INFORMATION TO USERS

This reproduction was made from a copy of a document sent to us for microfilming. While the most advanced technology has been used to photograph and reproduce this document, the quality of the reproduction is heavily dependent upon the quality of the material submitted.

The following explanation of techniques is provided to help clarify markings or notations which may appear on this reproduction.

1. The sign or “target” for pages apparently lacking from the document photographed is “Missing Page(s)”. If it was possible to obtain the missing page(s) or section, they are spliced into the film along with adjacent pages. This may have necessitated cutting through an image and duplicating adjacent pages to assure complete continuity.

2. When an image on the film is obliterated with a round black mark, it is an indication of either blurred copy because of movement during exposure, duplicate copy, or copyrighted materials that should not have been filmed. For blurred pages, a good image of the page can be found in the adjacent frame. If copyrighted materials were deleted, a target note will appear listing the pages in the adjacent frame.

3. When a map, drawing or chart, etc., is part of the material being photographed, a definite method of “sectioning” the material has been followed. It is customary to begin filming at the upper left hand corner of a large sheet and to continue from left to right in equal sections with small overlaps. If necessary, sectioning is continued again—beginning below the first row and continuing on until complete.

4. For illustrations that cannot be satisfactorily reproduced by xerographic means, photographic prints can be purchased at additional cost and inserted into your xerographic copy. These prints are available upon request from the Dissertations Customer Services Department.

5. Some pages in any document may have indistinct print. In all cases the best available copy has been filmed.
A spin-polarized low-energy electron diffraction study of a magnetized nickel(111) surface

Mulhollan, Gregory Anthony, M.A.
Rice University, 1987
PLEASE NOTE:

In all cases this material has been filmed in the best possible way from the available copy. Problems encountered with this document have been identified here with a check mark 

1. Glossy photographs or pages 
2. Colored illustrations, paper or print 
3. Photographs with dark background 
4. Illustrations are poor copy 
5. Pages with black marks, not original copy 
6. Print shows through as there is text on both sides of page 
7. Indistinct, broken or small print on several pages 
8. Print exceeds margin requirements 
9. Tightly bound copy with print lost in spine 
10. Computer printout pages with indistinct print 
11. Page(s) lacking when material received, and not available from school or author. 
12. Page(s) seem to be missing in numbering only as text follows. 
13. Two pages numbered . Text follows. 
14. Curling and wrinkled pages 
15. Dissertation contains pages with print at a slant, filmed as received 
16. Other 


University Microfilms International
RICE UNIVERSITY

A SPIN-POLARIZED LOW-ENERGY ELECTRON DIFFRACTION STUDY OF A MAGNETIZED NICKEL(111) SURFACE.

by

GREGORY ANTHONY MULHOLLAN

A THESIS SUBMITTED IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE DEGREE

MASTER OF ARTS

APPROVED, THESIS COMMITTEE:

G. K. Walters
Professor of Physics, Chairman

F. B. Dunning
Professor of Physics and of Space Physics and Astronomy

G. S. Mutchler
Professor of Physics

Houston, Texas

April, 1987
ABSTRACT

Spin-polarized low energy electrons from a GaAs source were used to probe a magnetized nickel(111) surface. Intensity curves as well as spin-orbit and exchange induced polarization asymmetries were obtained for the (00)-20°, (00)-30°, (00)-45°, (10) and (-10) beams. Comparison with a preliminary theoretical study yields no quantitative information on the magnetic moment per surface atom.
ACK-ACK-ACK-KNOWLEDGEMENTS

Many people made this thesis possible. Professor G. K. Walters was my thesis director and provided guidance and insight during the investigation. Professor F. B. Dunning was a continual source of needed ideas and enthusiasm.

Merci beaucoup to Dr. Ali Koymen, who was my resident guru and co-worker over the months. A long-distance thanks to Dr. Keith Jamison, who built the SPES and began my journey into the land of polarized electrons. Thanks to Xia Zhang, who was always around when needed. To everyone else in the lab who provided help and guidance, my deepest thanks.

Finally, my most special thanks go to my cats Azrael and Hagatha, without whose continual support and encouragement all this would never have been possible. PPHT!
| I.  | Introduction               | 1 |
| II. | Introduction to Ferromagnetism | 2 |
| III. | Magnetism at Surfaces      | 5 |
| IV. | LEED Theory                | 7 |
|     | A.  Electrons as Waves     | 7 |
|     | B.  Coherence Area         | 8 |
|     | C.  Kinematic Theory       | 9 |
|     | D.  Dynamic Theory         | 10 |
|     | E.  Inner Potential        | 15 |
|     | F.  Inelastic Scattering   | 15 |
|     | G.  Comparing Theory and Experiment | 17 |
| V.  | SPLEED                      | 17 |
|     | A.  Symmetry Properties    | 20 |
|     | B.  Theoretical Results    | 22 |
|     | C.  Experimental Quantities | 23 |
|     | D.  Sensitivity            | 23 |
| VI. | Vacuum Apparatus           | 24 |
|     | A.  SPES                    | 26 |
|     | B.  Mott Polarimeter       | 26 |
|     | C.  Main Chamber            | 27 |
|     | 1.  SPLEED                  | 27 |
|     | 2.  AES                     | 27 |
3. Sample Mounting 28
4. Le Reste 28
D. Optical Magnetometry 30
  1. Theory 30
  2. Experiment 32

VII. The Sample 35
  A. Cleaning the Crystal 35
  B. Diffraction Peaks 41
  C. Magnetization of the Sample 41
  D. Stray Fields 42

VIII. Experimental Data and Discussion 45
  A. Data 45
  B. Error 57

IX. Conclusion 62
X. Bibliography 65
I. INTRODUCTION

Low energy electron diffraction, (LEED), has proven to be a useful tool for surface structure determination.\(^1\) Spin-polarized low energy electron diffraction, (SPLEED), which was pioneered in this laboratory,\(^2\) exploits the influence of spin-orbit scattering to provide additional information. For even moderate Z materials, e.g., copper, this probe permits more precise structural determinations.\(^3\) Spin-orbit induced asymmetries are measured using a source of partially polarized electrons. These electrons, with a polarization of \(\approx 27\%\), are obtained from a room temperature solid state source previously constructed in this laboratory.\(^4\)

Additionally, scattering due to exchange effects in a magnetic sample may be determined separately from spin-orbit induced scattering effects.\(^5\) Due to the strong surface sensitivity of electron scattering, exchange asymmetries should reflect the magnetic properties of the topmost layers. A recent theoretical study has predicted enhanced magnetic moments at two surfaces of nickel.\(^6\) Using a geometry designed to facilitate comparison with theoretical calculations, a SPLEED study of the nickel (111) surface was undertaken to test the present ability of SPLEED to extract layer by layer information on the magnetic moment per atom.
II. INTRODUCTION TO FERROMAGNETISM

Microscopically, there are three types of behavior of matter in an applied magnetic field—diamagnetism, paramagnetism and ferromagnetism. Only in the case of ferromagnetism, however, does the effect of the applied field persist after its removal.\textsuperscript{7}

Diamagnetism's cause may be viewed classically as a direct consequence of Faraday's Law of Induction, i.e., free charge carriers act to resist change in their magnetic environment. In atoms, be they neutral or ionic, an orbital current, with sign given by Lenz's Law, and hence a magnetic moment are induced; the net effect is negative susceptibility. This is far the most common behavior, exhibited by materials with no unpaired electron spins, such as most atoms, molecules, liquids and solids, or ions within ionic crystals. A positive susceptibility is found in those materials which contain a net spin imbalance or non-zero orbital momentum energy levels which a magnetic field may mix into the ground state.\textsuperscript{8} Figure 1 below illustrates the temperature dependence of diamagnetism and the various forms of paramagnetism.\textsuperscript{9}

The ferromagnetism group may be subdivided into several categories, namely, ferromagnetism, antiferromagnetism, ferrimagnetism, helical magnetism, parasitic magnetism, etc.\textsuperscript{10,11} All are characterized by a macroscopic regularity in spin orientation which persists after field removal, or in some cases spontaneously (e.g. antiferromagnetism). The distinction in spin ordering is shown diagramatically in figure 2. It should be apparent that a single species may be cross-classified magnetically, but as the order of discussion is also the ordering in magnitude of effect,
Figure 1: Temperature dependence of diamagnetic and paramagnetic susceptibilities.
Figure 2: Spin ordering in ferromagnetic-like materials.
detection of both behaviors is often difficult.\textsuperscript{13}

While microscopic qualities subdivide the ferromagnetic-like materials, the macroscopic quality which distinguishes ferromagnets is hysteresis, the memory the material exhibits of its magnetic history. A generic hysteresis curve is shown below in figure 3.

Crystalline forms of ferromagnets exhibit an anisotropy in their behavior which periodic ordering introduces. For example, the easiest direction of magnetization for nickel is along the [111] axis. Temperature affects this anisotropy energy, e.g., the aforementioned easy axis shifts to the [110] direction at approximately 100° C.\textsuperscript{15}

III. MAGNETISM AT SURFACES

Loosely, matter is 'bulk' if translation in any direction away from the region of interest in steps yields a semicontinuum. Clearly, a surface, a division between dissimilar regions, e.g., crystal and vacuum or a solid and liquid phases, does not satisfy this criterion. Such inhomogenous systems are often very difficult to treat analytically, in comparison to homogenous matter\textsuperscript{16} and in particular, crystalline materials which allow the application of periodic boundary conditions.\textsuperscript{17} Surfaces have not, however, been entirely ignored since they are of great importance in many fields\textsuperscript{18,19} and have sparked rapidly growing theoretical interest in recent years.

In addition to complexity, symmetry breakage leads to interesting phenomena.\textsuperscript{20} For example, surface crystalline and electronic band structures and magnetic characteristics can be quite different from the corresponding bulk qualities.\textsuperscript{21} Magnetically, interest was noticeably peaked
Figure 3: A generic hysteresis curve.

Region:
I. Reversible initial permeability range
II. Irreversible processes, i.e., wall displacements and rotation magnetization
III. Rotation magnetization
IV. Saturation region

$V_c$: Virgin curve, curve from initially demagnetized material
$H_c$: Coercive field, $H$ necessary to return $B$ to zero
$B_r$: Remanence field, value of $B$ persisting when applied $H$ removed.
by reports of surface 'dead' layers by Liebermann et. al. in Fe\textsuperscript{22}, Co and Ni\textsuperscript{23}, with those in nickel persisting to absolute zero. This work led to considerable controversy in the literature\textsuperscript{24,25,26,27,28}. Since this period, however, numerous spin-sensitive spectroscopies have determined the nickel (hkl) surfaces to be ferromagnetic,\textsuperscript{29,30,31,32,33} with a linear dependence of the mean surface magnetic moments on temperature.

Exact determination of a mean magnetic moment per surface atom from experimental data is not simple. Many spectroscopies are plagued with theoretical ambiguities in their interpretation, e.g. spin-polarized metastable atom deexcitation spectroscopy and electron capture spectroscopy. Other probes, such as SPLEED, which in many respects are well modeled, are computationally unwieldy for extraction of physical parameters. It is the purpose of this study to determine if SPLEED is adequate to detect degradation or enhancement of surface magnetic moment compared to that in the bulk.

IV. LEED THEORY

A. Electrons as waves

The majority of information in modern physics is obtained through scattering experiments. Since de Broglie's assertion in 1924 that matter should exhibit undulatory characteristics,\textsuperscript{34} progress of this form of experimentation developed rapidly. A consequence of this proposed wave–particle duality is a unitarily simple formula which yields the vacuum wavelength of an electron.
\[ \lambda = \frac{12.3}{E_{[\text{eV}]^{1/2}}} \, \text{Å}. \]

Since most crystalline solids have lattice spacings on the order of 4 angstroms, electrons in the 10-250eV energy range (\( \lambda \) a few angstroms) are ideal probes of such structures. Proof of electron waves was soon found by Davisson and Germer in their classical experiment,\(^{35}\) which was conducted in analogy with then known X-ray diffraction techniques since extensive bulk characteristics had, at that time, been catalogued. Further quantitative use of this technique, however, waited upon the development of easily attainable ultra-high vacuum in the laboratory.

**B. Coherence Area**

The discussion below is based on the assumption that each incident electron is well represented as a plane wave with precisely defined momentum and energy. Experimentally, however, some spread in energy and momentum is always present. This leads to a difference in phase of the incident wave for different points on the surface. Thus, it is possible to define a coherence area, coplaner with the surface, over which this phase change is \( \leq 2\pi \). Wavefunctions with phase \( \geq 2\pi \) are incoherent with respect to the reference point and do not contribute to diffraction effects. For typical electron beams, this area is \( \approx 10^4 \text{Å}^2 \). The small size limits the sensitivity of LEED to gross surface defects since each electron illuminates a separate area of the crystalline surface.\(^{37}\)
C. Kinematic Theory

The electron's charge renders it subject to strong interactions with other charged particles. One result is strong scattering probabilities for what would be in X-ray scattering considered miniscule depth penetration, i.e., a few angstroms. In this light, it is possible to construct a theory of such scattering paralleling X-ray diffraction theory, but accounting for the rapid attenuation of an electron beam due to the strength of the coulombic interaction. This is the basis of the so-called kinematic, or single scattering, theory. A full discussion, which the outline below follows, may be found in a comprehensive volume by Pendry. Consideration of only single scattering has two implications. The first is that ion-core scattering, which is the primary mechanism (valence electrons contribute little), must be relatively weak and the second is that account must be taken of rapid attenuation upon increasing depth of the incident wave function. This is done by including an imaginary term in the expression of the inner potential \( V_0 \) of the applicable Hamiltonian. Thus, even in the absence of ion cores, the incident wavefunction, \( \phi \), would exhibit absorption. As an example, this energy dependent imaginary potential takes on a value for most materials of \( \approx -4 \text{ eV} \), implying a mean free electron path of only 5 angstroms for 100 eV incident electrons.

Two equivalent \( T = 0 \text{ K} \) methods exist for computing the resultant wavefunctions. In the first, a spherical wave expansion is employed with the added requirement of a periodic ordering of scatterers (ion-cores). Alternatively, this periodicity may be taken into account a priori by forcing
a solution to obey the surface net structure, modulo reciprocal net vector, by creating a resultant wavefunction, $\phi$, through a fourier series in momentum, or $k$ space. I.E.,

$$\phi = \sum_{k'} b_{k'} \exp[i k' \cdot r],$$

where

$$k' = k'_{||} + k'_{\perp} \text{ and}$$

$$k'_{||} = k_{||} + g,$$

where $g$ is a reciprocal net vector. The latter method immediately suggests the use of another X-ray popularized tool, the Ewald sphere, which is capable of illustrating what diffraction pattern a detector, usually a phosphorescent screen in LEED, will intercept. Such a construction is shown in figure 4. Further discussion of these two methods may be found in the Dynamic Theory section along with more detail of the inner potential. The assumption of weak ion-core scattering, while valid for large unit cell noble gas crystals, is generally inadequate. Figure 5 of a theory-experiment comparison illustrates this point.

D. Dynamic Theory

The shortcomings of the kinematic theory led to the formulation of the dynamic theory, which more realistically incorporates multiple scattering into its structure. Discussions of the spin-averaged theory are to be found in works by Pendry and Woodruff. In addition to strong ion
Figure 4: Ewald sphere with reciprocal rods, as opposed to the three dimensional points, are shown emanating from the real space crystal. The location of the maxima in k space is found by the interception of a sphere of radius |k_0| with these rods. One reciprocal net vector \( a^* = b \times n(2\pi/A) \), is shown, the other, \( b^* = a \times n(2\pi/A) \), lies perpendicular to the crystal surface normal and at some non-zero angle to \( a^* \), where A is the area of the unit mesh. Complete discussion of surface crystallography and its associated labeling may be found in a paper by Wood.\(^{38}\)
Figure 5: Comparison with experiment (—) of kinematic (—) intensities reflected from a (100) nickel surface at normal incidence, (00) beam.\textsuperscript{39}
core interaction, the mechanisms of inelastic scattering and inclusion of finite temperature effects will be discussed. While the latter two topics may be included in kinematic theory, their effect is not sufficient to overcome the single scattering assumption's damage.

Once again, the scattering from the individual ion-cores is found in the usual partial wave expansion, \( f(E, \theta) = (2i \nu)^{-1} \sum_{l=0}^{\infty} (2l+1)(\exp(2i\delta)-1)P_l(\cos \theta), \)

of plane waves in spherical harmonics. For electron energies less than 150 eV is it usually sufficient to maintain only the first six to eight phase shifts in the expansion. A simple argument by Pendry suggests the largest value of \( l \) necessary is \( |k|R_m \), where \( R_m \) is the muffin-tin radius, which is defined below.

As in kinematic theory, either the \( L \) or the \( K \) representation may be used with equally valid success and difficulty. The \( L \) method employs the summation over a finite slab of material with multiple scattering represented as partial wave events until through inelastic and spherical wave dispersion the wavefunction's amplitude goes to zero. The \( K \) method treats scattering within layers as two dimensional Bloch waves and between planes as plane waves with well defined momentum. For the \( L \)-representation, the main concern is how large a slab to consider, while for the \( K \)-representation the number of \( K \) valued plane waves between layers determines the ultimate calculation time, with three dimensional matrices describing the transmission and reflection between layers. Figure 6 illustrates the beam method. By taking advantage of crystalline symmetries, it is often possible to reduce computation time by at least two
Figure 6: In the beam method, scattering between planes is represented by plane waves with well defined momentum. Within planes, scattering is treated as two dimensional Bloch waves.
orders of magnitude.

E. Inner Potential

The scattering potential within a crystal may be represented by regions of spherically symmetric ion-core potentials, and interstitial regions with constant potential. Usually termed muffin-tin, this approximation has a proven record of success in bulk band calculations.47 Figure 7 illustrates the form of this potential. The use of a constant potential between spheres is further justified since the impinging electron creates a correlation hole in the conduction or valence band electrons, yielding the assumed form between ion cores.49 Truncation of the muffin-tin potential at the crystal surface is an inaccurate representation of the actual potential. The actual form of the surface potential approximates an exponential type decay which may lead to the presence of surface states.50

F. Inelastic Scattering

Attenuation of the elastic component of the scattered electron beam is accounted for in the same way as in the kinematic formulation, by introducing an imaginary part of the inner potential, termed the optical potential when of this form. Mechanisms responsible for such losses are the creation of plasmons and magnons and scattering from phonons. Typical single phonon loss energies are of the order of 1 meV,51 those due to plasmons are approx. 10 eV52 and losses by magnon excitation are of the order of 10 meV.53 Clearly, plasmon loss is readily discriminated against,
Figure 7: Real part $V_r$ of effective electron-crystal potential, without shape approximation (——) and in muffin-tin form (•••••). $E_{vac}$, $E_F$ and $E_{mt}$ denote the vacuum, Fermi and muffin-tin levels, respectively. $V_{or}$ is the real part of the inner potential, and $\phi$ the work function. Surface potential barrier without (——) and with (•••••) image asymptotes. Muffin-tin potential approximation sketched in plane perpendicular to surface (containing the atomic nuclei (●)). $R_m$ is the radius from one ion-core to the edge of the spherically symmetric potential region.48
with energy resolution of 1eV common in LEED systems. For room
temperature experiments, phonon loss is unavoidable and it widens the
diffraction maxima in addition to causing overall intensity loss. For
spin-averaged LEED, it is sufficient to scale the individual atomic
scattering factors by \( \exp[-M] \), where \( M \) is the Debye - Waller factor, which
at high temperatures \( \propto T \), a result well founded in studies of bulk
materials.\(^{54}\) Magnon excitation cross-sections are estimated to be an order
of magnitude lower than those for plasmon excitations\(^{55}\) and hence are a
minor channel for energy losses.

6. Comparing Experiment and Theory

The simplest method for comparison between theoretical and
experimental I-V profiles is the subjective eyeball test. While this may
appear to be a crude method of judgement, no universally accepted method
exists for quantifying the quality of a theoretical fit to the data. This is
not to say, however, that various reliability factors,(R-factors), which
weigh the differences between experiment and theory according to various
schemes, do not exist.\(^{56,57}\) As a rule, the adjustable parameters in the
dynamical LEED calculation are determined by minimizing the R-factor. An
example is shown below in figure 8.

V. SPLLEED

The inclusion of spin effects into diffraction theory requires the use
Figure 8: R-factor contour as function of top layer spacing and inner potential. The presence of a local minimum determines the theoretically adjustable parameters.56
of a relativistically correct Hamiltonian.\textsuperscript{59} A discussion of the complete relativistic formalism may be found in recent compilations.\textsuperscript{60,61} Briefly, one begins with an expansion of the Dirac Hamiltonian correct to order $c^{-2}$.

\[ H = \frac{\mathbf{p}^2}{2m} + V(E, \mathbf{r}) - \mathbf{\sigma} \cdot \mathbf{B}(E, \mathbf{r}) - \frac{\mathbf{p}^4}{8m^3 c^2} + \frac{\hbar}{8m^2 c^2} \Delta V(E, \mathbf{r}) + \]

\[ \frac{\hbar^2}{4mc^2} \mathbf{\sigma} \cdot [\nabla V(E, \mathbf{r}) \times \mathbf{p}]. \]

The first three terms are the Pauli Hamiltonian, which describes the energy of nonrelativistic spin $1/2$ particles in a magnetic field; the last three terms are: (1) a relativistic mass correction term, (2) the Darwin term\textsuperscript{62} and (3) the spin-orbit coupling term, which for a central force has the form $\lambda \mathbf{\sigma} \times \mathbf{L}$. Exchange scattering arises from electron spin-spin interaction, which may be represented as spin interaction with an effective magnetic field. The third term of the Dirac Hamiltonian represents this effect, where $\mathbf{B}$ is the magnetic field within the crystal. Use of this particular Hamiltonian is consistent with the band theory of ferromagnetism implying that the resultant exchange scattering prediction will have little validity near the Curie temperature. Of course, non-magnetic systems are immune to this problem. The exact solution of the above Hamiltonian is difficult. It is possible to simplify it by constructing and solving two equivalent spin-dependent potentials in analogy with a spin-averaged one.

The methodology underlying this construction is as follows. First, the electronic structure of a ground state, ($T=0$), ferromagnetic system is solved,\textsuperscript{63} based in the method originally proposed by Kohn and Sham.\textsuperscript{64} Next, the single electron excitation, modeled as an inhomogenous system with a slowly varying ground state and spin density, is characterized.\textsuperscript{65} As a
comparison, the spin-averaged potential obtained from the spin dependent potentials varies little from the muffin-tin form. (see figure 8) Exchange and coulomb correlation induced potential shifts are found in both the spherically symmetric potential and the interstitial regions with the magnitude of the latter being typically of order 10 eV for the elemental ferromagnets. The imaginary potential responsible for beam attenuation is computed in much the same way as in the spin-averaged case, with the difference that two potentials, one for each magnetization direction, must be calculated. The abovementioned modifications are incorporated into a \( K \)-representation type formalism, the intimate details of which are found in the review of Feder.\(^{67} \) As in the LEED case, the number of beams taken between slabs is determined by the condition of convergence of the diffracted beam intensities.

A. Symmetry Properties

Without recourse to the mathematical details, which are to be found in papers by Feder\(^ {68} \) and Dunlap,\(^ {69} \) some underlying symmetry properties may be stated. The pertinent scattering geometry is shown below in figure 9. For nonferromagnetic systems, time reversal (switching positions of gun and detector) brings about the usual compliance with the reciprocity theorem, i.e., the measured total intensity and spin-orbit asymmetry experience no change. Ferromagnetic systems, however exhibit sign reversal in the exchange and unpolarized asymmetries, (defined below) Experimentally, this fact presents no difficulty. The existence of mirror planes parallel or perpendicular to the scattering plane may bring about total suppression of
Figure 9: Scattering geometry with mean magnetization direction, $\mathbf{M}$, perpendicular to scattering plane. $\mathbf{P}$ defines the polarization of the beam, which is incident at an angle $\theta$ to the surface normal $\mathbf{n}$. 
one or more asymmetries in certain beams.\textsuperscript{70}

**B. Theoretical Results**

The solution of the Dirac equation is expressed in terms of four different scattered beam intensities, which may be noted $I^{\sigma \mu}$, with $\sigma(\mu)$ taking on $\pm$ values for incident spin parallel or antiparallel to some arbitrary quantization axis (mean sample magnetization vector). These quantities may be combined into four more physically meaningful expressions:

\[
\begin{align*}
I_{\text{total}} &= I = I^{++} + I^{--} + I^{+-} + I^{-+} \\
A_{s0} &= (I^{++} + I^{+-} - I^{-+} - I^{--})/I \\
A_{ex} &= (I^{++} + I^{--} - I^{+-} - I^{-+})/I \\
A_{u} &= (I^{++} + I^{+-} - I^{-+} - I^{--})/I.
\end{align*}
\]

$I_{\text{total}}$ is simply the additive combination of all scattered intensities, $A_{s0}$ is a measure of the scattering asymmetry due to spin-orbit coupling, $A_{ex}$ is a measure of pure exchange effects and $A_{u}$ is the measure of an unpolarized beam's asymmetry in scattering from a magnetized sample upon magnetization direction reversal. Physically, this last effect may be understood in that the unpolarized beam becomes partially polarized from experiencing initial spin-orbit scattering; the resultant beam is then exchange scattered.\textsuperscript{71}
C. Experimental Quantities

While it is experimentally possible to measure the above quantities directly, the differences in absolute intensities may be quite small and any minor perturbations in the beam trajectory (such as those from residual magnetic fields) can introduce appreciable errors in the measurements. With this in mind, the experimental asymmetries most accurately measurable with the lockin technique described below are:

\[ A^\mu = (|\mu^+ - |\mu^-|)/(|\mu^+ + |\mu^-|) \]

with \( \mu = + \) or \(-\), which may be related to the calculated asymmetries by

\[ A^{50} = (A^+ + A^-)/2, \text{ and} \]
\[ A^{ex} = (A^+ - A^-)/2. \]

Both of these terms contain mixing with \( A_\nu \), but as this asymmetry is approximately an order of magnitude smaller than \( A_{50} \) or \( A_{ex} \), its effect is negligible to first order.\(^{72,73}\)

D. Sensitivity

SPEEED is no less subject to thermally diffused scattering than conventional LEED.\(^{74}\) As in the dynamic model, thermal effects may formally be accounted for by the addition of a decay term in scattered intensities, although the validity of this is questionable. Clearly, the presence of multiple scattering sites limits the usefulness of this
approximation, particularly in the case of the polarizations. In fact, in a
kinematic model (single scattering), $A_{so}$ would not even change with
temperature. Temperature dependence in $A_{ex}$ is mainly due to the overall
decline of the magnetization with increasing temperature.

In general, the exchange asymmetry is not directly proportional to the
surface magnetization. Certain points in energy, however, may belie this
fact. As an example, $A_{ex}$ as calculated by Tamura et al. exhibits a jump
from 15% to 25% at 20 eV when the topmost layer's magnetic moment per
atom changes from the bulk value to an enhancement of 50%. The
corresponding I and $A_{so}$ are entirely unaffected by this adjustment
indicating that structural shifts may be determined from intensity and
spin-orbit information alone, with $A_{ex}$ reflecting a measure of surface
magnetism.

VI. VACUUM APPARATUS

Most components of the apparatus are housed within three
differential-ion pumped stainless steel chambers which are pictured below
in figure 10. The spin-polarized electron source, (SPES), chamber and main
experimental chamber routinely maintain $1 \times 10^{-10}$ torr base pressures, while
the Mott chamber, which is not bakable, only reaches $1 \times 10^{-8}$ torr. Details of
construction and subsequent evolution of each may be found in previous
works, with a brief overview below presenting current modifications.
Figure 10: Schematic of vacuum apparatus showing relative locations of SPES, main experimental chamber and Mott polarimeter.
A. SPES

The SPES provides the experimenter with a clean, vertically partially polarized beam of electrons. Cleanliness is accomplished through the use of a solid state electron source as outlined by Pierce et al.\textsuperscript{79} The particularities of this laboratory's version may be found in the dissertation of Jamison.\textsuperscript{80} To achieve emission with alternating up (+) and down (−) spin polarization, five hundred hertz helicity modulated 807 nm radiation from an AlGaAs diode laser\textsuperscript{81} is focused on a negative electron affinity, (NEA), GaAs(100) p-doped crystal face, whose NEA state has been brought about by a previously deposited Cs-O layer. M\textsubscript{j} selection rules allow theoretical emission of +/-50% polarized electrons, but this apparatus, at room temperature, typically yields polarizations, \((n_{up}-n_{down})/(n_{up}+n_{down})\), of 27+/−2%. The polarization of the beam is determined through the use of a 100kV Mott polarimeter.

B. Mott Polarimeter

The theoretical principles behind the Mott scattering technique are thoroughly described in Kessler.\textsuperscript{82} Basically, the polarization of an electron beam may be measured by detecting asymmetries in the electron current backscattered from high Z materials. Spin-orbit coupling causes this left-right anisotropy. The location of the Mott chamber and accelerating column are shown in figure 10 and a detailed description of its operation may be found in O'Neill's PhD dissertation.\textsuperscript{83}
C. Main Chamber

1. SPLEED

The beam from the SPES is used in conjunction with a commercial LEED optics and faraday cup assembly as shown above. This system allows the experimenter to collect conventional LEED I-V profiles simultaneously with polarization asymmetry measurements by modulating the incident beam's polarization. The unmodulated (unpolarized) faraday cup signal corresponds to 1/2 of the spin-averaged signal, while the 500 hz signal detected via a lock-in amplifier, gives the difference term, \( I_{\text{up}} - I_{\text{down}} \), as defined above.

2. AES

A second set of LEED screens, four grids with a coaxial electron gun, is positioned in the Mott-ward side of the chamber. Conventional LEED patterns may be viewed with this set, but its main purpose is to conduct Auger Electron Spectroscopy (AES). A high energy electron, typically 2-5 keV, initially core-ionizes the target atom. The resultant hole is filled by decay of a higher energy level electron in an Auger process, where by an energy-conserving second electron is simultaneously ejected.

The kinetic energy of the emitted electron is characteristic of the emitting atom. Extensive tabulations for the elements exist, rendering the detection of Auger electrons a sensitive chemical analysis tool. It is possible to detect the Auger current versus energy by modulating the retarding grid's potential slightly, e. g., a 2 \( V_{pp} \) signal on a potential of 250 Volts, and detecting the resultant fundamental frequency current. Doing so, however, results in the search for very small signals within a large
background signal since a grid system operates as a high pass filter. It is much easier to detect the derivative of the current signal since by the mean value theorem, a signal peak must pass through a region of zero slope-implying the existence of positive and negative going derivative peaks. Additionally, the background's contribution is greatly suppressed. Practically, this taking of the derivative is accomplished by phase-sensitive detection of the second harmonic signal. A simple Taylor series expansion shows that for good energy resolution (of order 1 eV), this harmonic is almost exclusively composed of the signal's derivative.\textsuperscript{66}

3. Sample Mounting

The sample is mounted on the end of an off-axis manipulator described elsewhere.\textsuperscript{67} The motion in the $\phi$ direction was, however, disabled since its presence was not required. The nickel crystal was separately mounted in an Armco C-shaped magnet. See figure\textsuperscript{11} for a schematic. The magnet serves to saturate the crystal's initial magnetization, allow for its reversal and to act as a keeper for minimizing stray magnetic fields. The difficulties with such a circuit will be addressed in the experimental discussion.

4. Le Reste

Ion milling, or sputtering, may be carried out by the use of a commercial ion gun. Pressures are measured using a nude ion guage or quadrupole mass spectrometer. The latter is important for monitoring inlet gas purity. A minor structural change was effected in the gas handling system to enable rapid switching of gasses available at the variable rate
Figure 11: Armco C-magnet mount with crystal (hatched region) in place.

The magnetizing field is applied (reversed) by driving (reversing) current in the coils.
leak valve. This change is shown diagramitically below in figure 12.

D. Optical Magnetometry

1. Theory

In order to conduct experiments on a magnetic sample, it is necessary to characterize its magnetic state. One way of doing this is to take advantage of the change in polarization of light reflected from a magnetic surface. Non-magnetic materials reflect linearly polarized light without change. Magnetized surfaces, however, perturb the polarization's direction and phase, giving rise to elliptical polarization. Detection of this phenomenon, the magneto-optic Kerr effect, (MOKE), or its close associate, reflectance magneto-circular dichroism, RMCD, gives a direct measure of the magnetic condition of the sample. Macroscopically, the effects may be attributed to two different complex indices of refraction for right and left circularly polarized light. Such a decomposition is plausible since a linearly polarized wave may be written as the superposition of left and right circularly polarized waves. In nonferromagnets, Zeeman splitting of the energy levels is the major contributor to changes in the conductivity tensor, which may be related to the index of refraction. Ferromagnets, however, in which the average angular momentum is quenched, are unaffected by the Zeeman splitting since a first order expansion yields it proportional to the average angular momentum. Instead, a shift in the wavefunctions themselves, via spin-orbit interaction, gives rise to the different indices.
Figure 12: DIN type diagram of gas handling system. The large chamber is the main experimental chamber. The gas bottles contain argon and oxygen. Note the presence of the sorption pump which enables changing of the gasses.
2. Experiment

Three different geometries exist for magneto-optic measurements as shown and discussed in figure 13. Two slightly different arrangements were used to detect the RMCD from a bulk nickel crystal with the transverse geometry. The first\textsuperscript{92} employs the use of right and left circularly polarized light, while the second\textsuperscript{93} employs the time honored method\textsuperscript{94} of a direct measurement of change in reflectivity. Both set-ups are shown below in figure 14.

In the first arrangement, 632.8 nm radiation from a helium-neon laser passes through a prism polarizer set at 45° to the fast axis of a 50kHz photoelastic modulator.\textsuperscript{95} After reflection, the beam passes through an analyzing polarizer, by which as much of the 50kHz signal is canceled as possible and then through a condensing lens to focus the beam on the face of an AC coupled photodiode. This output is fed to a PAR 124A lock-in amplifier set for the fundamental, which is, for small scattering angles (9.5° in our case), dependent only on RMCD.\textsuperscript{96} The resultant signal then goes to an X-Y chart recorder, whose abscissa is scanned with the magnetizing field. Particular to this method is a high sensitivity of the modulator to beam drift.

The second and simpler method employs the same beam path with (1), the removal of the modulator, (2), the initial polarizer being set at 90° to M and (3), the insertion of a DC-coupled photodiode whose output is fed to an electrometer with variable coarse zeroing.\textsuperscript{97} The electrometer's output then drives the ordinate of the recorder. The analyzer is initially set slightly off from extinction and any change in reflectivity, which may be treated as an effective rotation, shows up as a DC signal at the photodiode
Figure 13: Three possible geometries for measuring the Kerr effect. The polar effect, with $\mathbf{M}$ perpendicular to the surface, is the largest effect. The longitudinal (transverse) effect has $\mathbf{M}$ in plane and parallel (perpendicular) to the scattering plane. The ordering in magnitude is the order of listing.$^{91}$
Figure 14: Modulator and DC type RMCD measurement systems. The modulator(DC) system consists of all component blocks shown excepting those outlined in dash-dots(dashes). The components are:

I. Helium-Neon laser
II. Polarizing Polarizer
III. Piezo-electric Modulator
IV. Sample-magnetized perpendicular to plane of paper
V. Analyzing Polarizer
VI. Condensing Lens
VII. AC(DC) coupled photodiode
VIII. Lock-in amplifier
IX. Electrometer
X. x-y Chart Recorder
output. Due to DC coupling, and the small size of the signal, all ambient light must be kept from the diode's surface.\textsuperscript{98}

**VII. THE SAMPLE**

The sample was obtained from Cornell University,\textsuperscript{99} with both sides cut and polished to within .5° of the (111) plane as determined by a Laue camera. Magnetization was along the [1\textit{TO}] axis, which is nickel's second easiest magnetization direction. None of the (111) normals are coplaner with the (111) plane. This disallows relaxation of the chosen magnetization axis into the easiest direction due to surface anistropy energy.\textsuperscript{100} Figure 15 shows the physical orientation of the crystal and how it is cut from a bulk crystal. The (1\textit{TO}) plane, which is the scattering plane for this geometry, is also a mirror plane for the fcc lattice.

**A. Cleaning of the Crystal**

It is known that of the three most commonly studied faces of nickel, the (111) face is the easiest to clean, although for high enough bulk concentrations, the resultant surface carbon concentration may take the form of a graphitic layer due to the matching of the (111) and planer graphite lattice constants.\textsuperscript{101} After pumpdown and bakeout, the surface contaminants shown by AES were sulphur and carbon. One hour of sputtering with a 600 eV beam followed by an anneal to 575°C was sufficent for permanent sulphur removal. Two methods exist for permanent surface carbon removal. Normal Ar\textsuperscript{+} sputtering is the first and desorption as CO by
Figure 15: (a) Orientation of the (111) plane with respect to the face centered cubic lattice. (b) Crystallographic orientation of the sample. The C-magnet is attached to the narrow ends of the crystal. The outlined plane is the scattering plane.
titration with O₂, is the second. Both of these methods were employed. An initial oxygen treatment of 1x10⁻⁸ torr of O₂ with crystal temperature of 300°C, was successful in fully removing the surface carbon with no detectable O₂ residue on the surface. Subsequent Ar⁺ sputtering, however, which is necessary to remove contamination by residual gasses in the experimental chamber, caused the carbon feature to reappear. While it seems plausible that methane from the ion pumps, which are switched off during sputtering, was being cracked on the surface by the ion beam, a comprehensive study has shown this to not be the case.¹⁰² Partial pressure of CO was below the 1x10⁻¹¹ torr range and could not contribute to fast carbon accretion. Since the crystal temperature was kept at 300°C, we did not expect any oxygen to be incorporated into the bulk¹⁰³ and hence any carbon depletion would be at the surface and near surface only.

For this reason, we had to utilize a method to draw the bulk carbon to the surface where it could be removed by Ar⁺ sputtering or desorbed as CO. To use this process effectively, it is necessary to understand the mechanisms of carbon migration within the crystal. Three distinct temperature regimes may be defined. The high temperature, or 'lattice gas,' phase is the lowest in surface coverage due to dissolution into the bulk. Second is the intermediate-temperature segregation region, where uniform carbon coverage exists in the presence of a surface and solid reservoir, in analogy to condensation at a surface from a surrounding gas. At low temperatures, carbon precipitates from the bulk onto the surface. The exact values of the segregation and precipitation temperatures are bulk concentration dependent.¹⁰⁴ Thus, we might expect to clean the crystal by cooling it to precipitate carbon from the bulk and then sputtering it away.
Reheating then permits the diffusion of deeper bulk carbon to the near-surface region, from which it is brought to the surface by cooling, etc. The decision to use 600°C as the bulk diffusion temperature was governed by the need to use a temperature higher than the expected daily anneal temperature of 400°C. The anneal temperature was chosen to be above nickel’s Curie temperature (358°C), and was limited by the rather inefficient heating achievable with the large C-magnet assembly. These cycles were continued until the carbon Auger feature was approximately one half its initial size.

In an effort to speed the removal of the carbon, one prolonged four hour session of 500°C, 2x10⁻⁸ torr O₂, CO desorption was utilized, since we could remove the carbon atoms diffusing to the surface without actually sputtering the surface away. Figure 16 illustrates the carbon AES signal as a function of time. At these elevated temperatures, O₂ diffusion into the bulk is possible, but it was not seen by AES. As expected, the carbon peak reappeared after cooling the crystal, but two 1keV beam sputters followed by anneals to 600°C decreased the carbon coverage to below 2%, i.e., to within the noise level of the AES system. A comparison of the clean and dirty nickel AES spectra is shown below in figure 17. All subsequent sputters were conducted at 600eV with a 400°C anneal to remove surface imperfections. The anneals were accomplished by simple radiative and electron bombardment heating. The anneal temperature was held for only five minutes, but the 1.5 hour long cooling period to room temperature proved to be sufficient time at an elevated temperature to eliminate sputter damage as was seen by the sharpness of the LEED pattern.
Figure 16: Auger signal of 272 eV carbon feature as a function of exposure time in an oxygen atmosphere.
Figure 17: (a) AES spectrum of nickel sample prior to any cleaning. The peaks due to sulphur, carbon and nickel are indicated by the elemental symbol. (b) AES spectrum of clean crystal. Location of diffraction peaks is shown along with high energy nickel features.
B. Diffraction Peaks

The presence of diffraction peaks which might be mistaken for elemental peaks in the AES spectra requires an explanation. Diffraction peaks have been observed in nickel\textsuperscript{105} and copper and cobalt\textsuperscript{106}. The energies for which these peaks are seen closely correspond to the energies where peaks in X-ray absorption are observed. Such peaks occur at energies for which the ejected photoelectron has an optimum energy and momentum direction for propagation through the crystal. This explains the size of the AES signal at these energies; the effective escape depth for secondaries will be larger for those matching diffraction conditions. For nickel, the observed diffraction peak energies are 121, 140, 161, 173, 204, 241, 290 and 388 eV. The peak locations are noted in figure17. Additionally, the sharpness of these diffraction peaks is indicative of surface cleanliness and surface order. This serves as an additional, albeit extremely qualitative, check for a clean and well structured surface.

C. Magnetization of the Sample

The magnetic behavior of the sample was characterized before insertion into vacuum and after completion of the experiment. Changing the crystal's magnetic state was accomplished using a slowly ramping DC current into the coils shown in figure 11. RMCD measurements determined the currents (fields) necessary to achieve saturation. Initially a current of 10 amperes was required, but the value as measured after removal from vacuum had decreased to 1.0 ampere. Several factors could help to bring
about this change.

First, impurity concentration can drastically affect the saturation field in the regime of low concentrations. The removal of most bulk carbon enables saturation to be reached with a smaller field as shown below for iron in figure 18. Second, it is possible that better mechanical contact between the keeper and crystal resulted in a higher flux into the crystal. However, the order of magnitude of such an effect is too small to account for the observed change (e.g., an electromagnet with a deliberate air gap may exhibit a 40% loss of flux from the gap vicinity)\textsuperscript{108} Third, the role that magnetostriction played in the crystal's behavior must be considered since the sample was clamped on the edges. With continued annealing, we expect the crystal to move so as to minimize its stress. This would result in an increase in the saturation field required since nickel has negative magnetostriction.\textsuperscript{109} Finally, it is possible that when the annealing process removed surface damage the magnetic properties of the crystal were altered. The first hypothesis seems most probable since our initial carbon peak showed no decrease upon heating to 500°C (this corresponds to \(\approx 0.01\%\) carbon), and after final cleaning, no carbon precipitation was detected at room temperature (\(\leq 0.001\%\) carbon).\textsuperscript{110}

D. Stray Fields

Stray magnetic fields must be minimized to avoid bending the probing electron beam. A simple calculation shows that for fields below 30 milligauss, the total deflection in our apparatus is \(\leq 1\text{mm}\) for 10 eV electrons and even lower for higher energy electrons. This corresponds to
Figure 18: Effect of impurities on magnetic properties of iron. Annealing at 1400°C in hydrogen reduces the carbon content from about 0.02% to less than 0.001%.107
an angular deviation of less than .25°, a value which is acceptable for LEED measurements. However, Hall-probe measurements demonstrated .5 gauss stray vertical field 2mm from the sample face, which clearly had to be reduced.

The field's origin may be understood by a simple argument. The C-magnet provides a path by which flux is routed through the crystal. If the coercive field of the magnet, $H_{c \text{-magnet}}$, were zero, then only flux due to the crystal would be present in the magnetic circuit after the magnetizing current was turned off. For $H_{c \text{-magnet}}$ nonzero, we must add to the flux due to the crystal, flux from the C-magnet. Since the nickel crystal had achieved saturation, any additional impression of flux 'leaked' out from the crystal. This same argument, however, suggests a way to reduce these stray fields provided the coercive field of the nickel crystal, $H_{\text{nickel}}$, is larger than $H_{c \text{-magnet}}$. Application of $-H_{c \text{-magnet}}$ to the system reduced the flux due to the C-magnet to approximately zero, while maintaining the crystal's saturation state. The size and direction of the measured stray field did not appreciably change during the course of the experiment.

The necessary current to reduce the stray fields to an average value of 20 milligauss was measured to be 1.0 amperes, the same value as measured for $H_{c \text{-magnet}}$. It should be clear, however, that the crystal's change in $H_{\text{nickel}}$ to 1.0 amperes was problematic. Reproducability difficulty was first noted in the exchange asymmetry of the (-10) beam. By choosing a point in energy where the spin-orbit asymmetry was zero and scanning the crystal horizontally, regular variations in the magnitude and sign of the signal from the lock-in were observed. These regions were postulated to correspond to oppositely oriented domains since all the asymmetry signal would be due to
exchange scattering only. It was not possible to test this assumption using RMCD measurements since the crystal could not be scanned in this probe's direction while in situ. RMCD measurements from different parts of the crystal, however, confirmed saturation in the individual domains. A horizontally scanned RMCD measurement, performed after sample removal, confirmed this, resulting in the following variation in signal across one vertical position as seen in figure 19a. Both SPLEED asymmetry and RMCD measurements indicated several domains of alternating direction of over 2mm in width and running nearly the length of the crystal. The 'fracturing' of the crystal into magnetic domains is explicable by the hysteresis loop shown in figure 3. The backdriving current brought the crystal to the value of $H_c$, as is shown in figure 19b, a region of domain rotation and wall migration.

VIII. EXPERIMENTAL DATA AND DISCUSSION

A. Data

Intensity and spin-orbit and exchange asymmetry versus energy curves were taken for the (00) beam at 20°, 30° and 45° included angles and the (10) and (-10) beams at normal incidence. They are shown below in figures 20-34. Error bars are indicative of statistical deviations between separate data collections. All data have been shifted for the difference in work functions between the GaAs source and the nickel crystal (5.35eV) and have had the intensity contribution from diffuse scattering subtracted.
Figure 19: (a) Domain widths as determined by RMCD measurements. The hatched and clear regions correspond to oppositely oriented domains. The RMCD signal from each is equal in magnitude to that from the magnetized, but not backdriven, crystal. (b) Hysteresis curve obtained from nickel sample with modulator type RMCD measurement. Note that this single pass signal shows drift over the time (10 s) required for its acquisition.
Figures 20-34: Intensity and spin-orbit and exchange asymmetry versus energy plots for the (00) beam at 20°, 30° and 45° and the (10) and (-10) beams at normal incidence. Curves are (---) θ-1°, (---) θ, and (●●●) θ+1°. Theoretical curves (---) are shown where available.\textsuperscript{114} Asymmetry bars shown on the (00) 45° beam are after Kuyatt.\textsuperscript{113}
Ni(III) (10) Spin-Orbit Asymmetry

Energy [eV]

Asymmetry [??]

30 40 50 60 70 80 90 100 110 120 130 140 150 160
Ni(111) (10) Exchange Asymmetry

Asymmetry [X]

Energy [eV]
As a test of spin-orbit and exchange asymmetry separation, data was collected with the sample demagnetized by an AC current. The resulting spin-orbit asymmetry is shown with that from the magnetized sample for the (00) 30° beam in figure 35 and it illustrates agreement within our limits of reproducibility. Heating above the curie temperature was not sufficient to demagnetize the sample since small residual fields from the keeper would remagnetize the sample as it cooled.

Early intensity data collected by Demuth and Rhodin\textsuperscript{111} for the (00) 20°, (10) and (-10) beams, shown below in figure 36, compares favorably with our own. For historical completeness, a comparison of the (00) 45° intensity curve is made with two obtained by Davisson and Germer in the 1920's\textsuperscript{112} and their actual values of the spin-orbit asymmetry\textsuperscript{113} (probably instrumental asymmetries) are indicated in figures 37 and 27.

A preliminary theoretical study\textsuperscript{114} of the Ni(111) system is also shown below and compared to our data. While agreement is good in some regions, it is not sufficient to discern between a surface magnetic moment value per atom equal to the bulk value or enhanced over the bulk value by 10%. It is hoped that further progress in SPLEED theory will enable extraction of a surface magnetic moment per atom from available data, but at present, this is not possible.

B. Error

The problems associated with calibration of the SPLEED asymmetry signal due to variable instrumentation gains and statistical uncertainty have been previously discussed.\textsuperscript{115} Two possible sources of error remained.
Figure 35: Comparison of spin-orbit asymmetry obtained from extraction process(—) and from demagnetized sample(—) for (00) beam.
Figure 36: Early intensity data for (00) 20°, (10) and (-10) beams.\textsuperscript{111}
Figure 37: Davisson and Germer's and this experiment's (error bars) intensity data for (00) 45° beam.¹¹²
Spurious deflection caused by stray magnetic fields has been discussed above. We tested for this effect by examining the intensity signal for different magnetization directions. Insignificant variance was seen. The remaining source was uncertainty in the orientation of the sample.

First, the method used to set normal incidence is intrinsically subject to error. While the \( \phi \) motion of the manipulator was disabled, some change in this angle is possible by using the tilt platform of the manipulator. Rotation of \( \theta \) through 90° will show any deviation of \( \phi \) with respect to the LEED screen horizontal. Accuracy of setting \( \phi = 0^\circ \) is estimated to be \( \pm 1.0^\circ \).

Setting \( \theta = 0^\circ \) must be done by an indirect method. This is because at normal incidence, the specular beam returns along the incident beam path and hence is not visible on the phosphored screen. The total angle through which the crystal must rotate from beam disappearance to reappearance was measured to be \( \approx 4.0^\circ \). Uncertainty in this angle arises from difficulty in detecting the reemergent beam. Half this angle must be the amount necessary to be added to the disappearance angle to achieve normal incidence.

Finite beam width and varying incident beam current contribute to uncertainty in the exact point of disappearance. These limit this method's accuracy and reproducibility. To account for the absolute uncertainty, data was taken over three 1° increments, including the calculated value of normal incidence. Reproducability was better than 0.5°, as is seen in the (-10) beam 97 eV intensity feature.
IX. CONCLUSION

A spin-polarized low energy electron diffraction experiment was undertaken to test this probe's ability to determine surface layer by layer magnetization. An NEA GaAs crystal was used as a source of polarized electrons. The sample, a nickel (111) crystal, was magnetized using a C-shaped magnet. A sufficient quantity of data was obtained for comparison with theory. These studies do not, as of yet, yield quantitative information on the magnetic moment per surface atom. SPLEED, however, is capable of determining if a surface is magnetized. This feature may prove useful in future studies of the magnetic properties of ultra-thin films, which recent theoretical\textsuperscript{116} and experimental\textsuperscript{117} works have shown to possess novel magnetic characteristics.

In view of SPLEED's current inability to yield quantitative information on surface magnetic moments, its usefulness as a probe in future studies must be assessed with respect to other possibilities. Among these are spin- and angle-resolved photoemission (SPARPES), ECS, SPMDS, torsional magnetometry and polarization- and angle-resolved secondary electron spectroscopy (PARSELS).

SPARPES shares with SPLEED a well developed theoretical framework and the characteristic of signal-averaging over the first few surface layers. Presence of an in-house He-I (hv = 21.2 eV) light source together with (soon) a miniature Mott polarimeter makes study of surface ferromagnetism induced band splitting a definite possibility. As with SPLEED, no current photoemission spectrum calculations exist for magnetic adlayers on non-magnetic substrates. Unlike SPLEED, however, it is possible to make
(albeit rough) direct inferences as to the magnetic state of such adlayers from measured polarizations.

The two probes which sample the exponential tail of surface electronic wavefunctions, ECS and SPMDS, both provide measures of long range ferromagnetic order. ECS can, in two electron capture, measure short range order \( \approx 10-20\text{Å} \). Both of these methods are not fully theoretically developed, but they do yield data relevant to the surface magnetic moments. These probes currently exist in this laboratory.

Each of the above probes was dependent on the surface in question being exposed to vacuum. In torsional magnetometry, this is not necessary. So-called sandwiches—consisting of alternating layers of magnetic and non-magnetic elements—may have their layer-integrated moments and anisotropies measured in this way. It is extremely difficult to measure monolayer moments using this method.

PARSELS is relatively new as a surface probe. Universal curves predict large \( \approx 30\text{Å} \) escape depths for zero kinetic energy secondaries. This would seem to limit their surface specificity. However, recent studies have been indicative of short \( \approx 5\text{Å} \) escape depths.\textsuperscript{116} Experimentally, this probe is attractive since true secondaries are present in quantities two orders of magnitude higher than elastically scattered electrons. Additionally, depth profiling may be accomplished by varying the primary electron energy as well as the analyzing energy. Separation of loss features from the true secondary emission may be accomplished by modulating the sample potential independent of primary source and analyzer potential. Detection of this modulated signal then must yield only the true secondary current. The loss dependent current will be independent of the modulation
since the loss features are fixed with respect to the primary beam energy.

For ease in detection and depth profiling, PARSELS appears to be the most attractive probe for future use. In addition to probing the magnetic properties of the topmost layer, it would enable us to study the variance in magnetic moment through thin films and superlattices with reasonable spatial resolution perpendicular to the surface. The study of the angular dependence of secondary emission from non-magnetic surfaces should be in itself an interesting topic for future research. However, angular-dependent polarization information from magnetic surfaces will yield insight into the anisotropy properties of surfaces and thin films.
BIBLIOGRAPHY


39. Pendry, 1974, p. 84.


43. Pendry, 1974.


47. Ashcroft, et al., chapter 11.


50. Ziman, p. 196.


52. Pendry, 1974, p. 27.


54. Ziman, chapter 2.


59. Lind, et al.
60. Feder, 1985, chapter 4.
70. Feder, 1985, p. 150.
74. Rabbiteye blueberries, however, are heat resistant.
76. Tamura, et al.


81. Mitsubishi Electric Corporation # ML5401A.


84. Varian Model C 981-0127/98/-0137


86. Woodruff, et al., chapter 3.1.3.1.


91. Carey, et al.


93. Bader, et al.


96. Sato.

97. Keithley Electrometer Model 610C.

98. It is interesting to note that the 60 hz modulation in the room fluorescent lights may be seen at these signal levels with even small light leakage.

99. Addis, B. Materials Science Dept., Cornell University, Ithica, N. Y.

100. Gradmann, p. 179.


114. Feder, R., Private communication.


