NBSIR 80-2156 (FDA)

Surface Analyses of Medical Lens Implants By X-Ray Photoelectron Spectroscopy

Terrence J. Jach

Surface Science Division
Center for Thermodynamics
Molecular Science
National Bureau of Standards
U.S. Department of Commerce
Washington, DC 20234

September 1980

Final

Medical Devices Laboratory
Food and Drug Administration
Silver Spring, MD 20910

Surface Analyses of Medical Lens Implants by X-Ray Photoelectron Spectroscopy

Terrence Jach

Surface Science Division

National Bureau of Standards

CONTENTS

INTRODUCTION

A number of manufacturers presently offer plastic lenses as surgical implants to replace human eye lenses which are damaged or imperfect. The compatability and safety of these lenses with human tissue is dependent, in part, on their surface composition. It is therefore desirable to examine these lenses by a method which is sensitive to elements in the outermost few atomic layers of the surface. It is possible that due to contamination or a manufacturing procedure, a lens which shows low levels of a toxic element in bulk analysis may have a high concentration of that element a few atoms thick at the surface.

We have examined, using the surface-sensitive technique of X-Ray Photoemission Spectroscopy (XPS), individual samples of thirteen different lenses supplied by manufacturers to the Federal Drug Administration. The purpose of the study was to look for elements which were not ordinarily expected to be present in the polymer. The surface of each lens was analyzed twice: first, as it came from the package, and second, after removing the outermost layer of surface material by a brief period of ion sputtering. To within the limits of elemental detectability which will be outlined below, only two samples showed any elements other than carbon and oxygen.

METHOD

The lenses supplied by the FDA consisted of one each of thirteen different types produced by eight different manufacturers. A list of the lenses is included as Appendix A. It was assumed that each lens was precleaned by the manufacturer to whatever extent was considered adequate before sterilization. Each lens was removed from its sterile package using cleaned forceps and mounted directly on a sample carousel in the XPS chamber.

The chamber was pumped down to an ultimate pressure of 10^{-9} torr (10^{-7} Pa) before measurement.

Using a Physical Electronics[1] 04-151 X-Ray Tube with Mg anode and a model 15-255 Analyzer, photoemission spectra were taken over a binding energy range of 0-1000 eV. The photoelectron pulses were integrated for 1 sec and the spectrum advanced at 1 ev/s. Minimum elemental concentrations of 3% (atomic) were visible. Due to the low photocurrents involved, charging effects on the highly-insulating lenses were negligible.

Two XPS spectra were taken of each sample lens. The first was made of each lens without any surface treatment. The second spectrum was taken after 5 minutes of sputtering the lens with an argon-ion beam from a Physical Electronics[1] 04-161 Sputter Ion Gun.

The ion beam removed the surface constituents of the sample by bombardment. Although the sputter rate is highly dependent on the nature of the sample, a rough estimate of the sputter rate was 10 Å/minute (1nm/min). It was assumed that removal of 50 Å (5nm) of surface was adequate to get beneath water and organic matter which may have covered the surface after manufacturing. The depth of the surface layer removed was small enough to insure that any residual surface contamination from molding dies or the chemical process of lens manufacture should still be visible.

The spectra of the lenses were calibrated with respect to nickel and gold targets on the carousel which extablished peak positions to within 0.5eV. The circular area viewed by the analyzer varied between 1 and 3mm in diameter. The area sampled was large enough compared to the lens diameter that some slight features in the background of lens spectra may have been due to elements on the sample carousel at the periphery of the sampled area. Where this was a question, repeat spectra were taken with the sample slightly displaced. Only those elements which showed up consistently in the repeat spectra are cited here.

Positive identification of each element was made only after direct correlation of more than one spectral line with lines for that element in published spectra.[2]

RESULTS

In the first set of lens measurements (samples unsputtered) the spectra all showed large peaks due to carbon and oxygen, as might be expected of hydrocarbon samples. The ratio of carbon to oxygen signals for each lens is tabulated in Appendix B. In addition, sample No. 9 (the Intermedics lens) showed a small trace of chlorine before sputtering.

After 5 minutes of sputtering, the ratio of carbon to oxygen changed. Generally the oxygen peak decreased to about 30% of its pre-sputtered value, and the carbon peak increased by about 50%. The ratios of carbon to oxygen signals for each lens is tabulated in Appendix B. Sample No. 12(Iolab lens) showed a slight trace of nitrogen on the surface after sputtering.

These net changes indicate that the oxygen present on the original surface was probably a contamination (e.g., H_2O) or oxidation of the plastic.

Summary

We have obtained X-Ray Photoemission Spectra on the surfaces of 13 different implantable plastic eye lenses to study elemental constituents which occur on the surfaces as a result of manufacturing or handling. The spectra were taken on the surfaces as removed from their packaging and then again after sputtering to remove about 50 Å (5nm) of surface material. All lenses showed strong carbon and oxygen peaks both before and after sputtering, which would be considered normal for a plastic surface.

No foreign elements were detected with concentrations greater than 3% before sputtering except for one lens which showed detectable amounts of chlorine. This peak disappeared after sputtering. On one lens a detectable amount of nitrogen appeared after sputtering.

References:

1. Commercial equipment is identified to specify the measurement conditions and is not intended as an endorsement of this manufacturer by the National Bureau of Standards.

2. C. D. Wagner, W. M. Riggs, L. E. Davis, J. F. Moulder, and G. E. Muilenberg, Handbook of X-Ray Photoelectron Spectroscopy (Perkin-Elmer, Physical Electronics Div., Eden Prairie, Minn., 1979)

Appendix A: Summary of Lenses Tested

Sample No. (Also Figure No.)	FDA Assigned No.
1. Precision-Cosmet Co. Inc. (Kelmann II) KL-5722-X-B Lot 922	80-59
2. Precision-Cosmet Co. Inc. (Azar II) AR-2156-X-A Lot 867	80-60
3. Precision-Cosmet Co. Inc. (Tennant) AC-43298-X-A	80-61
4. Coburn Optical, Professional Products Div. MK VIII (Choyce) Control No. 171905	80-65
5. Coburn Optical, Professional Products Div. MK IX (Choyce) Control No. 216101	80-64
6. Heyer Schulte Medical Optics Ctr. Model AC-10 Cat. No. 013-1160-115 Lens No. 10791991	80-56
7. Heyer Schulte Medical Optics Ctr. Model AC-20 Cat. No. 013-2175-120 Lens No. 11791173	80-62
8. California Intraocular Lens Co. Cilco AC4 Serial 504295 Lot A1492	80-63
9. Intermedics Intraocular Inc. Model 018 Serial No. 08472	80-53
10. Surgidev Corp. In-Troc Style 10 Cat. No. S7610195 Ser. No. 103481	80-54
11. McGhan Medical Corp. Implens 60 Cat. No. 65-66205 Serial No. S01-073583	80-55
12. Iolab Corp. Model 91 Control No. 062879 91-U 687	80-57
13. Iolab Corp. Model 91 Control No. 081479 91-50 260	80-58

Appendix B.

Sample	FDA No.	Ratios of XPS peak Amplitudes, $\frac{C(15)}{O(15)}$ Before sputtering	After sputtering
1	80-59	1.1	2.2
2	80-60	0.90	1.8
3	80-61	0.86	2.1
4	80-65	0.90	3.8
5	80-64	0.82	3.1
6	80-56	0.80	2.5
7	80-62	0.81	4.8
8	80-63	0.96	3.1
9	80-53	0.79	3.9
10	80-54	1.1	4.4
11	80-55	0.81	5.3
12	80-57	0.82	5.0
13	80-58	0.90	4.1

Note for all Figures

The following elemental lines appear in each x-ray photoelectron spectrum and are not individually labelled:

Element (line)	Energy Position (on Binding Energy Scale.)
C (1s)	283 eV
O (1s)	531 eV
O (KVV Auger)	745, 767 eV
C (KLL Auger)	990 eV

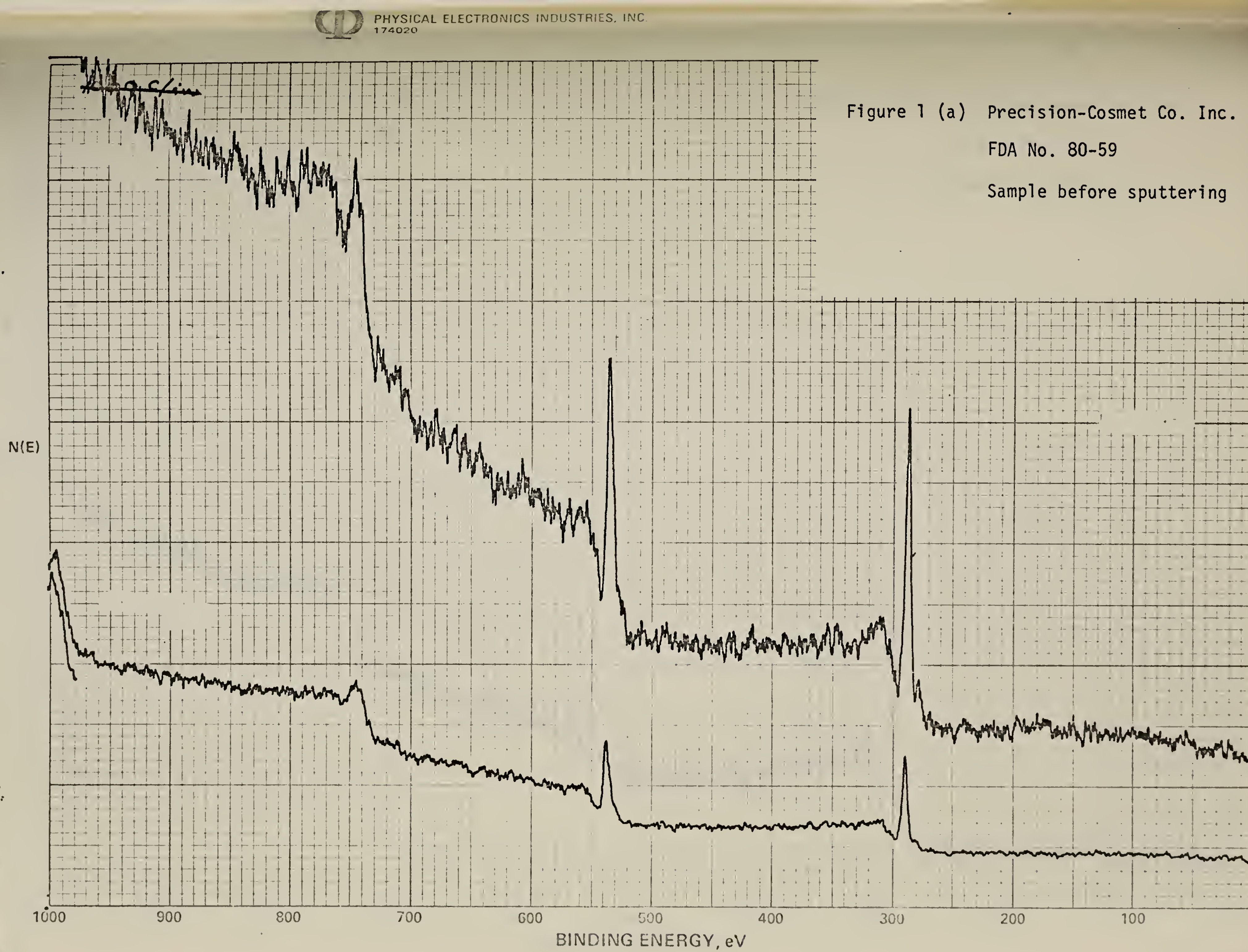


Figure 1 (a) Precision-Cosmet Co. Inc.

FDA No. 80-59

Sample before sputtering

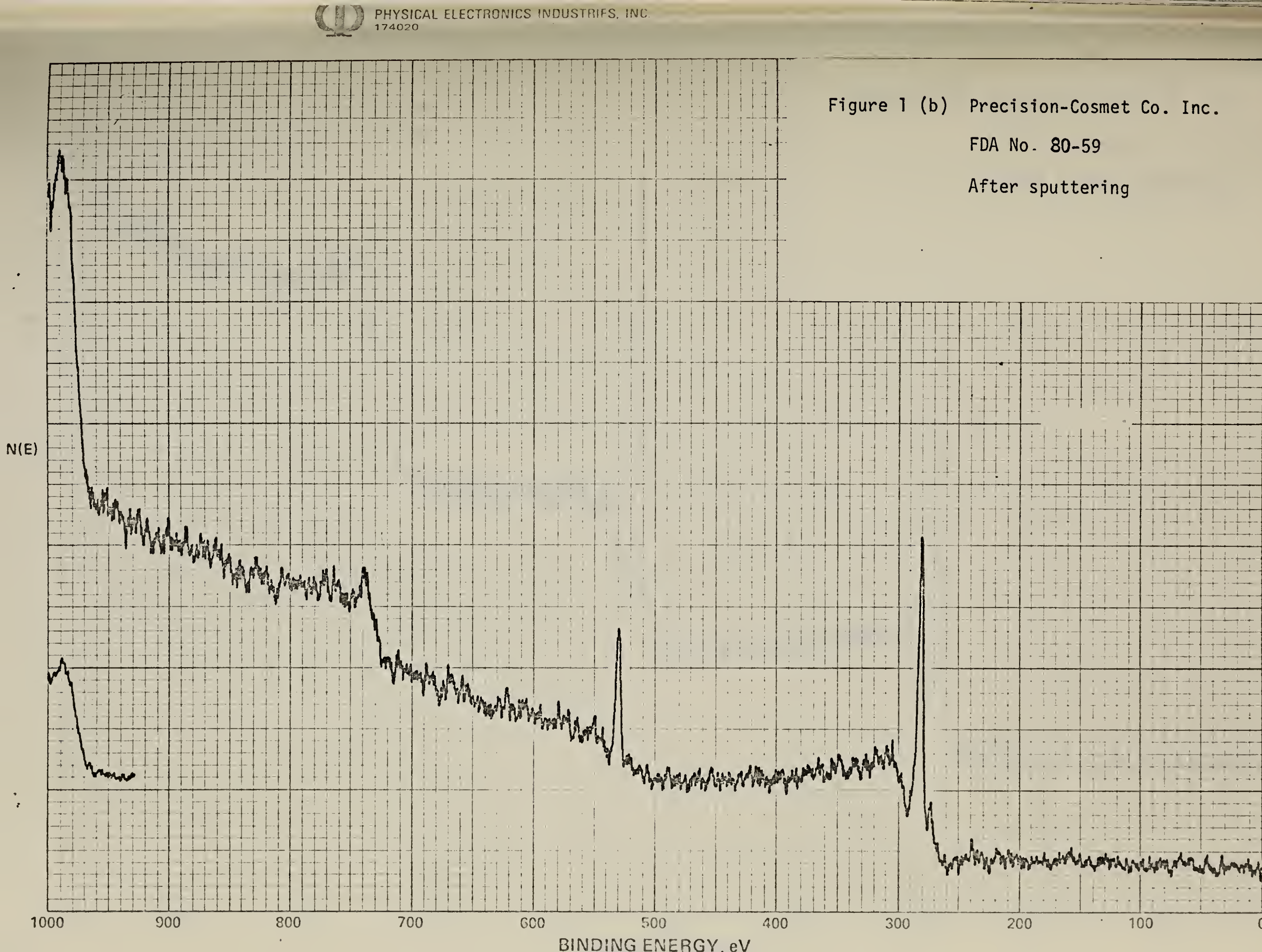


Figure 1 (b) Precision-Cosmet Co. Inc.

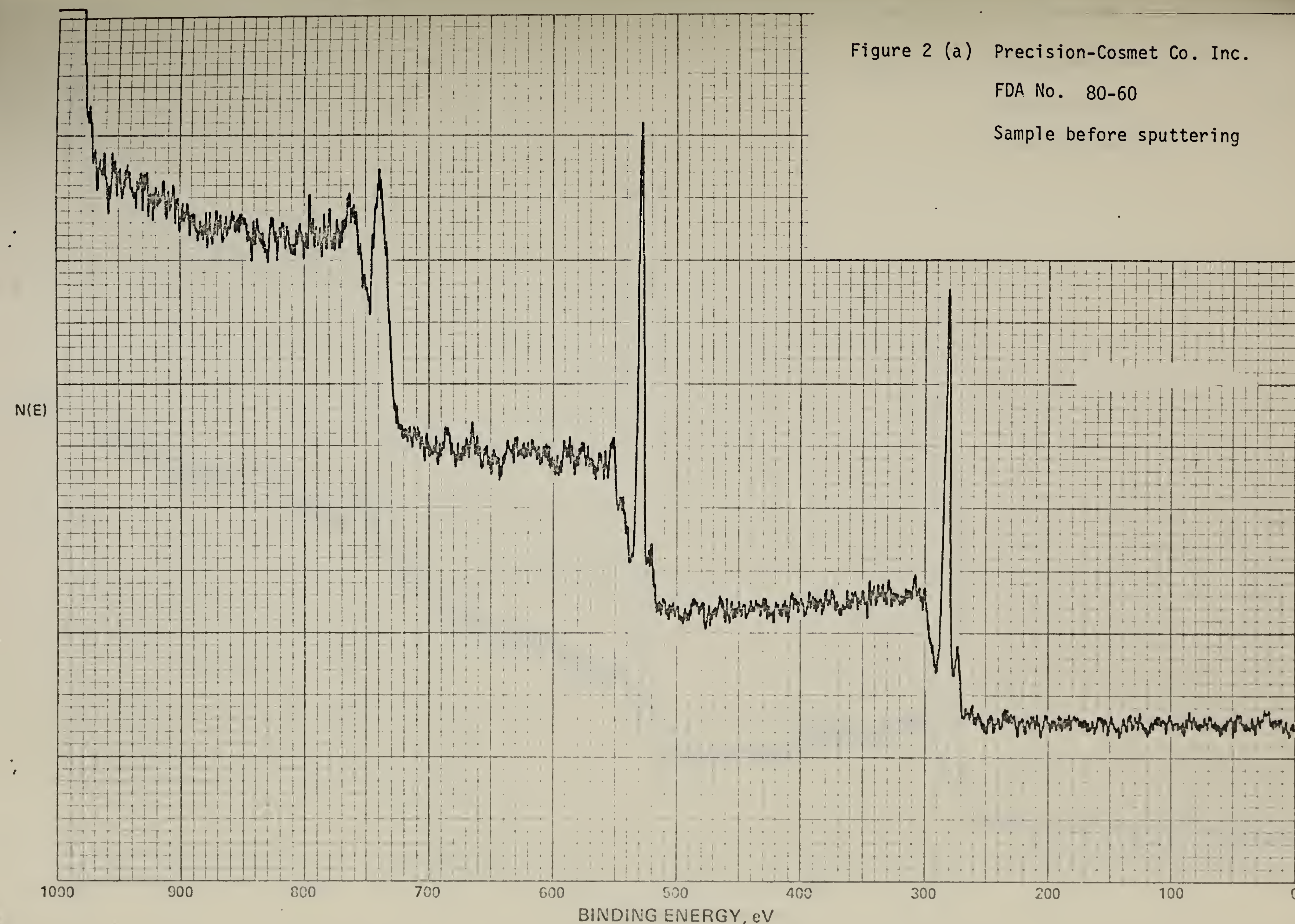
FDA No. 80-59

After sputtering

Figure 2 (a) Precision-Cosmet Co. Inc.

FDA No. 80-60

Sample before sputtering



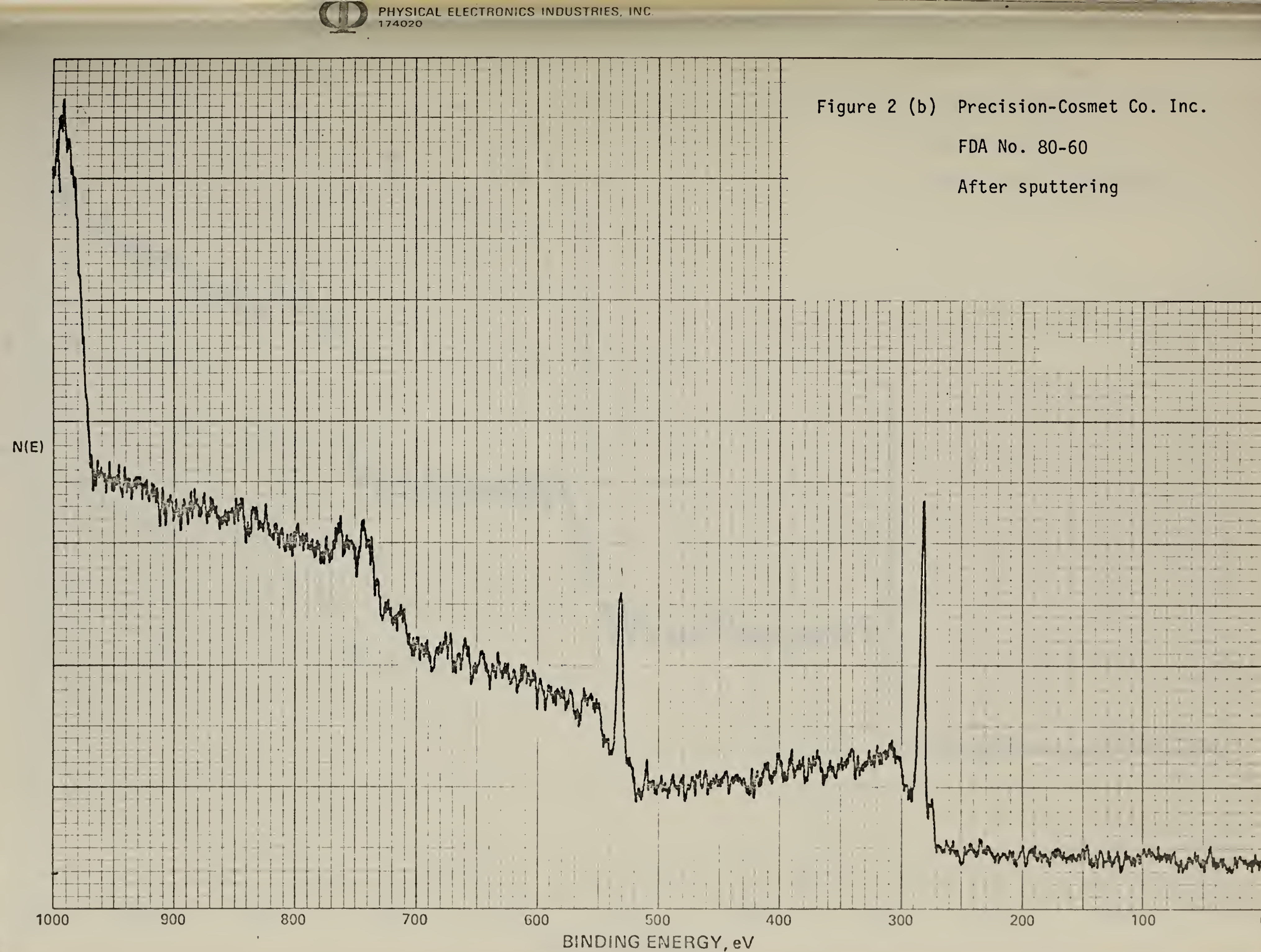
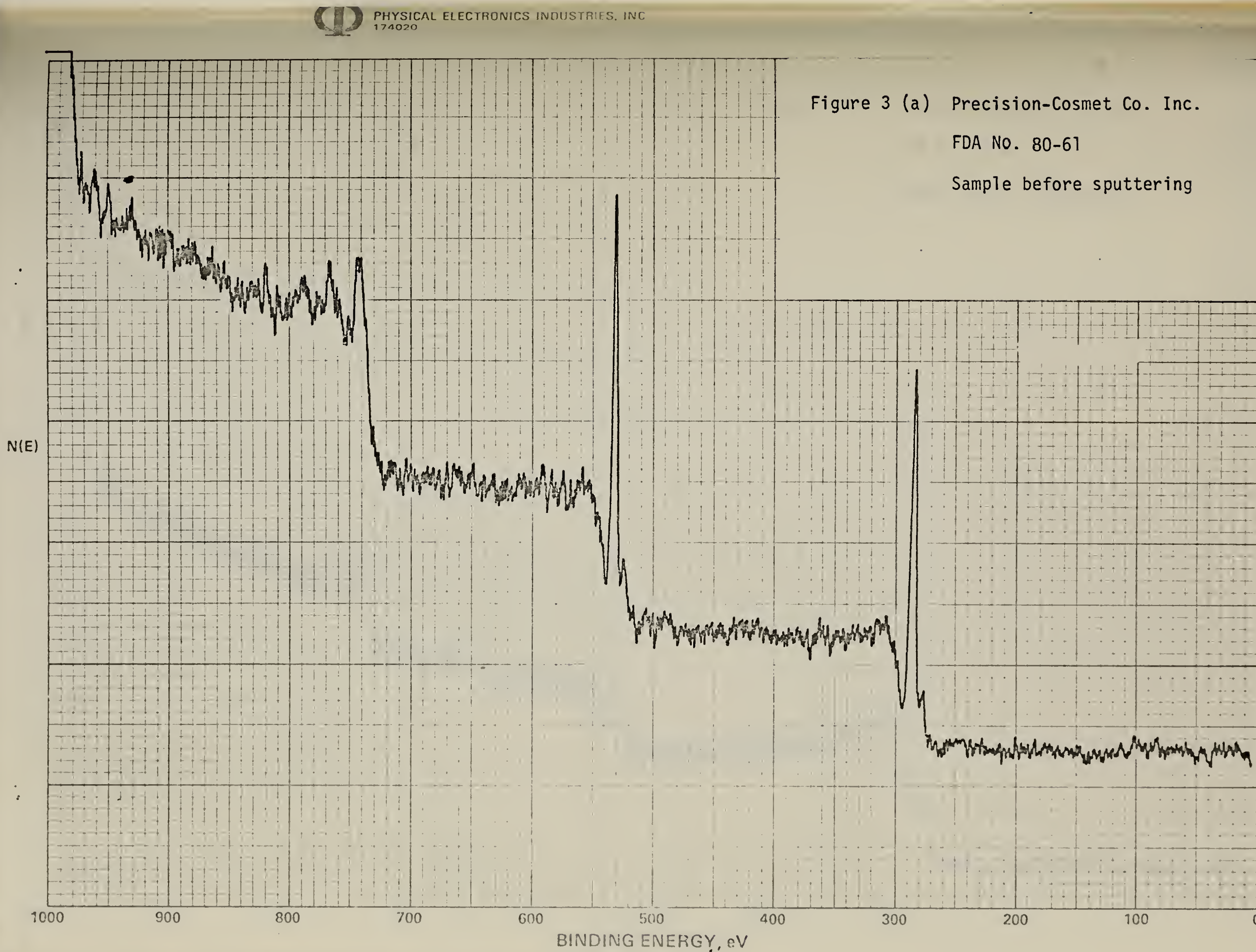

PHYSICAL ELECTRONICS INDUSTRIES, INC.
174020
Figure 2 (b) Precision-Cosmet Co. Inc.
FDA No. 80-60
After sputtering
N(E)
1000
900
800
700
600
500
400
300
200
100
0
BINDING ENERGY, eV

Figure 3 (a) Precision-Cosmet Co. Inc.

FDA No. 80-61

Sample before sputtering



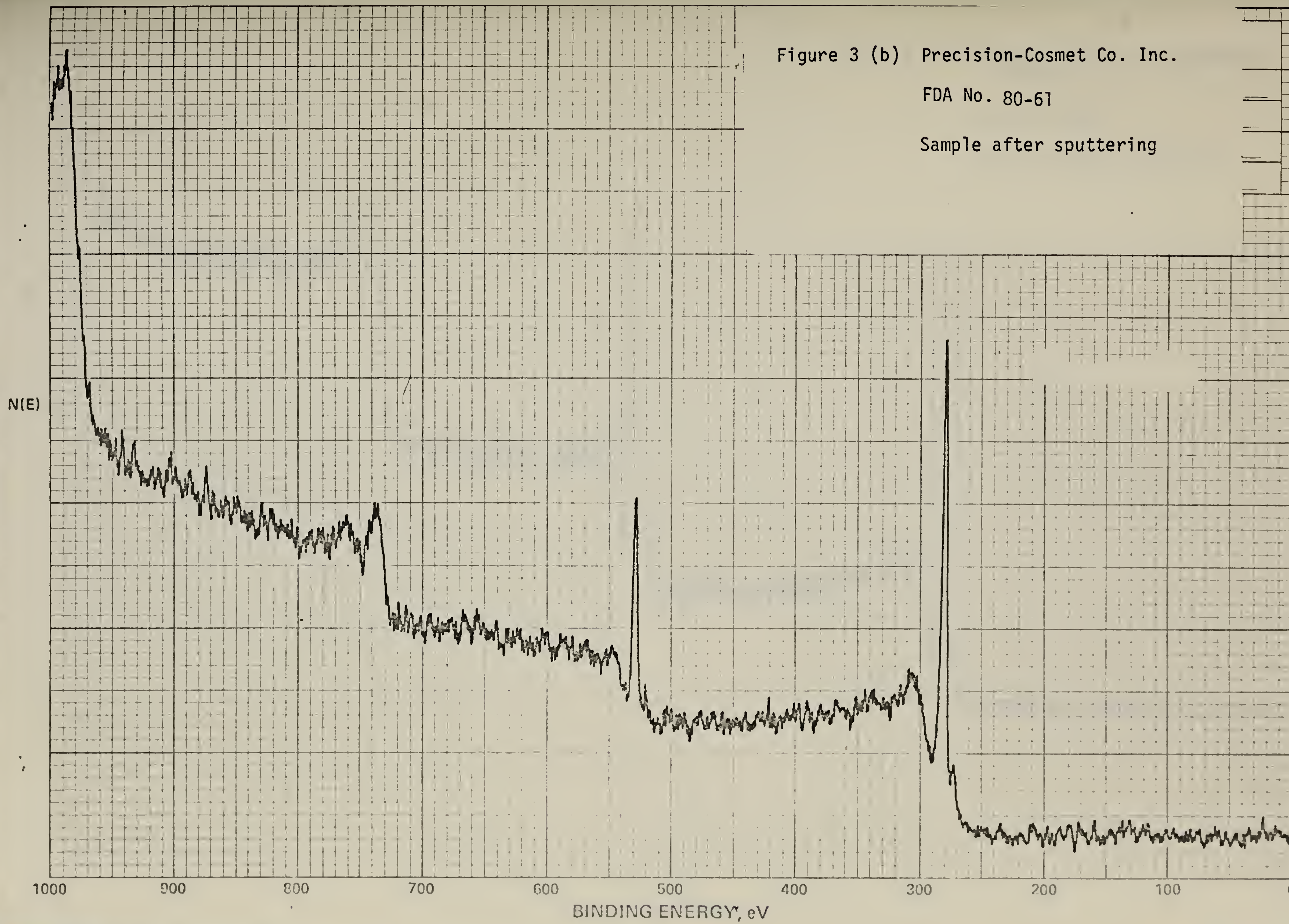


Figure 3 (b) Precision-Cosmet Co. Inc.

FDA No. 80-61

Sample after sputtering

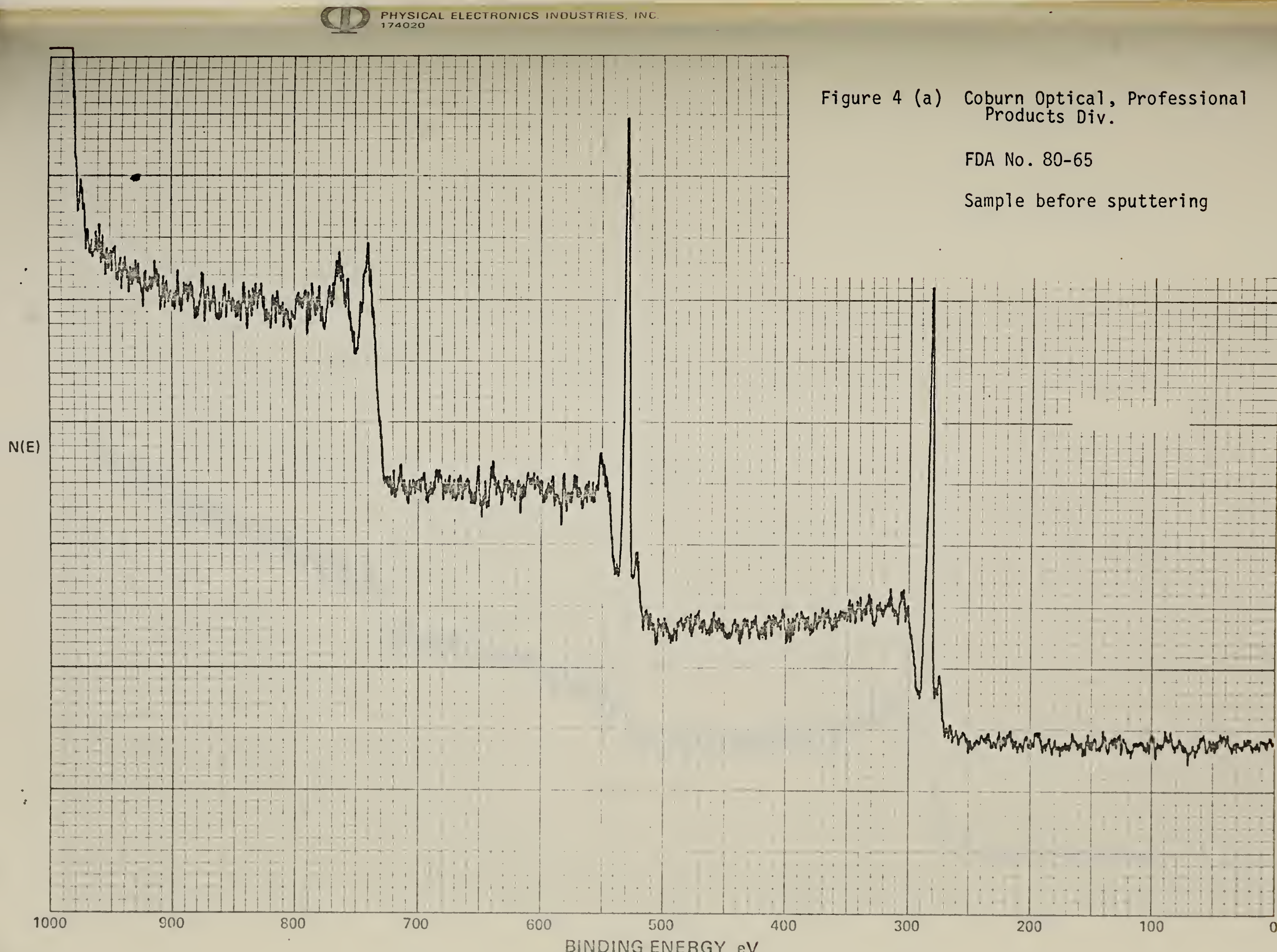


Figure 4 (a) Coburn Optical, Professional Products Div.

FDA No. 80-65

Sample before sputtering

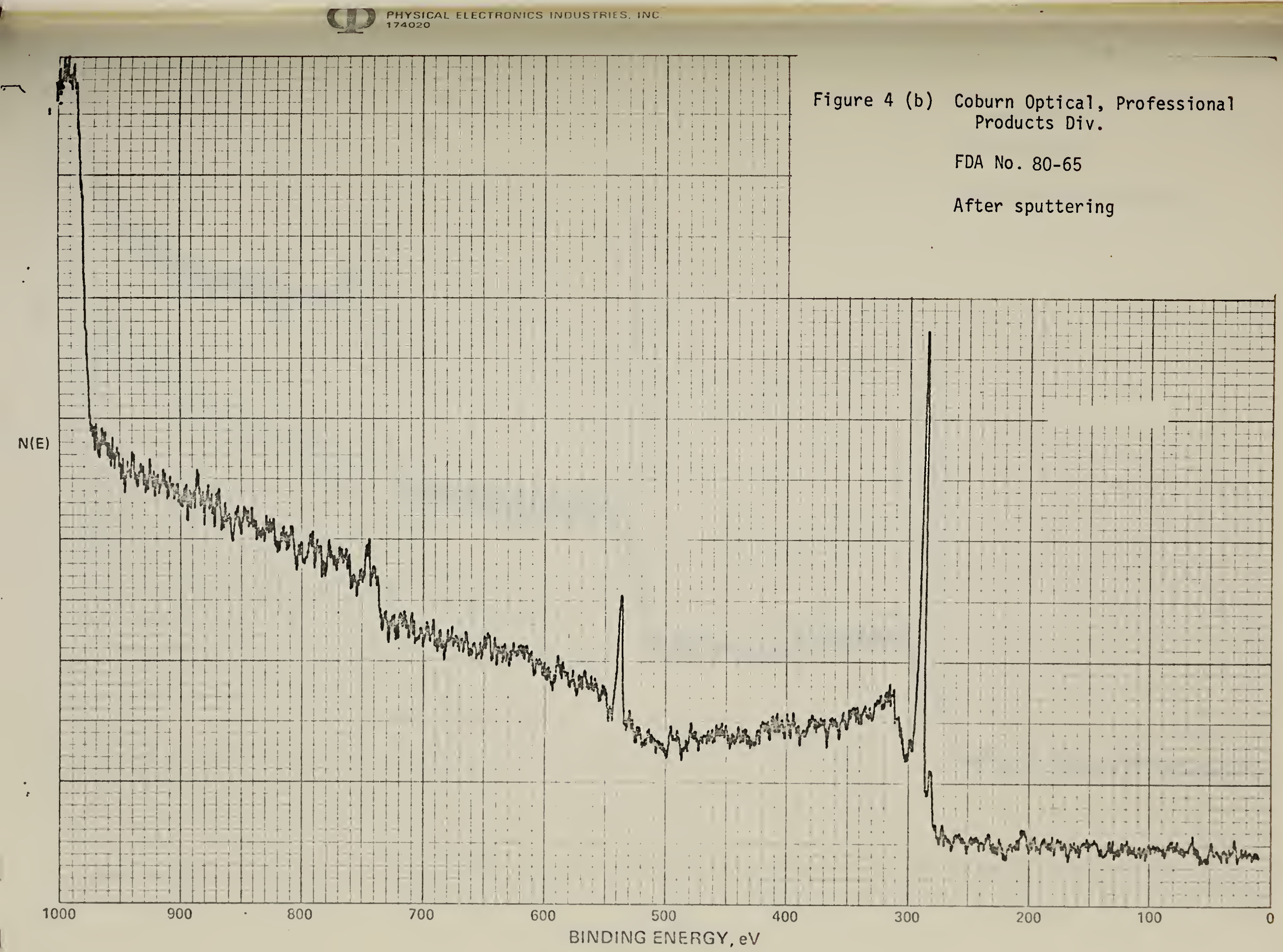


Figure 4 (b) Coburn Optical, Professional Products Div.

FDA No. 80-65

After sputtering

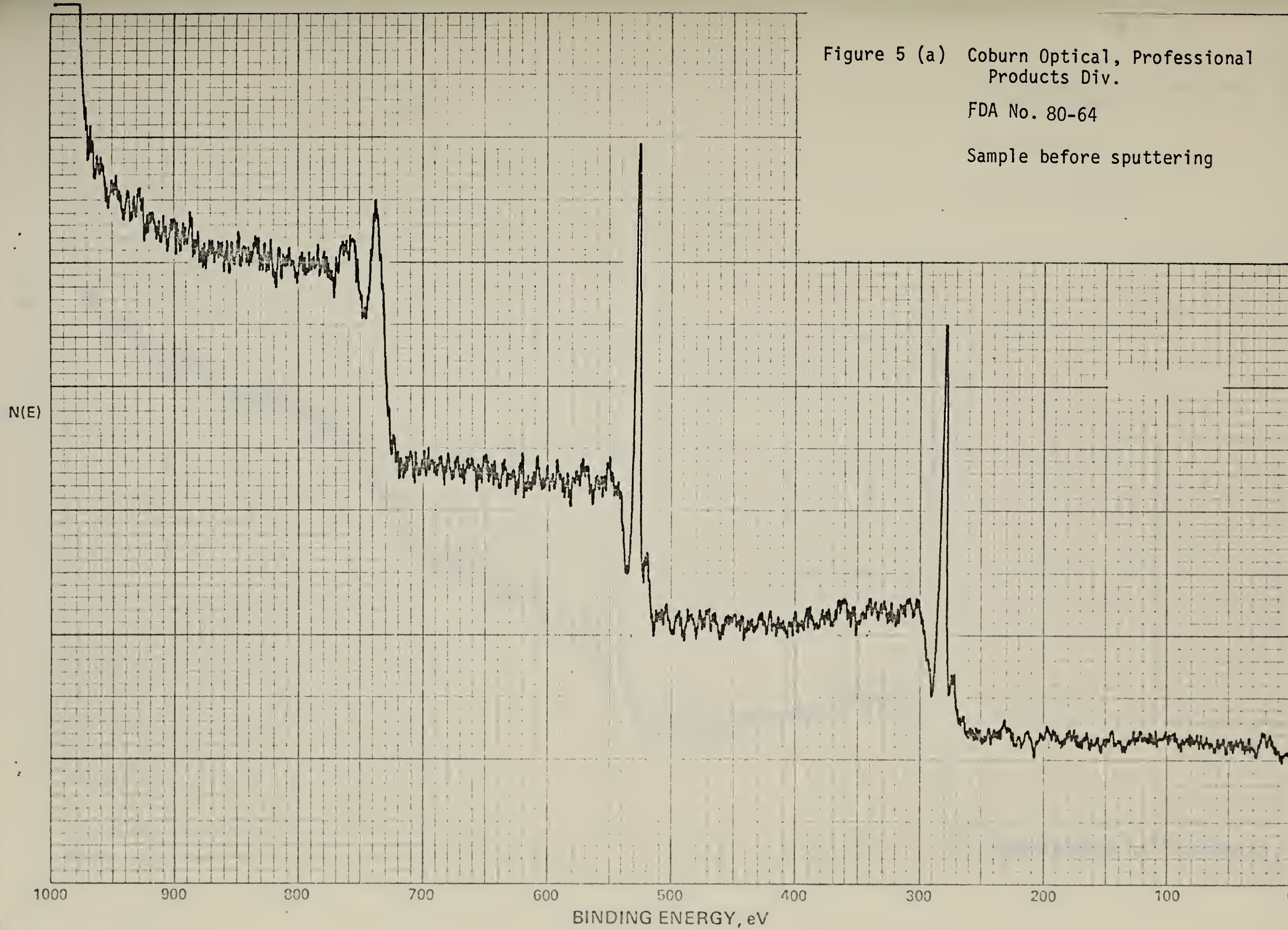


Figure 5 (a) Coburn Optical, Professional Products Div.

FDA No. 80-64

Sample before sputtering

Figure 5 (b) Coburn Optical, Professional Products Div.

FDA No. 80-64

After sputtering

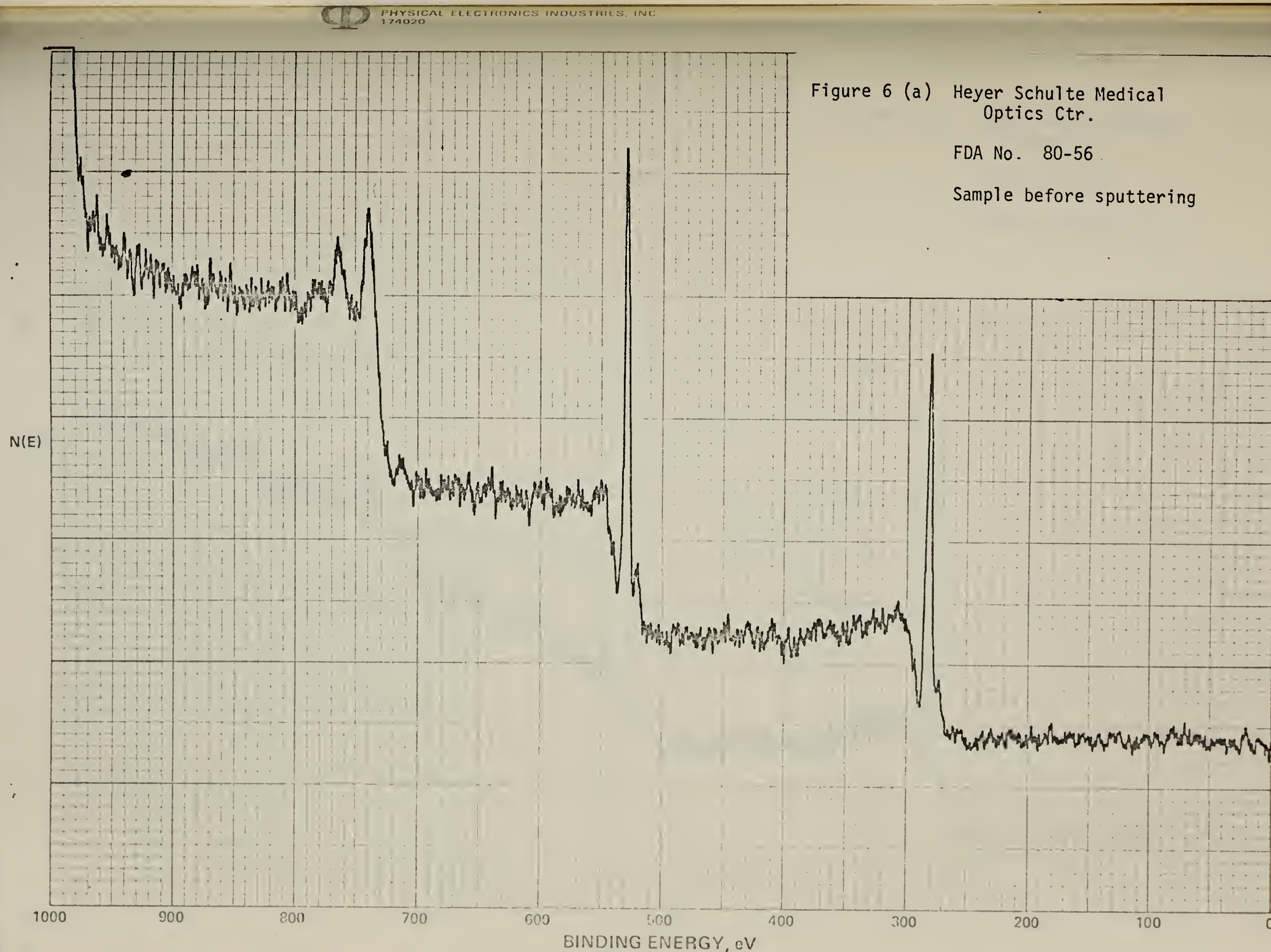


Figure 6 (a) Heyer Schulte Medical Optics Ctr.

FDA No. 80-56

Sample before sputtering

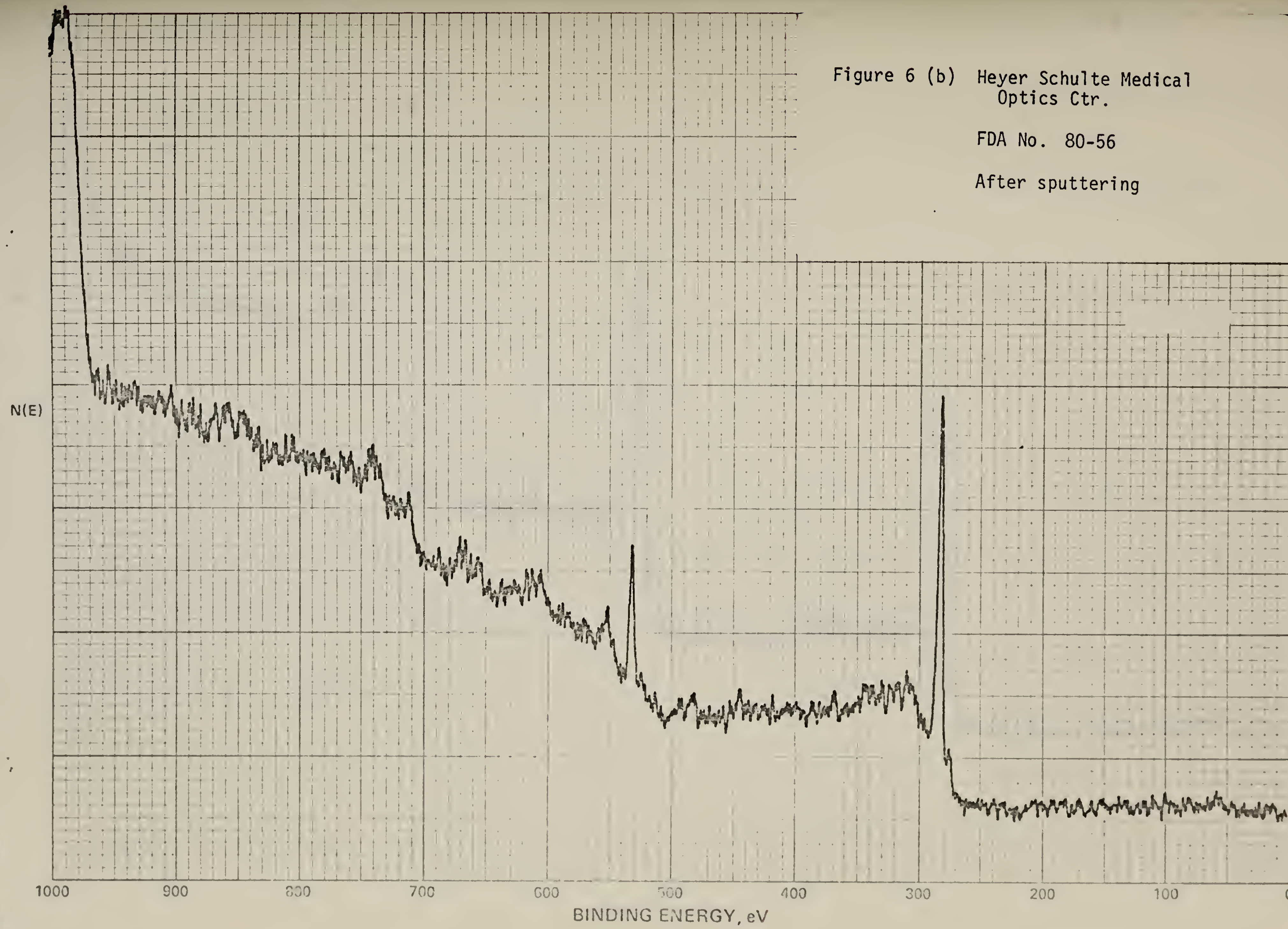


Figure 6 (b) Heyer Schulte Medical Optics Ctr.

FDA No. 80-56

After sputtering

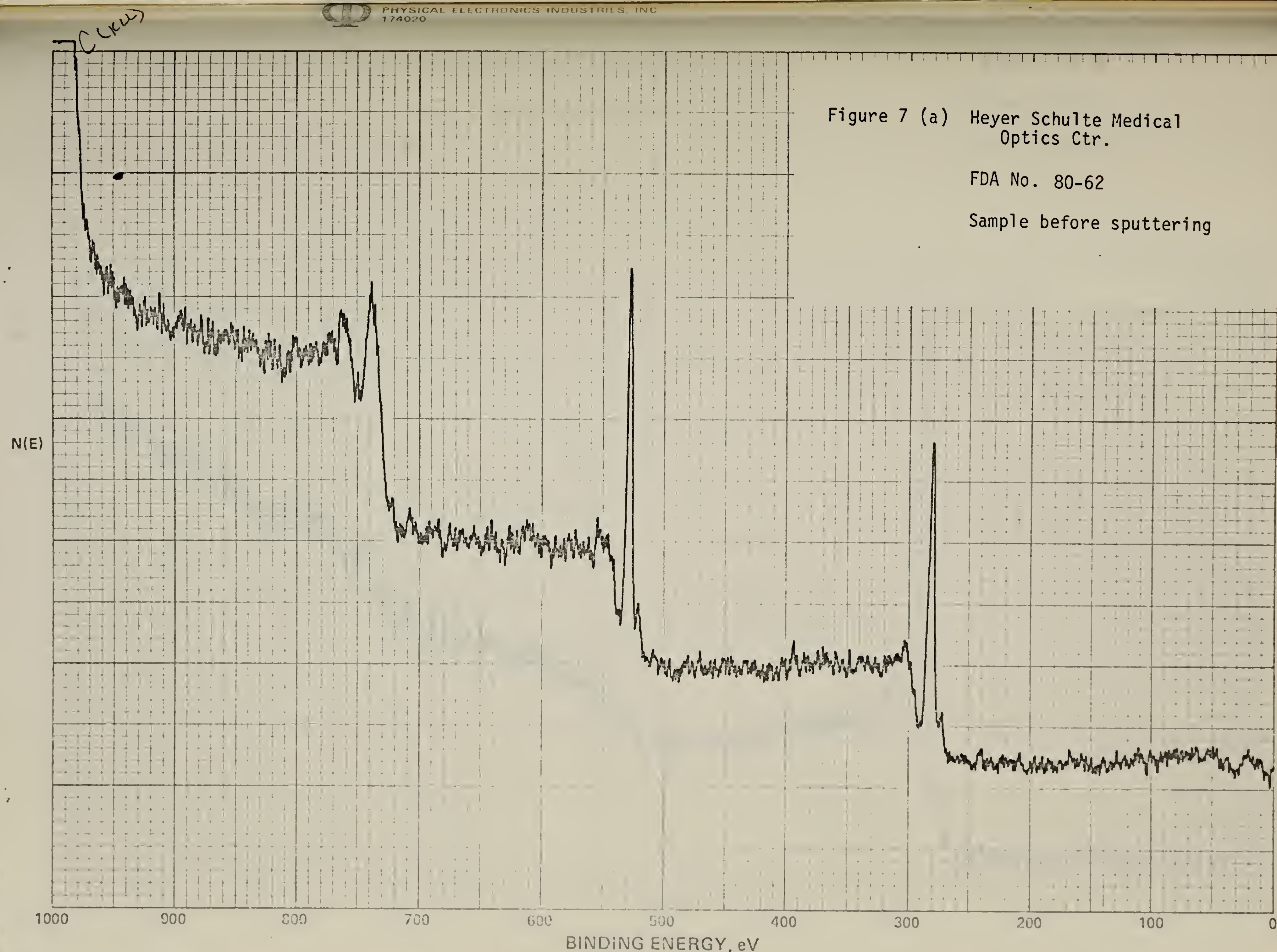


Figure 7 (a) Heyer Schulte Medical Optics Ctr.

FDA No. 80-62

Sample before sputtering

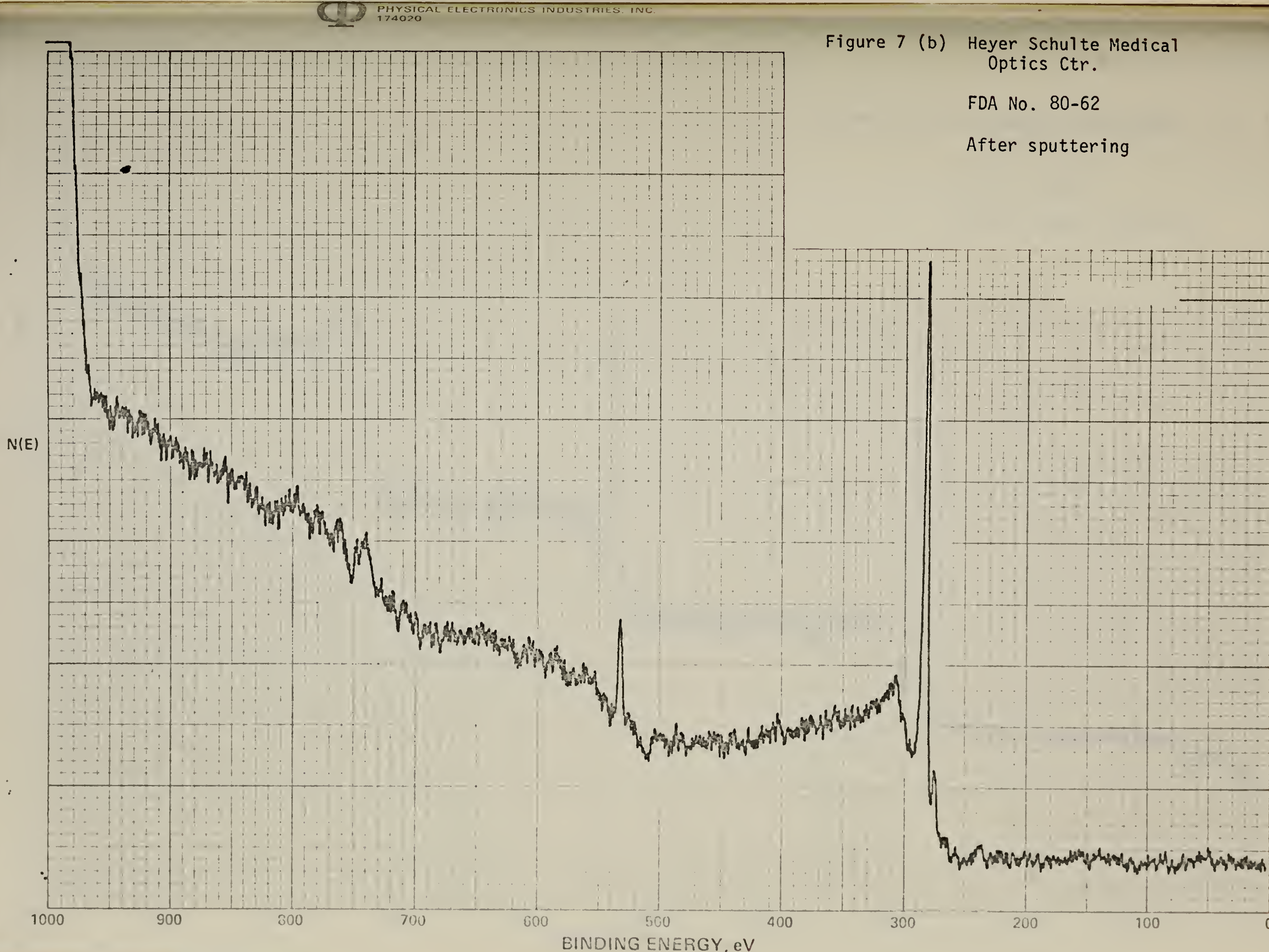


Figure 7 (b) Heyer Schulte Medical Optics Ctr.

FDA No. 80-62

After sputtering

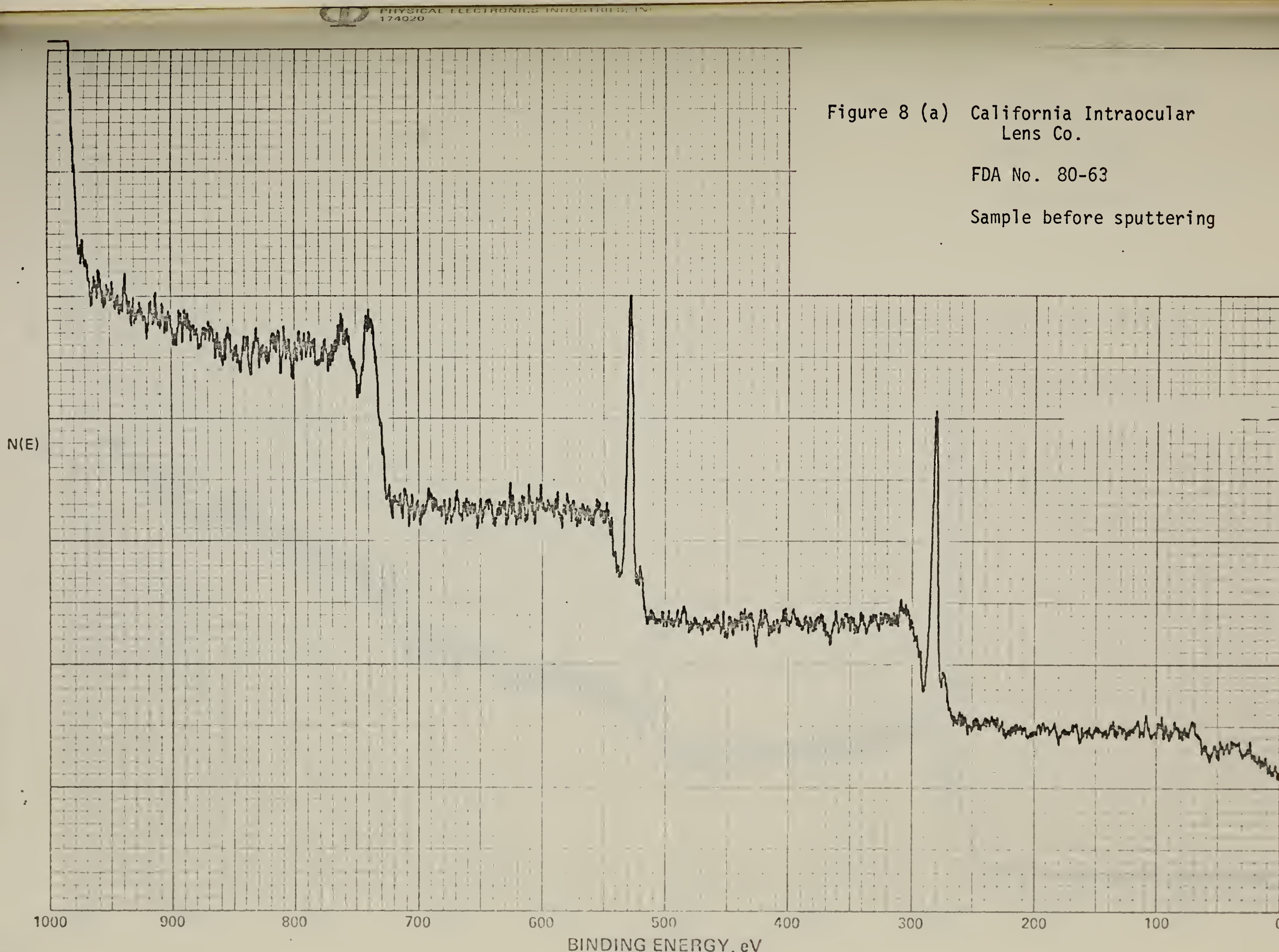
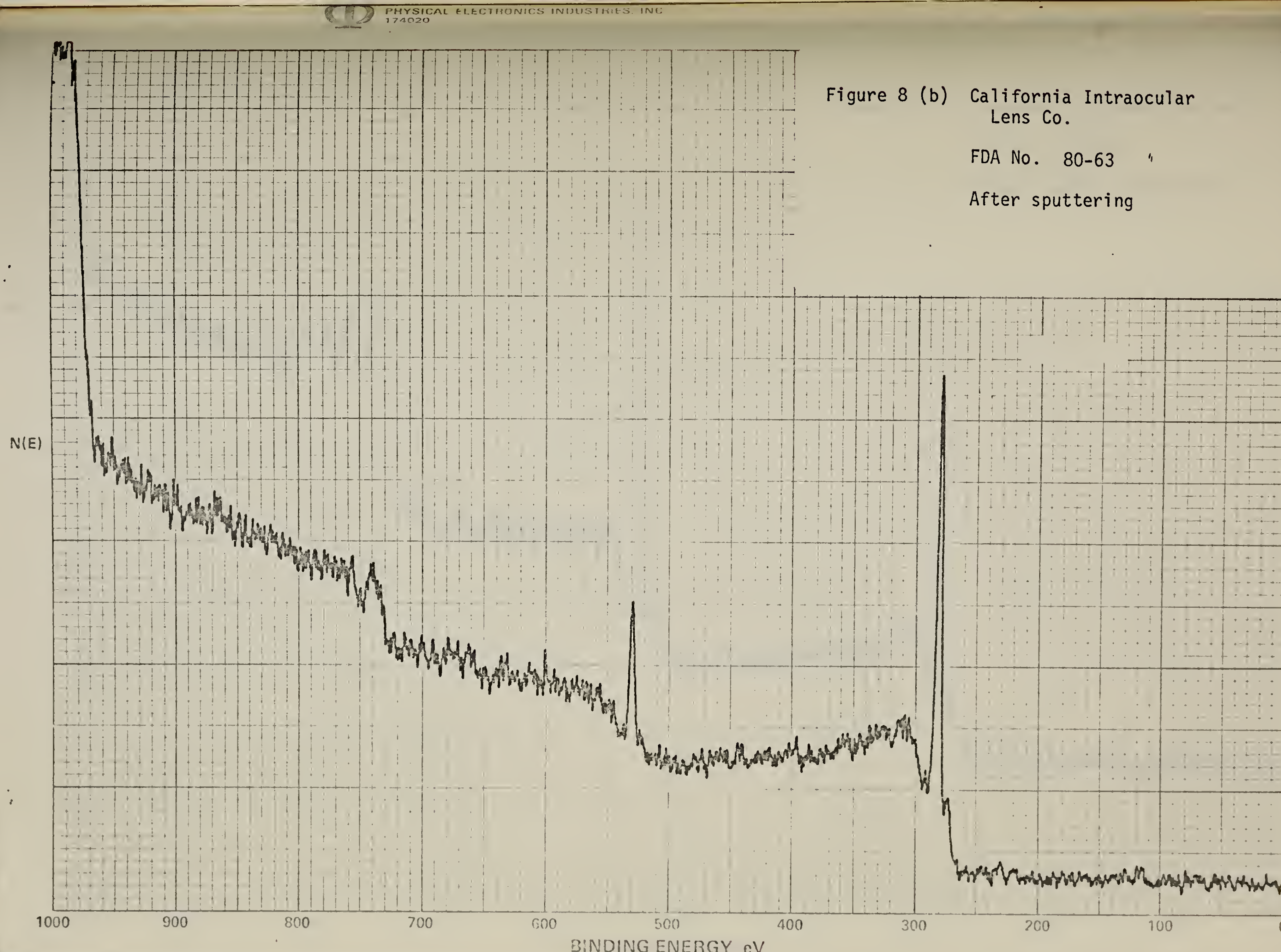


Figure 8 (a) California Intraocular
Lens Co.

FDA No. 80-63

Sample before sputtering


PHYSICAL ELECTRONICS INDUSTRIES, INC
174020
Figure 8 (b) California Intraocular
Lens Co.
FDA No. 80-63
After sputtering
N(E)
1000
900
800
700
600
500
400
300
200
100
0
BINDING ENERGY, eV

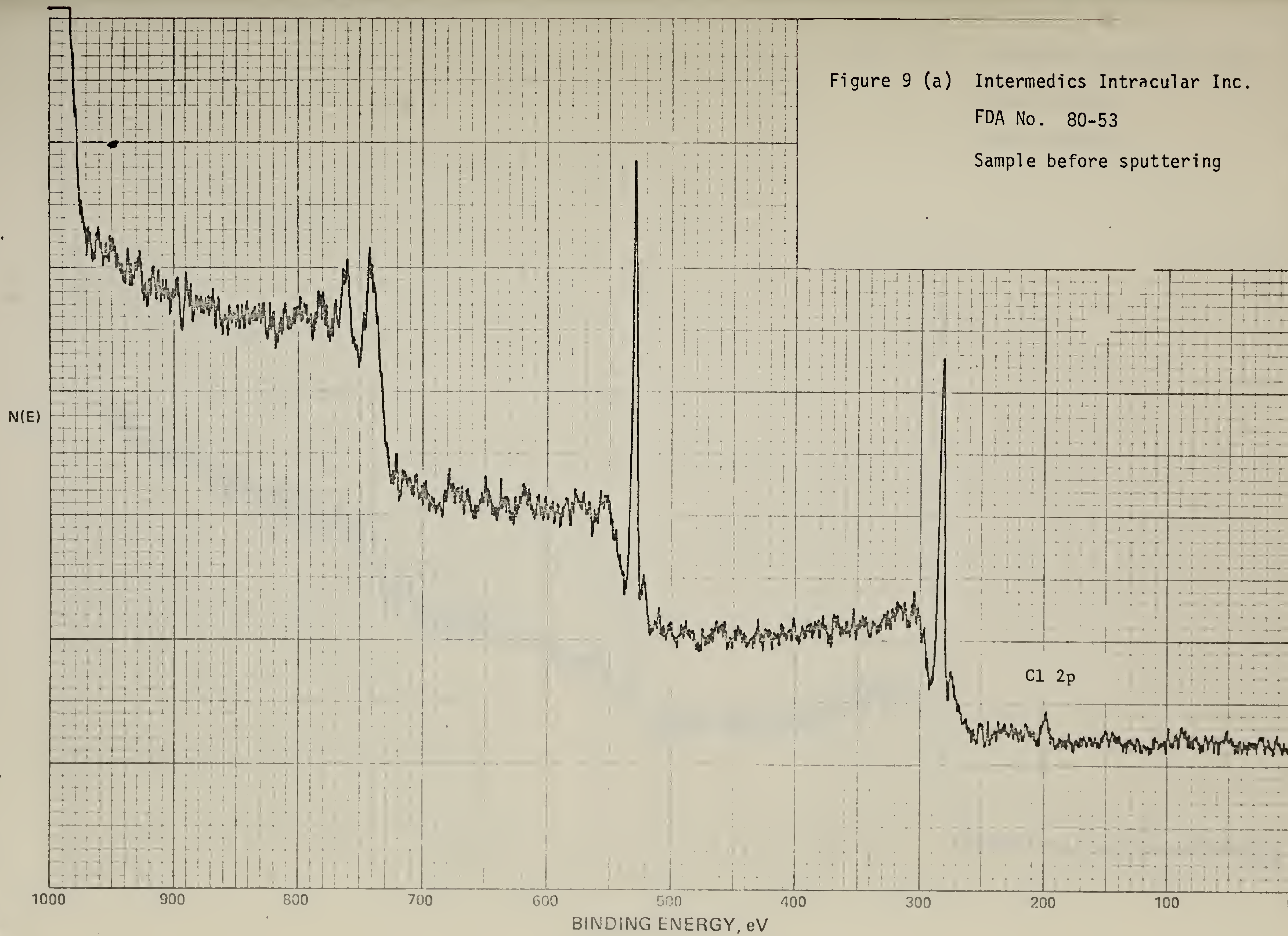
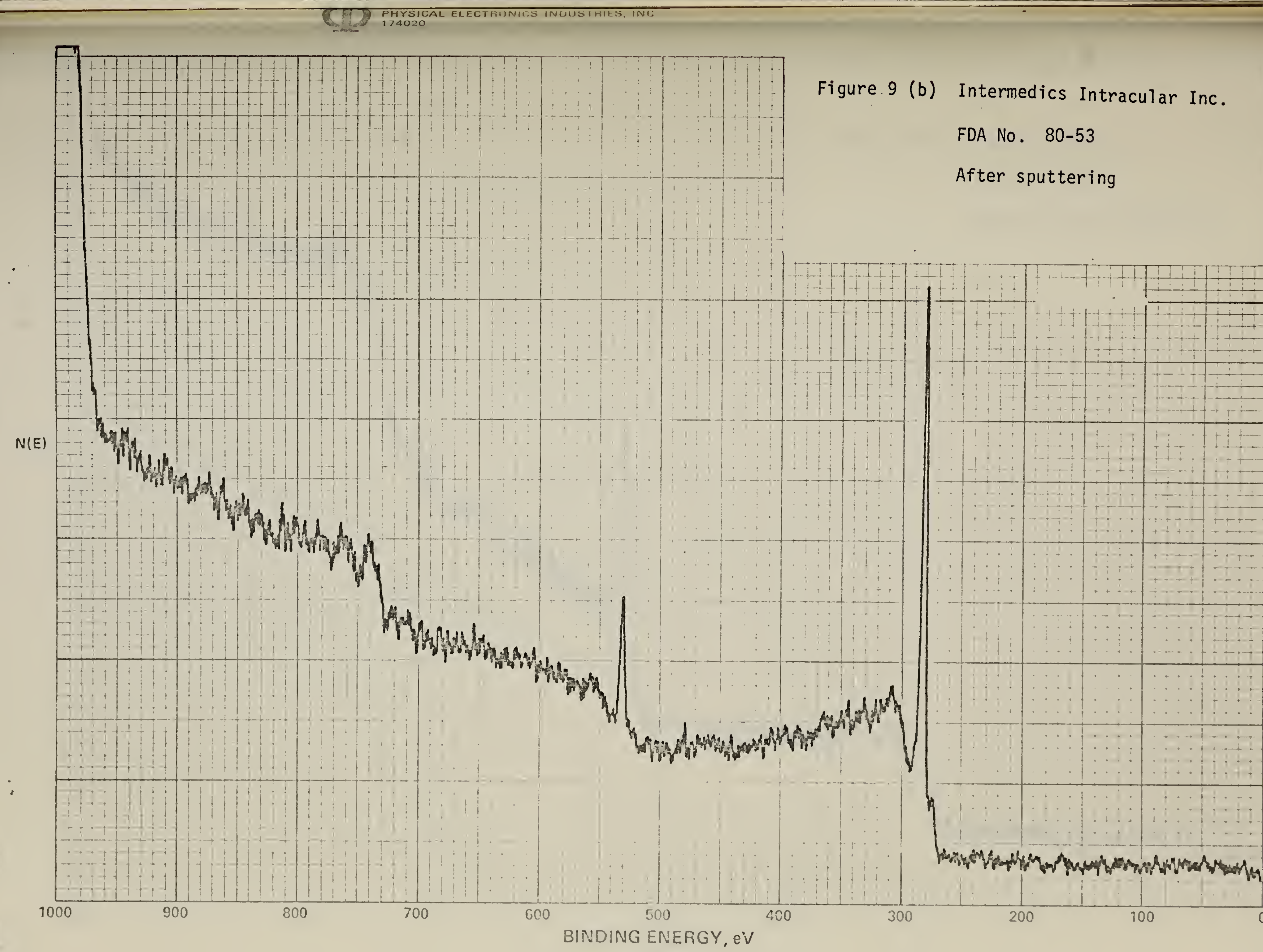


Figure 9 (a) Intermedics Intracular Inc.
FDA No. 80-53
Sample before sputtering


PHYSICAL ELECTRONICS INDUSTRIES, INC
174020
Figure 9 (b) Intermedics Intracular Inc.
FDA No. 80-53
After sputtering
N(E)
1000
900
800
700
600
500
400
300
200
100
0
BINDING ENERGY, eV

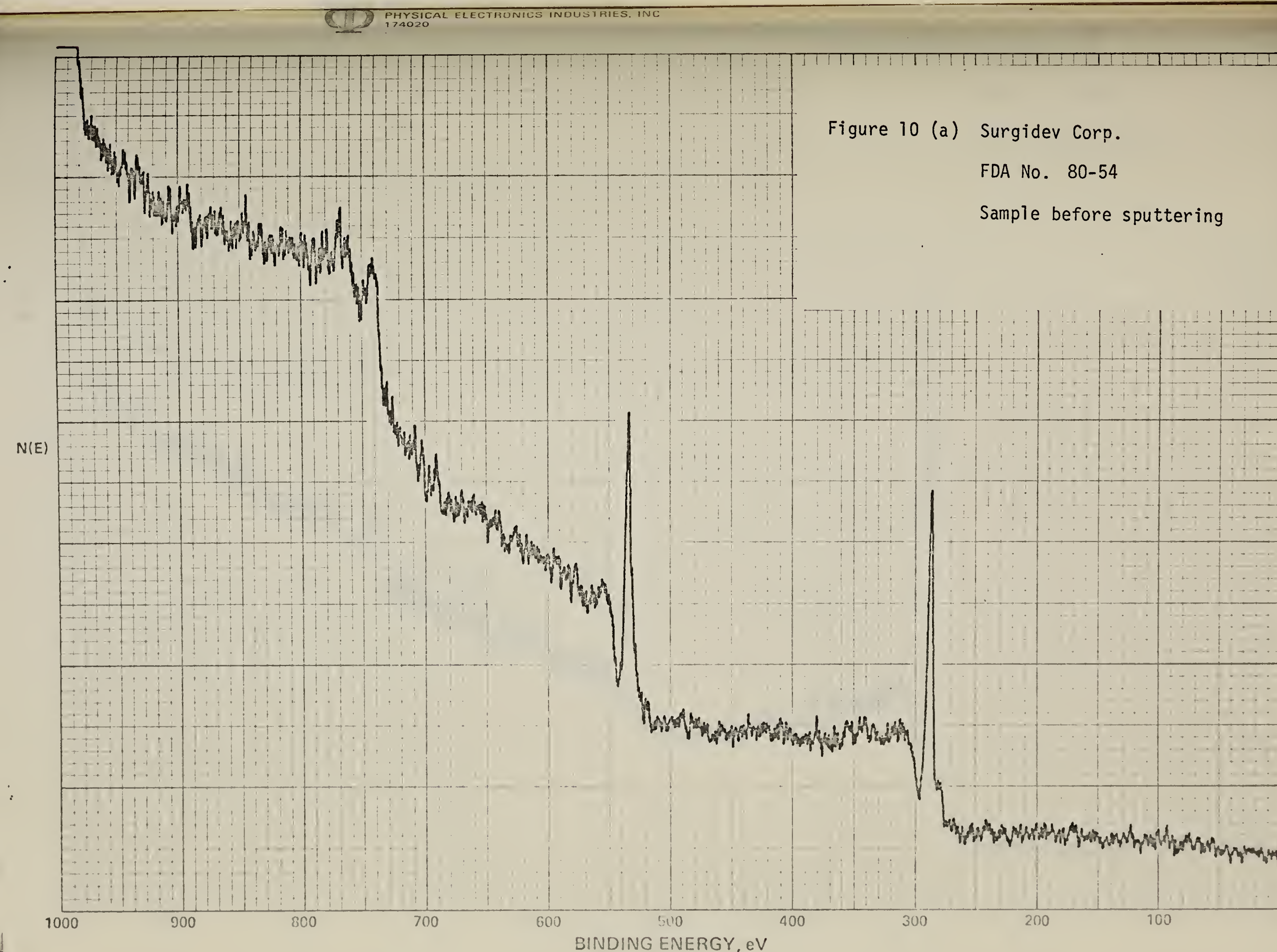
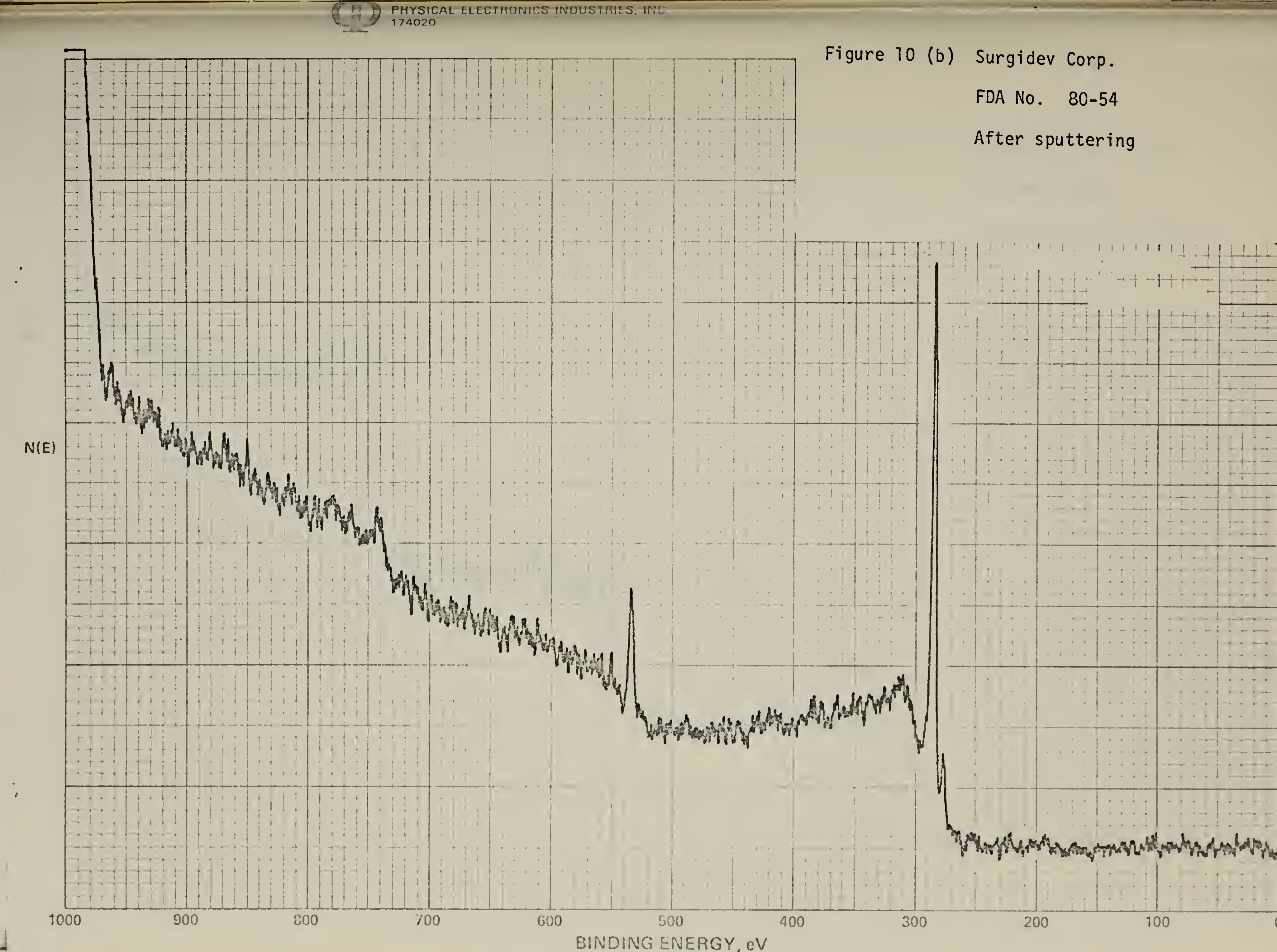

PHYSICAL ELECTRONICS INDUSTRIES, INC
174020
Figure 10 (a) Surgidev Corp.
FDA No. 80-54
Sample before sputtering
N(E)
1000
900
800
700
600
500
400
300
200
100
0
BINDING ENERGY, eV

Figure 10 (b) Surgidev Corp.

FDA No. 80-54

After sputtering



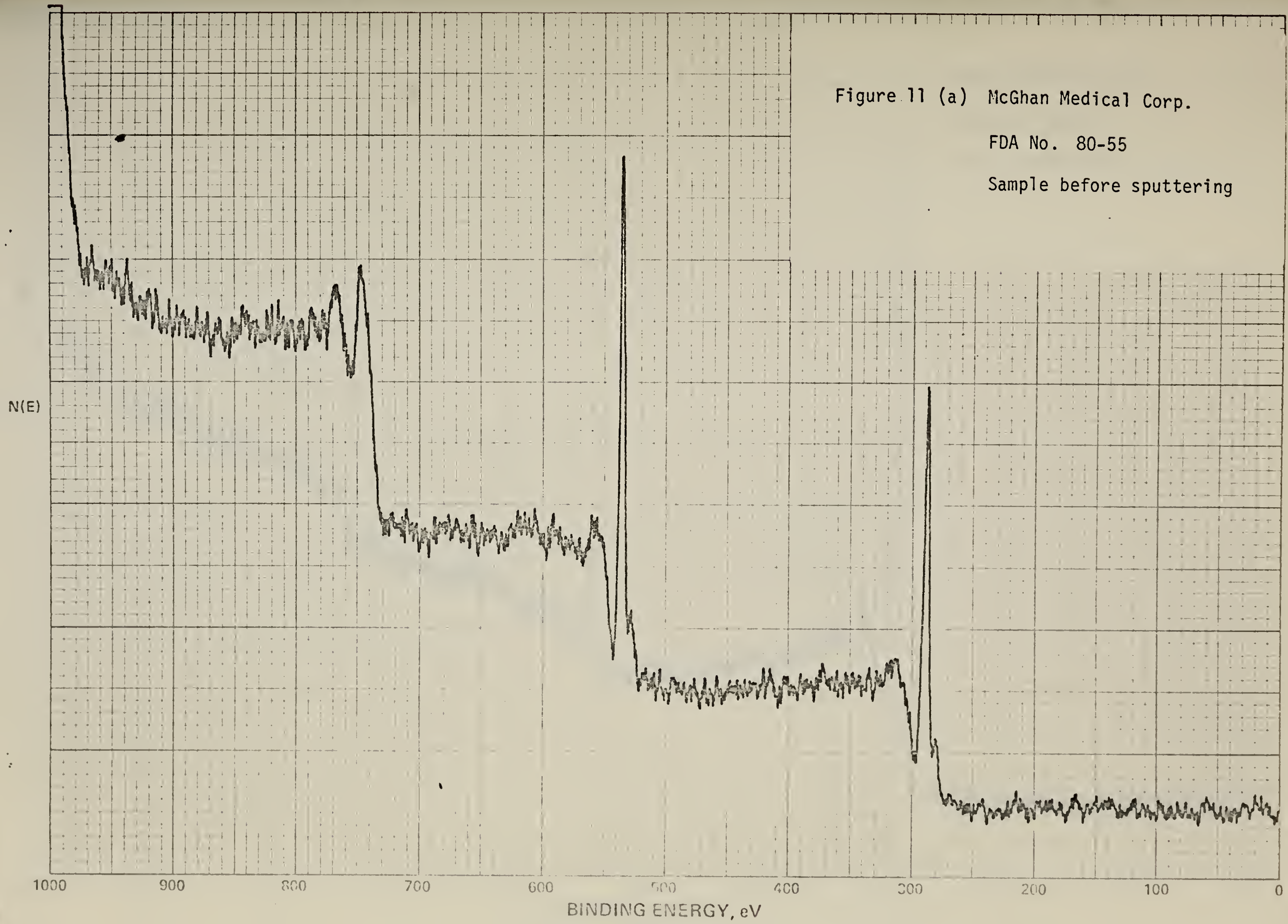


Figure 11 (a) McGhan Medical Corp.
FDA No. 80-55
Sample before sputtering
N(E)
1000
900
800
700
600
500
400
300
200
100
0
BINDING ENERGY, eV

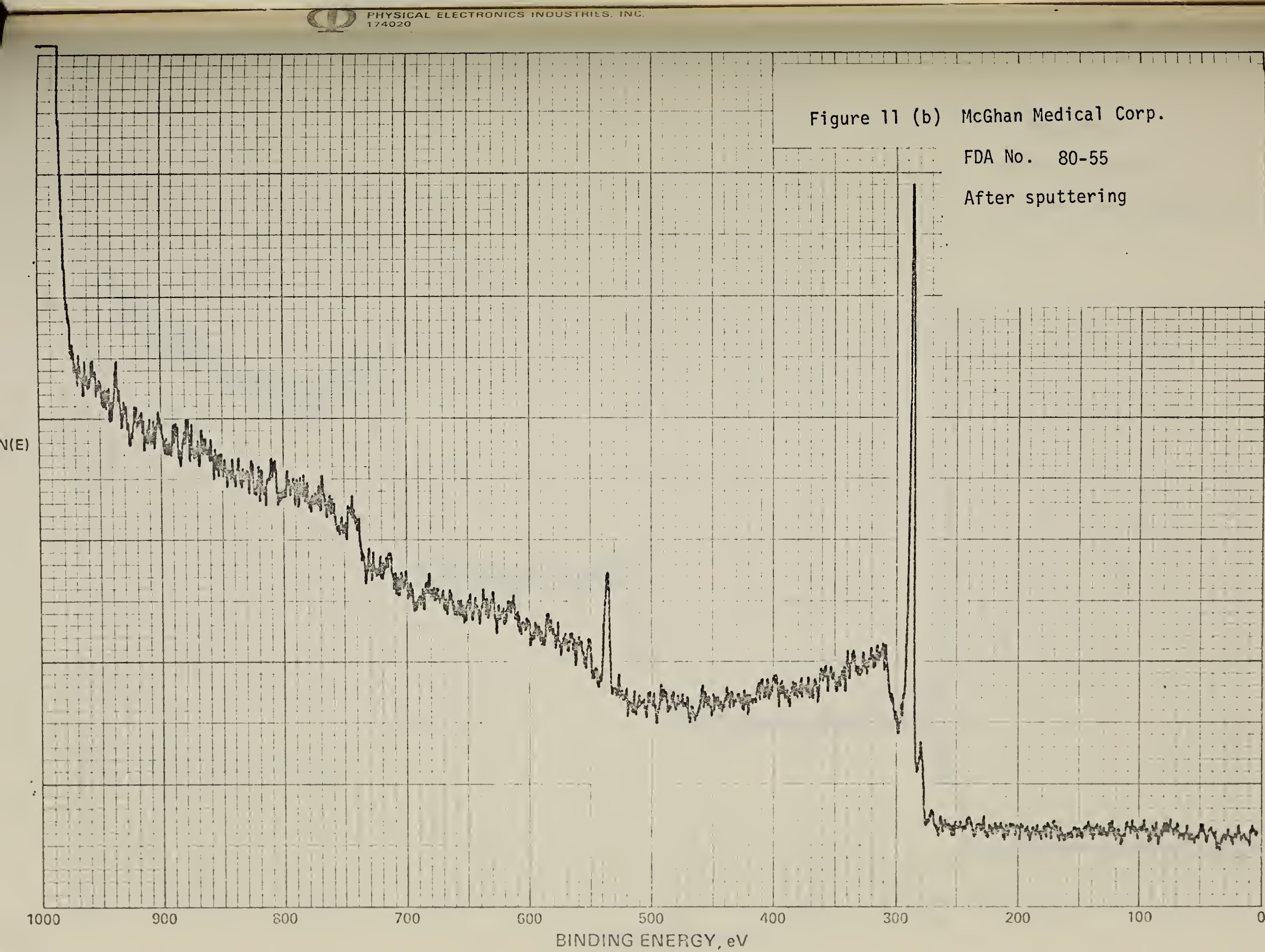


Figure 11 (b) McGhan Medical Corp.

FDA No. 80-55

After sputtering

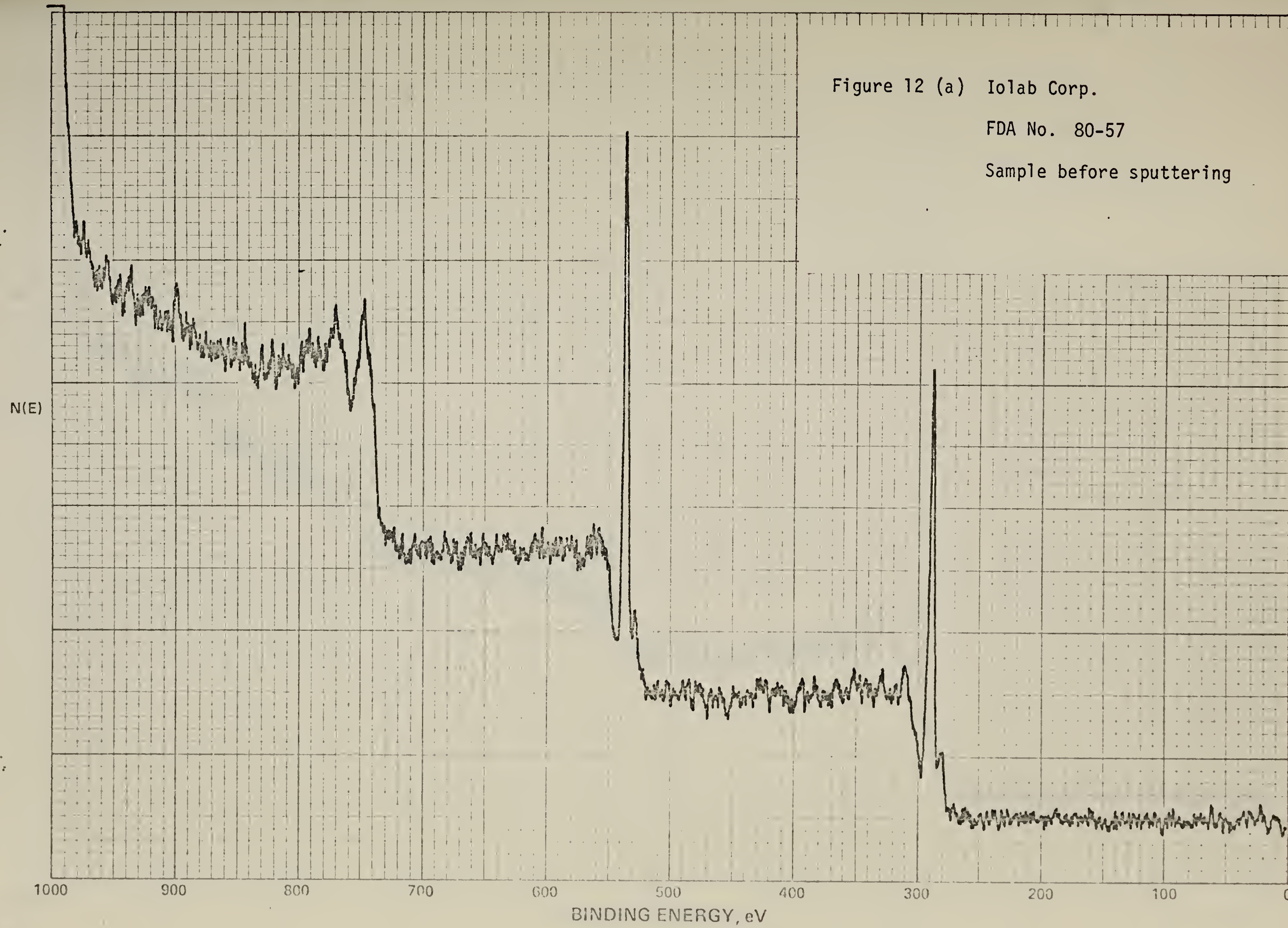
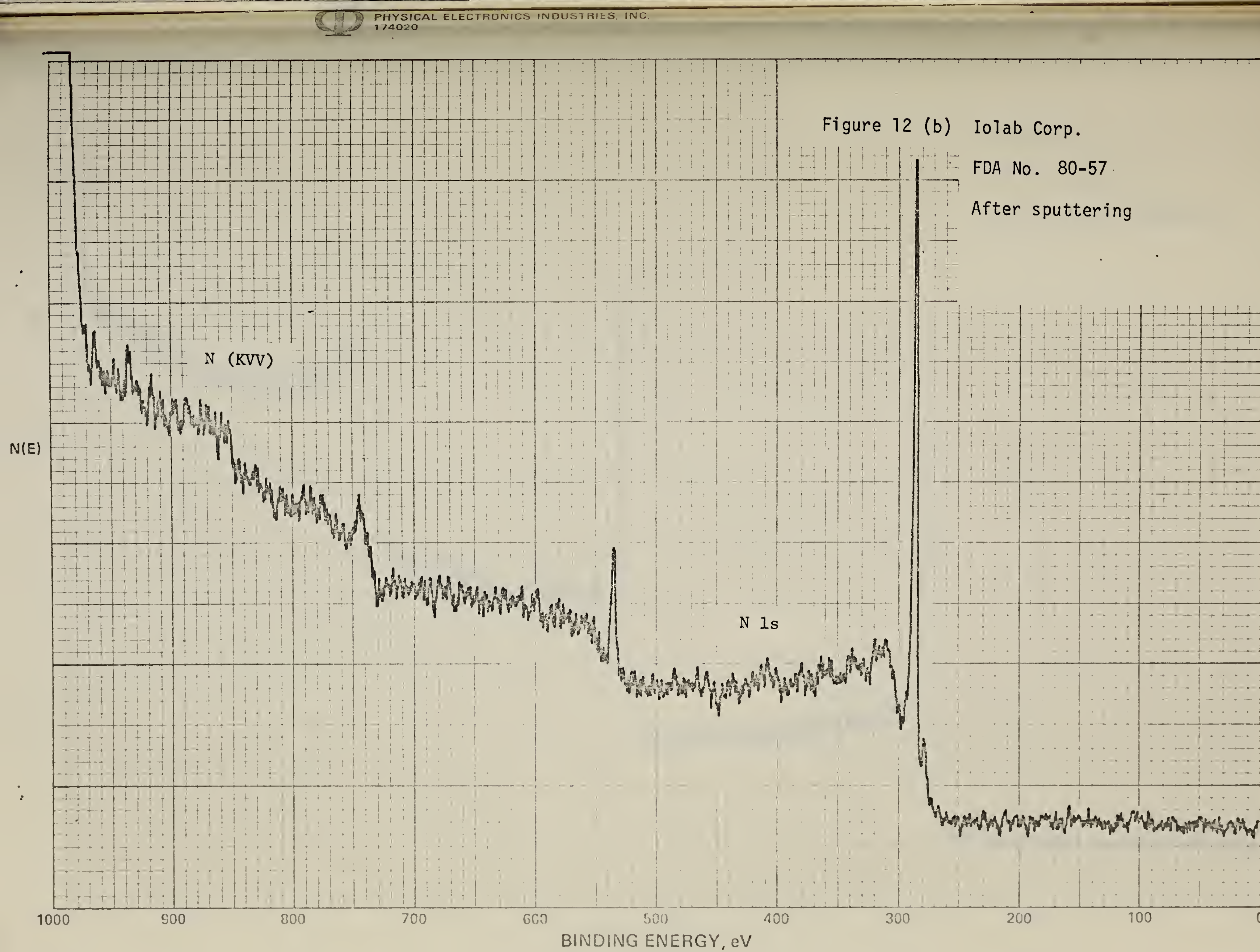
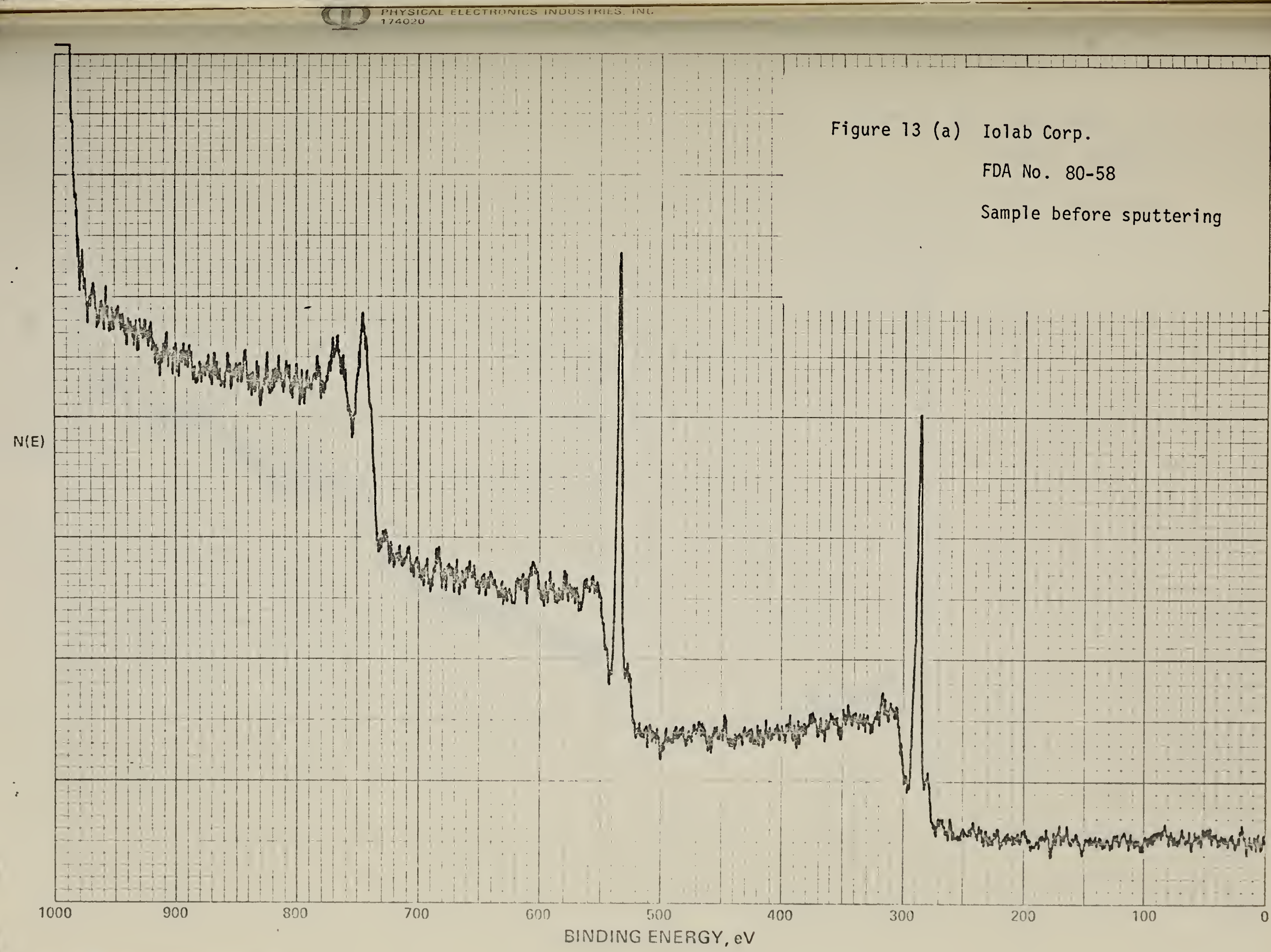


Figure 12 (a) Iolab Corp.

FDA No. 80-57

Sample before sputtering


PHYSICAL ELECTRONICS INDUSTRIES, INC.
174020
Figure 12 (b) Iolab Corp.
FDA No. 80-57
After sputtering
N (KVV)
N 1s
N(E)
1000
900
800
700
600
500
400
300
200
100
0
BINDING ENERGY, eV


PHYSICAL ELECTRONICS INDUSTRIES, INC.
174020
Figure 13 (a) Iolab Corp.
FDA No. 80-58
Sample before sputtering
N(E)
1000
900
800
700
600
500
400
300
200
100
0
BINDING ENERGY, eV

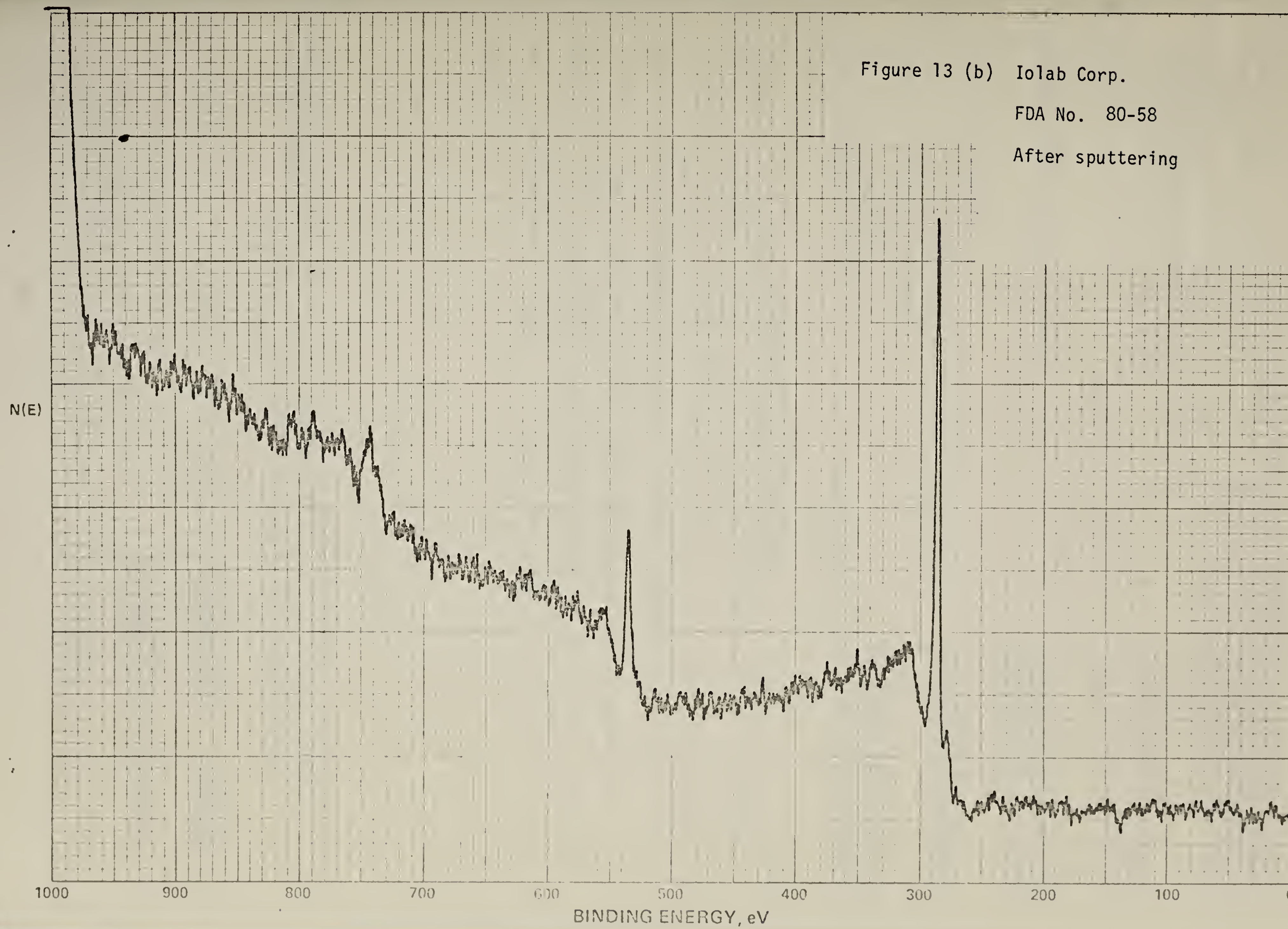


Figure 13 (b) Iolab Corp.

FDA No. 80-58

After sputtering

NBS-114A (REV. 9-78)

U.S. DEPT. OF COMM. BIBLIOGRAPHIC DATA SHEET	1. PUBLICATION OR REPORT NO. NBSIR-80-2156	2. Gov't. Accession No.	3. Recipient's Accession No.

4. TITLE AND SUBTITLE

Surface Analyses of Medical Lens Implants by X-Ray Photoelectron Spectroscopy

5. Publication Date

October 1980

6. Performing Organization Code

7. AUTHOR(S)

Terrence Jach

8. Performing Organ. Report No.

9. PERFORMING ORGANIZATION NAME AND ADDRESS

NATIONAL BUREAU OF STANDARDS
DEPARTMENT OF COMMERCE
WASHINGTON, DC 20234

10. Project/Task/Work Unit No.

541-1457

11. Contract/Grant No.

Task 80-07, NBS-BMD Interagency Agreement

12. SPONSORING ORGANIZATION NAME AND COMPLETE ADDRESS *(Street, City, State, ZIP)*

Mechanics and Materials Branch
Medical Devices Laboratory
Food and Drug Administration
Silver Spring, MD 28110

13. Type of Report & Period Covered

Final

14. Sponsoring Agency Code

15. SUPPLEMENTARY NOTES

☐ Document describes a computer program; SF-185, FIPS Software Summary, is attached.

16. ABSTRACT *(A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here.)*

Thirteen types of intraocular lens manufactured for human implantation were examined by X-Ray Photoemission Spectroscopy (XPS). The elements present on the surface of each lens were detected before and after cleaning by ion bombardment. All lenses showed strong surface coverage of the elements carbon and oxygen. Trace amounts of chlorine on one lens and nitrogen on another lens were observed.

17. KEY WORDS *(six to twelve entries; alphabetical order; capitalize only the first letter of the first key word unless a proper name; separated by semicolons)*

Intraocular lenses; surface analysis; X-ray photoelectron spectroscopy.

18. AVAILABILITY ☐ Unlimited

☒ For Official Distribution. Do Not Release to NTIS

☐ Order From Sup. of Doc., U.S. Government Printing Office, Washington, DC 20402, SD Stock No. SN003-003-

☐ Order From National Technical Information Service (NTIS), Springfield, VA. 22161

19. SECURITY CLASS (THIS REPORT)	21. NO. OF PRINTED PAGES
UNCLASSIFIED	
20. SECURITY CLASS (THIS PAGE)	**22. Price**
UNCLASSIFIED	

USCOMM-DC