as present convention dictates, then according to Fig. 6-2 the scattering crosssection as a function of increasing energy loss, $\Delta E$, must exhibit the following behavior (where it is assumed that the elastic region extends down to $\sim$1485 eV): a relatively fast rise to maximum, followed by a long, monotonic decline through $\Delta E \equiv 1050$ eV ($E_e \equiv 450$ eV), followed by a monotonic rise for $\Delta E > 1050$ eV ($E_e < 50$ eV). (Where it has also been assumed that the fundamental nature of the scattering processes remains the same throughout the energy domain.) Such an observation is suitable for comparison to theory.

A relatively successful and detailed theory of inelastic electron-electron scattering has been developed by Gryzinski [1965]. For a single inelastic scattering event, Gryzinski found that, as a function
of $\Delta E$, the probability of transferring a given amount of energy scales with increasing $\Delta E$ as follows: a fast rise to maximum, followed by a shallow, monotonic decline. No further structure—e.g., a subsequent increase in the crosssection with increasing $\Delta E$—is predicted. Thus, based on our observations, the notion that the $>450$ eV electrons are (mostly) BSE’s is in good agreement with Gryzinski’s predictions. If, however, one also assumes that the 50-450 eV population is comprised of BSE’s, our observations and the theory are at odds qualitatively. If we insist on maintaining the premise that we are dealing with BSE’s, then either the theory is in error, or some relatively exotic scattering process—one that possesses a cross section that increases with increasing $\Delta E$—is at work. Certainly, either of these scenarios is possible. A much simpler solution, however, is to abandon the BSE assumption and instead populate the 50-450 eV region primarily with SE’s.

The notion that the LET electrons are primarily SE’s is consistent with Gryzinski’s theory. To see this, it is important to realize that the $\Delta E$ in the theory refers to energy gains as well as energy losses. That is, for some arbitrary frame of reference, the two particles in a two-particle interaction will, in general, have different kinetic energies; their relative magnitudes—i.e., which is greater and which is less—is purely a function of the reference frame, and one can always switch to a frame where these relative magnitudes are reversed. Thus, the theory predicts a scattering cross section as a function of $\Delta E$, regardless of whether $\Delta E$ is positive or negative. Seen in this light and assuming the 0-450 eV electrons to be primarily SE’s, one observes precisely the behavior in intensity predicted by theory: a relatively fast rise to maximum, followed by a monotonic decline.

**Angular Behavior**

If the above scenario is correct—that LET electrons are primarily SE’s and IP electrons are primarily BSE’s—one might reasonably expect that the angular distributions of the two populations differ. Since representation of the data as AR spectra is not particularly well suited for examination of angular distributions, this piece of analysis will be performed in Chap. 7, where a more suitable representation is presented.
6.5 (c) Elastic Peaks: Intensities, Structure, and Behavior as a Function of Emission Angle

For purposes of discussion, we consider the elastic peak region to be approximately 1485-1505 eV. (Note: precisely what constitutes elastic scattering is not often addressed in the literature: apparently, it is generally assumed that “elastic” means perfectly elastic. But are there occasions in which one should consider electrons of energies 0.95\(E_p\) elastically scattered? 0.90\(E_p\)? It may be that processes in which absolutely no energy lost and processes in which only very little energy is lost—5%, say—do not differ significantly, or it may be that such processes are fundamentally distinct. Further consideration of this question may prove worthwhile, though will not presented here.)

**Double Peaks**

The double elastic peak is the result of an approximately 5-V peak-to-peak ripple in the electron gun power supply, producing an essentially bi-energetic primary beam (see Sec. C.2 in Appendix C). The 1497 eV peak is the larger of the two (a result the specific shape of the power supply ripple); consequently, for convenience, remaining discussion on the elastic peak will refer to the 1497 peak only. As a practical matter, it is not likely that the bi-energetic beam is significantly affecting SE production, though the presence of a double peak complicates the process of identifying fine structure in the quasi-elastic region.

**Intensity: Comparison with Theory**

Regarding the elastic peak itself, that the peak locations, onset energies, and widths are the same for all angles is expected, as these parameters are completely dependent on the characteristics of the incident primaries—namely, their energy and distribution. Regarding intensity, Jablonski et al. [1993] have performed Monte Carlo calculations specifically for elastic backscattering of 1.5 keV electrons normally incident on polycrystalline gold for both relativistic and nonrelativistic cases (unfortunately, they do not state explicitly the energy range they consider elastic). They use their results to calculate an AR yield due to elastically backscattered electrons at \(\alpha = 25^\circ\), and obtain results of \(\eta(25^\circ)_{\text{elastic}} = -0.036 \ \text{sr}^{-1}\) (relativistic) and \(\eta(25^\circ)_{\text{elastic}} = -0.030 \ \text{sr}^{-1}\) (nonrelativistic). Since an AR BSE spectrum for \(\alpha = 25^\circ\) was obtained in this investigation (see Fig. 6-3), an approximate comparison can be made. Specifically,
calculating the area under the curve for this spectrum's elastic peak, taken as 1486-1506 eV, yields the result \( \eta(25^\circ)_{\text{elastic}} = 0.007 \)—about a factor of five below Jablonski's relativistic result, and a factor of four below his nonrelativistic result. However, it is not entirely clear what Jablonski considers an elastically scattered electron—i.e., are 0.95\(E_B\) electrons elastic? 0.90\(E_B\) electrons? 0.85\(E_B\) electrons? This distinction could account for the discrepancy between his result and that of the present work. For example, taking elastic electrons to be those of energy \(E_e \geq 0.90E_B\) (1350-1505 eV) results in a calculated AR elastic yield for the \(\alpha = 25^\circ\) spectrum of \(\eta(25^\circ)_{\text{elastic}} = 0.021\)—in much better agreement with Jablonski—and using \(E_e \geq 0.86E_B\) gives Jablonski’s relativistic result of \(\eta(25^\circ)_{\text{elastic}} = 0.036\). Of course, disagreement between Jablonski’s calculation and our experiment may also be due to inadequacies in Jablonski’s model; examination of this model is certainly warranted, though will not be attempted here.

**Angular Behavior: Comparison with Theory**

Behavior of the AR elastic peaks with changing \(\alpha\) also lends itself to comparison with theory. Jablonski [1991] has calculated an angular distribution for the elastic peak, normalized to the elastic emission at \(\alpha = 25^\circ\). His results are presented in Fig. 6-11, along with results of this investigation. Agreement is excellent. Particularly worth noting is the prediction by Jablonski of increasing elastic yield with increasing \(\alpha\) in the range \(45^\circ < \alpha < 65^\circ\)—in agreement with the behavior of the 46° and 53° data observed in the present work. Measurements at higher \(\alpha\)'s need to be accomplished for further confirmation of Jablonski’s predictions.

### 6.5 (d) Elastic Peak Fine Structure

The presence of both the double elastic peak and the region of negative AER yields complicates the identification and analysis of fine structure. Also somewhat limiting is the 1 eV resolution of the data (better-than-1 eV resolution is required for some features). Nevertheless, fine structure has been observed and meaningful analysis is possible.
Comparison Measured BSE Yields with Theory (Surface D6)

FIGURE 6-11. Angular distribution of elastic yield as computed by Jablonski [1991] and measured in the present work (Surface D6). $E_B = 1.5$ keV, normal incidence.
REELS Analysis

Fine structure in the elastic peak seen in Fig. 6-9 is consistent with the results of reflection electron energy loss spectra (REELS) measurements presented in Fig. 6-12 (accomplished at a separate facility on sample A [LB IV, p. 23]). Of interest in Fig. 6-12 are the peaks at 2009.7 eV ($\Delta E = -2.7$ eV) and 2005.3 eV ($\Delta E = -6.1$ eV), closely matching the results of Fig. 6-9, which show peaks at $-3$ eV and $-6$ eV relative to the 1497 eV elastic peak.

Possible Sources

One common source of fine structure such as that observed in Fig. 6-12 is energy loss due to plasmon damping. In the case of gold, however, the proximity of the d-band to the conduction band (the d-band is actually completely contained within the conduction band) complicates calculation of the bulk and surface plasmon energies; to date, no such values for these energies have been located in the literature, making it difficult to determine whether or not plasmon damping is a candidate for the loss peaks in Fig. 6-12 (and Fig. 6-9). In addition to plasmon excitations, there also exist single-particle excitation states from the d-band to the Fermi level at approximately 3 eV and 6 eV [Citrin et al., 1978]; these states agree well with the observed structure at $-3$ eV and $-6$ eV both in the REELS data and our surface D6 measurements. Identification of the specific mechanisms responsible for the observed fine structure may prove relevant to analysis of fine structure in the SE peaks (see 6.6 (d) below): preferential absorption of specific energy increments is in general a reversible process, giving rise to complementary decay processes capable of liberating specific quanta of energy (e.g., creation and annihilation of plasmons). Thus, depending on the mechanism involved, if an absorption peak is observed in the high-energy portion of a spectrum, one may be justified in looking for complimentary excitation peak at low energies.

6.5 (e) Inelastic Peaks

The data acquisition profile of the AR spectra presented above is not particularly well suited to investigation of the 450-1485 eV energy range since the data points in this region were taken at 100 eV
FIGURE 6-12. Reflected electron energy loss (REELS) spectra for sample A, performed for ultrasonically cleaned surface (upper curves) and sputtered surfaces (lower curves). $E_B = 2012$ eV.

Intervals (see Table D.1 in Appendix D)—thus any fine structure in the data within this region would not have been observed. Still, the data are sufficient for a few comments.

**Intensity as a Function of Energy**

Behavior of the IP intensity with emission energy was discussed in detail in [6.5 (b)]. Here it is simply reiterated that this behavior is completely consistent with present inelastic scattering theory [Gryzinski, 1965].

**Negative AER Yields**

One aspect of the AR spectra in this range that draws immediate attention is the observation of negative AER yields for $\alpha \leq 35^\circ$ in the region 1460-1490 eV. The negative AER yields are clearly an instrument related effect, since in order to obtain negative $N(\alpha',E')$'s the number of electrons being collected by the detector must actually be increasing as the negative bias on the detector is increased. Though the specific source of the systematic error has not yet been identified, one possible candidate is leakage current from the negatively biased detector. Diagnostic measurements to date, however, have been
unable to detect any leakage current to $10^{-14}$ A—the detection limit of the instrument—and examination of the data reveals that leakages of least $-6 \times 10^{-13}$ A would be required to account for the observations. Thus it appears that the difficulty is somewhat more exotic, defying all attempts at explanation to date.

**Angular Behavior**

The changing nature of the peak falloff with increasing $\alpha$ for $E_e > -950$ eV, noted in Fig. 6-3 (b), is an interesting feature of the data. Specifically, as one moves from 950 eV to 1350 eV (approximately), the falloff of the intensity with increasing $\alpha$ appears to increase dramatically, becoming more "elastic-like." This shift in angular behavior may indicate a transition at this energy between dominant interactions creating the IP electrons. As the data as presented in Fig. 6-3 (b) are not well suited to anything more than a cursory angular analysis, detailed examination of the angular distribution of the various energy subpopulations is deferred to Chap. 7.

6.5 (f) Low-Energy Tails

As with the inelastic peak regions, the AR spectra of this investigation were not geared toward detailed investigation of the 50-450 eV energy range. Most interesting in the data is the evidence of fine structure in the 80-100 eV range depicted in Fig. 6-8. If the LET electrons are indeed SE’s, as the above discussion regarding the origin of the LET electrons suggests, this fine structure may be a consequence of competing interactions. Rosier and Brauer [1981b] have derived angle- and energy-dependent cross sections for various SE production mechanisms for aluminum; qualitative comparison of their results with the spectra of this investigation reveals similarity between the fine structure signatures observed and those expected in the regions where transition occurs from one dominant interaction to another. Further investigation along this line is warranted and should begin with acquisition of data at a finer resolution.

6.6 Low-Energy ($< 50$ eV) Spectra Analysis

The diagnostic evaluation presented in Appendix C indicates that the quality of the low-energy AR spectra measurements are excellent for all but the very lowest energies ($E_e < -10$ eV), where ambient
field effects become measurable. In this section we will examine the intensities and shapes of the AR SE spectra, their behavior as a function of emission angle, the issue of where one logically draws the distinction between SE’s and BSE’s, and the SE peak fine structure.

6.6 (a) Low-Energy AR Spectra Intensities

As with the > 50 eV portion of the AR spectra, one quick assessment of the intensities comes from integrating the AR SE spectra over the range 0-50 eV to obtain calculated AR yields. This has been done and the results are in good agreement with the measured values (≤ ~10% for surface D6, and ≤ ~5% for surface D7—see Table 6.4). Analogous to Fig. 6-10, a comparison of the partial AR yields of the low-energy peak (0-50 eV)—i.e., the AR SE yields—with partial yields of the other features is presented in Fig. 6-13. In the figure, the partial yields are represented as a fraction of the total AR yield, $\sigma(\alpha')$, as a function of $\alpha$. The results show that the 0-50 eV population comprises by far the largest portion of the emitted electrons at a given angle, and that this portion increases with increasing $\alpha$, primarily at the expense of the LET (50-450 eV) population.

Variations in the low-energy peak intensities between the two surfaces (see Fig. 6-6) are attributed to the effect of ambient fields. The figure makes clear that the effect is restricted to energies less than ~5
eV; however, as the distribution is peaked at these energies, the resulting discrepancy can be significant. The apparent under counting of SE’s by the RD alluded to in [5.7 (b)] is now completely understandable: it is due primarily to an undercounting of the < 5 eV population as a result of ambient field effects.

6.6 (b) AR SE Spectra Behavior as a Function of Emission Angle: The Low-Energy AER Distribution Function

Rösler and Brauer [1981a] have developed a fundamental angle-energy SE distribution function for nearly-free-electron metals [Eq. (2.12)]; it is the only such distribution to be found in the literature. Evaluation of their expression for the specific case of gold, however, is beyond the scope of this work. In the absence of an AR energy distribution against which to compare the data, a simple expression can be derived from existing semi-empirical theory; specifically, Eq. (2.8)—the angle-integrated expression for the SE energy distribution formulated by Chung and Everhart [1974]—is a suitable starting place:

$$N(E_e) = \frac{k}{E_b} \frac{E_e}{(E_e + \phi)}$$

(2.8)

(where $k$ is a material constant and $\phi$ is the work function). Two quick adjustments can be made to this formulation to adapt it for use as an AR energy distribution. First, to account for the sample bias, as well as the effect of any work function difference between the sample and the detector, we add an offset term to the energy: $E_e \rightarrow E_e - E_{off}$. Second, since all previous evidence suggests that SE’s follow a cosine distribution, Eq. (6.1) can be scaled with a cosine. Thus we have

$$N(E_e) = \frac{k}{E_b} \frac{E_e - E_{off}}{(E_e - E_{off}) + \phi} \cos \alpha$$

(6.1)

Figure 6-14 depicts a fit of the $\alpha = 17^\circ$ spectrum from surface D7 to Eq. (6.1). The result is encouraging. In particular, the entire spectrum is well described by the function, though the fit is noticeably better for $E_e < \sim 20$ eV. The values of $E_{off} = 2.45$ eV and $\phi = 7.4$ eV are reasonable, as $E_{off}$ is meant to account for the $-1.5$ V sample bias and the work function of clean gold is $\sim 5$ eV. (The specific value of $k$ is unimportant to this discussion, as its purpose here is merely to serve as a proportionality...
Comparison with Theory

Surface D7: $\alpha = 17^\circ$

**FIGURE 6-14.** Fits of (a) Eq. (6.1) and (b) Eq. (6.2) to the surface D7, $\alpha = 17^\circ$, AR low-energy spectrum. $E_B = 1.5$ keV, normal incidence.
constant.) Using the values of $k$ and $\phi$ obtained from this fit, a set of spectra for all of the surface D7 spectra has been constructed with Eq. (6.1) and the results displayed in Fig. 6-15(b). The figure shows reasonable agreement between the calculated curves and the data, though there is room for improvement. Specifically, the intensities of the predicted spectra are too high at all energies, and the function does not exhibit the observed increase in $E_{\text{onset}}$ and $E_{\text{peak}}$ with increasing $\alpha$.

The model can be improved if we recall that the work function represents a minimum energy (above the Fermi energy) required by an SE to escape the surface, and then recognize that for an electron with momentum vector $\vec{p}$ at some angle $\alpha$ with respect to the surface normal, the minimum energy required for escape scales inversely with $\cos \alpha$; i.e., the electron sees an effective work function given by $\phi_{\text{eff}} = \phi/\cos \alpha$. Thus, we have

$$N(E_e) = \frac{k}{E_b} \frac{(E_e - E_{\text{off}}) \cos \alpha}{\left(\frac{E_e - E_{\text{off}}}{\cos \alpha} + \frac{\phi}{\cos \alpha}\right)^2}$$

This formulation does not produce a shift in $E_{\text{onset}}$, though it does produce a shift in $E_{\text{peak}}$, as well as lower the predicted magnitudes. Following the same procedure as that used for Eq. (6.1), the $\alpha = 17^\circ$ data were fit to Eq. (6.2) and the fit parameters used to create a family of spectra ($k = 138,235$, $E_{\text{off}} = 2.45$, $\phi = 7.1$ eV). Results are depicted in Fig. 6-15(c). Referring to Table 6.2, surface D7 exhibits a $\Delta E_{\text{peak}}$ of $+1.0 \text{ V}$ from $\alpha = 17^\circ$ to $\alpha = 55^\circ$; detailed examination of the Eq. (6.2) results reveals a predicted $\Delta E_{\text{peak}}$ of $+1.6 \text{ V}$ over the same angular range. Quantitative comparison of the spectra intensities is not quite so good, as the predicted curve intensities now fall off too quickly with increasing emission angle. It is possible—even likely—that at least a portion of the disagreement in intensity between the model of Eq. (6.2) and the data is due to focusing of the lower-energy electrons toward the surface normal by the negative sample bias. In an attempt to eliminate this effect as a source of error, Eq.(6.2) was fit to the $E_e > 7 \text{ eV}$ data for the $\alpha = 17^\circ$ spectrum. The resulting fit did produce slightly increased intensities ($\sim 5\%$) at all energies, though agreement with the data was not significantly improved. It is also possible that the increase in $E_{\text{onset}}$ with
Figure 6-15. Comparison of surface D6 AR SE spectra to simple distribution functions.
increasing $\alpha$ is partially or wholly due to the negative sample bias; recent modeling has indicated this might be so [Nickles et al., 1999].

Equations (6.1) and (6.2) represent only a rudimentary attempt to obtain a suitable AER distribution from Chung and Everhart’s semiempirical, angle-integrated formulation; their relative success suggests that continued efforts are warranted.

6.6 (c) SE’s vs BSE’s: Where Does the SE Tail End?

Examination of the AR spectra at higher energies—in the vicinity of 450 eV—has already produced good reason to suspect that SE’s dominate the emitted electron population well beyond the conventional 50 eV cutoff. Analysis of the low-energy AR spectra lends further support to this notion and reveals some additional results.

The 50 eV Division

Based on the persistent convention of separating SE’s and BSE’s at 50 eV, it is reasonable to ask if there are any distinguishing features at 50 eV which might point to a transition between the two populations. This question can be addressed with the surface D6 data. Figure 6-16 depicts five D6 AR spectra in the range 0-450 eV plotted on a logarithmic scale. Two distinct transition zones present themselves: the decrease in slope of the distribution observed in the D7 data is again visible at about 20 eV, and also at ~100 eV. Thus, based on the AR spectra, the 0-450 eV electrons can be divided into three separate populations: the SE peak to ~20 eV (Region I); 20-100 eV (Region II); and 100-450 eV (Region III). Though the spectra slopes do reduce further and become “bumpier” at ~50 eV—features which have loosely been termed fine structure—the change is less abrupt than at the other locations. Nothing definitive presents itself as a transition zone at 50 eV.

Given no further knowledge of the spectra behavior beyond 100 eV, one might be inclined to regard either the 20 eV or 100 eV discontinuities as an SE-BSE transition zone. However, given the analysis of [6.5 (b)], this notion is weakened considerably. But if the behavior at 20 eV and 100 eV is not
FIGURE 6-16. Surface D6 low-energy AR spectra revealing trends in electron energy distributions. Data points included on the 17° spectrum are the same for all angles. $E_B = 1.5$ keV, normal incidence; $\Phi_s = -1.5$ V.
due to a shift from SE to BSE dominated emission, what be causing the observed behavior? As a matter of educated speculation, it is suggested that all three regions in Fig. 6-16 may be comprised primarily of SE’s, but with differing production mechanisms dominating in each region. Angular analysis presented in Chap. 7 will provide additional insight into this hypothesis.

The 20 eV Division

Referring to Fig. 6-14, it is interesting to note that the fits appear to worsen a bit for $E_e > -20$ eV. This deviation of the fit from the data is more evident when viewed on a logarithmic scale and is shown for all angles investigated on surface D7 in Fig. 6-16. While the fit goes astray at approximately 20 eV for all angles, a weak trend with increasing $\alpha$ is evident in Fig. 6-17: deviation of the fit from the data begins closer to 25 eV at 17° and gradually decreases monotonically to ~15 eV at 55°. Similar analysis has not been performed in detail for the surface D6 AR SE spectra, though the low-energy inset in Fig. 6-2 reveals the same signature for the D6 17° spectrum. The model deviates from the data where the slope of the tail flattens; the changing character of the spectrum indicates a transition from one population to another—perhaps transition from SE’s to BSE’s, or from SE’s produced primarily by one mechanism to those produced by another. The weak but definite angular dependence of the transition location may provide valuable clues to answering this question.

6.6 (d) SE Peak Fine Structure

Though the reliability of the AR spectra data drops for energies less than ~5 eV, there is statistically significant evidence for the presence of fine structure at several emission angles. Based on the results and discussion regarding the elastic peak fine structure [6.5 (d)], there is the inclination to look for a correlation between the ~3 eV and ~6 eV loss peaks and the shoulder that appears on the SE peaks in Fig. 6-7. The shoulder is located in the proper neighborhood for correlation—at ~5 eV. Complicating the precise determination of the shoulder’s location is the possibility that the SE peak data may be skewed somewhat by any difference in work functions between the detector (nonuniformly aquadag-coated aluminum) and the gold sample (recall the discussion of [4.7 (b)]). Adding to the uncertainty is the
FIGURE 6-17. Fits of Eq. (6.2) (solid lines) to surface D7 AR SE spectra (circles), demonstrating deviation of the fits from the data at ~20 eV for all angles.
question of origin. The detailed SEE calculations of Rösler and Brauer [1981b] for aluminum resulted in a predicted shoulder in the SE peak due specifically to enhanced SE production resulting from bulk and surface plasmon decay into single-electron states. Their predictions are in good agreement with experiment, as such structure has been observed in clean aluminum [Krane, 1978; Everhart et al., 1976; Henrich, 1973; Powell and Woodruff, 1972]. The appearance of the shoulder in the spectra in Fig. 6-7 is qualitatively very similar to that predicted by Rösler and Brauer (R&B). However, it appears more likely that the observed EP loss peaks are due to single-electron excitations in the d-band rather than plasmon damping, and whether or not this process is capable of enhancing SE production in the reverse process is not yet clear. Also, it is possible that there are plasmon frequencies at these energies; as mentioned above [6.5 (d)], the calculations have not been performed (or at least found). Finally, the apparent angular dependence of the shoulder is another clue; the R&B calculations were not performed for angle-resolved spectra (though their formulation is sufficiently general to support such calculations). It would seem that this aspect of the fine structure is an important clue to the ultimate resolution of its origin.

6.7 Discussion

While examination of the AR spectral data is not yet complete, analysis to this point has yielded two important results:

1. Evidence suggesting that SE's dominate the emitted electron population through energies significantly greater than 50 eV;

2. Evidence suggesting regions of differing dominant production mechanisms for both SE's and BSE's.

The first result is based on Fig. 6-2 and fits to the SE peaks, which indicate that the AR spectrum can reasonably be broken into four regions: (i) low-energy peak, extending to ~20 eV, (ii) inelastically scattered electrons in the range 20-450 eV, whose inelastic scattering cross sections apparently decrease with increasing energy, (iii) inelastically scattered electrons in the range 450-1460 eV, whose inelastic scattering cross sections apparently increase with increasing energy (through ~1350 eV), and (iv) elastically
scattered electrons. The primary origins of the electrons comprising regions (i) and (iv) should be relatively clear (SE’s and PE’s, respectively). To infer the origins of the remaining electrons, we have argued as follows: first, that the 10-1460 eV population is comprised of both PE’s that have lost energy through inelastic interactions with the sample, and resident sample electrons that have acquired some of this energy to become SE’s; second, that the specific interactions involved in energizing the SE’s in the 20-1460 eV range are inelastic electron-electron collisions between the PE’s and resident sample electrons (as opposed to plasmon excitation and decay, for instance). In this case, whether viewed from the perspective of the PE’s or the SE’s, inelastic scattering theory predicts diminishing scattering cross sections for increasing energy transfer $\Delta E$. If we now assume SE’s dominate the 20-450 eV population and BSE’s dominate the 450-1460 eV population, then instead of saying the 20-450 eV electrons have decreasing cross sections with increasing $E_e$ and the 450-1460 electrons have increasing cross sections with increasing $E_e$, we can say that both populations have decreasing cross sections with increasing $\Delta E$—as expected. Proceeding from this result, the entire region from the SE peak location (~2 eV) to 450 eV will now simply be termed the SE tail, while the region from 450 eV to the IP peak location (~1330 eV) will be termed the BSE tail.

The second result is based on Figs. 6-3 (b) and 6-16, which—based on the perceived changing character of the spectra slopes and possibly energy-resolved angular distributions—reveal three separate regions in both the SE and BSE tails. They are, respectively: approximately 2-20 eV, 20-100 eV, and 100-450 eV in the SE tail, and approximately 450 eV-950 eV, 950-1330 eV, and 1330-1460 eV in the BSE tail. The changing character of the SE and BSE energy and angular distributions may be consistent with transition zones between regions of differing dominant production mechanisms. To this point, however, the evidence only suggests that such may be the case. Before detailed investigation of specific theoretical predictions regarding varying production mechanisms is undertaken, the angular distributions as a function of emission energy need to be examined. This analysis is now presented.
CHAPTER 7
SURFACE PLOTS AND ENERGY-RESOLVED ANGULAR DISTRIBUTIONS

Looking at the data in as many ways as possible is an important aspect of data analysis. In the previous chapter the AER yields were viewed as AR spectra, depicted individually and together, plotted on linear and logarithmic scales. In this chapter, two additional means of depicting the data are explored: surface plots and energy-resolved (ER) angular distributions. Creating surface plots simply involves plotting the sets of AR cross sections of Chap. 6 together in a single, three-dimensional representation—the hope being that such a representation of the data will bring previously unidentified trends into specific relief. Creating ER angular distributions is also a natural extension of our analysis thus far, simply shifting focus from the “E” part of AER to the “A” part.

7.1 AR Spectra Surface Plots

Figures 7-1 and 7-2 are surface plots of the D6 and D7 AR spectra, with the intensities plotted logarithmically. Depiction of the data in this manner reveals nothing new in the D6 measurements; i.e., no new features are immediately evident. However, an aspect of the D7 data not previously noted is evident in Fig. 7-2: a region of enhanced intensity—termed the “mid-angle ridge” in the figure—spanning the full 0-50 eV energy range, between the angles of 25° and 41°. That the feature is not present in the D6 data is curious; it may indicate some important difference between the two surfaces, or may be attributable to a change external to the sample—the result of differing ambient field conditions, or perhaps improper location of the beamspot on the sample center, to name two possibilities. Future measurements specifically looking for this feature will be helpful.
FIGURE 7-1 Surface plot of surface D6 AR spectra: (a) 0-1500 eV, and (b) 0-50 eV. Vertical axis is logarithmic, scaled in exponents.
AR Spectra Surface Plot
Surface D7

**FIGURE 7-1. Continued.**
FIGURE 7-2. Surface plot of surface D7 AR spectra set. Vertical axis is logarithmic, scaled in exponents.
7.2 The Distinction Between AR Energy Distributions and ER Angular Distributions

Energy-resolved angular distributions and angle-resolved energy distributions are, in general, two entirely different sets of information. However, if one has obtained a complete set of either AR energy distributions (spectra) or ER angular distributions, one has obtained the same information. The difference really manifests itself in how one chooses to view the information. Viewing individual AR spectra (as in Chap. 6), one is investigating the behavior of electrons emitted at a given angle as a function of energy; that is, the detector has been positioned to collect electrons at a particular emission angle and then swept through an energy profile, stopping to make a measurement at (ideally) every energy. Conversely, viewing individual ER angular distributions, one is investigating the behavior of a given energy population as a function of angle; it's as if one has set their detector to measure only electrons at a particular energy (or small energy range) and then swept the detector about the sample, stopping to make a measurement at each angle. In this investigation, a set (not a complete set, but a representative one) of AR spectra has been obtained. Thus far, examination of the data has focused primarily on their nature as energy distributions. In this chapter we will continue our analysis by taking sets of energy distributions, constructing sets of angular distributions (as a function of energy), and analyzing the results—mindful of the previous chapter's conclusions.

7.3 The Value of ER Angular Distributions

Knowledge of how a particular energy population behaves with respect to emission angle is valuable to SEE investigations on two levels, both of which are rooted to the same notion: that the angular distributions of electrons leaving a surface are tied to the mechanisms which created them. On the first level, such an effect can aid in distinguishing between SE's and BSE's; on the second level, the effect may aid in identifying SE and BSE subpopulations created by different underlying mechanisms and provide clues as to the specific nature of the mechanisms. Theories based on detailed analysis of proposed
production mechanisms for both SE's and BSE's have been offered [2.3 (b)]; they make specific predictions regarding the energy and angular distributions resulting from various flavors of electron-electron, electron-ion, and electron-surface interactions, and are in need of evaluation. Such evaluation can only be accomplished with detailed sets of ER angular distribution data for a wide range of materials and incident energies, and calculations specific to the sample materials studied.

7.4 Constructing ER Angular Distributions from AR Spectra

Given the AR spectra sets described in Chap. 6, construction of ER angular distributions is accomplished by organizing the AER yields by energy rather than angle. Results can be viewed either as surface plots or cross sections of the surface plots cut along the angular axis (just as AR spectra are cross sections of the surface, cut along the energy axis). An example of this is presented in Figure 7-3. In Fig. 7-3(a) one sees the family of AR spectra; in Fig. 7-3(b) the spectra have simply been organized differently, according to energy rather than angle. While surface plots of the raw data are interesting to view, they are not particularly well-suited for examination of angular distribution as a function of energy, given the large spread in intensity as a function of energy. To facilitate accurate comparison of, say, 3 eV electrons, with, say, 20 eV electrons—whose intensity is considerably less—each ER angular distribution can be normalized to the intensity of the lowest angle—17° in this case. An example is given in Fig. 7-4. (When analyzing the normalized ER energy distributions of this investigation, it is important to realize that the distributions displayed at various energies are actually averages of energy ranges, centered on that energy: for example, the distribution plotted at 450 eV is actually the distribution for the 400-500 eV population, since data were taken at 100 eV intervals in this portion of the energy domain.)

7.5 Results and Analysis

Figures 7-5 and 7-6 are normalized ER angular distributions for the complete D6 and D7 data sets, respectively. The coloring and shading have been chosen in an attempt to best illustrate the important
FIGURE 7-3. Example of conversion from (a) AR spectra to (b) ER angular distributions (surface D6).
Normalized ER Angular Distributions

Figure 7-4. Example of normalized ER angular distributions (constructed from surface D6 AR spectra).
features in each figure. Considerable structure in the ER angular distributions is evident in both figures.

Examination of these data is accomplished with the analysis of Chap. 6 in mind. Specifically, we wish to investigate:

1. changes in the distributions at 20 eV, 50 eV, and 100 eV, based on perceived change in slopes of the individual AR spectra;

2. changes in the distributions near 450 eV, where a transition in the nature of the AR energy spectra is observed;

3. the region around 950 eV, looking at changes in the inelastic peak.

For convenience, examinations of the data are accomplished separately for the low-energy (0-450 eV) and high-energy (450-1460 eV) regions.

7.5 (a) Low-Energy ER Angular Distributions (0-450 eV)

Figure 7-7 and 7-8 depict low-energy (0-50 eV) portions of the D6 and D7 data. Apparent in both figures are coherent structure in the angle-energy surfaces. Most interesting is the pronounced "mid-angle ridge" in Fig. 7-8. Consistent with the surface plot of Fig. 7-1(b), this ridge is not evident in Fig. 7-7. Also very noticeable in both 7-7 and 7-8 are a number of energy-aligned ridges. In particular, plots indicate two well defined structures at ~40 eV and 45 eV.

Regarding the changing nature of the angular distribution with energy, the following trends are observed in both Figs. 7-7(b) and 7-8(b). Starting at ~5 eV, intensity falloff with angle increases fairly quickly to ~10 eV, remains roughly steady to ~20 eV, and then begins to fall off more quickly, on average, through 50 eV. Thus, the change in the nature of the energy distribution at ~20 eV, noted in the spectra, appears to coincide with a change in the nature of the angular distribution. This behavior is further illustrated in Fig. 7-9—a cross section of Figs. 7-7 and 7-8 comparing the angular distributions of several representative energy populations. The decidedly unique behavior of the 2-5 eV population is taken to be a consequence of their susceptibility of deflection by ambient electrostatic fields.
Normalized ER Angular Distributions
Surface D6

FIGURE 7-5. Normalized ER angular distributions for surface D6 (0-1500 eV).
Normalized ER Angular Distributions
Surface D7

FIGURE 7-6. Normalized ER angular distributions for surface D7 (0-50 eV).
Normalized ER Angular Distributions
for Surface D6
(Low-Energy)

**FIGURE 7-7.** Normalized ER angular distributions constructed from surface D6 AR spectra: (a) oblique view, showing relief in the surface and (b) head-on view, showing intensity falloff with energy. Colored circles are the actual AER yield data.
Normalized ER Angular Distributions for Surface D7 (Low-Energy)

FIGURE 7-8. Normalized ER angular distributions constructed from surface D7 AR spectra: (a) oblique view, showing relief in the surface and (b) head-on view, showing intensity falloff with energy. Colored circles are the actual AER yield data.
0-50 eV Cross Sections

Figure 7-9. Angular distributions for selected energy populations in the 0-50 eV range for (a) surface D6 and (b) surface D7.
Progressing beyond 50 eV, Fig. 7-10 depicts angular distributions through 450 eV. In this figure one notes somewhat jumbled behavior in the 50-100 eV range, particularly at emission energies of approximately 80 eV and 100 eV. Beyond 100 eV, the behavior remains constant until ~200 eV, where it becomes slowly more cosine-like (see Fig.7-11) through 450 eV. In Fig. 7-11 comparison of the various angular behaviors to cosine and screened Rutherford distributions shows that the 50 eV population more closely resembles so-called “BSE” behavior, while the higher energies closely match the cosine-like “SE” behavior. Thus our brief analysis of the energy distributions above 50 eV has revealed some sort of transition at ~ 200 eV not noted in the AR spectra analysis.

7.5 (b) High-Energy ER Angular Distributions (450-1500 eV)

Figure 7-12 depicts ER angular distributions in the range 0-1400 eV. The region beyond 1400 eV was omitted for clarity (see Fig. 7-5 for a depiction of the full energy range). This figure, more than any other, illustrates the varied nature of the SE and BSE populations emitted from the sample. Above 450 eV the figure shows a leveling-off of the angular distributions, which becomes very cosine-like (see Fig. 7-13) until approximately 950 eV, where they begin a move toward more elastic-like distribution, finally reaching a distribution that falls off much faster than the elastic (screened Rutherford) behavior. There are two important points in all of this behavior. First, distinct transitions in the ER angular distribution behaviors are observed at approximately 450 eV and 950 eV, coinciding with the changing behavior of the energy distributions. Second, whatever the kind of electron comprising the >450 eV population—SE or BSE—it is clear that the experiences of these electrons, i.e., their histories of interactions, vary considerably with emission energy.

Cross sections of several energies in the elastic region are presented in Fig. 7-14, together with a screened Rutherford cross section [Eq. (5.1)] for reference. It is interesting and unexpected to note that all of the quasi-elastic electrons fall off much faster with increasing emission angle than predicted by screened Rutherford scattering. Certainly this result deserves further investigation.
FIGURE 7-10. Normalized ER angular distributions through 450 eV (surface D6): (a) oblique view, depicting relief and (b) head-on view, depicting intensity behavior with emission energy.
0-450 eV Cross Sections
(Surface D6)

Figure 7-11. Angular distributions for selected energy populations in the range 0-450 eV (surface D6).
FIGURE 7-12. Normalized ER angular distributions through 1400 eV (surface D6): (a) oblique view, depicting relief and (b) head-on view, depicting intensity behavior with emission energy.
0-1400 eV Cross Sections
(Surface D6)

FIGURE 7-13. Angular distributions for selected energy populations in the range 0-1400 eV (surface D6).
Elastic Peak Cross Sections  
(Surface D6)

Figure 7-14. Angular distributions for selected energy populations in the elastic peak (surface D6).
7.6 Discussion

Examination of the ER angular distributions compliments and supports the results of the AR spectra analysis. Most importantly, it further muddies the distinction between SE’s and BSE’s. Whereas close examination of energy distributions (Chap. 6) has cast doubt on the 50 eV division, examination of the angular distributions has cast doubt on distinctions based on theoretical predictions of angular behavior. That is, from the data above, cosine-like behavior—generally regarded as SE behavior—is observed in both low energy (10 eV) and higher-energy (900 eV) populations, as is more elastic (screened Rutherford)-like behavior (e.g., 50 eV and 1000 eV)—generally regarded as BSE behavior. Regarding the observed angular behavior of elastic peak electrons, strong deviation from screened Rutherford cross section is potentially very important; difficulties with the primary beam, however (the 5 V peak-to-peak ripple), may be playing a role. Further analysis should be deferred until measurements can be made with a more stable source of incident primary electrons.

Electron Histories

Theoretical predictions of a cosine distribution for secondaries arise from interactions between the internally produced SE’s with the ionic cores [Rosier and Brauer, 1981b]. Thus it may be reasonable to suggest that the cosine-like behavior of the higher-energy (500-1000 eV) behavior is the result of multiple interactions of the incident primaries (with both ionic cores and resident electrons). Further, deviation from a cosine distribution may serve as a measure of how many interactions a given electron—SE or BSE—has experienced. This line of reasoning suggests that if most of the, say, 50 eV electrons are indeed SE’s, then they have been produced by a single (or very few) relatively large energy transfer interactions, and then undergone few interactions with the solid prior to emission. Precisely why this distinction would be a function of emission energy is not clear. However, more detailed analysis can be performed, given AER data such as that presented above and detailed theoretical formulations such as those of Rosler and Brauer [1981a,b] and Ganachaud and Cailler [1979a, 1979b].
It is initially unclear whether or not the hypothesis based on the AR spectra—that SE’s and BSE’s dominate above and below ~450 eV, respectively—is supported by the observed ER angular behavior. While a change in the character of the angular behavior is observed at ~450 eV, it is not the kind of fundamental change observed in the energy spectra; rather, it appears as a transition to a population that has undergone sufficient interactions within the sample to have become isotropic. Still, the energy dependence of the angular distribution depicted in Fig. 7-12(b) could be consistent with the 450 eV division between SE’s and BSE’s if we assume that (i) the average amount of energy lost or gained in a single interaction by both SE’s and BSE’s is small, and (ii) the more interactions a given electron undergoes, the more isotropic its distribution. In this case, BSE’s that have undergone greater energy losses have, on average, experienced more interactions and should have more cosine-line distributions. Looking at Fig. 7-12(b), this is observed as one moves from relatively low energy loss at the left of the figure to relatively high energy loss near the center. Similarly, SE’s that have experienced greater energy gains will have, on average, experienced more interactions and thus as one moves from low energy gains to high energy gains, the distribution should become more cosine-like. Again referring to the figure, this behavior is observed as one moves from approximately 100 eV to 500 eV. (This discussion is not meant to apply to the SE’s with energies less than ~20 eV, as their behavior appears to be of a fundamentally different nature.) Based on this purely qualitative discussion, more detailed evaluation of the observations against inelastic scattering theory is certainly warranted. In particular, such analysis might indicate the average amount of energy lost and gained per interaction for the BSE’s and SE’s, respectively, and how many interactions and of what type are required to produce isotropic populations.

Classification of the Emitted Electrons

Based on the analysis of Chaps. 6 and 7, it is suggested that the emitted electrons can be divided into five general categories:

1. SE Peak: 0-20 eV
2. Inelastic SE Tail: 20-450 eV
3. *Inelastic BSE Tail*: 450-950 eV
4. *Inelastic Peak*: 950-1485 eV
5. *Elastic Peak*: 1485-1505 eV

This classification scheme is illustrated in Fig. 7-15. The energy ranges are approximate, specific to this investigation (1.5 keV electrons normally incident on gold), and angle-dependent.

### 7.7 Conclusions

Having analyzed both AR spectra and ER angular distributions, the conclusions of [6.2] are updated:

1. Both AR spectra and ER angular distributions support a separation between SE and BSE dominance at ~450 eV;

2. The angular distribution of the SE and BSE populations is far from uniform: transitions from one angular behavior to another occur relatively smoothly, and at energies in general agreement with those identified as transition energies in the AR spectra;

3. For energies less than ~100 eV, changes in the angular distributions could be indicative of the roles of fundamentally different production processes;

4. For energies greater than ~100 eV, changes in the angular distributions are not attributed to transitions between entirely different production mechanisms, but to different numbers of interactions.

That SE’s may dominate the population of electrons leaving the sample through energies significantly greater than 50 eV (450 eV, or 0.3\(E_h\), in this investigation) would be a significant result if verified. On a practical level, such an outcome could mean significantly altered SE and BSE yields (increased and decreased, respectively), depending on material and incident energy, than were previously thought. More importantly, the fundamental manner in which SEE is viewed will need to change. Figure 7-16 depicts how SEE and electron backscattering is generally portrayed in the literature versus current observation. Clearly, a great deal of room exists for improved understanding. Further investigation should begin with additional measurements—examining the AER SE and BSE distributions of various materials for a broad range of incident energies, and for more complete and finer energy and angular ranges.
Classification of Emitted Electrons

**FIGURE 7-15.** Schematic depicting five general categories of emitted electrons as a function of emission energy. (Surface D6, $\alpha = 17^\circ$)
FIGURE 7-16. Comparison between (a) theory and (b) measurement of the angular distributions of emitted SE’s and BSE’s as a function of emission energy.
CHAPTER 8

CONCLUSION

The purpose of this chapter is to settle the dust a bit—to summarize what I believe are the principal results of this work, and to put down on paper a few suggestions for any who may choose to follow and extend this work.

8.1 Principal Results—Summary of Instrument Performance and Experimental Findings

The principal achievements of this investigation include:

(i) evaluation of instrument and method
(ii) acquisition and analysis of new measurements
(iii) experience.

Details of (i) and (ii) are discussed below; (iii) is mentioned to underscore that the completion of this dissertation does not mark the end of SEE measurement at Utah State University, more the end of the beginning, and the experienced gained in this investigation is already playing a role in plans for continued work [Chang et al., 1999].

8.1 (a) Instrument Performance

As a new design, the instrument and methods of this investigation were in need of thorough evaluation. With the conclusion of the set of measurements described in this report, this objective has been met. Specifically, we find that:

1. The RD design has proven extremely effective, with an energy resolution that is more than adequate for the intended purpose; surface charging within the detector is an issue which still needs to be addressed [see Appendix C];

2. Excellent agreement between directly measured AR yields and those obtained by integrating the AR spectra is a convincing verification of spectra measurement technique and the data reduction process [6.5(a); 6.6(a)];
3. The presence of ambient electric fields within the chamber apparatus (CA) precludes accurate AR spectra measurements below ~10 eV; these fields are likely the result of electrostatic charging of oxidized aluminum surfaces, abundant within the CA [C.3];

4. Negative sample biasing in association with very-low energy AR measurement is not a viable technique for the present geometry: low-energy SE’s are focused toward the surface normal, severely distorting their angular distribution [C.2];

5. Instabilities in the electron beam and a relatively low limit (100 Hz) on the data acquisition presently preclude Auger signal acquisition, inhibiting our ability to perform adequate in situ surface characterization [3.1(b); 3.1(c)];

6. The high-voltage power supply used to bias the RD is limited to 1 V increments at voltages above 1000 V, limiting resolution to 1 eV at high energies and preventing identification of energy loss peaks near the elastic peak [C.1(c)];

7. A 5-V peak-to-peak ripple in the electron gun power supply results in an essentially bienergetic electron beam, with energies ~5 eV apart; this feature has proved useful in assessing the RD energy resolution, though will ultimately be troublesome when attempting to identify energy loss peaks near the beam energy [C.1(c)];

8. The total yield determination scheme is wholly inadequate; relative $\delta$ and $\eta$ comparisons between surfaces are possible, but absolute yield determinations are plagued by substantial systematic errors resulting from SE collection by the tertiary samples [4.4(b); 4.7(b); A.2].

Strategies for overcoming some of these difficulties are presented in [8.2].

8.1 (b) Measurements and Analysis

Acquisition of a representative set of simultaneous angle- and energy-resolved (AER) SE and BSE measurements is by far the most important achievement of this investigation. The data were used to construct AR spectra and ER angular distributions which were examined both as surface plots and cross sections. Most significant in the analysis to date are the following:

1. Evidence strongly suggesting that SE’s dominate the population of emitted electrons through energies significantly greater than 50 eV [6.5(b); 6.6(c); 7.5(a)];

2. Indications that different SE and BSE production mechanisms dominate at different emission energies [6.6(c); 7.5];
3. No evidence of angular behavior incorporating refraction at the surface, hypothesized by Baroody, at any energy [7.5].

Total yield and AR yield measurements were used in a primarily diagnostic role, revealing a number of subtleties regardig SEE observation—the importance of work function, and the need for a field free environment.

8.2 Suggested Instrument Modifications

As a result of experience gained in this investigation, a number of improvements to instrument and technique suggest themselves:

1. Sample redesign. Evidence suggests that a substantial portion of the electric field problems noted in the AR measurements, as well as the large systematic error in the total yield measurements, is a result of poor sample arrangement. Specifically, removing the primary sample from the large sample holder currently being used, removing the present sample heater and thermocouple, and eliminating the tertiary samples altogether should improve the situation considerably. While the loss of annealing capability is not preferable, ion sputtering should maintain the ability to produce a clean surface. Regarding the tertiary samples, ancillary measurements indicate that the return current to the primary, even under conditions of positive bias, is not as significant as once thought [LBIII, p. 81y]—eliminating the need for the tertiary samples. Finally, reorienting the sample vertically and mounting the electron gun and RD in the horizontal plane will allow for all AR measurements to be made for a single azimuthal angle.

2. Aquadag coating of the chamber apparatus. Also contributing to the ambient electric field difficulties are aluminum oxide surfaces within the chamber apparatus (CA). To eliminate the charging of these surfaces that appears to be occurring, reduce the production of ancillary SE’s, and eliminate work function differences among the CA surfaces, it is suggested that all surfaces within the CA be coated with colloidal graphite. One possible difficulty with this approach is the introduction of a considerable volume of carbon into the chamber: the impact on carbon contamination of the sample surface will need to be assessed in situ. If it is found that this solution is unworkable, it is suggested that the aluminum components within the CA be remade, perhaps with titanium, and gold plated (to prevent oxide formation). One disadvantage of this scheme is the increased production of tertiary SE’s by the gold.
3. *Ground sample and all surfaces.* In order to ensure as field-free an environment as possible, the sample and all surfaces within the chamber apparatus should be at the same potential for AER measurements at the very lowest energies; tying everything to earth ground is the simplest way to accomplish this. Sample floating may still be used without difficulty for measurements at energies greater than ~15 eV.

4. *New, smaller detector.* Since the effect of ambient electric fields is directly proportional to the sample-detector distance, reducing this distance is desirable. Using a smaller detector—of the same basic design—should facilitate this change. Also, it will be helpful to coat the interior of the new detector with aquadag, ensuring the surfaces within the detector are uniform, immune to surface charging, and have the same work function as most other surfaces in the CA. In fact, such a detector has already been constructed; all that remains is to put it to use.

5. *New or improved electron gun and faster data acquisition.* Resolution of Auger signals will require considerably less noisy data than that currently being collected. Evidence suggests that a significant portion of the present noise level is due to instability in the primary beam; reducing this instability may or may not require a new electron gun. Coupled with a more stable beam, faster data acquisition will allow computer averaging of more data points, further reducing uncertainty in the signal. Also, the 5-V peak-to-peak ripple in the electron gun power supply needs to be eliminated; this may or may not be possible with the present Varian gun by repairing the HV power supply.

6. *Scheme for RD biasing resolution at high voltage.* Resolution of energy loss peaks near the beam energy, necessary for surface and bulk plasmon investigation, will require energy resolution on the order of 0.1 eV at energies above 1 keV. To effect such a capability with the present HV power supply, it may be possible to set the HV supply at a given voltage and then float the HV supply with a lower-voltage, higher resolution supply.

One final suggestion, designed exclusively to improve angle-resolved measurements, is implementation of a double sphere design similar to that used by Jonker [1951]. An initial design scheme is presented in Fig. 8-1. The sample is located at the center of two concentric conducting spheres. The inner sphere and sample are held at ground, creating a field free region inside the inner sphere, while the outer sphere and attached detector rotate in the horizontal plane about the sample. The outer sphere can be biased as desired, and the resulting electrostatic field in the region between the two spheres is ideally radial. Each sphere has a slot running about the equator—to allow for the escape of SE’s and BSE’s in the
Double-Sphere Detector-Sample Design

![Diagram of double-sphere detector-sample design](image)

**Figure 8-1.** Schematic depicting double-sphere design for measurement of angle-resolved SE spectra.
case of the inner sphere, and to allow primary beam access to the sample in the case of the outer sphere. The interior of the both spheres is coated with colloidal graphite (aquadag) to prevent surface charging, reduce tertiary SE production, and provide for a uniform work function. Total yield determination with this scheme may be possible through some sort of biasing scheme for the sample and inner sphere.

Several variations of the double sphere design are also possible. First, one could likely eliminate the inner sphere altogether without serious affect: it is the field-free aspect of the design that is most important, and removing the inner sphere while grounding the outer sphere preserves this quality. Replacing the spheres with cylinders (single or double) is second option, and such a design may prove easier to fabricate.

As presented here, the single or double sphere (or cylinder) design leaves a number of important details unaddressed. Beam current measurement, total yield determination, ion beam access for sputtering, and sample heating are a few examples of additional considerations.

8.3 Recommendations for Further Research

Three tasks present themselves as the most immediate and most logical next steps once improvements to the instrument have been effected:

1. *Further AER measurements for polycrystalline gold.* Full sets of AER data should be acquired for a range of incident energies and the results examined for the same five categories of emitted electrons as observed in this investigation. If the analysis of this investigation is correct, one would expect the same telltale transition signatures to appear in the AR spectra and ER angular distributions, but at different energies. The data should also be examined for evidence of the mid-angle ridge noted in the surface D7 ER angular distributions [7.4 (a)].

2. *Acquisition of AER measurements for polycrystalline aluminum.* Data for additional materials should be acquired for comparison with gold, and since specific, detailed theoretical calculations have been made for SEE from aluminum (*e.g.*, Rösler and Brauer [1981b] and Ganachaud and Cailler [1979b]), aluminum seems the logical choice for the next material investigated.
3. **Comparison with theory.** Once additional data have been acquired, detailed comparison with the theories of Rösl er and Brauer [1981a, 1981b], Jablonski [1991], Jablonski et al. [1993, 1989], Gryźinski [1965], and Michaud and Sanchez [1984] should be attempted. Such analysis will involve examination of fine structure in the SE peak, as well as an attempt to account for the observed ER angular behavior over the entire range of emission energies.

Also, additional investigations along the following lines come to mind:

4. **Fine structure attributed to plasmon excitation and decay.** Fine structure due to plasmon excitations—referred to as energy loss peaks—are well-documented features found near the elastic peak [Reimer, 1993]. Fine structure attributed to plasmon decay has been observed in integrated SE spectra, near the SE peak, for clean aluminum [2.3 (b)]. To my knowledge, the angular dependence of these phenomena remains entirely uninvestigated experimentally.

5. **Contamination studies.** As thicker and thicker contaminant layers are deposited, SE and BSE production is affected at greater and greater depths within the surface. Examining the AR yields of various energy populations—obtained from AR spectra—as a function of contamination depth may prove beneficial for understanding details of SE and BSE transport.

6. **Negative sample biasing.** Since SE’s originate within the sample, SE energies—as measured by the detector—are affected by sample bias, while BSE energies are not. Examining spectra taken under varying conditions of negative sample bias, therefore, may prove useful in identifying true secondaries, complementing the AER data.

Finally, it cannot be overstated that exploring new ways to look at the same data has proved invaluable in this investigation. Progressing from a linear plot of the AR spectra to a log plot suggested new ways to classify the emitted electrons, moving to a surface plot revealed significant fine structure in the low-energy realm, and plotting the AER data as ER angular distributions rather than AR spectra uncovered a rich variety of angular behavior. Thus it is strongly recommended that additional means of organizing and displaying the AER data be actively explored. One example that immediately comes to mind is looking at normalized AR spectra, analogous to the normalized angular distributions of Chap. 7.
8.4 Concluding Remarks

This investigation began approximately seven years ago with the initial goal of acquiring AER SE measurements for conductors, for the somewhat vague reason that there did not seem to be any such measurements in the literature. Throughout the data acquisition process, and even much of the analysis, focus remained primarily on the low-energy (< 50 eV) population. Eventually, however, it was realized that the data were rich with new information throughout the entire range of energies investigated. Detailed analysis has been accomplished for some of the data—much of it pertaining to instrument evaluation. Analysis for much more of the data has been accomplished to varying degrees—some only just begun. It may seem that much of the analysis presented is incomplete, and that is correct. Nevertheless, such analysis has been included in the hope of stimulating further work. One could spend a career following all that has been begun and suggested in these pages. If one could just graduate....
REFERENCES


APPENDICES
APPENDIX A

CALCULATIONS

A.1 Random Error in $\delta$ and $\eta$ Determinations

Estimates of the random error in the total SE and BSE yield determinations, based on multiple measurements and calculated in the usual manner, are presented in Tables A.1 and A.2. In the case of single location measurements (Table A.1), random error can be attributed primarily to variations in the incident beam current. In the case of multiple-location measurements, random variations in surface morphology and cleanliness are likely to be additional contributing factors.

A.2 Systematic Error in $\delta$ and $\eta$ Determinations

A.2 (a) The Gamma Factor

With the primary and tertiary samples biased to +50 V, it is reasonable to assume that at least some of the SE’s emitted by the primary are collected by the tertiaries, rather than returning to the primary as desired. Referring to Fig. A-1, the SE’s subject to collection are those with trajectories taking them into the shaded regions—the regions above the tertiary samples. From the figure, which is drawn nearly to scale, it is easy to see that about half of the SE’s are emitted into these regions, and therefore the maximum number of SE’s that might be collected by the tertiaries is about 50%. In the development that follows, $s(0)$, $s(50)$, $r(0)$, $r(50)_{true}$, $r(50)_{meas}$, $se$, and $bse$, and $b$ are the sample currents at 0 V and +50 V, the return (tertiary sample) current at 0 V, the actual and measured return currents at +50 V, and the SE, BSE, and beam currents, respectively. We have

$$s(0) = b - bse - se + r(0)$$

$$s(50) = b - bse - \gamma \cdot se + r(50)_{true}$$

where $\gamma$ is defined as the fraction of SE’s collected by the tertiary samples, and $r(50)_{true}$ is given by

$$r(50)_{true} = r(50)_{meas} - \gamma \cdot se.$$  After a bit of manipulation, we can write the expressions
TABLE A.1. Standard deviations for single-location measurements for selected surfaces. ($E_B = 1.5$ keV)

<table>
<thead>
<tr>
<th>Surface</th>
<th># of Measurements, Locations</th>
<th>$\sigma_\tau$ (each location)</th>
<th>Fractional Uncertainty (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>$\sigma_\delta$</td>
<td>$\sigma_\eta$</td>
</tr>
<tr>
<td>A0i</td>
<td>3 meas. on each of 4 locations</td>
<td>0.003</td>
<td>0.002</td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.006</td>
<td>0.003</td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.01</td>
<td>0.008</td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.003</td>
<td>0.003</td>
</tr>
<tr>
<td>A3a</td>
<td>17 meas. on 1 location (center)</td>
<td>0.001</td>
<td>0.001</td>
</tr>
<tr>
<td>A6</td>
<td>3 meas. on each of 5 locations</td>
<td>0.003</td>
<td>0.003</td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.000</td>
<td>0.000</td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.006</td>
<td>0.003</td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.003</td>
<td>0.000</td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.003</td>
<td>0.007</td>
</tr>
<tr>
<td>D2</td>
<td>5 meas. on 1 location</td>
<td>0.004</td>
<td>0.003</td>
</tr>
</tbody>
</table>

TABLE A.2. Standard deviations for multiple-location measurements for selected surfaces ($E_B = 1.5$ keV)

<table>
<thead>
<tr>
<th>Surface</th>
<th># of Measurements, Locations</th>
<th>$\sigma_\tau$ (all locations)</th>
<th>Fractional Uncertainty (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>$\sigma_\delta$</td>
<td>$\sigma_\eta$</td>
</tr>
<tr>
<td>A0i</td>
<td>15 meas. over 4 locations</td>
<td>0.008</td>
<td>0.003</td>
</tr>
<tr>
<td>A6</td>
<td>15 meas. over 5 locations</td>
<td>0.006</td>
<td>0.003</td>
</tr>
<tr>
<td>D2</td>
<td>9 meas. over 3 locations</td>
<td>0.004</td>
<td>0.003</td>
</tr>
<tr>
<td>D3</td>
<td>4 meas. over 3 locations</td>
<td>0.009</td>
<td>0.005</td>
</tr>
</tbody>
</table>
FIGURE A-1. SE’s emitted into the shaded regions are subject to collection by the tertiary samples when the primary and tertiary samples are biased to +50 V.

\[ se = (1 - 2\gamma)^{-1} \left[ s(50) - r(50)_{\text{meas}} - s(0) + r(0) \right] \]  \hspace{1cm} (A.1)

\[ bse = b - (1 - 2\gamma)^{-1} \left[ s(50) - r(50)_{\text{meas}} \right] + \frac{2\gamma}{1 - 2\gamma} \left[ s(0) - r(0) \right] \]  \hspace{1cm} (A.2)

for the corrected SE and BSE currents.

A.2 (b) Estimating Gamma

Note that the numerators in Eqs. (3.9) and (3.16) represent the SE and BSE currents, respectively, in the case where $\gamma$ is zero. Therefore we see from Eq. (A.1) that the effect of taking the tertiary-sample-collected SE’s into account is to increase $\delta$; that is, not accounting for this effect will underestimate $\delta$ by a factor of $1 - 2\gamma$, always (recall our above assumption that $\gamma < 0.5$). Looking at Eq. (A.2), the effect on $\eta$ of including $\gamma$ is less clear; the effect of the second term in the equation is to reduce $\eta$, while that of the third term is to increase $\eta$.

Fortunately, we can easily remove the ambiguity by realizing that we must have $\delta + \eta = \sigma$ (a constant). Therefore, if the effect of including $\gamma$ is to increase $\delta$, it must also be that $\eta$ is decreased. Returning to Eq. (A2), this means that we must have

\[ \frac{\text{2nd term}}{\text{3rd term}} > 1 \]

Imposing this constraint, along with the additional facts that (i) from my measurements it is always the case that $[s(50) - r(50)_{\text{meas}}] < [s(0) - r(0)]$, and (ii) we must have $\eta > 0$, leads to the expression for $\gamma$.
Applying expression (A.3) to data set D3.01 (one of the data sets for surface D3) gives an ultimate upper bound on $\gamma$ of approximately 16%. Based on our derivation of Eq. (A.3), this says $\gamma$ equals 0.16 if $\eta = 0$, for an incident beam energy of $E_B = 1.5$ keV (the beam energy for data set D3.01). If we actually knew what $\eta$ was, we could refine this estimate of $\gamma$. Fortunately, we do have a clue. In order to obtain a more accurate value for $\gamma$ assume that:

(i) the value of $\eta = 0.46$ obtained by Thomas and Pattinson [1970] for $E_B = 1.5$ keV is correct (also the value obtained via integration of the $\eta(\alpha')_{\text{meas}}$ values obtained for surface D6);

(ii) at least one of the sample D surfaces is similar to that of Thomas and Pattinson, and that collection of SE’s by the tertiary samples is the only reason my calculated $\eta$ for this surface deviates from the T&P value.

Then from Eq. (A.2) we have

$$\eta = 0.46 = \frac{b_{\text{se}}}{b}$$

which implies

$$0.46b = \frac{b(1 - 2\gamma) - [s(50) - r(50)_{\text{meas}}] + 2\gamma [s(0) - r(0)]}{1 - 2\gamma}$$

Solving for $\gamma$ yields

$$\gamma = \frac{0.54b - [s(50) - r(50)_{\text{meas}}]}{2(0.54b) - 2[s(0) - r(0)]} \quad (A.4)$$

Reviewing the sample D data, we find that surface D3 has the lowest value for $\eta$ (0.54), while surface D8 has the highest (0.70). Using the data from these two surfaces, we can estimate realistic upper and lower bounds on $\gamma$ and, therefore, upper and lower bounds on the systematic errors in $\delta$ and $\eta$. Beginning with data set D3.01a and applying Eq. (A.4), we calculate a lower bound of $\gamma = 0.03$, meaning approximately 3% of the SE’s emitted from the primary sample are collected by the tertiary samples when both primary
and tertiary samples are biased to +50 V. Thus we have $(1 - 2\gamma)^{-1} = 1.06$, which implies a systematic error in $\delta_{\text{meas}}$ of

$$
\% \text{Error} (\delta) = \frac{\delta_{\text{meas}} - \delta_{\text{true}}}{\delta_{\text{true}}} = \frac{\delta_{\text{meas}} - 1.06\delta_{\text{meas}}}{1.06\delta_{\text{meas}}} = -6\% 
$$

and a systematic error in $\eta_{\text{meas}}$ of

$$
\% \text{Error} (\eta) = \frac{\eta_{\text{meas}} - \eta_{\text{true}}}{\eta_{\text{true}}} = \frac{\eta_{\text{meas}} - (\eta_{\text{meas}} - 0.06\delta_{\text{meas}})}{\eta_{\text{meas}} - 0.06\delta_{\text{meas}}} = +15\% ,
$$

where we have used the fact that $\sigma = \text{constant}$, which implies

$$
\eta_{\text{true}} + \delta_{\text{true}} = \eta_{\text{true}} + 1.06\delta_{\text{meas}} = \eta_{\text{meas}} + \delta_{\text{meas}}
$$

or

$$
\eta_{\text{true}} = \eta_{\text{meas}} - 0.06\delta_{\text{meas}}
$$

Repeating this procedure on data set D8.01a leads to an upper bound on $\gamma$ of $\gamma = 0.10$, or

$$(1 - 2\gamma)^{-1} = 1.25$$

which translates to systematic errors in $\delta_{\text{meas}}$ and $\eta_{\text{meas}}$ of $-20\%$ and $+51\%$, respectively.

**A.2 (c) Changing Surface Conditions**

It is important to note that as a result of SE collection by the tertiaries, changes in a surface’s SEE characteristics are accompanied not only by a change in $\delta$, but by a concomitant change in $\eta$ as well.

Specifically, we see that a reduction in the actual number of SE’s being emitted is reflected by a reduction in the measured currents $I_{s(50)}$ and $I_{r(50)}$; referring to Eqs. (3.9) and (3.16), the result is a decrease in the measured values of both $\delta$ and $\eta$. Conversely, increasing the number of emitted SE’s has the reverse effect on $I_{s(50)}$ and $I_{r(50)}$, resulting in increased measured values for both $\delta$ and $\eta$. Note that implicit in this example is the assumption that $\gamma$ is constant. If we abandon this assumption, it is possible for a change in SEE to result in oppositely directed changes in $\delta$ and $\eta$. To illustrate this point, consider the example in Table A.3. In Case 2 in the table, the sample has undergone some process (e.g., sputtering) which has lowered the SE yield, but left the BSE yield unchanged. Also, $\gamma$ has increased in Case 2, with
TABLE A.3. How changing surface conditions might affect $\eta$ via a change in $\gamma$

<table>
<thead>
<tr>
<th>Case 1 (Initial Surface)</th>
<th>Case 2 (decreased $\delta$, increased $\gamma$)</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>SE collection by tertiaries</strong>: $\gamma = 0.05$</td>
<td><strong>SE collection by tertiaries</strong>: $\gamma = 0.10$</td>
</tr>
<tr>
<td>Actual $\delta$: 1.00</td>
<td>Actual $\delta$: 0.96</td>
</tr>
<tr>
<td>Actual $\eta$: 0.40</td>
<td>Actual $\eta$: 0.40</td>
</tr>
<tr>
<td>Actual return currents (i.e., not including erroneous SE collection by the tertiaries)</td>
<td>Actual return currents (unchanged)</td>
</tr>
<tr>
<td>$I_{r(0)} = -3$</td>
<td>$I_{r(0)} = -3$</td>
</tr>
<tr>
<td>$I_{r(50)} = -20$</td>
<td>$I_{r(50)} = -20$</td>
</tr>
</tbody>
</table>

| $I_b = -100$ | $I_b = -100$ |
| $I_{\delta(0)} = +37$ | $I_{\delta(0)} = +33$ |
| $I_{r(0)} = -3$ | $I_{r(0)} = -3$ |
| $I_{\eta(50)} = -75$ | $I_{\eta(50)} = -70$ |
| $I_{r(50)} = -25$ | $I_{r(50)} = -30$ |

\[\delta_{\text{meas}} = 0.90 \quad \eta_{\text{meas}} = 0.50\]

\[\delta_{\text{meas}} = 0.77 \quad \eta_{\text{meas}} = 0.59\]
the result that as δ decreases, η actually increases. A mechanism which might simultaneously result in a decreasing δ and increasing γ (and therefore an increasing η) is discussed in [4.7 (b)].

A.3 Total SE and BSE Yield Determinations for a Negatively Biased Sample

Using the notation defined in [A.2], consider the currents absorbed by and emitted from a sample under the conditions of sample bias depicted in Fig. A-2, where we have assumed that

\[ b_{se}(0) = b_{se}(-10) = bse. \]

We see that the sample currents at 0 V and -10 V, respectively, are given by

\[
\begin{align*}
    s(0) &= b - s_{e}(0) - bse + r(0) \\
    s(-10) &= b - s_{e}(-10) - bse + r(-10)
\end{align*}
\]

We can eliminate \( b \) and \( bse \) by subtracting, which gives

\[
se(-10) - se(0) = s(0) - s(-10) - r(0) + r(-10)
\]

But we already have an expression for the 0-V SE current [3.3 (d)]:

\[ se(0) = s(50) - r(50) - s(0) + r(0) \]

Substituting, we have

\[
se(-10) = s(50) - s(-10) - r(50) + r(-10)
\] (A5)

FIGURE A-2. Currents to and from a sample under different conditions of sample bias.
Thus we can calculate the SE yield of a negatively biased sample via an analogous equation to Eq. (9):

$$
\delta(-10)_{\text{meas}} = \frac{I_{se(-10)}}{I_B} = \frac{I_{S(50)} - I_{r(50)} - I_{S(-10)} + I_{r(-10)}}{I_B}
$$

(A6)

### A.4 SE and BSE Deflection Due to Ambient Electric and Magnetic Fields

The force on a particle of charge $q$ and mass $m$ in the presence of an electric field $\vec{E}$ and a magnetic field $\vec{B}$ is given by the Lorentz force law

$$
\vec{F} = q[\vec{E} + \vec{v} \times \vec{B}] = m\vec{a}
$$

An ambient electric field alone will accelerate an SE or BSE with a magnitude

$$
|\vec{a}| = \frac{q|\vec{E}|}{m}
$$
during the time $t$ that it moves over the sample to the detector distance, $d$: $t = d, \sqrt{m/2E}$, where the electron's velocity $v$ is related to its energy $E$ via $v = \sqrt{2E/m}$. If we assume $\vec{E}$ perpendicular to the electron's initial velocity, we can solve for the resulting deflection $\Delta d$ over the sample-detector distance:

$$
\Delta d_{\text{electric}} = \frac{1}{2} at^2 = \frac{q E}{4E} \frac{d_i^2}{d}\]

We will consider $\vec{E}$ to be significant when $\Delta d$ is comparable to the size of the detector aperture, $\sim 1.5$ mm. We can then solve for the minimum transverse electric field strength required to effect a significant deflection as a function of electron energy:

$$
E_{\perp} = \frac{4E \Delta d}{qd_i^2}
$$

This relation is displayed graphically in Fig. A-3, where $\Delta d = 1.5$ mm and $d_i = 62$ mm for this apparatus.

For an ambient magnetic field, $\vec{B}$, consider the geometry depicted in Fig. A-4. An electron of energy $E$ is emitted from the point $(r,0)$ towards the detector, and is deflected in a circular arc by an ambient, transverse $\vec{B}$ directed out of the page. As before, we will consider $\vec{B}$ to be significant when it
Effect of Electric and Magnetic Fields on Electron Trajectories

Figure A-3. Minimum transverse electric and magnetic fields required to deflect an SE or BSE out of the first RD aperture, as a function of electron energy.
Magnetic Field Effects on Electron Detection

**Figure A-4.** Deflection of an SE or BSE due to an ambient, transverse magnetic field.
results in a transverse deflection (relative to the electron’s initial velocity vector) $\Delta d$ comparable to the size of the detector aperture, over the sample-detector distance, $d$. By solving for the maximum Larmour radius, $r$, associated with this deflection (as shown in the figure), we can determine the minimum transverse magnetic field strength necessary to effect such a deflection.

We have $x^2 + y^2 = r^2$, where $x = r - \Delta d$ and $y = d$, giving

$$
\Delta d_{\text{magnetic}} = \frac{1}{qB_\perp} \left\{ \sqrt{2mE} - \sqrt{2mE - (qB_\perp d)^2} \right\}
$$

Demanding that for $x = r - 1.5 \, \text{mm}$, $y = 62 \, \text{mm}$, solving for the Larmour radius yields $r \approx 1.3 \, \text{m}$. Then from the defining equation for the Larmour radius,

$$
r = \frac{mv}{qB_\perp} = \frac{1}{qmB_\perp} \sqrt{2E}
$$

we can write

$$
B_\perp = \frac{1}{qm} \sqrt{2E}
$$

This relation is depicted graphically in Fig. A-3.
APPENDIX B

SAMPLE SURFACE SUMMARY

Measurements in this investigation were accomplished for 30 different surfaces on three separate samples of polycrystalline gold. A surface on a given sample was considered to have “changed” whenever the sample was subjected to any process with a reasonable chance of altering its SEE characteristics—e.g., ion sputtering, annealing, and prolonged energetic electron bombardment. The three separate gold samples were designated A, B, and D. Individual surfaces are labeled with the following nomenclature: the first letter designates the sample (A, B, or D), and the following numbers and letters designate a new surface. For example, A0i and A0ii are different surfaces on the same sample (sample A). There is no significance to the fact that some surfaces are distinguished by an additional letter in the label (e.g., A4 and A4a) and some are not (e.g., A5 and A6). A different label always means a new surface from the standpoint of secondary electron emission.

Sample A: Following chemical cleaning and flaming [4.1 (b)], the sample was stored for ~1 hr. in distilled, deionized water prior to its insertion into the vacuum chamber and subsequent immediate pumpdown.

Surface

A0i original washed and flamed surface;
system bakeout (~72 hrs. at 150°C);
annealed (210°C for 48 hrs.)

A0ii electron beam bombardment of sample center (~10 hrs. at 10 μA/cm²);
annealed (210°C for 48 hrs.)

A0iii Ar⁺ sputtered (45 min., 2.8 keV, normal incidence, ~1 μA/cm²)

A0iv annealed (210°C for ~24 hrs.)

A1 vacuum exposure (~2x10⁻¹⁰ Torr for ~1 month);
electron beam bombardment of sample center (~5 hrs. at 10 μA/cm²)

A1a atmosphere exposure (~1 hr.);
system bakeout (~72 hrs. at 150°C)
A2 Ar⁺ sputtered (for 15 min., 500 eV, normal incidence, \(~0.1 \mu A/cm^2\))

A3 annealed (220°C for \(~15\) hrs); surface hot (\(~220°C\))

A3a surface A3 cooled to room temperature

A3b electron beam bombardment of sample center (45 hrs. at \(~10 \mu A/cm^2\))

A4 additional electron beam bombardment of sample center (120 hrs. at \(~1000 \mu A/cm^2\))

A4a Argon exposure (\(~90\) min. at \(4 \times 10^{-4}\) Torr)

A5 annealed (350°C for \(~6\) hrs.)

A6 Ar⁺ sputtered (16 hrs., 500 eV, normal incidence, \(~0.1 \mu A/cm^2\)); annealed (250°C for 1 hr.)

A7 annealed (300°C for \(~10\) hrs.)

A7a electron beam bombardment of sample center (30 min. at \(~10 \mu A/cm^2\))

A7b further electron beam bombardment of sample center (50 min. at \(~10 \mu A/cm^2\))

A7c electron beam bombardment of top center location (76 min. at \(~10 \mu A/cm^2\))

A8 Ar⁺ sputtered (45 min., 2.6 keV, various angles 0-90°, \(~1.5 \mu A/cm^2\)); annealed (260°C for 2 hrs.)

A8a electron beam bombardment of sample center (8 hrs. at \(~10 \mu A/cm^2\))

A9 annealed (300°C for \(~10\) hrs.)

A9a electron beam bombardment of sample center (40 min. at \(~10 \mu A/cm^2\))

A10 annealed (300°C for \(~30\) hrs.)

A10a electron beam bombardment of sample center (60 min. at \(~10 \mu A/cm^2\))

A11 Ar⁺ sputtered (15 min., 2.6 keV, normal incidence, \(~1.5 \mu A/cm^2\))

Sample B: Following chemical cleaning and flaming, the sample was stored in distilled, deionized water for \(~5\) months before vacuum chamber insertion.

Surface

B1 original washed and flamed surface; atmosphere exposure (\(~1\) hr.); system bakeout (\(~72\) hrs. at 150°C)
B2 annealed (210°C for ~15 hrs.);
Ar⁺ sputtered (15 min., 2.5 keV, 75° incidence, ~1.5 μA/cm²)

B2a electron beam bombardment of sample center (6 hrs. at ~10 μA/cm²)

B3 Ar⁺ sputtered (15 min., 2.5 keV, 75° incidence, ~1.5 μA/cm²)

B3a electron beam bombardment of sample center (17 hrs. at ~10 μA/cm²)

**Sample D:** Following chemical cleaning and flaming, the sample was stored in distilled, deionized water for ~8 months prior to vacuum chamber insertion.

**Surface**

D1 original washed and flamed surface;
system bakeout (~72 hrs. at 150° C);
annealed (215°C for 20 hrs.);
Ar⁺ sputtered (15 min., 2.5 keV, 75° incidence, ~1.5 μA/cm²)

D1a electron beam bombardment of sample center (11 hrs. at ~10 μA/cm²)

D2 Ar⁺ sputtered (15 min., 2.5 keV, 75° incidence, ~2 μA/cm²);
annealed (215°C for ~20 hrs.)

D3 atmosphere exposure (~48 hrs.);
pumpdown and system bakeout (~72 hrs. at 150° C)

D4 Ar⁺ sputtered (15 min., 2.5 keV, 75° incidence, ~1.5 μA/cm²)

D5 annealed (210°C for ~22 hrs.);
Ar⁺ sputtered (15 min., 2.5 keV, 75° incidence, ~1.5 μA/cm²)

D5a electron beam bombardment of sample center (60 min. at ~10 μA/cm²)

D6 Ar⁺ sputtered (20 min., 2.5 keV, 75° incidence, ~1.5 μA/cm²)

D6a electron beam bombardment of sample center (31 hrs. at ~10 μA/cm²)

D7 Ar⁺ sputtered (15 hrs., 500 eV, 75° incidence, ~0.3 μA/cm²)

D7a electron beam bombardment of sample center (30 hrs. at ~10 μA/cm²)

D8 Ar⁺ sputtered (21 hrs., 500 eV, 75° incidence, ~0.3 μA/cm²)

D9 Ne⁺ sputtered (19 hrs., 500 eV, 75° incidence, ~0.2 μA/cm²)

D10 under UHV (~2 months);
several short duration (\(-1\) hr) atmosphere exposures, followed by pumpdowns;
additional vacuum exposure (\(-10^{-9}\) Torr for \(-2\) months, no bakeout)

D11 \(\text{Ar}^+\) sputtered (17 hrs, 500 eV, 75° incidence, \(-0.3\) \(\mu\)A/cm²)

D12 annealed (222°C for \(-60\) hrs.)
APPENDIX C
ANGLE-RESOLVED DIAGNOSTIC MEASUREMENTS

Evaluation of the strengths and weaknesses of the instrument and method with respect to AR observations has comprised a substantial portion of this investigation. Results of these diagnostic measurements are crucial to the proper evaluation of the AR-yield and -spectra measurements presented in Chaps. 5-7. Generally, it is found that the instrument is performing well, with the exception of some nagging field affects. In particular, AR BSE observations show no hint of difficulty, though ambient electric fields—apparently due to surface charging within the chamber apparatus—are affecting accurate measurement of the lower energy SE's. Detailed descriptions of the data and analyses leading to these and other conclusions are presented below.

C.1 Rotatable Detector

C.1 (a) RD Alignment

Prior to vacuum chamber insertion, the RD was aligned on the sample center. Inside the vacuum, the alignment was checked by locating the electron beam on the sample so as to maximize the RD signal; if the RD were properly aligned, the signal would be maximized when the beam was on the sample center, regardless of RD location about the sample. The following were noted:

1. In general, $I_{RD(0)}$ was not maximized at the same beam location as $I_{RD(-50)}$.

2. Maximum $I_{RD(-50)}$ occurred for a beam location of sample center for $0 < \phi_{RD} < 120^\circ$; for $\phi_{RD} > 120^\circ$, however, maximum $I_{RD(-50)}$ occurred at locations progressively nearer the top of the sample.

The first of these results suggests that the trajectories of the low energy electrons are being significantly affected between the sample and detector. Evidence presented in [C.3] will identify ambient electric fields as the cause. At first glance, the second of the above results would seem to indicate that the RD is not properly aligned on the sample center. On further consideration, however, this behavior is just as it ought to be. When the RD is placed at low and middle emission angles, the signal is maximized when the beam...
is on sample center. At high emission angle placement, however, moving the beamspot to the top of the sample moves it closer to the detector by ~6mm, where the solid angle subtended by the detector is larger, therefore collecting more signal. Thus it appears that the RD is adequately aligned.

C.1 (b) RD-Produced Secondaries

Some of the electrons entering the RD—those entering at angles slightly off-centerline—strike the interior surfaces of the 1st and 2nd apertures. It is reasonable to assume that SE’s are produced at these surfaces as a result, and that some of these RD-produced secondaries are subsequently collected by the RD, contaminating the signal. In order to quantify this affect, an AR spectrum was obtained with the sample biased to −50 V. At this sample bias, all SE’s produced at the sample will have energies of at least 50 eV as they enter the detector; RD-produced SE’s, however, will be peaked around 2 eV. By sweeping the detector from 0 V to −50 V, one should obtain a spectrum of the RD-produced secondaries. Results are depicted in Fig. C-1. Because the signal due to the RD-produced SE’s was so small, the data were quite noisy; applying a standard smoothing algorithm, however, clearly reveals a typical SE spectrum (LB IV, p. 16y). Comparison of the RD-produced SE signal with that of the sample-produced SE’s [Fig. C-1(b)] shows the RD-produced portion comprises nearly all of the signal at ~1.5 eV, falling to about 5% by ~3 eV.

C.1 (c) Energy Resolution

Estimates of the RD’s energy resolution can be made both experimentally and theoretically. Theoretical estimation is based on the geometric considerations of sample-RD distance, RD aperture sizes, and beamspot size, and results in a value of approximately 0.02%. That is, for an RD bias of −1000 V, the RD theoretically collects electrons with energies $E > 1000.2$ eV. Experimental estimation of the RD’s energy resolution can be accomplished given a spectrum and a feature with a known width about a known energy. There exists such a feature in this investigation.

Resulting from a 5 V peak-to-peak ripple in the electron gun power supply’s HV output, the electron beam is actually bi-energetic; i.e., the beam is comprised of an electron population centered about
Figure C-1. (a) RD-produced secondaries and (b) comparison of RD- and sample-produced SE signals for surface D7. $E_B = 1.5$ keV.
two energies—at approximately $E_B \pm 2.5$ eV in this case [LB III, P. 90w]. Thus, for a given beam energy, there should exist two elastic peaks in the BSE spectra, centered about the beam energy and with an approximately 5-eV separation. Figure C-2 is a portion of a typical AR spectrum for this investigation, depicting the elastic BSE peak for $E_B = 1500$ eV. Data are collected at 1 eV intervals. In this spectrum, the two peaks of the bi-energetic beam are clearly resolved; thus a conservative estimate of the instrument’s energy resolution is 5 eV out of 1500 eV, or $\sim 0.3\%$. A more realistic estimation of energy resolution is obtained by looking at the half-width at half-maximum of the two peaks. Doing so gives a value of about 1.0 V out of 1500 eV, or $\sim 0.07\%$, in surprisingly good agreement with theoretical estimates given above. Here it is noted that above 1100 V, the high voltage power supply used to bias the RD has a lower limit of 1 V per energy step, inherently limiting energy resolution to 1 eV.

On the low-energy side, energy resolution is estimated by looking at the half-width at half-maximum for the low energy side of the SE peak. Examination of several AR SE spectra in this manner for surface D6 leads to a value of $\sim 0.5$ eV. Thus we can estimate the overall energy resolution as $0.5$ eV $\pm 0.03\%$ of $E_B$. 

\[ \text{Double BSE Peak} \]
\[ \alpha = +17^\circ \]
\[ E_B = 1500 \text{ eV} \]

\begin{figure}[h]
\centering
\includegraphics[width=\textwidth]{double_bse_peak.png}
\caption{Angle-resolved spectrum depicting double BSE peak for surfaces D6 and B3. $E_B = 1.5$ keV.}
\end{figure}
C.1 (d) Charging within the RD

Figure C-3 depicts a number of AR SE spectra taken for several RD positions on surface D7. Referring to Fig. C-3(a), trials D7.18, D7.21, and D7.22 were originally taken in order to quantify repeatability of AR spectra. While all three trials indicate excellent repeatability, something odd is happening with D7.22; specifically, \( N(E) \) is negative up to \(-2.8\) eV. This means that as the RD was stepping up in voltage through \(-2.8\) volts, the current being collected by the RD was actually increasing. Clearly this should not be happening, and similar behavior is not observed for either of the other two trials. The one notable difference between D7.22 and the other two trials is that D7.22 was begun immediately upon completion of the 91-minute long D7.21—i.e., as soon as D7.21 was complete the RD bias was reset to 0 V and a new sweep begun; the beam was not removed from the sample surface and no other measurements were performed in the interim. By contrast, D7.18 was performed the previous day as the first measurement of the day, and D7.21 was performed \(-24\) hrs. later. It was therefore hypothesized that something in the chamber apparatus or RD charged during D7.21, and was discharging during the first few min of D7.22.

To further test this charging hypothesis, additional AR spectra—trials D7.32, D7.33, and D7.34—were run. D7.32 was the first measurement of the day and required 91 min. to complete, D7.33 was begun exactly one minute following the completion of D7.32, and D7.34 was begun forty min after the completion of D7.33 (with the beam placed in the sample FC in the interim). Results are depicted in Fig. C-3(b) and would seem to support the charging theory, as D7.33 exhibits the discharging signature and D7.34 does not. The question, then, is what is charging: is it something in the inside the magnetic shield, or something inside the RD? Charging exterior to the RD can be ruled out if the following is considered: during the short interim between D7.32 and D7.33 (and between D7.21 and D7.22), the beam remained on the sample surface—i.e., nothing exterior to the RD should have changed noticeably during the interim. Thus, if something exterior to the RD were charging during D7.32 (and D7.21), there would be no reason for it to suddenly discharge during the first few min of D7.33 (and D7.22). What did change between the end of one trial and the beginning of the next was the RD bias; it was reset from approximately \(-53\) V.
FIGURE C-3. Diagnostic AR SE spectra identifying the presence of electrostatic charging within the RD. $E_B = 1.5$ keV
to 0 V. Therefore, it would appear that whatever is charging is doing so in connection with the RD bias (that is, the bias on the FC located inside the RD).

The next question, then, is whether the charging is strictly a result of placing a negative bias on the RD FC, or whether an incident electron beam—i.e., an incident RD signal—is also required. To answer this question, one final set of diagnostic spectra—trials D7.38 and D7.39—were taken. Trial D7.38 was the first measurement of the day, and again required 91 min. to complete; D7.29 was begun 30 min. after the completion of D7.38. During the intervening period between the two trials, the beam was located in the sample FC and the RD bias remained at -50 V; after 30 min, the RD bias was set to 0 V and D7.39 was immediately begun. Results are depicted in Fig. C-3(c); they show no sign of discharging during D7.39. Thus the combined conditions of electrons entering the detector and a biased FC in the detector are required in order to effect whatever charging is occurring inside the RD; once one or other of these is removed, the discharge time is ~5–10 min.

It is hypothesized that the charging within the RD is occurring on the back surface of the RD's 2$^{nd}$ aperture (see Fig. 3-2). As the negative RD bias steps up during a spectrum measurement, fewer and fewer electrons that make it through the 2$^{nd}$ aperture are collected by the FC. The rejected electrons must go somewhere, and the back wall of the 2$^{nd}$ aperture is the most likely collecting surface. This surface was coated with colloidal graphite, and if this coating is acting as a weakly insulating layer, one can imagine how a slight negative charge might accumulate.

C.2 Sample Biasing

For an ideal geometry, negative sample biasing results in an electric field directed radially outward from the sample center to the various grounded surfaces surrounding the sample. Nominally, the effect is to energize all of the SE's emerging from the sample center along their initial trajectories (by the amount $e\Phi_S$, where $\Phi_S$ is the sample bias), reducing the deflecting affects of ambient transverse electric and magnetic fields. Secondaries emerging from the sample with trajectories initially directed toward the RD
are accelerated toward the grounded first aperture while maintaining their direction of flight; the result is an accurate AR SE yield determination, with an AR SE energy spectrum shifted by $e\Phi_S$.

AR BSE measurements should not be affected by negative sample biasing. Owing to their much greater energies, BSE’s are far less susceptible to stray fields and sample biasing, therefore, should not significantly alter which BSE’s are collected by the detector; as a result, $\eta(\alpha')$ determinations will not be affected. Furthermore, AR BSE spectra should not be shifted, as backscattered electrons originate in the primary electron beam. As the PE’s impinge on the negatively biased sample their energy is reduced by $e\Phi_S$; as they reemerge from the sample, however, and make their way to the detector, their energies are increased by $e\Phi_S$, resulting in zero net energy change. (It is interesting to note that in principle, this fact may provide a method for distinguishing between true SE’s and true BSE’s.)

In an effort to assess the value of negative sample biasing for the somewhat less-than-ideal geometry of the present apparatus, trial AR-yield and -spectra measurements were accomplished under varying conditions of negative sample bias. (It is noted the test spectra were intended to reveal large-scale trends as a function of sample bias—e.g., effects on magnitude and location of the SE peak; thus, a relatively coarse (0.5–2 eV) energy resolution was used.) Results are presented in Figs. C-4, C-5, and C-6; they reveal the following general trends:

1. Negative sample biasing does induce changes in $\delta(\alpha')$, depending on $\alpha'$; specifically, for increasingly negative $\Phi_S$, $\delta(\alpha')$ increases for $0^\circ < \alpha < -55^\circ$, and decreases for $\alpha > -55^\circ$ (Figs. C-4, C-5);

2. For $\Phi_S = 0$ volts, the function $\delta(\alpha)$ is reasonably well described by a cosine law; though for increasingly negative $\Phi_S$, $\delta(\alpha)$ deviates progressively further from a cosine law (Fig. C-4);

3. Negative sample biasing, at least through $-5$ V, has no affect on $\eta(\alpha')$ determinations, nor on the function $\eta(\alpha)$ (Fig. C-4);

4. A negative sample bias of $\Phi_S$ does energize SE’s by an amount of $-e\Phi_S$ (Figs. C-5, C-6);

5. Significant changes in AR SE spectra as a result of negative sample biasing (to about $-20$ V) are restricted to energies less than $-15$ eV—i.e., the collection of SE’s with energies $E > -15$ eV is not significantly affected by negative sample biasing (Figs. C-5, C-6).
The Effect of Sample Bias on AR Yields

FIGURE C-4. Angle-resolved SE and BSE yields as measured for surface D6 under varying conditions of negative sample bias, $\Phi_S$. Random errors are of the order of the marker sizes. Cosine fits are of the form $\delta(\alpha) = \delta(0) \cos(\alpha)$. 
Negative Sample Biasing
Surface B2

Actual Spectra

Corrected for Sample Bias

**Figure C-5.** Diagnostic AR SE spectra as measured for surface B2 under varying conditions of negative sample bias ($\Phi_s$) for emission angles of (a) $-19^\circ$, (b) $+53^\circ$, and (c) $+65^\circ$. $E_B = 1.5$ keV.
Negative Sample Biasing
Surface D1

Figure C-6. Diagnostic AR SE spectra, as measured for surface D1 under varying conditions of negative sample bias for emission angles of $-17^\circ$ [(a) and (b)] and $-19^\circ$ [(c) and (d)]. $E_B = 1.5$ keV.
These observations are generally consistent with the expected results of negative sample biasing, discussed above; collection of low-energy \( (E < -15 \text{ eV}) \) SE’s is enhanced—at least for the case of \( \alpha < -50^\circ \)—and BSE collection is unaffected. However, something is not quite right; the fact that, for \( \alpha > -55^\circ \), collection of low-energy \( (E < -15 \text{ eV}) \) SE’s decreases with increasingly negative \( \Phi_S \) is not expected. Detailed study of these and other data (not presented here) has lead to the conclusion that this affect is an artifact of the sample biasing scheme and sample-RD geometry [Nickles et al., 1999]. Specifically, the electric field produced between the sample and RD is not radially directed; rather, it is essentially perpendicular to the sample surface, as depicted in Fig. C-7. The affect of this field geometry is to focus the SE’s toward the normal—where the strength of the focusing is a function of SE energy. Furthermore, as the primary / tertiary sample geometry is not symmetric with respect to RD location about the sample (see Fig. 3-3), the effect of negative sample biasing on low-energy SE collection is not only function of emission angle, \( \alpha \), but also of RD position, \( \varphi_{RD} \). For the RD positioned generally at the front of the sample \( (0^\circ < \varphi_{RD} < -70^\circ) \), increased low-energy SE collection is observed; for the RD positioned generally to the side of the sample \( (70^\circ < \varphi_{RD} < -110^\circ) \), low-energy SE collection is essentially unaffected; for the RD positioned generally behind the sample \( (110^\circ < \varphi_{RD} < -180^\circ) \), decreased low-energy SE collection is observed. See Nickles, et al [1999] for further discussion.

**Figure C-7.** Schematic depicting the effect on SE trajectories of an electric field due to negative sample biasing.
C.3 Ambient Field Effects

Knowledge of the electrostatic field environment between the sample and RD is crucial to meaningful AR measurement analyses. Since direct measurement of ambient electric fields is not possible with this instrument, its presence and severity must be inferred through indirect evidence. In this vein, the following three behaviors were noted throughout the investigation:

1. The results of \( \delta(\alpha') \) determinations were dependant on beam-current magnitude; specifically, for \( I_B > -170 \text{ nA} \), the \( \delta(\alpha') \)'s decreased with increasing \( I_B \). The affect was not noticed for beam currents less than 170 nA, and no similar affect for \( \eta(\alpha') \) was observed;

2. The 0-V RD signal was at a maximum upon placing the beam on sample center, and would gradually decrease over the next \( \sim 2 \text{ min.} \), stabilizing at a value 6–10% below the initial reading;

3. Placement of the beam in the sample FC for the first time on a given day would result in a small but consistent return current registering on the tertiary samples—about 600 fA for an \( \sim 150\text{-nA} \) beam current. (Minimizing this “beam-in-cup” return current is, in fact, how the beam is centered in the sample FC.) With the beam remaining in the FC, the beam-in-cup return current would remain approximately constant indefinitely. Placing the beam on the sample surface for several min, however, and returning it to the FC consistently had the result of essentially eliminating the beam-in-cup return current.

These observations are consistent with the presence of a transverse electric field in the region between the sample and detector. It is hypothesized that this field is the result of slight electrostatic charging of one or more surfaces within the chamber apparatus—charging resulting from the spray of electrons created when the beam is placed on the sample surface. With the beam on the sample surface, the charging occurs over several min, and its magnitude is in some manner proportional to the beam current. The resulting ambient electric field deflects some low-energy SE’s out of the RD, accounting for points (1) and (2), and deflects stray electrons away from the tertiary samples, accounting for point (3). It is noted that (1) and (2) could also be the result of some kind of charging occurring within the RD itself, however such a thing by itself would not account for (3); thus, it appears there must be some charging exterior to the RD, within the
chamber apparatus. To further characterize the nature of the ambient $\vec{E}$—its constancy, repeatability, and strength—a number of additional diagnostic measurements were made; results are presented below.

**C.3 (a) Spectra Repeatability (Same Surface)**

Given the presence of an ambient $\vec{E}$ within the chamber apparatus, it is important to determine its variability from spectrum to spectrum; that is, does the field remain relatively constant, or does it vary dramatically? This question speaks to the reproducibility of AR spectra for a given surface, and the answer will manifest itself in the random error in each data point in the spectra. Figures C-8 and C-9 depict repeat spectra performed for several emission angles on surfaces D7 and D9, respectively; they reveal excellent repeatability for a given surface. It is emphasized that these data do not speak to the overall accuracy of the AR SE spectra, but do demonstrate that the ambient field environment within the chamber apparatus is relatively consistent from trial to trial for a given surface.

**C.3 (b) Spectra Repeatability Between Surfaces**

The nature of the ambient $\vec{E}$ within the chamber apparatus is further investigated by comparing AR spectra results for surfaces D6 and D7—surfaces prepared in a similar manner and whose AR spectra, therefore, should be similar. Results are depicted in Fig. C-10. The data reveal excellent agreement between the two sets of AR spectra for energies above roughly 5 eV; agreement for lower energies, however, is less consistent. Specifically, after correcting for the $-1.5$-V sample bias, spectra for the two surfaces differ noticeably below 5 eV for emission angles of $17^\circ$, $25^\circ$, and $35^\circ$. For the remaining two angles for which comparisons are made, $46^\circ$ and $53^\circ$, agreement in these lower energies is much better.

These results are consistent with a differing ambient electric field environment within the chamber apparatus for the two surfaces. Specifically, it is hypothesized that the transverse $\vec{E}$ is somewhat greater at the lower emission angles for surface D7, affecting the trajectories of up to 7-eV SE’s (change in the oxidized aluminum surfaces surrounding the sample is likely to lead to such a difference). In particular, the process of backfilling the chamber with argon and sputtering the sample and surrounding surfaces (the ion beamspot is considerably larger than the primary sample) is a plausible mechanism by which the charging
Repeat AR SE Spectra (Surface D7)

**FIGURE C-8.** Repeat AR SE spectra for three different emission angles on surface D7 ($E_B = 1.5$ keV, $\Phi_B = -1.5$ V)
Repeat AR SE Spectra (Surface D9)

Figure C-9. Repeat AR SE spectra performed on surface D9 ($E_B = 1.5$ keV, $\Phi_S = -1.5$ V).
Repeatability Between Surfaces

**FIGURE C-10.** Comparison of surface D6 and D7 AR SE spectra for selected emission angles. The data have been adjusted to compensate for the $-1.5$ V sample bias. $E_B = 1.5$ keV
characteristics of surfaces within the chamber apparatus might be affected. One might argue that perhaps changes in the sample surface itself are responsible for the spectra differences, and indeed such a possibility remains open. The two surfaces were not sputtered in an identical manner (see Appendix B) and it is conceivable that differences in the sputters produced the differing AR spectra. Furthermore, some level of surface contamination may be playing a role. The fact that only low-energy SE’s are being affected, however, makes a differing $\vec{E}$ a more plausible explanation. One would expect that changes in surface morphology or composition should lead to an effect on SE’s of all energies, not just the lower-energy SE’s; such an effect is in fact observed when comparing AR spectra from clean and contaminated surfaces [see Sec. C.4]. Thus it is concluded that ambient fields within the chamber apparatus are not, in general, reproducible from surface to surface.

C.3 (c) Spectra Symmetry About the Beam Axis

The sample-RD geometry is such that certain ranges of emission angles are accessible by placing the RD either to the left or the right of the normally incident primary beam. By convention, emission angles measured via placement of the RD to the left of the beam axis (as one faces the sample) are referred to as negative, while those to the right are positive. Comparing AR spectra between positive/negative $\alpha$ pairs allows for further characterization of the ambient $\vec{E}$ within the chamber apparatus; assuming a surface where SE emission is symmetric with respect to azimuthal angle [3.1(c)], any discrepancies between $\pm \alpha$ pairs should be due to asymmetries in the field environment.

With this in mind, Fig. C-11 compares a number of $\pm \alpha$ pairs for surfaces D6 and D7. While the spectra for $\alpha = \pm 17^\circ$ [Figs. C-11 (a), (c)] are in excellent agreement, results for $\alpha = \pm 19^\circ$ and $\alpha = \pm 74^\circ$ make it clear that AR SE spectra measurements are not, in general, symmetric about the beam axis. Specifically, disagreement again occurs for the lower-energy SE’s. This result is consistent with the those presented above, further supporting the notion that ambient electric fields within the chamber apparatus are having a significant impact on SE’s with energies below about 10 eV.
Symmetry about the Beam Axis

\[ \phi_3 = -1.5 \text{ V} \]

**Surface D6**

- **Figure C-11.** Symmetry check of AR SE spectra for various angles on surfaces D6 and D7 \((E_B = 1.5 \text{ keV})\).
C.4 Surface Contamination Effects

It has been noted that one effect of surface contamination through carbon deposition is to lower the total SE yield, $\delta$ [4.4]. Based on this result, one might ask whether all SE’s are affected equally, or if certain energies are attenuated preferentially. Addressing this question, Fig. C-12 depicts two AR SE spectra measurements performed on surface D6 at $\alpha = +17^\circ$—one soon after the surface was sputtered, and the other following ~23 hrs. of electron beam bombardment, when total yield measurements had decreased by ~11% (presumably due to the beam-induced ESD of carbon). Looking at the %-difference curve plotted in the figure, one readily observes that the effect of a contaminating carbon film is to reduce SE production at all energies relatively uniformly. Note that this signature is significantly different than those seen in Figs. C-10 and C-11, where the differences in like-angle spectra are assumed due to ambient field effects.

C.5 Sample Current as a Measure of Beam Current

The AR spectrum measurement technique described in [3.2(b)] requires constant beam-current measurement while the beam is located on the sample center. This is accomplished by assuming that any change in sample current is due to a change in beam current; thus, monitoring the sample current should be tantamount to monitoring the beam current. For example, a 10% increase in sample current in this process

<table>
<thead>
<tr>
<th>$I_b$ (nA)</th>
<th>% change</th>
<th>$I_s$ (nA)</th>
<th>% change</th>
</tr>
</thead>
<tbody>
<tr>
<td>-148</td>
<td></td>
<td>+95</td>
<td></td>
</tr>
<tr>
<td>-175</td>
<td>+15.4%</td>
<td>+111</td>
<td>+14.4%</td>
</tr>
<tr>
<td>-200</td>
<td>+12.5%</td>
<td>+128</td>
<td>+13.3%</td>
</tr>
<tr>
<td>-225</td>
<td>+11.1%</td>
<td>+144</td>
<td>+11.1%</td>
</tr>
</tbody>
</table>
Effect of surface Contamination on AR Spectra

FIGURE C-12. Comparison of AR SE spectra for clean and contaminated surfaces ($E_B = 1.5$ keV, $\Phi_S = -1.5$ V).
is regarded as a 10% increase in beam current. To determine the validity of this assumption, a number of sample current measurements were accomplished for varying beam currents (as measured by placing the beam in the large FC in the primary sample), and the percent change in $I_s$ compared to that in $I_B$. Results are presented in Table C.1. They show that changes in $I_s$ are indicative of proportionate changes in $I_B$ to within $\pm 7\%$.

C.6 Diagnostic Summary

Results of the diagnostic measurements presented above are summarized as follows:

1. SE's produced within the RD (as a result of SE's and BSE's entering the detector) are not a significant source of signal contamination;

2. The RD has an experimentally determined energy resolution of about $0.5 \text{ eV } \pm 0.0003E_B$;

3. Electrostatic charging within the RD results when a negative bias on the RD FC acts to filter some of the SE's and BSE's entering the detector; this charging may or may not affect AR spectra results;

4. Surfaces within the chamber apparatus are becoming electrostatically charged, stemming from the spray of SE's and BSE's within the CA when the beam is on the sample surface; SE collection is being altered by a maximum of $5-10\%$ at any given angle as a result, while BSE collection is unaffected;

5. Ambient electric fields between the sample and detector are impacting the collection of SE's with energies below $-10 \text{ eV}$; the significance of the effect appears to be greater for $\alpha' > -55^\circ$;

6. Negative sample biasing has the effect of focusing SE's toward the surface normal, resulting in anomalously high SE collection; for negative $\Phi_s$ in the range $0-5 \text{ V}$, this focusing is significant for electron energies below $-15 \text{ eV}$;

7. A sample bias of $\Phi_s = -1.50 \text{ V}$ achieves the best balance between effectively counteracting the effects of ambient electric and magnetic fields, while minimizing the focusing effect of a negative $\Phi_s$; at this sample bias, AR measurements for $\alpha' < -55^\circ$ are the most reliable;

8. Contamination appears to affect SE production roughly equally at all energies, producing a noticeably different signature than the effect of ambient fields;
9. Changes in the sample current are an excellent indicator of proportionate changes in the beam current.
APPENDIX D

DATA REDUCTION

As accomplished in this investigation, raw data for AR spectra determination consists of sample current measurements, RD current measurements, and RD bias measurements. Manipulation of the data as it is being collected is termed conditioning; manipulation of the data subsequent to its collection is termed reduction. Important details of data collection, conditioning, and reduction are reported below.

D.1 Data Collection and Conditioning

RD- and sample-current measurements were collected simultaneously by computer at 100 Hz (see [3.1(e)] for hardware and software details). The collection software then averaged 5 seconds worth of data (500 points), applied a linear correction function to each 500-pt. average (compensating for a slight systematic error incurred in the A/D conversion), and recorded the resulting RD/sample current pairs. Eleven such pairs were recorded for each RD bias over a period of 55 sec. RD bias measurements were accomplished manually, by simply reading the value off of the power supply. The standard RD biasing profile and precision is given in Tables D.1 and D.2.

D.2 Sample Current Reduction

Reduction of the raw sample current data consisted of two simple stages: filtering —i.e., removing unreliable data—and normalizing. Filtering was necessary as the first 500-pt. average in each set of 11, recorded by the computer for each RD bias, was found to be unreliable (a difficulty with the collection software); every 11th sample current measurement was therefore discarded, beginning with the first. Normalizing all of the sample current measurements of a given spectrum to a single value was accomplished as a means of correcting the raw RD currents for changes in the beam current (see [D.3] below). Choice of the specific normalization factor is unimportant from the standpoint of analysis (LB IV,
### TABLE D.1. Standard AR spectra RD biasing profile.

<table>
<thead>
<tr>
<th>Voltage Range (−V)</th>
<th>Step Size (−V)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0-1.5</td>
<td>0.50</td>
</tr>
<tr>
<td>1.75-2.00</td>
<td>0.25</td>
</tr>
<tr>
<td>2.10-4.00</td>
<td>0.10</td>
</tr>
<tr>
<td>4.25-11.50</td>
<td>0.25</td>
</tr>
<tr>
<td>12.00-16.50</td>
<td>0.50</td>
</tr>
<tr>
<td>17.50-21.50</td>
<td>1.00</td>
</tr>
<tr>
<td>23.50-53.50</td>
<td>2.00</td>
</tr>
<tr>
<td>55.0-115.0</td>
<td>5.0</td>
</tr>
<tr>
<td>125.0-165.0</td>
<td>20.0</td>
</tr>
<tr>
<td>205.0-250.0</td>
<td>45.0</td>
</tr>
<tr>
<td>350.0-1250.0</td>
<td>100.0</td>
</tr>
<tr>
<td>1300.0-1350.0</td>
<td>50.0</td>
</tr>
<tr>
<td>1360.0-1470.0</td>
<td>10.0</td>
</tr>
<tr>
<td>1475.0-1485.0</td>
<td>5.0</td>
</tr>
<tr>
<td>1486.0-1510.0</td>
<td>1.0</td>
</tr>
</tbody>
</table>

### TABLE D.2. Accuracy of RD biasing.

<table>
<thead>
<tr>
<th>Voltage Range (−V)</th>
<th>Accuracy</th>
</tr>
</thead>
<tbody>
<tr>
<td>0-1.1</td>
<td>0.03% ± 0.0065 V</td>
</tr>
<tr>
<td>1.1-11</td>
<td>0.03% ± 0.0024 V</td>
</tr>
<tr>
<td>11-110</td>
<td>0.03% ± 0.024 V</td>
</tr>
<tr>
<td>110-1100</td>
<td>0.04% ± 0.24 V</td>
</tr>
<tr>
<td>1100-1550</td>
<td>0.04% ± 1 V</td>
</tr>
</tbody>
</table>
p. 25y); as a matter of logic and convenience, sample current measurements for a given data set were normalized to the first 500-pt. average.

D.3 RD current reduction

Reduction of the raw RD current data consisted of three stages: filtering, scaling, and averaging. Filtering was accomplished in the same manner as described above for the sample current: every 11\textsuperscript{th} 500-pt. average, beginning with the first, was discarded. Scaling of the raw RD current measurements was necessary to account for changes in the beam current over the course of a spectrum. Since the RD current is proportional to the beam current, and since changes in the beam current are reflected by proportionate changes in the sample current [C.5], scaling the RD current to the normalized sample current removes the distorting effect of a changing beam current. Once scaled, the ten 500-pt. averages for each RD bias were averaged into a single 5000-pt. average, and these values divided by the solid angle subtended by the detector to give units of [nA sr\textsuperscript{-1}]; this data could then be plotted against the RD bias to get an integrated-current vs bias curve, as depicted in Fig. D-1. Differentiating this data and dividing by the initial beam current then gives the desired AR spectrum $N(\alpha', E)$ in units of [emitted electrons per steradian per eV per incident electron], also depicted in the figure. Examining the necessity of scaling the RD current measurements with the changing sample current, examples of RD current vs bias curves and AR spectra, calculated with and without this scaling, are presented in Fig. D.2.
Figure D-1. Integrated current and AR spectrum plots typical of this investigation ($E_g = 1.5$ keV, $\Phi_0 = -1.50$ V).
Importance of RD Current Scaling

Surface D7, $\alpha = +55^\circ$

Figure D-2. (a) RD current vs. bias and (b) AR spectrum depicting the importance of scaling the RD current data with the sample current data ($E_B = 1.5$ keV, $\Phi_S = -1.50$ V).
CURRICULUM VITAE

Robert E. Davies
(March, 1999)

Professional Objective
Application and development of my broad range of scientific, technical, and leadership/management skills and experience in the realms of science policy, science education, and public policy.

Education
PhD, Physics  Utah State University  (1998)
MS, Physics  Utah State University  (1996)
Fellow, NASA Graduate Student Researcher’s Program  (1995–1998)
One year graduate study, Russian and Soviet space systems, Moscow Aviation Institute and Lavochkin NPO, Moscow, Russia  (1993–94)
BS*, Meteorology  Texas A&M University  (1987)
BS, Physics  South Dakota State University  (1986)

*Although I have taken all meteorology courses required for this degree, Texas A&M requires completion of a Texas History course before formally granting a BS. I did not take such a course, and therefore am not in possession of an official BS from Texas A&M; I include it among my education credits, however, as it accurately reflects the level of my formal study in the field.

Skills and Experience
Science/Technical
- Experimental Physicist – PhD, electron-surface interactions; ultra-high vacuum physics, charged particle detection, electrical and mechanical systems design and construction, automated data acquisition, data reduction and analysis
- Space Environment / Space Physics – 4 yrs graduate study of ionospheric physics, magnetospheric physics, plasma physics, solar-terrestrial interactions
- Spacecraft-Environment Interactions – 7 yrs graduate work physics of spacecraft charging, parasitic power loss and “snapover” of high-voltage solar arrays (collaboration with Space Environmental Effects Branch at NASA LeRC)
- Charged-Particle Detection – extensive experience designing, constructing, and operating retarding potential analyzer (RPA)-type detectors
- Meteorologist – BS, 4 yrs experience synoptic and mesoscale forecasting, U.S. Air Force; meteorological satellite specialist, extensive training on/use of GOES and DMSP receiving equipment, infrared and visual imagery, uses and limitations

Space Operations
- Skipper project – 250 kg scientific spacecraft studying optical bowshock emissions: beginning-to-end project member; exposure to all phases of spacecraft design and mission planning, including vehicle propulsion, attitude control, thermal control, power systems, communications, data storage and transmission, launch and in-orbit operation
- Russian space systems – studied Russian/Soviet launch systems, spacecraft, control systems, space-based power systems, ion propulsion, lunar and interplanetary probes at Moscow
Aviation Institute (MAI); authored, two manuals on Russian spacecraft and launch systems; all work accomplished in the Russian language

- **Russian engineering/design philosophy experience** — studied astronautical engineering and design philosophy at MAI and Lavochkin NPO; active participant in joint U.S.–Russian spacecraft project “Skipper” (attitude control system), work included periodic technical interchange meetings with Russian scientists and engineers

**Leadership/Management**

- **Officer and Meteorologist, United States Air Force (4 years)** — SAC Weather Support Unit shift supervisor, personnel supervisor; project officer, major facilities and equipment upgrade project
- **Air Force ROTC (4 years)** — extensive leadership and managerial training including one semester as Cadet Corps Commander; four-year scholarship winner, Commandant’s Award winner (top Field Training graduate out of over 300 cadets), Governor’s Cup recipient (top SDSU AFROTC graduate, 1986), Distinguished Graduate and Regular Commission recipient (top 5% nationwide of 1986 AFROTC graduates)
- **PhD project manager** — conceptualized, designed, and executed PhD investigation from beginning to end, including acquisition of funding for the project (NASA Grant #NGT3-52302); managed $66,000 budget over 3 years

**Publications**


Davies, R.E., and L.D. Peel, “The Soviet Lunar Program” (Moscow Aviation Institute and Lavochkin NPO, Moscow, Russia) 155 pp., 1994


Davies, R.E., and J.R. Dennison, "The effect of negative sample bias on secondary electron emission from spacecraft surfaces," to be submitted to *Journal of Spacecraft and Rockets*, 1999