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QUANTITATIVE ANALYSIS OF THIN FOILS
USING BACKSCATTERING MEASUREMENTS

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ABSTRACT

Backscattering of 1.25 MeV protons and alpha particles
from thin foils is used for a non-destructive quantitative ele-
mental analysis of thin foils. The detection of helium that
had previously been implanted into a 1350 $\overset{o}{A}$ palladium foil and
the analysis of a composite aluminum and parylene foil are
presented as examples of this technique.

INTRODUCTION

Backscattering of charged particles from an accelerator is a well-known method that has been used for several years to analyze material surfaces.[1-5] It has also been used to analyze ion-implanted layers[6] and, more recently, to analyze 300 to 2000 Å thick amorphous oxide layers.[7] This technique readily lends itself to the analysis of surfaces and can be used to detect and measure small amounts of elements present.

In this paper we discuss the application of back-scattering measurements to the elemental analysis of thin foils. This method was used to analyze 600 to 3500 Å thick, freestanding, polycrystalline foils, and provides for a rapid, non-destructive, quantitative analysis of the sample. Analyses of two specific foils are presented as examples. The first case uses backscattering of 1.25 MeV protons to detect helium that had previously been implanted into a 1350 Å thick palladium foil.[8] The second case uses backscattering of 1.25 MeV alpha particles to quantitatively analyze a composite aluminum and parylene foil.

By converting the energy scale of a backscattered spectrum to an atom-mass scale, we can identify the elements associated with a target provided that they are located close

to its surface (as shallow dopants or as surface components in thick targets or when associated with foils). The scale conversion may be accomplished using the following formula, based simply on the kinetics of elastic scattering:

$$\frac{E}{E_o} = \left[\frac{M_1 \cos \theta_s + (M_2^2 - M_1^2 \sin^2 \theta_s)^{\frac{1}{2}}}{M_1 + M_2} \right]^2 \qquad . \tag{1}$$

This relationship is discussed by Mayer, Eriksson, and Davies.[4] E_o and E are the energies of the projectile just prior to and immediately after the scattering event. M_1 is the mass of the projectile and M_2 the mass of the target atom. θ_s is the scattering angle in laboratory coordinates.

This energy-mass relationship is insensitive to small changes in θ_s in the backward direction. In the present experiment, we may assume $\theta_s = 180°$ without a significant loss of accuracy and thus simplify the conversion expression to:

$$\frac{E}{E_o} = \left[\frac{M_2 - M_1}{M_1 + M_2} \right]^2 \qquad . \tag{2}$$

Table I lists the calculated values of E/E_o for $\theta_s = 160°$ and 180° for both protons and alpha particles ($M_1 = 1.01$ and 4.00) for several values of M_2 calculated using [Equation 1]. Comparison of the E/E_o values in Table I supports the use of the simplified relationship for target masses of 4 or greater and shows the insensitivity of the backscattering angle.

The system was calibrated by backscattering from thick
(> 1 micron) samples of Be, Al, Cu and Au. These particular
elements were chosen because of their availability and the fact
that their masses range the periodic table. The backscattered
spectra from these elements obtained by using a 1.25 MeV alpha
particle beam are shown in Figure 1. These spectra exhibit typical
thick-target backscattering in that any element present, which is
lighter in mass than the substrate, occurs as a small peak on the
plateau produced by the substrate. An example of this is the
oxygen peak seen in the aluminum spectrum in Figure 1. Thick-
target backscattering spectra normally mask the effects of a
lighter mass element causing its detection to be difficult;
however, when a heavy mass element is present in a lighter mass
substrate it may be associated with an easily detectable peak
that lies to the right of the substrate's edge. An example of
this is the carbon and oxygen peaks appearing in the beryllium
spectrum in Figure 1.

A calibration curve can be established by associating
the channel number at the half-plateau value of a thick sample
spectrum to the calculated E/E_0 ratio. When a thin sample
(e.g., 1000 Å thick) like a foil or thin oxide layer is used
instead of a thick substrate, the channel number associated
with the peak in the spectra is used. Using copper as an

example, we can calculate the E/E_0 ratio expected for an alpha
particle projectile to be:

$$\frac{E}{E_0} = \left[\frac{M_2 - M_1}{M_1 + M_2} \right]^2 = 0.776$$

and if E_0 = 1.25 MeV, E = 0.97 MeV. Thus the copper edge or
half plateau value occurring at channel number 101 corresponds
to an energy ratio of 0.776 or a final energy of 0.97 MeV. In
a similar manner, we may calculate the remainder of the energy
ratios for the spectra shown in Figure 1. These values, along
with their corresponding channel numbers, are listed in Table II.
These values establish a calibration curve for an incident beam of
1.25 MeV alpha particles, and this curve of E/E_0 vs. channel
number is shown in Figure 2.

As an aid in identifying unknown peaks in a back-
scattering spectrum, the E/E_0 ratios for elements expected to
be present should be calculated and plotted on the calibration
curve. The channel numbers associated with these E/E_0 ratios
can be read directly from the calibration curve and can serve as
an identification aid during the analysis. The E/E_0 calculation
is simply reversed to identify unknown peaks that do not appear
at a calibration point or at an expected element channel number.

To determine the absolute amounts of elements present,
we must know the elastic scattering cross sections. In most cases,

this may be calculated using the Rutherford scattering relation-
ship. While, in principle, absolute measurements can be made
directly (this involves the measurement of the integrated beam
current and the solid angle subtended by the detector), in practice
one usually normalizes to a known calibration standard. The
areas under the peaks in the backscattered spectrum then provide
us with relative measurements. That is, if we know the absolute
amount of one of the elements present or we have a calibrated
sample, we can calculate the amount of the others present
since we can determine their relative areas from the spectrum.

The Rutherford scattering relationship

$$\frac{d\sigma}{d\Omega} = 1.3 \times 10^{-27} \left(\frac{Z_1 Z_2}{E_o}\right)^2 \left(\frac{M_1 + M_2}{M_2}\right)^2 \frac{1}{\sin^4 \theta_{s/2}} \tag{3}$$

is also discussed by Mayer, Eriksson and Davies.[4] In this
relationship M_1 is the projectile mass, M_2 is the target atom
mass, and Z_1 and Z_2 are their respective atomic numbers. θ_s is
the laboratory scattering angle and E_o is the initial energy of
the projectile. This expression, based on simple Coulomb
scattering, is valid provided we are using alpha particles and
scattering from targets with masses of carbon or greater.

The Rutherford scattering relationship may be simpli-
fied to provide a backscattered yield for a thin target. Since

we have a fixed geometry we can reduce this expression to

$$Y \propto \left[\frac{Z_1 Z_2}{E_o}\right]^2 \left[\frac{M_1 + M_2}{M_2}\right]^2 X_2 \quad . \tag{4}$$

Y is the backscattering yield for a particular target element, and X_2 is the number of atoms/cm^2 for that element. The yield Y corresponds directly to the area under a peak in the back-scattering spectrum.

Care must be exercised when backscattering from the lighter elements since both nuclear reactions and nuclear elastic scattering must be considered. Nuclear reaction components may introduce false mass peaks into the spectra or may enhance the area under one or more of the peaks in the backscattered spectra. The analysis for elements heavier than boron is free from these effects when alpha particles are used; however, care must be used when measuring the absolute amount of carbon present with a proton beam since the nuclear scattering cross section for this event is larger than the value predicted by Rutherford scattering theory.

EXPERIMENTAL DETAILS

The foils were analyzed by measuring the energy of the back-scattered particles from an incident beam of 1.25 MeV protons or alpha particles provided by a 2 MeV Van de Graaff accelerator. Particle beams produced in an accelerator are

easily collimated in both energy and position. This allows us to look at a small solid angle of the backscattered particles and to assume that the energy spread caused by kinetics is small compared with the energy resolution of the detection system.

The target foils were positioned perpendicular to the incident beam in a 10^{-6} torr range vacuum chamber as shown in Figure 3. A partially depleted, silicon, surface-barrier detector with an active area of 25 mm^2 and a resolution of 16 keV at full width half maximum for alpha particles was positioned 13 cm from the target at a scattering angle of 166°. The output of the detector was fed through a charge-sensitive amplifier and a pulse shaping network to a multichannel pulse-height analyzer. A schematic diagram of the electronics setup is shown in Figure 4. This combination provided for a rapid data collection system with a minimal background and a linear response for backscattered particles with energies of 200 keV to 1.5 MeV.

The target foils were bombarded for varying periods of time until approximately 5×10^4 counts were accumulated in the primary peak channels. Reliable target bed -current measurements were not obtained, but the target current was believed to be on the order of a nanoampere. (This is based on a beam current of 1 to 3 micro amperes on the final collimator, 0.08 cm in diameter, positioned 16.5 cm from the target.)

RESULTS

^{4}He$^+$ Ions in a Pd Foil

The energy spectrum of 1.25 MeV protons backscattered
from a thin (1350 Å) Pd foil is shown in Figure 5; ^{4}He$^+$ ions to
a dose of less than 2×10^{16} ions/cm^2 had previously been
implanted into the foil. This spectrum is typical of that from
a thin (600 - 3500 Å) foil and consists of sharp, well defined
peaks; a peak due to the presence of helium is clearly seen.
The thick-target spectrum in Figure 5 is presented for comparison
purposes; it illustrates the helium peak that one would observe
if a thick Pd target had been implanted with ^{4}He$^+$ ions to the
same depth and dose as in the thin foil.

The He peak in the backscattered spectrum is greatly
enhanced over Rutherford scattering predictions due to a large
nuclear scattering cross section for protons on ^{4}He. The only
experimental cross section data available[9] show that for 1.73 MeV
protons on ^{4}He the elastic scattering cross section is more than
10 times greater than the Rutherford cross section. Using known
cross sections or calibrations standards, the backscattering
here could be made quantitative.

6 μg/cm^2 of Al on Parylene

A composite foil of 6 μg/cm^2 of aluminum deposited upon
a thin parylene* foil was analyzed using a 1.25 MeV alpha particle

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* Parylene N (C_8H_8), a product of Union Carbide Corporation.

beam. The backscattered spectrum is shown in Figure 6; for comparison purposes, the backscattered spectrum from a 2000 Å thick, free-standing parylene foil is shown in Figure 7.

Comparison of the backscattered spectrum with the values contained in Table II allows us to identify the large peaks in Figure 6 as Al, O, and C. A calculation similar to the one described earlier allows us to verify that the small peak appearing between the C and O peaks as nitrogen. Since these peaks represent thin samples, the channel number associated with the peak is used for identification.

Using the yield [Equation 4] and the fact that the area under the Al peak in Figure 6 corresponds to 6 $\mu g/cm^2$ of that material, we can quantitatively analyze this foil. The absolute amount of oxygen present is calculated as an example of this method of analysis. Putting the values for Z_2 and M_2 into Equation (4) for both O and Al provides:

$$Y_{Al} = 223.1 \frac{X_{Al}}{E_o} \text{ and } Y_0 = 84.5 \frac{X_0}{E_o} \quad .$$

The ratio of the areas under these peaks in Figure 6 gives $Y_{Al}/Y_0 = 9.2$. Using Avagadro's number allows us to convert the amount of aluminum present to atoms per square centimeter. Thus 6 $\mu g/cm^2$ corresponds to $X_{Al} = 1.34 \times 10^{17}$ atoms/cm^2. Thus, from the above:

$$x_C = \frac{(223.1)\,(\text{\dots}\cdot 10^{17})}{(64.5)\,(\text{\dots})} = 3.9 \times 10^{16} \text{ atoms/cm}^2.$$

Thus the initial amount of oxygen present is 3.9×10^{16} atoms/cm^2. In a similar manner we can calculate the amount of nitrogen to be 2.0×10^{14} atoms/cm^2 and carbon to be 9.4×10^{17} atoms/cm^2. Assuming unit density this amount of C implies that we have a parylene layer approximately 1800 Å thick. This calculated thickness is in close agreement with the thickness (2000 ± 200 Å) of a similarly prepared parylene foil that was measured with an interferometer.

CONCLUSION

Charged particle backscattering has been used as a non-destructive tool for the quantitative elemental analysis of thin foils. It is especially suited to the detection, measurement, and study of the lighter elements when they are associated with a heavier mass substrate. An incident beam of 1 to 1.25 MeV protons or alpha particles may be used to sample sub-surface as well as surface layers of foils of up to 3500 Å thick. The technique has the capability of detecting and measuring elements present in concentrations of less than a tenth of a mono-layer (10^{14} atoms/cm^2) with a mass of helium or greater and therefore is a valuable asset in studying diffusion (Ref. 4, p. 137) of the lighter elements in heavier mass foils.

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Figure Captions:

Figure 1: 1.25 MeV alpha particle backscattered spectra from thick samples of beryllium, aluminum, copper and gold used for calibration. The $\natural$ symbol indicates the half-plateau or peak value used to define the calibration curve shown in Figure 2.

Figure 2: Calibration curve for backscattered spectra measured with a 1.25 MeV incident alpha particle beam.

Figure 3: Schematic diagram of experimental assembly.

Figure 4: Block diagram of the electronics assembly.

Figure 5: Backscattered spectrum of a 1350 $\overset{\circ}{A}$ Pd foil that had been implanted with $^4He^+$ ions. Data taken at θ_s = 160° with a surface barrier detector. An incident beam of 1.25 MeV $^1H^+$ ions was provided by a Van de Graaff accelerator. The dashed curve represents the backscattered spectrum of a thick Pd sample that had been implanted with $^4He^+$ ions to the same depth and dose as the foil. Approximate counting time 10 minutes.

Figure 6: Backscattered spectrum of a composite foil of 6 $\mu g/cm^2$ of Al deposited upon a layer of parylene. Data taken at θ_s = 160° with a surface barrier detector. An incident beam of 1.25 MeV $^4He^+$ ions was provided by a Van de Graaff accelerator. Approximate counting time 20 minutes.

Figure 7: Backscattered spectrum of a 2000 Å parylene foil. Data taken at θ_s = 160° with a surface barrier detector. An incident beam of 1.25 MeV $^4He^+$ ions was provided by a Van de Graaff accelerator.

Table Captions:

Table I: Calculated values of the final to initial energy ratio
(E/E_o) for both protons and alpha particles backscattered from
selected target masses at laboratory scattering angles of 160°
and 180°.

Table II: Calculated E/E_o ratios for the calibration element
spectrums shown in Figure 1 with their associated channel numbers.
The E/E_o ratios were calculated for alpha particles with an initial
energy of 1.25 MeV at a laboratory scattering angle of 180° using
the relationship:

$$\frac{E}{E_o} = \left[\frac{M_2 - M_1}{M_1 + M_2}\right]^2 \quad .$$

TABLE I

M_2	Protons $M_1 = 1.01$		Alpha Particles $M_1 = 4.00$	
	$\theta_s = 160°$	$\theta_s = 180°$	$\theta_s = 160°$	$\theta_s = 180°$
	E/E_O	E/E_O	E/E_O	E/E_O
4.00	0.367	0.360	– –	– –
9.01	0.646	0.640	0.156	0.148
16.00	0.783	0.778	0.371	0.360
32.06	0.884	0.882	0.614	0.605
107.87	0.964	0.964	0.866	0.878
180.95	0.973	0.976	0.918	0.916
196.97	0.976	0.980	0.924	0.922

TABLE II

Element	E/E_{o}	Channel Number
Be	0.148	30
C	0.250	42
O	0.360	54
Al	0.550	75
Cu	0.776	101
Au	0.922	117

TARGET FOIL

COLLIMATOR

INCIDENT BEAM

1.25 MeV
$^1H^+$ or
$^4He^+$

$\theta_s = 160°$

BACKSCATTERED
BEAM

DETECTOR