UCID-16607

LAWRENCE LIVERMORE LABORATORY
University of California/Livermore, California

BETA-RADIATION BACKSCATTER MEASUREMENTS OF ALUMINUM ON PLASTIC

Grover M. Taylor

October 19, 1974

Prepared for U. S. Atomic Energy Commission under contract no. W-7405-Eng-48

DISCLAIMER

BETA-RADIATION BACKSCATTER MEASUREMENTS OF ALUMINUM ON PLASTIC

ABSTRACT

The beta-backscatter method of measuring the thickness of an aluminum coating on a plastic substrate is described. The degree of accuracy that can be obtained for various assumed calibration standards and counting times is estimated. A 95% confidence level of ±2% is estimated when each count is for 1000 s, and five standards are available with an estimated accuracy of ±0.4%.

INTRODUCTION

The feasibility of measuring the thickness uniformity of an 11.43-μm aluminum layer on a plastic substrate 127-μm thick is being investigated. This report is a preliminary description of the beta-radiation backscatter method of measurement, which has several advantages: measurements (1) can be made from one side of the specimen without access to the other side, (2) can be limited to include only a very small area of the plating, and (3) will give a result that is averaged over that area. Some details of this method are discussed, and the order of accuracy anticipated for it is given. The problem of measurement time is also discussed.

THE BETA-RADIATION BACKSCATTER METHOD

This method of measuring the coating thickness of one material on a substrate of another has been developed as a result of the ready availability of various radioactive beta-radiating materials. The method depends on large-angle scattering of the beta particles as they penetrate solid materials; the scattering is dependent on the mass-per-unit area of the material being penetrated. It is not the author's purpose to give a detailed, radiation physics explanation of all the various reactions involved, but only to explain enough of the experiment to give a general idea of the measurements.

As the electrons from a radiation source move through a material, they lose their excess kinetic energy through a number of different reactions. Consequently, in a given material they penetrate a fairly well-defined range. This range is a function of the electron's energy and of the atomic number and physical density of the material. In the present case, the electrons must penetrate the aluminum plating layer twice to give a signal at least partially sensitive to the aluminum thickness. On the other hand, it is undesirable to have a beta energy high enough so the electrons can penetrate both plating and substrate twice, or the backscattered radiation becomes a function of the substrate thickness also. Promethium-147 appears to be the only available source that meets these criteria and is also suitable in other respects as a radiation source for this work.

The relative position of the components making up the input to the experiment is illustrated in Fig. 1. A radiation source, suitably mounted, emits beta radiation, some of which passes into the plating and substrate under examination. A small fraction of these betas undergo scattering through angles near 180° and enter the sensitive volume of the radiation counter. The dependence of the resulting count rate on plating thickness is used for the measurements.

The general pattern of expected results is shown in Fig. 2. All counting data must be reduced to the same time base to be valid, but for simplicity, we can assume that each count is for the same time. In Fig. 2, Cl is the count on the substrate and CN is the count on an infinite thickness of plating material. A horizontal line has been drawn to indicate these two unique values. Evidently any plating thickness from zero to infinity will give a count between these two. The curve drawn gives the relation expected.

ANALYTICAL MODEL

Although a theoretical solution accounting for all details of the beta-backscatter experiment is extremely complex and will not be discussed here, an analytical model developed at LLL some years ago[1] appears to be useful in the present case. The model can be shown as plausible on general grounds and its degree of applicability to a given specific case can then be determined by experimental data. An example of the model's equation is,

$$\ln\left[\frac{CN - C(i)}{CN - Cl}\right] = Al \cdot T(i),$$

where CN is the count rate for infinite plating thickness, Cl the count rate for zero plating thickness, C(i) the count rate for plating thickness T(i), and Al is determined by a least-mean-squares fitting routine. The type of graph expected from the equation is given in Fig. 3.

Actual measurements made on a set of foils of known thickness counted by the beta-backscatter method on a plastic substrate 127-μm thick are shown in Fig. 4. The values found for the foils, represented by small circles, fit the model very well. The poor fit of the data point at 30.5 μm causes no concern. It is in a region where the accuracy falls off rapidly and is determined by taking the small difference of two large numbers, each of which has an experimental uncertainty associated with it.

While this analytical model is useful as a calibration equation to determine unknown thicknesses and the expected accuracy for a given specific set of measurements, calculations based on it are too conservative. A simpler model is described in a later section and results of calculations from it are given.

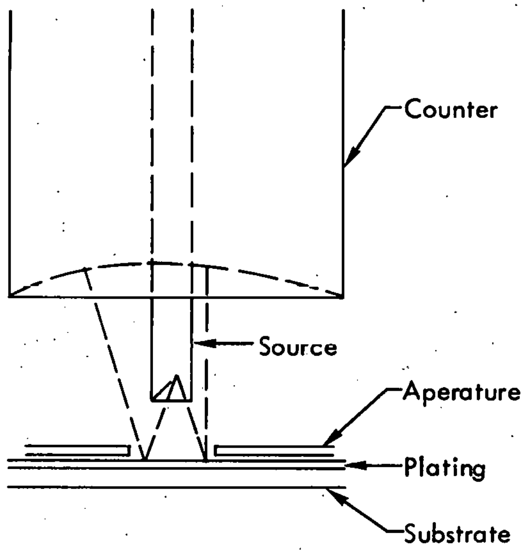

Fig. 1. Geometry of beta backscatter.

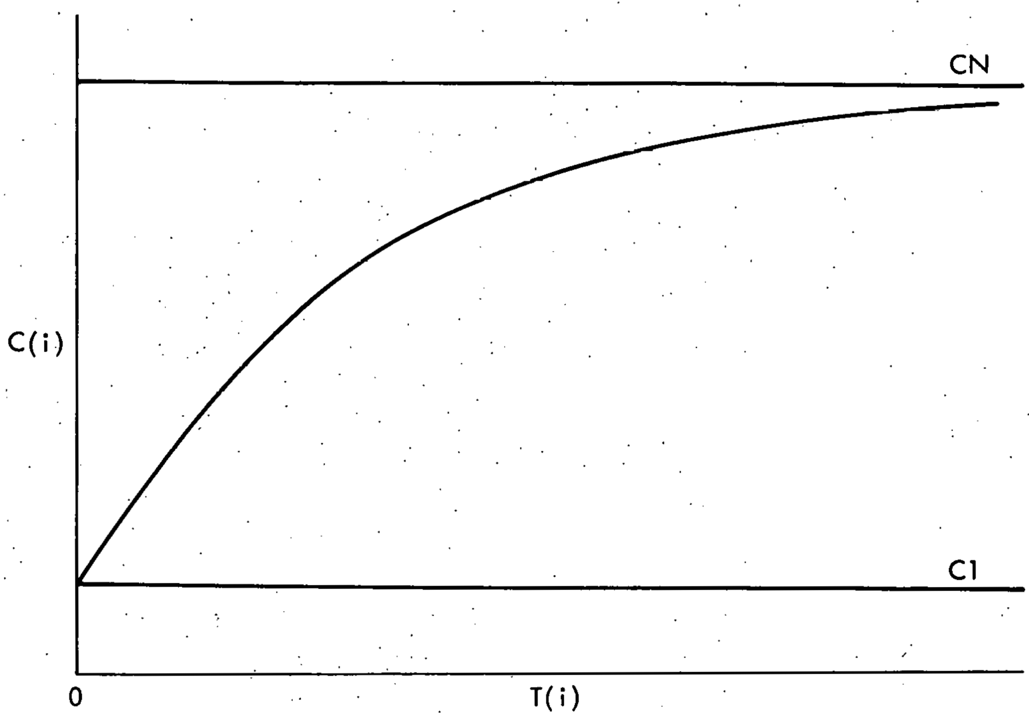

Fig. 2. Counting rate as a function of thickness.

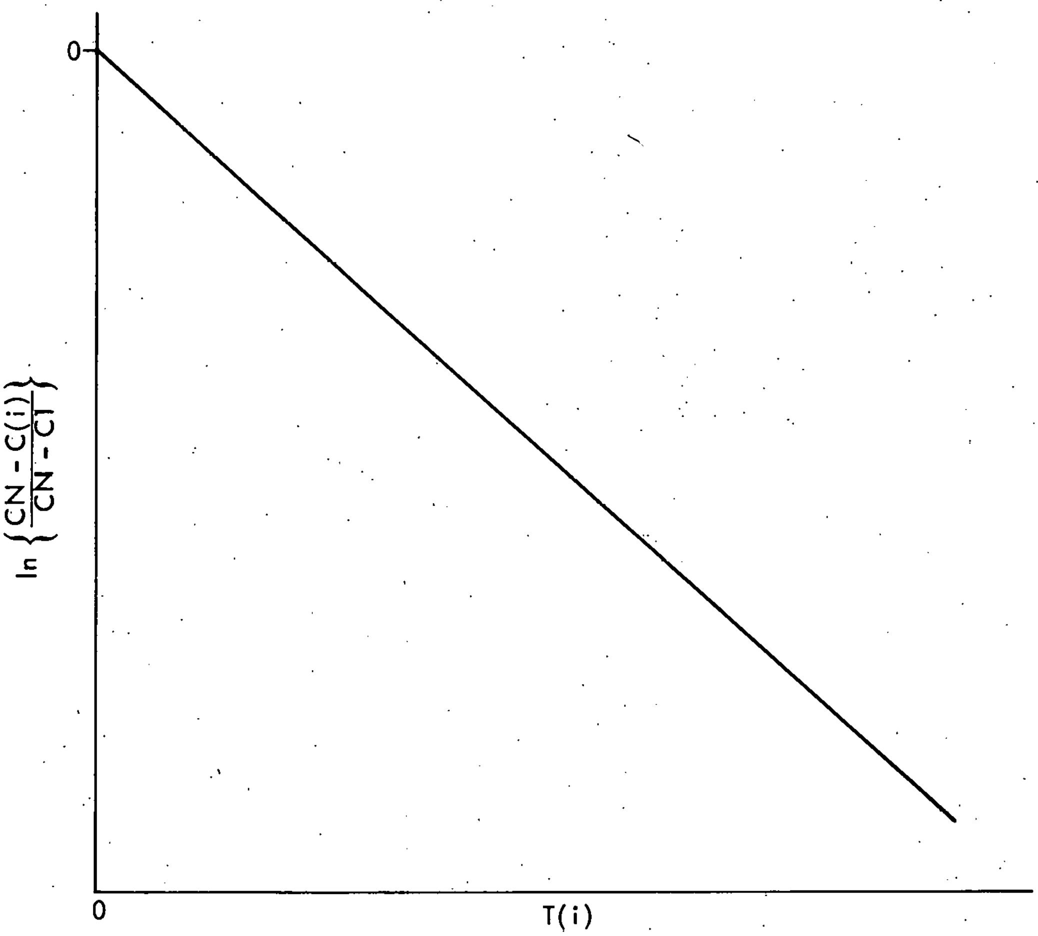

Fig. 3. Mathematical model for beta backscatter.

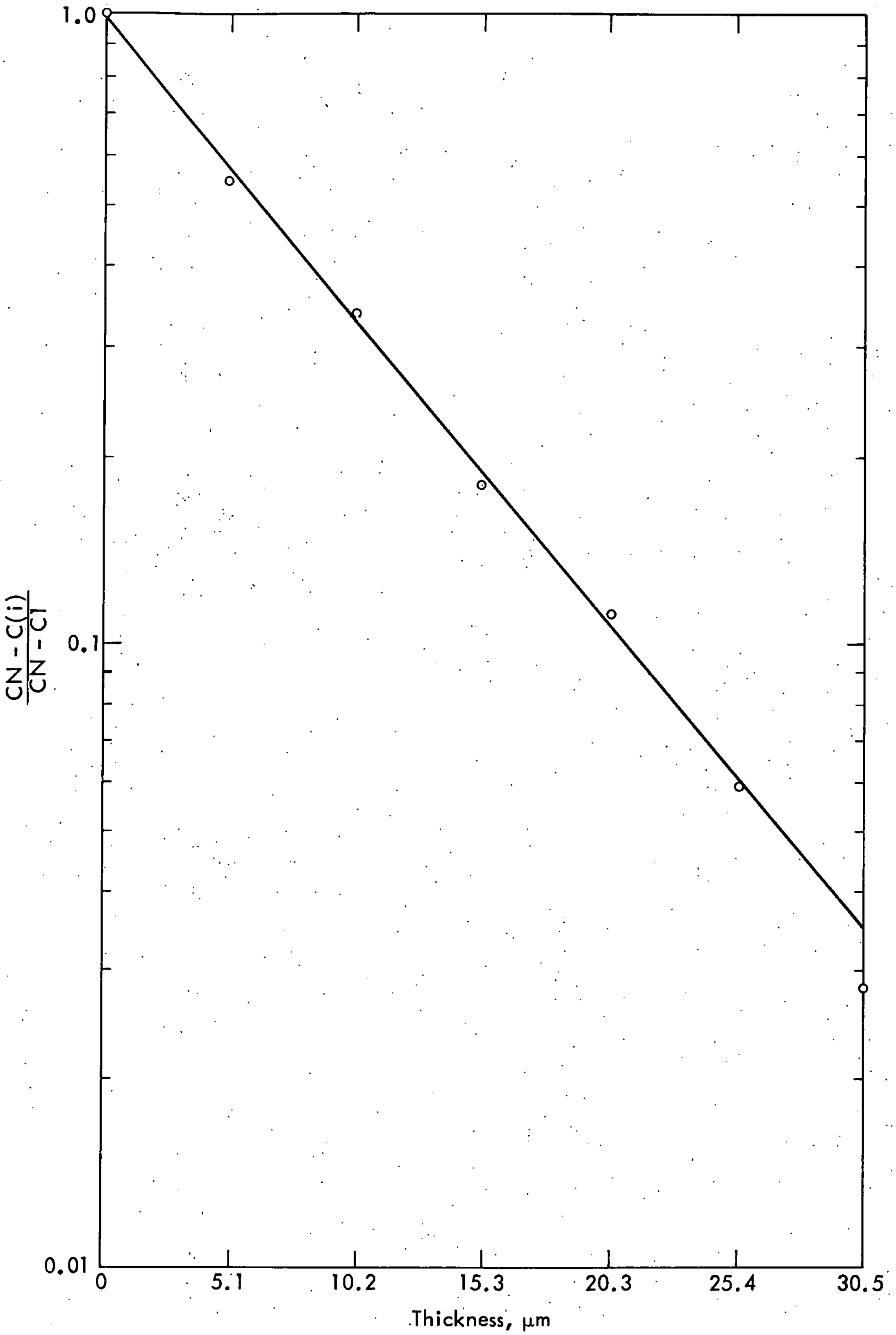

Fig. 4. Experimental data for beta backscatter.

RADIATION DETECTION EQUIPMENT

The detector usually used in beta-backscatter measurements is a thin-window, Geiger-Muller counter. The pulses from it are large and their amplitude is independent of the energy of the beta particle causing them, provided the counting rate is not too high. When the detector is used within the count-rate limitation and with good electronic equipment, the output of the system will obey Poisson statistics and the standard deviation of N counts will be given by $\sqrt{N}$. This assumption is used subsequently to calculate the expected accuracy for various specific cases.

The reason for the counting-rate limitation is that a Geiger-Muller counter has a dead time associated with it. For a short period of time after a count, it will either not respond to an additional particle in its sensitive volume or respond with a pulse of reduced amplitude. In either case, the validity of the Poisson distribution is destroyed and the actual accuracy is less than might be anticipated from a calculation based on the $\sqrt{N}$ assumption.

A dead time of about 10^{-4} s is not unusual for a Geiger-Muller counter would limit count rates to 10^{-3} counts/s for a 10% coincidence loss. The counts can be corrected, however, by the following formula:

$$CR(C) = \frac{CR(0)}{1 - \tau \cdot CR(0)},$$

where CR(0) is the observed rate in counts/s, τ is dead time in seconds, and CR(C) is the corrected rate in counts/s. If rates appreciably higher than 10^{-3} counts/s are attempted, the experimental error will be increased. For a high degree of accuracy, count rates must be limited to lower values.

For higher rates a photomultiplier-scintillation counter may be substituted for the Geiger-Muller counter. However, the output pulses of such a detector are roughly proportional to the energy deposited in the scintillator by the incident particle. Promethium-147 emits beta particles with a maximum energy of 0.23 MeV, a relatively low value. Any particle reaching the scintillator will have undergone scattering in the sample and will lose additional energy in penetrating the light-tight cover of the scintillator. As a consequence, many low-energy betas will be present in the spectrum and the photomultiplier output will contain many low-voltage pulses. To block noise pulses from the amplifier input, a discriminator is normally used to prevent voltage pulses smaller than some preselected value from being counted. The counting-rate output of the system may turn out to be such a rapidly varying function of this setting, however, that residual jitter in the threshold becomes a limiting factor on accuracy. The standard deviation may then be appreciably larger than $\sqrt{N}$.

Thus the question of whether or not the higher counting rate of the scintillation counter can be useful is contingent on whether the required degree of equipment stability can be attained. Probably this can be answered only by experiment.

STATISTICAL CONSIDERATIONS

To determine the plating thickness at a single point, a set of calibration data followed by a count for the point in question is necessary. The counts obtained in a calibration experiment are combined, either graphically or analytically, to obtain a calibration curve that is then used, with a reversal of the variables, to determine an unknown thickness from its counting rate. In the calibration samples, subsequently referred to as standards, two types of error may be introduced: (1) a bias because of a systematic error in determining their thickness, and (2) an error because of random variations in their measurement. Once the standards are chosen, both errors become systematic; but for analytical purposes, we will assume an a priori uncertainty in the standards and call it a random variable.

When the system is calibrated, known thicknesses are inserted and the corresponding counts determined. Each count is assumed to have an uncertainty of $\sqrt{N}$ associated with it and each standard is assumed to have either zero or a small uncertainty. The errors are combined to give an estaimte of the uncertainty in the fit parameters. An unknown to be measured would then have the uncertainty of its own count plus the parameter uncertainties involved in its expected accuracy. The general formula for combining errors is

$$\mathrm{VAR}\{F(X(i))\} = \sum_i \left[\frac{\partial F}{\partial X(i)}\right]^2 \mathrm{VAR}\{X(i)\},$$

where F(X(i)) is a function of the multiple variables X(i), VAR {} the square of estimated standard deviation and $\frac{\partial}{\partial X(i)}$ the partial derivative with respect to X(i).

SHORT-RANGE MODEL

As shown earlier, while the analytical model is valid through the entire useful range of the beta-backscatter method, its error analysis is too conservative if the variable is to be determined only over a very short range. An exponential over a short range can be represented fairly well by a straight line. Consequently, in addition to the analytical model, the following short-range model is used:

$$C(i) = A0 + A1 \cdot T(i),$$

where T(i) is the plating thickness, A0 and A1 are fit parameters, and C(i) is the count rate for T(i).

As in the analytical model, we assume the equipment is calibrated using a set of N standards. The data pairs C(i) and T(i) are used to obtain a least-mean-squares estimate of A0 and A1. The short-range equation is then used to find the unknown thicknesses associated with the experimentally determined counts. The propagation of errors is calculated as described previously except that A0 and A1 are correlated and we must take this into account when calculating the error in the unknown thicknesses from their counts

CALCULATIONS

A computer program was written so that the parameters listed in Table 1 could be changed from run to run to study each parameter's effect on the experimental accuracy of the final thickness determination. The results of some computer runs based on Wolberg's model[2] were as follows:

Table 1. Calculated uncertainties.

No. standards	Uncertainty standards (μm)	Measurement time (s)	S (%)	S corrected (%)	95% confidence level (%)
3	0	10	3.9	7.4	
3	0.127	10	4.0	7.6	
3	0.254	10	4.1	7.8	
3	0	100	1.2	2.3	
3	0.127	100	1.4	2.7	
3	0.254	100	1.8	3.4	
3	0	1000	0.4	0.8	
3	0.127	1000	0.8	1.5	
3	0.254	1000	1.3	2.5	10
5	0	10	3.7	4.5	
5	0.127	10	3.8	4.6	
5	0.254	10	3.9	4.7	
5	0	100	1.2	1.4	
5	0.127	100	1.3	1.6	
5	0.254	100	1.4	1.8	
5	0	1000	0.4	0.5	
5	0.127	1000	0.6	0.7	
5	0.254	1000	1.1	1.3	2

In the first column, the number of standards assumed is given; only three or five standards were considered. In the second column, the uncertainty value assumed in measuring the thicknesses of the standards is given. The measurement times listed in the third column are for a counting rate of 1,000 counts/s, a maximum reasonable rate for a Geiger-Muller counter.

Estimates for the standard deviation of a measurement at the midrange of the calibration are given in column four. These estimates are then corrected for the number of degrees of freedom and their new values listed in the next column. Finally, in the last column, two values are given for the 95% confidence level. These two values are for the maximum credible accuracy in terms of the quality of standards and the allowable measurement time. For both values, a single measurement would take 1000 s, so the method would be relatively slow.

The calculations of Table 1 do not take into account any variability for instrument instability or for positioning accuracy. It seems probable, however, that both would show up as noticeable factors in the final accuracy.

CONCLUSIONS

The beta-backscatter method appears to be a suitable way of determining the average mass-per-unit area of aluminum deposits on plastic in a very small area. The method is nondestructive and practical where a relatively long measurement time is available and where a standard deviation on the order of ±2% will be adequate. These conclusions are based on equipment already in use and on data obtained in a small number of measurements although the supporting calculations are approximate and give estimates rather than analytical answers.

The use of a scintillation counter would reduce by a large factor the time required to obtain enough counts for a high degree of statistical accuracy. Further experimental data is required to determine how accurate the data from a system using this type of detector may be, however.

Finally, we should note that the data obtained so far and the calculations described briefly here do not support the conclusion that this technique would be satisfactory for certifying the aluminum thickness to ±2%.

REFERENCES

1. J. D. Fox and G. M. Taylor, Mater. Eval. 28 (1), 17 (1970).
2. J. R. Wolberg, Prediction Analysis, (D. Van Nostrand Co., New Jersey, 1957).

JKP/lc