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EVALUATION OF A TECHNIQUE FOR MEASURING ION ENERGY LOSS IN VAPOR-DEPOSITED MATERIALS

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16. Abstract

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SUMMARY

A technique for measuring ion energy loss values for vapor-deposited materials has been investigated. The technique employed vapor-depositing the desired thickness of material on a floated gold substrate target. The target was then placed in a proton beam while silicon semiconductor detectors monitored simultaneously the initial and exit beam energy. The investigation covered a wide range of target thicknesses (2 to 25 μm) and energies (0.5 to 3.4 MeV). A computer program was developed to reduce the data and correct for the gold substrate and the finite sample thickness. A comparison of these data with values reported in the literature indicates agreement to within ±1.6 percent over the tested ranges. Although the technique has been evaluated for protons in aluminum, it is expected to yield accurate results for other ions and/or materials.

INTRODUCTION

The problem of how energetic charged particles lose energy while traveling through matter has been under examination for many years. Both theoretical and experimental approaches have been applied to this problem and have resulted in much success. The Bethe (refs. 1 and 2) stopping-power equation determines the stopping-power function in terms of appropriate parameters over certain energy ranges. However, the problem that requires more investigation (ref. 3) is the attainment of good experimental and theoretical agreement in the lower energy ranges (less than 5 MeV/amu). Additions to the Bethe equation in the form of electron-shell corrections have refined it so that its values agree with experimental values over a wide range of particle energies. At the lower energies (less than 2 MeV/amu) the shell correction terms which are experimentally determined will add their uncertainty to the stopping-power equation. It has been found, however, that the data scatter for stopping-power experiments is as large as 10 percent for some materials (ref. 3). Therefore, more accurate experimental data are necessary to augment the theoretical equations in the low energy region.

In many cases, past experiments have used rolled or hammered target foils whose thickness must be questioned when the foil thickness is on the order of micrometers. This is one cause for error that may have introduced the discrepancies found in reported data. Also, it is physically difficult and in some cases impossible to obtain self-supporting films for such thicknesses. One successful technique has been the use of vapor-deposited films in place of the rolled and hammered foils. However, to make these films self-supporting, a supporting substrate must be dissolved. This is difficult and impractical for some materials. In this report, the desired target material was vapor-deposited on an inert gold film (0.1 micrometer thick) which supported the sample. The use of the inert gold film allows the examination of any vapor-deposited material regardless of its chemical activity.

In other experiments, spectrometers have been used to obtain energy measurements. Although these are accurate, they are difficult and time consuming to use. For the present method, surface barrier energy detectors with special very thin windows replaced bulky spectrometers. These detectors are both accurate and efficient and thereby reduce the amount of time necessary to obtain experimental data.

The purpose of this report is to evaluate this method for obtaining energy loss (dE/dx) measurements. A comparison of these data with other published data is made for dE/dx results of protons in aluminum, for which accurate data are known (ref. 3). The energy range investigated is between 0.500 and 3.400 MeV.

SYMBOLS

E_F degraded proton energy

E_0 incident proton energy

ΔE average energy lost by proton in traveling through sample

dE/dx rate of energy loss

E_1 energy of Am^{241} alpha particle

E_2 energy of alpha particle as measured by detector with source angle of 0

E_3 energy of alpha particle as measured by detector with source angle of θ

$\Delta E_\alpha = E_2 - E_3$

Δx sample thickness

Δx_1 thickness of inactive gold window on detector

θ angle measured from detector normal to alpha source

EQUIPMENT

Accelerator

The source of energetic protons used in this experiment was a 4-MeV Van de Graaff particle accelerator and its associated beam transport system (fig. 1). An analyzing magnet (mass-energy product 16) in conjunction with a slit stabilizing system maintained the beam energy to within ±2 keV of a set energy. Since semiconductor particle detectors were used to measure the beam energy, it was necessary to obtain an independent energy calibration for the system. This was done with a nuclear magnetic resonance (NMR) technique (ref. 4) to measure the magnetic field of the analyzing magnet. The absolute energy calibration depended on two proton-neutron threshold reactions:

At 1.565 MeV,

$$^{51}_{23}V + ^{1}_{1}H \rightarrow ^{1}_{0}n + ^{51}_{24}Cr$$

and at 1.881 MeV,

$$^{7}_{3}Li + ^{1}_{1}H \rightarrow ^{1}_{0}n + ^{7}_{4}Be$$

Since the direct proton beam flux was too high for the detector equipment, it was necessary to decrease the beam intensity from 10^{13} to 10^{4} particles/cm^2-sec. This was accomplished in the target chamber, shown in figure 2, which contained a 0.034-cm-diameter aperture and a 0.1-μm-thick gold scattering foil. Only those protons which scattered 45° from the beam direction were active in the experiment. The energy of the protons was corrected for the energy lost during scattering. After the scattering, the low-intensity monoenergetic protons impinged normal to the sample and to the energy detector.

Sample Construction

Each sample consisted of aluminum vapor-deposited onto a 0.1-μm-thick gold support layer. First, the gold layer was produced by a separate deposition onto a soap-covered optical slide and its thickness was measured by interference methods (ref. 5).

The gold film was then transferred to a sample holder (fig. 3) by floating the gold from the slide in water. The gold substrate was placed over the 1.4-cm^2 hole on the sample holder. Then, the desired sample thickness of aluminum was vapor-deposited onto this gold support. Simultaneously, the vapor-deposited aluminum was being collected on an optical monitoring slide. After each deposit, the thickness of aluminum on the monitoring slide was measured by interference techniques. Therefore, the thickness of aluminum on the gold substrate was determined. After obtaining the desired aluminum thickness, the sample was clipped over a detector and placed in an evacuated target chamber (fig. 3). As shown in this figure, a direct-beam aperture existed in the sample holder. With this geometry, the energy detector would simultaneously measure not only the proton energy through the sample (E_F) but also the incident proton energy (E_0). As with the scattering foil, corrections for the energy lost in the gold support layer were taken into account.

Energy Detector System

Charged particles that enter the energy detector produce pulses whose amplitude is a linear function of the particle energy. These pulses were first amplified and then fed into a multichannel pulse-height analyzer. Figure 4 shows the biased output of this analyzer for a typical experiment. The quantity "channel number" is a calibrated function of particle energy for a specific gain of the electronics system. To ascertain that this gain remained constant throughout the duration of the experiment, an external precision pulse generator was coupled to the electronics system. Prior to and following each test, pulses with amplitudes of 0.50, 1.00, and 2.00 (arbitrary units) were fed into the system. The channel into which these pulses fell would determine the electronic linearity as well as any system gain-drift. The actual pulses from the detector are labeled in figure 4 as E_0 and E_F. The quantity $\Delta E = E_0 - E_F$ is defined as the average energy lost by a proton in traveling through the sample. If Δx (sample thickness), E_0, and ΔE for $0.500 \text{ MeV} < E_0 < 3.400 \text{ MeV}$ are known, it is possible to determine the dE/dx function over that energy range by calculation.

Corrections to Pulse-Height Data

It was necessary to correct the data from the multichannel analyzer. The three corrections took into account energy loss in the gold scattering foil, in the gold sample support subfilm, and in the inactive gold energy-detector window. The first two of these were simple to correct. Since these gold films were made and measured in this laboratory, the film thicknesses were accurately known from interference measurements. With these thicknesses known, the energy loss in the films was taken into account to correct the quantity ΔE. It was more difficult, however, to determine the necessary correction for the inactive gold window on the energy detector. This was accomplished,

though, by use of a separate experimental setup which is shown in figure 5. In an evacuated chamber, a collimated alpha particle source (Am^{241}) produced a beam incident on the energy detector. By measuring the alpha-particle energy with the detector and changing the incident angle from 0 to θ degrees, an energy shift $\Delta E_\alpha = E_2 - E_3$ was measured. It is possible to express the energy read by the detector with the beam incident at θ degrees as

$$E_3 \approx E_1 - \frac{dE_1}{dx}\frac{\Delta x_1}{\cos\theta}$$

where: E_1 is the initial alpha-particle energy, dE_1/dx is the rate of alpha-particle energy loss in gold, and Δx_1 is the thickness of the inactive gold window. Thus, the thickness of the inactive gold window can be expressed as

$$\Delta x_1 = \frac{\Delta E_\alpha \cos\theta}{1 - \cos\theta}\left(\frac{dE_1}{dx}\right)^{-1}$$

where dE_1/dx is assumed to be constant over Δx_1. This assumption is correct for small Δx_1, which is applicable here. The thickness of gold on the detector as determined by this method was less than 0.02 μm and resulted in less than $\pm$0.02 percent error.

CALCULATIONS

The corrected pulse-height data were used in the calculations of the final dE/dx function. These calculations involved a complete set of measurements of ΔE, Δx, and E_0 over the energy region of interest. For this experiment, increments of 0.500 MeV were used in the region 0.500 to 3.000 MeV except for the highest energy run at 3.400 MeV. To determine the dE/dx function, it is necessary to consider the following integral:

$$\Delta x = \int_{E_F}^{E_0} \left(\frac{dE}{dx}\right)^{-1} dE \qquad (1)$$

From equation (1), it is now assumed that the $(dE/dx)^{-1}$ function can be expressed as a polynomial expansion function. Therefore, $(dE/dx)^{-1}$ has the form

$$\left(\frac{dE}{dx}\right)^{-1} = \sum_{i=0}^{N} a_i F^i \qquad (2)$$

where a_i represents the undefined coefficients and $F = f(E_0)$. Therefore, applying equation (2) to equation (1) results in

$$\Delta x = \int_{E_F}^{E_0} \sum_{i=0}^{N} a_i F^i \, dE \tag{3}$$

For each of the M test runs, this integral was evaluated and an $N \times M$ matrix containing the undefined coefficients was obtained. Therefore, the form of the solution is expressible as

$$\Delta x_j = \sum_{i=0}^{N} a_i \left(F_j\right)^i \qquad (j = 1,\ldots,M)$$

If $M >> N$, it is possible to apply a least-squares fit to the solution matrix and thereby obtain values for the coefficients a_i. Since $\lim_{\Delta x \to 0} \frac{\Delta E}{\Delta x} = \frac{dE}{dx}$, the integral in equation (3) was divided by ΔE and the limit taken. Therefore, equation (3) becomes

$$\frac{dE}{dx} = \lim_{\Delta x \to 0} \frac{\Delta E}{\Delta x} = \frac{\Delta E}{\int_{E_F}^{E_0} \sum_{i=0}^{N} a_i F^i \, dE} \tag{4}$$

The evaluation of this equation with the computer-determined values for a_i is the final result of this experiment.

RESULTS AND DISCUSSION

Since this is an investigation of a technique, it is necessary to compare the results with those of other published papers. For example, Janni (ref. 6) has compiled an extensive listing of proton dE/dx values in various materials. His report compiles results from both experimental data and theoretical calculations so that the tabulated values below 1.0 MeV represent smoothing and interpolating (and in some instances extrapolating) of a significant amount of current available data. Table I shows a comparison of data for aluminum from Janni (ref. 6), the present report, Allison and Warshaw (ref. 7), Barkas and Berger (ref. 8), Bichsel (ref. 9), and Nielsen (ref. 10).

Care must be exercised in using vapor-deposited materials because it is necessary to use a sufficiently thick sample to obtain bulk material density. Otherwise, the film-thickness measurements will indicate a thicker sample than actually exists. For example,

since bulk density for vapor-deposited aluminum is obtained in samples thicker than 1.6 μm (ref. 11), no aluminum samples thinner than this value should be used. In this report, the sample thicknesses were from 1.8 to 25 μm. With these samples, the maximum deviation between data from this report and these of Janni was +1.6 percent at 0.500 MeV. Between 1.0 and 3.4 MeV the maximum deviation was ±0.9 percent.

In the present investigation, the total experimental error is less than ±2 percent. The absolute experimental error is broken down as follows:

Parameter	Percent error	Cause of error
E_0	±0.02	Beam slit width
ΔE	±.7	Detector system resolution
Corrections to raw data	±.1	Scattering foil and sample support
Δx	±1.2	Sample thickness resolution

It is expected that the use of this technique with other ions and/or materials will also result in accurate data.

By use of a computer program, the experimental dE/dx results have been reduced to equation (4), which may be applied to the energy region 0.5 to 3.4 MeV to give dE/dx results within the specified error. From equation (4) then,

$$\frac{dE}{dx} = \frac{\delta\, 10^3}{\sum_{i=1}^{4} \left[A_i Y^i\right]_{E_0-\delta}^{E_0+\delta}} \tag{5}$$

where dE/dx is in keV/μm and

E_0 = incident proton energy

Y = variable of integration

δ = small energy increment ($\approx$5 keV)

and the coefficients A_i are to be evaluated as

$A_1 = 3.280$ $\quad$ $A_3 = -0.433$

$A_2 = 4.250$ $\quad$ $A_4 = 0.047$

In figure 6 is shown a plot of this function where the solid line indicates the tested region. Note that there are no inflection points in the dashed extrapolated segment of the curve. The smoothness of the dE/dx function indicates that evaluation of equation (5) in small energy increments is a valid operation.

CONCLUDING REMARKS

A technique for measuring ion energy loss in vapor-deposited materials has been investigated. The technique utilizes silicon energy detectors and self-supporting samples vapor-deposited on a gold substrate. The results obtained are in the form of an energy loss (dE/dx) equation for protons in aluminum which may be evaluated with an experimental error of ±2 percent in the region of this investigation (between 0.500 and 3.400 MeV). A comparison of this data with Janni's published data indicates an overall deviation of ±1.6 percent. Although the technique has been evaluated for protons in aluminum, it is expected to yield accurate results for other ions and/or other materials.

Langley Research Center,
National Aeronautics and Space Administration,
Langley Station, Hampton, Va., August 8, 1969,
124-09-21-04-23.

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TABLE I.- COMPARISON OF REPORTED VALUES OF dE/dx

FOR PROTONS IN ALUMINUM

Proton energy, MeV	Values of dE/dx, keV/μm, from –					
	Present report	Ref. 6	Ref. 7	Ref. 8	Ref. 9	Ref. 10
0.500	69.1	68.0	67.5	---	---	---
1.000	46.8	47.0	47.8	---	46.7	---
1.400	38.0	38.0	36.5	---	37.8	---
1.596	34.8	34.8	---	---	---	34.5
2.000	30.1	29.9	30.2	30.3	29.9	---
2.032	29.8	29.6	---	---	---	29.2
2.400	26.5	26.3	---	---	---	---
2.495	25.7	25.7	---	---	---	25.9
3.000	22.5	22.5	---	---	22.5	---
3.400	20.3	20.5	---	---	---	---

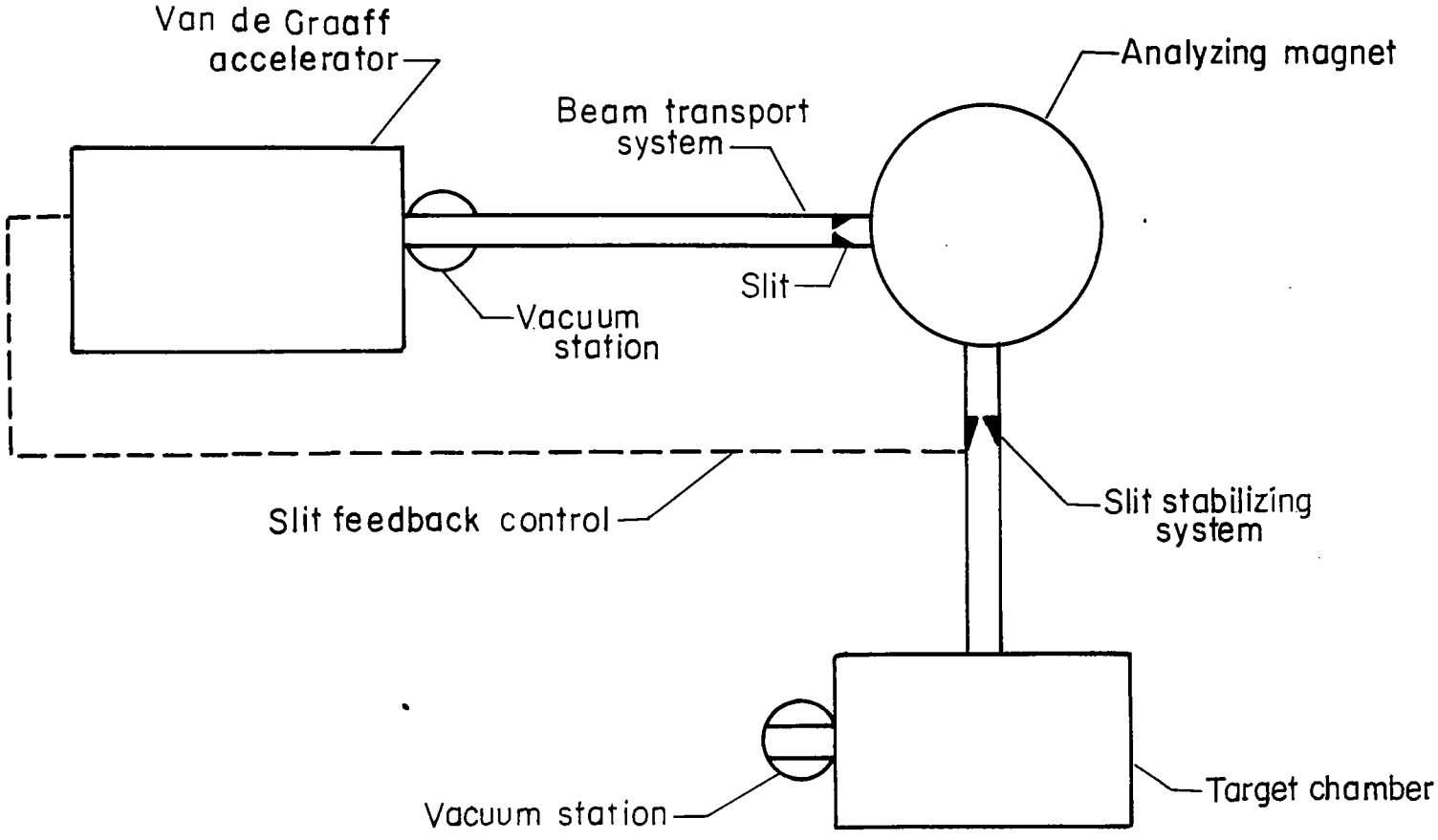

Figure 1.- Test equipment.

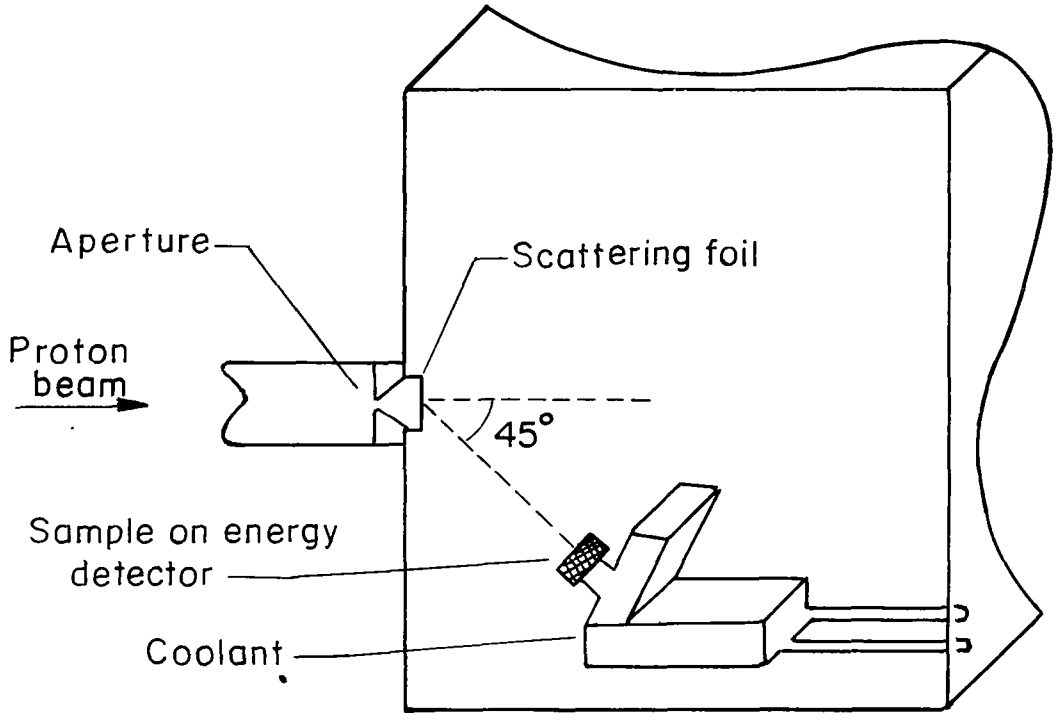

Figure 2.- Vacuum target chamber.

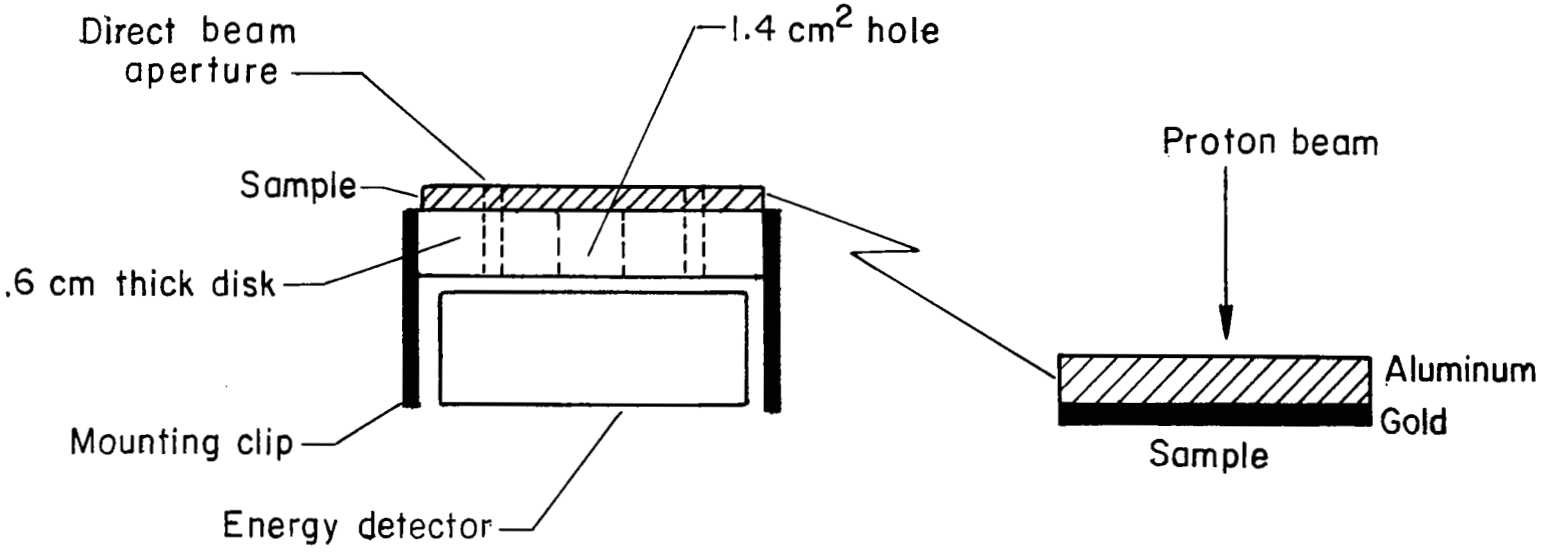

Figure 3.- Sample holder (exaggerated sample size).

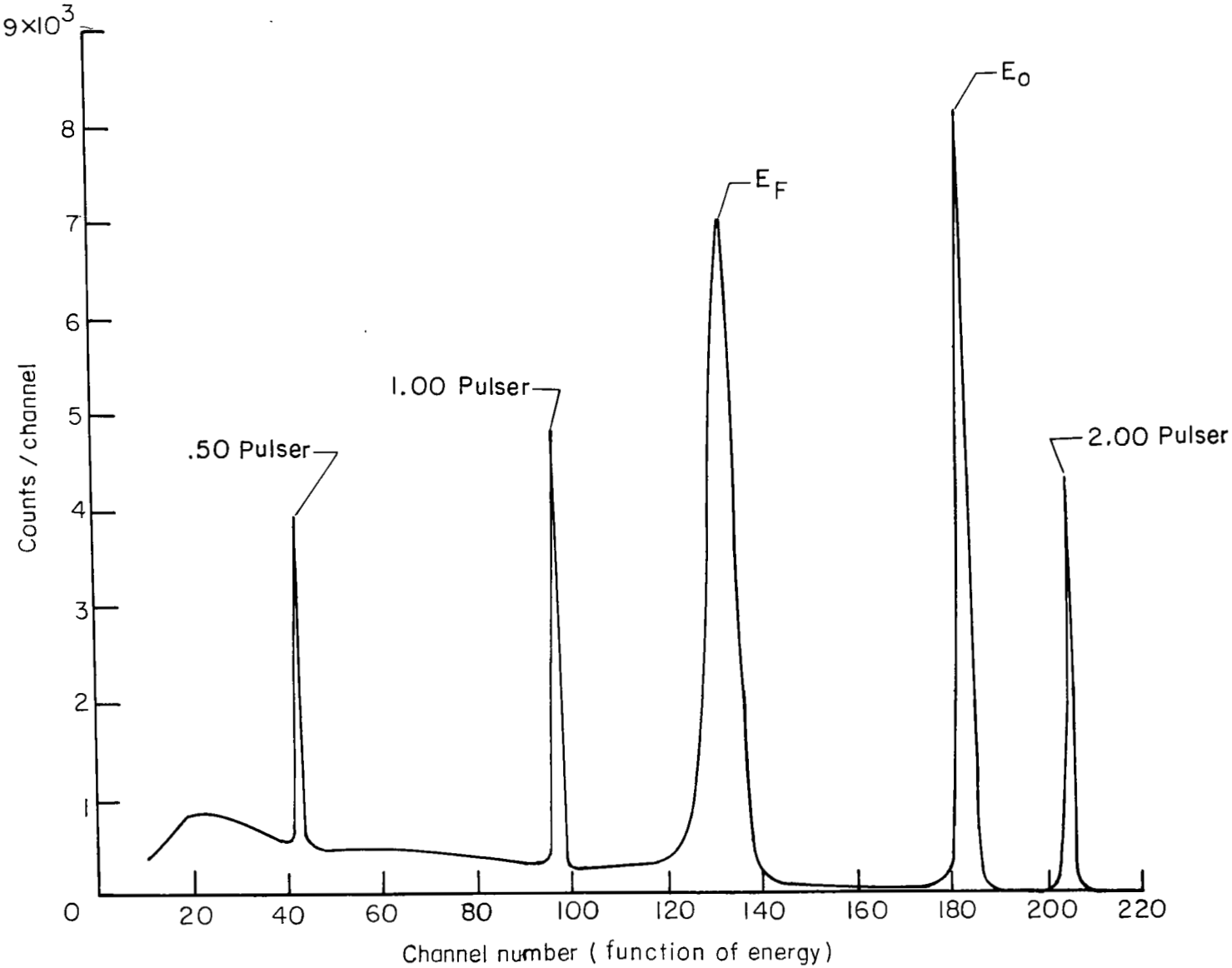

Figure 4.- Output of pulse-height analyzer for typical test run.

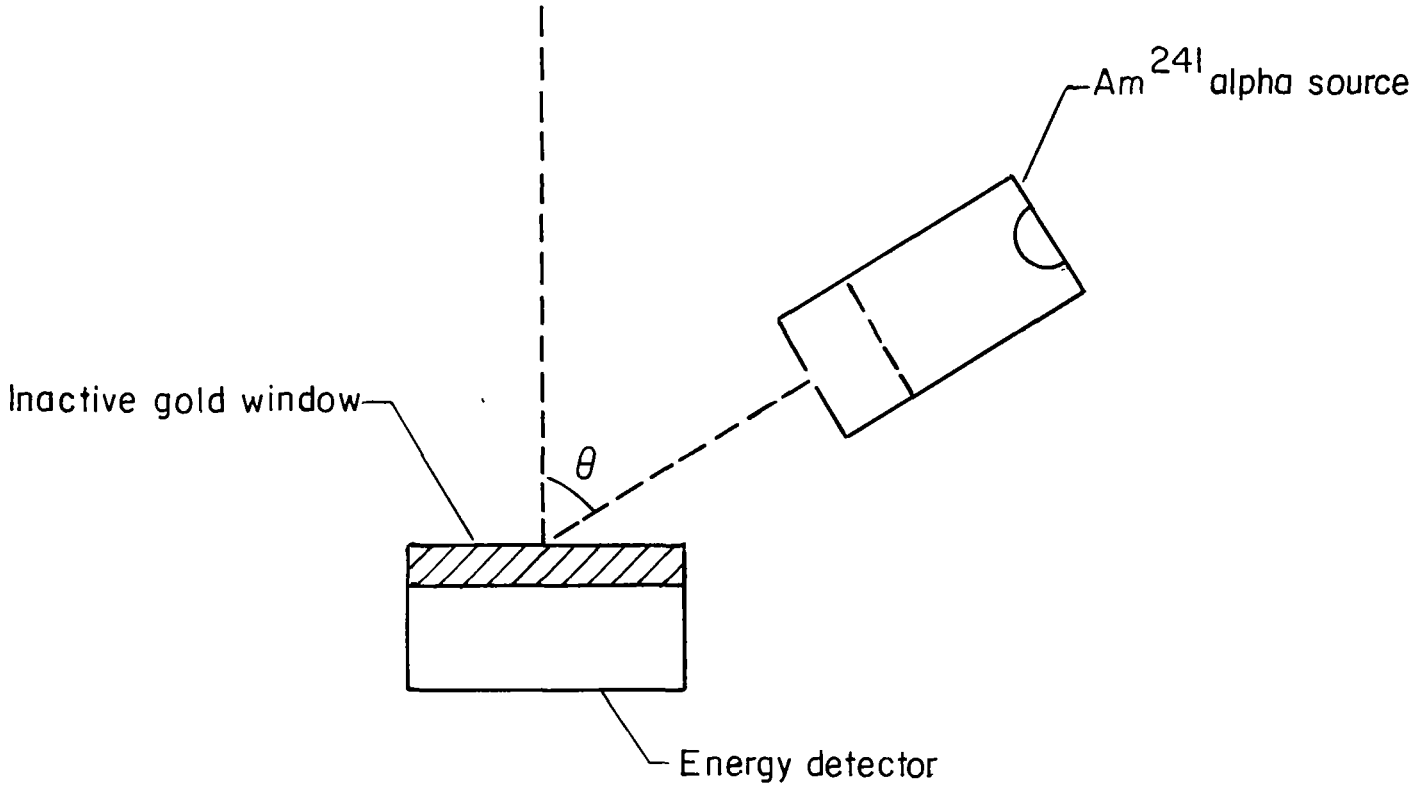

Figure 5.- Setup for determination of gold window thickness.

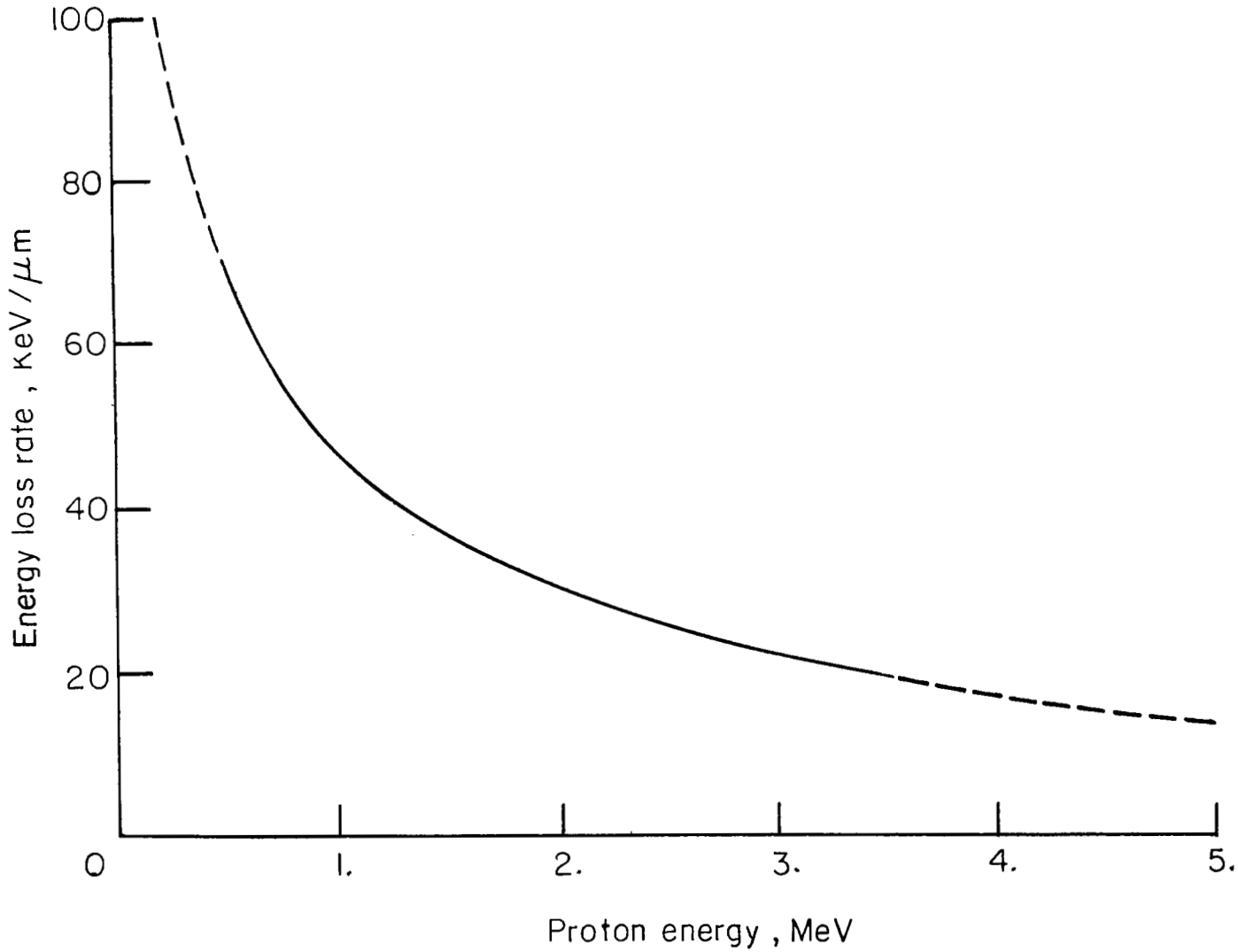

Figure 6.- Energy loss rate dE/dx for protons in aluminum. Solid line indicates range of this investigation; dashed lines are extrapolated.