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Beta Backscatter Measurements of Aluminum Ion Plating on U-0.75 Ti Parts

K. W. Dolan

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BETA BACKSCATTER MEASUREMENTS OF ALUMINUM ION PLATING ON U-0.75 Ti PARTS

K. W. Dolan
Acceptance Technology Division 8444
Sandia National Laboratories, Livermore

ABSTRACT

The beta-backscatter method of measuring thin laminates on thick substrates was evaluated for possible production inspection applications of aluminum ion plating on U-0.75 Ti parts. The choice of radiation source, the effects of surface curvature, and background radiation from uranium isotopes were examined. Measurements of aluminum coatings on U-0.75 Ti test coupons and cylindrical parts were made with the β-backscatter technique and compared to α-transmission measurements.

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BETA BACKSCATTER MEASUREMENTS OF ALUMINUM ION PLATING ON U-0.75 Ti PARTS

Introduction

The β-backscatter method of measuring the thickness of thin laminates on thick substrates was evaluated for possible production inspection applications for aluminum ion plating on U-0.75 Ti parts. The aluminum ion plating in the production application has a minimum thickness of 5 μm (200 μin), and a maximum thickness of 13 μm (500 μin). X-ray fluorescence techniques, as well as an α-transmission technique,[1] are both applicable to inspection of thin coatings of aluminum on U-0.75 Ti, but both techniques require that the part be inspected in vacuum. Desirable features of the β-backscatter technique are that it does not have a vacuum requirement (i.e., the measurement can be done in air), and counting periods are much shorter than with either the α-transmission of X-ray fluorescence techniques. Important aspects of the β-backscatter method which this study addresses are (1) the sensitivity of the measurement, (2) the choice of radiation source, (3) the effect of surface curvature on the measurement, and (4) the effect of background radiation from uranium isotopes in the substrate material.

Beta-Backscatter Method

In the β-backscatter method, a β-emitting radioisotope source is positioned near the surface to be inspected. A fraction of the β-ray flux incident on the material surface is backscattered due to large angle scattering. The backscatter intensity is proportional to Z^n, where Z is the atomic number of the material, and n is an empirical constant with a value 0.7 to 0.8.[2] A thin layer of low Z material on a thick substrate of higher Z material reduces the backscatter intensity. Increasing the thickness of the surface layer beyond approximately one-fifth the range of the most energetic β-particle gives a backscatter intensity equivalent to an infinitely thick layer of the surface material.

The backscatter flux is measured with a thin window radiation detector. Geiger-Mueller (G-M) tubes with thin mylar or mica windows are used for this purpose, as are plastic scintillation/photomultiplier tube detectors with thin beryllium windows. The detectors can be used in either the count rate mode or the current mode, depending on the source strength.

The choice of a β-ray source is determined by the thickness and atomic number of the surface coating. Higher energy β-sources are used for thicker, higher atomic number coatings, while lower energy β-sources are used for better sensitivity with thinner, lower atomic number coatings. Two commonly used isotopes for measuring aluminum laminates with thicknesses in the range of interest here are promethium-147 (2.6 y half-life, 0.22 MeV maximum β-energy), and thallium-204 (4.0 y half-life, 0.76 MeV maximum β-energy). The maxiumum aluminum thicknesses that can be measured with these sources in the backscatter mode are 50 μm (2.0 millinch) with Pm-147, and 300 μm (12.0 millinch) with Tl-204.

The backscatter measurement is geometry dependent. The gage aperture, position of the source relative to the aperture and detector, and the standoff distance of the gage from the surface being inspected are all parameters of the measurement. The source position is adjusted to maximize the backscatter flux. When using a collimated source, an optimum source position exists that minimizes the difference in the backscatter flux between flat and curved specimens. In practice, the difference between flat and curved specimens can be made negligible by proper positioning of the collimated source, provided the diameter of the gage aperture is less than $2(2D)^{1/2}$, where D is the curved surface diameter.[3] When this difference is negligible NBS traceable standards, which are flat surface standards, can be used for calibration.

Equipment

The β-backscatter gage for the present study uses an annular G-M tube with a thin mylar window. The G-M tube is enclosed in a shielded holder, as shown in Figure 1. The holder is shielded with lead to attenuate external radiation, and lined with beryllium to minimize internal reflections of the β-flux. The gage platen, i.e., that part of the gage that normally contacts the surface under inspection, is also made of beryllium. The platen orifice that defines the area under inspection is 6.4 mm (0.25 in) diameter. The β-source is a collimated source contained in the hollow nose of a needle point holder. The source holder is inserted through the cylindrical open core of the annular G-M tube, with the tip of the holder positioned 1.5 mm (0.06 in) from the external surface of the orifice.

The counting unit used with the G-M tube gage is a commercially available unit* containing a charge sensitive amplifier, discriminator, and scaler with 0.5 MHz count rate capability. The bias voltage on the G-M tube was set to the plateau level of the count rate response curve to provide insensitivity to small variations in the bias voltage while providing maximum output. The discriminator level was set to eliminate essentially all but signal pulses.

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*Miniscular Model No. MS-2, Eberline Instrument Corporation, Santa Fe, NM

β-Source Evaluation

Two different β-sources were evaluated for the coating thickness measurements. A Pm-147 source with maximum β-energy of 0.22 MeV, and a Tl-204 source with maximum β-energy of 0.76 MeV were used to obtain count rate versus coating thickness calibration curves. The calibration standards were thin foils of aluminum of known thicknesses pressed over flat coupons of U-0.75 Ti. Measurements were made with the gage platen in contact with the surface of the standard. A number of measurements were made on each calibration standard to obtain a statistical uncertainty of less than ± 1%. The results of these measurements are given in Figure 2. These data show that the sensitivity of the Pm-147 measurement is a factor of 3 to 5 greater than that of the Tl-204 measurement for the coating thicknesses of interest, 5 to 13 μm (200 to 500 μin) Al. For a statistical uncertainty of ± 1% in the count rate, the calibration curves of Figure 2 show that a thickness measurement of 5 μm (200 μin) Al will have an uncertainty of ± 0.21 μm (± 8.3 μin) using a Pm-147 source, and ± 1.1 μm (± 42 μin) using Tl-204. At a thickness of 13 μm (500 μin) Al, the corresponding uncertainties are ± 0.36 μm (± 14 μin) with Pm-147, and ± 1.3 μm (± 50 μin) with Tl-204. On the basis of these results, we conclude that Pm-147 is the preferred source for measuring Al coatings in the range 5 to 13 μm (200 to 500 μin) on thick U-0.75 Ti substrates.

Surface Curvature Effects

The effect of surface curvature on β-backscatter measurements with the present gage was determined by measuring the backscatter count rate from cylinders of various diameters. Measurements were made for two different material configurations. In one configuration, a thick gold foil was laid over the cylindrical surface, giving backscatter from an essentially infinite thickness of high Z material of the desired surface curvature. In the second configuration, a thin aluminum foil was laid over the gold substrate foil, giving backscatter from a low Z coating over a thick high Z substrate. The same foils were used on each of the cylinders to insure constant substrate and surface layer thicknesses. The gold foil used here was 25 μm (1.0 millinch) thick, and the aluminum foil was 3.0 μm (0.12 millinch) thick. The aluminum surface foil is partially transmissive to the Pm-147 β-particles, while the gold substrate foil is opaque.

Results of the β-backscatter measurements on cylindrical surfaces are given in Figure 3 where the backscatter count rate is plotted as a function of cylinder diameter. The flat surface results are included in this figure as a cylinder of infinite diameter. Each datum point is the mean value of five separate measurements, and the associated error bar is the standard deviation of the mean. The results show that the backscatter count rate is a constant for cylindrical surfaces with diameters greater than 50 mm (2.0 inches), and equal to the backscatter rate from a flat surface sample of the same composition.

Thus, we conclude that for a gage with platen orifice 6.4 mm (0.25 in) diameter or less, flat surface standards can be used directly to provide calibration for measurements on cylindrical surfaces with diameters 50 mm

(2.0 inches) or greater. This allows the use of flat surface NBS traceable standards for calibration purposes.

Calibration

The calibration standards used here were made from multiple layers of single sheet aluminum foil pressed over flat U-0.75 Ti coupons. The single sheet aluminum foil was 3.04 ± 0.25 μm (120 ± 10 μin) thick, while the dimensions of the coupon surface area were 12.5 x 25.0 mm (0.5 x 1.0 in). The thickness of the single sheet foil was quite uniform, significantly better than the thickness uncertainty, while the density of the foil was equal to 100% dense material, 2.70 g/cm^3. Multiple layers of the single sheet foil gave effective coating thicknesses of 6.10 ± 0.36, 9.14 ± 0.43, 12.2 ± 0.5, 15.2 ± 0.6, and 18.3 ± 0.6 μm (240 ± 14, 360 ± 17, 480 ± 20, 600 ± 23, and 720 ± 25 μin). More accurate NBS traceable standards are desirable for production inspection, but for the present technique development study, the accuracies of the standards were adequate. Uncertainties associated with the thickness of calibration standards are bias uncertainties, while those associated with count rate measurements are statistical uncertainties.

Since the U-0.75 Ti substrate material is radioactive, the β-backscatter gage is subjected to a background radiation flux that originates in the part being inspected. In addition to background radiation received directly through the gage orifice, a number of X-rays and γ-rays from the part have sufficient energy to penetrate the shielding of the gage. Thus, the background rate is dependent on the geometry of the part relative to the position of the gage.

The background rate is determined by taking a measurement of the count rate with the gage source removed. The results of background measurements on the present calibration standards are given in Table 1. It is seen that the background rate is essentially a constant for these standards, independent of the aluminum coating thickness. Also given in Table 1 are the total count rate with the Pm-147 source in place in the gage, and the resultant backscatter rate (i.e., the difference between the total rate and the background rate). The backscatter rate is just the rate at which β-particles from the gage source, backscattered from the sample, enter the detector. The backscatter rate as a function of aluminum coating thickness, obtained from the calibration standards, is used to define the calibration curve of Figure 2. The thickness of aluminum coating on U-0.75 Ti parts is determined by using this curve, where the backscatter rate is the measured total rate corrected for background from the part under inspection.

Applications

Cylindrical Parts

Aluminum ion plating thicknesses on cylindrical U-0.75 Ti parts were measured with the β-backscatter techniques developed here. Part diameters were greater than 50 mm (2.0 inches), so that flat surface calibration standards could be used directly for calibration purposes. The measuring procedure included a measurement of the background counting rate with the β-source removed from the gage, and then a measurement of the total rate with the β-source in place in the gage. Measurements were made at a number of points on both the inside and outside surfaces of each part. The ion plating was found to be quite uniform over each surface, within the statistical uncertainty of the measurement. However, the plating on the inside surface of each part was significantly less than that on the outside surface. Inside coating thicknesses were found to be approximately 75% of outside coating thicknesses.

The calibration standards used in these measurements were the same as those used in α-transmission measurements[1] on the same parts, allowing a direct comparison of the results of the two techniques without bias uncertainties. The results of the β-backscatter measurements are given in Table 2, together with the comparable α-transmission results. The measured thicknesses of the aluminum ion plating ranged from 1.2 to 2.0 μm (50 to 80 μin), with statistical uncertainties of approximately ± 15% with the β-backscatter technique, and ± 5% with the α-transmission technique. The β-backscatter values given in Table 2 are consistently larger than the comparable α-transmission values, although the results are essentially within the combined standard deviations in each case.

The background rate with the β-backscatter gage for measurements on inner surfaces of the parts was typically 22,000 counts/min, while the background rate on outer surfaces was typically 16,000 counts/min. This compares with the background rate from the calibration standards of approximately 10,000 counts/min. The signal to background ratio for measurements on inner surfaces of the parts was typically 0.73, while that for measurements on outer surfaces was approximately 0.96. The use of a stronger β-source would provide larger signal to background ratios, and more accurate thickness values than those obtained here.

Corrosion Test Coupons

Aluminum equivalent thicknesses of aluminum ion plating, including corrosion residue, on U-0.75 Ti corrosion test coupons were also measured with the β-backscatter technique. The dimensions of the corrosion test coupons were similar to those of the calibration samples, and background rates were essentially identical with those from the calibration samples.

A total of ten corrosion test coupons were inspected, with measurements obtained at from four to six locations on each side of the coupons. Mean values obtained from the series of measurements are given in Table 3. In every case but one, the measured values at the separate locations on a side were within the statistical uncertainty of each other and the mean value,

indicating that the coating thickness, including residue, was uniform over the coupon surface to within the uncertainty of the measurement. In the case of coupon number 3, side 2, the thickness measured at two locations near one edge, points 3 and 4, were 1.8 μm (70 μin) thicker than that measured over the rest of the surface (see Table 3). The measurements were repeated on this coupon and several other coupons verifying that the values obtained were repeatable within statistical uncertainties.

The test coupons were found to have coatings of two basic thicknesses. Half of the coupons had aluminum equivalent coatings of approximately 5 μm (200 μin), and the other half had coatings ranging from 10 to 15 μm (400 to 600 μin). The β-backscatter gage used here with the Pm-147 source gave accuracies ranging from ± 0.13 μm (± 5 μin) for coatings approaching zero thickness to ± 0.33 μm (± 13 μin) for coatings up to 19 μm (750 μin), not including bias errors introduced by uncertainties in the calibration foils. The bias error from the present set of calibration foils gives an additional uncertainty of ± 8%.

An α-transmission gage, calibrated with the same set of foils, was used to measure the coating thickness on one of the coupons for comparison.[1] For coupon number 8, the α-transmission gage gave an average aluminum equivalent thickness of 4.75 ± .08 μm (187 ± 3 μin) as compared to 4.83 ± 0.51 μm (190 ± 20 μin) from the β-backscatter gage (statistical uncertainties only). These values show good agreement, and are well within the statistical uncertainties of the two measurements.

Conclusions

The β-backscatter technique for measuring the thickness of thin laminates on thick substrates was evaluated for possible production inspection applications of aluminum ion plating on U-0.75 Ti cylindrical parts. The preferred source for these measurements was determined to be promethium-147 (0.22 MeV maximum β-energy), which provides an accuracy of ± 0.2 μm (± 8.3 μin) with ± 1% counting statistics at the minimum aluminum thickness of interest, 5.0 μm (200 μin). The effect of surface curvature on the backscatter rate was found to be negligible for cylindrical parts with diameters greater than 50 mm (2.0 inches), where the inspection area defined by the gage orifice at contact is less than 6.4 mm (0.25 inch) diameter. The background radiation from the natural decay of uranium isotopes in the substrate material was found to be an important effect, for which correction is necessary when establishing calibration curves from finite area calibration standards for application to large area production parts. Comparison of the present β-backscatter measurements on cylindrical parts and test coupons gave excellent agreement with more accurate α-transmission measurements. Desirable features of the β-backscatter method are relatively short measurement periods, of the order of minutes, and no vacuum requirement,as is the case with X-ray fluorescence and α-transmission. These features, together with the accuracy as determined here, and the independence from surface curvature effects, lead us to recommend that this technique be used in the production inspection application.

REFERENCES

1. K. W. Dolan, to be published.
2. L. R. Zumwalt, Nucleonics 12, 55 (1954).
3. B. B. Jotte and L. E. Miller, Plating and Surface Finishing, 62, 567 (1975).

TABLE 1

Counting Rates From Calibration Standards, Aluminum Covered U-0.75 Ti Flat Surface Coupons, Measured with the β-Backscatter Gage

Standard	Thickness of Aluminum Cover (μm)	Thickness of Aluminum Cover (microinch)	Background Rate (counts/min)	Total Rate (counts/min)	Backscatter Rate (counts/min)
1	0.0	0.	10340	27870	17530
2	3.0	120.	10200	24140	13940
3	6.1	240.	10270	20920	10650
4	9.1	360.	10130	18330	8200
5	12.2	480.	(10120)*	16520	6400
6	15.2	600.	(10120)*	15240	5120
7	18.3	720.	10110	14480	4370

*Interpolated Value

TABLE 2

Measurements of Aluminum Coating Thickness on Cylindrical U-0.75 Ti Parts Using β-Backscatter Gage and α-Transmission Techniques

Part Number	Cylindrical Surface	Aluminum Thickness (μm)*	
		β-Backscatter Gage	α-Transmission Technique**
X013	Outer	1.7 ± 0.2	1.6 ± 0.05
X013	Inner	1.2 ± 0.3	1.0 ± 0.05
X014	Outer	2.0 ± 0.2	1.8 ± 0.1
X014	Inner	1.5 ± 0.2	1.1 ± 0.1

*Uncertainties are statistical uncertainties only.

**See Reference 1.

TABLE 3

β-Backscatter Measurements of Coating Thicknesses on U-0.75 Ti Corrosion Coupons. Coating Includes Aluminum Ion Plating and Corrosion Residue. Thickness Values are Aluminum Equivalent Thicknesses.

Coupon No.	Coupon Identification	Side	Location(s)**	Aluminum Equivalent Thickness* (μm)	(microinches)
1	37.8141	1	1-4	4.6 ± 0.5	180 ± 20
		2	1-4	4.8 ± 0.5	190 ± 20
2	37.5288	1	1-4	5.8 ± 0.3	230 ± 10
		2	1-4	5.3 ± 0.3	210 ± 10
3	37.2894	1	1-4	16.0 ± 0.5	630 ± 20
		2	1-2,2-6	13.0 ± 0.5	510 ± 20
		2	3-4	14.7 ± 0.5	580 ± 20
4	36.8535	1	1-4	5.8 ± 0.3	230 ± 10
		2	1-4	5.1 ± 0.5	200 ± 20
5	50.6	1	1-5	13.0 ± 1.3	510 ± 50
		2	1-5	10.4 ± 0.8	410 ± 30
6	50.7	1	1-5	4.6 ± 0.3	180 ± 10
		2	1-5	4.6 ± 0.5	180 ± 20
7	50.8	1	1-5	16.8 ± 0.8	660 ± 30
		2	1-5	14.0 ± 0.8	550 ± 30
8	50.9	1	1-5	4.6 ± 0.5	180 ± 20
		2	1-5	4.8 ± 0.5	190 ± 20
9	50.10	1	1-4	14.5 ± 0.5	570 ± 20
		2	1-5	12.4 ± 0.5	490 ± 20
10	50.11	1	1-5	12.7 ± 0.8	500 ± 30
		2	1-5	15.5 ± 1.8	610 ± 70

*Uncertainties given are statistical uncertainties only. Uncertainty of calibration foil thickness contributes an additional bias uncertainty of ± 4% to ± 6% not included in the values given here.

**Separate locations inspected each side of coupon, numbered 1 through n, each 6.4 mm (0.25 inch) diameter.

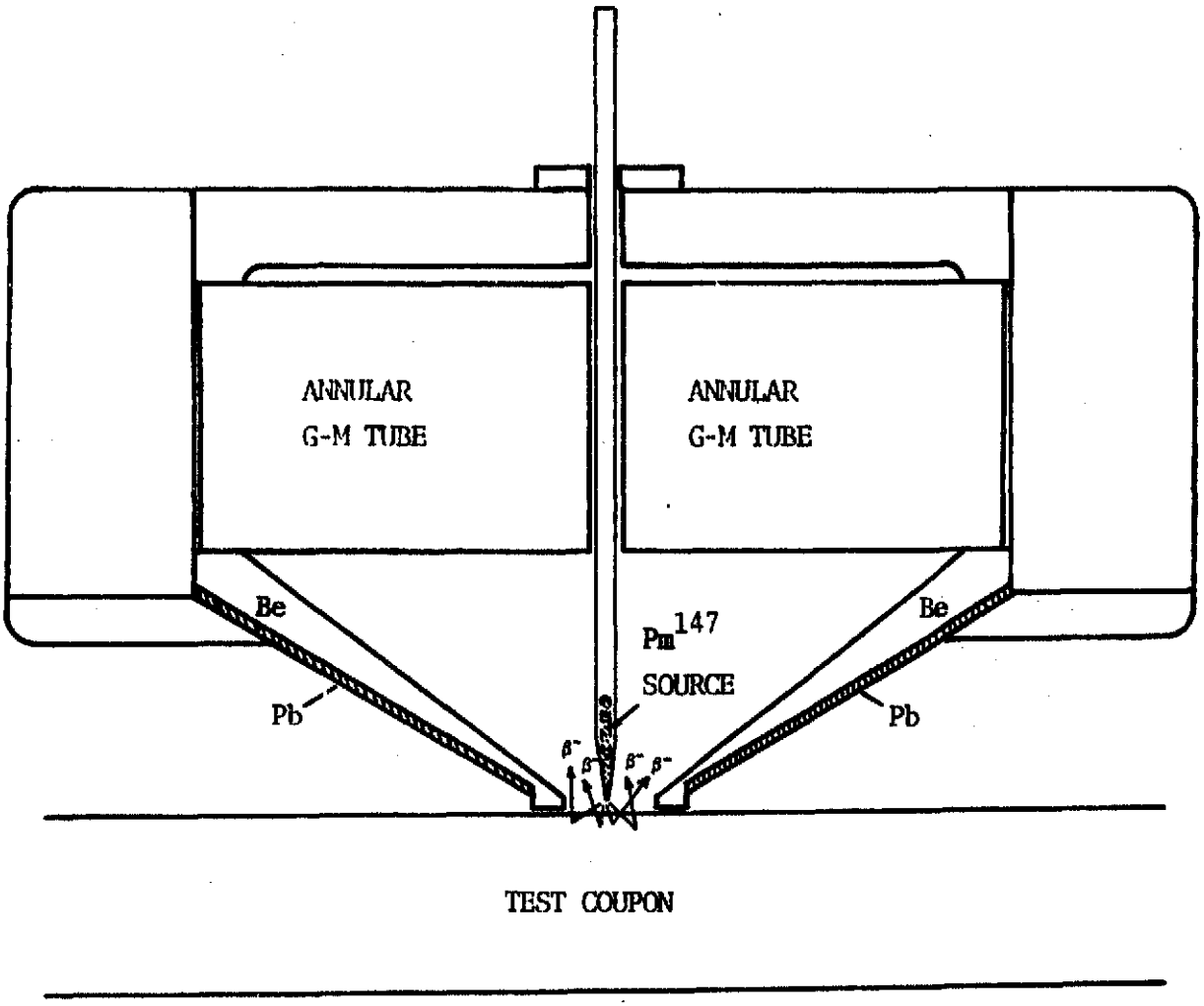

Figure 1. Schematic representation of β-backscatter gage in test configuration.

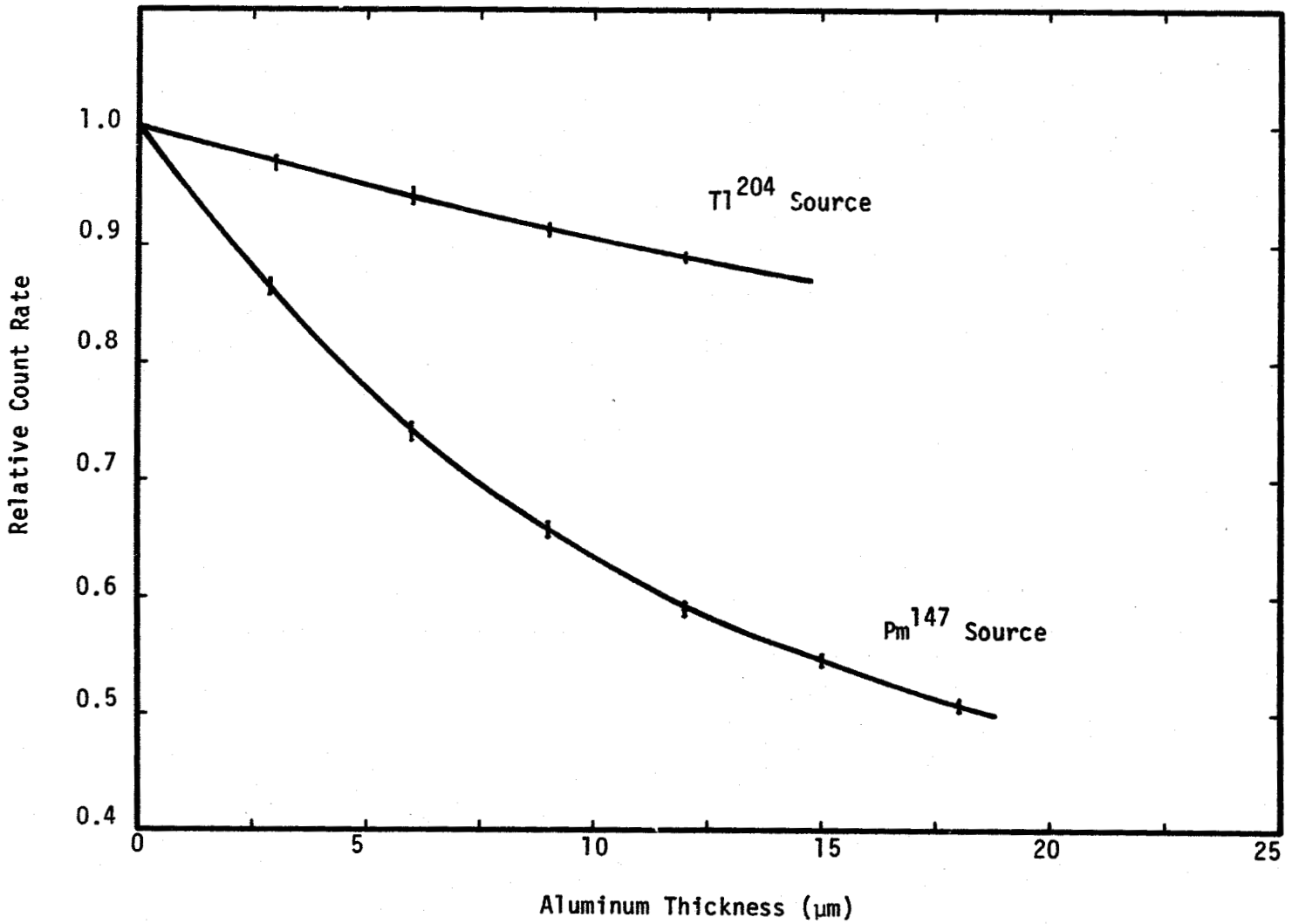

Figure 2. Sensitivity of β-backscatter measurement for aluminum coating on U-0.75 Ti substrate with different β-sources.

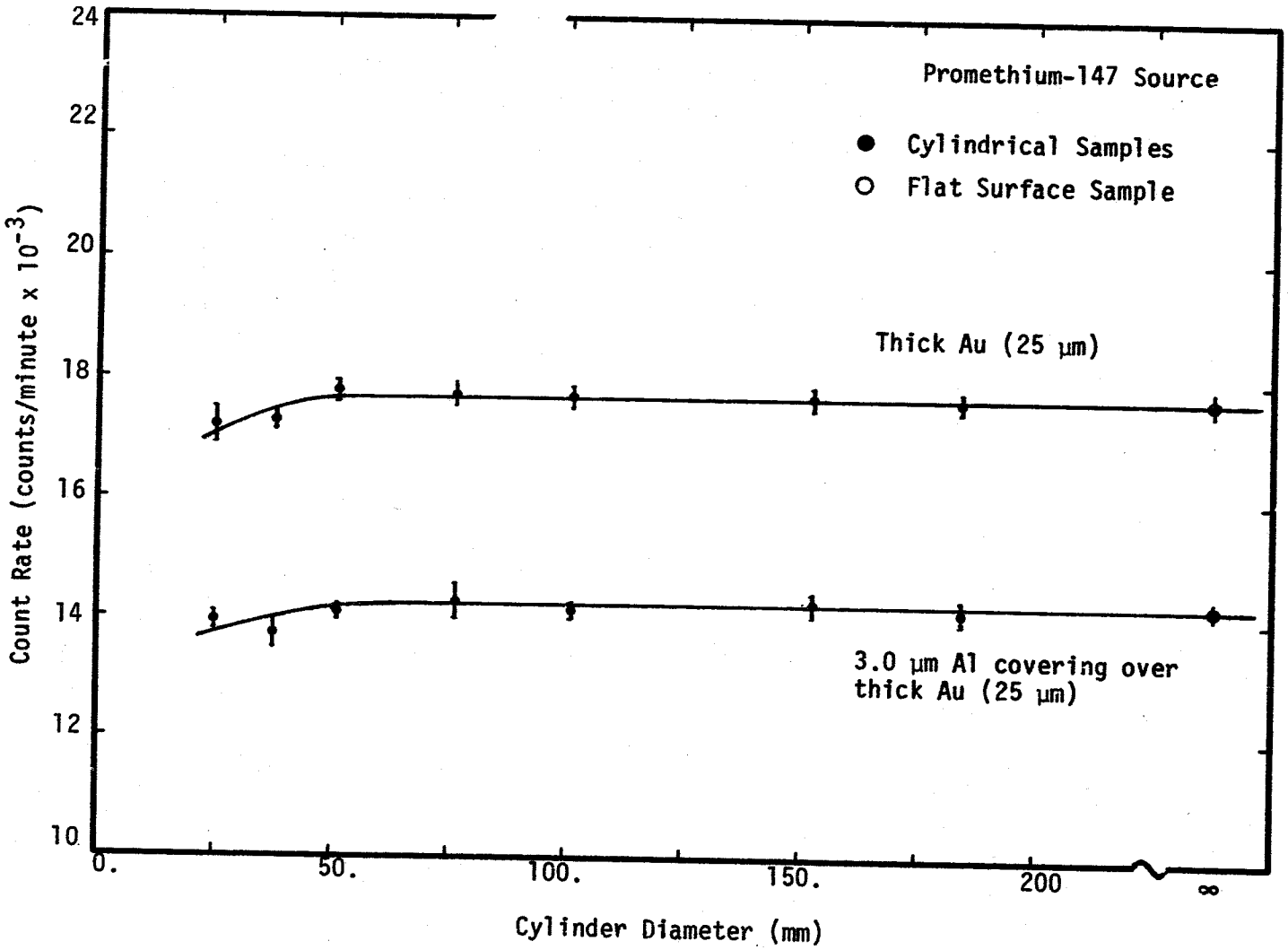

Figure 3. Effect of surface curvature on β-backscatter measurement using a promethium-147 source and gage orifice 6.4 mm (0.25 inch) diameter.

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