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WANL-TME-1189
June 15, 1965

Westinghouse Astronuclear Laboratory

BETA-BACKSCATTER THICKNESS MEASUREMENTS OF DEPOSITED NIOBIUM CARBIDE ON FLAT GRAPHITE SURFACES

(Title Unclassified)

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BETA-BACKSCATTER THICKNESS MEASUREMENTS OF DEPOSITED NIOBIUM CARBIDE ON FLAT GRAPHITE SURFACES

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SUMMARY

A low cost, portable beta-backscattering thickness gage ("Micro-Derm") was purchased from Unit Process Assemblies, Inc., to investigate thickness measurements of flat niobium-carbide coatings over a graphite substrate. Original coating thickness considerations included a range of 1-5 mils. Subsequent considerations have included relatively thin coatings up to 0.0005 inch for both fueled and non-fueled element exterior surfaces. Preliminary performance data is described in the thickness range of 1 to 5 mil niobium carbide. Within the scope of this work, these data indicate that Micro-Derm beta-backscattering measurements can be achieved within ten percent of the results obtained by metallographic direct measurements.

Subsequent to the initial investigation of the Micro-Derm as an inspection tool for measuring niobium-carbide coating thickness, the basic electronic unit was modified by the addition of a preamplifier, a voltage regulator, and an electronic counter. These additions to the original equipment strengthened the output of the detector system and provided for a digital read-out of beta-backscatter count. The purpose for these modifications was to increase the reliability of the coating thickness determinations.

I. INTRODUCTION

At the start of this investigation, beta-backscattering equipment was available for measuring the niobium-carbide thickness in the channels of support blocks and elements. No facilities were available for accurately and non-destructively measuring the exterior flat surface coating thickness. In lieu of a non-destructive test, qualifying specimens representative of each furnace run were sectioned, and the coating thickness measured in filar units at 200X magnification. The acceptance standards for exterior flat surfaces on support blocks included 1.5 mils minimum at the nozzle end, graduating to 2 mils minimum at the element-junction end, with a maximum thickness of 5 mils anywhere.

The coating interface of niobium carbide to ATJ graphite is normally irregular, as shown in the sketch in Figure 1. This irregularity causes difficulty in making precise optical measurements. It is assumed that beta-back-reflection will tend to average the diffusion of niobium carbide into the graphite. It has been observed that the coating interface with LTV grade graphite is considerably more regular, as shown in the photomicrograph, Figure 2.

This investigation describes the Micro-Derm equipment, calibration procedures, and compares Micro-Derm thickness results with those produced by optical means.

II. EQUIPMENT DESCRIPTION

A. Operating Principle

The Micro-Derm thickness gage employs the principle of beta-radiation back-scattering as a means of determining various thicknesses. This gage consists of a probe system connected to an electronic system for interpretation and presentation of data.

B. Radioisotope Probe System

The probe system is basically a metal stand with an integral beta-ray source and beta-ray detector. The top of the stand is milled flat to hold the specimens during thickness measurements. Below and supporting the specimens are located the various size aperature plates, which confine the beta-ray impingement. Directly below the aperature opening is the Strontium-90 isotope sealed in the end of a metal tube. The source to specimen distance is variable in 1/32 inch increments. Surrounding the tube source is the back-reflection detector.

1. Beta Source

The Strontium-90 source is rated as having an activity of 1.0 micro-curies. No beta absorption curves have been made with this isotope to determine the energy spectrum of the source.

2. Detector

The detector is a Geiger-Muller Tube which has a 0.0003-inch mica window. In operation, the gas contained in the tube ionizes when bombarded with beta particles. During ionization, an electrical charge is produced in proportion to the amount of radiation.

3. Aperature Plates

The aperature plates supplied with the instrument include:

Circular Aperature (Diameter, Inch)		Dome Height (Inches)
0.250	x	0.0
0.156	x	0.125
0.125	x	0.09375
0.093	x	0.0
0.062	x	0.0
0.047	x	0.0

Slit Aperature

Length	Width	Dome Height
.156"	.045"	.1625"
.125"	.030"	.125"
.125"	.030"	0.0

Special aperature plates may be purchased to suit the size of the specimen. Typical aperature designs are shown in Figure 3.

C. Basic Electronic Unit

The Micro-Derm basic electronic unit counts the backscattered radiation received as electrical impulses from the Geiger-Muller Tube and displays them as a direct reading on the scale of a precision meter. The scale used during the early part of this program is linear (0-100 units). It is intended to be used for calibration; however, it was used for this as well as reading coating thickness of unknowns.

The M-D unit contains a precision timer (0.001 minute accuracy) that may be pre-set to read minutes and one-hundredths of minutes.

A normal-reverse switch is installed to permit reading of coatings which are either higher or lower in atomic number than the base material.

A rate switch is mounted for convenience to speed or slow up the recording of electrical impulses from the Geiger-Muller Tube. Six settings are available. The Number 1 setting is for fast recording. Number 2 will read half as fast as Number 1. Each setting will read exactly one-half as fast as the next lowest setting.

III. EQUIPMENT CALIBRATION

A. General

The beta-backscattering principle permits the measurement of thin layers of one substance on another base material, providing that there is significant difference in atomic number or density between the two materials (regardless of whether they are metallic or non-metallic, magnetic or non-magnetic). Referring to the graph in Figure 4, niobium and graphite have atomic numbers of 6 and 41, respectively. With the wide spread in atomic numbers, it is considered practical to use beta-backscattering methods for thickness determinations.

B. Standards

Niobium foil was obtained from Kawecki Chemical Company. To supplement the range of standards used in this investigation, a number of niobium-carbide coated graphite specimens were re-checked by optical methods and used throughout this investigation. The range of standards is shown in Table 1.

C. Determination of Optimum Test Parameters

The determination of optimum source distance, aperature size, rate switch position, and radiation count time are set forth in the Micro-Derm Operating Instructions. These parameters were investigated for the three aperature sizes: 0.062", 0.093", and 0.250". These comparative data are shown in Table 2.

The criteria for selecting optimum operating conditions are the lowest values in columns 11 and 12 of Table 2 for any given aperature size. The optimum time (T_o) shown in column 11 can be calculated using the formula:

$$T_o = \frac{KB^2}{2^{R-1} \times N}$$

where: K = a constant depending on statistical accuracy desired (K = 5000)
B = figure of merit: $B = \frac{100}{100 - \text{Div. Reading}}$
R = rate switch position
$N = 200 \left(20 + \frac{1000 - \text{check point dial reading}}{20}\right)$

TABLE 1

BETA-BACKSCATTER CALIBRATION STANDARDS

Material Base	Material Coating	Niobium Thickness (Inches)	NbC (1) Nb x 1.235 (Inches)	NbC (2) Coating (Inches)	NbC (3) (Modified) (Inches)
Graphite	Niobium Foil	.00075	.000925	------	.00133
Graphite	Niobium Foil	.0013	.00161	------	.00201
Graphite	Niobium Foil	.0022	.00272	------	.00312
Graphite	Niobium Foil	.0032	.00395	------	.00435
Graphite	Niobium Foil	.0041	.00507	------	.00548
Graphite	Niobium Foil	.0052	.00642	------	.00683
Graphite	Niobium Foil	.0064	.0079	------	.0083
Graphite	Niobium Foil	.0072	.0089	------	------
Graphite	NbC	------	------	.0004	------
Graphite	NbC	------	------	.0005	------
Graphite	NbC	------	------	.00055	------
Graphite	NbC	------	------	.0007	------
Graphite	NbC	------	------	.00124	------
Graphite	NbC	------	------	.002	.0024

(1) $\frac{\text{Compound Thickness}}{\text{Niobium Thickness}} = \frac{\text{Metal Density}}{\text{Compound Density}} \times \frac{\text{Molecular Compound, Wt.}}{\text{Molecular Wt., Metal}} = \frac{8.5}{7.8} \times \frac{104.9}{92.9} = 1.235$

(2) Metallurgical Lab Thickness

(3) Refer to Figure 20

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TABLE 2

DETERMINATION OF MEASUREMENT-TIME AND RATE-SWITCH POSITION

	1 Combination	2 Source Type	3 Source Number	4 Aperature W x L x H Dia. x H (0 Set = 980)	5 D_{sw}	6 Source Pos. 20 - H + D_{sw}	7 Tube Position	Optimum Time (T_o) and β Determination: 8 Div. Rdg.	9 Chkpt Dial Rdg.	10 Rate Pos. Used	11 T_o Min.	12 β	Rate-Switch Position Determination: 13 Zero Set Dial Rdg.	14 Elapsed Time	15 Actual Time Chosen	16 F = (15)/(14)	17 Rate Switch Position
1	Calib 062 D-O	Sr^{90}	880	.062 D-O	2	22	3	48	640	2	1.35 1.168	1.92 1.925	730	1.91	1.168	.612	1
2	Calib 062 D-O	Sr^{90}	880	.062 D-O	3	23	3	54	457	2	1.30 1.092	2.18 2.175	660	1.38	1.092	.793	1
3	Calib 062 D-O	Sr^{90}	880	.062 D-O	4	24	3	61	418	2	1.63 1.52	2.57 2.56	540	1.17	1.52	1.30	2
4	Calib 062 D-O	Sr^{90}	880	.062 D-O	1	21	3	51	875	1	2.42	2.04					
5				0 Set - 981													
6	Calib 093 D-O	Sr^{90}	880	.093 D-O	2	22	3	32	339	2	.54	1.46	863	.25	1.0	4.0	3.0
7	Calib 093 D-O	Sr^{90}	880	.093 D-O	3	23	3	38.5	179	2	.57	1.65	810	.34	1.0	2.94	3.0
8	Calib 093 D-O	Sr^{90}	880	.093 D-O	4	24	3	46.5	179	2	.70	1.87					
9	Calib 093 D-O	Sr^{90}	880	.093 D-O	1	21	3	35.0	850	2	1.30	1.54					
10				0 Set - 981													
11	Calib .250 D-O	Sr^{90}	880	.250 D-O	2	22	3	23.5	688	3	.73	1.31					
12	Calib .250 D-O	Sr^{90}	880	.250 D-O	3	23	3	22.0	508	3	.44	1.28	918	.08	1.0	12.5	4
13	Calib .250 D-O	Sr^{90}	880	.250 D-O	4	24	3	23.0	370	3	.44	1.30	910	.095	1.0	10.5	4
14	Calib .250 D-O	Sr^{90}	880	.250 D-O	5	25	3	25.0	374	3	.49	1.34					

M. D. Serial No. ______ P. S. Serial No. ______ Name J. R. Steele Date 1-25-65

*See Calculations

The figure of merit (B) and optimum time (T_o) may be taken from the alignment chart shown in Figure 5. These optimum conditions do not necessarily represent the actual test parameters that will be used, but serve as a basic guide. In these determinations, the dial position was adjusted to read exactly 100 on the linear scale, using the thickest niobium foil. This adjustment is noted in column 9 as the check point dial reading. The base material, graphite, is then read and noted in column 8.

The rate position switch is for convenience and is actually an electronic control which simply permits the meter needle to move upward half as fast (for a given setup) when switched to the next higher position.

D. Meter Zero Positioning

In all the data presented, the linear dial was adjusted to read zero for the time cycle, using bare graphite over the aperature. Should current fluctuations cause a drift in the zero point after the completion of a coating reading, the reverse-normal switch can be flipped to the reverse position momentarily; and the zero reading is thrown up scale for reference. If the up scale 0 reading does not correspond to the original up scale 0 reading, this amount of deviation may be subtracted (deviation is positive), or added (deviation is negative) to the meter reading. The 0 set dial will correct for drift in the up scale 0 reading without repeating the beta-scan cycle.

E. Calibration Curve Adjustment

Normally, the Micro-Derm has good reproducibility. However, to avoid making new calibration curves due to voltage fluctuations and resulting meter drift, it was found that all points could be brought back on the original curves by simply adjusting the check point dial.

F. Calibration Curves

Calibration curves were plotted for the three flat aperatures in an effort to determine the effect of time on reproducibility and the range of coating thickness that will appear in a linear curve.

1. Aperature 0.250 D-O

Initially, two curves were plotted from data (Table 4) exposing the calibration standards to 0.5 minute and 1.0 minute beta scan. The techniques may be noted in Table 3 as Techniques 126-1 and 126-2 (1.0 minute). No effort was made to bring the two curves (Figure 6) together. It may be noted that the linear portions of the curves are from: 0.4-1.2 mils, 1.24-3.5 mils (extrapolated), and 3.5-5.0 mils. Each point on the curve was plotted from an average of 10 consecutive readings on each standard used. Little difference is noted in the general shape of these two curves.

To obtain a better comparison of the effect of scan time on the linearity of the curve, the two curves at 0.5 minute and 1.0 minute were brought together. To do this, Techniques 127-1 and -2 (Table 3) were used to produce the data in Table 5. These data were then plotted in Figure 7. Very little difference exists in these two curves.

The above four sets of data are plotted on log-log paper (Figure 8).

2. Aperature 0.062 D-O

This is the smallest diameter aperature initially received with the Micro-Derm. From the optimum parameters in Table 2, it was expected that a time cycle greater than one minute would be needed for any degree of accuracy. The first calibration curve (Figure 9) was made, using 1.8 minutes in Technique 122-1 (Table 3). The various points (Table 6) deviated from the curve to a greater degree than when using the 0.250 aperature.

To find the full effect of time on the calibration curves, the scan period was varied from 1.0-2.0 minutes, as shown in Techniques 128-1, -2, -3, and -4 (Figure 3). The calibration data is shown in Table 6. The representative curves are shown in Figure 10. It was not concluded if increased time improved the accuracy since single readings were made in Techniques 128-3 and -4. It is to be noted that the Micro-Derm settings were identical for Techniques 128-3 and -4 except for the rate switch and time.

TABLE 3
MICRO-DERM OPERATION TECHNIQUES

1 Technique Number	2 Date	3 Source Type	4 Source Number	5 Aperature Dia. x H	6 Source Position	7 D_{sw}	8 Tube Pos.	9 Chkpt Dial	10 O-Set Dial	11 Rate Pos.	12 TO Min.
126-1	1-26-65	Sr^{90}	880	0.250 D-O	24	4	3	739	955	3	0.50
126-2	1-26-65	Sr^{90}	880	0.250 D-O	24	4	3	739	930	3	1.00
127-1	1-27-65	Sr^{90}	880	0.250 D-O	24	4	3	674	926	2	0.50
127-2	1-27-65	Sr^{90}	880	0.250 D-O	24	4	3	649	926	3	1.00
122-1	1-22-65	Sr^{90}	880	0.062 D-O	22	2	3	310	638	1	1.80
128-1	1-28-65	Sr^{90}	880	0.062 D-O	23	3	3	960	853	2	1.00
128-2	1-28-65	Sr^{90}	880	0.062 D-O	23	3	3	728	797	2	1.50
128-3	1-28-65	Sr^{90}	880	0.062 D-O	22	2	3	601	818	1	1.00
128-4	1-28-65	Sr^{90}	880	0.062 D-O	22	2	3	601	818	2	2.00
126-3	1-26-65	Sr^{90}	880	0.093 D-O	22	2	3	848	914	2	0.75
126-4	1-26-65	Sr^{90}	880	0.093 D-O	22	2	3	440	893	2	1.00
126-5	1-26-65	Sr^{90}	880	0.093 D-O	22	2	3	740	918	3	1.25
126-6	1-26-65	Sr^{90}	880	0.093 D-O	22	2	3	939	920	3	1.25
126-7	1-26-65	Sr^{90}	880	0.093 D-O	22	2	3	656	890	2	1.00
130-1	1-30-65	Sr^{90}	880	0.093 D-O	22	2	3	647	902	2	1.00
201-1	2-1-65	Sr^{90}	880	0.093 D-O	22	2	3	647	904	2	1.00

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TABLE 4
CALIBRATION STANDARDS

STANDARD BASE MATERIAL			PO3	PO3	PO3	PO3	Foil	ATJ	Foil	PO3	Foil	Foil	Foil	Foil	Foil
STANDARD THICKNESS (NbC)			.0004	.0005	.00055	.0007	.00093	.00124	.00161	.002	.00272	.00395	.00507	.00642	.0079
Date	Technique	M-D Reading													
1-26	126-1	1	19.0			23.5	28.0	33.0		38.0	43.5	48.5	50.5		
	(.5 Min.)	2	" "			23.75	" "	32.5		" "	43.0	47.5	50.0		
		3	" "			23.0	27.75	33.0		37.5	43.5	48.5	" "		
		4	" "			24.0	28.0	" "		" "	42.0	48.0	" "		
	0.25	5	" "			" "	" "	" "		" "	43.0	" "	" "		
	Aperature	6	" "			" "	" "	" "		37.0	" "	" "	" "		
		7	" "			" "	27.5	" "		37.5	" "	" "	" "		
		8	" "			" "	28.0	" "		38.0	42.5	" "	" "		
		9	19.5			23.75	27.5	32.5		37.5	43.0	49.0	50.5		
		10	19.0			" "	28.0	33.0		38.0	42.5	48.0	" "		
		Average	19 05			23.775	27.85	32.9		37.65	42.9	48.15	50.15		
		Avg/4	4.76			5.94	6.975	8.23		9.42	10.72	12.03	12.52		
1-26	126-2	1	38.0			49.5	57.0	68.0		75.5	83.0	95.0	98.5		
	(1.0 Min.)	2	39.5			" "	" "	67.0		76.0	83.5	94.5	98.0		
		3	" "			49.0	" "	67.5		" "	" "	95.0	99.0		
		4	" "			" "	57.25	67.0		77.0	84.5	94.0	98.0		
	0 25	5	" "			48.5	57.0	68.0		76.0	84.0	95.0	" "		
	Aperature	6	38.5			49.5	58.0	67.0		" "	" "	" "	" "		
		–	" "			49.25	56.5	67.5		" "	" "	95.5	" "		
		8	39.0			49.0	58.0	68.0		" "	" "	" "	" "		
		9	" '			49.75	" "	" "		" "	" "	94.5	" "		
		10	38.5			48.5	" "	" "		" "	83.5	95.0	" "		
		Average	38.95			49.1	57.4	67.6		76.05	83.8	94.9	98.15		
		Avg/4	9 73			12.25	14.34	16.85		19.0	20.95	23.7	24.5		

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TABLE 5
CALIBRATION STANDARDS

STANDARD BASE MATERIAL			PO3	PO3	PO3	PO3	Foil	ATJ	Foil	PO3	Foil	Foil	Foil	Foil	Foil
STANDARD THICKNESS (NbC)			.0004	.0005	.00055	.0007	.00093	.00124	.00161	.002	.00272	.00395	.00507	.00642	.0079
Date	Technique	M-D Reading													
1-27	127-1 (.5 Min.)	1	34.0	37.5		43.0	51.0	61.0		67.0	75.5	83.0	88.0		
		2	34.0	" "		" "	50.5	60.5		67.0	74.5	84.0	" "		
		3	33.0	36.5		42.5	" "	59.5		67.5	75.0	86.0	87.5		
	0.25 Aperature	4										85.0			
		Average	33.7	37.1		42.8	50.7	60.3		67.2	75.0	84.5	87.83		
		Avg/4	8.42	9.275		10.7	12.7	15.1		16.75	18.7	21.1	21.95		
1-27	127-2 (1.0 Min.)	1	33.0	37.0		42.0	50.0	59.0		66.0	75.0	85.0	87.0		
		2	33.5	" "		43.5	" "	" "		67.5	" "	84.0	87.5		
		3	33.0	" "		42.5	" "	" "		68.0	74.0	" "	88.0		
	0.25 Aperature	Average	33.3	37.0		42.7	50.0	59.0		67.2	74.8	84.4	87.5		
		Avg/4	8.33	9.25		10.65	12.5	14.75		16.8	18.7	21.1	21.83		

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TABLE 6
CALIBRATION STANDARDS

STANDARD BASE MATERIAL			PO3	PO3	PO3	PO3	Foil	ATJ	Foil	PO3	Foil	Foil	Foil	Foil	Foil
STANDARD THICKNESS (NbC)			.0004	.0005	.00055	.0007	.00093	.00124	.00161	.002	.00272	.00395	.00507	.00642	.0079
Date	Technique	M-D Reading													
1-22	122-1	1	23.5	28.0	27.5	34.0	43.5	56.0	65.5	64.0	81.0	94.0			
	(1.8 Min.)	2	22.0	" "	28.0	33.0	43.0	" "	64.5	68.0	79.0	" "			
		3	24.5	26.0	29.5	35.0	" "	" "	63.4	67.5	81.0	" "			
	.062	4	23.0	28.5	30.0	34.0	42.0	" "	64.0	68.0	79.0	" "			
	Aperature	Average	23.2	27.7	28.75	34.0	42.8	56.0	64.3	66.8	80.0	94.0			
		Avg/4	5.8	6.93	7.18	8.5	10.7	14.0	1o.1	16.7	20.0	23.5			
1-28	128-1	1	27.5	30.5		34.75	43.5	52.0		63.0	71.5	87.0	91.0		
	(1.0 Min.)	2	27.5	" "		35.0	42.0	54.5		63.5	71.0	85.0	90.0		
		3	27.5	" "		" "	43.0	54.0		62.5	72.0	87.0	89.5		
	.062	4					42.0	53.0		64.0		85.0	91.0		
	Aperature	Average	27.5	30.5		34.9	42.6	53.5		63.3	71.5	86.0	90.5		
		Avg/4	6.88	7.63		8.72	10.65	13.33		15.8	17.85	21.5	22.6		
1-28	128-2	1	30.5	34.0		40.0	44.5	57.5		64.5	71.0	84	90.0		
	(1.5 Min.)	2	29.5	35.0		39.0	46.0	57.0		64.0	72.	85	89.0		
		3	31.0	33.5		38.0	" "	" "		64.0	73.	84.5	89.5		
	.062	4	31.0	34.5			48.0	56.0				84			
	Aperature	5						56.5							
		Average	30.6	34.2		39.0	46.2	56.75		64.0	72.	84	89.5		
		Avg/4	7.67	8.55		9.75	11.5	14.2		16.0	18.	21	22.4		
1-28	128-3	1	29.5	31.5		38.5	45.0	56.0		66.0	72.0	84.5	88.0		
	(1.0 Min.)														
	.062 Aper.	reading/4	7.48	7.88		9.62	11.25	14.0		16.5	18.0	21.1	22.0		
1-28	128-4	1	30.0	32.0		39.0	46.5	55.0		63.5	72.0	85.0	89.5		
	(2.0 Min.)	2									72.0	85.5	89.0		
	.062 Aper.	reading/4	7.5	8.0		9.75	11.6	13.75		15.9	18.0	21.2	22.4		

The same typical curves were produced with the 0.062" diameter aperature as with the 0.250" diameter aperature.

3. Aperature 0.093 D-O

With this medium-size aperature, three curves were drawn to show any effect of variation in time. The techniques are shown in Table 3 as 126-3, -4, and -5. The Micro-Derm readings are shown in Table 7 for the time intervals of 0.75, 1.0, and 1.25 minutes. The three curves are shown in Figure 11. The one-minute curve represented as Technique 126-4 appears the most accurate. It can be noted in Figure 11 that the values are in inches that each Micro-Derm scale division represents for the given linear portion of the curve. There is some advantage in increasing the slope of the curve since the error from each Micro-Derm scale division becomes less. These same data are plotted on log-log scale in Figure 12.

Since Technique 126-5 appeared to extend the low range curve from 1.24 mils to 1.42 mils following a linear curve, it was decided to repeat this and increase the slope of the curve at the same time, using Technique 126-6 (Table 3). Single readings (Table 7) were used to plot the curve (Figure 13). The upper limit of the low-range linear curve was apparently extended to 1.46 mils, where it intersects the projection from the middle-range linear curve. The data from Technique 126-6 are also plotted on log-log scales (Figure 14), which correspond closely to Technique 126-3.

Technique 126-7 (Table 3) was designed to increase the slope of the curves and to bring the low and medium-range curves together (as shown in Curve 126-4, Figure 12). This curve, however, shown in Figure 15, is similar to Figure 13 without a distinct intersection point.

Technique 130-1 is similar to Technique 126-7 and is used for computing actual niobium-coating thickness in the following section. The data for this technique (130-1) is shown in Table 8. Representative calibration curves are shown in Figures 16 and 17.

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TABLE 7
CALIBRATION STANDARDS

STANDARD BASE MATERIAL			PO3	PO3	PO3	PO3	Foil	ATJ	Foil	PO3	Foil	Foil	Foil	Foil	Foil
STANDARD THICKNESS (NbC)			.0004	.0005	.00055	.0007	.00093	.00124	.00161	.002	.00272	.00395	.00507	.00642	.0079
CONVERTED THICKNESS(NbC)															
Date	Technique	M-D Readings													
1-26	126-3	1	32.0			40.5	50	58.5		67.0	74.0	85.0	89.0		
	(.75 Min.)	2	34			41.5	49	58.0		66.0	" "	84.5	88.0		
		3	33.5			43.5	" "	59.5		66.0	" "	85.5	90.0		
	.093	4	34			41.0	50	59.0		66.0	" "	86.0	90.0		
	Aperature	5	32.5			41.0	49.5	58.5		67.5	" "	" "	89.5		
		Average	33.2			41.4	49.7	48.7		66.5	74.0	85.2	89.3		
		Avg/4	8.3			10.35	12.42	14.7		16.6	18.5	21.3	22.3		
1-26	126-4	1	27.0	29.5		34.0	41.0	48.0		53.5	61.0	71.0	74.0		
	(1.0 Min.)	2	27.5			35.0	39.5	49.0		55.0	61.5	71.5	73.0		
	.093	3	26.5			33.5	40.5	48.0		54.5	60.5	71.5	73.5		
	Aperature	Average	27.0	29.5		34.2	40.3	48.3		54.3	61.0	71.3	73.5		
		Avg/4	6.75	7.38		8.54	10.08	12.1		13.52	15.23	17.8	18.37		
1-26	126-5	1	23.5	26.0		30.0	35.0	41.0		49.0	54.5	62.0	65.0		
	(1.25Min.)	2	23.0	26.0		29.5	35.5	" "		" "	54.0	" "	66.0		
	.093	3	24.0	27.0		30.0	35.0	" "		" "	53.5	" "	65.0		
	Aperature	Average	23.5	26.3		29.8	35.2	41.0		49.0	54.0	62.0	65.3		
		Avg/4	5.88	6.57		7.44	8.78	10.25		12.22	13.5	15.5	16.3		
1-26	126-6 (1.25Min.)	1	34.0	37.0		42.0	49.0	57.5		69.5	77	88.0	92.0		
	.093 Aper.	reading/4	8.5	9.25		10.5	12.23	14.25		17.35	19.25	22.0	23.0		
1-26	126-7 (1.0 Min.)	1	32.5	36.0		41.5	49.25	57.5		67.5	73.5	86.0	90.0		
	.093 Aper.	reading/4	8.13	9.0		10.4	12.3	14.35		16.85	18.35	21.45	22.5		

TABLE 8
CALIBRATION STANDARDS

STANDARD BASE MATERIAL			PO3	PO3	PO3	PO3	Foil	ATJ	Foil	PO3	Foil	Foil	Foil	Foil	Foil
STANDARD THICKNESS (NbC)			.0004	.0005	.00055	.0007	.00093	.00124	.00161	.002	.00272	.00395	.00507	.00642	.0079
CONVERTED THICKNESS (NbC)							.00133	.00164	.00201	.0024	.00312	.00435	.00548	.00683	.0083
Date	Technique	M-D Reading													
1-30	130-1	1	32.5	35	35.5	39.5	47.5	58.0	62.0	65	71.5	83.5	86	90	
		2	31	"	35	39.0	46.5	56	61	65	73.5	83	86	90	90
		3	31	"	36	40.0	47	56	62.5	65.5	73	84.5	85.5	90	
		4	31.5	35.5	36	"	47	58	61	64	72.5	82	87	91	
		Average	31.5	35.1	35.6	39.6	47	57	61.8	64.8	70.3	83.3	86.2	90.3	
		Avg/4	7.87	8.775	8.9	9.9	11.75	14.25	15.45	16.2	17.54	20.8	21.5	22.6	
2-1	201-1	1	32.0	35	37.5	41.5	49.5	60.5	66.0	68.0	72.5	86.0	90.0	93	
		2	34.0	36.5	36.5	43.0	50.0	61.0	66.5	68.5	73.0	86.0	88	94	
		3	32.5	37	37.0	42.0	50.0	60.5	63.0	68	75.0		89	94.5	
		4	33.0	37.5	38.0		49.5	65.5	67.5	74.0	74.0			93	
		Average	32.8	36.8	37.2	42.2	49.75	60.75	65.3	68.0	73.8	86.0	89	93.7	
		Avg/4	8.22	9.12	9.32	10.55	12.45	15.2	16.3	17.0	18.4	21.5	22.2	23.42	
2-2	202-1	1	31	34.5	35.7	39.5	47	57	63	66	72	83	88	91	
		2					48		62		71				
		3									72				
		Average	31	34.5	35.7	39.5	47.5	57	62.5	66	71.7	83	88	91	
		Avg/4	7.75	8.63	8.83	9.875	11.87	14.25	15.6	16.5	17.93	20.72	22	22.72	
2-3	202-1	1	31	34.5	36	39.5	49	57.5	62	66					
		2					47	57	63						
		3					48	58.0	62						
		Average	31	34.5	36	39.5	48	57.5	62.3	66					
		Avg/4	7.75	8.63	9.0	9.875	12.0	14.35	15.58	16.5					

Since there were no major advantages in the use of one aperature size over another, except time, it was decided to read actual coating thicknesses using the medium-size (0.093 diameter) aperature described in Technique 130-1.

IV. COATING THICKNESS DETERMINATION

A. Range of NbC Thickness

A number of support blocks were selected from the Materials Department, Building 9, which had previously been sectioned and the coating thickness determined in the Metallography Lab. The method used is to section and polish the surface transverse to the coated surface and measure the thickness at 200X magnification in filar units. The filar units are multiplied by a factor (0.00177) to give thickness in mils. Blocks with a coating thickness range of 1.9-4.6 mils were selected for reading with the Micro-Derm.

B. Procedure

Using Technique 130-1 and the resulting curve previously described, 49 determinations were made on the Micro-Derm. Their distribution is shown in Figure 18, comparing Metallography Lab results with the Micro-Derm. As can be noted, nearly all points over the full range were to the left of the ideal correlation curve. By drawing a line through the average distribution, it was noted that the Micro-Derm was reading 0.0004" low over the full range.

C. Results

A new calibration curve was prepared (Figure 19) using values for calibration standards that were increased by 0.0004 (NbC) and a factor for Nb.

Relating the new calibration standard values (Table 8) of NbC with the thickness of the niobium foil, a correlation chart was prepared (Figure 20.) It is noted that the NbC/Nb ratio is not a uniform factor of 1.235, but graduates from 1.65 for 1 mil NbC to 1.285 for 7 mils Nb foil. This probably results from the difference in density of NbC with the increase in thickness. This may also be attributable to a transition of niobium carbide to pure niobium as the thickness increases.

The original Micro-Derm readings for the block coating were plotted on the curve shown in Figure 19. These data were then recorded on a new correlation chart (Figure 21). Nearly 100% of these points fell within 10% of the ideal correlation between the Metallography Lab and the Micro-Derm. A numerical presentation of the coating thickness comparison is shown in Table 9.

V. CONCLUSIONS*

From the above investigation in the use of a commercial beta-backscattering thickness gage, it can be concluded that:

A. Niobium carbide coating over graphite in a range of 1.9-4.5 mils can be read on the Micro-Derm within 10% of the results reported by metallography.

B. Within the scope of this investigation, there are three linear segments within the typical calibration curve. These may be due to:

1. Low activity of the beta source.

2. Density variations as the NbC thickness increases.

3. Possible transition of NbC to Nb as the thickness increases (should be confirmed by micro-probe or x-ray diffraction.

C. Since there appears to be no correction needed in the low range (0.4-1.24 mils) portion of the calibration curves presented, it is believed that the Micro-Derm gage is satisfactory for testing exterior surfaces of fueled and non-fueled elements where the upper limit is 0.5 mil thickness.

D. It is acknowledged that the Micro-Derm with its low activity source and Geiger-Muller Tube detector lacks the speed of the more elaborate solid-state detectors with electronic counters and digital converters.

---

*Supplementary conclusions may be found in Appendix D covering results following equipment modifications.

TABLE 9*
MICRO-DERM, NbC BETA-BACKSCATTER THICKNESS DETERMINATION

S/N			QCOI 36		Metallographic		Micro-Derm		Deviation		Micro-Derm		
(Ht)	Run Number	Type	Fig.	No.	Original	Recheck	Single	Average	Mils	Percent	Technique	Curve	Ap. Size
1010	142	J4	4	5	2.76		2.95		+.19		130-1	130-1	.093 D-0
				6	2.44		2.80		+.36				
678	Std 17	J4	4	21	2.97		3.13		+.14				
	Std 20			20	3.86		----		----				
896	Std 16	J4	4	21	2.87		2.88		+.01				
	Std 18			20	3.01		3.13		+.12				
1003	Std 0032	J4			3.20		3.13		-.07				
740	B 237	J8	4	20	4.39	4.43	4.05		-.38				
				21	4.48	4.38	4.12		-.26				
				2	3.86	3.90	4.14		+.24				
				3	3.98	3.96	3.94		-.02				
19855	B 237	J4	4	6	4.33	4.32		3.98	-.34				
				7	4.51	3.58		3.65	+.07				
				8	3.54	3.58		3.65	+.07				
				24	4.25	4.25		4.26	+.01				
				25	4.51	4.52		4.20	-.32				
				2	4.42	4.42		4.05	-.37				
				3	4.44	4.46		4.05	-.39				
				4	4.16	4.13		4.05	-.08				
19918	B 237	J7	4	6	3.54	3.54		3.83	+.29				
				7	2.65	2.67		2.88	+.23				
				8	2.44	2.40		2.70	-.26				
				21	4.25	3.72		3.83	+.11				
				22	4.14	4.03		4.26	+.23				
				2	3.72	3.72		3.80	+.08				
				3	4.46	4.07		4.05	-.02				
				4	4.33	4.39		4.02	-.37				
19876	B 237	J5	4	6	4.12			4.02	-.08				
				7	3.29	3.54		3.80	+.26				
				24	4.12			4.26	+.14				
				25	3.96	4.28		4.34	+.06				
				26	4.16			4.08	-.08				
				2	3.54	3.96		4.02	+.06				
				3	3.84	3.89		3.98	+.11				
				4	3.57	3.54		3.98	+.41				
				38	1.95			2.32	+.27				

*Page 1 of 2 pages

S/N (Ht)	Run Number	Type	QCOI 36 Fig.	QCOI 36 No.	Metallographic Original	Metallographic Recheck	Micro-Derm Single	Micro-Derm Average	Deviation Mils	Deviation Percent	Micro-Derm Technique	Micro-Derm Curve	Micro-Derm Ap. Size
110	B 237	J9	4	7	4.28			3.90	-.38		130-1	130-1	.093 D-0
				8	3.77			3.58	-.19				
				2	4.67	4.48		4.01	-.47				
				3	4.33	4.03		3.80	-.30				
	Porous Coating			4	4.21			4.01	-.20				
	on Block			5	4.74	4.54		4.24	-.30				
740	B 237	J8	4	20	4.39			4.24	-.15				
				21	4.48			4.35	-.13				
				2	3.86			3.90	+.04				
				3	3.98			4.01	+.13				
42	A55	J6	4	6	3.11	3.19	3.13		-.06				
						3.16	3.00		-.16				
						3.06	3.00		-.06				
						2.85	2.84		-.01				
						2.82	2.80		-.02				
						2.69	2.70		+.01				
						2.58	2.48		-.10				
						2.51	2.66		+.15				
						2.44	2.56		+.12				
						2.37	2.38		+.01				
						2.28	2.18		-.10				
						2.23	2.12		-.11				
						2.20	2.05		-.15				
						2.09	1.93		-.16				
						1.98	2.05		+.07				
						1.93	1.88		-.05				
				8	1.66	1.91	1.83		-.08				
495				43		1.95							
				44									
1212				43	(angle)	2.00							
				44									
				46	(angle)	2.10							
1482				41									
				42		2.30	2.58		+.28				
				43									

E. To make the Micro-Derm more versatile, an inexpensive slotted operature and hole-locating fixture may be purchased to read the flat surfaces between the channels.

F. The Micro-Derm is suitable for measuring very thin coatings (less than 0.5 mils) since the following isotope sources are available:

Carbon-14	1.155 Mev.
Premethium-147	0.22 Mev.
Thallium-204	0.76 Mev.
Radium-D	1.17 Mev.
Strontium-90	2.18 Mev.

G. The Micro-Derm may be equipped with output for connection to any standard external scaler to possibly improve the readout.

H. The repeatability of duplicating calibration readings over the wide range of standards is excellent where one Micro-Derm meter scale division represents approximately 0.0001 inches.

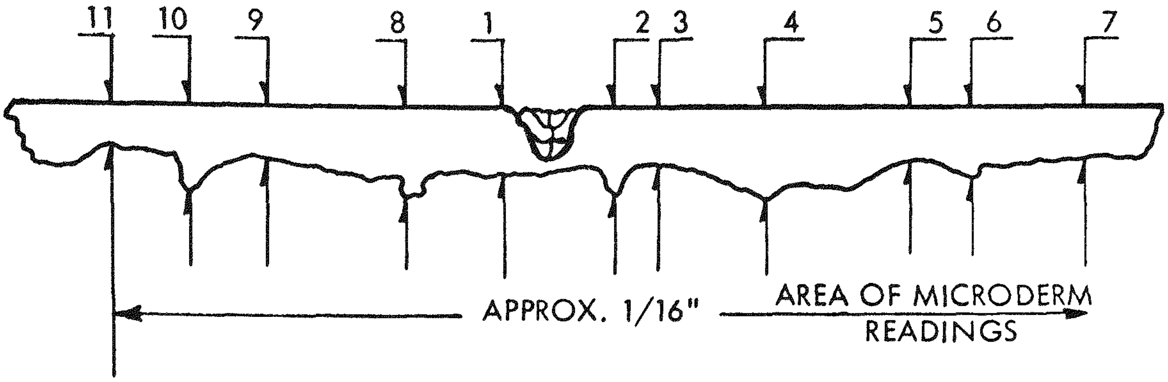
I. Little training is needed for technicians or inspectors to operate the Micro-Derm. Care is to be exercised in mishandling the Strontium-90 source tube, the Geiger-Muller Tube window, and coated parts.

J. Further work is justified in obtaining statistical data in the use of 0.062" and 0.093" operatures for NbC coatings.

K. There is a need for obtaining full-range calibration standards of NbC over graphite.

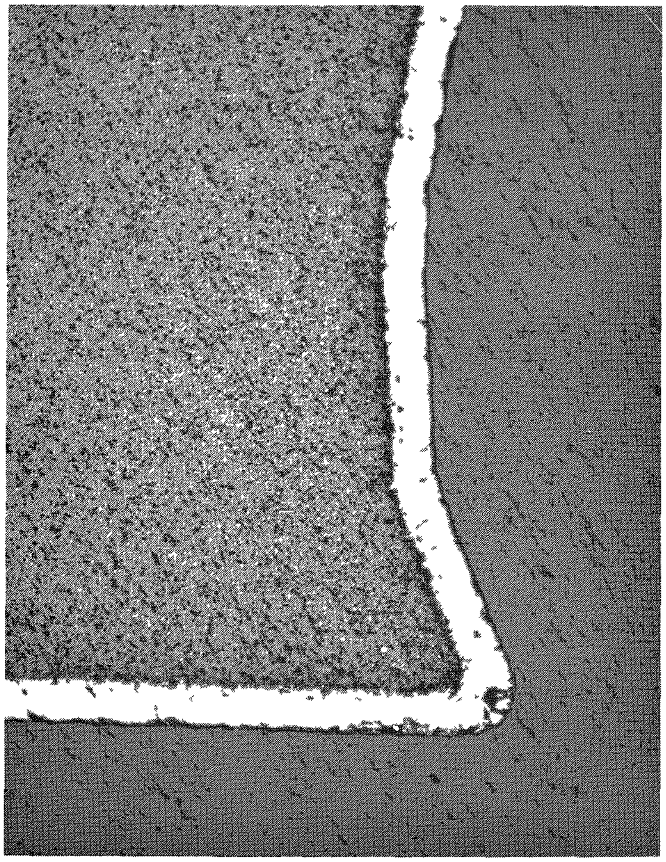
PROFILE OF CALIBRATION HT1003 (ATJ)

STANDARD AT 200 x POSITION 21 (INCH FROM END)



COATING THICKNESS (INCHES)	
METALLOGRAPH READINGS	
1	.00277
2	.00470 (SPIKE)
3	.00272
4	.00403 (SPIKE)
5	.00293
6	.00359 (SPIKE)
7	.00261
8	.00354 (SPIKE)
9	.00238
10	.00354 (SPIKE)
11	.00254
AV	.00321

FIGURE 1



COATED LTV GRAPHITE

7200 100X

FIGURE 2

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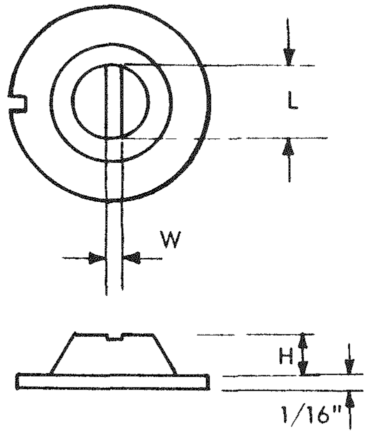
STANDARD DOME-APERTURE PLATES MD-AP

Slit-Aperture Type



Designation
W x L x H

W = Width of slit in thousandths of an inch.
L = Length of slit * (or diameter of dome) in 32nds of an inch.
H = Height of top of dome above level of top of flat aperture plate, in 32nds of an inch.

Example No. 1:

.062 x 7 x 3

Width of slit is 0.082"	Length of slit is 7/32"	Height of dome is 3/32"

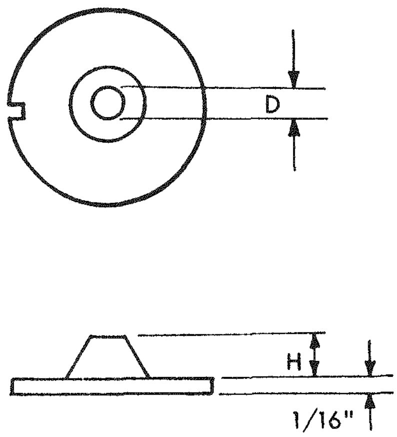
Example No. 2:
A flat aperture plate

.048 x 16 x 0

Width of slit is 0.048"	Length of slit is 16/32" 1/2"	H = 0 means flat plate

*The effective length of slit is slightly smaller than the dome diameter, due to the wall thickness of the dome side-walls.

Circular-Aperture Type



Designation
D x H

D = Diameter of circular aperture opening, in thousandths of an inch.
H = Height of top of dome above level of top of flat aperture plate, in 32nds of an inch.

Example No. 1:

156 D x 3

Aperture opening is .156" (5/32") I.D.	Height of dome is 3/32"

Example No. 2:
A flat aperture plate

.250 D x 0

Diameter of aperture in .250" I.D.	H = 0, means flat plate

FIGURE 3

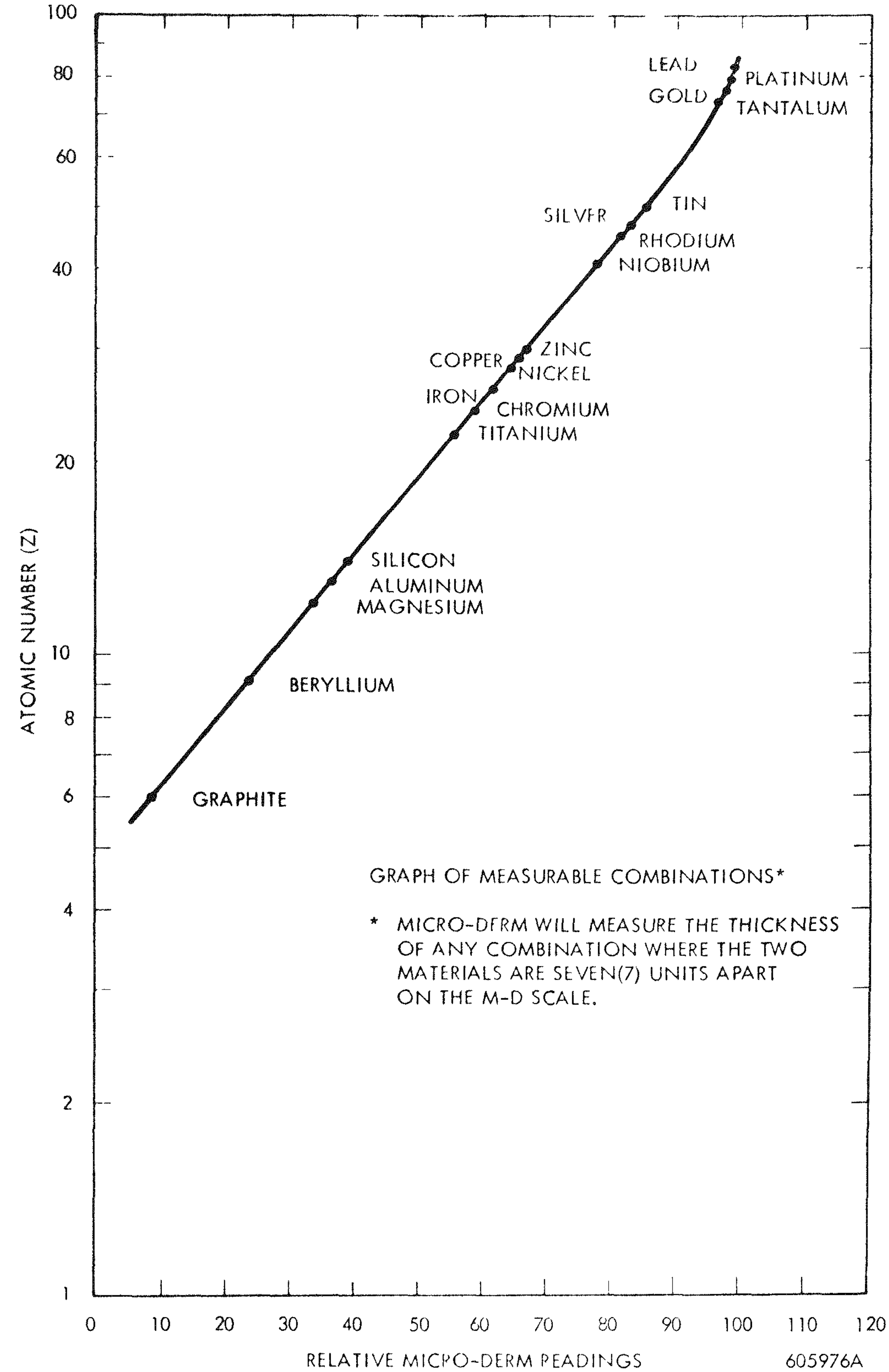
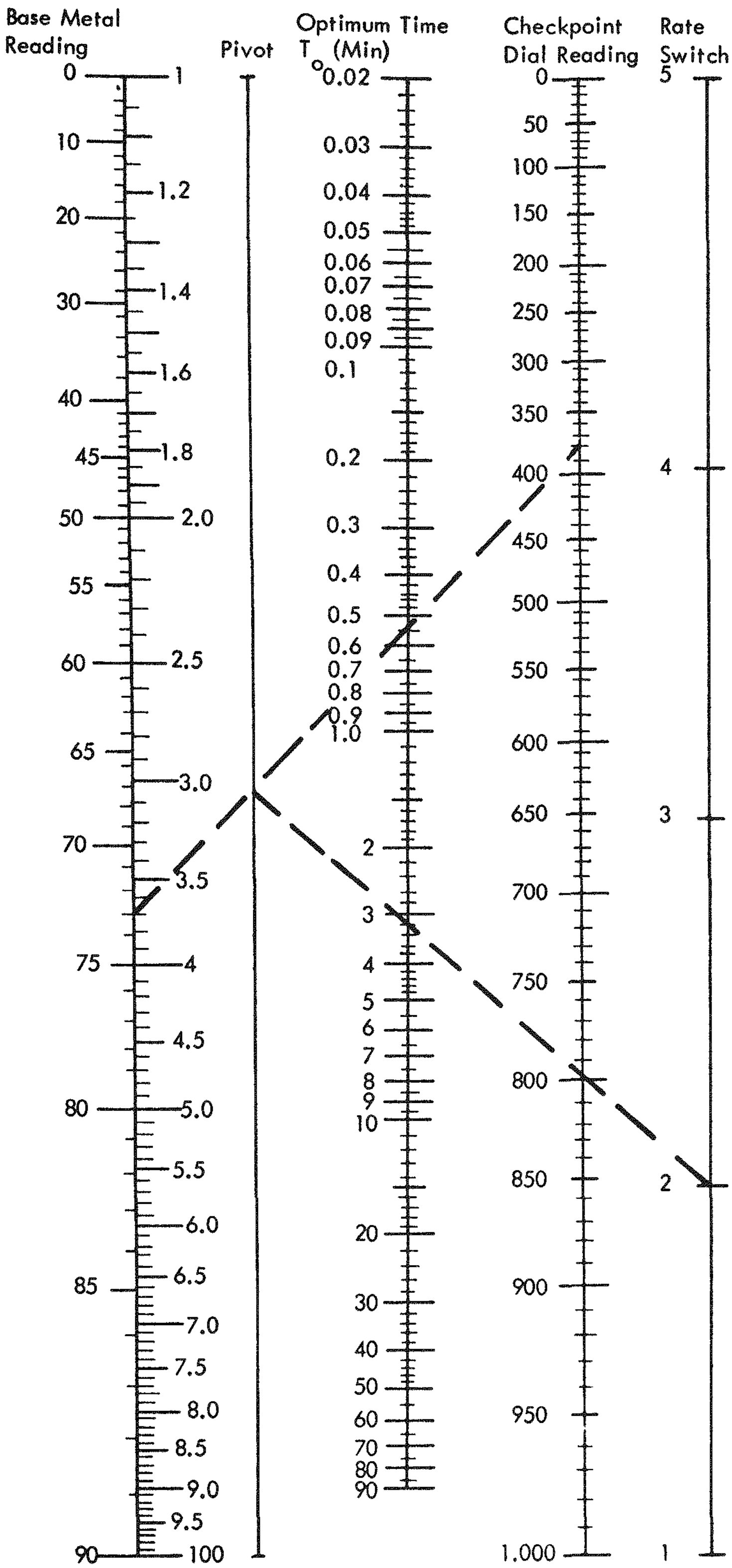

ATOMIC NUMBER (Z)
RELATIVE MICRO-DERM READINGS
605976A
LEAD
PLATINUM
GOLD
TANTALUM
TIN
SILVER
RHODIUM
NIOBIUM
ZINC
COPPER
NICKEL
IRON
CHROMIUM
TITANIUM
SILICON
ALUMINUM
MAGNESIUM
BERYLLIUM
GRAPHITE
GRAPH OF MEASURABLE COMBINATIONS*
* MICRO-DERM WILL MEASURE THE THICKNESS OF ANY COMBINATION WHERE THE TWO MATERIALS ARE SEVEN(7) UNITS APART ON THE M-D SCALE.

Figure 4



DETERMINATION OF T_o

This chart determines the optimum time, T_o, in terms of figures read directly from the MICRO-DERM. Readings are based on a one minute time period, as explained in the operating instructions.

Note that may be read from the chart directly to the right of the base metal reading.

Sample Drawn on Chart
(dashed lines at left)

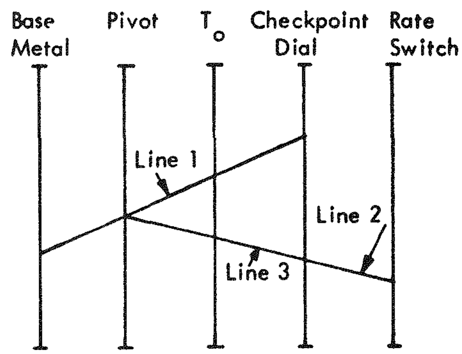
Base Metal Reading: 73

Checkpoint Dial Reading: 380

Rate Switch Position: 2

Optimum TIme: 3.2 Min.

Instrumctions



1. Draw line from "Base Metal Reading" to "Checkpoint Dial Reading".
2. From intersection of Line (1) with "Pivot" line, draw line to rate switch position.
3. Intersection of this second line with "T_o" line is optimum time in minutes.

Figure 5

605978A

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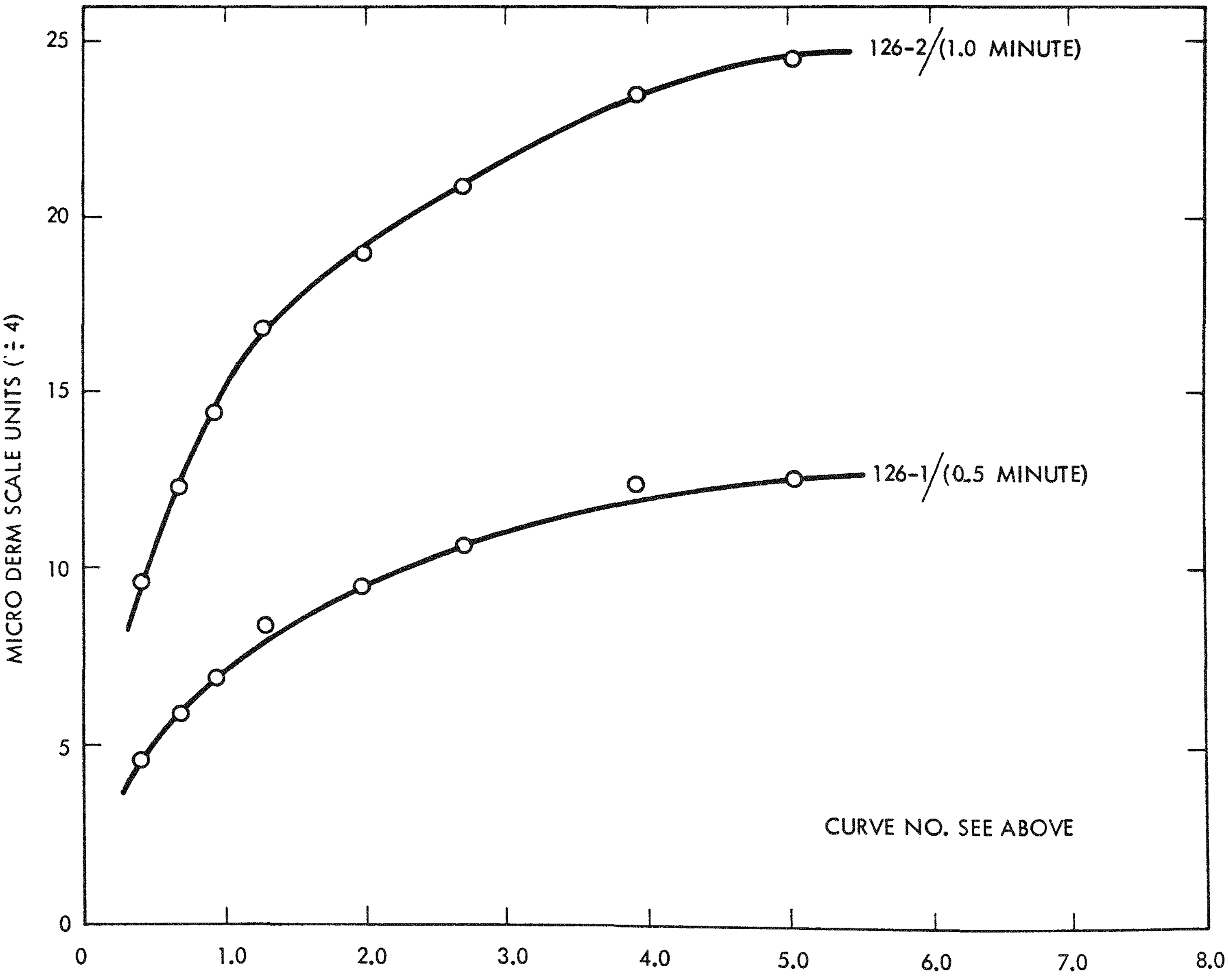

Figure 6

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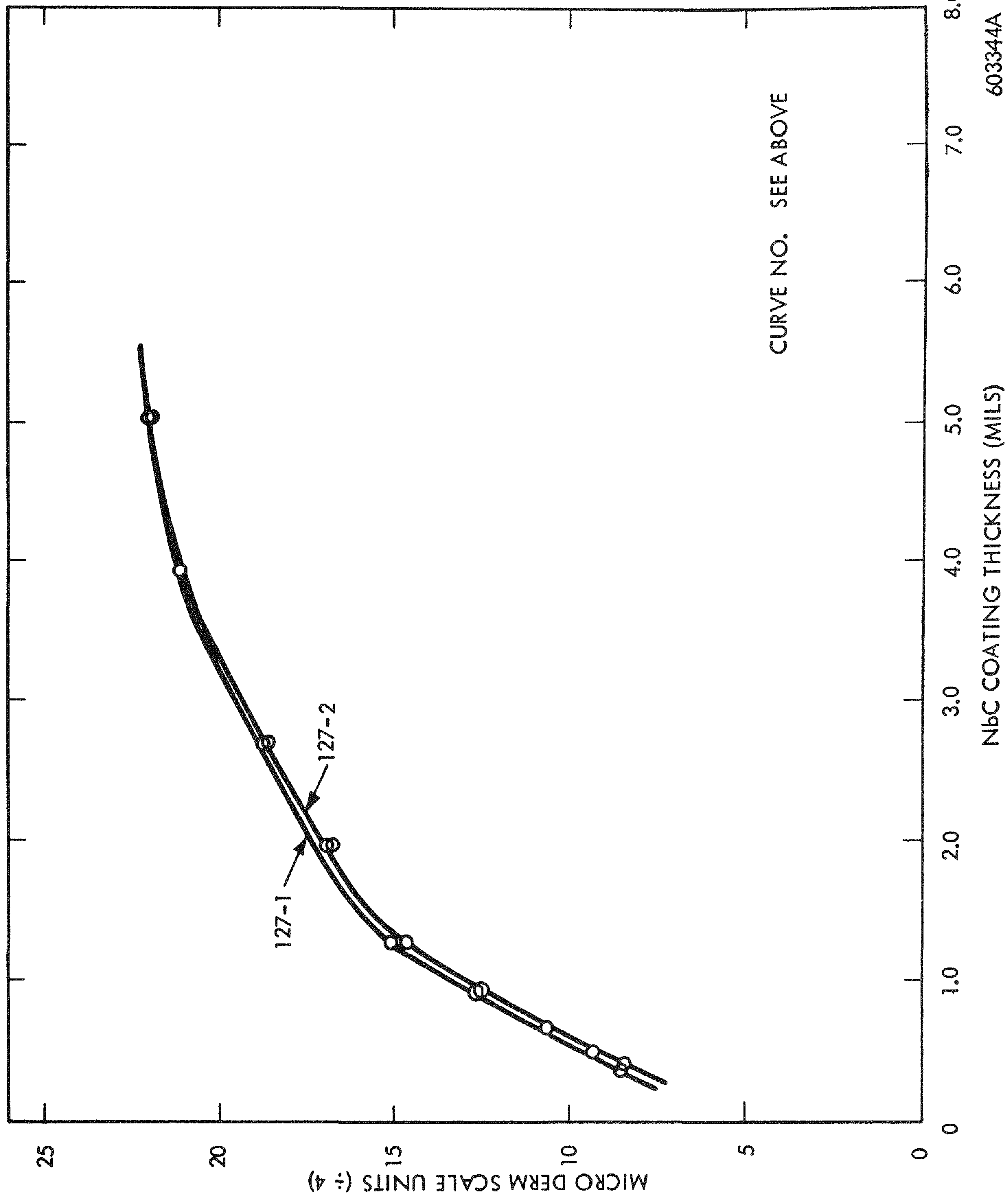
CURVE NO. SEE ABOVE
127-1
127-2
MICRO DERM SCALE UNITS (÷ 4)
0
5
10
15
20
25
1.0
2.0
3.0
4.0
5.0
6.0
7.0
8.0
NbC COATING THICKNESS (MILS)
603344A

Figure 7

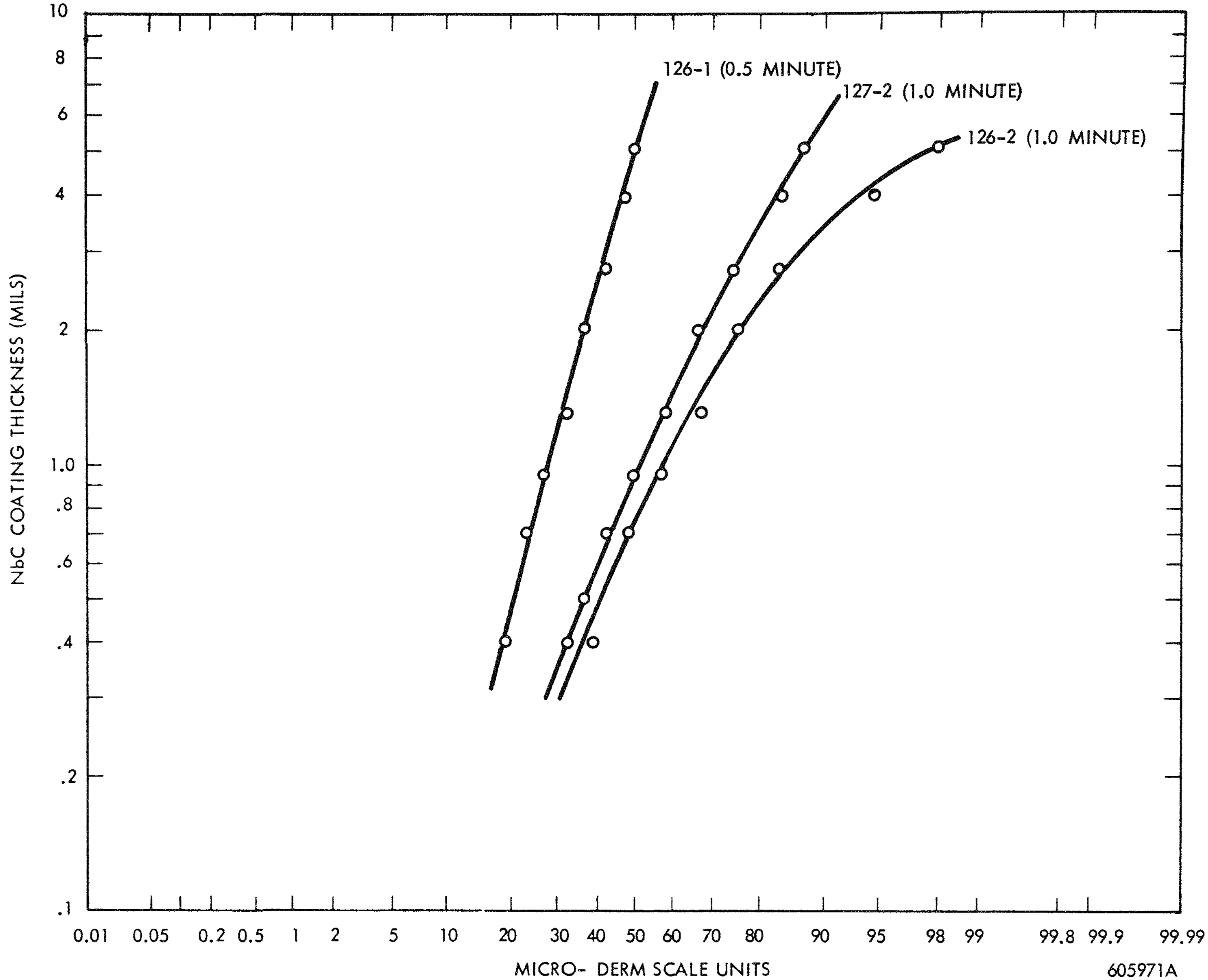
126-1 (0.5 MINUTE)
127-2 (1.0 MINUTE)
126-2 (1.0 MINUTE)
NbC COATING THICKNESS (MILS)
10
8
6
4
2
1.0
.8
.6
.4
.2
.1
0.01
0.05
0.2
0.5
1
2
5
10
20
30
40
50
60
70
80
90
95
98
99
99.8
99.9
99.99
MICRO- DERM SCALE UNITS
605971A

Figure 8

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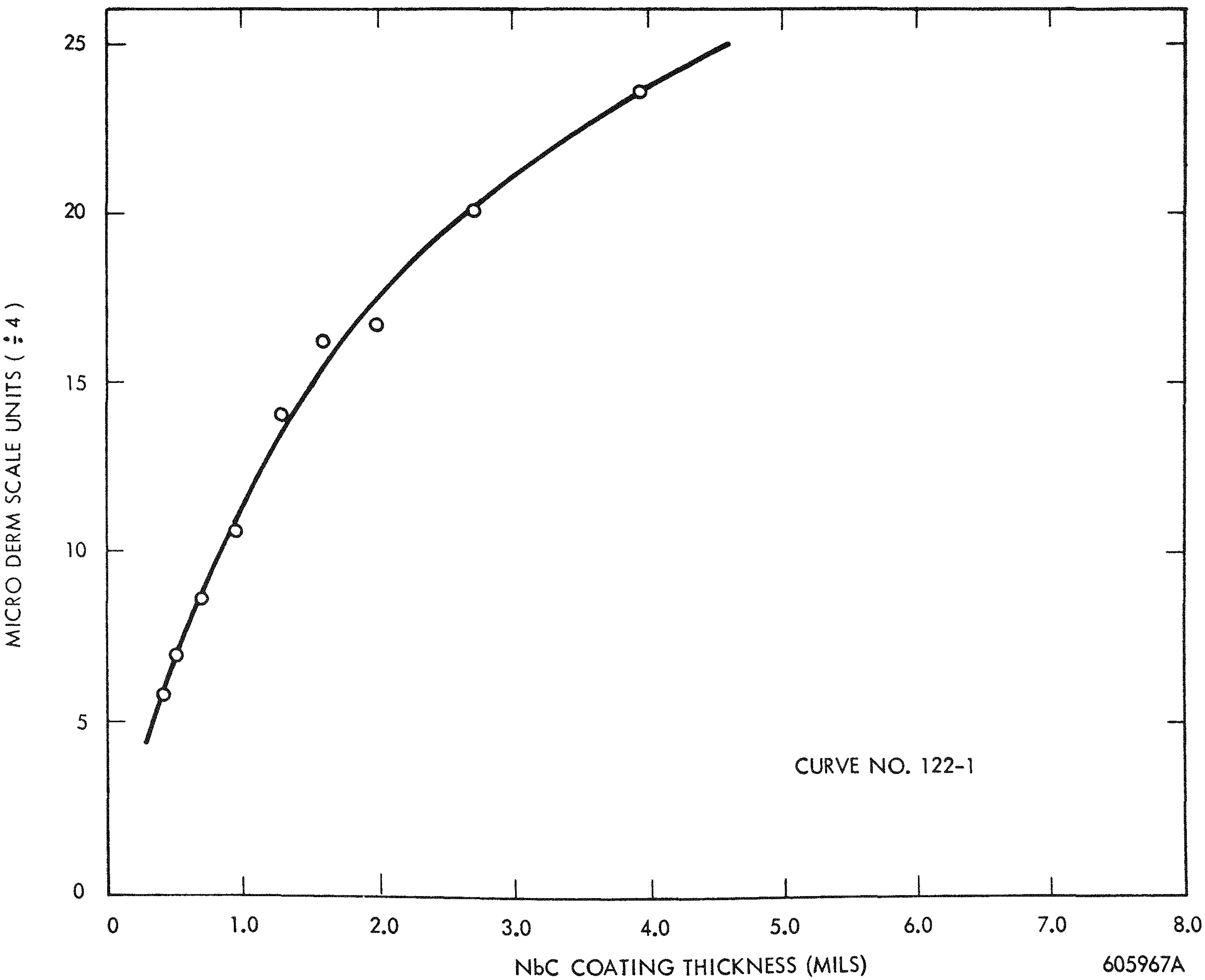

Figure 9

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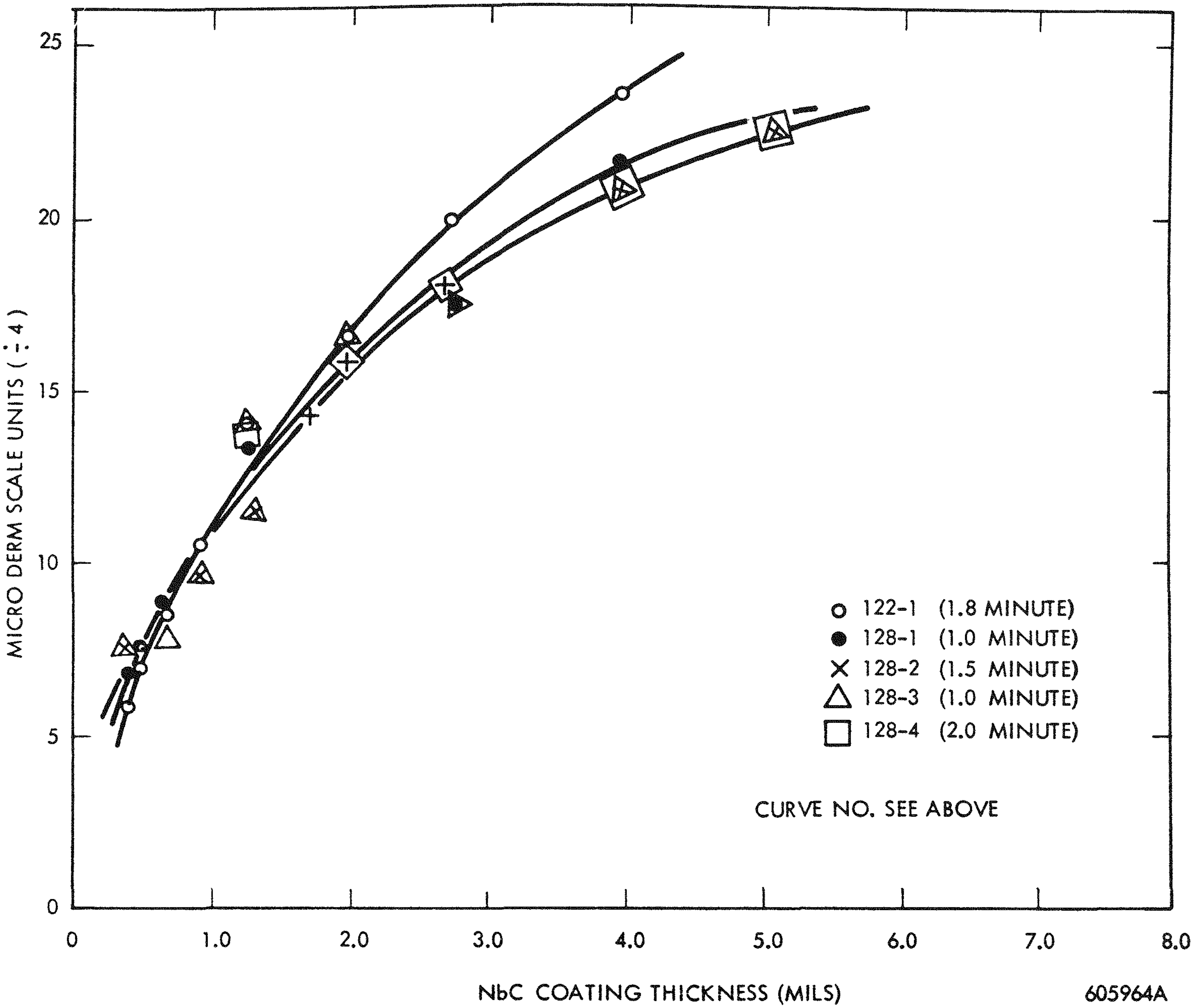

Figure 10

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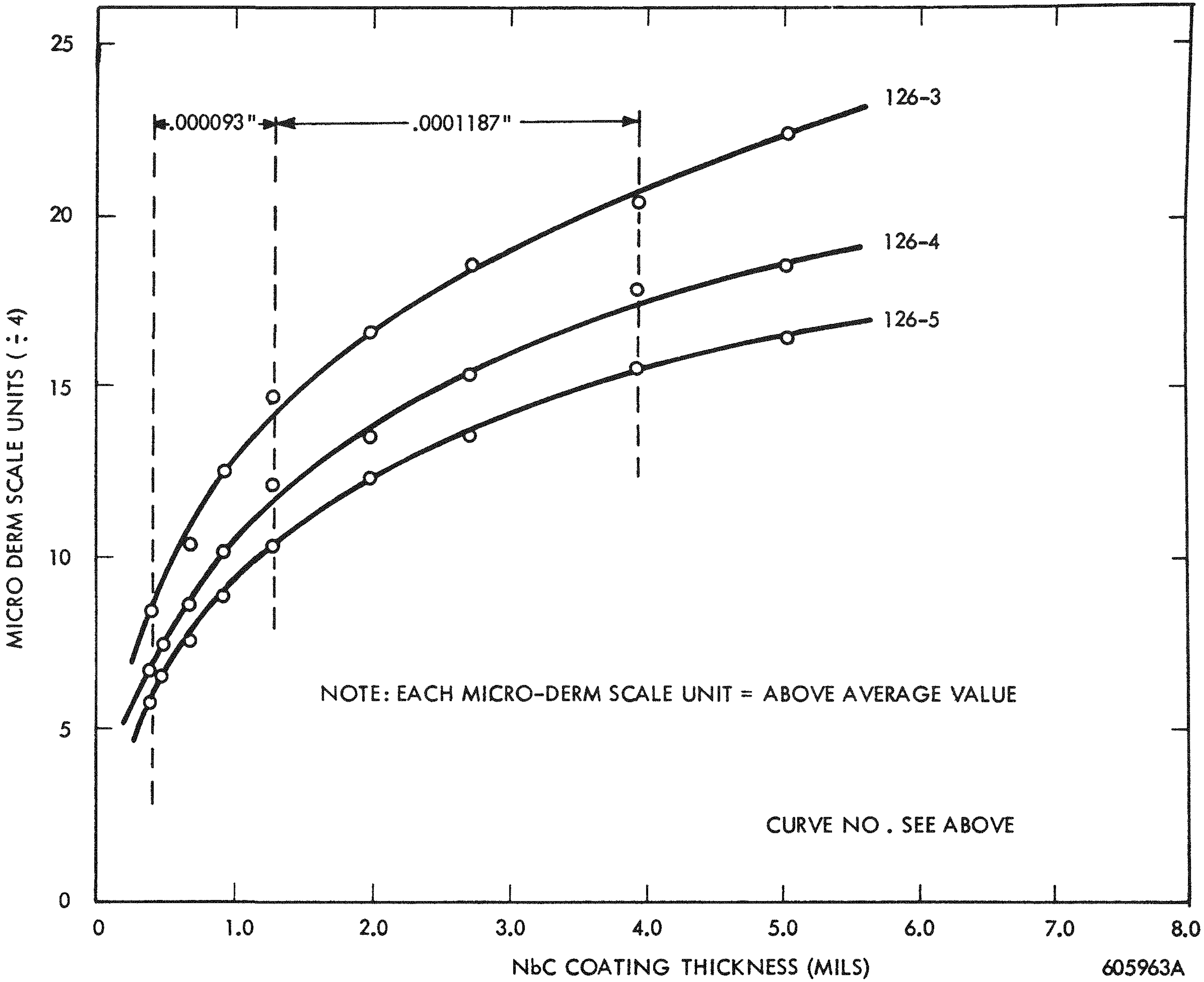

Figure 11

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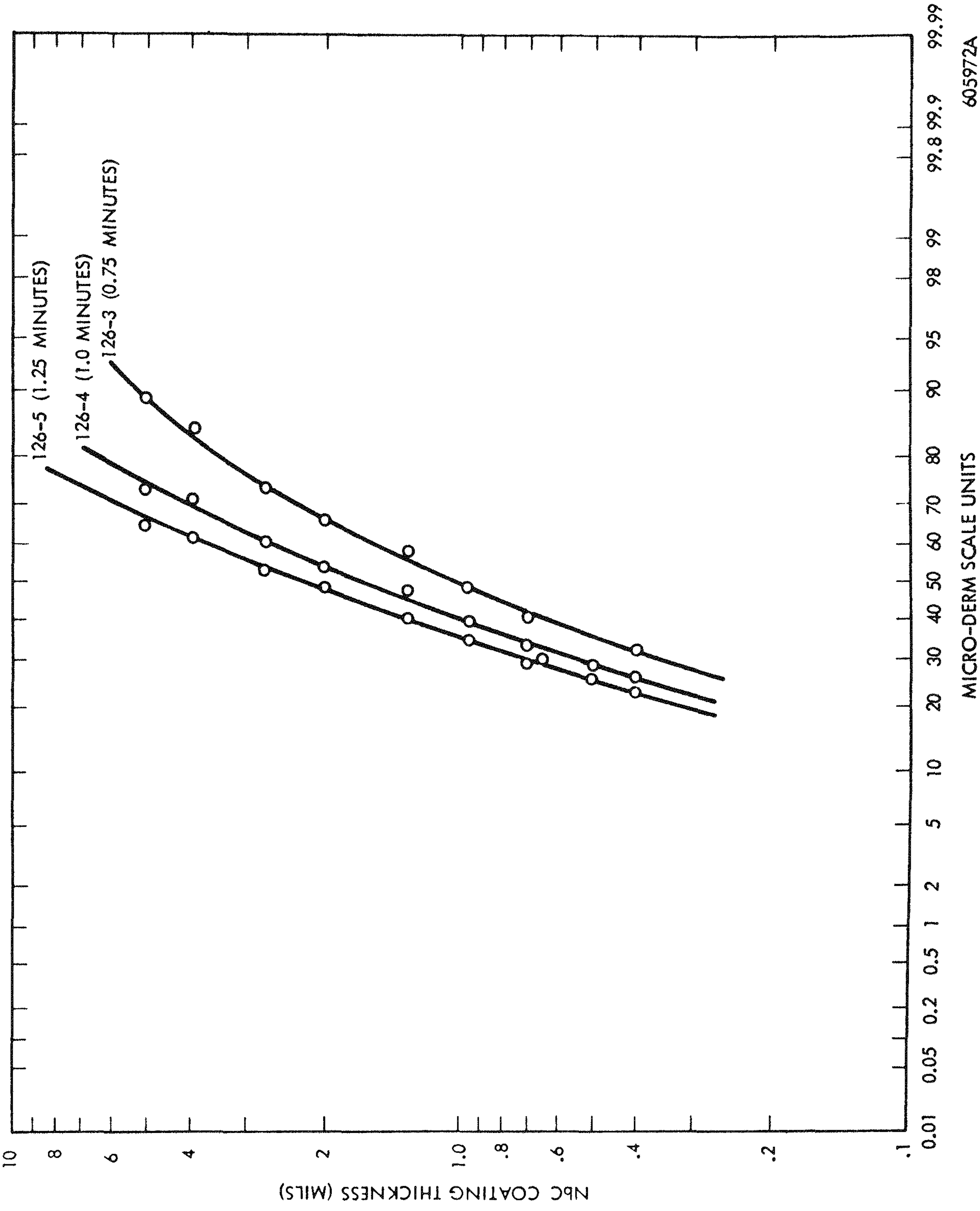
126-5 (1.25 MINUTES)
126-4 (1.0 MINUTES)
126-3 (0.75 MINUTES)
10
8
6
4
2
1.0
.8
.6
.4
.2
.1
NbC COATING THICKNESS (MILS)
0.01
0.05
0.2
0.5
1
2
5
10
20
30
40
50
60
70
80
90
95
98
99
99.8
99.9
99.99
MICRO-DERM SCALE UNITS
605972A

Figure 12

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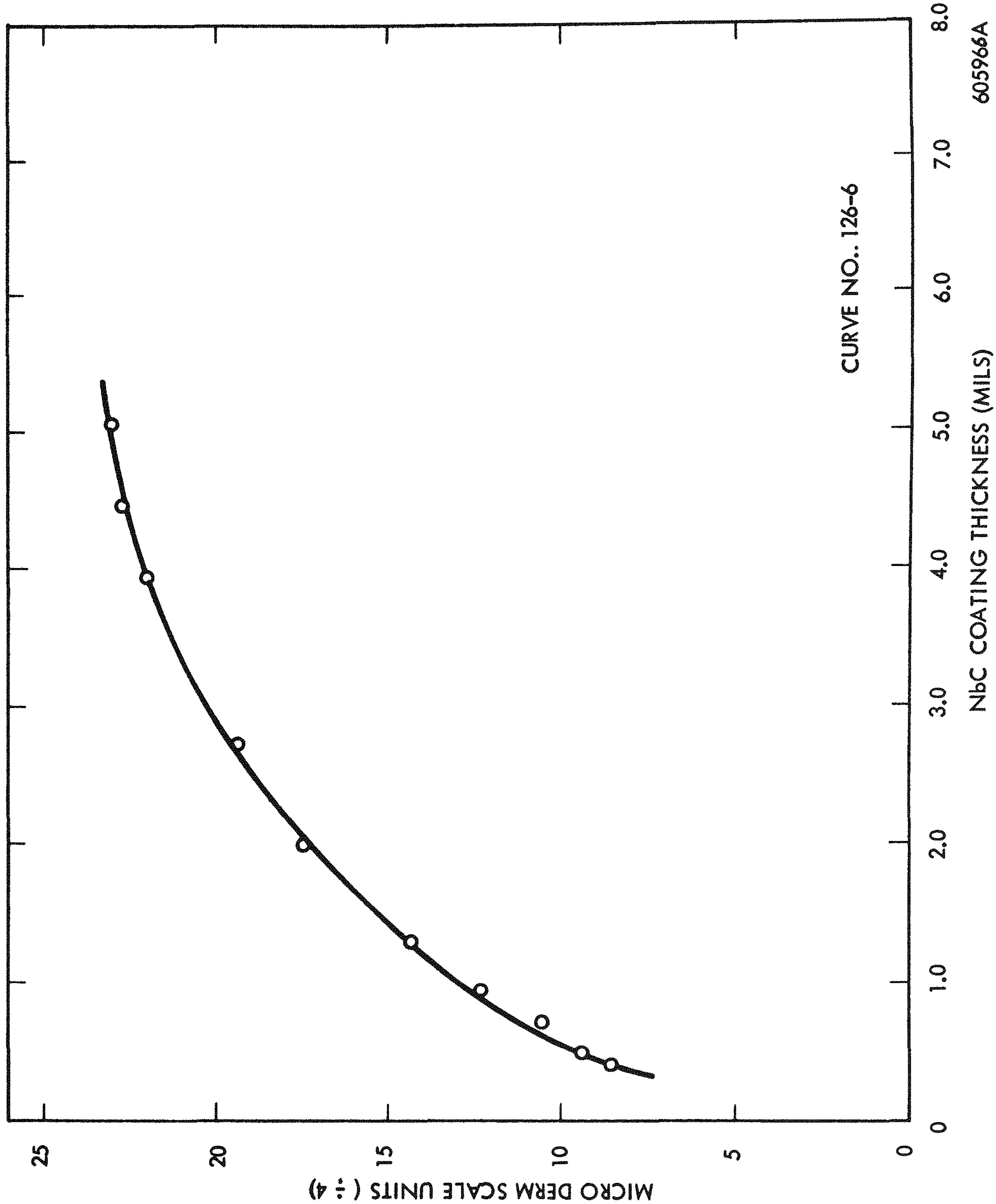

Figure 13

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RESTRICTED DATA
Atomic Energy Act - 1954

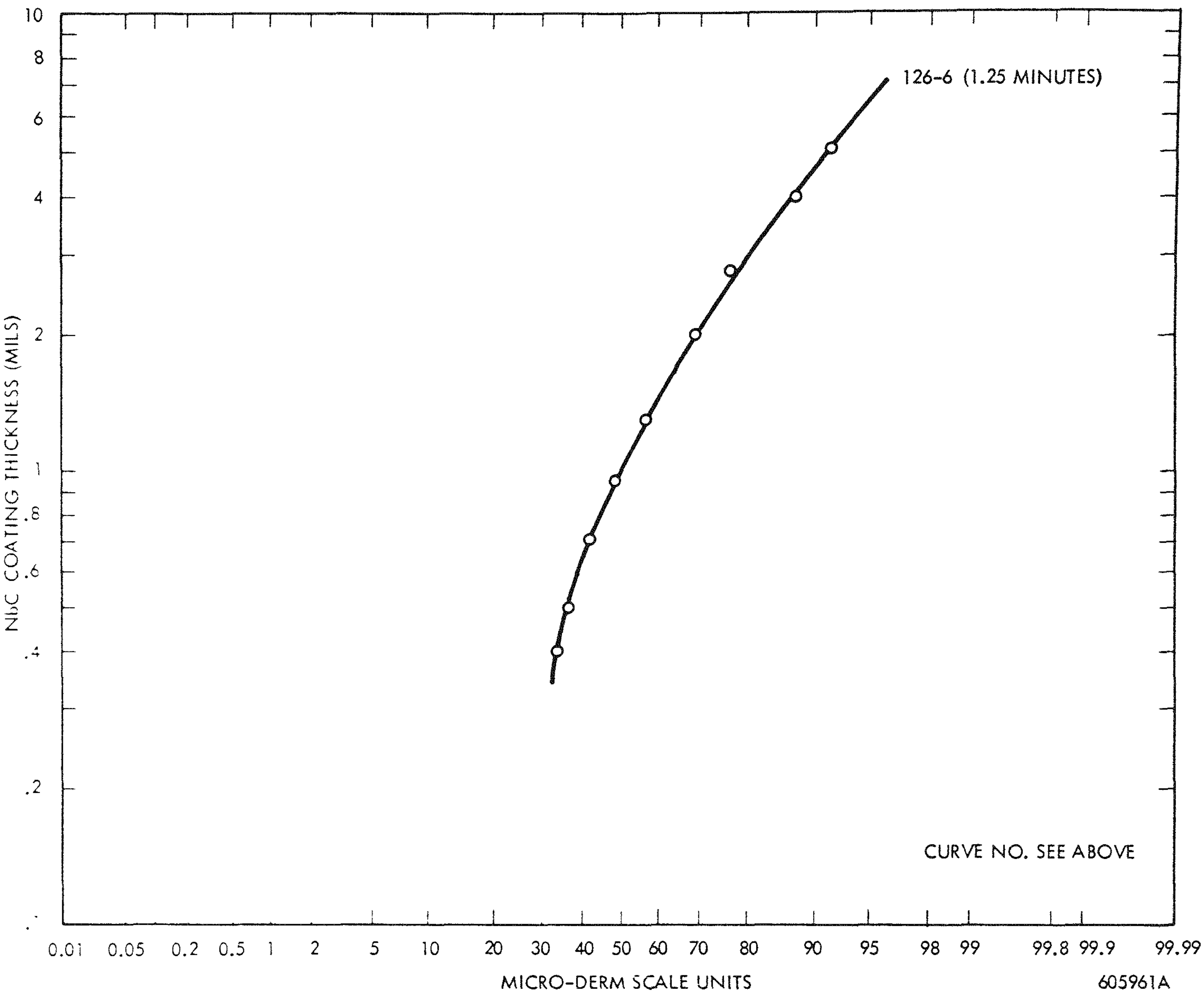

Figure 14

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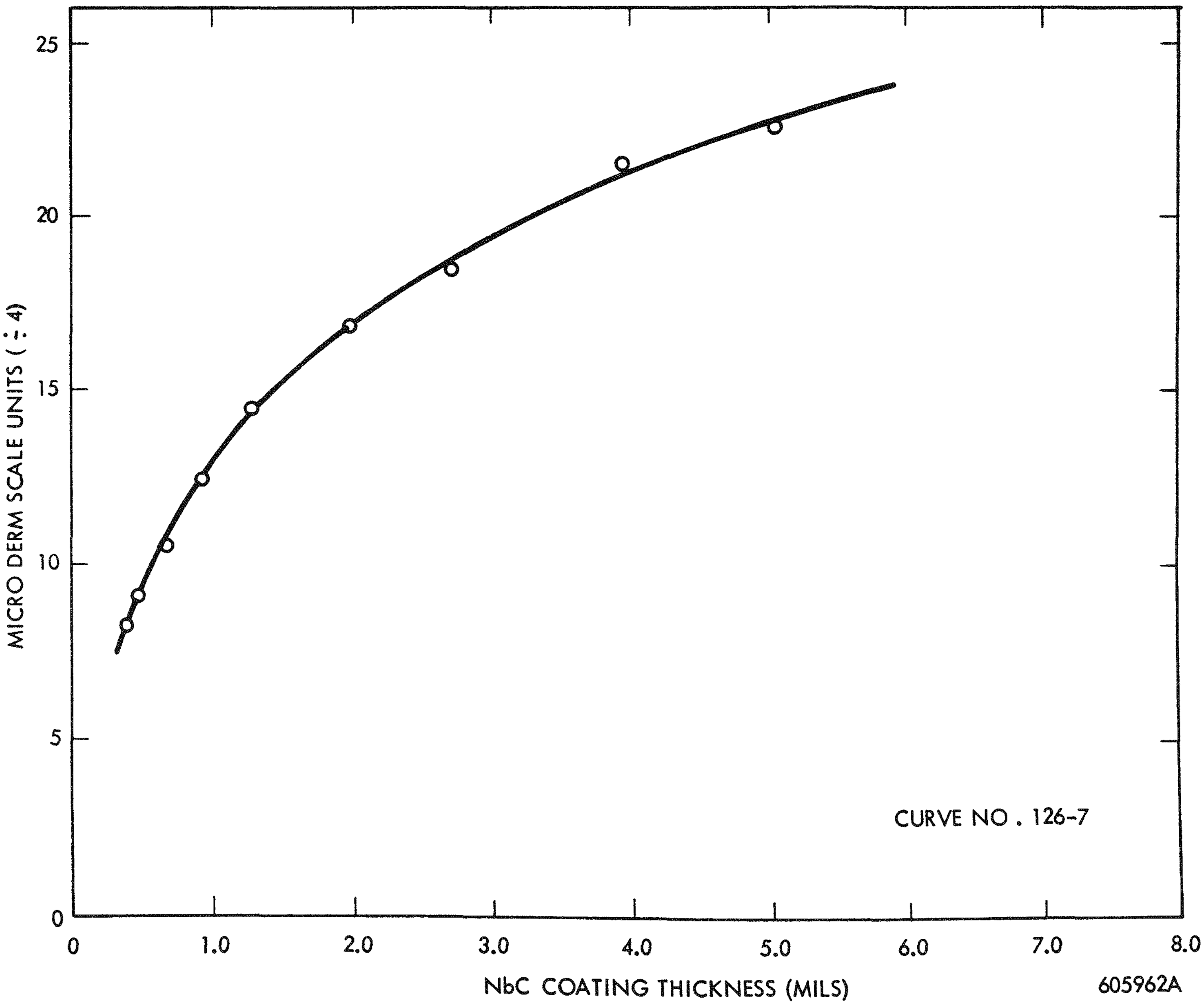

Figure 15

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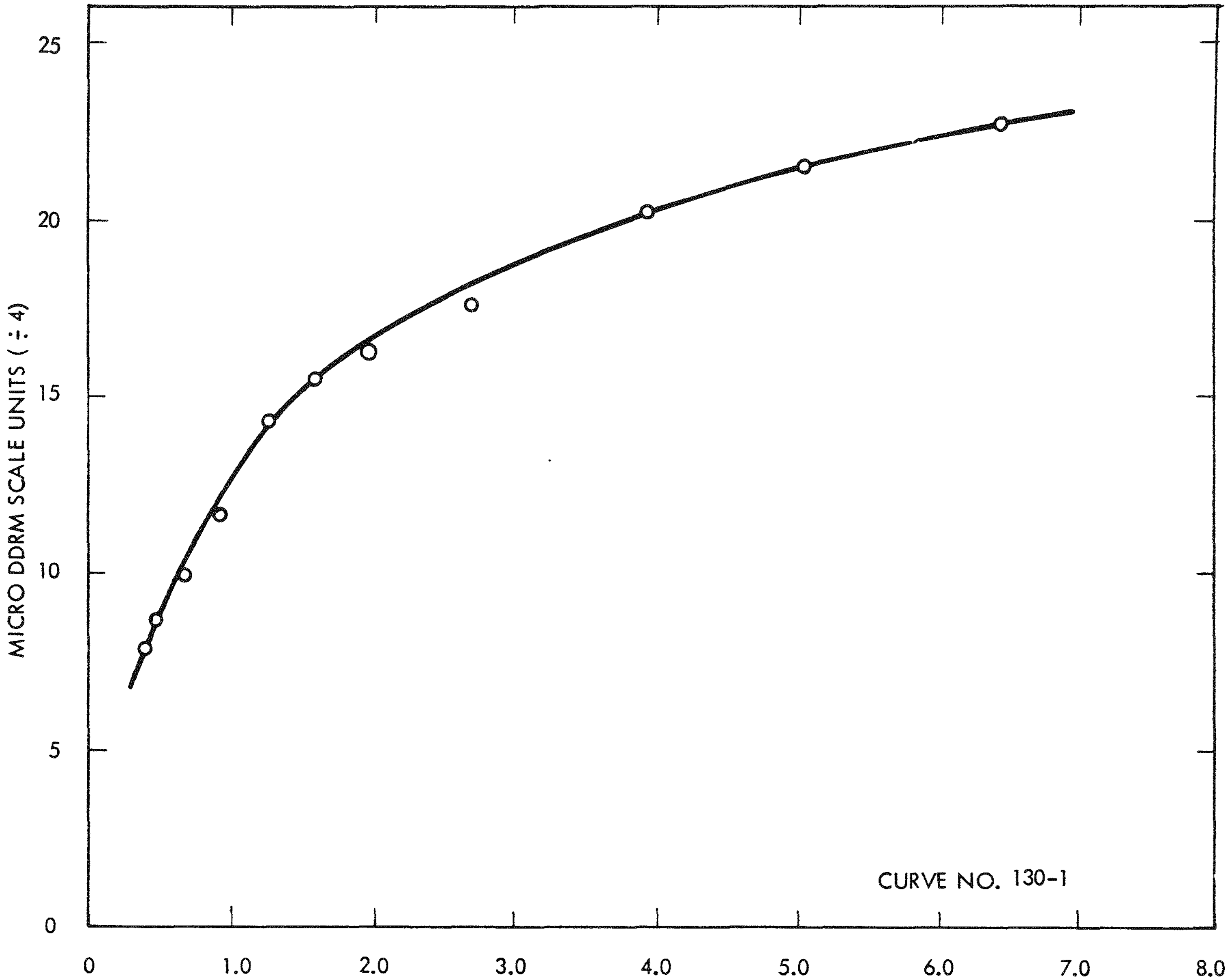

605965A

Figure 16

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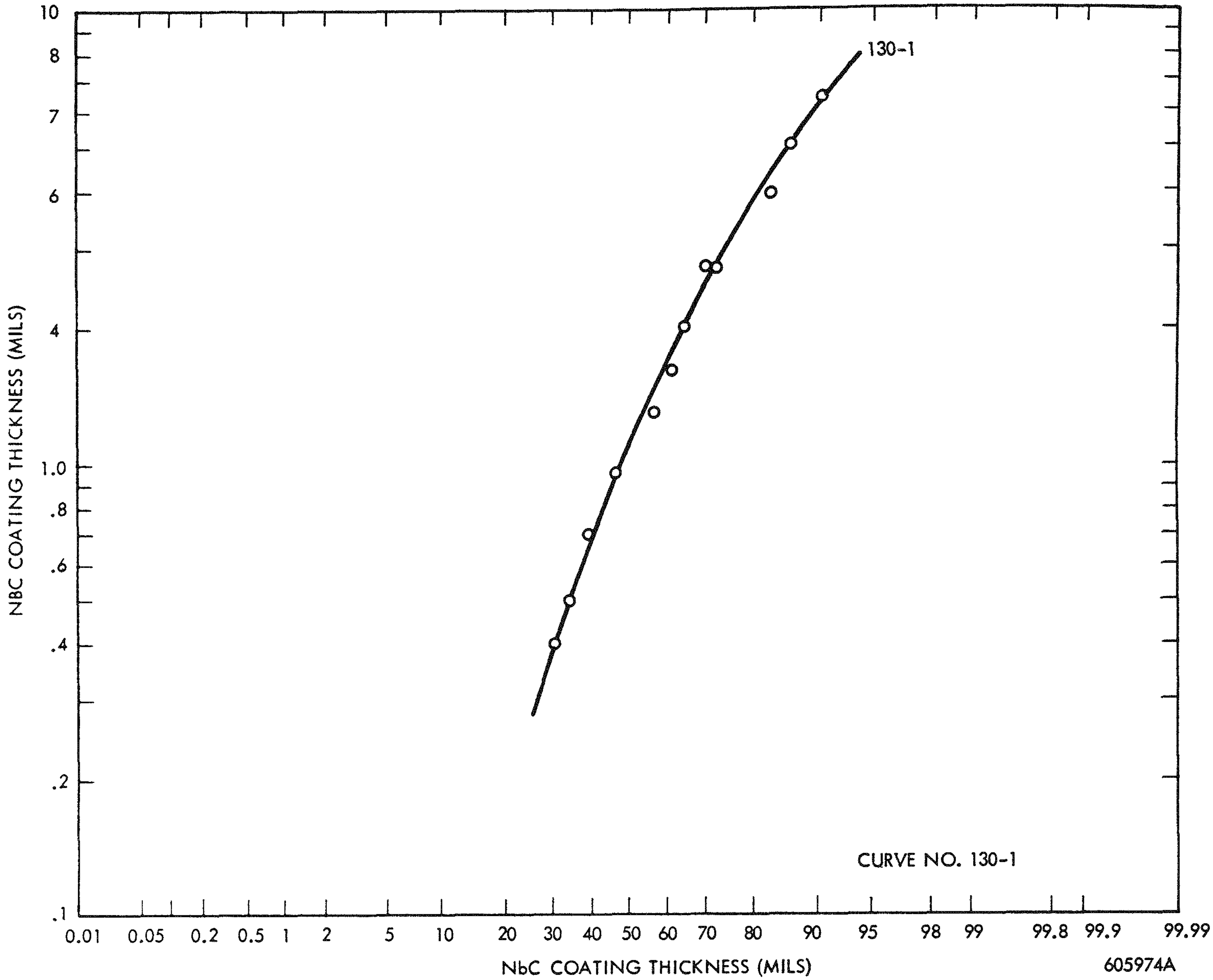

Figure 17

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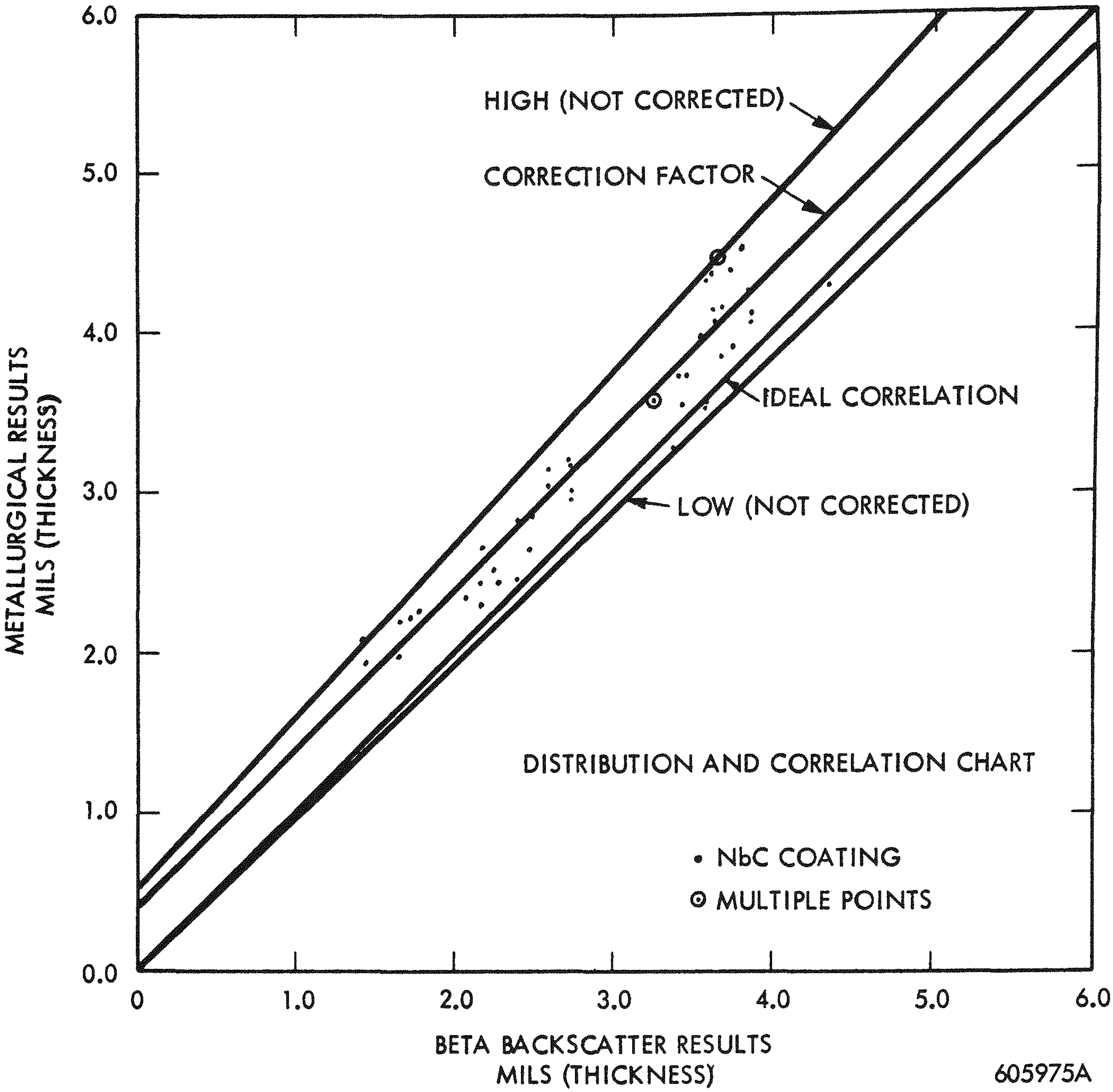
6.0
5.0
4.0
3.0
2.0
1.0
0.0
METALLURGICAL RESULTS
MILS (THICKNESS)
HIGH (NOT CORRECTED)
CORRECTION FACTOR
IDEAL CORRELATION
LOW (NOT CORRECTED)
DISTRIBUTION AND CORRELATION CHART
• NbC COATING
⊙ MULTIPLE POINTS
0
1.0
2.0
3.0
4.0
5.0
6.0
BETA BACKSCATTER RESULTS
MILS (THICKNESS)
605975A

Figure 18

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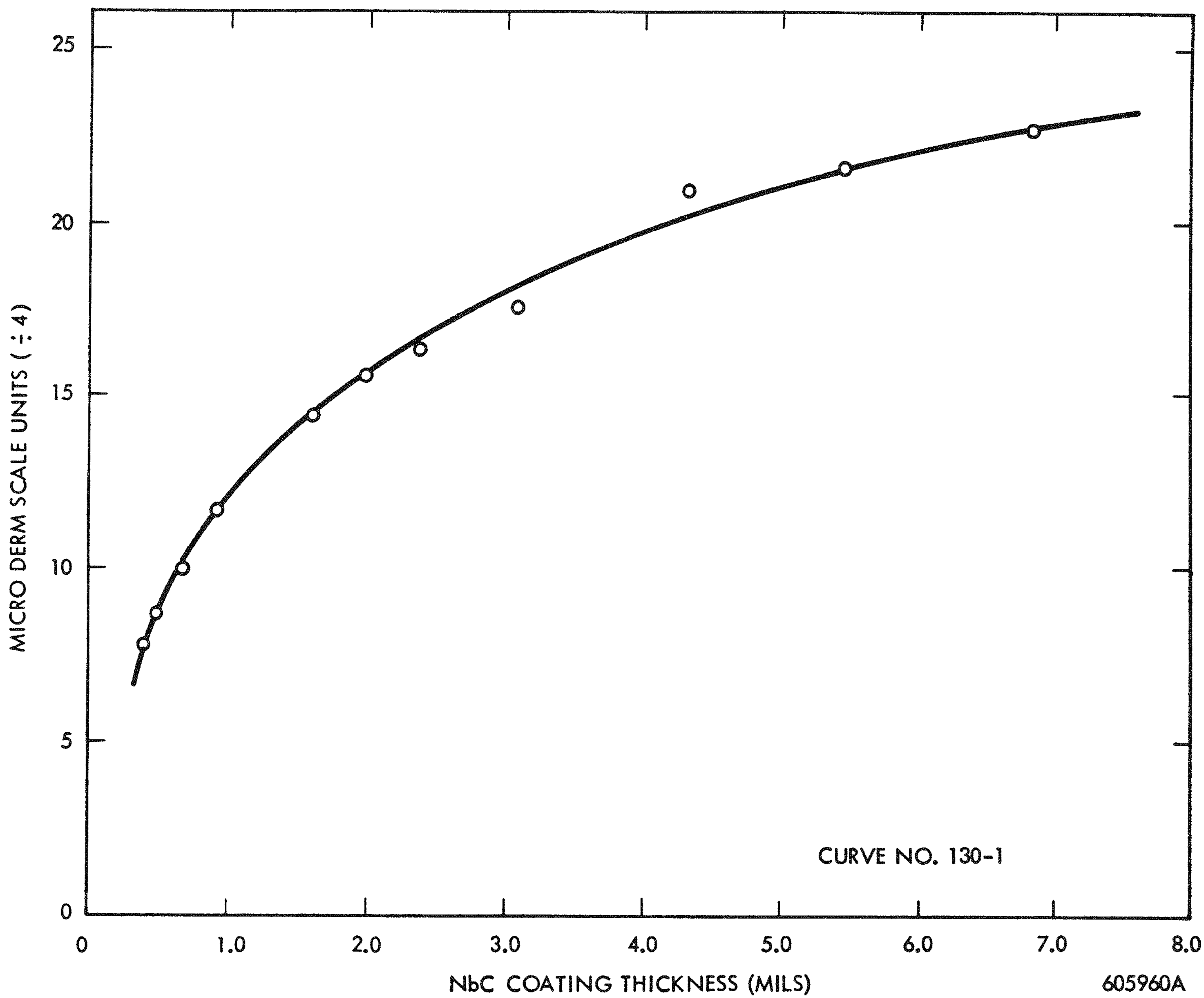

Figure 19

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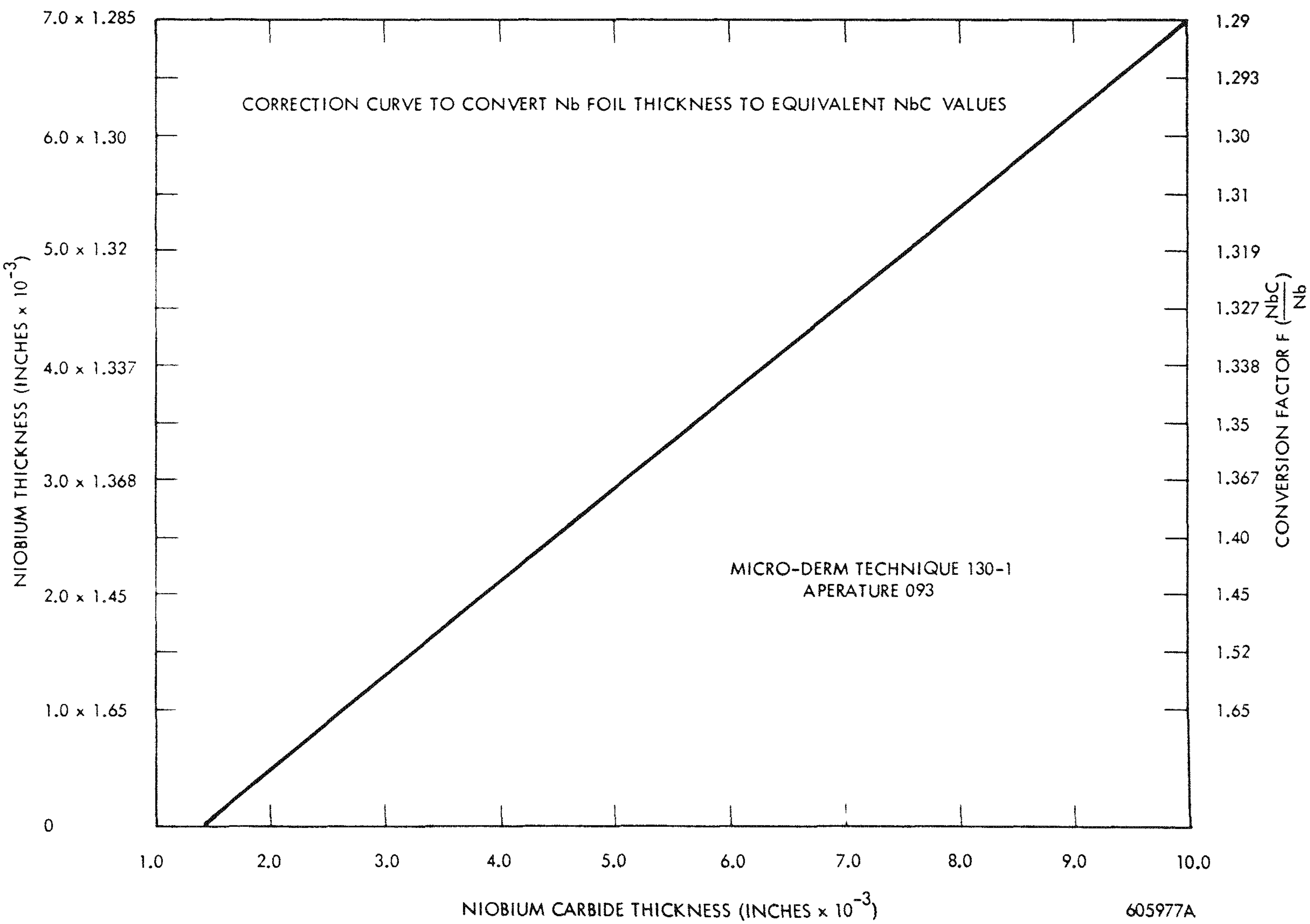

Figure 20

Astronuclear Laboratory

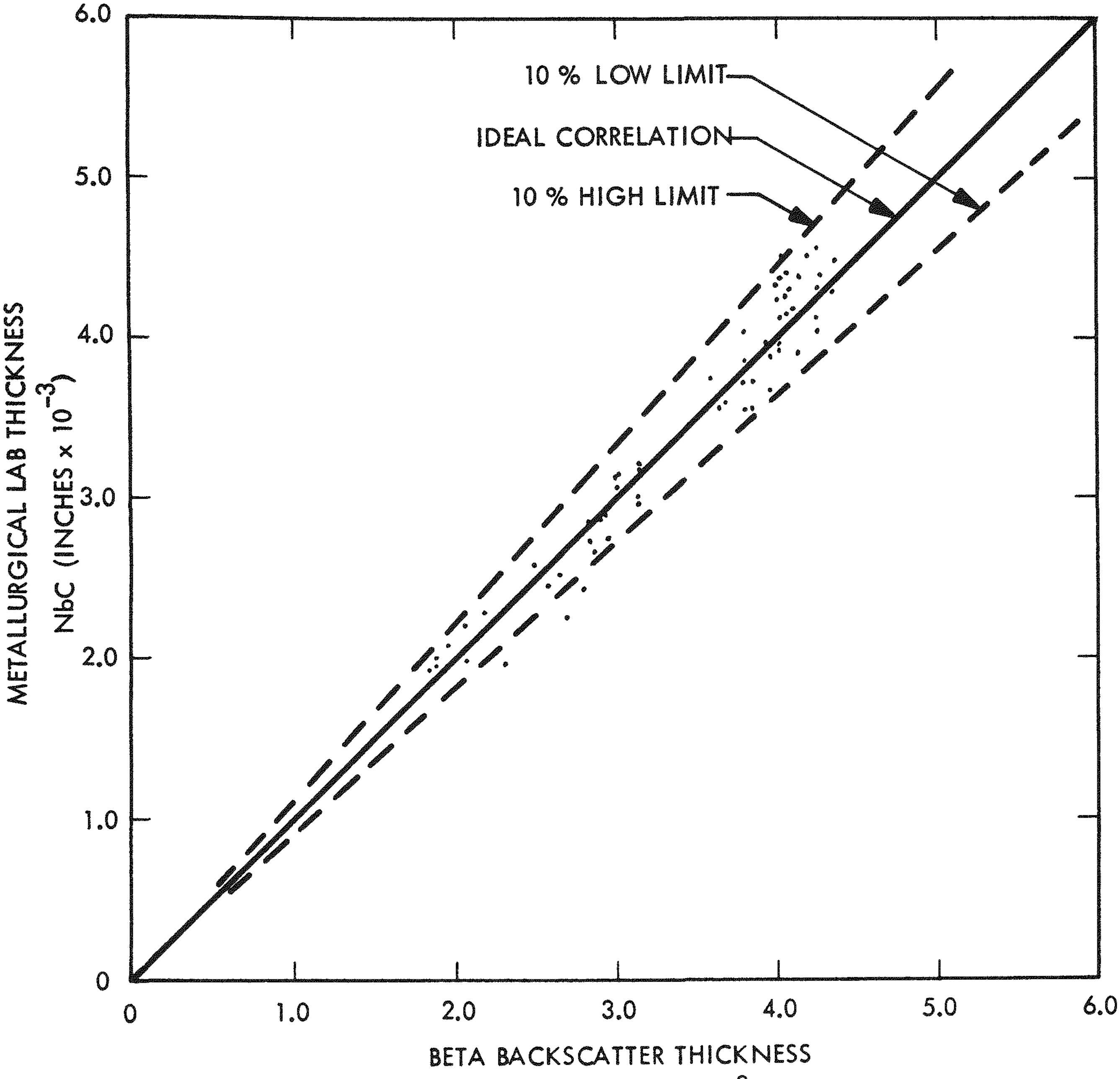

Figure 21

605973A

I. EQUIPMENT MODIFICATION

A. Purpose

The Micro-Derm basic electronic unit was modified to include a pre-amplifier in the detector circuit and outlet jacks for an external electronic counter.

The purpose for these modifications was to increase the reliability of the coating thickness determinations. With the low source strength of the Strontium-90 probe, the beta-backscatter count was slow in producing sufficient signal strength, which resulted in long scanning periods.

B. Pre-Amplification

The addition of a pre-amplifier circuit to strengthen the beta-backscatter count significantly changed the rate of count from the Geiger-Muller Tube and required a recalibration of the equipment. The intent of the addition of a pre-amplifier circuit was to give a greater number of counts per unit time and thus provide the possibility of shorter scan times and greater accuracy.

The relative increase in count with the pre-amplifier is not linear over the full range in coating thickness comparing the count as registered on the Micro-Derm S-1 linear scale. The scale deflection increased by 65% in the 1/2-mil thickness range and increased by 30% in the 4-mil range. With all the equipment variables (machine settings) the same, the addition of the pre-amplifier would permit a beta scan to be made in 25% less time. It was believed, however, that to decrease the time would decrease the total count with a sacrifice in accuracy. The recalibration of the equipment was, therefore, based on the original time of beta scan.

C. Electronic Digital Counter

The Micro-Derm S-1 linear scale which was originally used for indicating the beta-backscatter count during a time interval has 50 divisions, reading

0 to 100. From the original calibration curves for a thickness range between 2-4 mils, a scale division was equivalent to approximately 0.00022 inches of coating thickness. Since each of the 50 scale divisions was equivalent to 2 on the linear scale, a half-division was read by the operator for closer delineation of thickness. A typical 1/2 scale division was equivalent to 0.00011 inch of coating thickness.

The digital counter was added to the system to eliminate inaccuracies in interpreting one-half scale readings and thus give better reproducibility. It was desirable to have a digital counter that was adjustable to give direct coating thickness. Since none were available, a compromise was made; and a Beckman Model 7060U was used. This electronic counter consists of an electronic gate with controlling circuitry, a chain of six decimal-counting units, and three input-trigger channels. For use with the Micro-Derm, the function switch is placed at the C/B-A operation. A pulse from the input channel opens the gate, and pulses from a second channel are counted until a pulse from the third channel closes the gate.

Unfortunately, the Beckman Counter digital output cannot be regulated through the Micro-Derm controls, with the exception of the timing circuit. The Beckman Counter integrates the pulses from the Geiger-Muller Tube (beta-backscatter) in terms of digital units. These digital units, corresponding to calibration standard thickness, are then used to plot a calibration curve.

D. Voltage Regulation

Due to frequent and severe voltage fluctuations which affected beta-backscatter results, a Sola, Type CVS, constant voltage regulator was purchased for use with the Micro-Derm. The voltage regulation was excellent, and little or no drift was noted in the base material zero setting. The Micro-Derm and Beckman Electronic Counter were both tied into this regulator.

I. EQUIPMENT CALIBRATION

A. Calibration Standards

As noted in Section III, various thickness Nb foil was cemented to graphite and used as calibration standards. To be compatible with Metallography Lab reports on NbC thickness, the Nb foil thickness was multiplied by a factor proportionate to the foil thickness. This conversion was time-consuming, and it was decided to use only the NbC-coated graphite as calibration standards. Wherever it was possible, the new fine-grain coated graphite base was used for standards since the coating interface is more regular than that with the ATJ grade graphite.

B. Aperature Size

Two new aperatures were purchased specifically to test support block surfaces. To test the coating thickness on end surfaces between the channels, a flat slit aperature with slit dimensions of 0.030" width x 0.125" length was obtained. This provided an effective test area of 0.00375 square inches.

To test the support block coating thickness on the side of the hex near the nozzle end, a raised dome-type aperature was needed. For this purpose, an aperature was purchased with a slit opening of 0.030" width x 0.125" length. The dome height was 0.125". The slit was machined across the top of the dome (saw cut) leaving open ends. It was expected that the same calibration curve could be used as with the flat aperature since the same test area was used. Erratic results were achieved since the open ends of the slit (saw cut) permitted escape of beta-backscatter rays. A larger round dome (0.156" diameter x 0.125" dome height) aperature was substituted.

C. Calibration Curves

Two calibration curves were drawn using standards shown in Table B1. Additional points on the curves were plotted from the Micro-Derm readings of blocks that had been sectioned and reported by the Metallography Lab. The curves are shown in Figures B1 and B2. Direct reading charts were prepared from these curves giving the NbC coating thickness in mils corresponding to the Beckman Electronic Counter readout (see Tables B2 and B3).

TABLE B1
CALIBRATION STANDARDS

Techniques 524-1
520-1

Standard Identification	Location	Niobium Carbide (Mils)		Micro-Derm Counter Readout Technique Number	
		Met. Lab	Q. E. Recheck	520-1 (.156 D-4)	524-1 (.030)
1. PO-3	----	.0007	--	193 avg.	305 avg.
2. Foil	----	.00095	--	218 avg.	319 avg.
3. Ht X-1	No. 1	.00124	--	242 avg.	339 avg.
4. Foil	----	.00161	--	258 avg.	352 avg.
5. Ht X-2	No. 6	.00163	--	258 (alternate)	353 avg.
6.					
7. Ht X-1	No. 8	.00221	--	270 avg.	362 avg.
8. D-11	a	.00265	--	278 avg.	370.5 avg.
9. D-11	a(small)	.00292	--	283 avg.	372.5 avg.
10. T-4	b	.00315	--	287 avg.	375.5 avg.
11. Ht19918	6	.00354	--	295 avg.	382.0 avg.
12. Ht19918	21	(.00425)	.00384	299 avg.	385 avg.
13. Ht34	No. 22	.00425	--	305 avg.	391 avg.
14. T-4	c	.00506	--	314 avg.	398.5 avg.
15. T-1	c	.00575	--	318 avg.	405 avg.

TABLE B2
MICRO-DERM DIRECT READING CHART (BLOCK END FACES)

Date: May 25, 1965 | Aperature - .030-4-0 | Technique No. 524-1

Beckman Counter	NbC (Mils)	Beckman Counter	NbC (Mils)	Beckman Counter	NbC (Mils)
323	1.00	367	2.55	389.5	4.15
327	1.05	368	2.60	390	4.20
330	1.10	368.5	2.65	391	4.25
333	1.15	369	2.70	391.5	4.30
336	1.20	370	2.75	392	4.35
339	1.25	371	2.80	392.5	4.40
341.5	1.30	371.5	2.85	393	4.45
344	1.35	372	2.90	393.5	4.50
346	1.40	373	2.95	393.75	4.55
348	1.45	373.5	3.00	394.25	4.60
349	1.50	374.5	3.05	394.75	4.65
351	1.55	375	3.10	395	4.70
352	1.60	375.5	3.15	395.5	4.75
353	1.65	376.5	3.20	396	4.80
353.5	1.70	377.5	3.25	396.5	4.85
354	1.75	378	3.30	397	4.90
355	1.80	379	3.35	397.5	4.95
356	1.85	379.5	3.40	398	5.00
357	1.90	380	3.45	398.5	5.05
357.5	1.95	381	3.50	399	5.10
358	2.00	382	3.55	399.5	5.15
359	2.05	382.5	3.60	400	5.20
360	2.10	383	3.65	400.5	5.25
361	2.15	383.5	3.70	401	5.30
361.5	2.20	384.5	3.75	401.5	5.35
362.5	2.25	385	3.80	402	5.40
363	2.30	385.5	3.85	402.5	5.45
364	2.35	386.5	3.90	403	5.50
364.5	2.40	387	3.95	403.5	5.55
365.5	2.45	387.5	4.00	404	5.60
366.5	2.50	388.5	4.05	404.25	5.65
		389	4.10	404.75	5.70
				405	5.75

ACCEPTANCE LIMITS (Block)
Dome End - 2 Mil Minimum
Nozzle End - 1-1/2 Mil Minimum
Anywhere - 5 Mil Maximum

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WANL-TME-1189

TABLE B3
MICRO-DERM DIRECT READING CHART (BLOCK SIDES)

Date: May 24, 1965		Aperature - . 156-D-4		Technique No. 520-1	
Beckman Counter	NbC (Mils)	Beckman Counter	NbC (Mils)	Beckman Counter	NbC (Mils)
225	1.00	276	2.55	303.5	4.15
229	1.05	277	2.60	304	4.20
233	1.10	277.5	2.65	304.5	4.25
237	1.15	278.5	2.70	305	4.30
240	1.20	279.5	2.75	305.5	4.35
243	1.25	280.5	2.80	306	4.40
246	1.30	281.5	2.85	306.5	4.45
249	1.35	282.5	2.90	307	4.50
251	1.40	283	2.95	307.5	4.55
253	1.45	284	3.00	308	4.60
255	1.50	285	3.05	308.5	4.65
256	1.55	286	3.10	309	4.70
257	1.60	287	3.15	309.5	4.75
259	1.65	288	3.20	310	4.80
260	1.70	289	3.25	310.5	4.85
261	1.75	289.5	3.30	311	4.90
262	1.80	290.5	3.35	311.5	4.95
263	1.85	291.5	3.40	312	5.00
263.5	1.90	292.5	3.45	312.25	5.05
264.5	1.95	293	3.50	312.5	5.10
265.5	2.00	294	3.55	313	5.15
266.5	2.05	295	3.60	313.5	5.20
267.5	2.10	296	3.65	314	5.25
268.5	2.15	296.5	3.70	314.5	5.30
269	2.20	297.5	3.75	315	5.35
270	2.25	298	3.80	315.5	5.40
271	2.30	299	3.85	316	5.45
272	2.35	299.5	3.90	316.5	5.50
273	2.40	300.5	3.95	317	5.55
274	2.45	301	4.00	317.5	5.60
275	2.50	301.5	4.05	318	5.65
		302.5	4.10	318.5	5.70
				319	5.75

ACCEPTANCE LIMITS (Block)
Dome End - 2 Mil Minimum
Nozzle End - 1-1/2 Mil Minimum
Anywhere - 5 Mil Maximum

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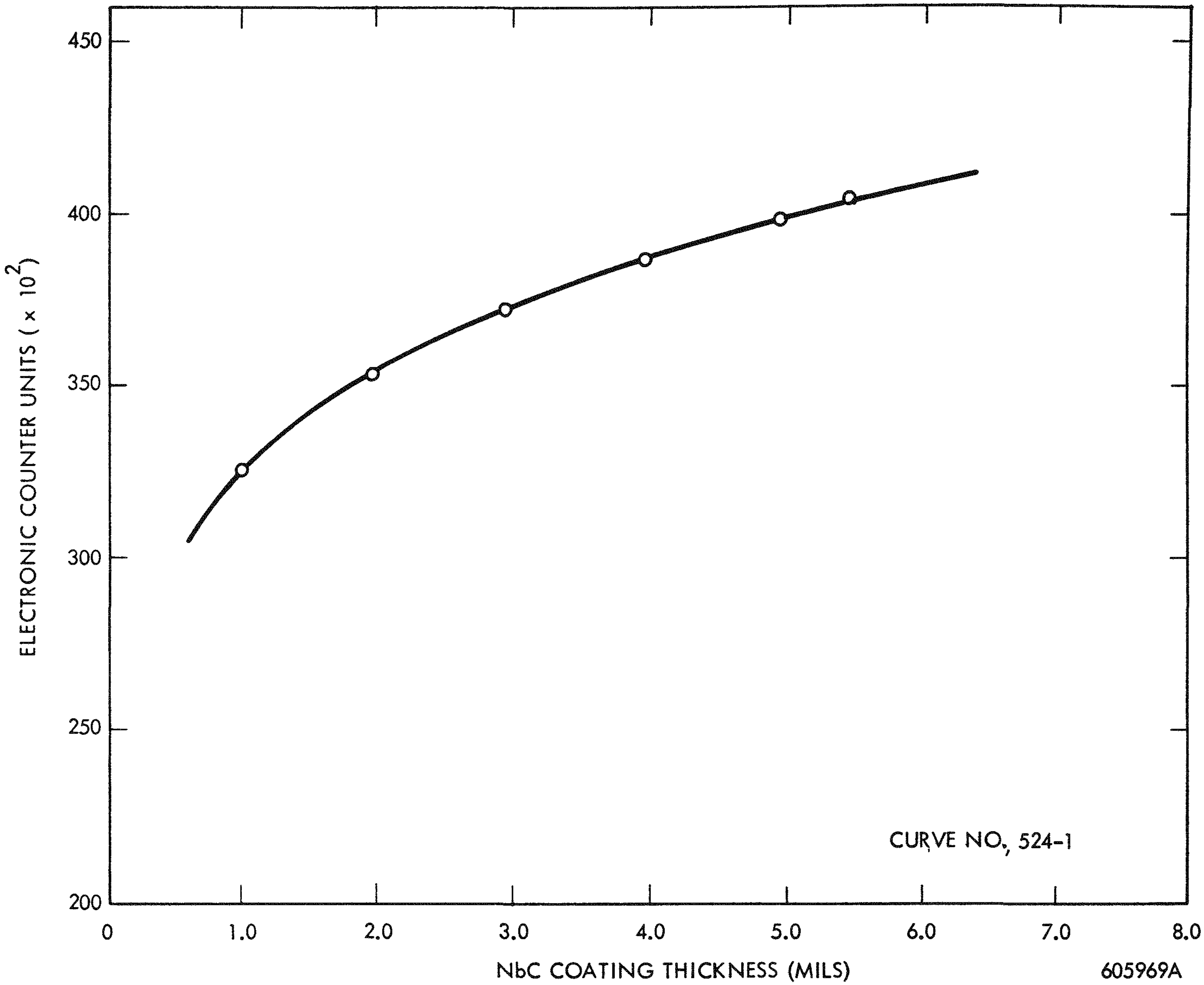

Figure B-1

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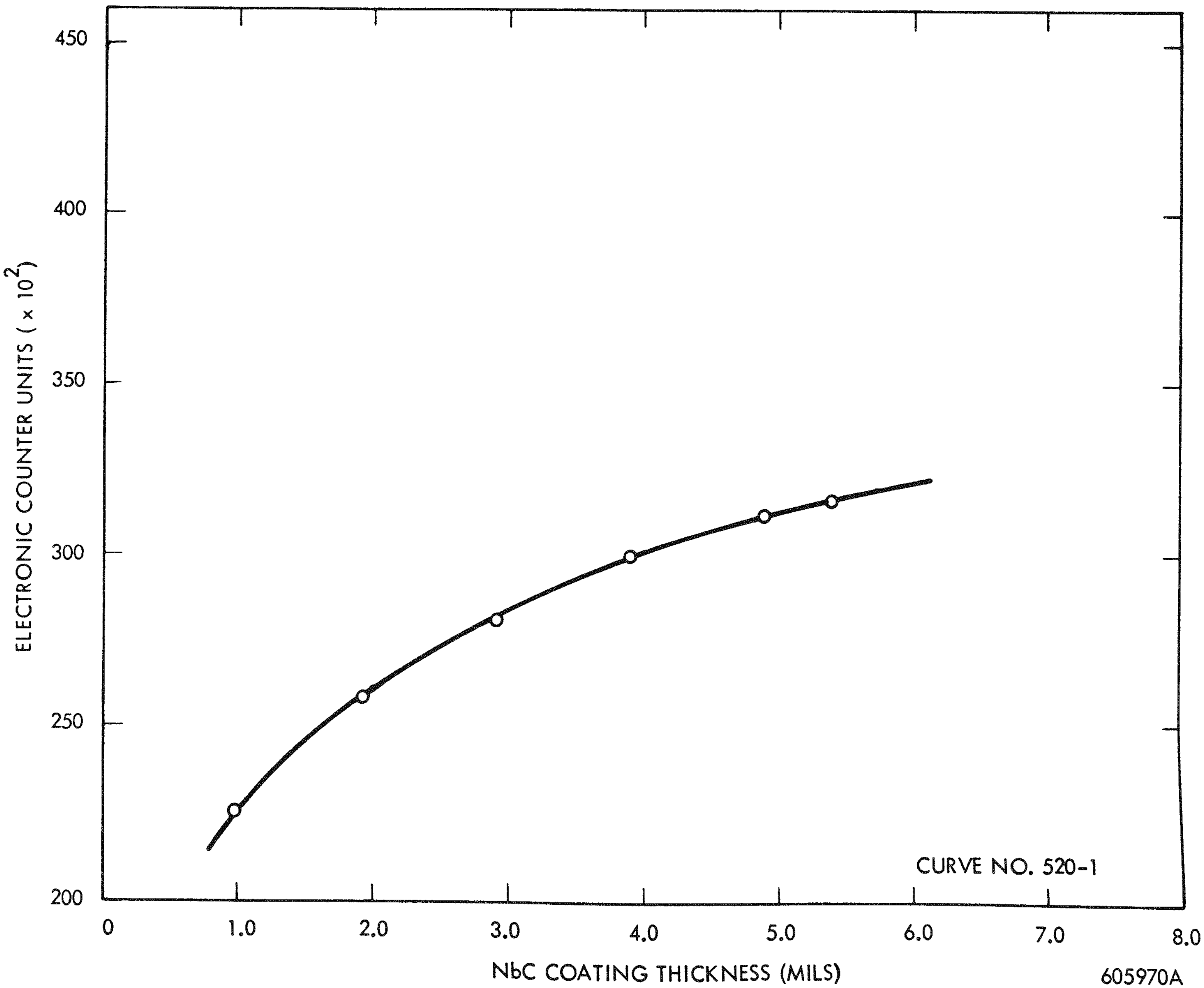

Figure B-2

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I. COATING THICKNESS DETERMINATION

A. Technique

The techniques used in producing the calibration curves (Figures B1 and B2) are shown in Table C1. With the addition of the electronic counter, the various Micro-Derm basic unit settings are unrelated to the electronic counter readout, with the exception of the time control. The check point dial, zero set dial, rate switch, and the reverse switch are now currently used to provide a zero setting for the base material. The measure switch and the timer are the only controls that are integrated with the Beckman Counter.

B. Procedure

The following procedure is followed in making a thickness determination using the modified Micro-Derm equipment:

1. Warm up equipment for 10 minutes.
2. Establish zero linear scale reading for graphite base material.
3. Place standard on aperature plate.
4. Turn display knob on counter to zero.
5. Turn Micro-Derm switch to measure.
6. Turn display knob on counter to infinity.
7. Compare countout with calibration curve or direct reading chart.
8. If average readout does not compare with the curve within 0.1-0.2 mils, adjust timer and repeat steps 2. through 8.
9. If standards agree, place unknowns over aperature and repeat above procedures using the direct reading charts to report results.

TABLE C1

MICRO-DERM CALIBRATION TECHNIQUE

	1 Technique No.	2 Date	3 Source Type	4 Source No.	5 Aperature Dia. X H	6 Source Pos.	7 D_{sw}	8 Tube Pos.	9 Chkpt Dial	10 C-Set Dial	11 Rate Pos.	12 To Min.	0 Up Pos.
1	520-1	5-20-65	Sr^{90}	880	.156-D-4	19	3	3	517	929	3	1.0	18.0
2	524-1	5-24-65	Sr^{90}	880	.030-4-0	23	3	3	517	884	3	1.5	40.0
3													
4													
5													
6													
7													
8													
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A Quality Engineering Procedure, No. 107A, was prepared to provide support block inspection criteria. This procedure further requires that a block representative of the top and bottom of a coating furnace run be tested. Should either of these two blocks fail to meet the thickness requirements, all blocks in the furnace run are to be beta-backscatter tested.

C. Results

During the period that the modified equipment was being investigated, the reproducibility of the thickness results from day-to-day was excellent. Over a period of one week, the zero point needed only minor adjustment. The standards over a full range of 0.9-6.0 mils did not vary more than 5% as average values.

A number of support blocks rejected for exceeding the low and high thickness limits were beta-backscatter tested. Where direct comparisons could be made with Metallography Lab results, the correlation was good. This numerical correlation is shown in Table C1. A correlation chart (Figure C1) shows the deviation of the Micro-Derm results from the reported Metallography Lab results.

TABLE C2 (Page 1 of 2)
NUMERICAL CORRELATION OF METALLOGRAPHY LAB AND MICRO-DERM NbC THICKNESS RESULTS

S/N (Ht)	Run Number	Type	QCOI 36		Metallographic		Micro-Derm		Deviation		Micro-Derm		
			Fig.	No.	Original	Recheck	Single	Average	Mils	Percent	Technique	Curve	Ap. Size
1223	B-252	Reg	4	5	3.72			3.35	-.37		524-1	524-1	.030-4-0
				6	3.09			3.25	+.16				
				7	3.03			3.09	+.06				
				20	3.38			3.49	+.11				
				21	3.49			3.53	+.04				
				22	3.38			3.26	-.12				
				1	3.11			3.20	+.09				
				2	2.90			3.08	+.18				
				3	2.83			2.90	+.07				
				4	3.09			2.75	-.24				
				23		1.58		1.35	-.33				
				24		1.73		1.35	-.38				
				25		1.64		1.35	-.29				
				26		1.62		1.32	-.30				
495	B-163	Reg	4	25	1.49	1.65		1.70	+.05				
A19876	B-237	J5	4	6	4.02		4.12		+.10				
				7	3.29	3.54	3.35		-.19				
				8	2.44	3.02	3.00		-.02				
				24	4.12		3.72		-.40				
				25	3.96	4.28	4.05		-.23				
				26	4.16		4.05		-.11				
				2	3.54	3.96	3.85		-.11				
				3	3.84	3.89	3.90		+.01				
				4	3.57	3.54	3.70		+.16				
				38	1.95		2.10		+.15				
				39	2.88		2.50		-.38				
A19918	B-237	J7	4	6	3.54	3.54	3.50		-.04				
				7	2.65	2.67	2.52		-.15				
				8	2.44	2.40	2.31		-.09				
				21	4.25	3.72	4.02		+.30				
				22	4.14	4.03	3.96		-.07				
				23	3.43	----	3.50		+.07				
				2	3.72	4.25			+.53				
				3	4.46	4.07	4.40		+.33				
				4	4.33	4.39	4.35		-.04				
				25	1.56	----	1.66		+.10				
				26	1.98	----	1.94		-.04				

S/N (Ht)	Run Number	Type	QCOI 36		Metallographic		Micro-Derm		Deviation		Micro-Derm		
			Fig.	No.	Original	Recheck	Single	Average	Mils	Percent	Technique	Curve	Ap. Size
42	A55	J6	4	6	3.11	3.28	3.03		-.25		524-1	524-1	.030-4-0
				7	2.33	2.62	2.62		0				
				8	1.66	2.02	1.77		-.25				
				1	3.43	3.54	3.45		-.09				
				2	3.54	3.54	3.50		-.04				
				3	3.54	3.50	3.50		0				
53	A-60	J4	4	1	1.66			1.54	-.12				
				2	1.35			1.26	-.09				
				4	3.54			3.40	+.16				
A19855	B-237	J4	4	6	4.33	4.32		4.25	-.07				
				7	4.51	3.58		3.65	+.07				
				8	3.54	3.58		3.30	-.28				
				24	4.25	4.25		4.35	+.10				
				25	4.51	4.52		4.35	-.17				
				2	4.42	4.42		4.35	-.07				
				3	4.44	4.46		4.40	-.06				
				4	4.16	4.13		4.35	+.22				
110	B-237	J9	4	7	4.28	4.68		4.70	+.02				
				8	3.77	3.80		3.80	0				
				2	4.67	4.48		4.50	+.02				
				3	4.33	4.03		4.20	+.17				
				4	4.21	4.37		4.35	-.02				
29	A-61	J5	4	6	1.77			1.55	-.22				
				7	3.54			3.05	-.49				
				8	3.78			3.50	-.28				
				24	1.77			1.80	+.03				
				25	3.66			3.45	-.21				

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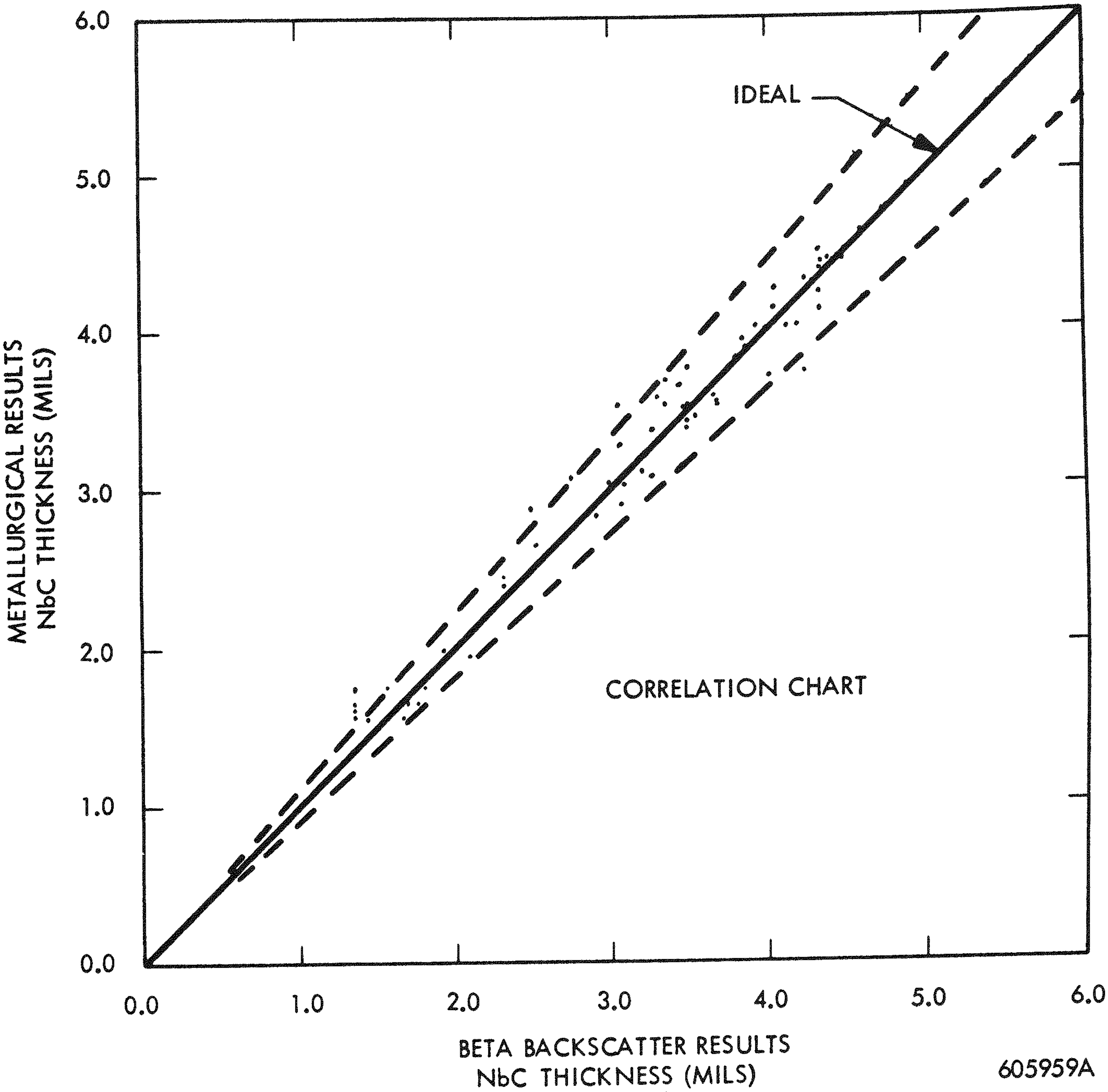
IDEAL
CORRELATION CHART
METALLURGICAL RESULTS
NbC THICKNESS (MILS)
BETA BACKSCATTER RESULTS
NbC THICKNESS (MILS)
0.0
1.0
2.0
3.0
4.0
5.0
6.0
605959A

Figure C-1

CONCLUSIONS

The following conclusions may be drawn following the experimental work covered by this report:

1. The modified Micro-Derm equipment may be used to determine the NbC coating thickness of the external surfaces of support blocks within the thickness range of 1.5-5.0 mils.

2. The Micro-Derm beta-backscatter results will agree with metallurgical optical determinations within 10% of any thickness in the range of 1.5-5.0 mils.

3. Further development work should be scheduled in acquiring instrumentation for increasing or decreasing the count on the Beckman Counter for calibration purposes.

4. In the event of failure in the Beckman Electronic Counter, the Micro-Derm linear scale may be used in substitution without loss of accuracy in coating thickness determinations.

5. Efforts should be continued to develop a beta-backscatter test system utilizing a higher source strength to reduce the testing time.

6. The test plan provided by QIP(L) No. 107, Revision 1, will provide a more adequate coverage of acceptance of support blocks than the present plan of testing one block per furnace run by metallographic methods.

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