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FUELS AND LUBRICANTS RESEARCH DIVISION

24 August 1988
File: 17-7958-842

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Naval Air Propulsion Center
Attn: Mr. R.A. Kamin, PE-33
1440 Parkway Avenue
Trenton, New Jersey 08628

Subject: **NTIAC Contract DLA 900-84-C-091 to Perform JFTOT Tube Deposit Thickness Measurements**

Dear Sir:

This letter reports the results of SwRI comparison of techniques for measuring the thickness of deposits formed on the surface of heated metal tubes during JFTOT testing of fuels.

The techniques evaluated in this program included optical birefringence, dielectric strength, and ion milling. A fourth method involving cross sectioning and direct measurement of deposit thickness at high magnification using electron microscopy would serve as referee if wide disagreement among the three primary techniques was encountered.

Considering the inherent difficulties in measuring deposits of less than 1 micrometer by any indirect approach, all three methods showed surprisingly good agreement when the data were plotted for comparison. Fortunately, the results of these tests exhibited close agreement. Difficulties during specimen preparation of the direct cross sections as well as resolution problems encountered during electron microscope examination resulted in images that were too marginal to be of value for this program.

Image enhancement equipment has recently been installed on the scanning electron microscope at SwRI. Through techniques involving image digitization, digital averaging of multiple image frames, and digital noise filtering algorithms, image quality can be significantly improved when poor signal-to-noise ratio is encountered. Although this equipment arrived too late to be of use in this program, it should greatly improve SwRI's ability to consistently perform cross-sectional measurements in future studies involving this type of deposit. The tube specimens for this program are being retained for evaluation with this improved equipment if funding can be identified.

The excellent overall agreement among the measurement techniques is indicative that each of the techniques is worthy of further study. Since these data represent tubes from only three JFTOT tests, it is difficult to attach any definitive statistical significance. The results, however, are encouraging, at least for this small group. Some interesting trends were noted in these data sets that should be observed in any future tests to determine if they are meaningful developing patterns or random aberrations within this rather small data set.



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Mr. R.A. Kamin
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Figure 1 is a plot comparing thickness results as determined by Auger ion milling and by dielectric strength as measured with the Deposit Measuring Device (DMD). The data points in Figure 1 represent only the data points for which birefringence data also exist. Figure 2 is a plot of Auger and DMD thickness data, but this plot contains all data points that were tested by these methods. Figure 3 plots the birefringence results compared to DMD results, while Figure 4 compares birefringence with Auger ion milling.

The DMD has shown in previous testing to be very useful in determining maximum deposit thickness and total volume of deposit. As seen in these plots, the DMD shows rather poor resolution with very thin deposits. This limited resolution has not proven to be a major problem. These very thin deposit zones represent so little of the total deposit volume on the tube that, in most cases, their contribution to the total deposit volume is too small to cause any major error in the calculation of total deposit volume. For most of the deposits encountered, the maximum thickness zone is sufficiently thick to be resolved by DMD. Direct comparison of DMD data to the other techniques presents a unique problem. The DMD tests a very small point at each location. Thus, small variations in texture of the tube or small localized variations in the deposit can affect the reading at any one location. Since many locations are tested to determine the total volume, small variations tend to be averaged out when all the locations are integrated to determine volume. The other techniques, however, examine a small spot at each location, rather than a point. While this spot is still small, it is many times larger than the point that is tested by DMD. Thus, this integration of thickness over a larger area tends to smooth the response at each location tested. Thus, DMD usually appears to have more scatter when individual locations are compared to the other techniques. For this type of direct comparison, it might be interesting to curve-fit the DMD data down the length of the tube for each quadrant, then compare the other techniques to the fitted data.

Figure 4 illustrates the ability of both birefringence and Auger ion milling to resolve very thin deposits, as shown by the very nice zero intercept of the plot. Note that tube 136 data represented by the "X" marks seem to be producing a different slope than the other two tubes that are represented by the "." marks. While all the data in Figure 4 show very good correlation, the fact that the data from tube 136 seem to be following a different slope could indicate that its deposit ion-milled at a different rate, which seems unlikely. It could also indicate that either the deposit on tube 136 or the test fuel that was present when the birefringence tests were made had a slightly different refractive index than was present with the other two tests.

As noted in the progress report dated 4 April 1988, the method used to index the tube quadrant locations was different at Naval Air Propulsion Center (NAPC) as compared to SwRI, since clockwise rotational indexing was used in one case and counterclockwise rotational indexing was used in the other. This difference in indexing caused the two data sets to agree on 0- and 180-degree positioning, but the locations of 90- and 270-degree positions were reversed. To avoid any confusion, all data and plots included in this current report have been adjusted to agree with the NAPC indexing approach.



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All data output from the DMD testing is in voltage units required to cause dielectric breakdown of the deposit. Previous work has shown that the dielectric strength of typical JFTOT deposits when tested by the DMD is 350 volts per micrometer of deposit thickness. All thickness calculations from DMD data were done using this ratio.

The data output from the Auger ion milling are in units of time. To establish calibration and to establish a baseline reference, a piece of tantalum foil with a known thickness of tantalum oxide on its surface was ion-milled until the oxide was removed. This procedure allowed calculation of milling rate for a tantalum oxide film under the test conditions that had been established. The JFTOT deposit locations were then milled, and milling times were recorded. The tantalum foil was milled at a new location after each JFTOT location was tested to recheck calibration and ensure that operating conditions had not changed. The JFTOT deposit thickness information collected by Auger ion milling thus represents thickness expressed as units representing tantalum oxide milling rates, since no standard exists to calibrate directly with the deposit. It was expected that the deposit would mill at a somewhat faster actual rate since it is primarily carbon and hydrogen. These elements are lighter than the oxygen and tantalum of the standard. Therefore, if the deposit did mill faster than the standard, then the indicated deposit thickness would be somewhat less than the actual thickness. Note in Figure 1 that in the thicker deposit zones where DMD has previously exhibited good resolution, the ion-milling values indicate less thickness. Note also in Figure 4 that for tubes 137 and 138, the ion-milling values are similarly low when compared to the birefringence values. These data sets indicate that the ion-milling thickness data need to be multiplied by a factor of approximately 1.4 to correct for faster milling of the deposit as compared to the standard. The data set from tube 136, however, indicates, in Figure 4, that no milling rate correction is needed since it has nearly a one-to-one slope.

Please write or call the undersigned at (512) 522-2594 if you have any questions regarding this project.

Very truly yours,



James G. Barbee
Engineering Technologist

JGB/sjd
(JGB1.B)

cf: Southwest Research Institute, Mr. L.L. Stavinoha
Nondestructive Testing Information Analysis Center, Mr. Frank Iddings

SECURITY CLASSIFICATION OF THIS PAGE

REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

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			Measurement		
			Thickness		
			Optical Birefringence		
			Dielectric Strength		
			Ion Milling		
			Nondestructive Testing		
19. ABSTRACT (Continue on reverse if necessary and identify by block number) The results of comparison of techniques for measuring the thickness of deposits formed on the surface of heated metal tubes during JFTOT testing of fuels is reported. The techniques evaluated included optical birefringence, dielectric strength, and ion milling.					
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22a. NAME OF RESPONSIBLE INDIVIDUAL R.A. Kamin			22b. TELEPHONE (Include Area Code)		22c. OFFICE SYMBOL PE-33

FIGURE 1

V
1.0
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1

0 0.1 0.2 0.3 0.4 0.5 0.6 0.7 0.8 0.9 1.0 1.1 V

11417 350 V/11 (CASE LOCATIONS AS PICTURED)

FIGURE 2

V
1.0
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1

0 0.1 0.2 0.3 0.4 0.5 0.6 0.7 0.8 0.9 1.0 1.1 V

FIGURE 3

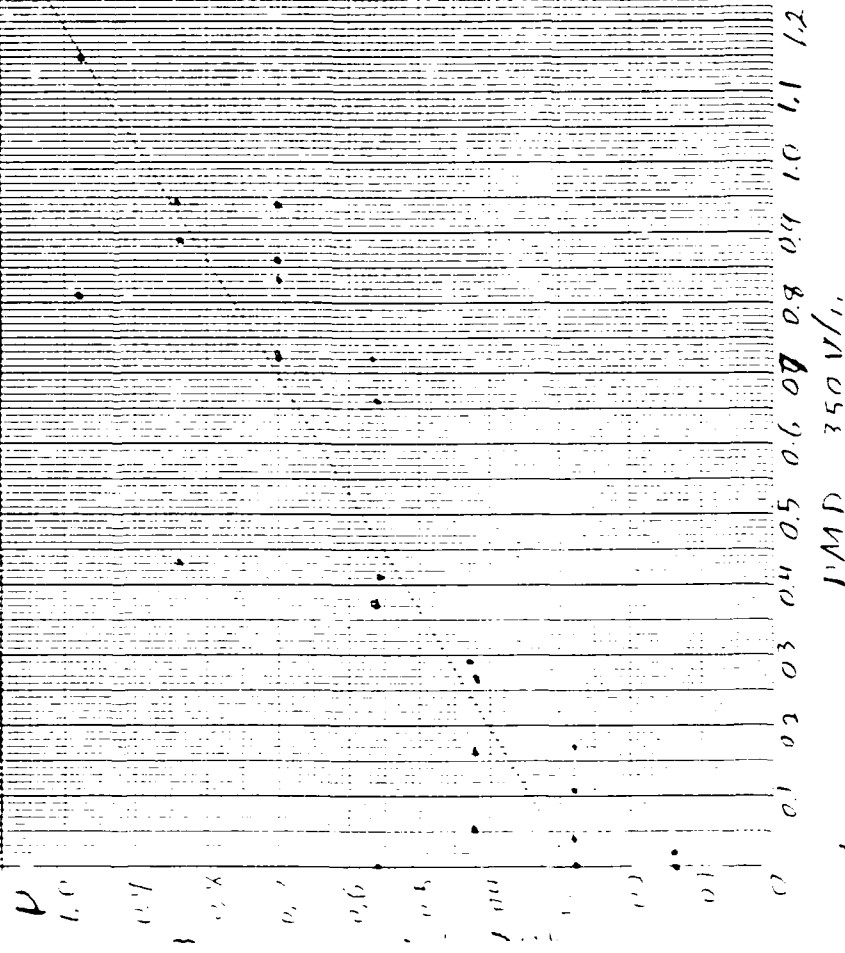
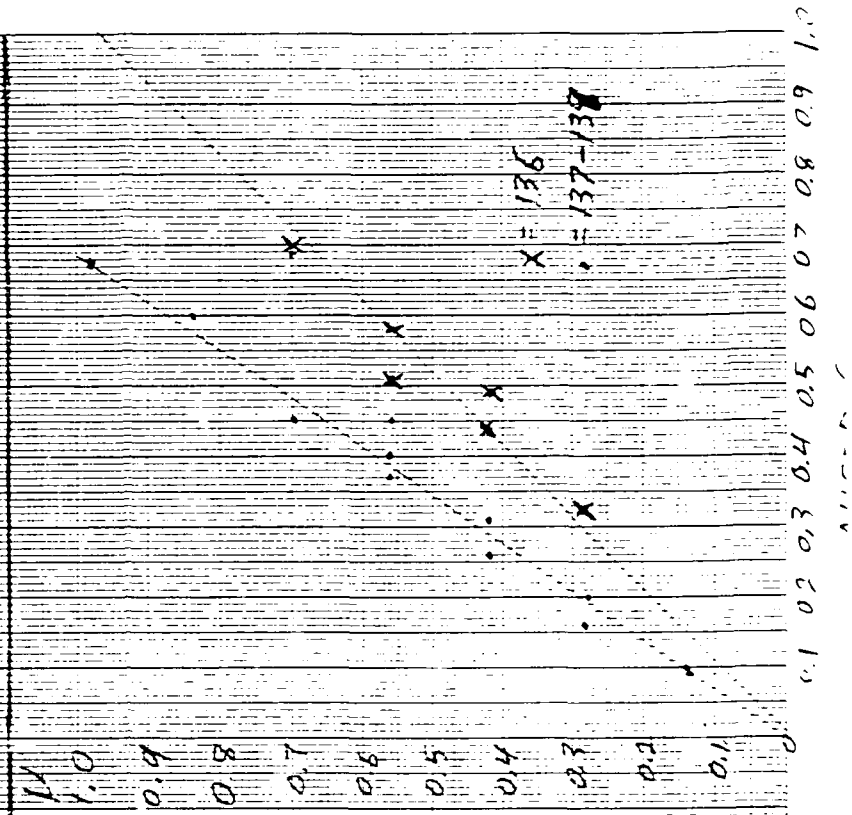


FIGURE 4



DMD DATA

DMD DATA FOR NAPC TUBE NUMBER 136

JFTOT, ASTM D 3241 DMD Test Results:

Maximum Deposit Thickness = 851×10^{-7} cm at Station Number 40
Total Volume of Deposit = 1369×10^{-7} cm³

DMD Dielectric Breakdown Voltage at Each Station Tested
Degree = Tube Rotation Average = Average Volts for Each Station

Station Number	0 Degree	270 Degree	180 Degree	90 Degree	Average Volts*
2	0	0	0	214	54
4	0	0	0	65	16
6	0	0	0	53	13
8	0	0	0	39	10
10	0	0	0	59	15
12	0	0	0	0	0
14	0	4	0	0	1
16	0	0	0	0	0
18	0	4	0	0	1
20	5	0	0	0	1
22	4	6	0	0	3
24	5	7	0	7	5
26	4	24	4	14	12
28	14	40	23	25	26
30	55	61	44	100	65
32	84	72	100	151	102
34	141	67	154	253	154
36	212	182	239	286	230
38	277	156	268	268	242
39	230	210	233	279	238
40	332	250	271	340	298
42	295	251	307	316	292
44	312	245	286	253	274
46	195	192	232	106	181
48	95	116	151	90	113
50	32	72	58	28	48
52	0	5	13	6	6
54	0	1	0	0	0
56	0	4	3	0	2
58	0	0	0	0	0

Total of Averages = 2402

*Note: 350 Volts = 1 Micrometer

(JGB.L4)

DMD DATA FOR NAPC TUBE NUMBER 137

JFTOT, ASTM D 3241 DMD Test Results:

Maximum Deposit Thickness = 974×10^{-7} cm at Station Number 44
 Total Volume of Deposit = 1687×10^{-7} cm³

DMD Dielectric Breakdown Voltage at Each Station Tested

Degree = Tube Rotation

Average = Average Volts for Each Station

Station Number	0 Degree	270 Degree	180 Degree	90 Degree	Average Volts*
2	0	0	0	0	0
4	0	0	0	0	0
6	0	0	0	0	0
8	0	0	0	0	0
10	0	0	0	0	0
12	0	0	0	0	0
14	0	0	0	0	0
16	0	0	0	0	0
18	0	0	0	0	0
20	4	0	0	5	2
22	5	0	0	0	1
24	5	5	6	3	5
26	2	18	8	12	10
28	7	5	33	29	19
30	17	38	47	48	38
32	71	75	57	88	73
34	139	148	112	142	135
36	219	158	150	219	187
38	276	241	269	273	265
39	234	251	266	260	253
40	379	254	327	331	323
42	372	284	366	310	333
44	350	291	337	385	341
46	291	288	151	357	272
48	276	253	218	292	260
50	170	180	217	208	194
52	100	129	142	141	128
54	83	48	116	53	75
56	27	23	84	41	44
58	0	0	0	5	1

Total of Averages = 2959

*Note: 350 Volts = 1 Micrometer

(JGB.L5)

DMD DATA FOR NAPC TUBE NUMBER 138

JFTOT, ASTM D 3241 DMD Test Results:

Maximum Deposit Thickness = 1117×10^{-7} cm at Station Number 44
Total Volume of Deposit = 1644×10^{-7} cm³

DMD Dielectric Breakdown Voltage at Each Station Tested

Degree = Tube Rotation

Average = Average Volts for Each Station

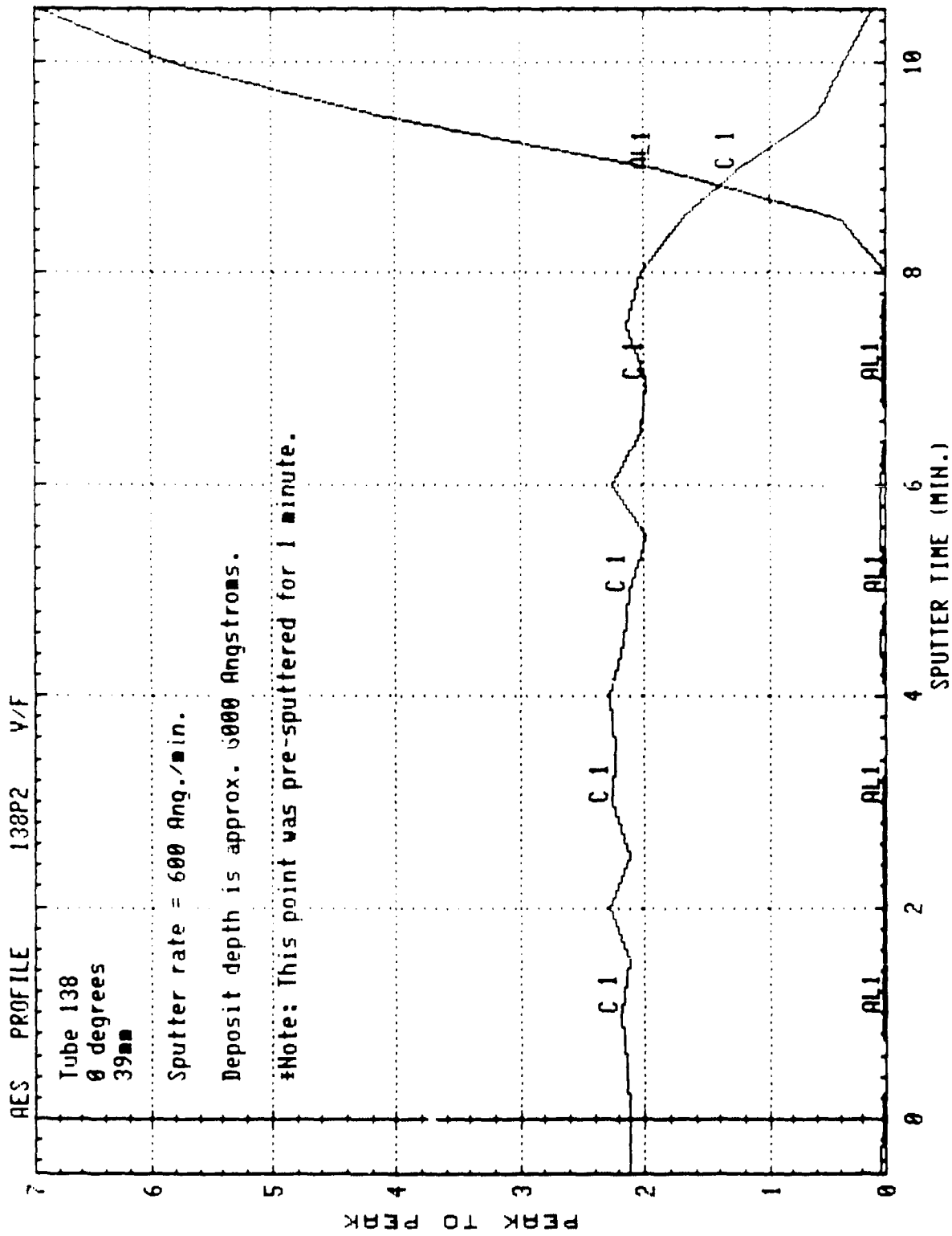
Station Number	0 Degree	270 Degree	180 Degree	90 Degree	Average Volts*
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4	0	0	0	0	0
6	0	0	0	0	0
8	0	0	0	0	0
10	0	0	0	0	0
12	0	0	0	0	0
14	0	0	0	0	0
16	0	0	0	0	0
18	0	0	0	0	0
20	0	0	0	4	1
22	0	0	0	5	1
24	0	0	0	0	0
26	6	0	0	5	3
28	0	6	6	7	5
30	43	0	10	7	15
32	121	118	17	21	69
34	139	169	167	131	152
36	239	254	292	197	246
38	294	303	403	304	326
39	317	241	208	305	268
40	375	329	419	340	366
42	315	401	275	396	347
44	369	398	364	434	391
46	332	351	328	402	353
48	286	0	327	0	153
50	137	5	223	4	92
52	130	6	123	0	65
54	64	4	46	0	29
56	0	5	6	0	3
58	0	0	0	0	0

Total of Averages = 2885

*Note: 350 Volts = 1 Micrometer

(JGB.L6)

AUGER ION MILLING DATA



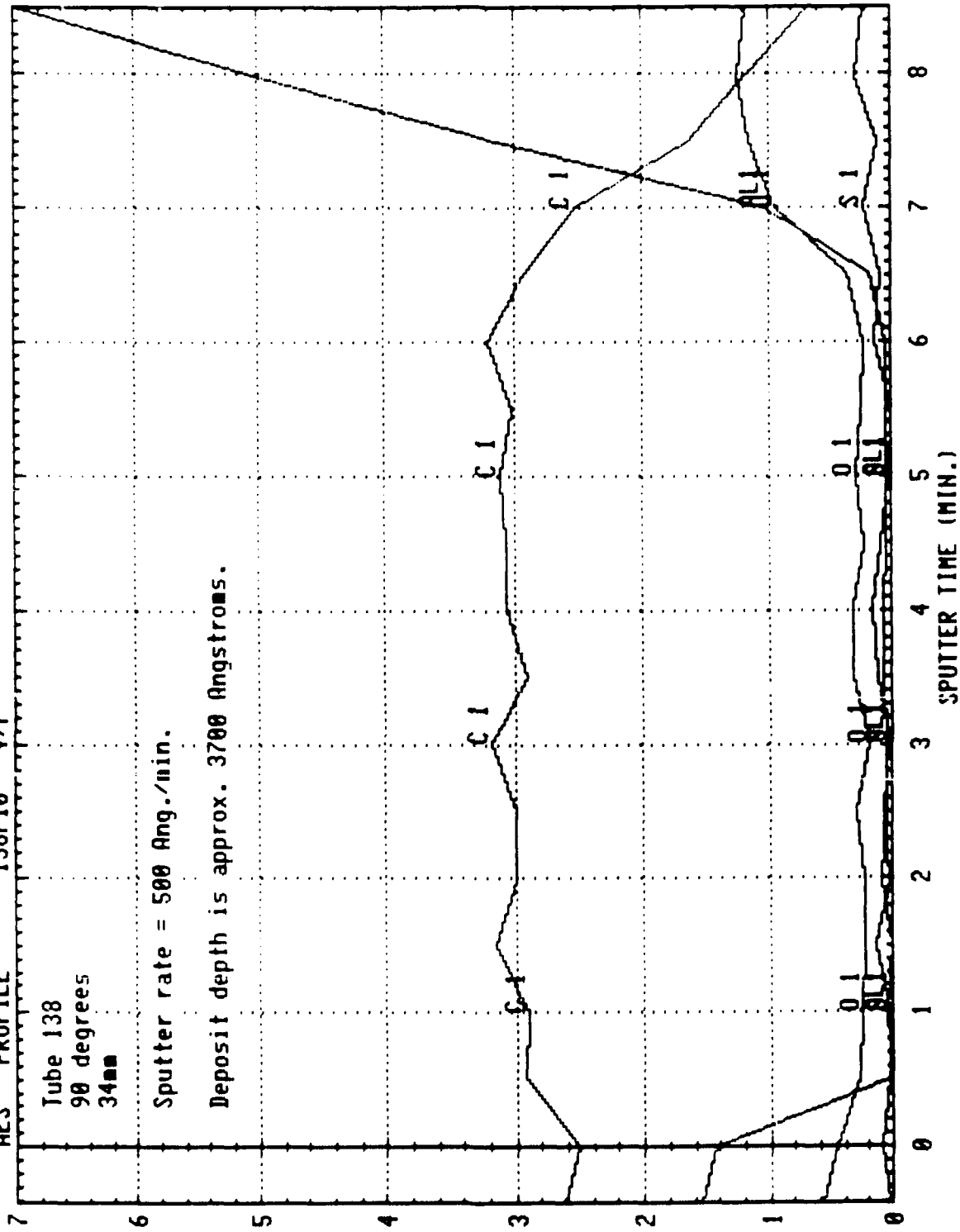
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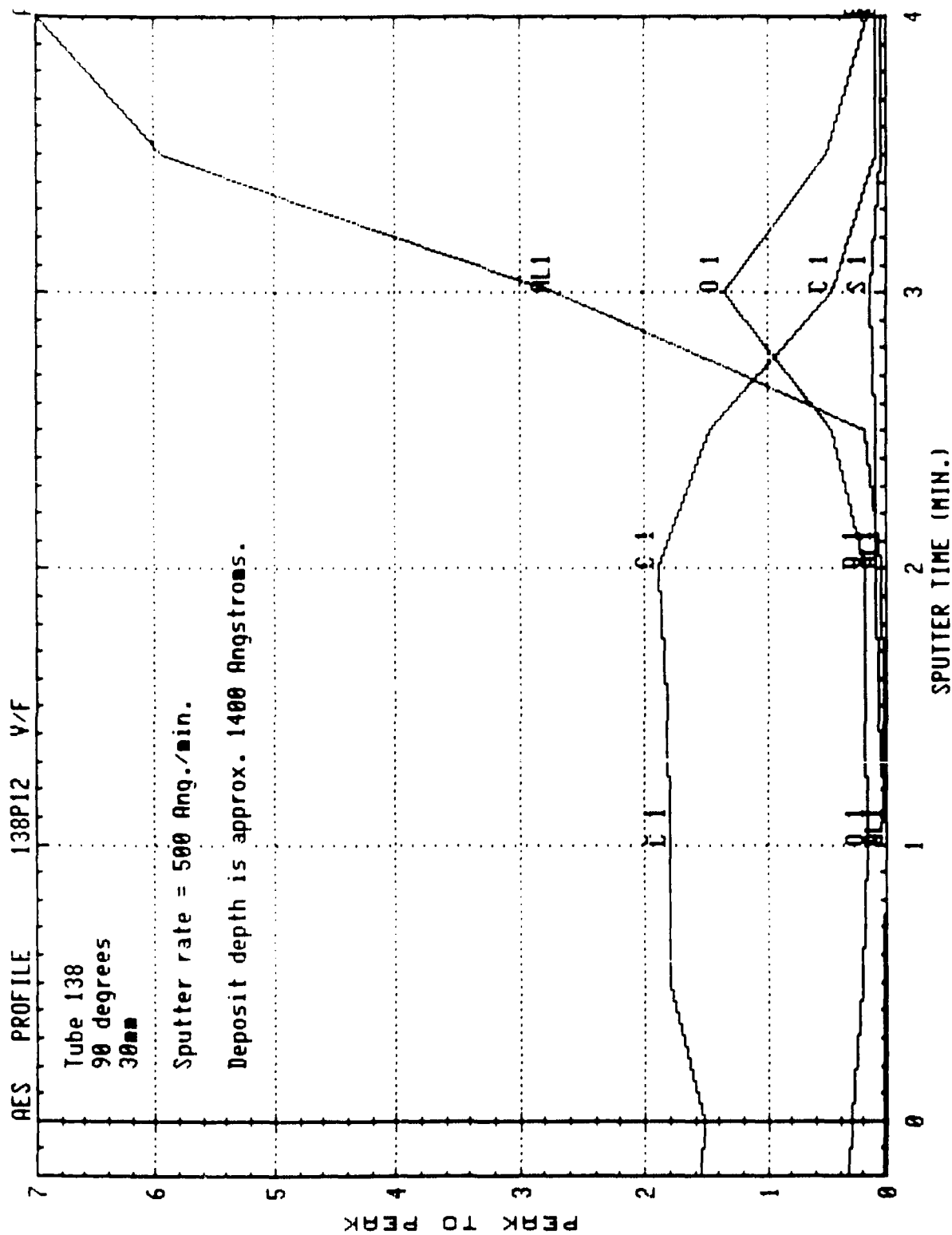
Tube 138
90 degrees
34mm

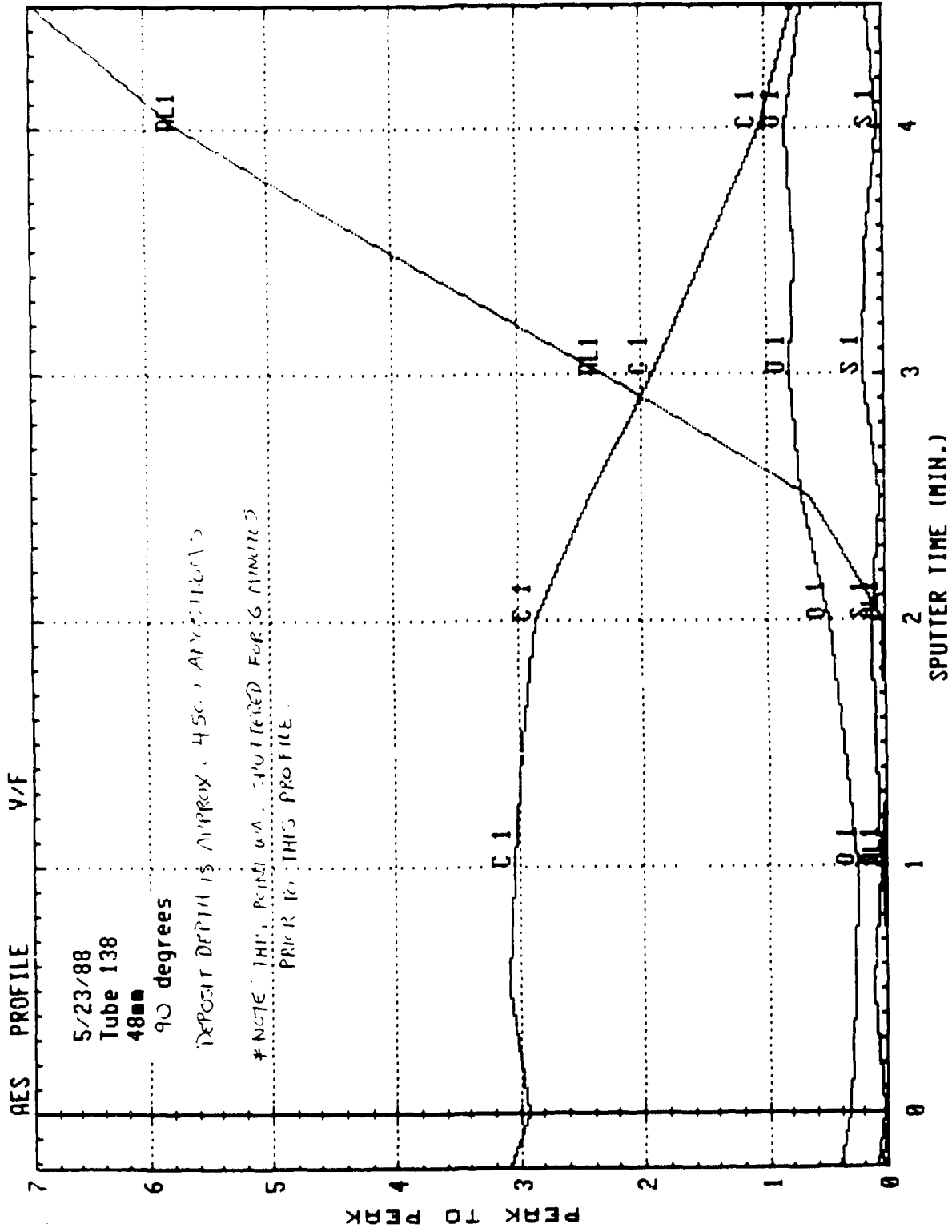
Sputter rate = 500 Ang./min.

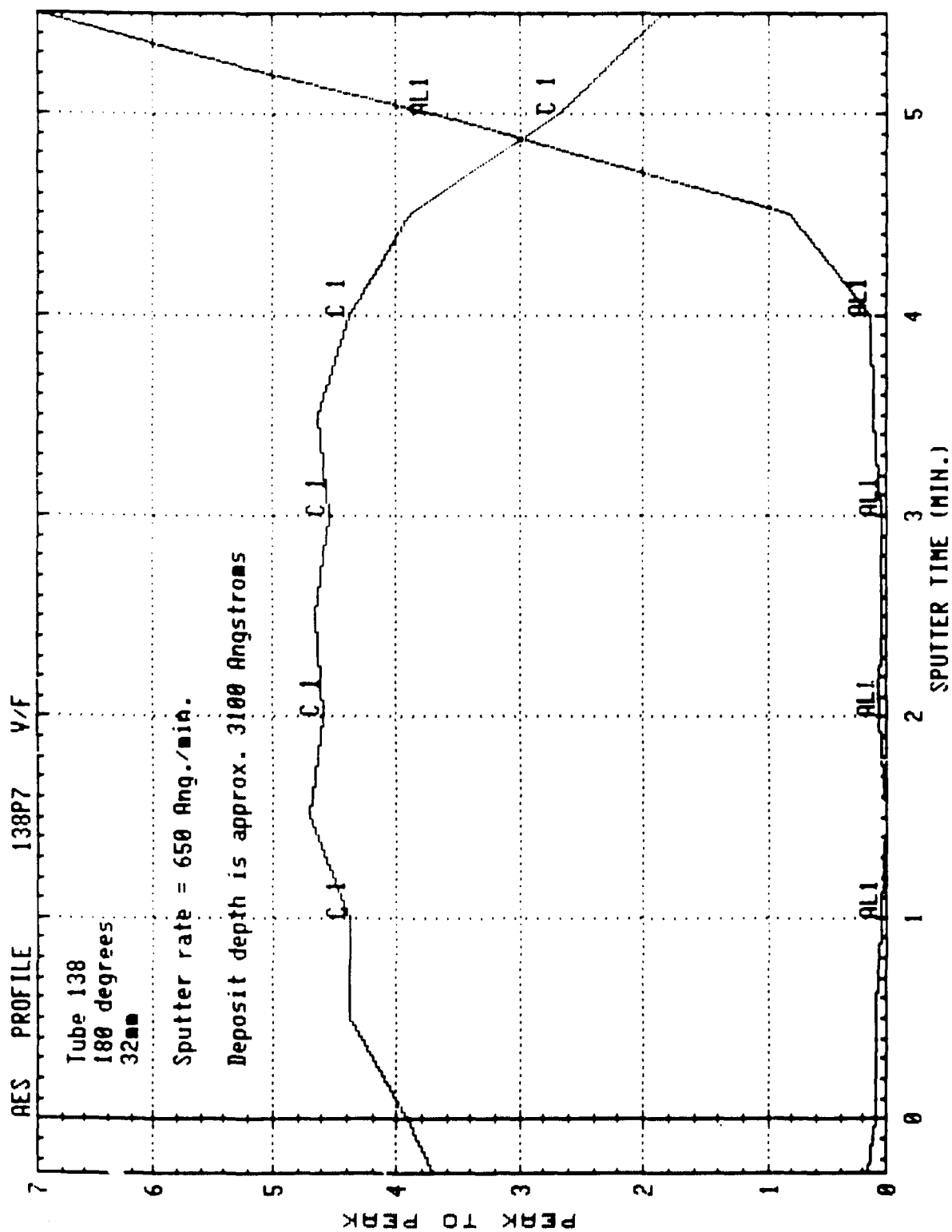
Deposit depth is approx. 3700 Angstroms.

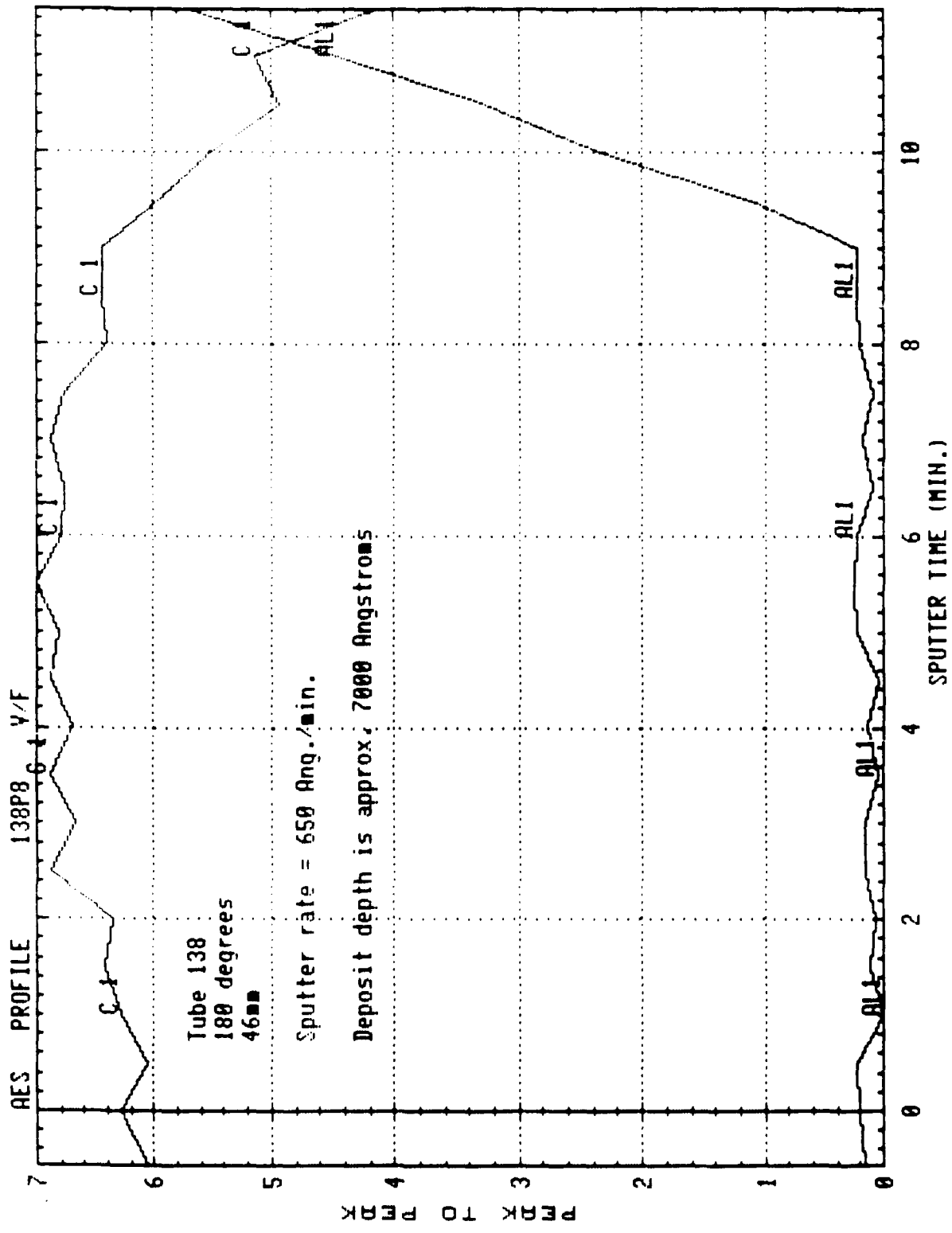
PERK TO PERK











AES PROFILE 138P9 V/F

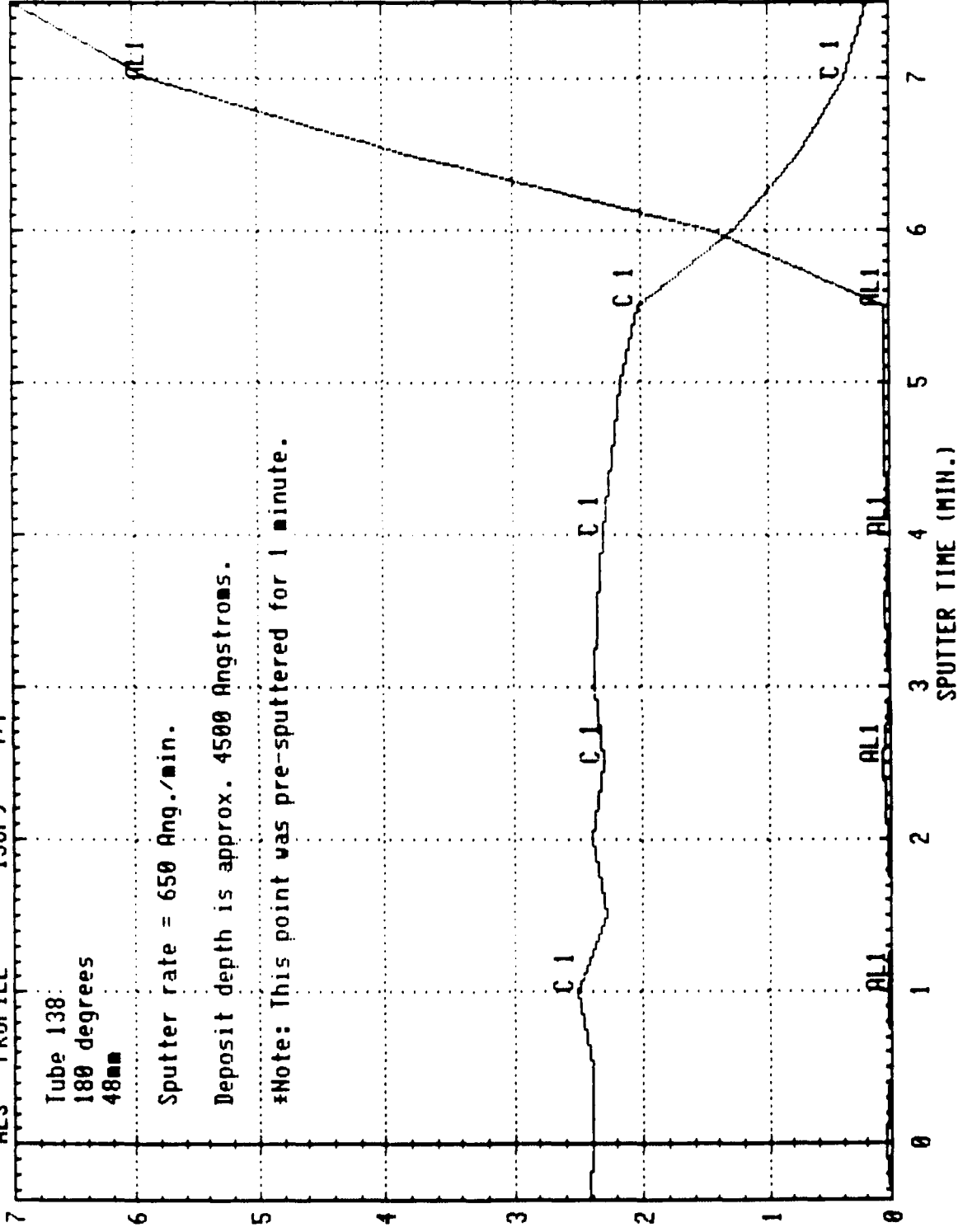
Tube 138:
180 degrees
48mm

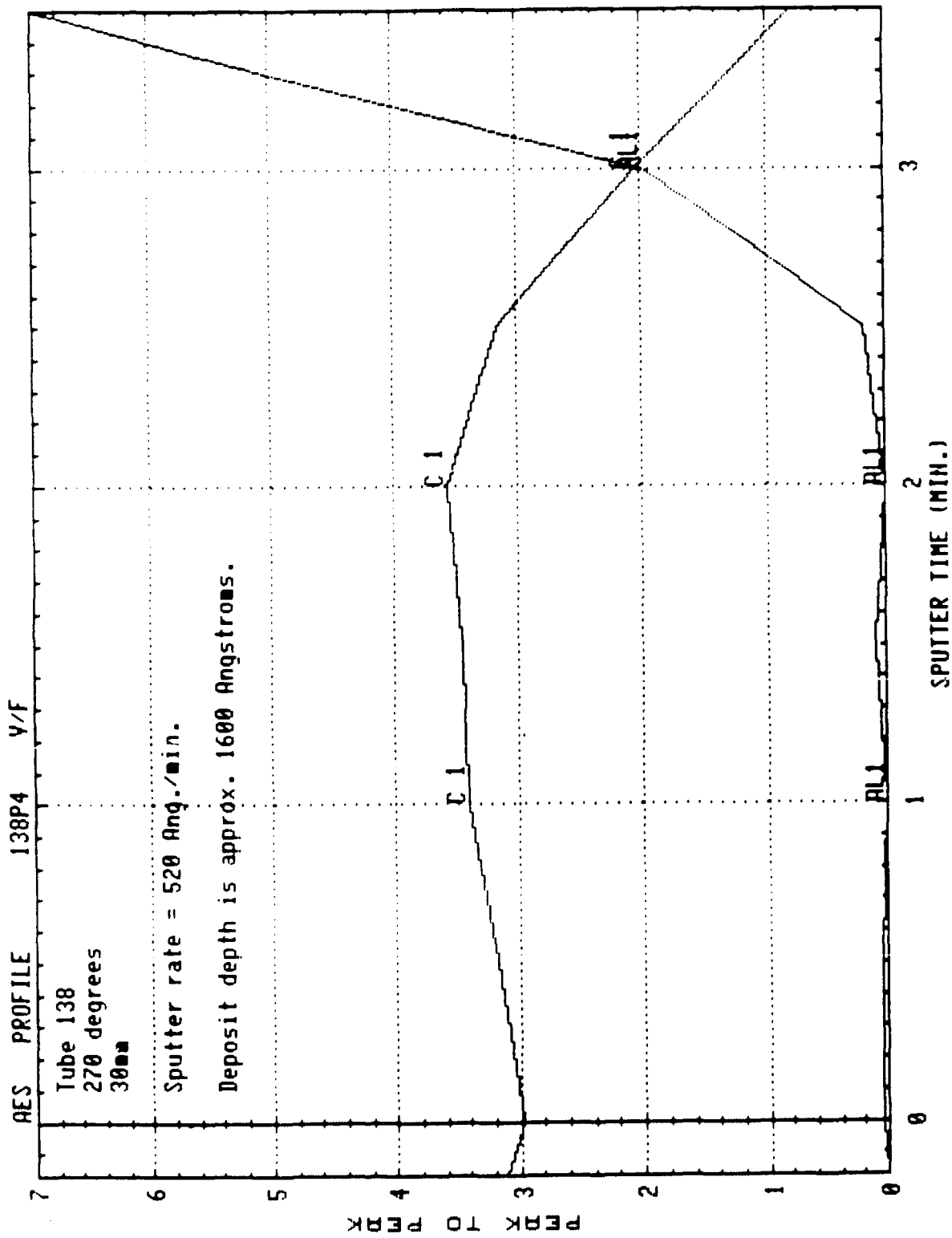
Sputter rate = 650 Ang./min.

Deposit depth is approx. 4500 Angstroms.

*Note: This point was pre-sputtered for 1 minute.

PEDK TO PEDK





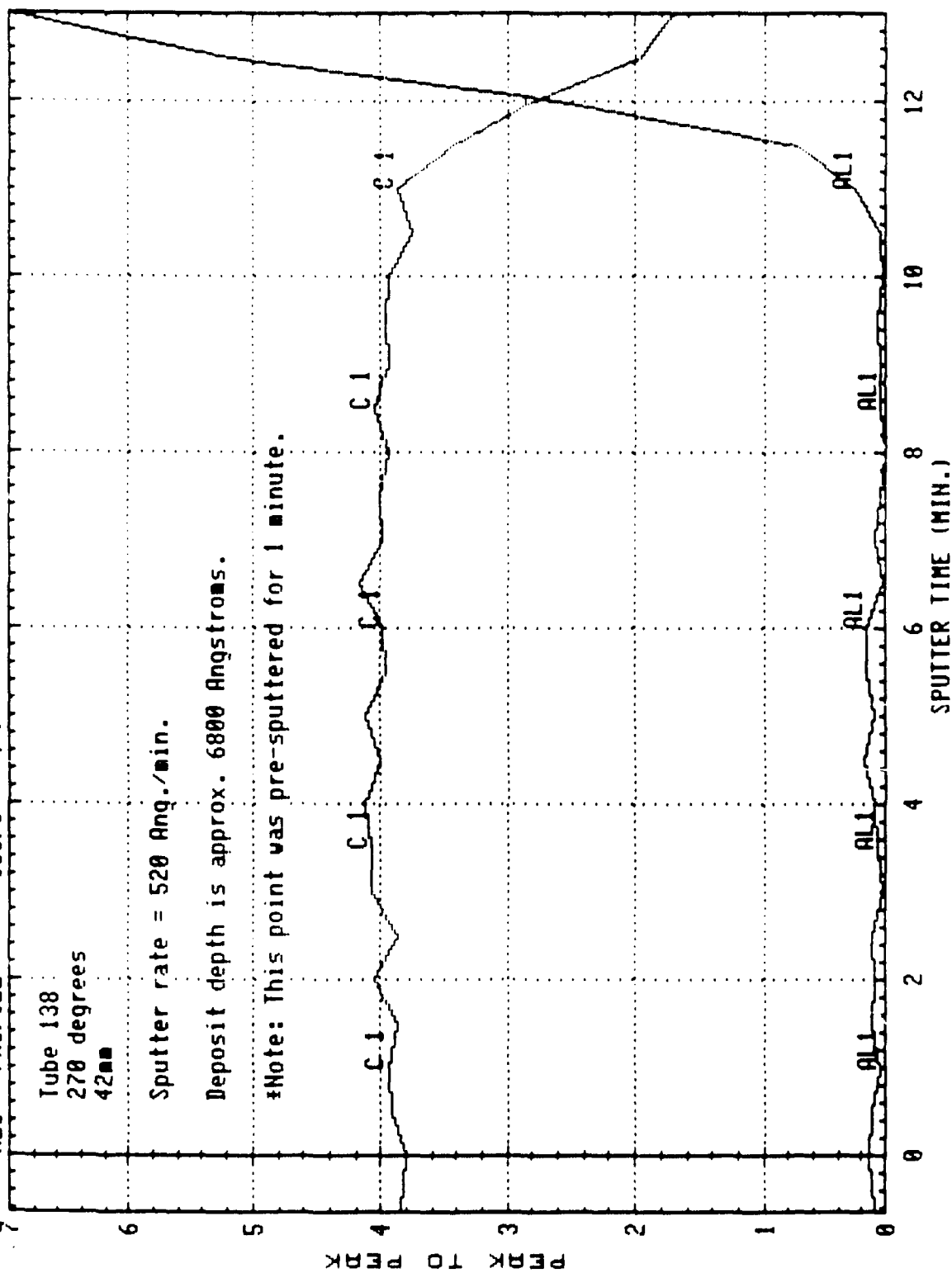
AES PROFILE 138P5 V/F

Tube 138
270 degrees
42mm

Sputter rate = 520 Ang./min.

Deposit depth is approx. 6800 Angstroms.

*Note: This point was pre-sputtered for 1 minute.



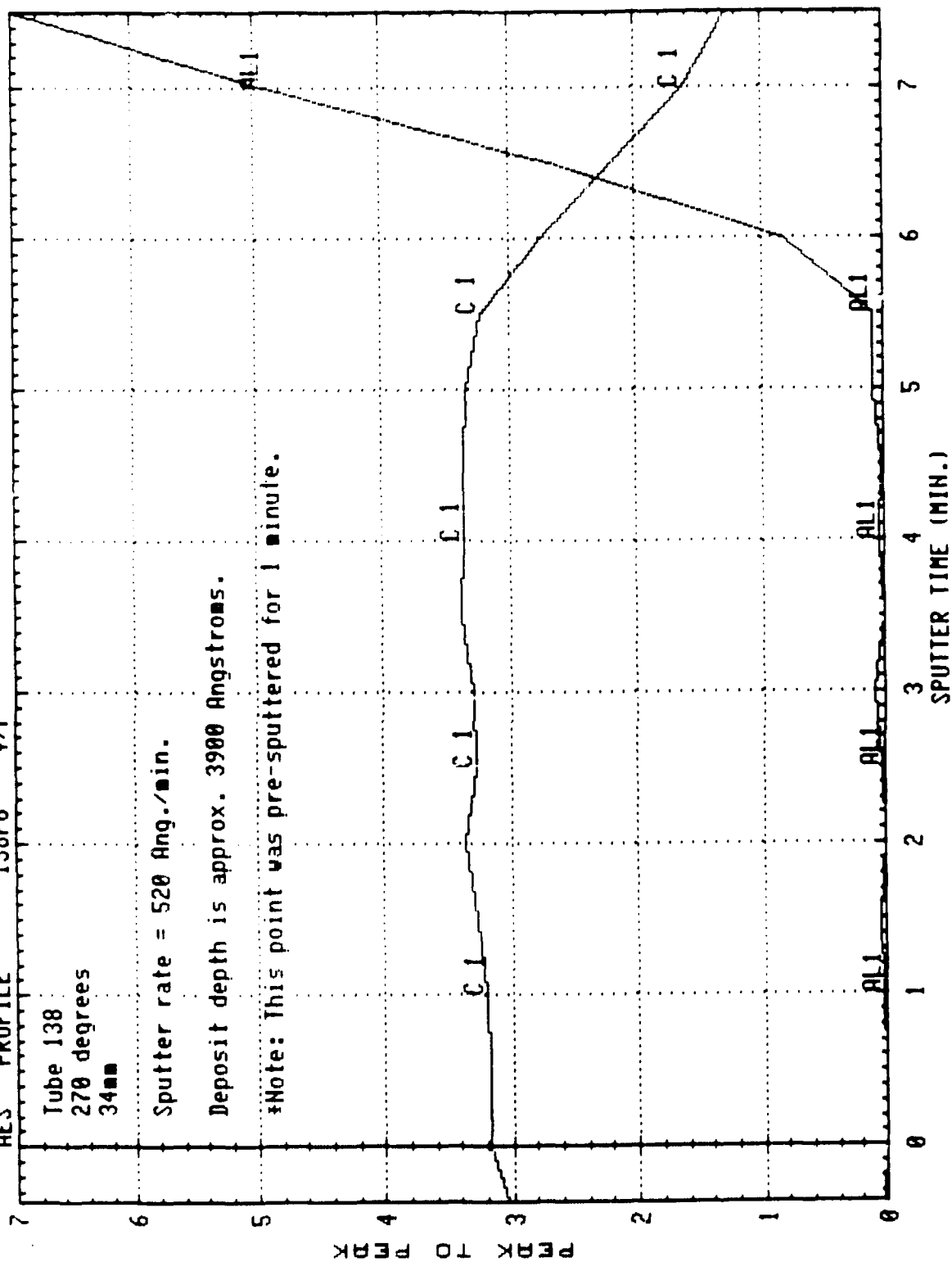
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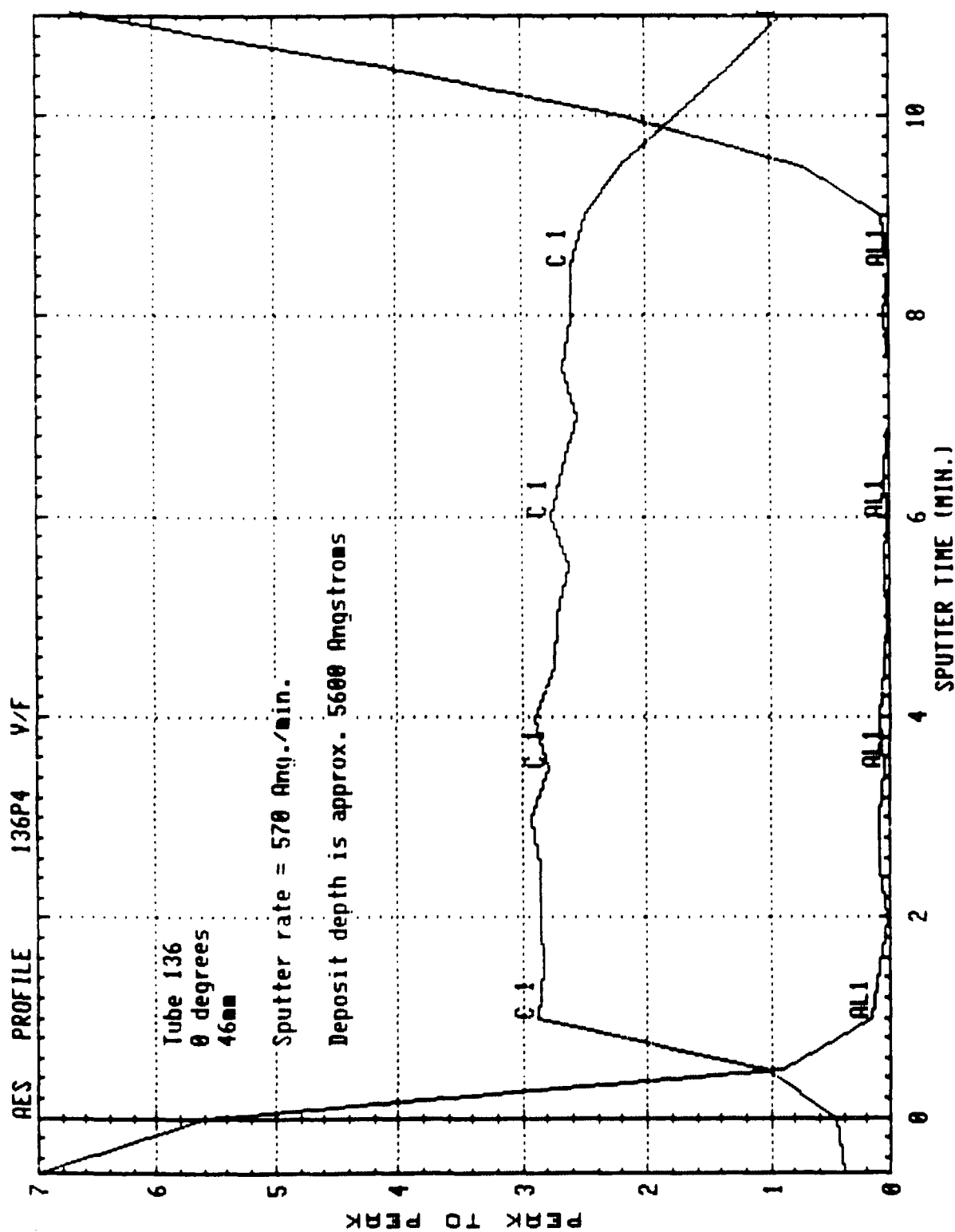
Tube 138
270 degrees
34mm

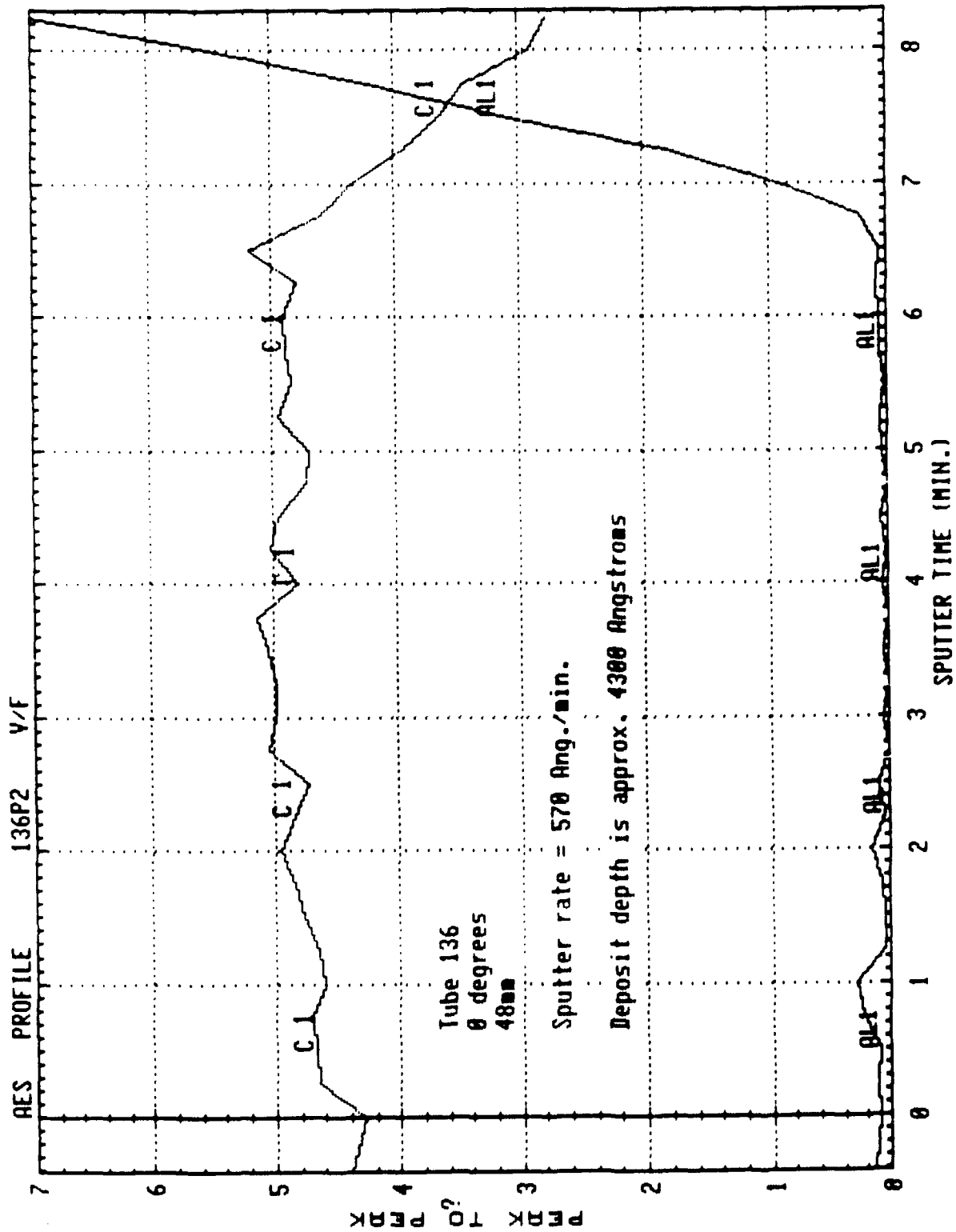
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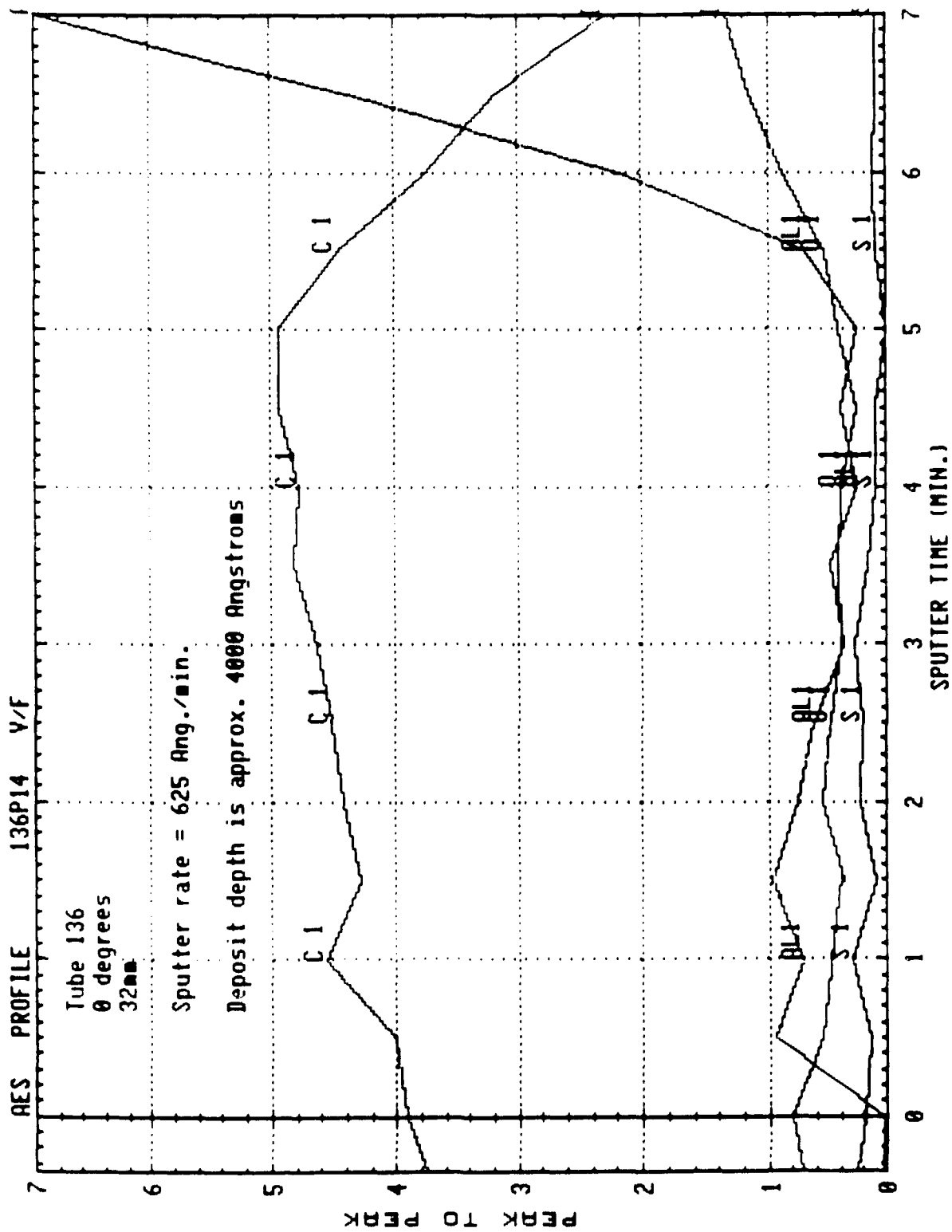
Deposit depth is approx. 3900 Angstroms.

*Note: This point was pre-sputtered for 1 minute.









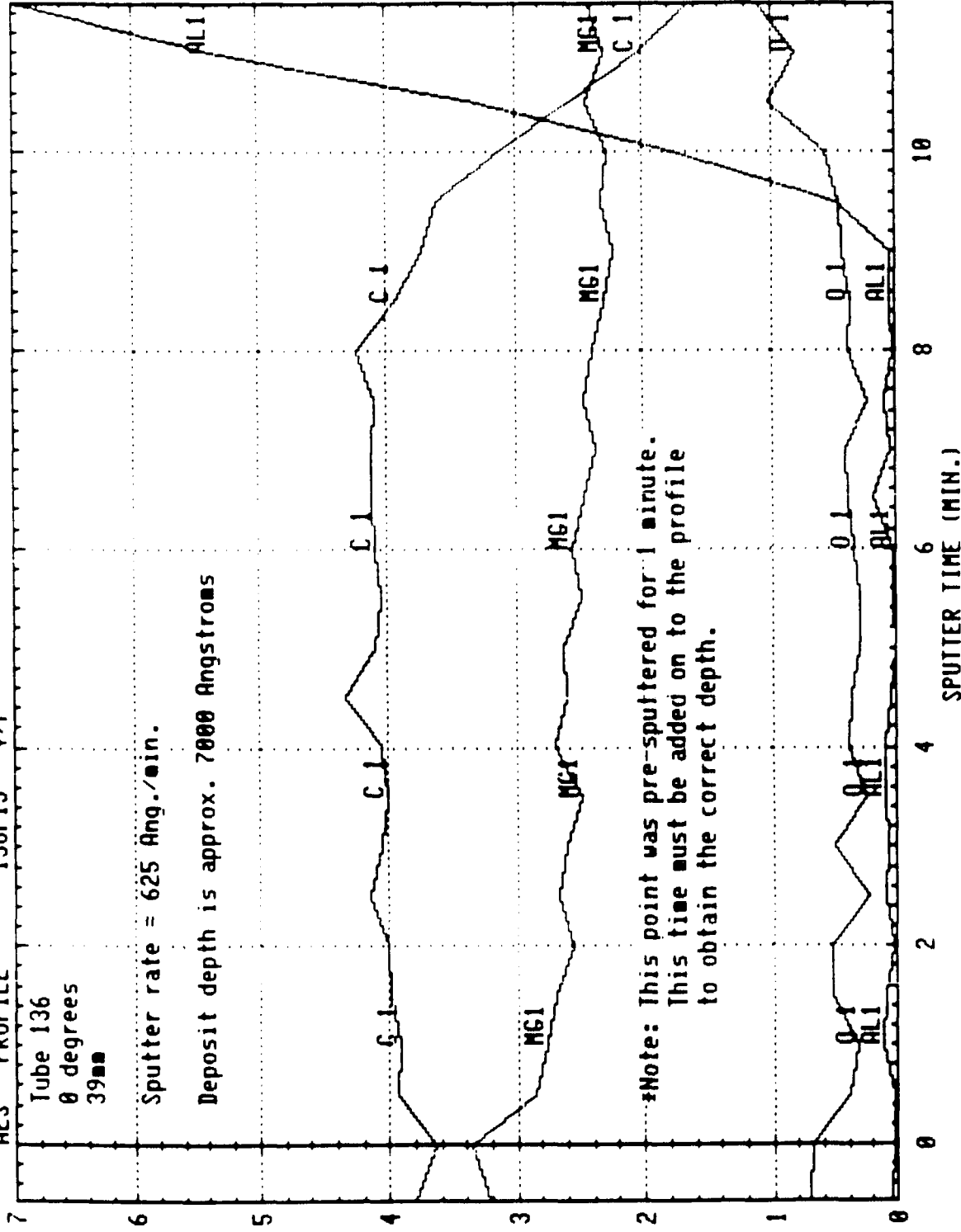
AES PROFILE 136P15 V/F

Tube 136
0 degrees
39mm

Sputter rate = 625 Ang./min.

Deposit depth is approx. 7000 Angstroms

PEAK TO PEAK



*Note: This point was pre-sputtered for 1 minute.
This time must be added on to the profile
to obtain the correct depth.

AES PROFILE 136P13 V/F

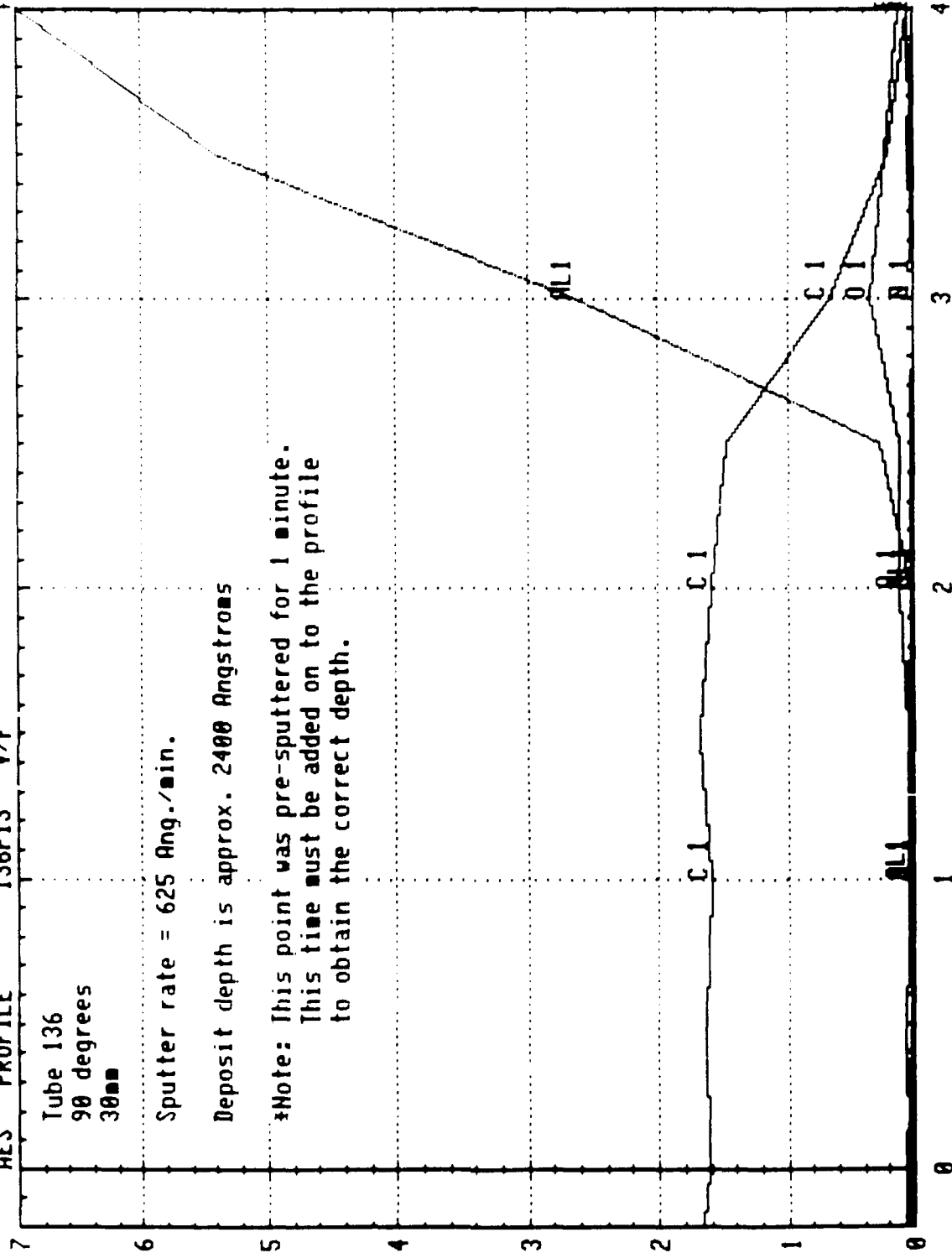
Tube 136
90 degrees
30mm

Sputter rate = 625 Ang./min.

Deposit depth is approx. 2400 Angstroms

*Note: This point was pre-sputtered for 1 minute.
This time must be added on to the profile
to obtain the correct depth.

PEAK TO PEAK



SPUTTER TIME (MIN.)

AES PROFILE 136P12 V/F

Tube 136
90 degrees
48mm

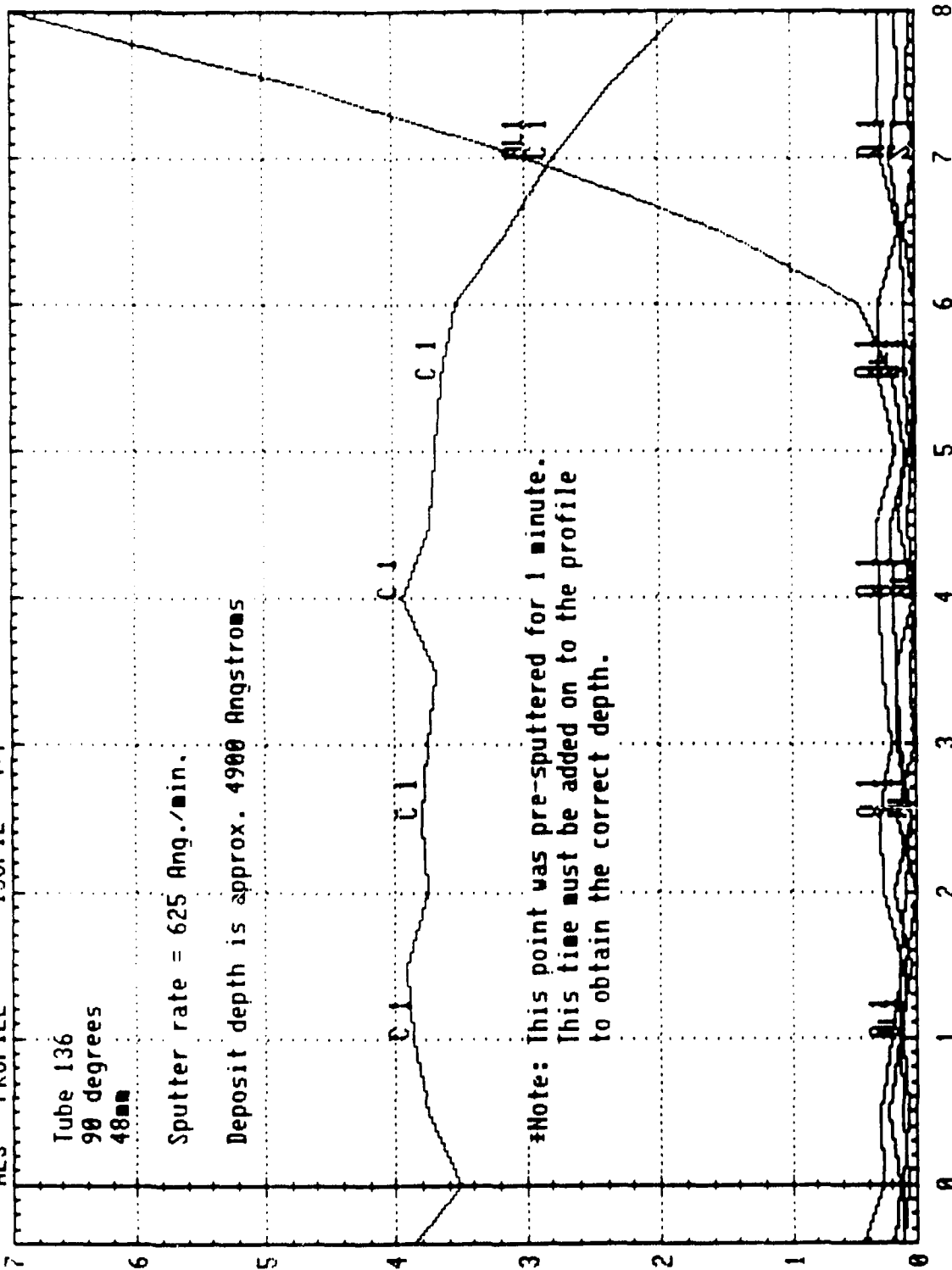
Sputter rate = 625 Ang./min.

Deposit depth is approx. 4900 Angstroms

PERCENT TO PEAK

SPUTTER TIME (MIN.)

Note: This point was pre-sputtered for 1 minute.
This time must be added on to the profile
to obtain the correct depth.



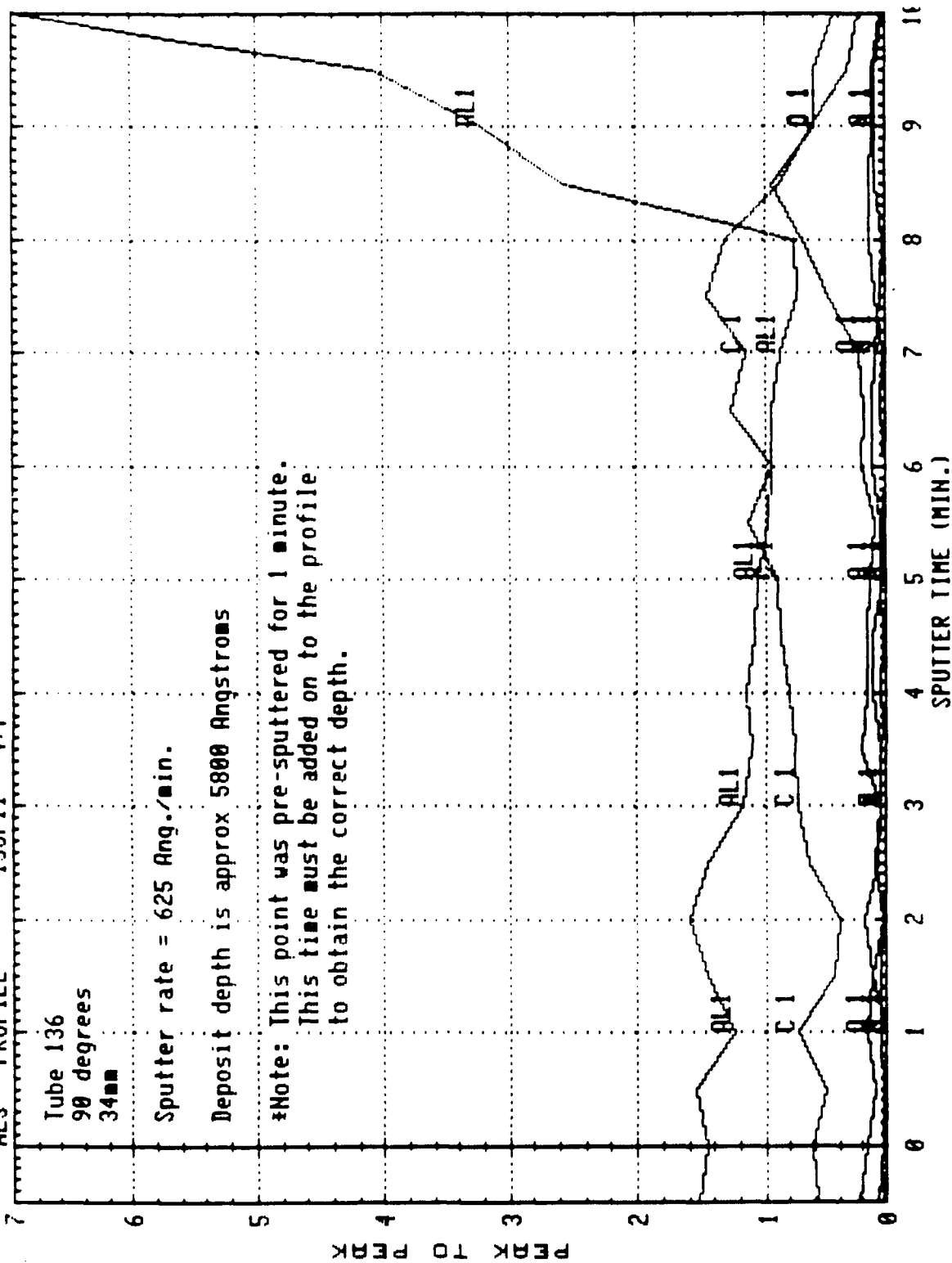
AES PROFILE 136P11 V/F

Tube 136
90 degrees
34mm

Sputter rate = 625 Ang./min.

Deposit depth is approx 5800 Angstroms

*Note: This point was pre-sputtered for 1 minute.
This time must be added on to the profile
to obtain the correct depth.



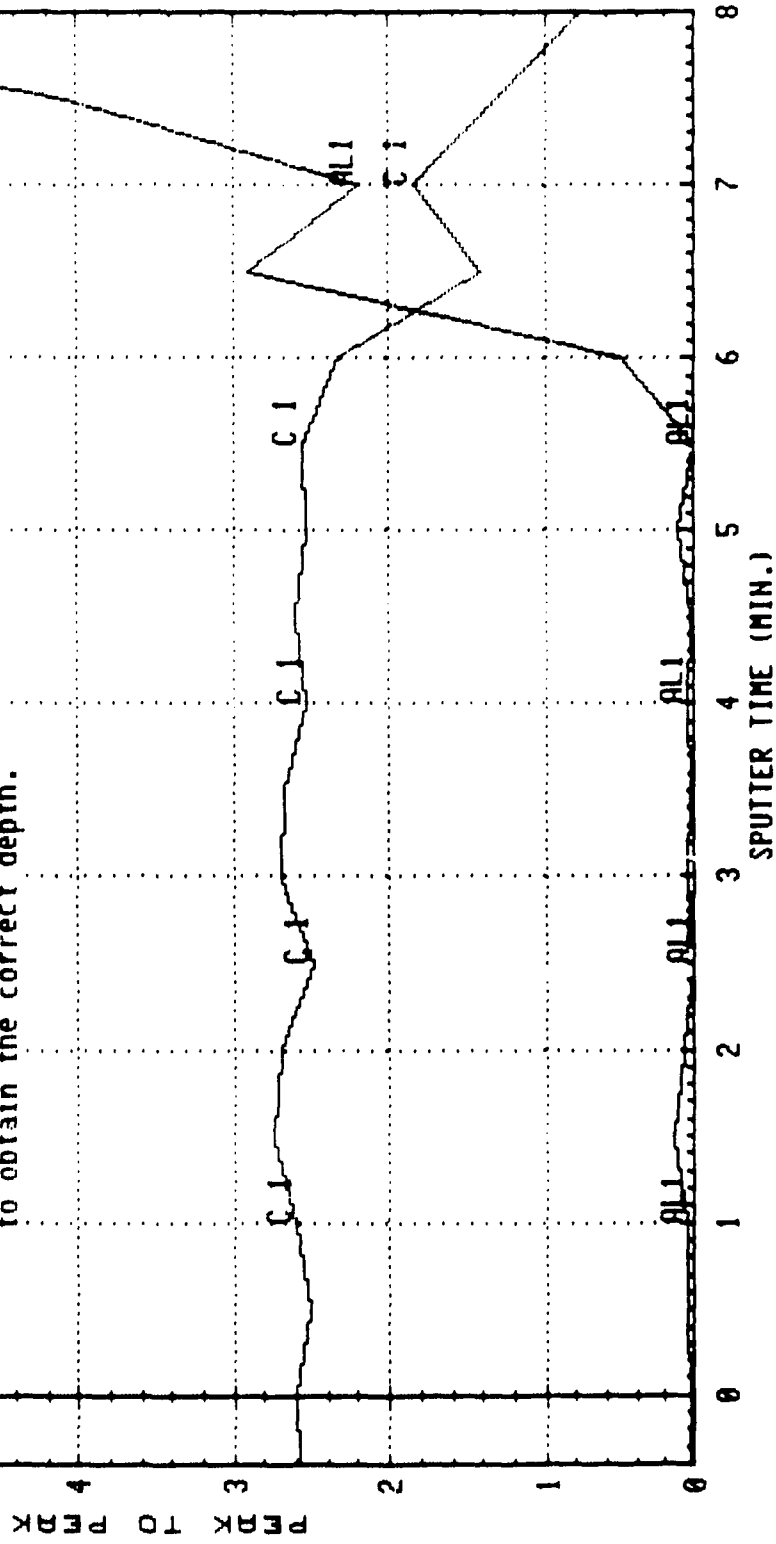
AES PROFILE 136P10 V/F

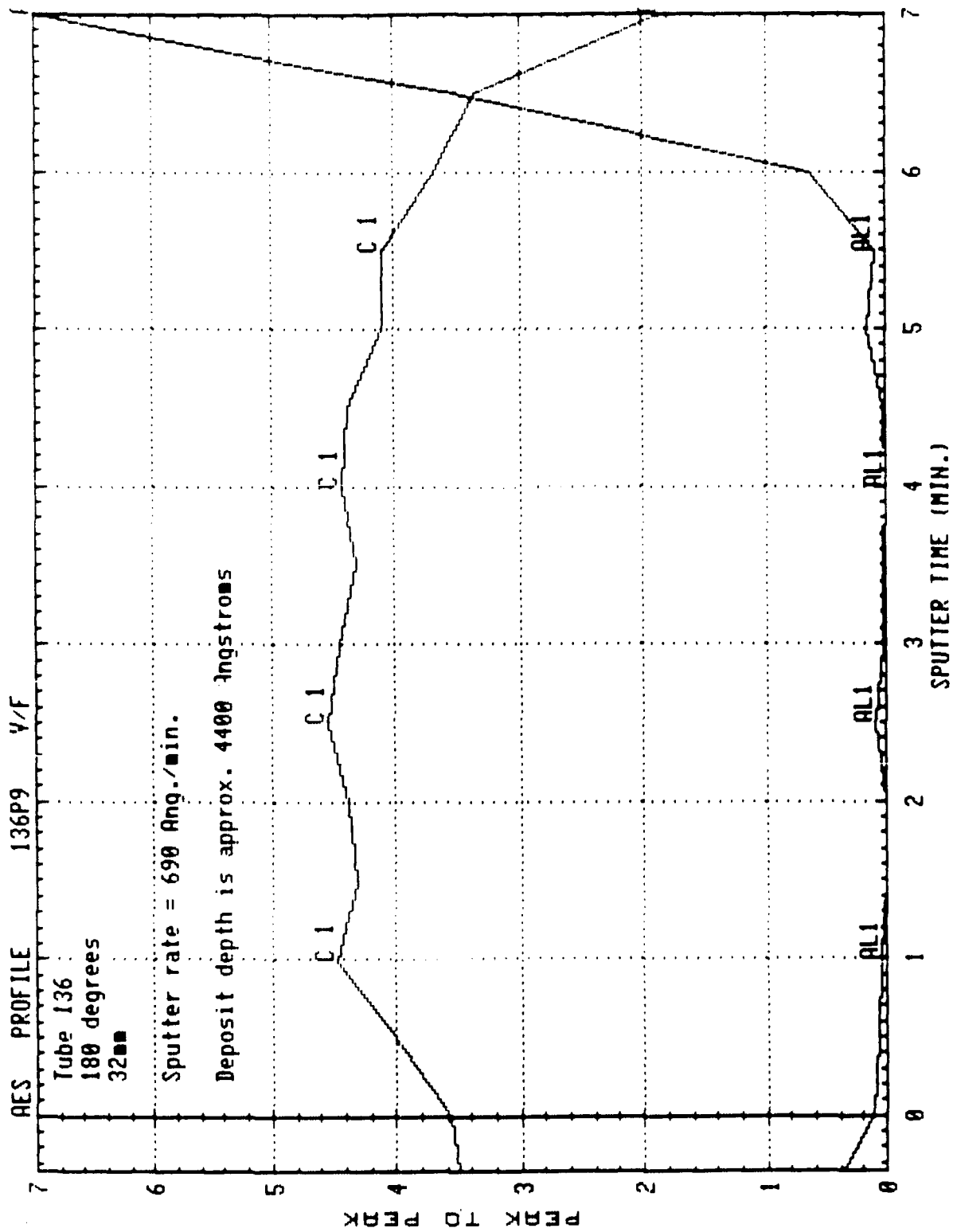
Tube 136
180 degrees
46mm

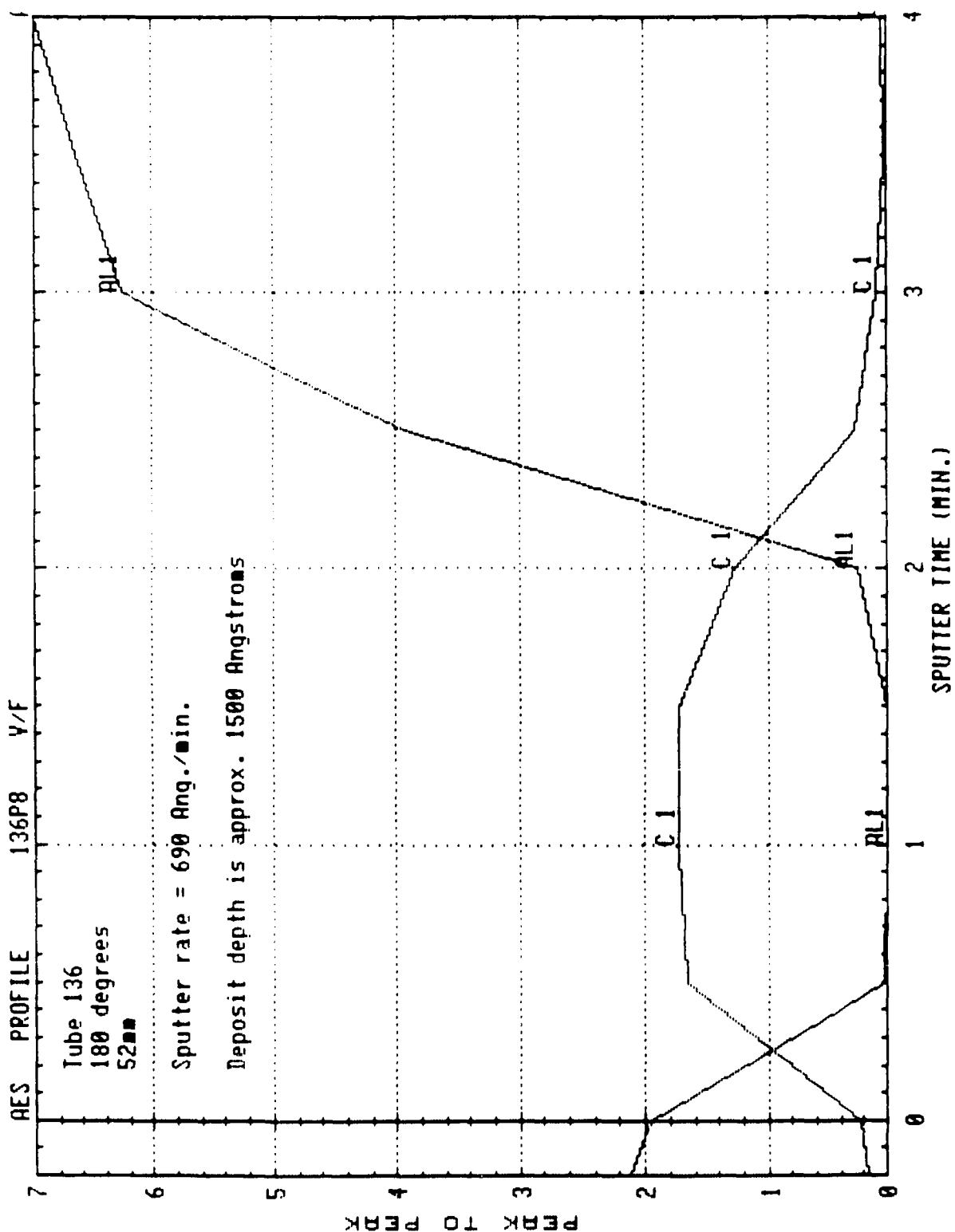
Sputter rate = 720 Ang./min.

Deposit depth is approx. 5100 Angstroms

Note: This point was pre-sputtered for 1 minute.
This time must be added on to the profile
to obtain the correct depth.





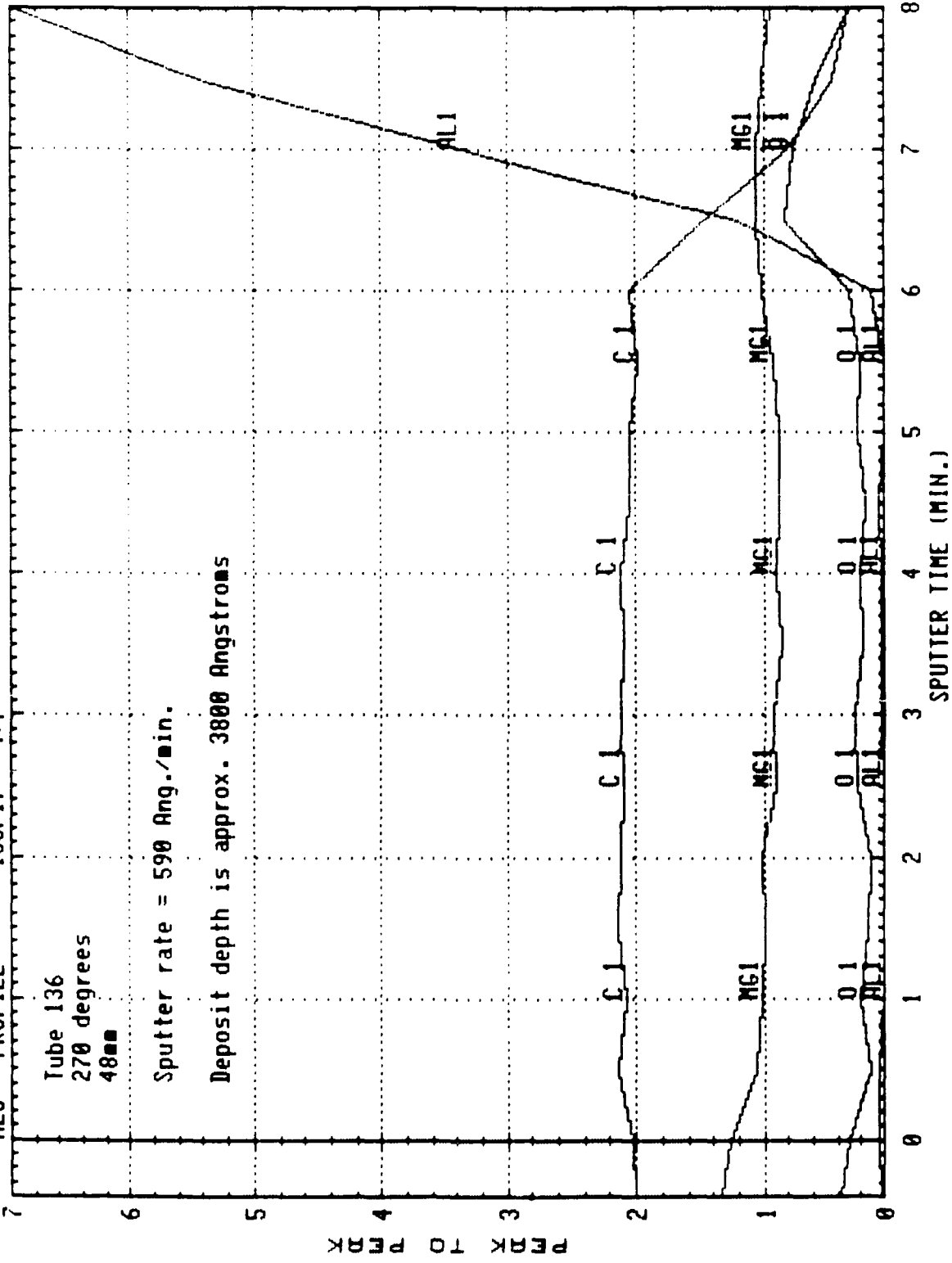


AES PROFILE 136P17 V/F

Tube 136
270 degrees
48mm

Sputter rate = 590 Ang./min.

Deposit depth is approx. 3800 Angstroms



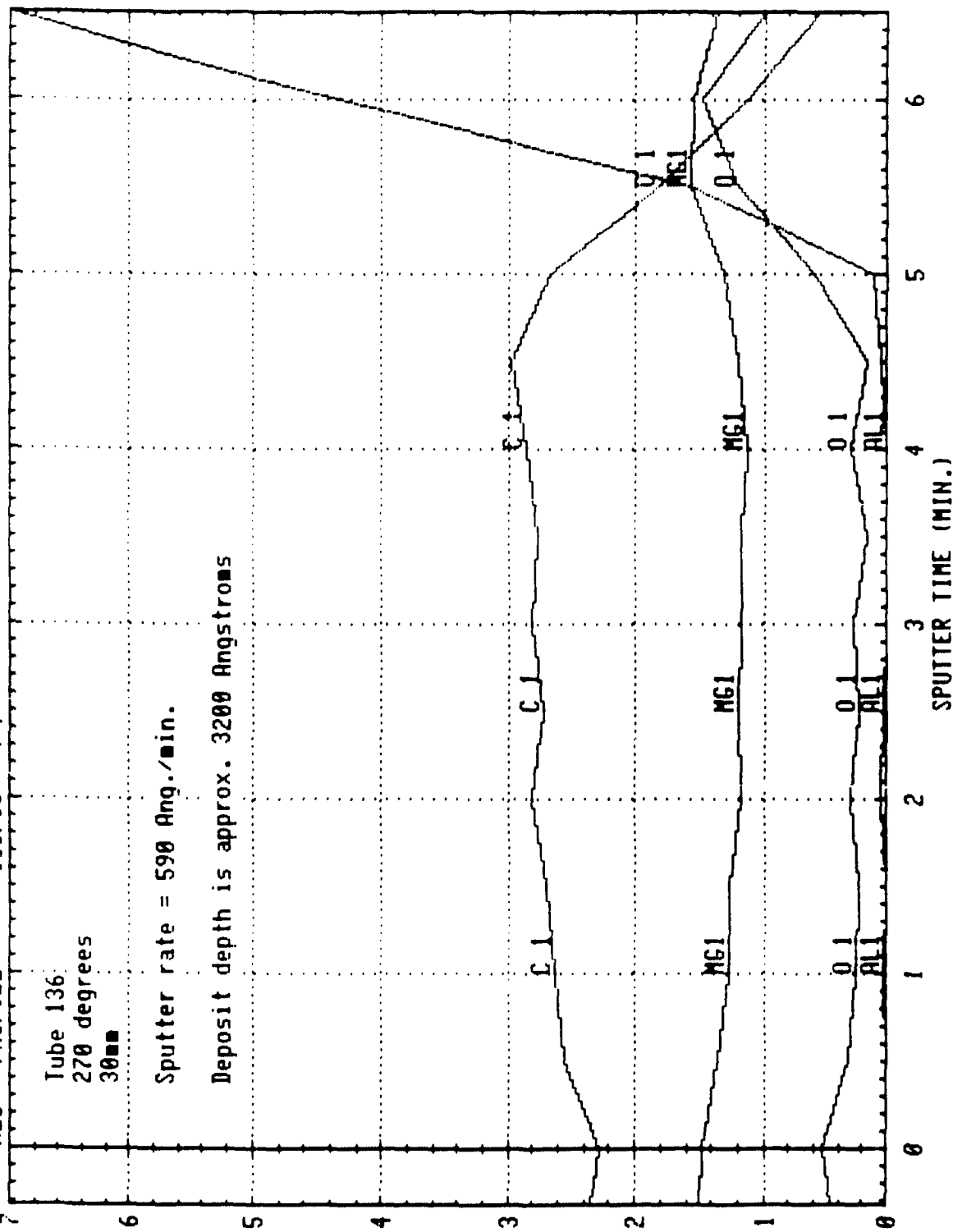
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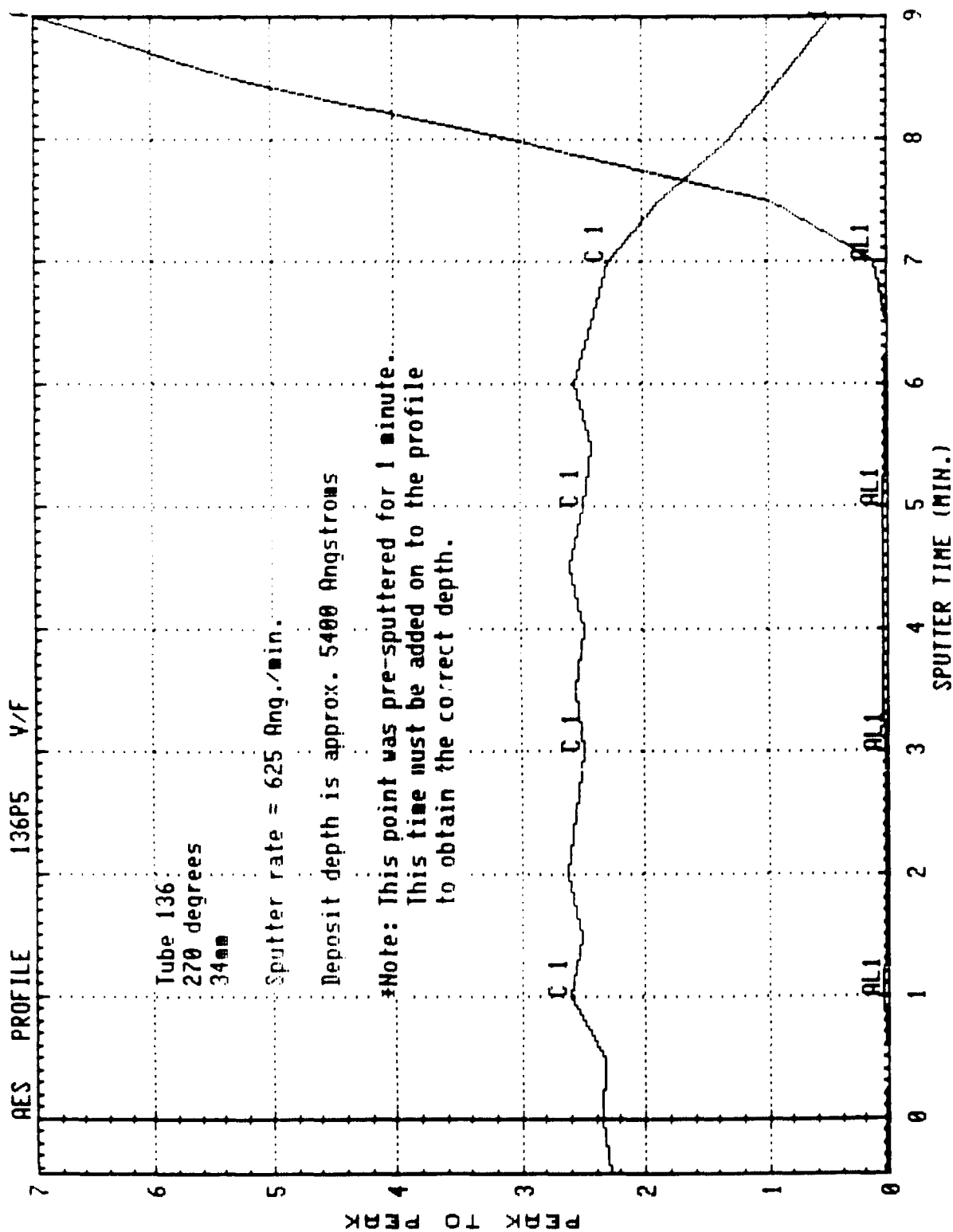
Tube 136
270 degrees
30mm

Sputter rate = 590 Ang./min.

Deposit depth is approx. 3200 Angstroms

PERK TO PERK





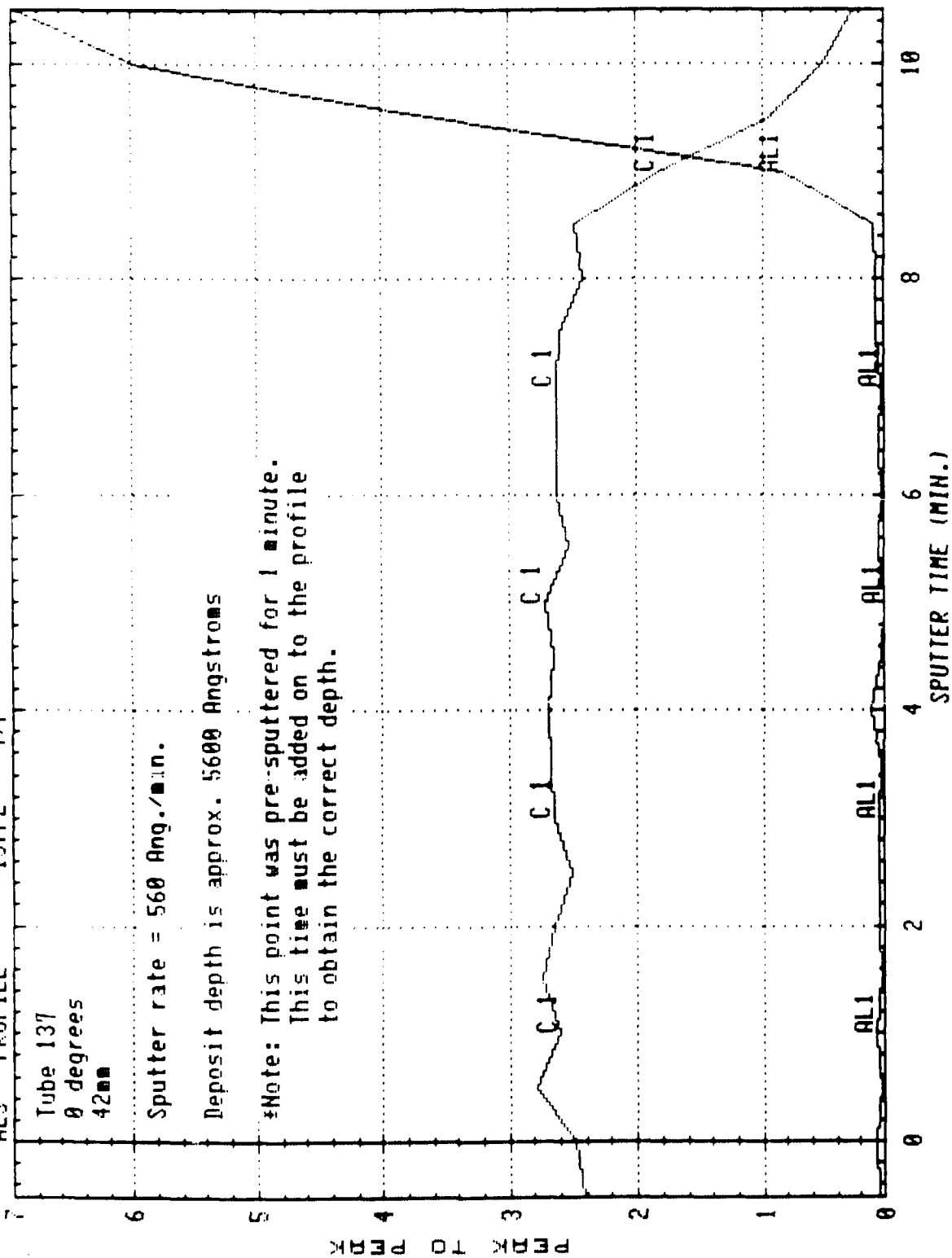
AES PROFILE 137P2 V/F

Tube 137
0 degrees
42mm

Sputter rate = 560 Ang./min.

Deposit depth is approx. 5600 Angstroms

*Note: This point was pre-sputtered for 1 minute.
This time must be added on to the profile
to obtain the correct depth.

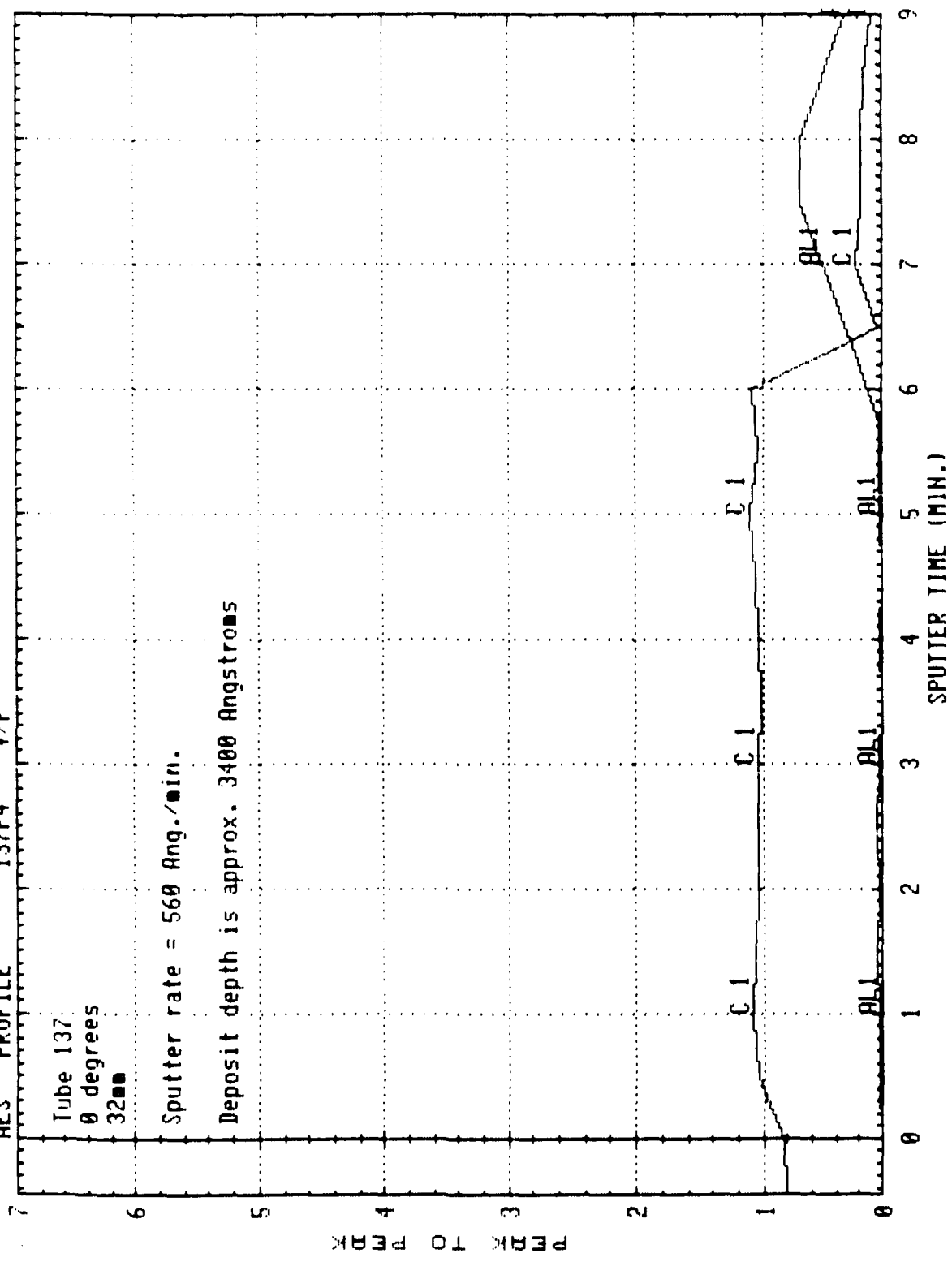


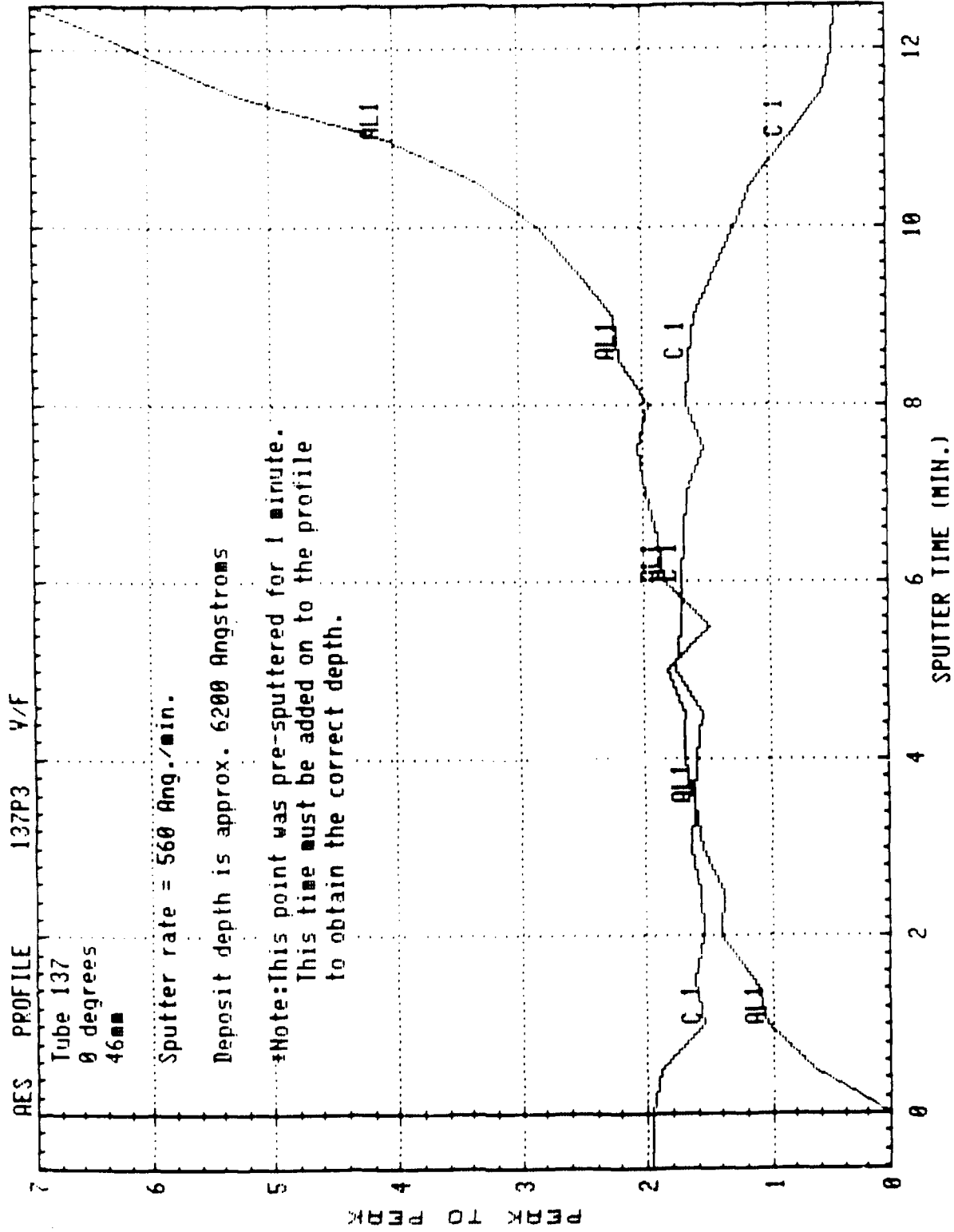
AES PROFILE 137P4 V/F

Tube 137
0 degrees
32mm

Sputter rate = 560 Ang./min.

Deposit depth is approx. 3400 Angstroms





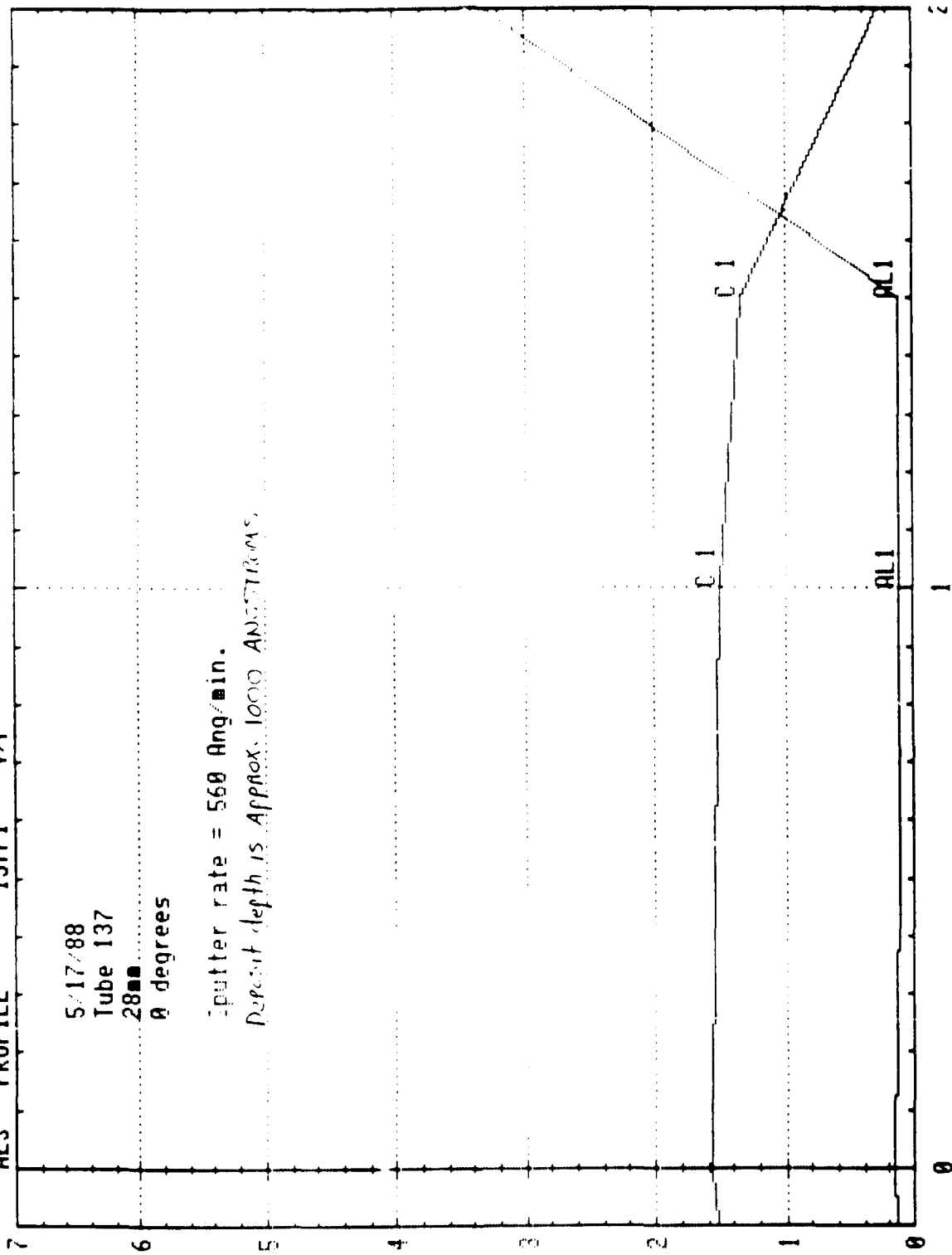
AES PROFILE 137P1 V/F

5/17/88
Tube 137
28mm
0 degrees

Sputter rate = 560 Ang/min.

Deposit depth is approx. 1000 Angstroms.

PEAK TO PEAK



SPUTTER TIME (MIN.)

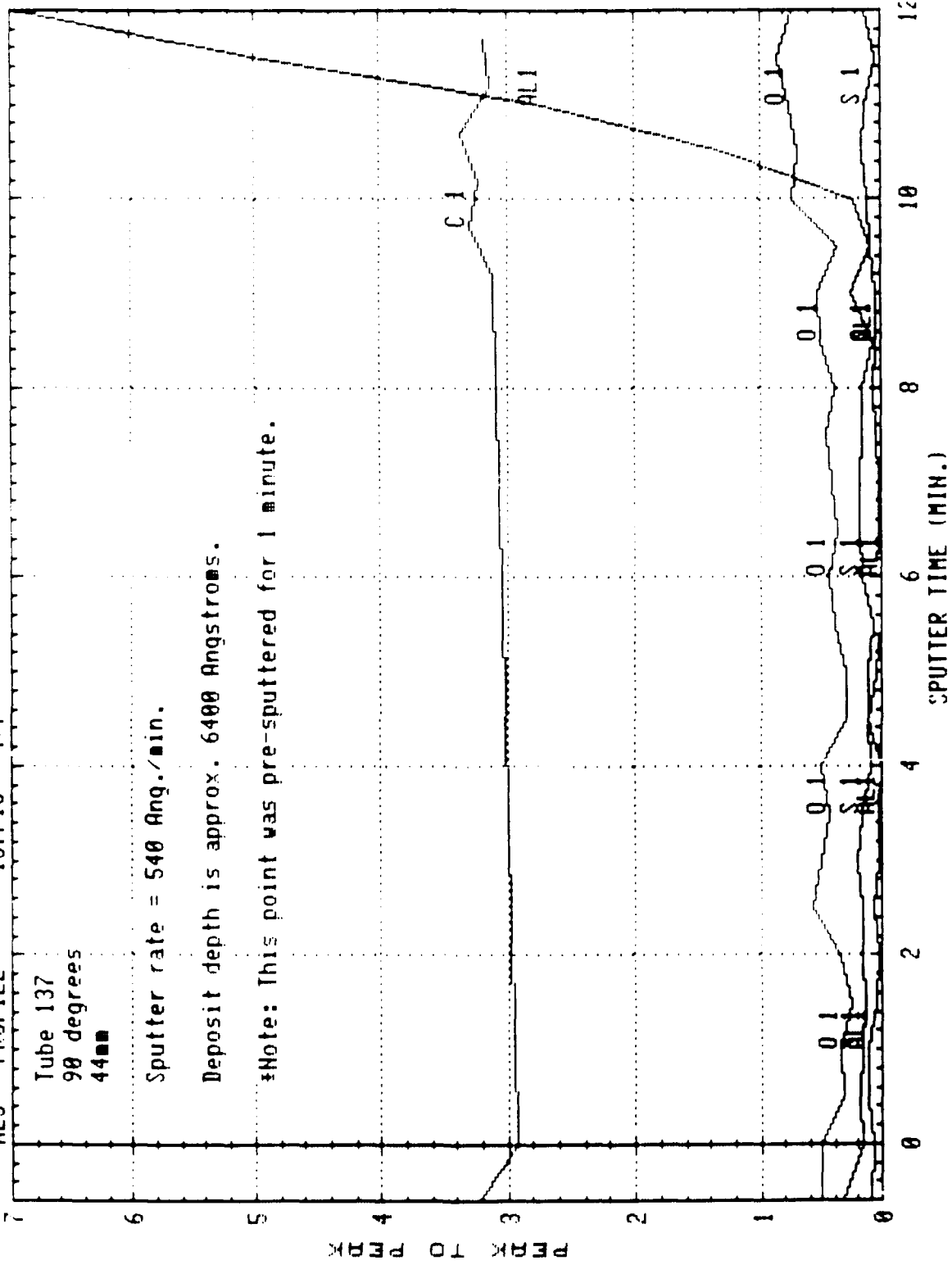
AES PROFILE 137P13 V/F

Tube 137
90 degrees
44mm

Sputter rate = 540 Ang./min.

Deposit depth is approx. 6400 Angstroms.

*Note: This point was pre-sputtered for 1 minute.



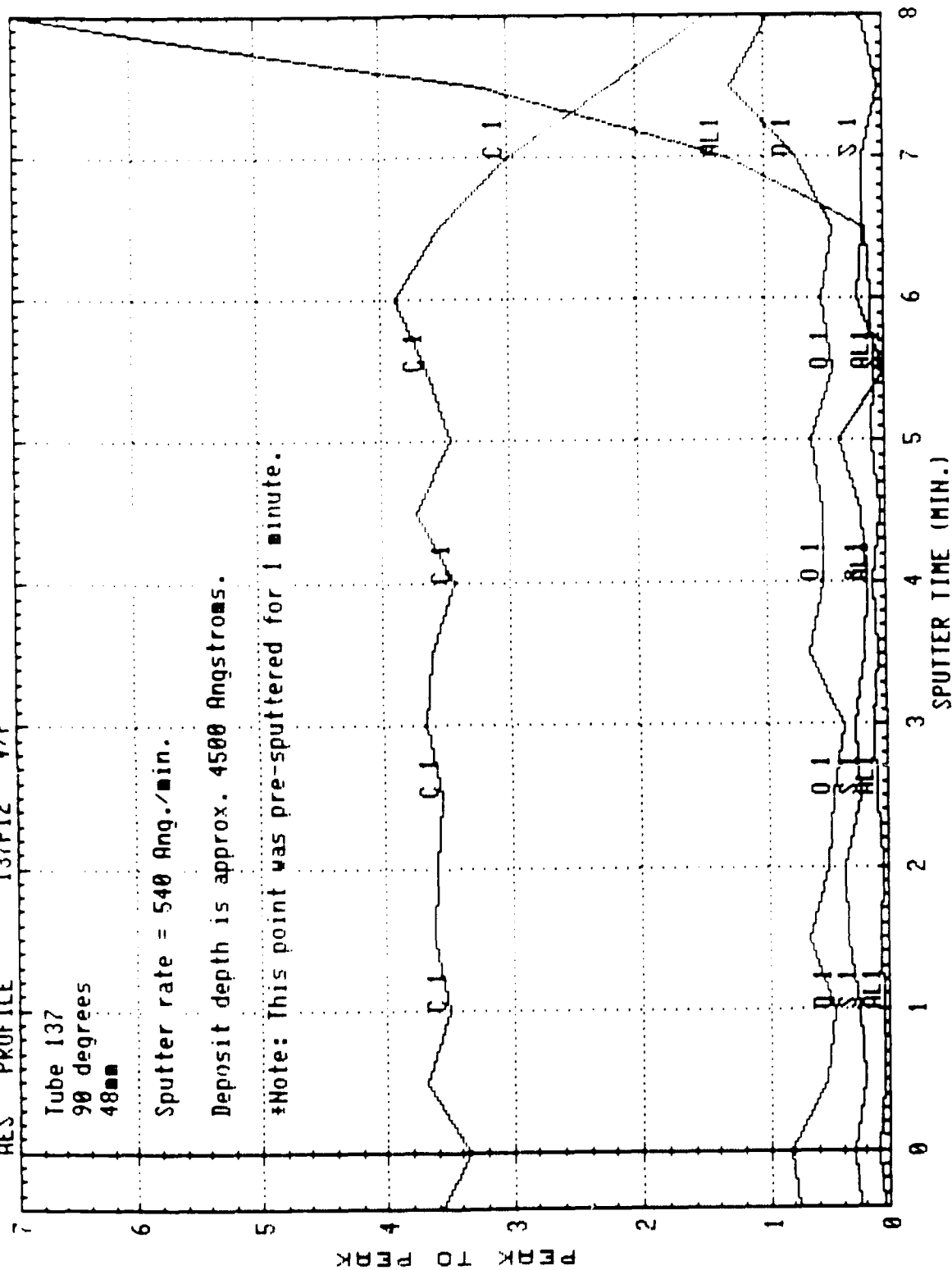
AES PROFILE 137P12 V/F

Tube 137
90 degrees
48mm

Sputter rate = 540 Ang./min.

Deposit depth is approx. 4500 Angstroms.

*Note: This point was pre-sputtered for 1 minute.



AES PROFILE 137P11 V/F

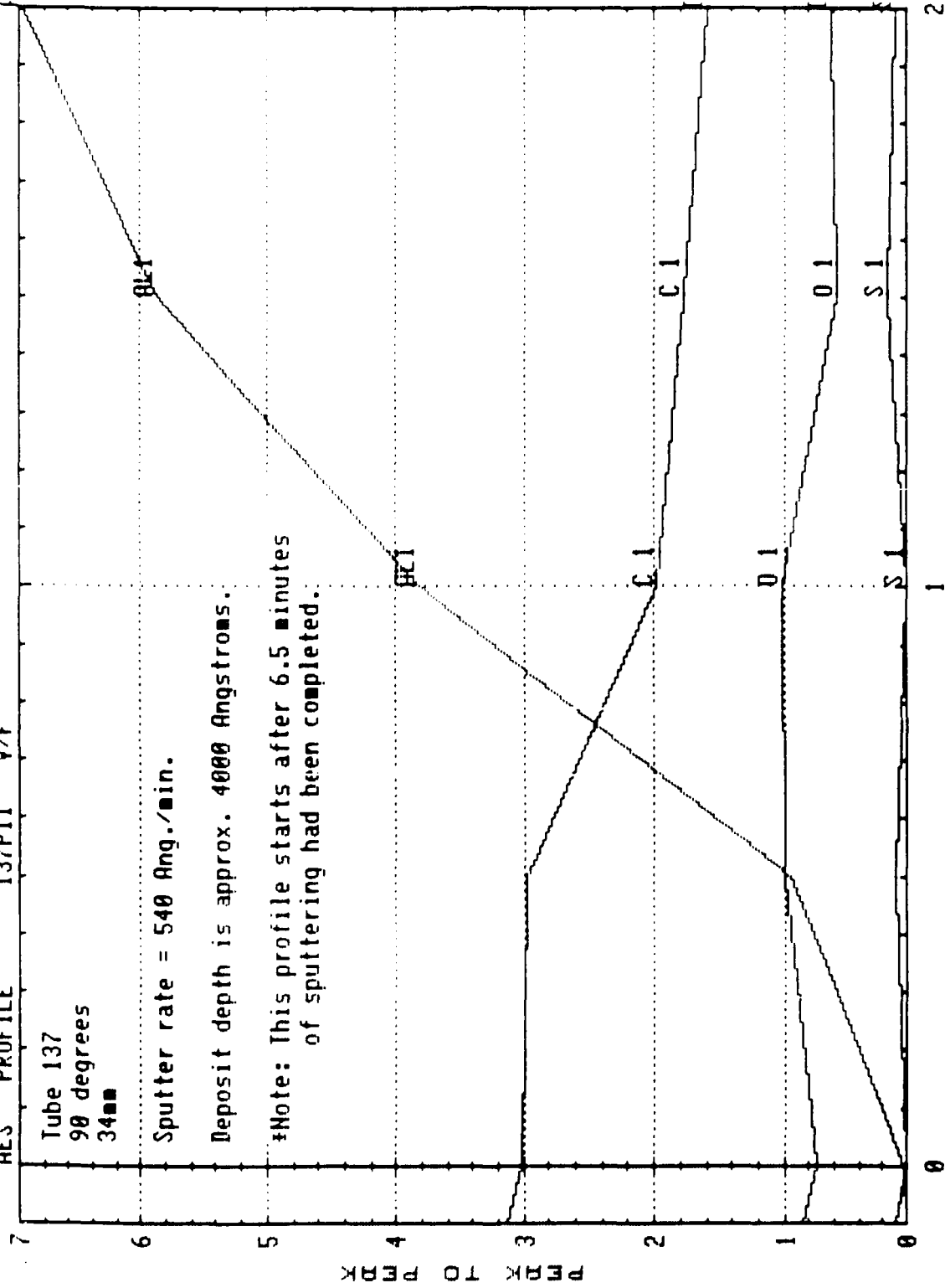
Tube 137
90 degrees
34mm

Sputter rate = 540 Ang./min.

Deposit depth is approx. 4000 Angstroms.

Note: This profile starts after 6.5 minutes
of sputtering had been completed.

PERK TO PERK



2

AES PROFILE 137P10 V/F

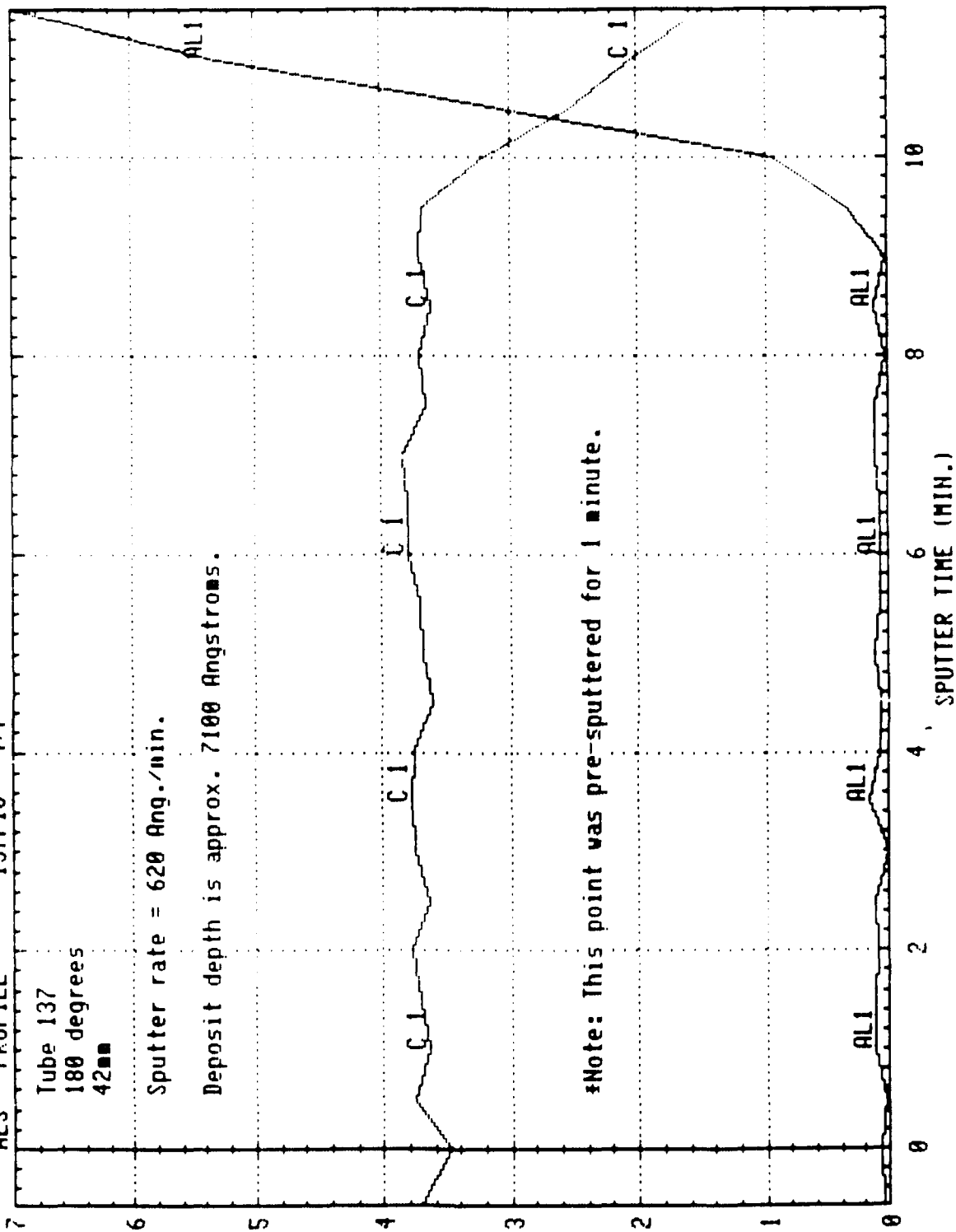
Tube 137
180 degrees
42mm

Sputter rate = 620 Ang./min.

Deposit depth is approx. 7100 Angstroms.

PERK TO PERK

*Note: This point was pre-sputtered for 1 minute.



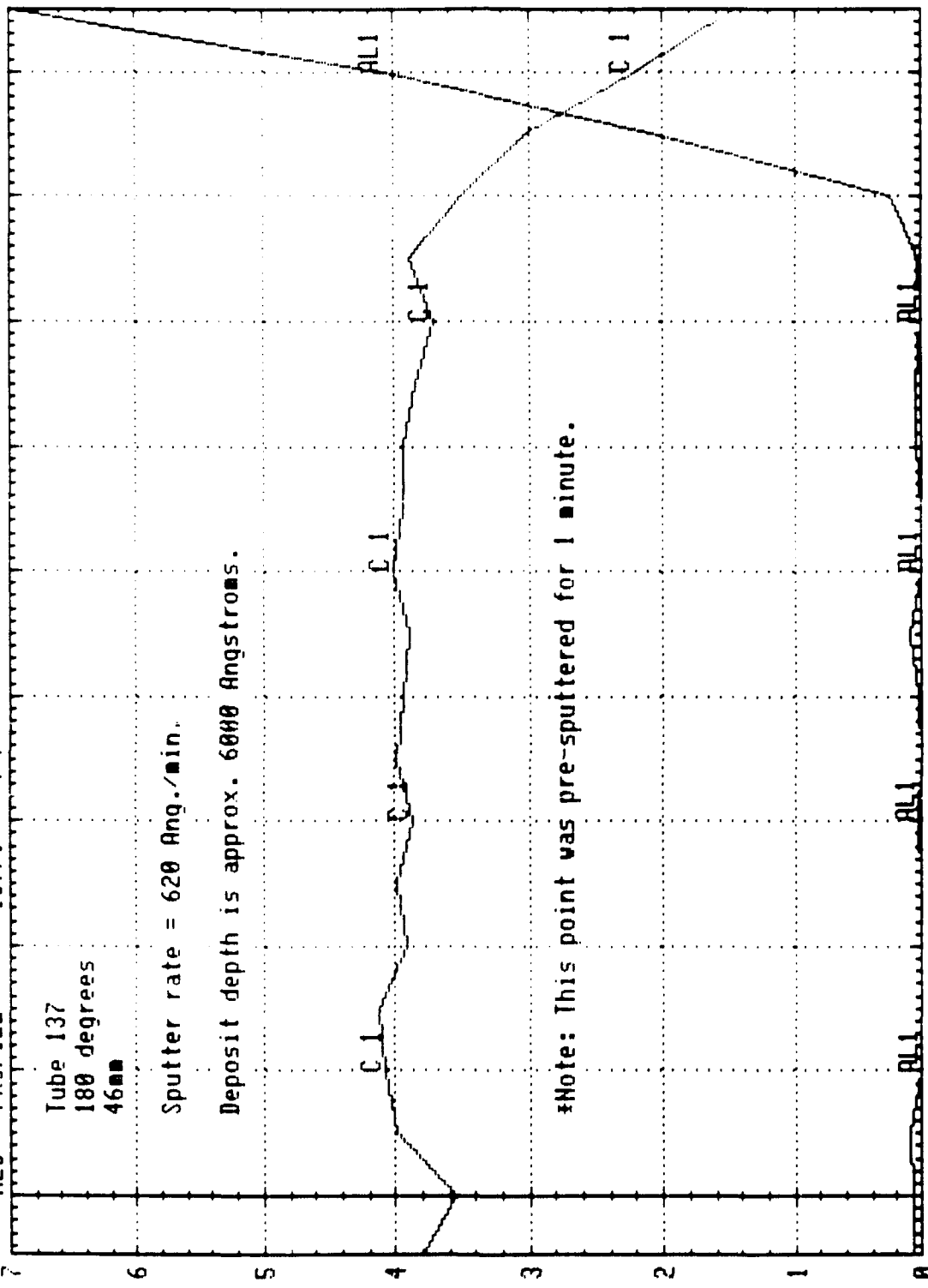
AES PROFILE 137P9 Y/F

Tube 137
180 degrees
46mm

Sputter rate = 620 Ang./min.

Deposit depth is approx. 6000 Angstroms.

PEAK TO PEAK



*Note: This point was pre-sputtered for 1 minute.

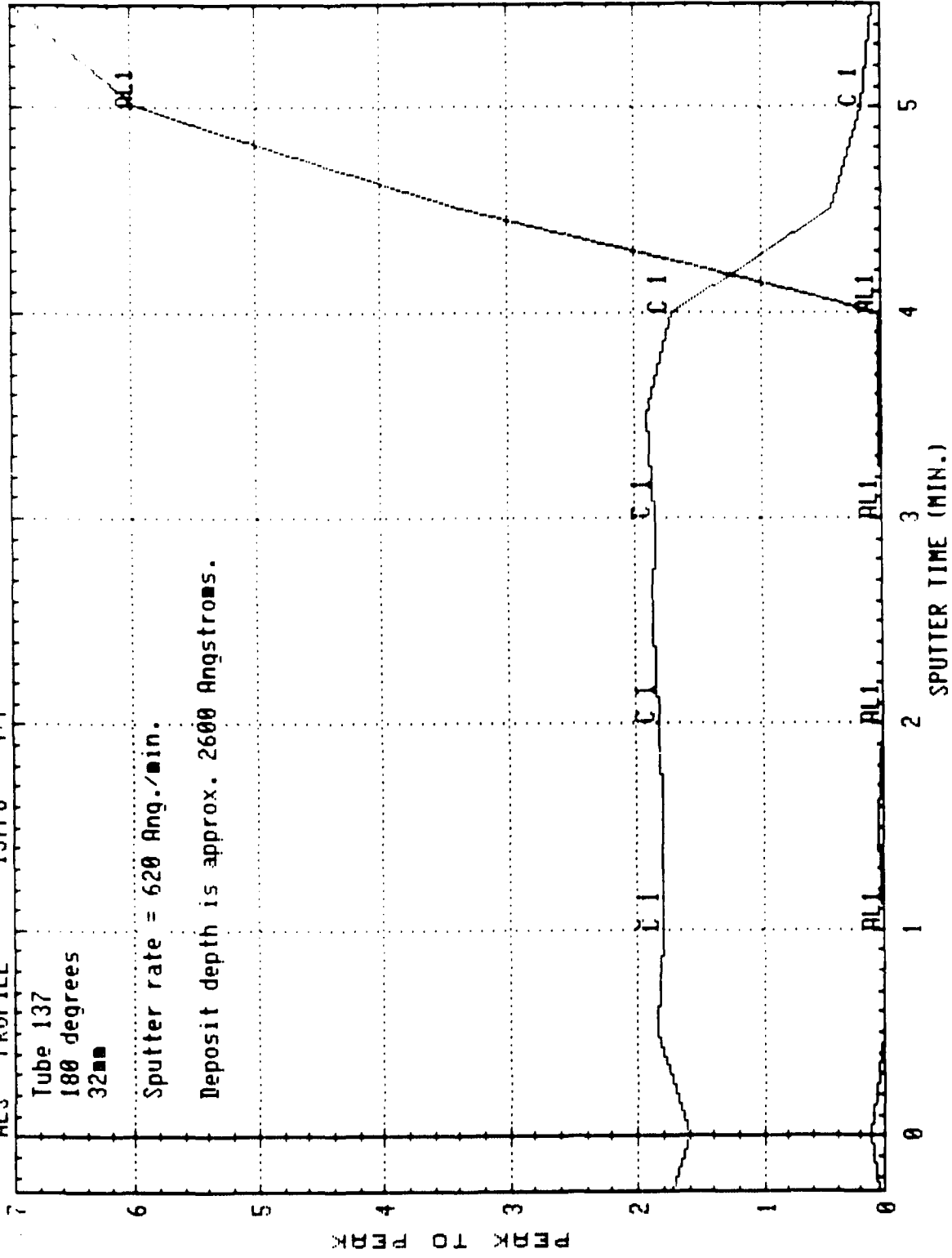
SPUTTER TIME (MIN.)

AES PROFILE 137P8 V/F

Tube 137
180 degrees
32mm

Sputter rate = 620 Ang./min.

Deposit depth is approx. 2600 Angstroms.



AES PROFILE 137P7 V/F

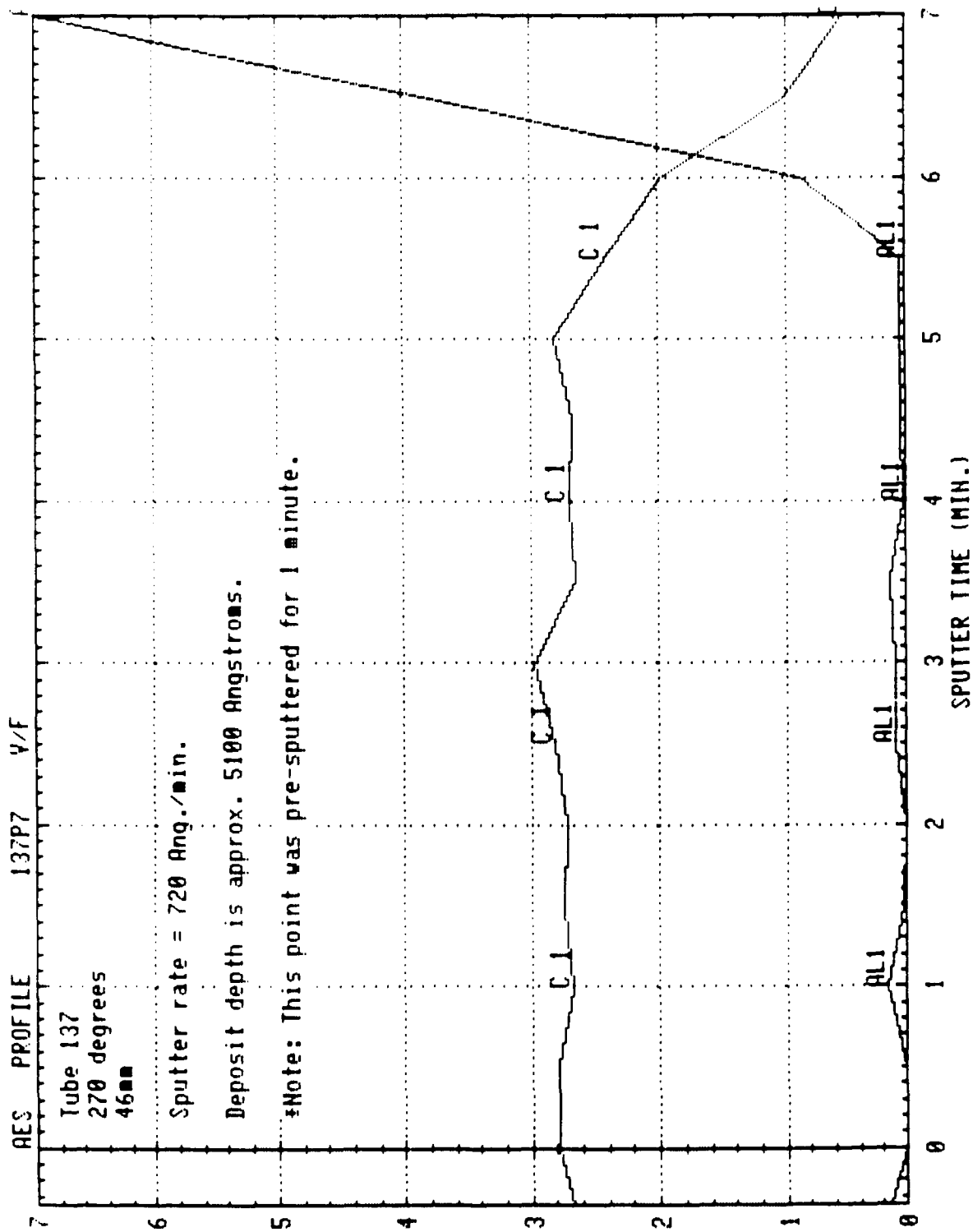
Tube 137
270 degrees
46mm

Sputter rate = 720 Ang./min.

Deposit depth is approx. 5100 Angstroms.

*Note: This point was pre-sputtered for 1 minute.

PEAK TO PEAK



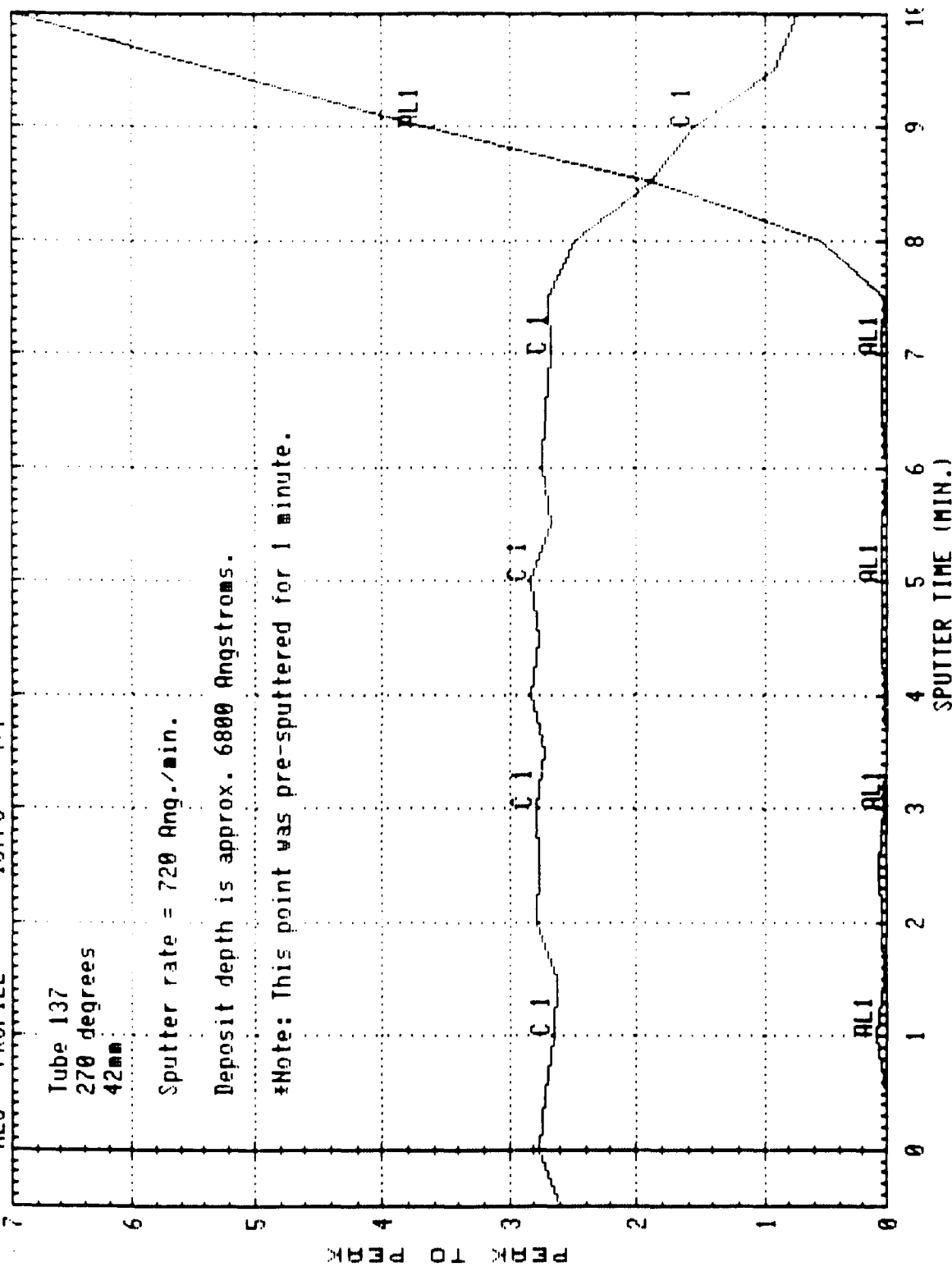
AES PROFILE 137P6 V/F

Tube 137
270 degrees
42mm

Sputter rate = 720 Ang./min.

Deposit depth is approx. 6800 Angstroms.

*Note: This point was pre-sputtered for 1 minute.



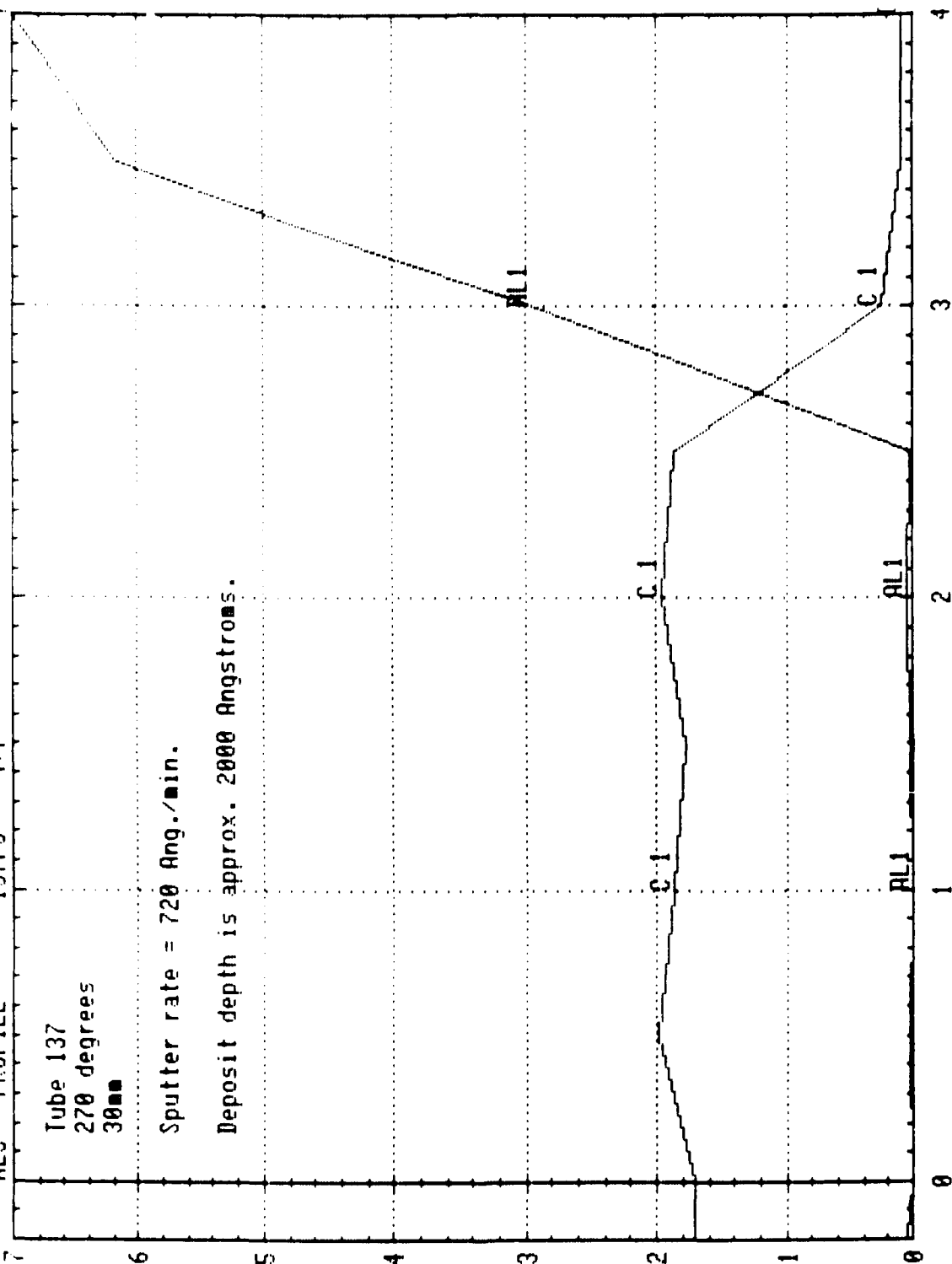
AES PROFILE 137P5 V/F

Tube 137
270 degrees
30mm

Sputter rate = 720 Ang./min.

Deposit depth is approx. 2000 Angstroms.

PERK TO PERK



SPUTTER TIME (MIN.)