Active

Project #: E-18-617 Cost share #:

Rev #: 0 OCA file #:

Work type : RES

Contract#: H-13010D

Mod #: (INIT + MOD #1) Document : PO Prime #:

Contract entity: GTRC

Subprojects ? : N

CFDA: N/A

Main project #: PE #: N/A

Project unit:

MAT ENGR

Unit code: 02.010.112

Project director(s):

CARTER W B

MAT ENGR (404)894-6762

Sponsor/division names: NASA / MARSHALL SPACE FLT CTR, AL

Sponsor/division codes: 105 / 005

Award period: 920731 920930 (performance) to 920930 (reports)

Sponsor amount New this change Total to date Contract value 19,844.00 19,844.00 Funded 19,844.00 19,844.00 Cost sharing amount 0.00

Does subcontracting plan apply ?: N

Title: ELECTRON SPECTROSCOPY FOR CHEMICAL ANALYSIS - SAMPLE ANALYSIS

PROJECT ADMINISTRATION DATA

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894-4820

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MARSHALL SPACE FLIGHT CENTER, AL

35812

PROCUREMENT OFFICE

NASA MARSHALL SPACE FLIGHT CENTER MARSHAL SPACE FLIGHT CENTER, AL

35812

Security class (U,C,S,TS) : U Defense priority rating : DO-C9

ONR resident rep. is ACO (Y/N):

N/A supplemental sheet

GIT X

Equipment title vests with: SEE SECTION IX. (<\$5,000 VESTS W/GIT UPON ACQUISITION.)

Sponsor

Administrative comments -

› INITIATION OF TWO-MONTHS, FIXED-PRICE PURCHASE ORDER. **NOTE: REPORTS TO BE& SUBMITTED THRU OCA/REPORTS COORDINATOR. NO DELIVERABLES SCHEDULE APPLICABLE.*

GEORGIA INSTITUTE OF TECHNOLOGY OFFICE OF CONTRACT ADMINISTRATION

NOTICE OF PROJECT CLOSEOUT

Close	eout Notice	Date :	11/03/92
Project No. E-18-617	Center No.	10/24	-6-R7581-0A0_
Project Director CARTER W B	School/Lab	MSE	
Sponsor NASA/MARSHALL SPACE FLT CTR, AL			_
Contract/Grant No. H-13010D	Contract E	ntity (ЭTRC
Prime Contract No.			
Title ELECTRON SPECTROSCOPY FOR CHEMICAL ANALYSIS	- SAMPLE AN	ALYSIS_	
Effective Completion Date 920930 (Performance) 920	930 (Report	s)	
Closeout Actions Required:		Y/N	Date Submitted
Final Invoice or Copy of Final Invoice Final Report of Inventions and/or Subcontracts Government Property Inventory & Related Certif: Classified Material Certificate Release and Assignment Other		Y Y Y N Y	
CommentsEFFECTIVE DATE 7-31-92. CONTRACT VALUE	\$19,844		
Subproject Under Main Project No.			
Continues Project No.			
Distribution Required:			
Project Director Administrative Network Representative GTRI Accounting/Grants and Contracts Procurement/Supply Services Research Property Managment Research Security Services	Y Y Y Y		
Reports Coordinator (OCA) GTRC Project File Other HARRY VANN FMD	Y Y Y Y		

NOTE: Final Patent Questionnaire sent to PDPI.

£ 18.6/1

ELECTRON SPECTROSCOPY FOR CHEMICAL ANALYSIS -- SAMPLE ANALYSIS

REPORT # LDEF-01

Prepared by:

W.B. Carter School of Materials Science and Engineering Georgia Institute of Technology Atlanta, GA 30332-0245

for:

NASA/MSFC Marshall Space Flight Center, AL 35812

under:

PO # H-13010D

11 September 1992

This document reports the results of ESCA analyses performed on the following samples:

IV-1, IV-4, IV-5, IV-8, IV-11, IV-68, IV-73

A description of the ESCA data precedes reports of the analyses.

Description of ESCA Data

Two types of ESCA spectra were collected 1) general surveys and 2) high resolution spectra. The ESCA spectrometer is calibrated such that the Au $4f_{7/2}$ photoemission peak appears at a B.E. of 84.0 eV.

General surveys were first taken of all samples over a binding energy (B.E.) range of -10 ev to 1090 eV. A single count, plotted on the vertical axes, corresponds to one detected photoelectron. An analyzer pass energy of 200 eV and an x-ray spot about 1 mm in diameter were used for the collection of general surveys. Semiquantitative analyses of the elemental compositions of the sample surfaces were performed from the general survey data. These analyses are based on the photoemission peak areas of the various elements represented in the spectra, their photoemission cross sections and the energy response of the analyses. Not included in these analyses are the effects of variable electron inelastic mean free paths in the samples or the influences of sample inhomogeneities.

High resolution spectra were taken over B.E. ranges corresponding to photoemission peaks of interest identified in the general surveys. High resolution spectra were collected using an analyzer pass energy of 50 eV and an x-ray spot of about 300 $\mu \rm m$ in diameter. Most high resolution spectra have been peak fitted with one or more Gaussian components. The energy, FWHM and area (total counts in Gaussian component) corresponding to each fitted component are displayed on the spectra.

At least two spots on each sample were examined as discussed in the reports below.

Reports on ESCA Analyses

Samples IV-1, IV-4, IV-5 and IV-11 produced similar spectra with the exception that IV-1 has a small amount of Ag present. Varying amounts of C were observed on these samples ranging from about 9 atomic percent near the center of IV-5 to about 20 atomic percent at one spot on IV-4. The semiquantitative 0 to Si ratios range from about 1.6 to 2.1. It must be stressed that these ratios are for the surfaces of the samples, which almost certainly are covered to some extent with water, adventitious carbon, O_2 , etc.

Samples IV-8, IV-68 and IV-73 were each unique among the IV designated samples. Each sample analysis is presented below.

IV-1: Exposed passivated Ti reflector

A spot near the center and a spot about midway between the center and the perimeter (off center region) of this sample were examined.

The surface of this specimen is composed primarily of O, Si and C with a small amount of Ag (~1 atomic %). Semiquantitatively, the ratio of O to Si is about 2:1.

The general surveys from each spot were similar and indicated the presence of O, Si, Ag and C. See figure 1 (center region) and figure 4 (off center region). Figure 4 indicates the possible presence of I. Semiquantitative analyses of the relative sizes of the photoemission peaks from the four elements common to both spectra yield the following:

Table 1: Semiquantitative Composition of Center Region of Sample IV-1 from Figure 1

Element	Approximate Atomic Percent
0	58
Si	30
C	11
Ag	1

Table 2: Semiquantitative Composition of Off Center Region of Sample IV-1 from Figure 5

Element	Approximate Atomic Percent
0	60
Si	28
С	11
Ag	1

Figures 2 and 3 are high resolution spectra of the Si 2s and 0 1s photoemission peaks, respectively, obtained from the center region. Each has been peak fitted with a single Gaussian peak. Figures 5 and 6 are similar high resolution spectra obtained from

the off center region. Both Si 2s spectra and both O 1s spectra are similar.

Figure 7 is a high resolution spectrum of the Ag 3d photoemission peaks $(3d_{5/2}$ and $3d_{3/2})$ obtained from the off center region of the sample. It has been fitted with two Gaussian components.

IV-4: Exposed Ti with protective overcoat

A spot near the center and a spot about midway between the center and the perimeter (off center region) of this sample were examined.

The surface of this specimen is composed primarily of O, Si and C. Figures 8 - 13 display the spectra taken on this sample. Tables 3 and 4 present the semiquantitative analyses obtained from the two general surveys taken on this sample.

Table 3: Semiquantitative Composition of Center Region of Sample IV-4 from Figure 8

Approximate Atomic Percent
54
30
16

Table 4: Semiquantitative Composition of Off Center Region of Sample IV-4 from Figure 11

Element	Approximate Atomic Percent
0	50
Si	31
С	19

IV-5: Exposed Ti, passivated

A spot near the center and a spot about midway between the center and the perimeter (off center region) of this sample were examined.

The surface of this specimen is composed primarily of O, Si and C. Figures 14 - 19 display the spectra taken on this sample. Tables 5 and 6 present the semiquantitative analyses obtained from the two general surveys taken on this sample.

Table 5: Semiquantitative Composition of Center Region of Sample IV-5 from Figure 14

57
34
9

Table 6: Semiquantitative Composition of Off Center Region of Sample IV-5 from Figure 17

Element	Approximate Atomic Percent
0	54
Si	33
С	13

IV-8: Exposed Al, Sio,

The surface of this specimen is about half covered with a dark material that appears "corroded". The region not appearing "corroded" appears "shiny". Two spots were examined - one located on a shiny region of the sample and one located on the "corroded" region. There are fingerprints on the side of the sample that was not examined.

Both spots examined indicate the presence of O, C and Ag. The "shiny" region also displays Si and a small amount of Na (~2 atomic %). The "corroded" region does not display Si or Na. The relative amounts of C and Ag differ substantially between the two regions. The "shiny" region displays about 18 atomic percent C

and about 2 atomic percent Ag while the "corroded" region displays about 57 atomic percent C and about 21 atomic percent Ag.

The O 1s spectrum from the "shiny" region is composed of at least two components. The larger of these is probably associated with silicon oxides and the smaller with metallic oxides. The O 1s spectrum from the "corroded" region displays a single Gaussian component, which is consistent with presence of a single oxide of Ag. The "corroded" region appears to be covered with primarily carbonaceous material and an oxide of silver.

Figures 20 - 27 display the spectra taken on this sample. Tables 7 and 8 present the semiquantitative analyses obtained from the two general surveys taken on this sample.

Table 7: Semiquantitative Composition of "Shiny" Region of Sample IV-8 from Figure 20

Element	Approximate Atomic Percent
0	55
Si	23
С	18
Ag	2
Ag Na	2

Table 8: Semiquantitative Composition of "Corroded" Region of Sample IV-8 from Figure 24

Element	Approximate Atomic Percent
0	22
С	57
Ag	21

IV-11: Exposed Al, Sio,

A spot near the center and a spot about midway between the center and the perimeter (off center region) of this sample were examined.

The surface of this specimen is composed primarily of 0, Si and C. Figures 28 - 33 display the spectra taken on this sample. Tables 9 and 10 present the semiquantitative analyses obtained from the two general surveys taken on this sample.

Table 9: Semiquantitative Composition of Center Region of Sample IV-11 from Figure 28

Element	Approximate Atomic Percent
0	53
Si	30
C	17

Table 10: Semiquantitative Composition of Off Center Region of Sample IV-11 from Figure 31

Approximate Atomic Percent
54
32
14

IV-68: Exposed tungsten

About half of one side of this sample was exposed during the LDEF flight. Two spots in the exposed area were examined - one spot near the center (center region) and one spot about midway between the center and the perimeter (off center region).

Both spots display O, C, Si and W. The center region displays substantially more C than the off center region.

The two high resolution scans of the W $4f_{7/2}$ photoemission peak indicate that its B.E. is about 35.5 eV. This B.E. is indicative of WO₃.^{1,2} WO₂ displays values for this B.E. 2 to 3 eV lower. The W4 $f_{7/2}$ B.E. from tungsten oxy-carbides are 1 to 2 eV higher.

It can thus be surmised that most of the W at or near the surface of this sample is in the form of WO_3 .

The O 1s spectra are composed of at least two components. The larger of these is probably associated with silicon oxides and the smaller with WO_3 .

Tables 11 and 12 present the results of the semiquantitative analyses obtained from the two general surveys taken from this sample. Figures 34 - 41 contain the spectra taken from this sample.

Table 11: Semiquantitative Composition of Center Region of Sample IV-68 from Figure 34

Element	Approximate Atomic Percent
0	49
Si	22
С	26
W	3

Table 12: Semiquantitative Composition of Off Center Region of Sample IV-68 from Figure 38

Element	Approximate Atomic Percent
0	59
Si	26
С	11
W	4

IV-73: Exposed tantalum

About half of one side of this sample was exposed during the LDEF flight. Two spots in the exposed area were examined - one spot near the center (center region) and one spot about midway between the center and the perimeter (off center region).

Both spots display O, C, Si and Ta. Substantial amounts of C exist on both spots. The two high resolution scans of the Ta $4f_{7/2}$ photoemission peak indicate that its B.E. is about 26.5 eV. This B.E. is indicative of Ta_2O_5 . The O 1s spectra are composed

of at least two components. The larger of these is probably associated with silicon oxides and the smaller with Ta_2O_5 .

Tables 13 and 14 present the results of the semiquantitative analyses obtained from the two general surveys taken from this sample. Figures 42 - 50 contain the spectra taken from this sample.

Table 13: Semiquantitative Composition of Center Region of Sample IV-73 from Figure 42

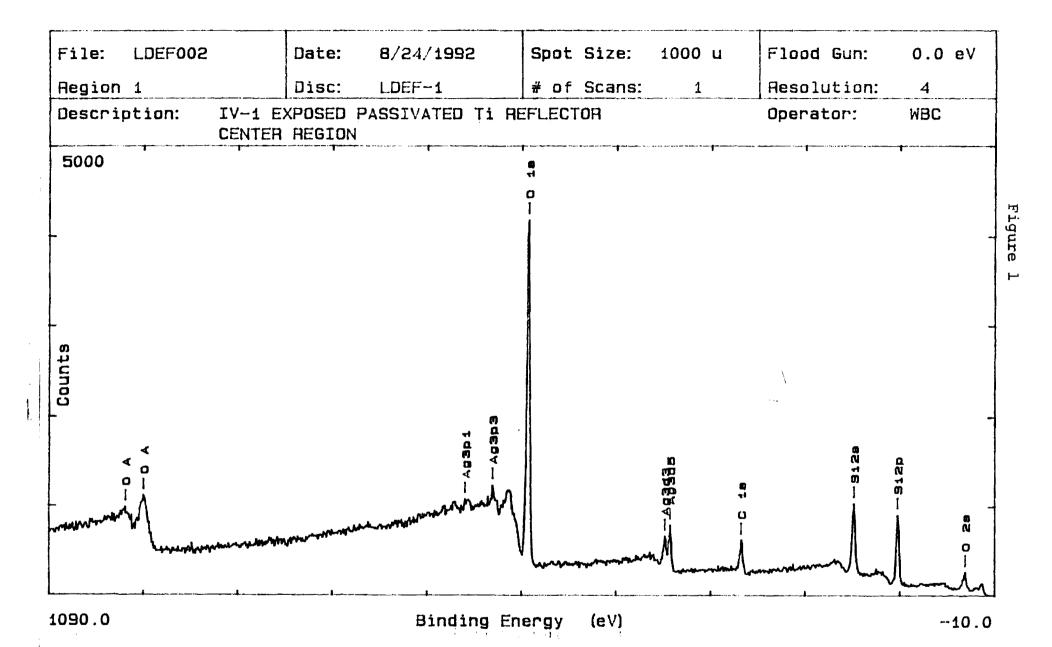
Element	Approximate Atomic Percent
0	46
si	15
C	34
Ta	5

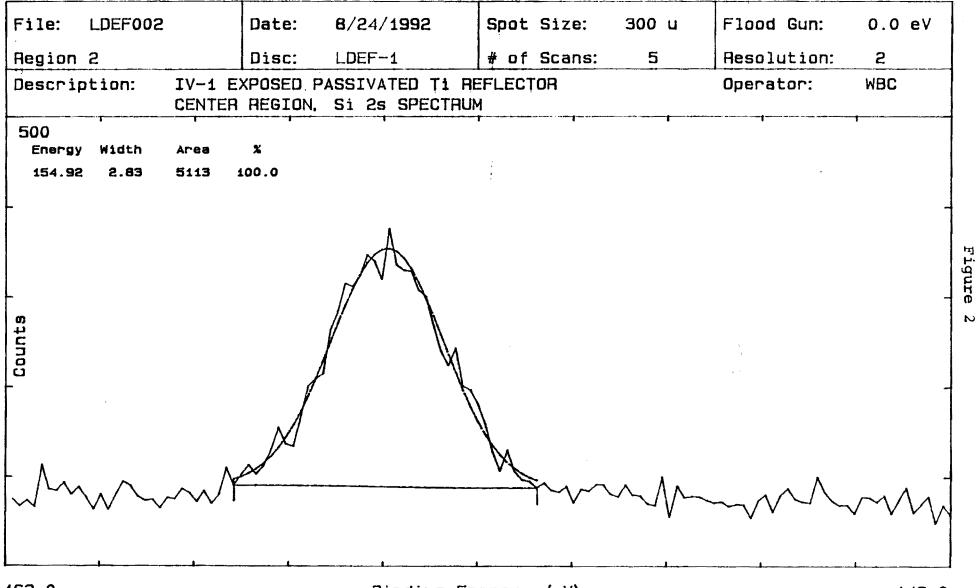
Table 14: Semiquantitative Composition of Off Center Region of Sample IV-73 from Figure 47

Approximate Atomic Percent
47
20
29
4

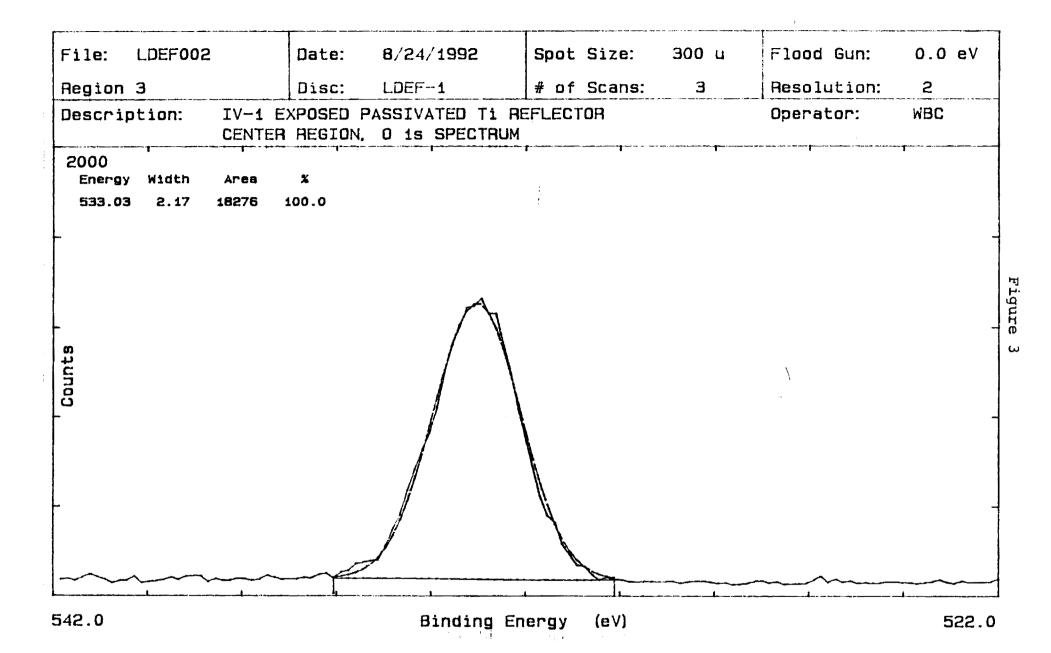
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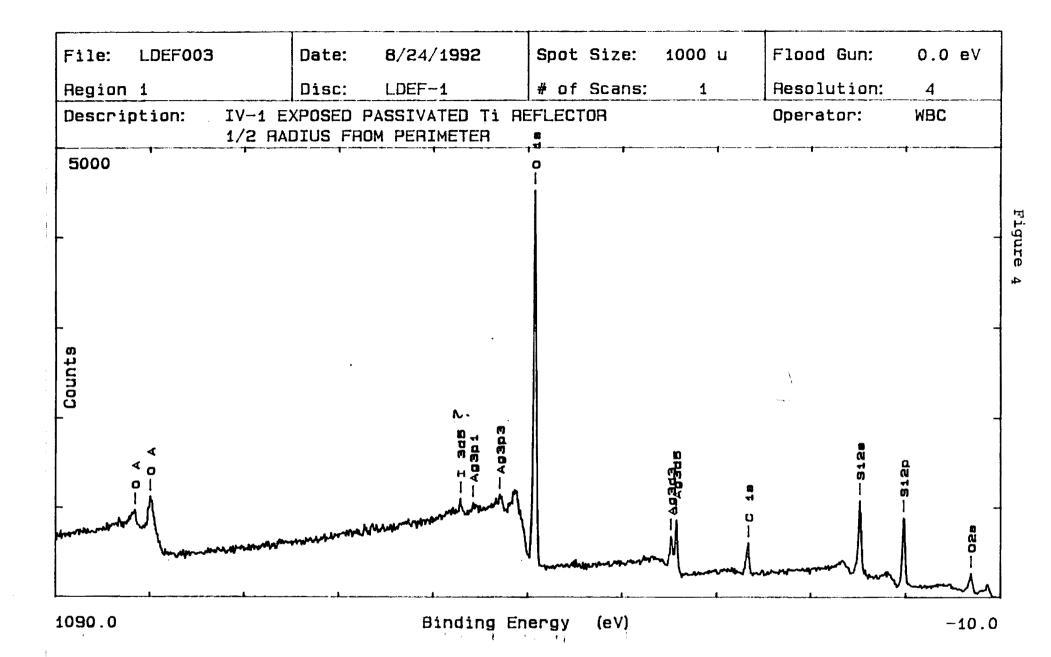
- 1.D.D. Sarma and C.N.R. Rao, J. Electron Spectrosc. Relat. Phenom. 20, 25(1980).
- 2.L. Salvati, L.E. Makovsky, J.M. Stencel, F.R. Brown and D.M. Hercules, J. Phys. Chem. <u>85</u>, 3700 (1981).

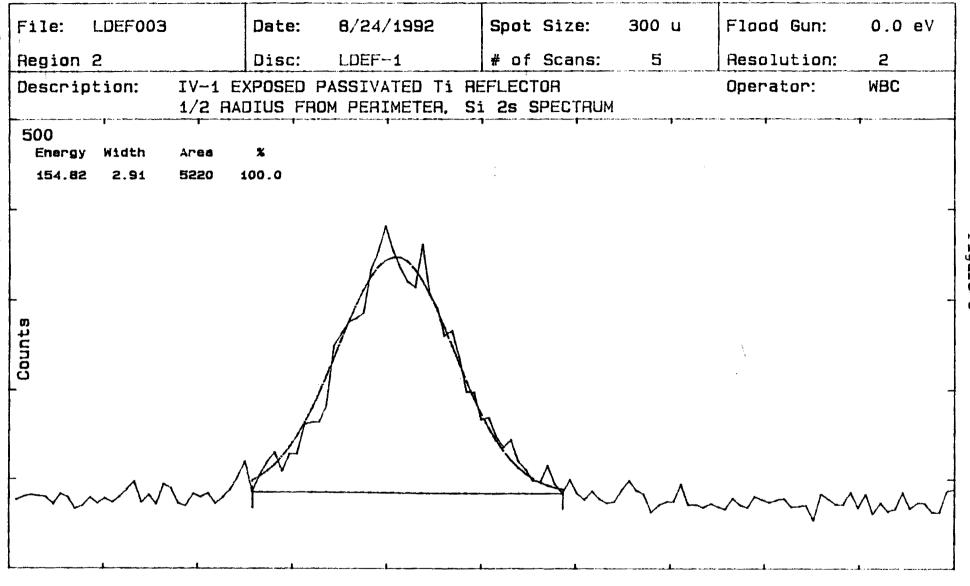




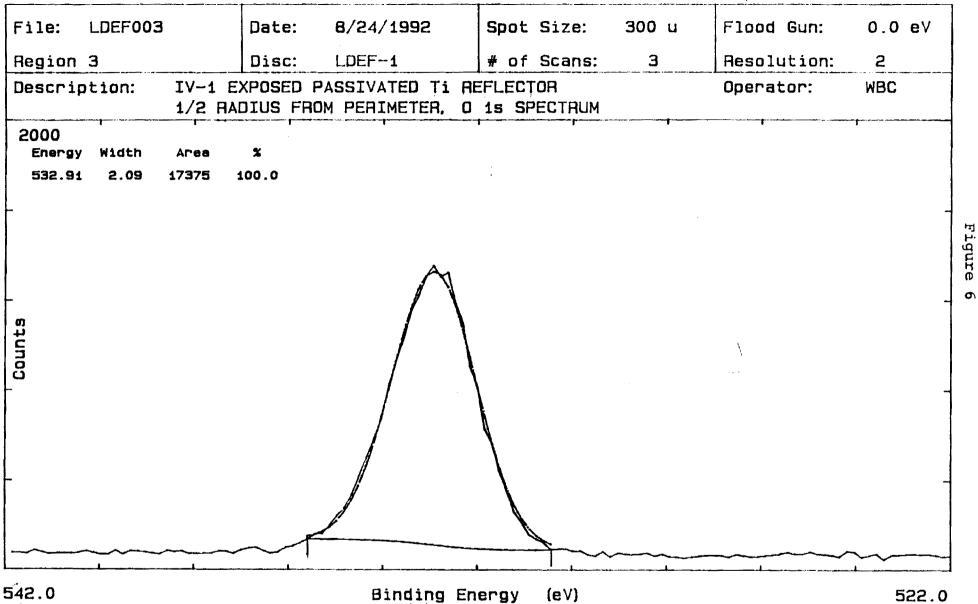
Binding Energy (eV)





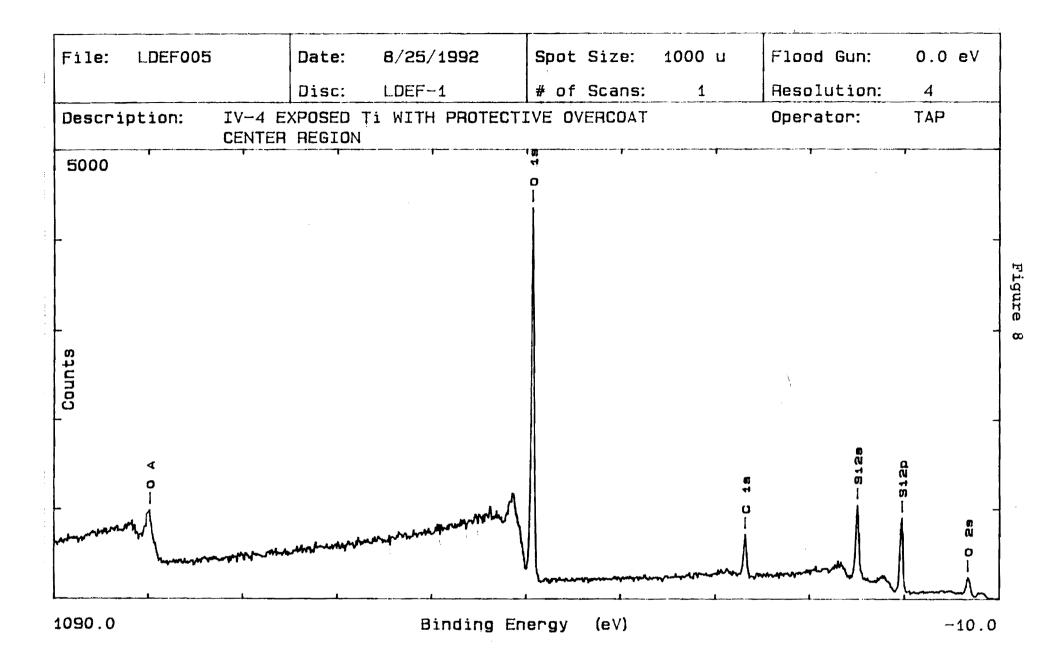


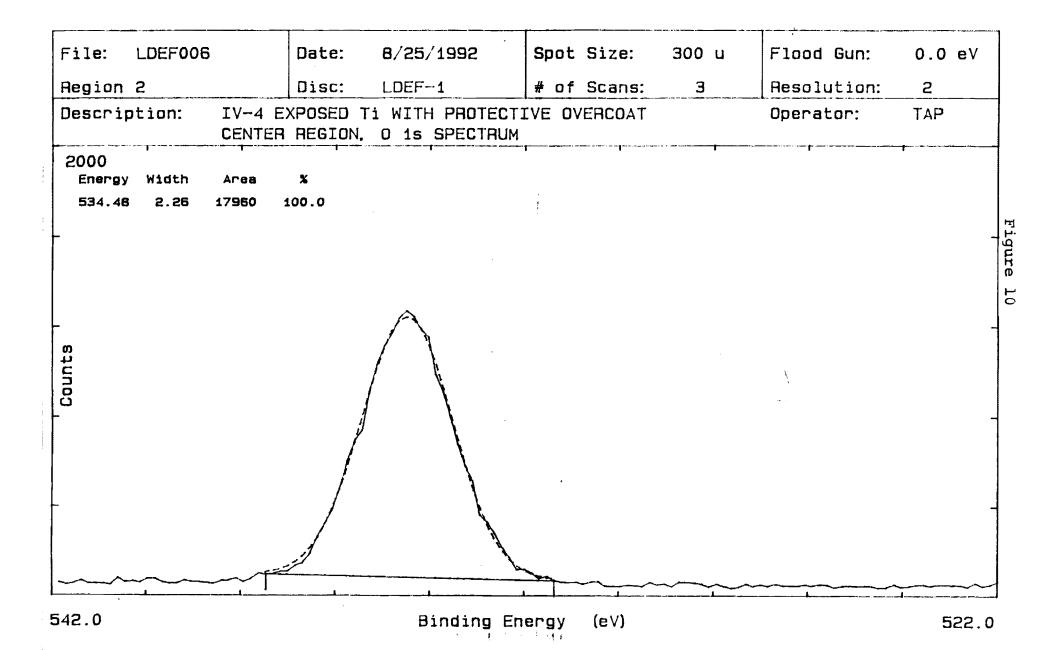
Binding Energy (eV)

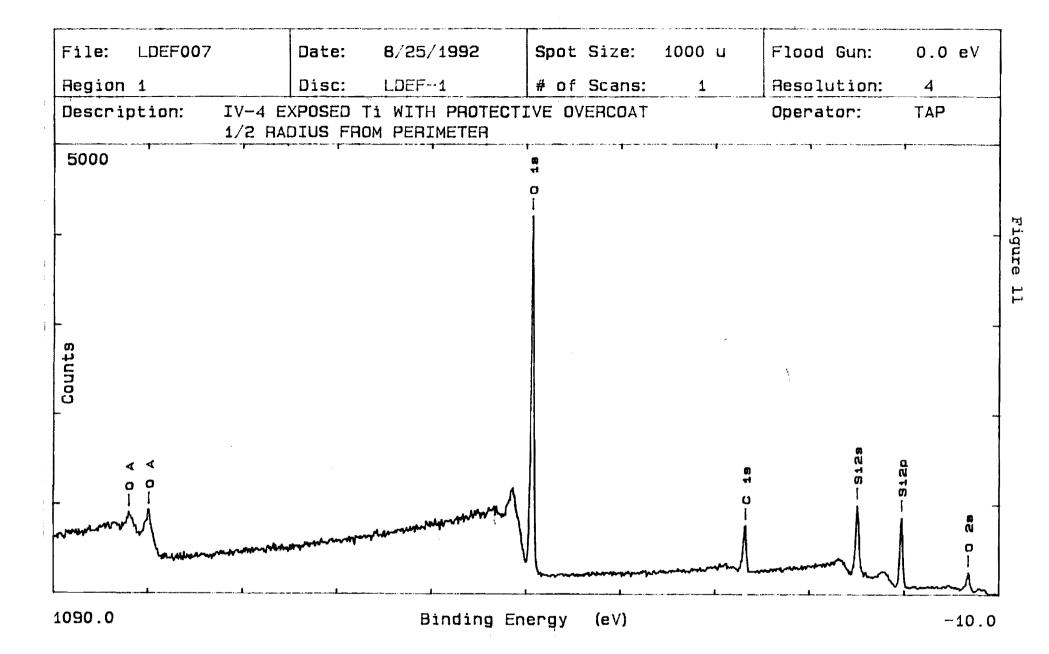


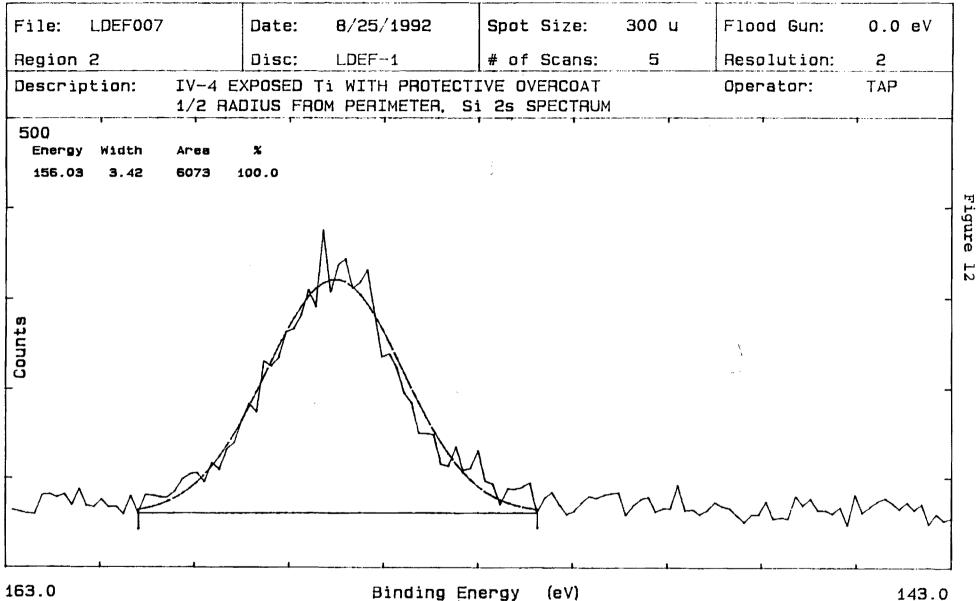
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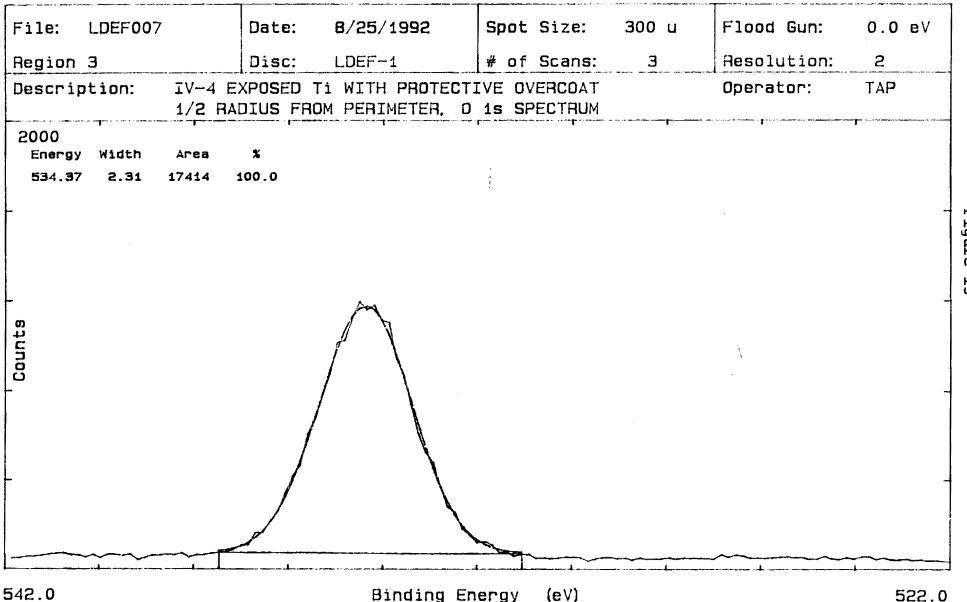




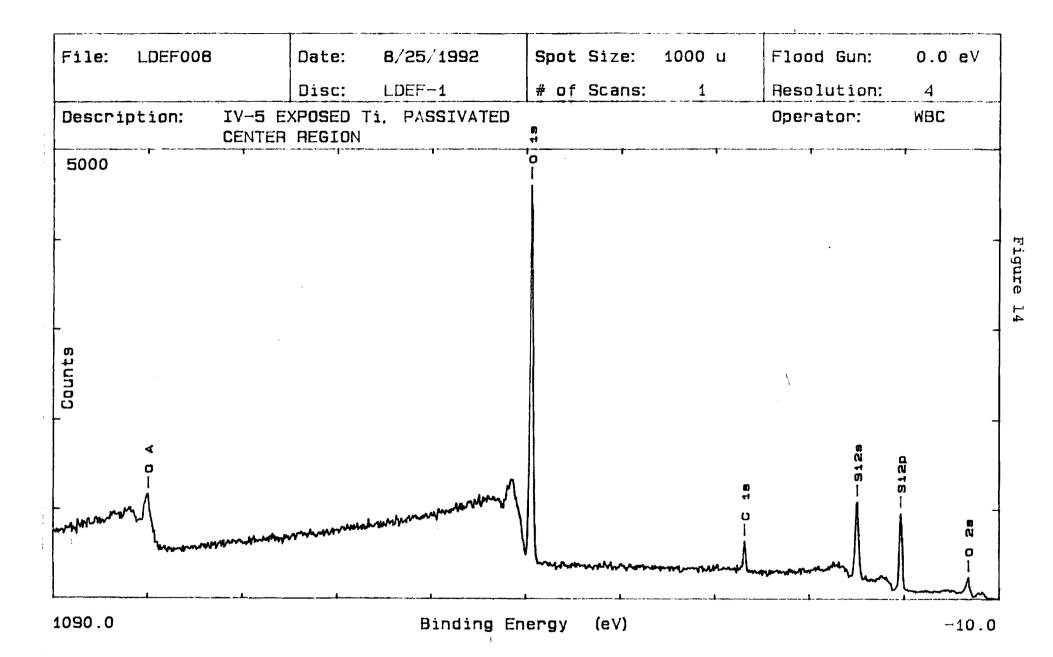


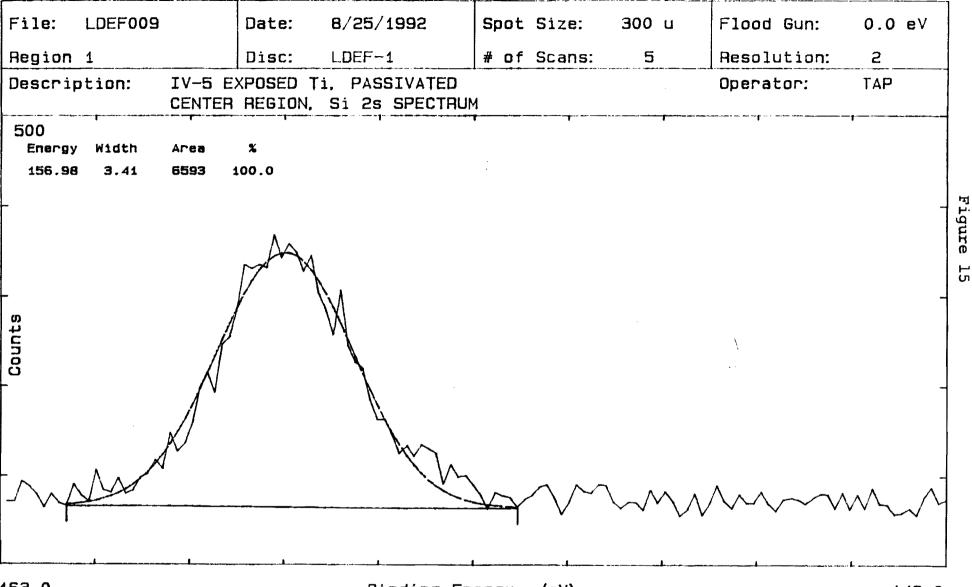


(eV) Binding Energy

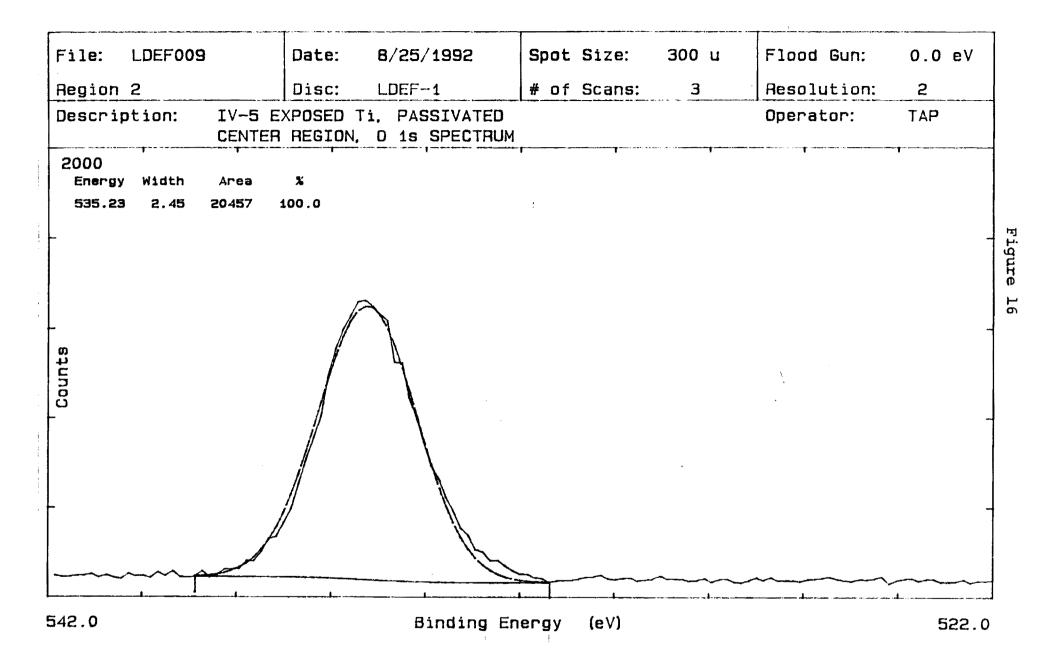


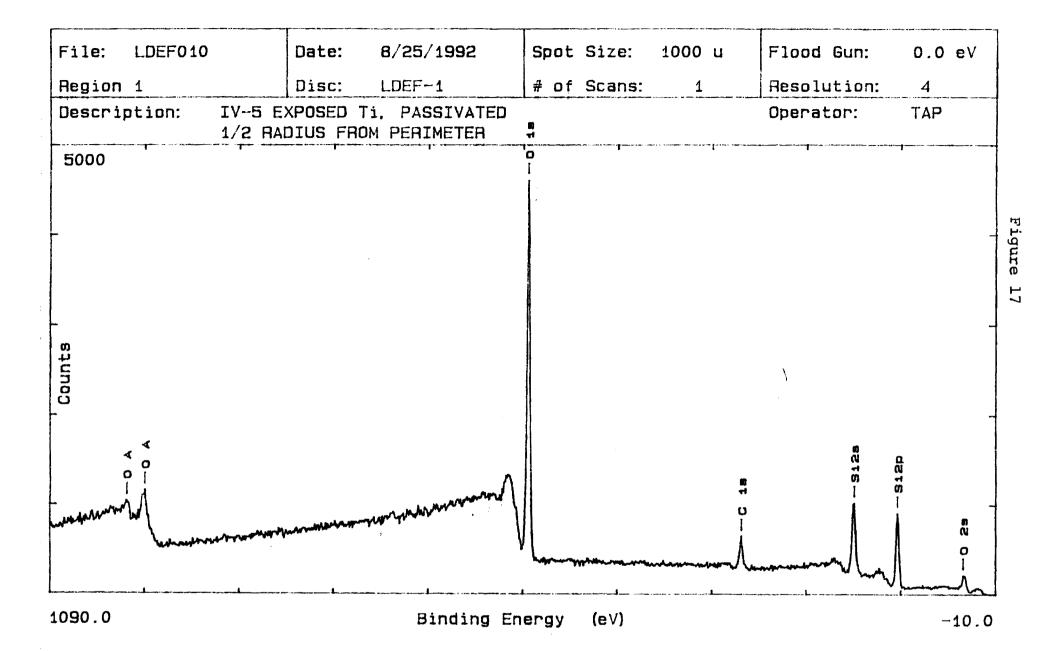
Binding Energy

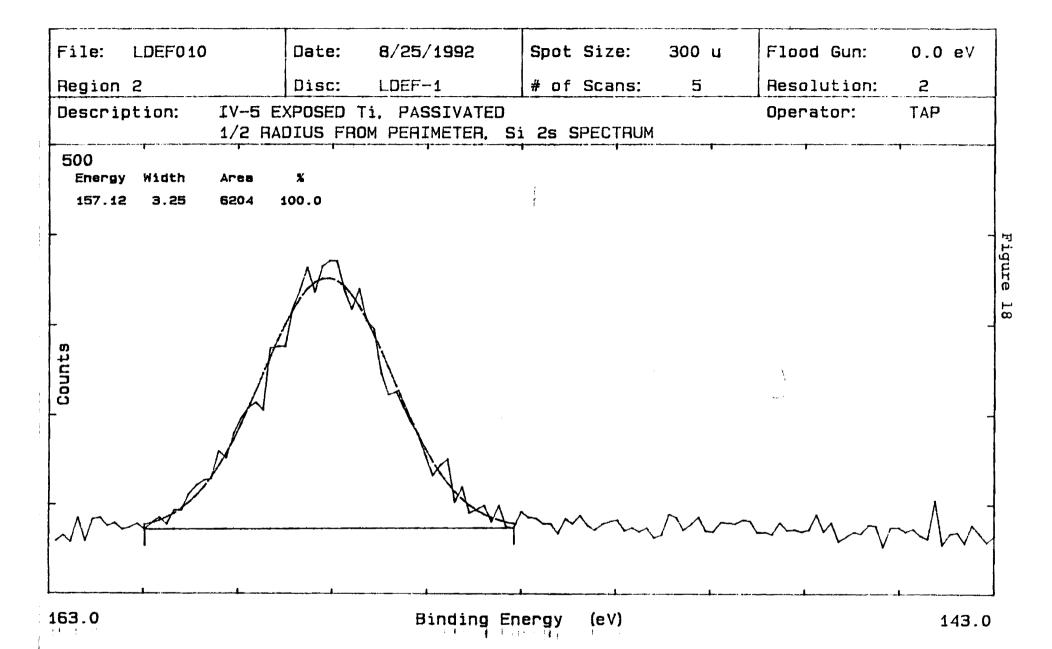


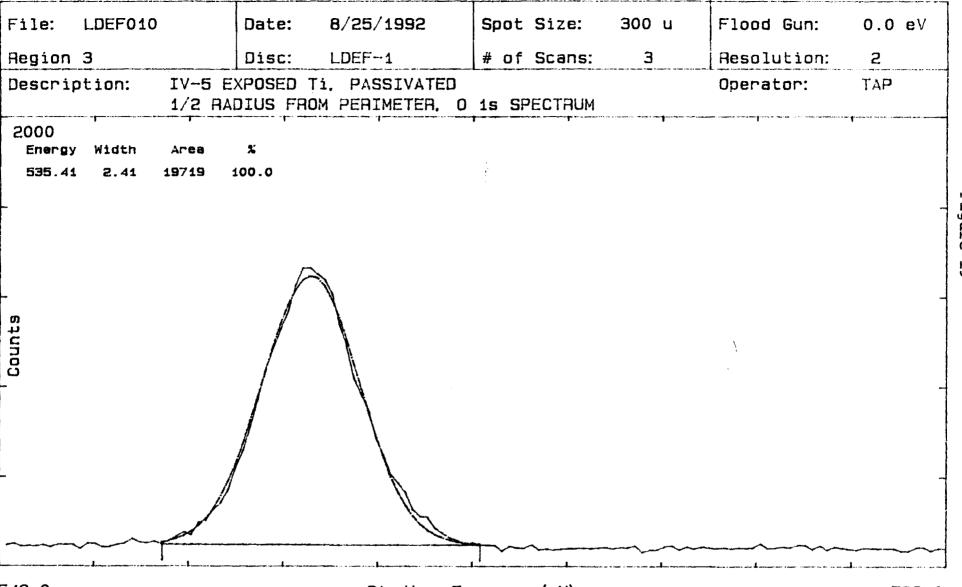


Binding Energy (eV)

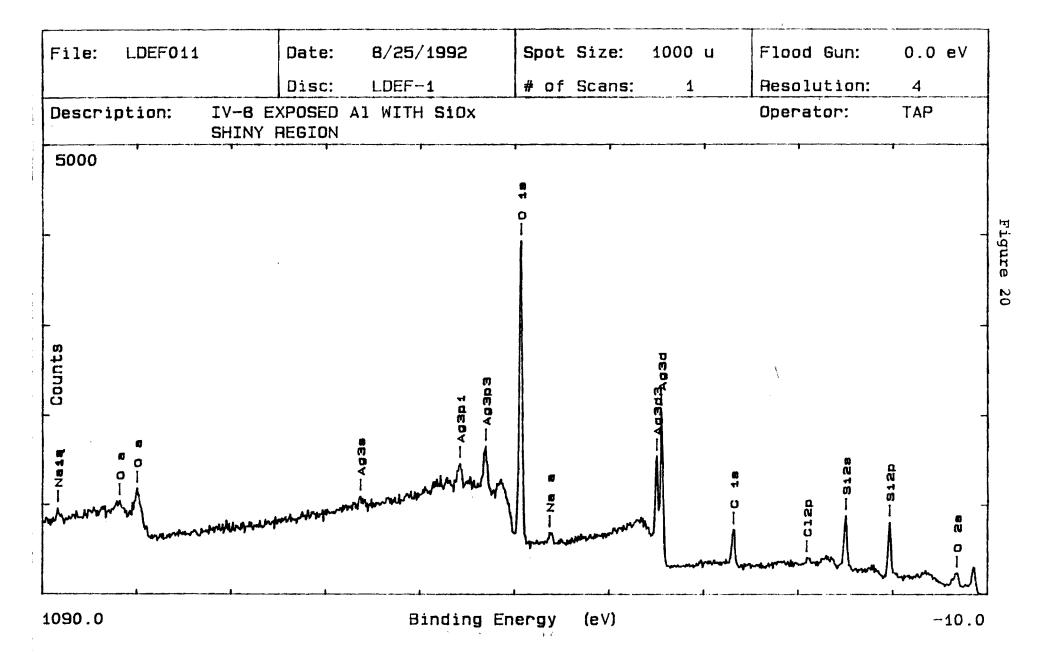








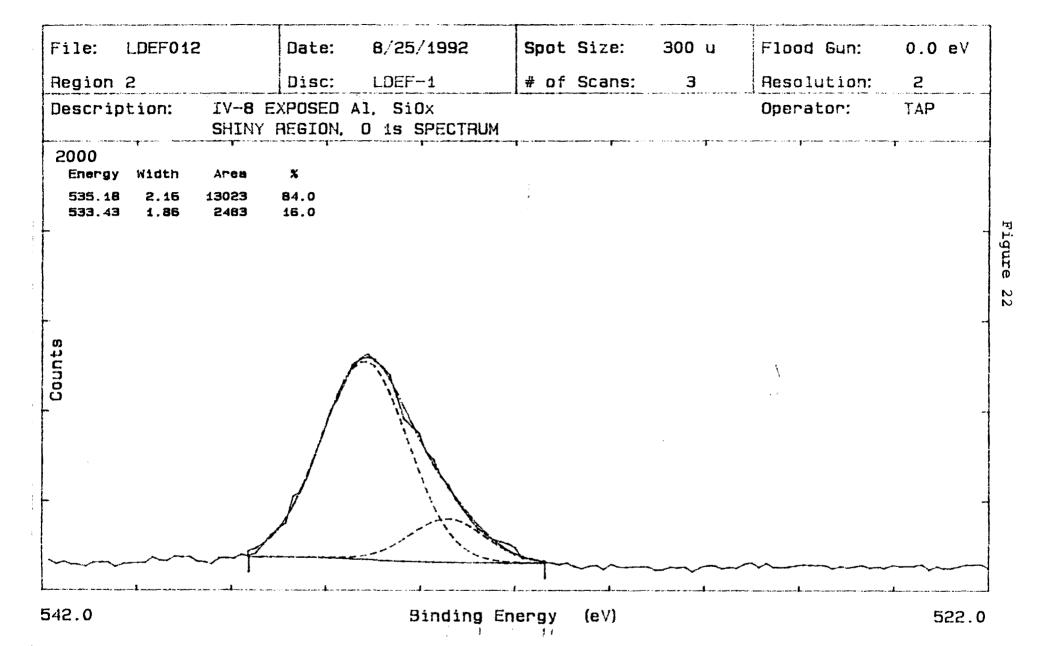
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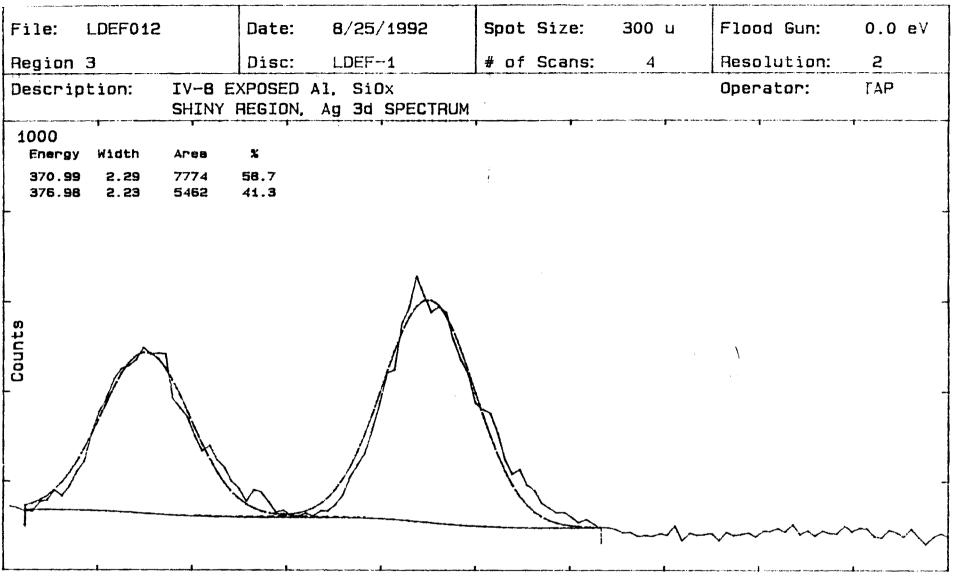


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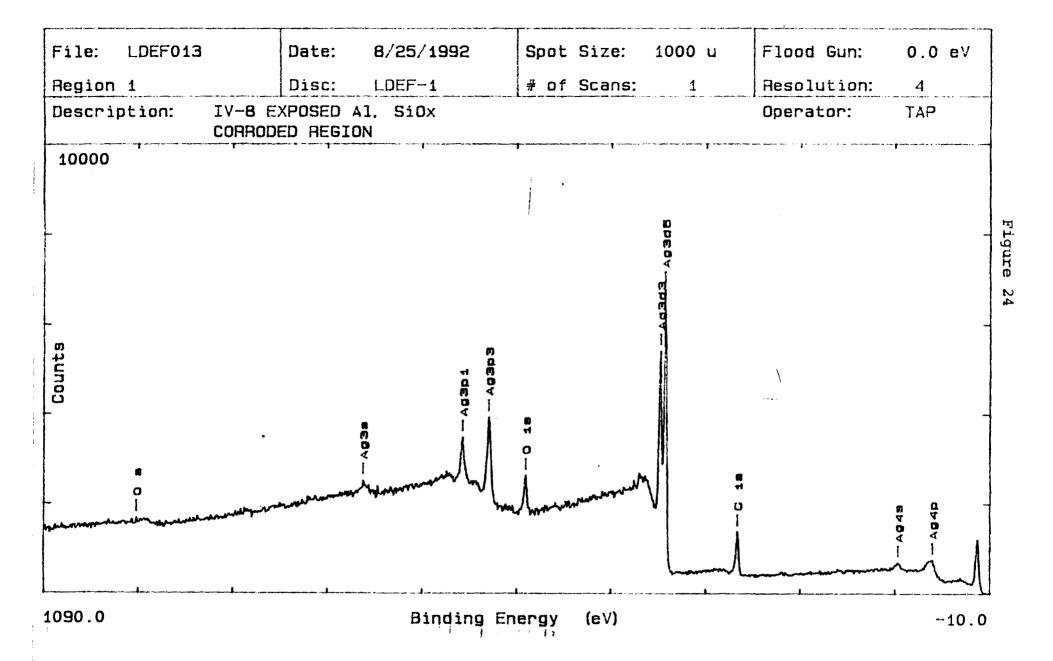
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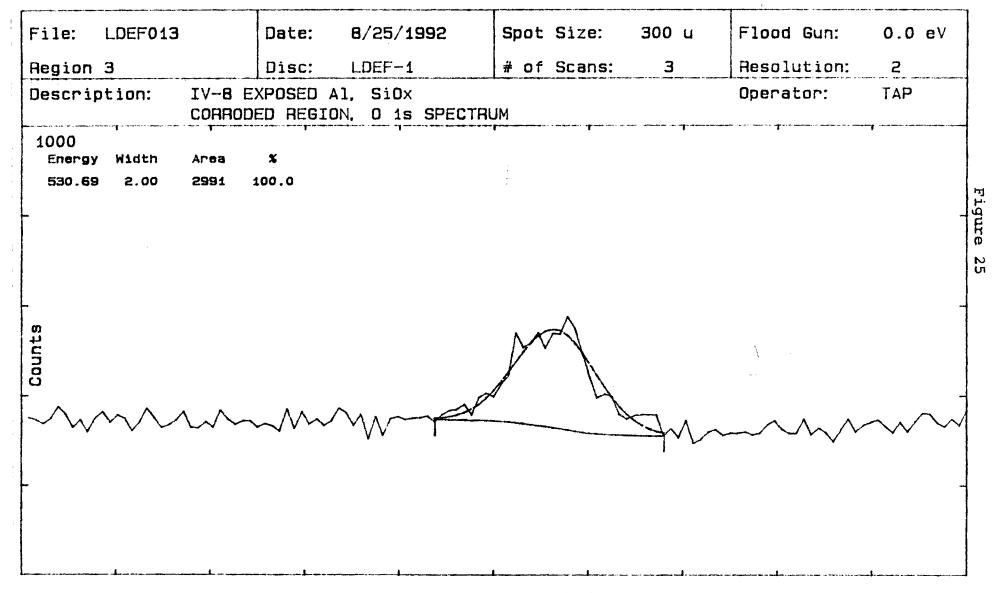
143.0



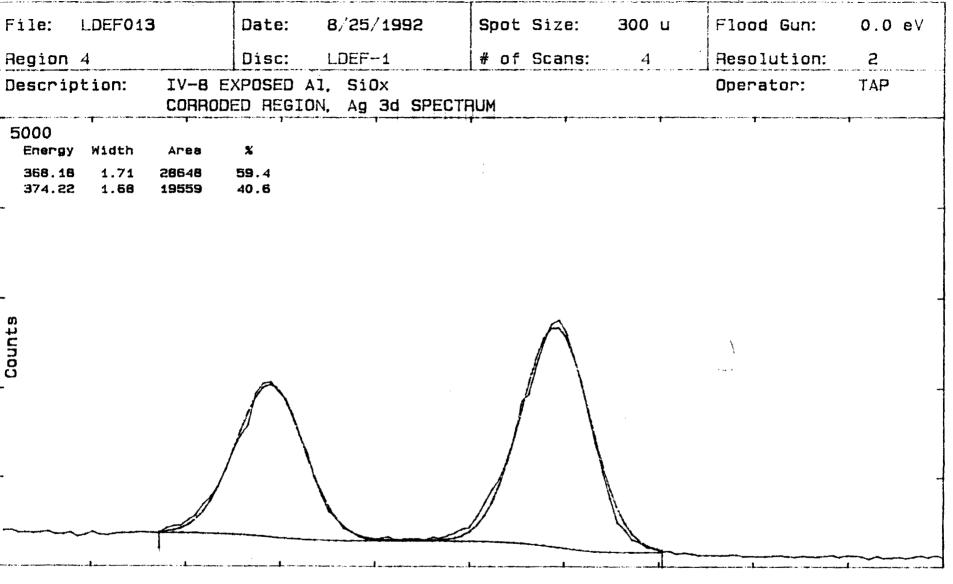


Binding Energy (eV)



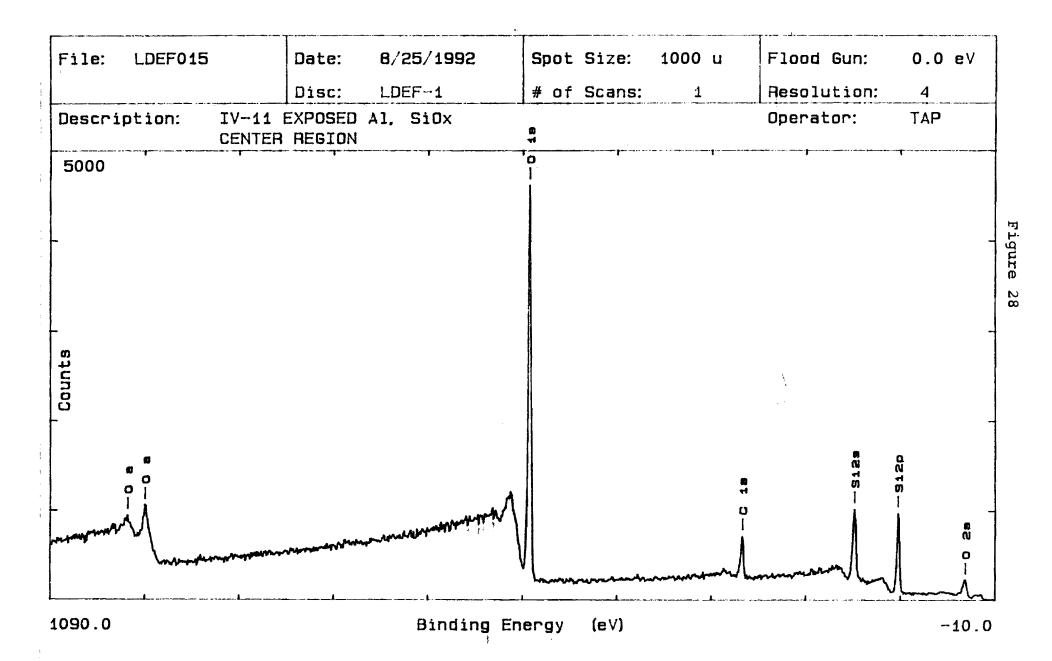


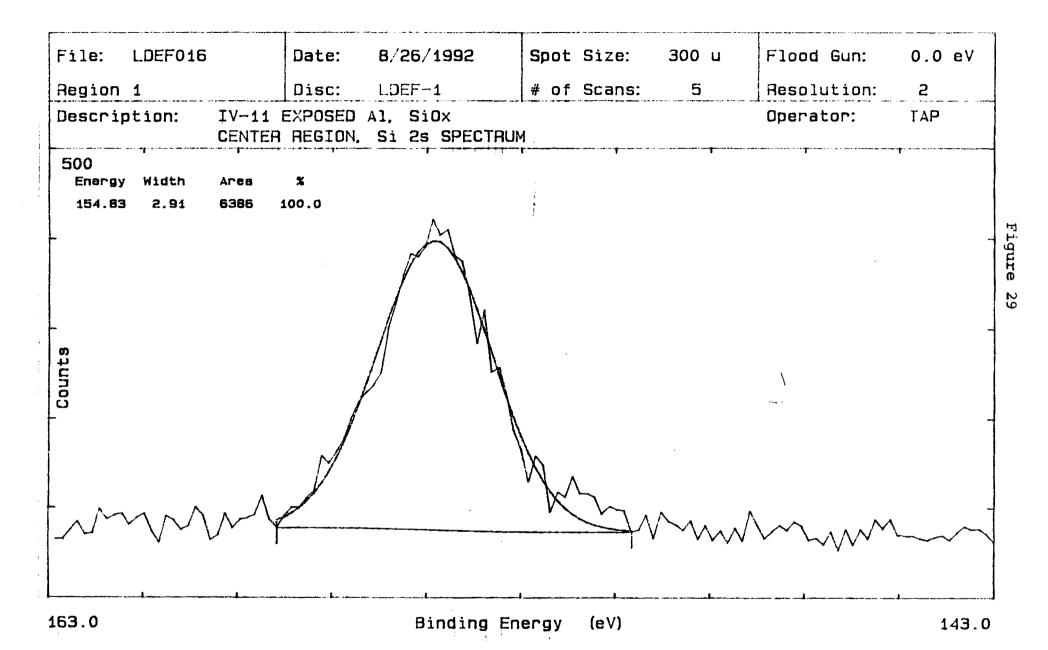
542.0 Binding Energy (eV) 522.0

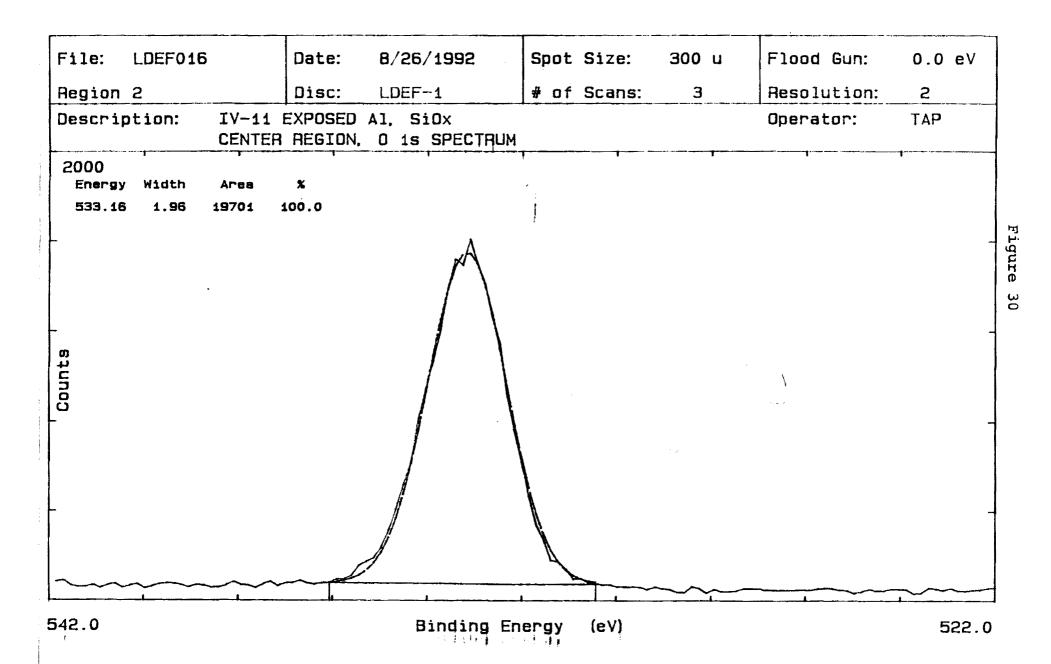


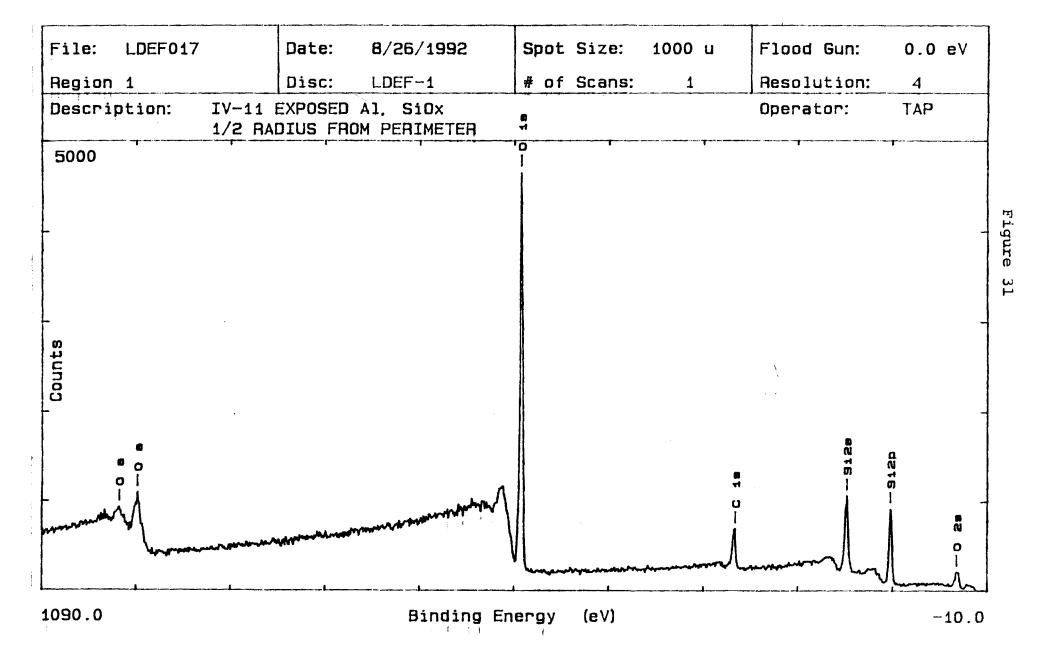
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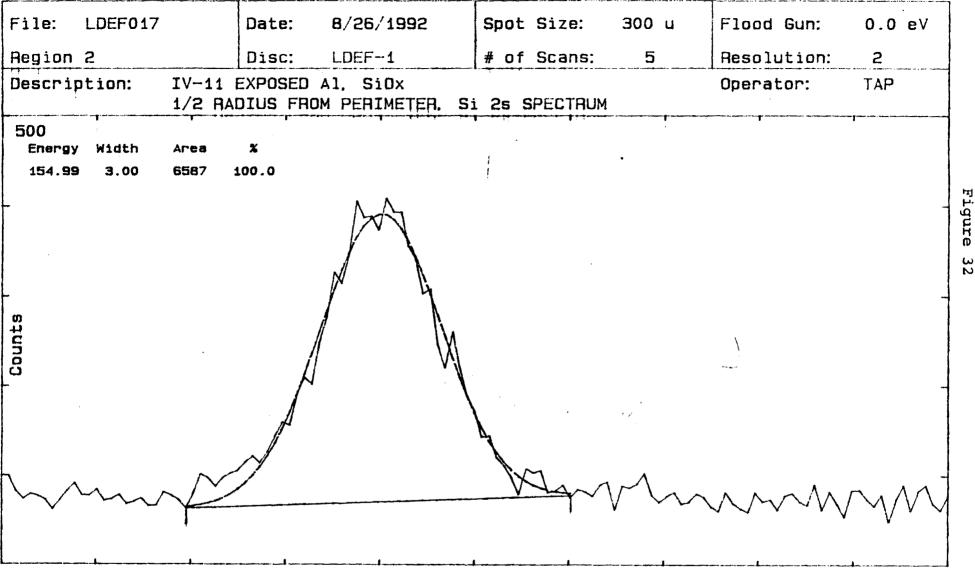
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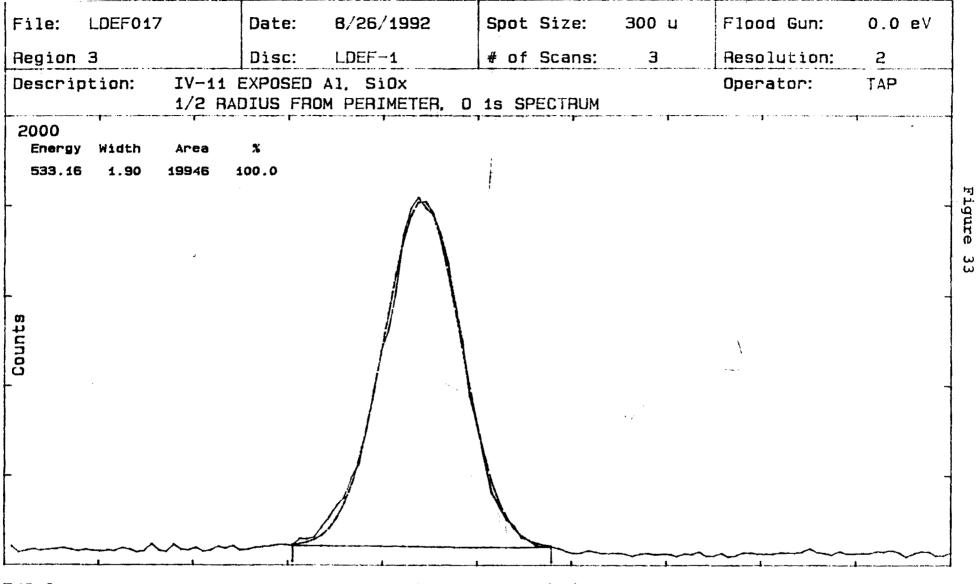




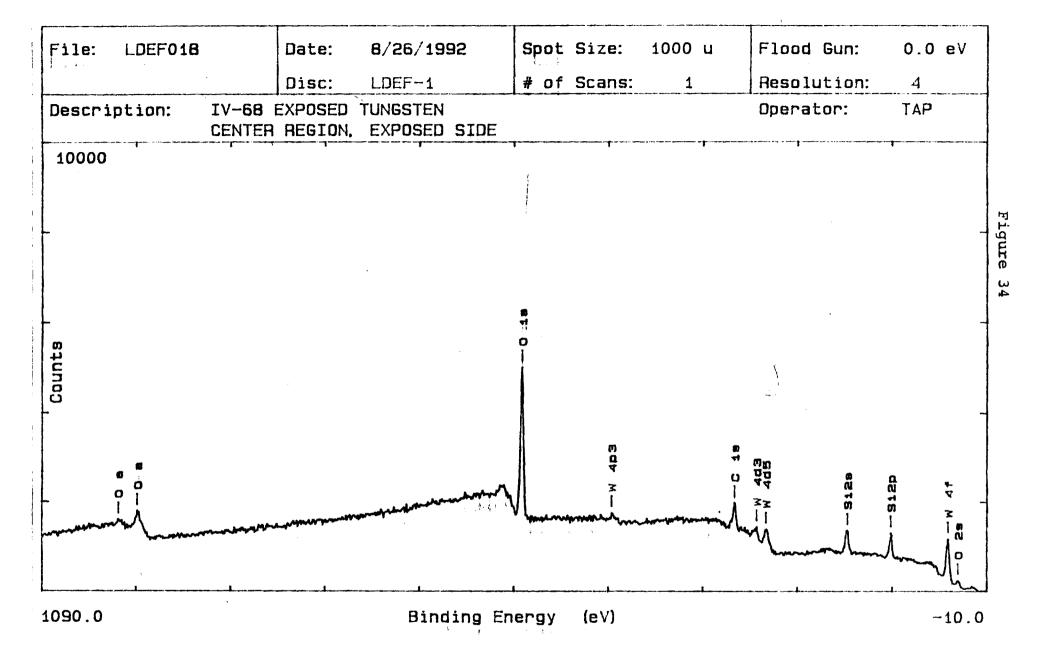


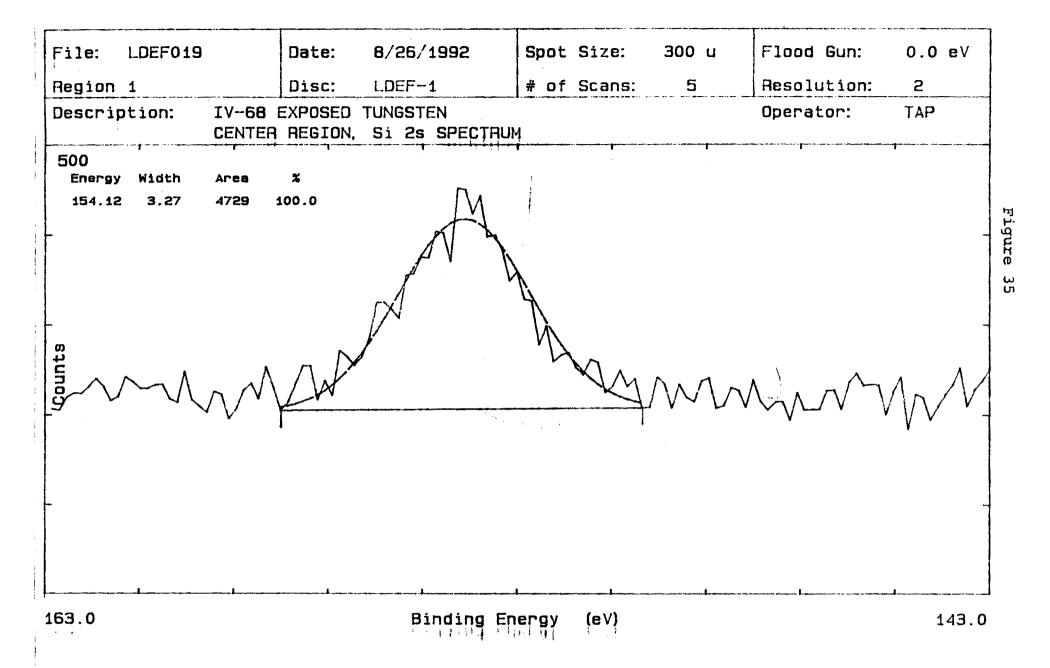


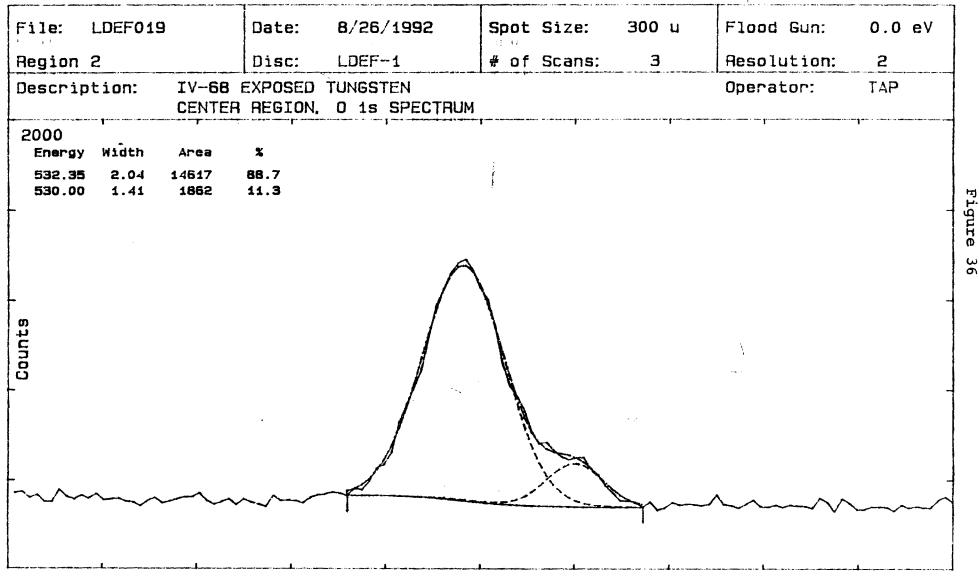
Binding Energy (eV)



Binding Energy (eV)





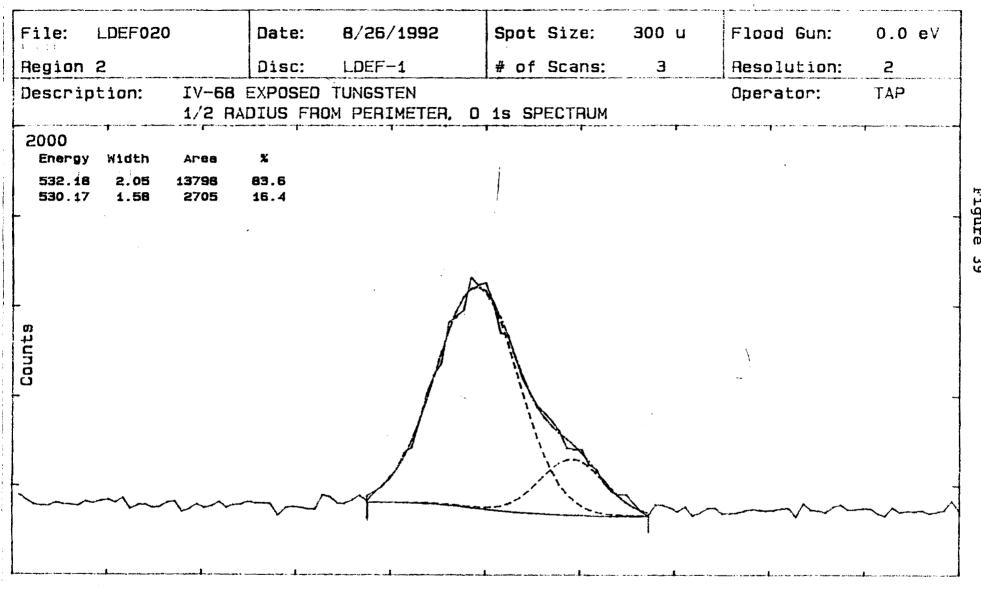


Binding Energy (eV)

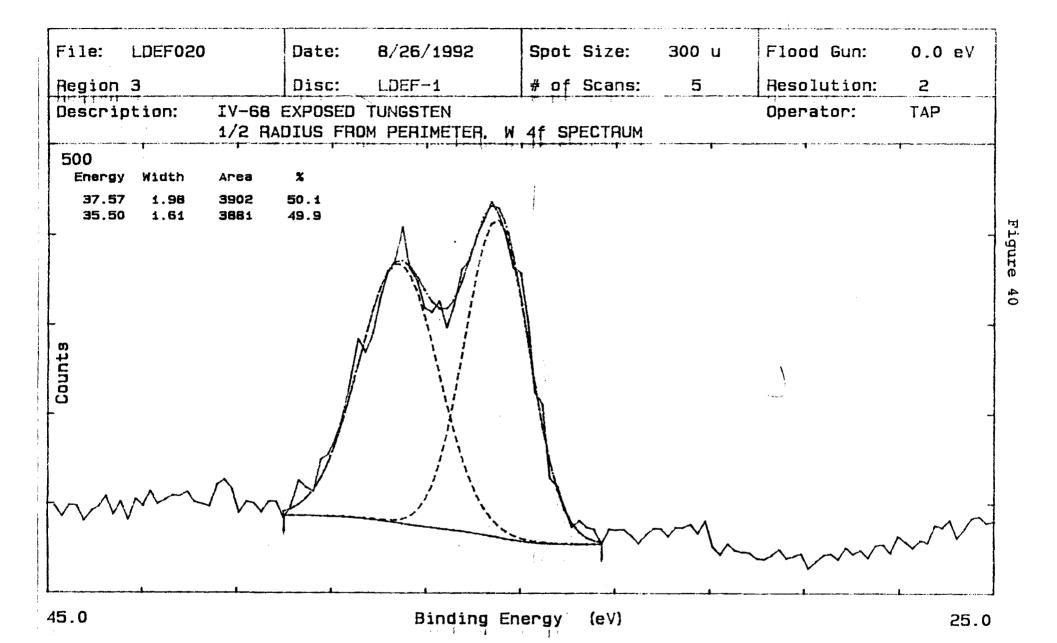
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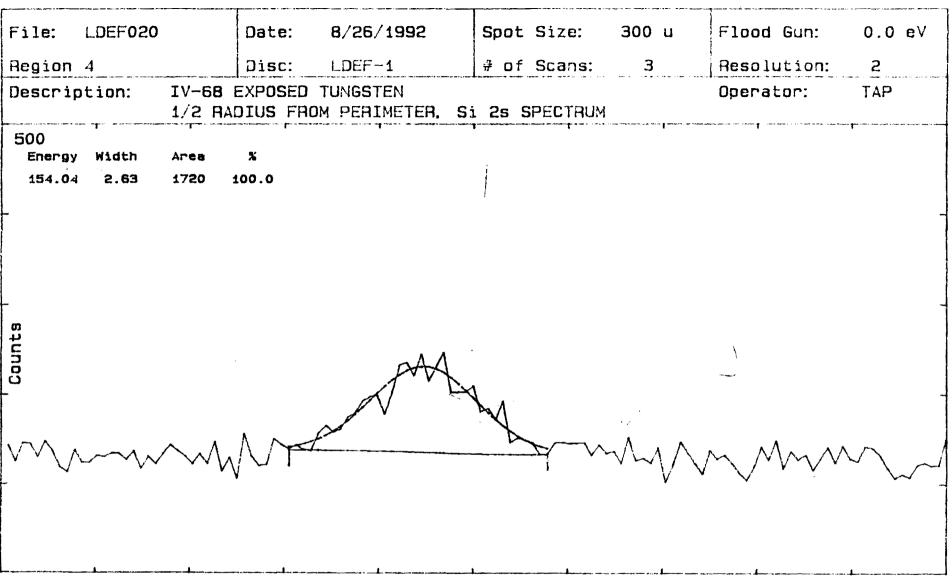
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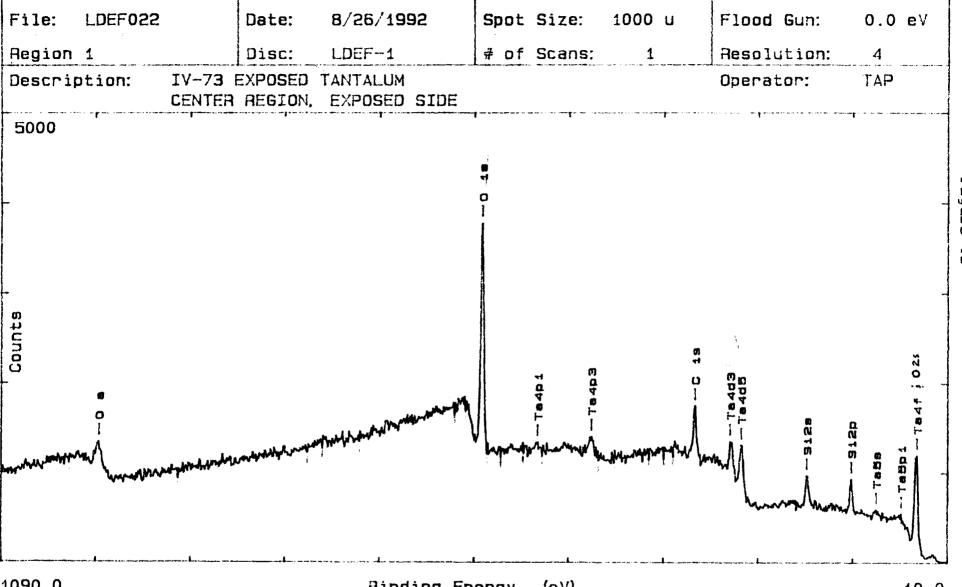


Binding Energy (eV)



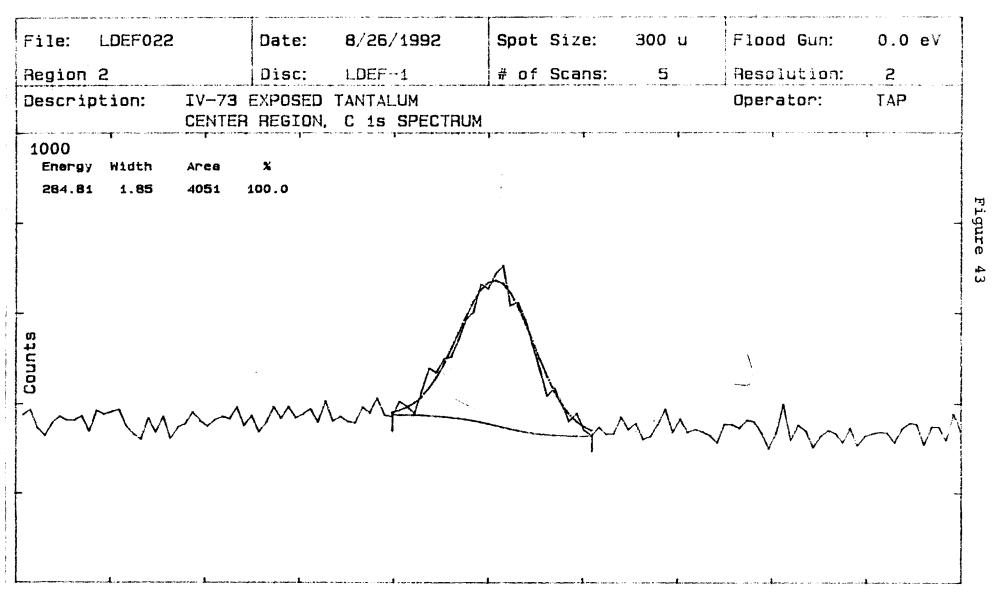


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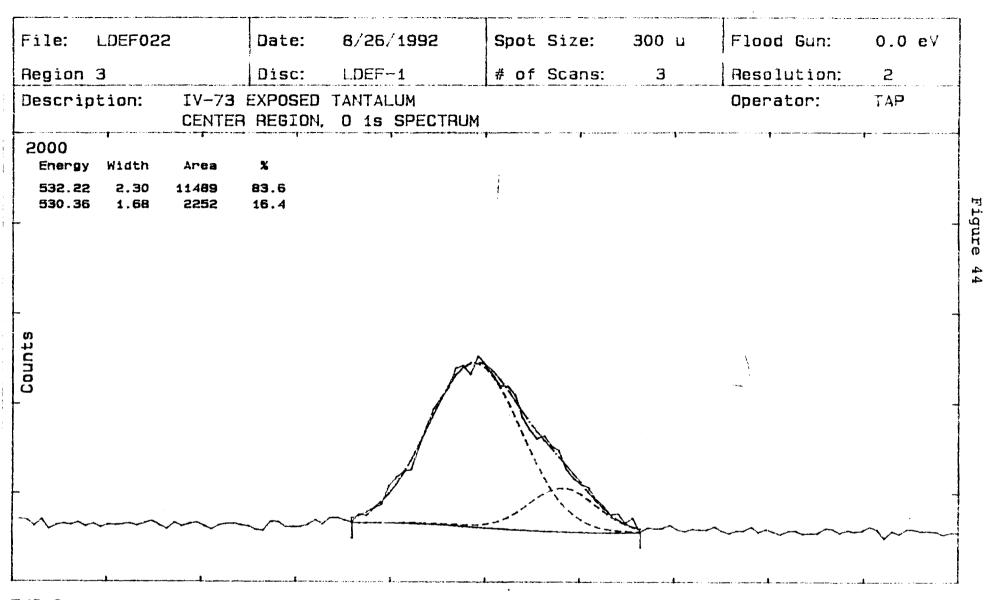


(eV) Binding Energy

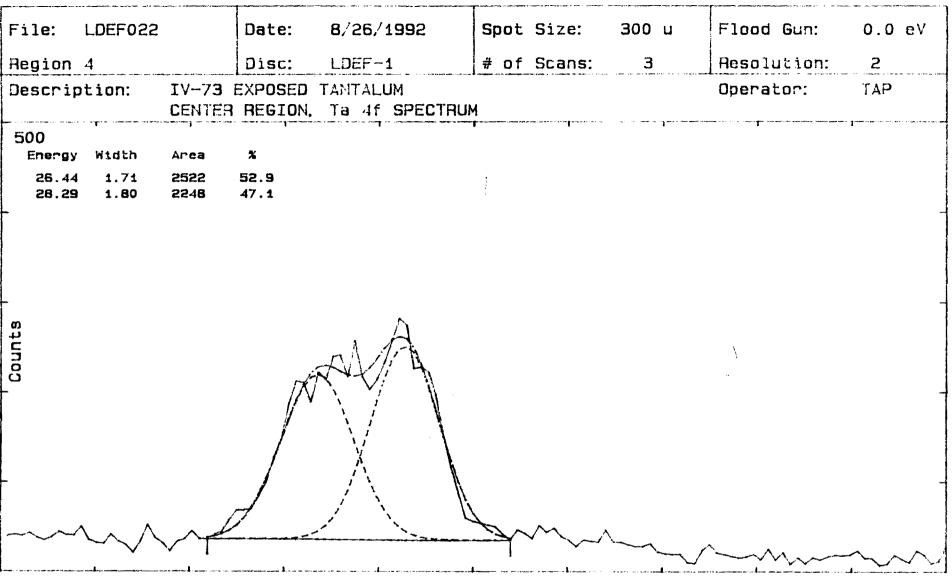
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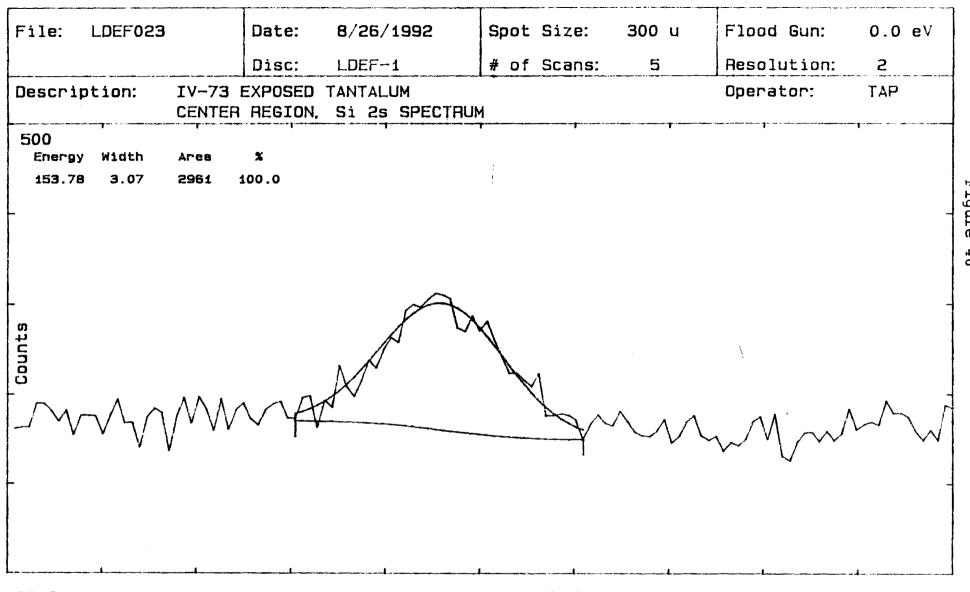
Binding Energy (eV)



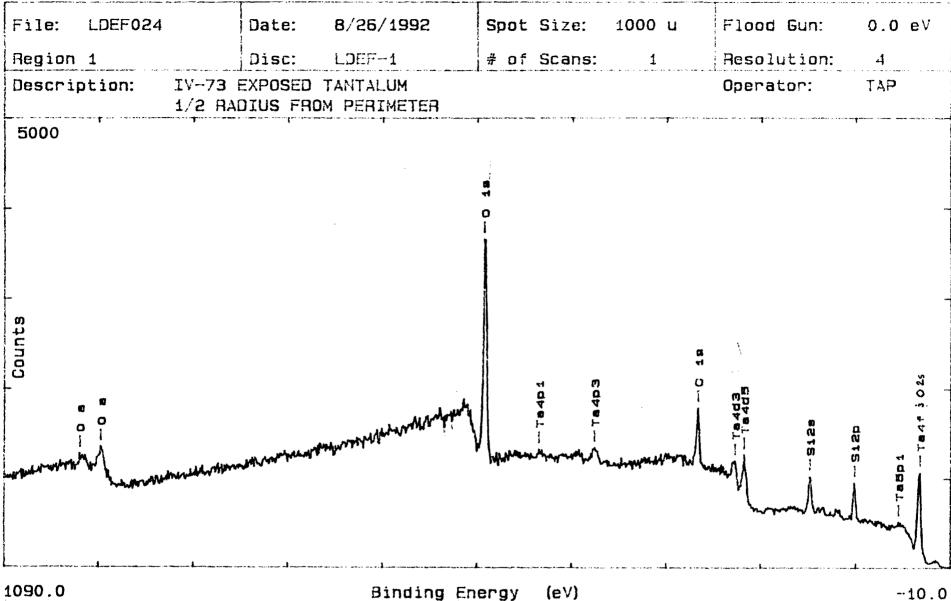
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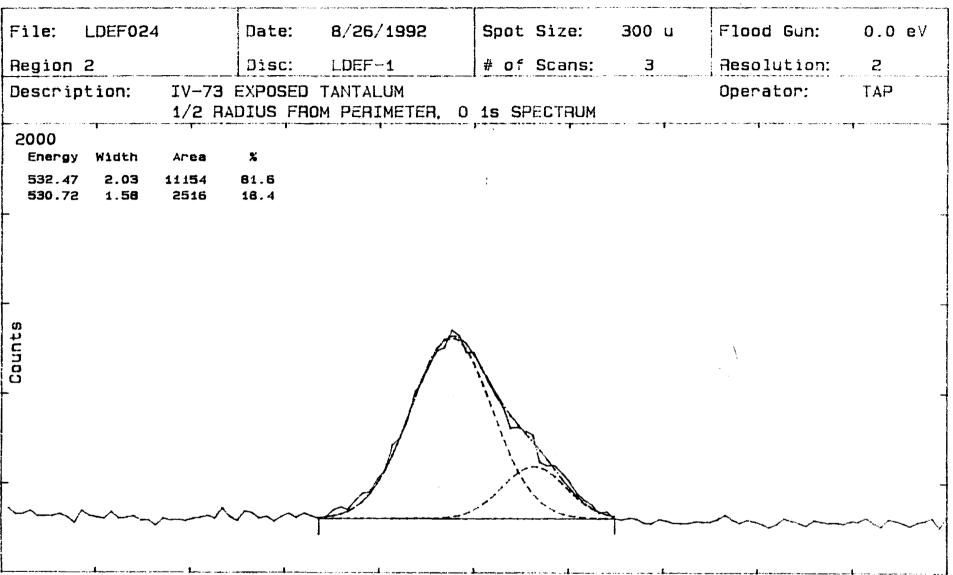
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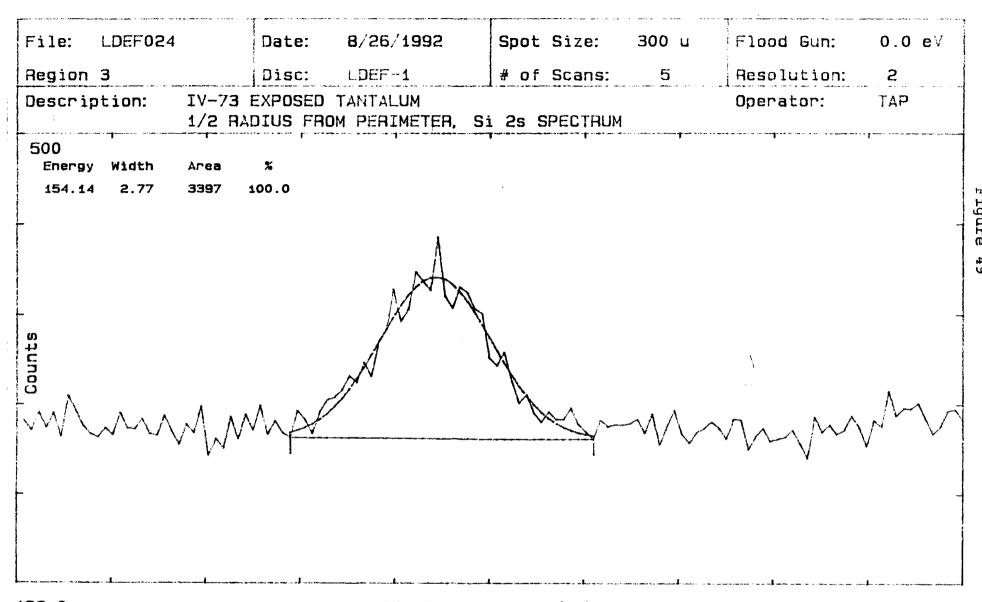
Binding Energy (eV)



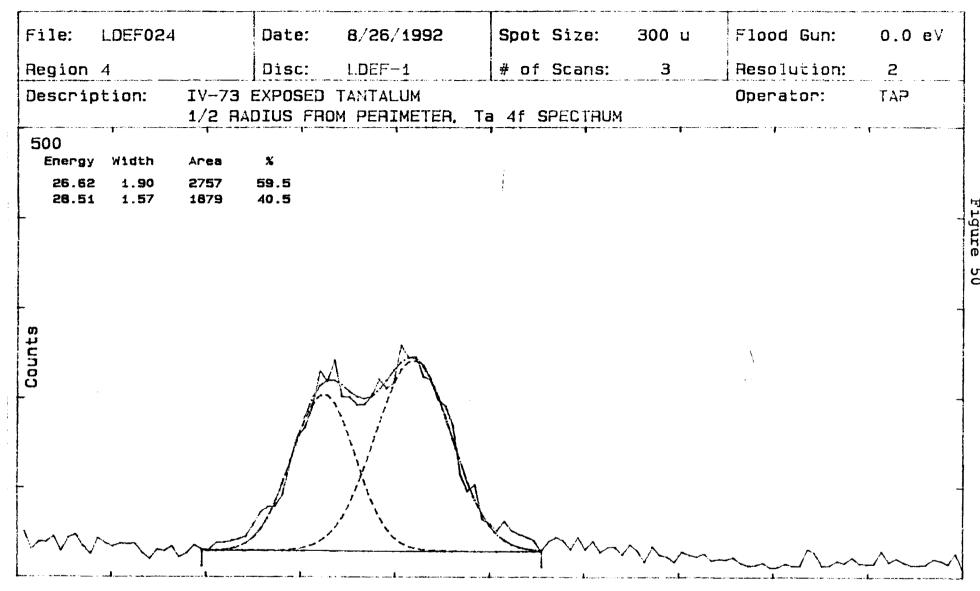
Binding Energy



542.0 Binding Energy (eV) 522.0



Binding Energy (eV)



Binding Energy (eV)

£15.7

# ELECTRON SPECTROSCOPY FOR CHEMICAL ANALYSIS -- SAMPLE ANALYSIS REPORT # LDEF-02

Prepared by:

W.B. Carter
School of Materials Science and Engineering
Georgia Institute of Technology
Atlanta, GA 30332-0245

for:

NASA/MSFC Marshall Space Flight Center, AL 35812

under:

PO # H-13010D

September 14, 1992

This document reports the results of ESCA analyses performed on the following samples:

CM01-15, CM01-24, CM01-31, CM01-45, CM02-15

A general description of the ESCA data is contained in previous report # LDEF-01. A spot near the center and a spot about midway between the center and the perimeter (off center region) of each sample were examined. After collecting data from two spots on each sample, a spot near the center of each was examined, sputtered for 2 minutes with 4 keV Ar⁺ ions and reexamined. Reports of the sample analyses follow.

#### Reports on ESCA Analyses

Prior to sputtering, all samples produced similar spectra comprised primarily of C, O and Si. The single exception being that the center region of sample CM01-24 displayed a small amount of Mg. Mg was not observed on the off center region of this sample nor was Mg observed near its center after sputtering. Varying amounts of C were observed on these samples prior to sputtering ranging from about 17 atomic percent on CM01-45 to about 38 atomic percent on CM01-15.

Sputtering affected the surface composition of each sample generally by reducing the atomic percent C present and by decreasing the O/Si ratio. The single exception to this is sample CM01-31 on which the O/Si ratio increased after sputtering. The C on sample CM01-45 was reduced below detectability by 2 minutes of sputtering.

Prior to sputtering, the samples did not charge under the x-ray beam. They did charge after sputtering. This indicates that the sample surfaces were initially covered with electrically conductive layers that were removed by sputtering.

#### CM01-15: Control mirror, SiO, on aluminum

Figures 1 - 8 are the spectra taken from the two spots prior to sputtering. Figure 9 is a spectrum taken of the center region which was then sputtered for two minutes prior to collecting the spectrum shown in figure 10.

Table 1: Semiquantitative Composition of Center Region of Sample CM01-15 from Figure 1 Prior to Sputtering

Approximate Atomic Percent
39
22
39

Table 2: Semiquantitative Composition of Off Center Region of Sample CM01-15 from Figure 5 Prior to Sputtering

Element	Approximate Atomic Percent
0	42
Si	20
С	38

Table 3: Semiquantitative Composition of Center Region of Sample CM01-15 from Figure 10 After Sputtering

Element	Approximate Atomic Percent
0	34
Si	32
С	34
C	

#### CM01-24: Exposed mirror, SiO, on aluminum

Figures 11 - 18 are the spectra taken from the two spots prior to sputtering. Figure 19 is a spectrum taken of the center region which was then sputtered for two minutes prior to collecting the spectrum shown in figure 20.

The presence of Mg near the center of this sample is indicated by the Mg Auger peak seen in the general survey of this area. It was not possible to quantify the amount of Mg present because of the unavailability of sensitivity data for Auger peaks appearing in ESCA spectra.

Table 4: Semiquantitative Composition of Center Region of Sample CM01-24 from Figure 11 Prior to Sputtering

Element	Approximate Atomic Percent
0	48
Si	29
C	23

Table 5: Semiquantitative Composition of Off Center Region of Sample CM01-24 from Figure 15 Prior to Sputtering

Element	Approximate Atomic Percent
0	51
Si	29
С	20

Table 6: Semiquantitative Composition of Center Region of Sample CM01-24 from Figure 20 After Sputtering

Element	Approximate Atomic Percent
0	54
Si	41
C	5

## CM01-31: Exposed mirror (windowed), SiO, on aluminum

Figures 21 - 26 are the spectra taken from the two spots prior to sputtering. Figure 27 is a spectrum taken of the center region which was then sputtered for two minutes prior to collecting the spectrum shown in figure 28.

Table 7: Semiquantitative Composition of Center Region of Sample CM01-31 from Figure 21 Prior to Sputtering

Element	Approximate Atomic Percent
o	51
si	30
c	19

Table 8: Semiquantitative Composition of Off Center Region of Sample CM01-31 from Figure 24 Prior to Sputtering

Element	Approximate Atomic Percent
o	51
Si	30
C	19

Table 9: Semiquantitative Composition of Center Region of Sample CM01-31 from Figure 28 After Sputtering

Element	Approximate Atomic Percent
0	63
Si	27
С	9

## CM01-45: Exposed mirror, SiO_x on aluminum

Figures 29 - 36 are the spectra taken from the two spots prior to sputtering. Figure 37 is a spectrum taken of the center region which was then sputtered for two minutes prior to collecting the spectrum shown in figure 38.

Table 10: Semiquantitative Composition of Center Region of Sample CM01-45 from Figure 29 Prior to Sputtering

Element	Approximate Atomic Percent
0	51
Si	31
C	18

Table 11: Semiquantitative Composition of Off Center Region of Sample CM01-45 from Figure 33 Prior to Sputtering

Element	Approximate Atomic Percent
0	52
Si	32
C	16

Table 12: Semiquantitative Composition of Center Region of Sample CM01-45 from Figure 38 After Sputtering

Element	Approximate Atomic Percent
O	58
Si	42

## CM02-15: Exposed mirror, SiO, on Al, Trailing Edge

Figures 39 - 44 are the spectra taken from the two spots prior to sputtering. Figure 45 is a spectrum taken of the center region which was then sputtered for two minutes prior to collecting the spectrum shown in figure 46.

Table 13: Semiquantitative Composition of Center Region of Sample CM02-15 from Figure 39 Prior to Sputtering

Element	Approximate Atomic Percent
o	38
si	25
c	37

Table 14: Semiquantitative Composition of Off Center Region of Sample CM02-15 from Figure 42 Prior to Sputtering

Element	Approximate Atomic Percent
<u> </u>	39
si c	27 34

Table 15: Semiquantitative Composition of Center Region of Sample CM02-15 from Figure 46 After Sputtering

Element	Approximate Atomic Percent
o Si	51 42
С	7

#### Silica Standard

In an effort to calibrate the ESCA technique in regard to O/Si ratios on  $SiO_x$  covered samples, spectra were collected from a piece of high purity silica  $(SiO_2)$ . The silica was first rinsed in acetone followed by a rinse in methanol. The silica was mounted on a sample holder under a fine nickel wire mesh screen, placed in the ESCA spectrometer and a general survey collected using the low energy electron flood gun. The nickel screen and flood gun controlled sample charging. The spectrum (figure 47) indicates the presence of O, C, Si and N in the approximate

atomic percentages displayed in table 16. The O/Si ratio from this spectrum is about 2.6.

After collecting this spectrum, the sample was heated in the ESCA system to an unknown temperature below 300°C. (The sample stage in which the sample holder sits was heated to 300°C.) After about three days at the elevated temperature, the spectrum shown in figure 48 was collected while the sample was hot. O, Si, C and N were present on the surface. Table 17 presents the results of the semiquantitative analysis of this spectrum. The O/Si ratio after heating is about 1.4.

The primary effect of heating seems to be to decrease the O/Si ration without affecting the C or N coverage. This is perhaps due to the desorption of water and other contaminants from the surface promoted by heating.

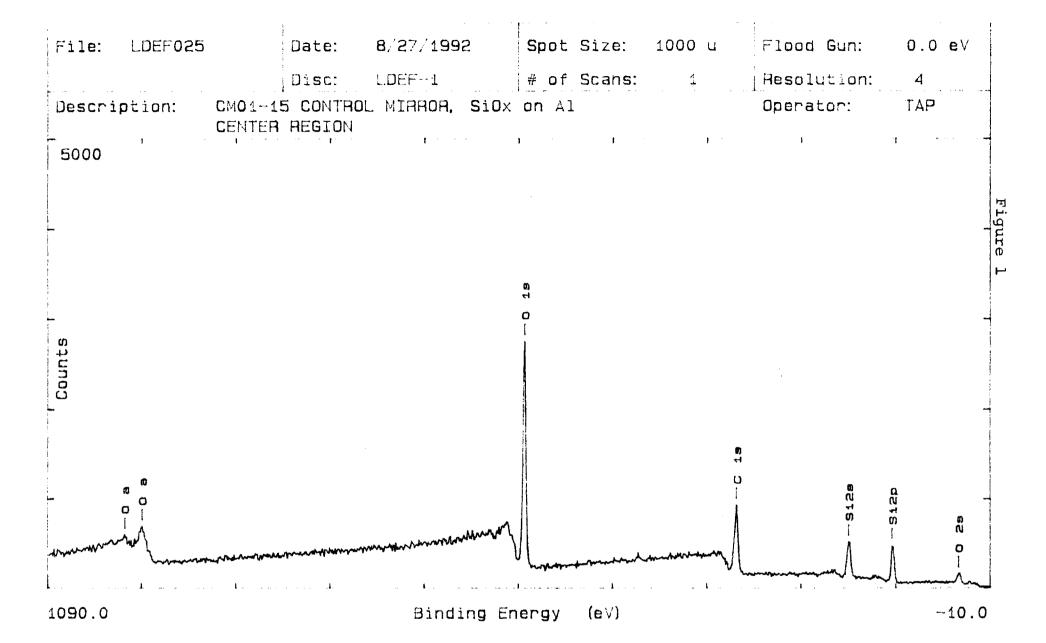
It is not feasible to sputter the silica surface while it is under the nickel screen. Because of the variability of the O/Si ratio, the substantial amounts of C and N present on the silica standard after heating and the inability to sputter clean the standard as it was observed, it is not possible to yet determine what semiquantitative O/Si ratio would be obtained from a clean SiO₂ surface.

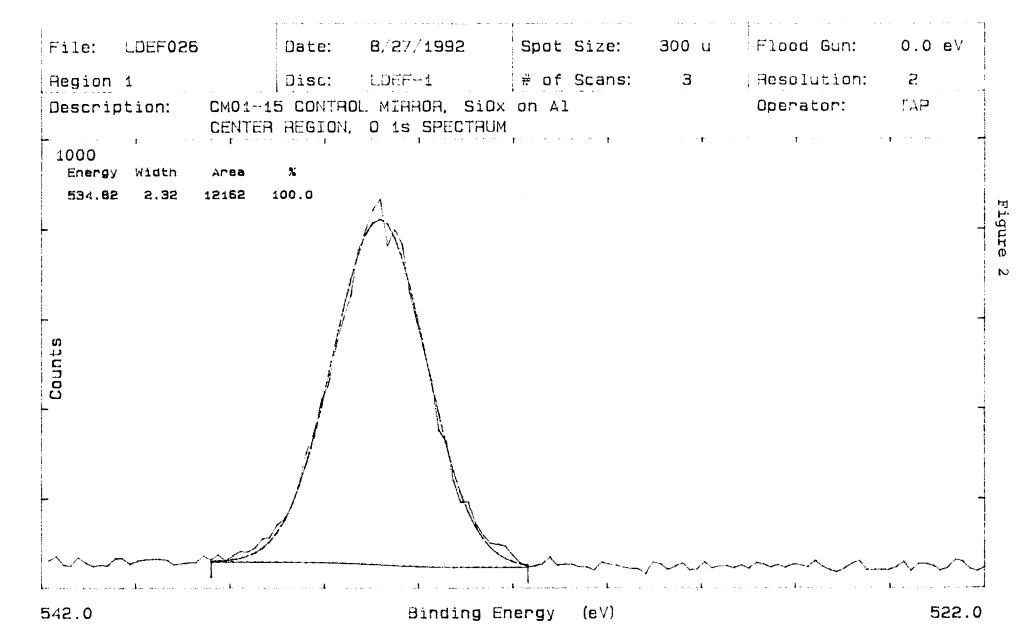
Table 16: Semiquantitative Composition of Silica Standard after Cleaning from Figure 47

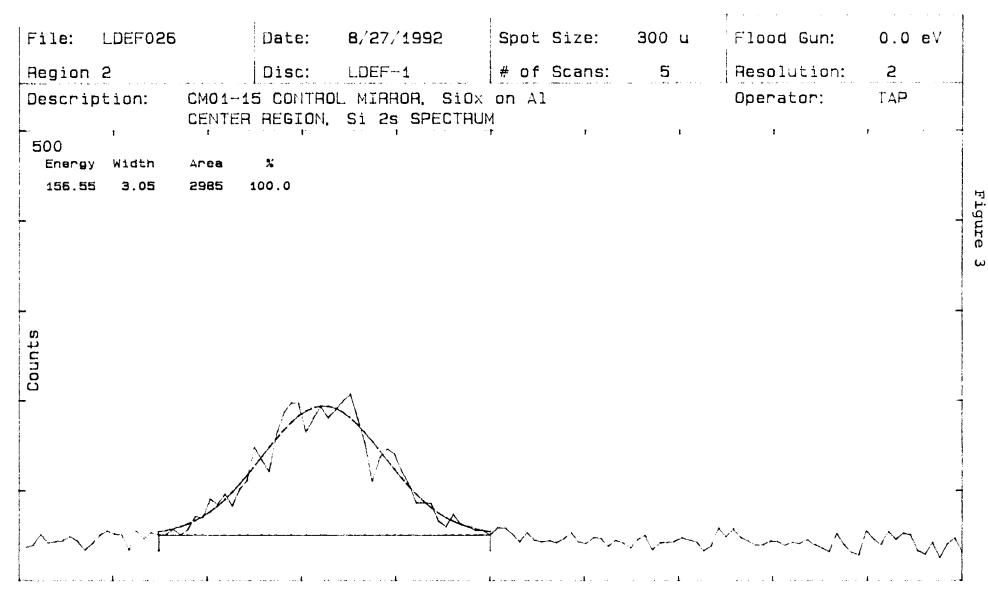
Element	Approximate Atomic Percent
0	42
Si	16
C	34
N	8

Table 17: Semiquantitative Composition of Silica Standard after About Three Days at an Elevated Temperature from Figure 48

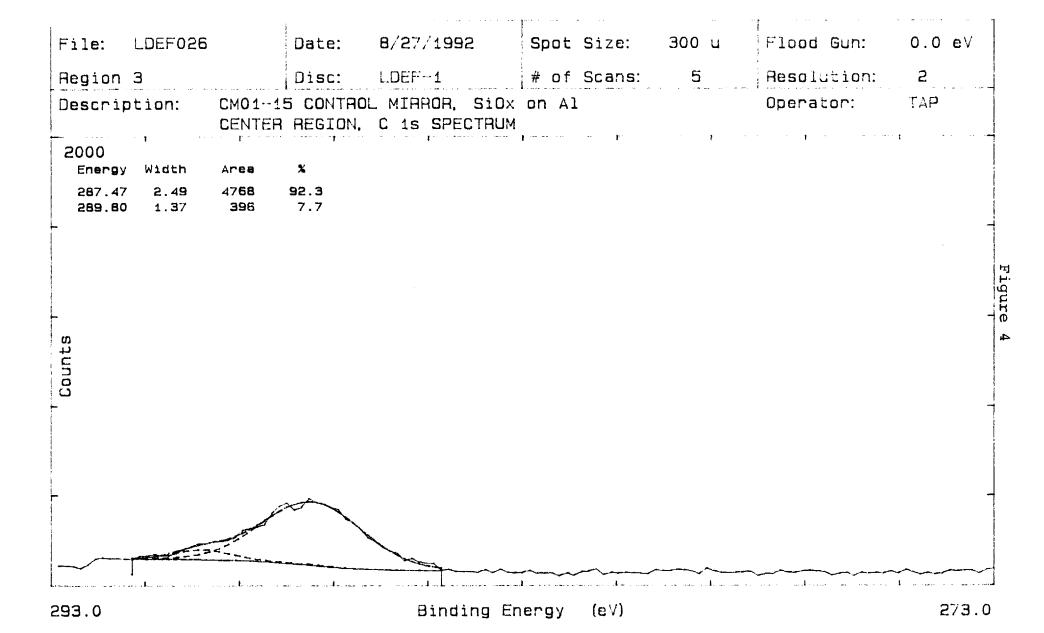
Element	Approximate Atomic Percent
0	34
Si	24
С	34
N	8

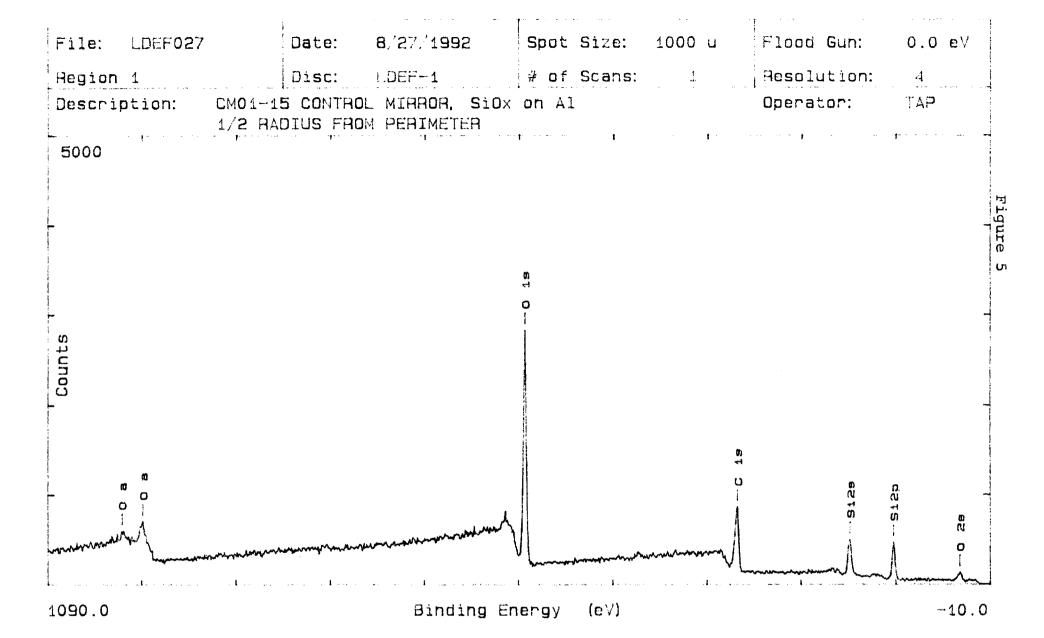


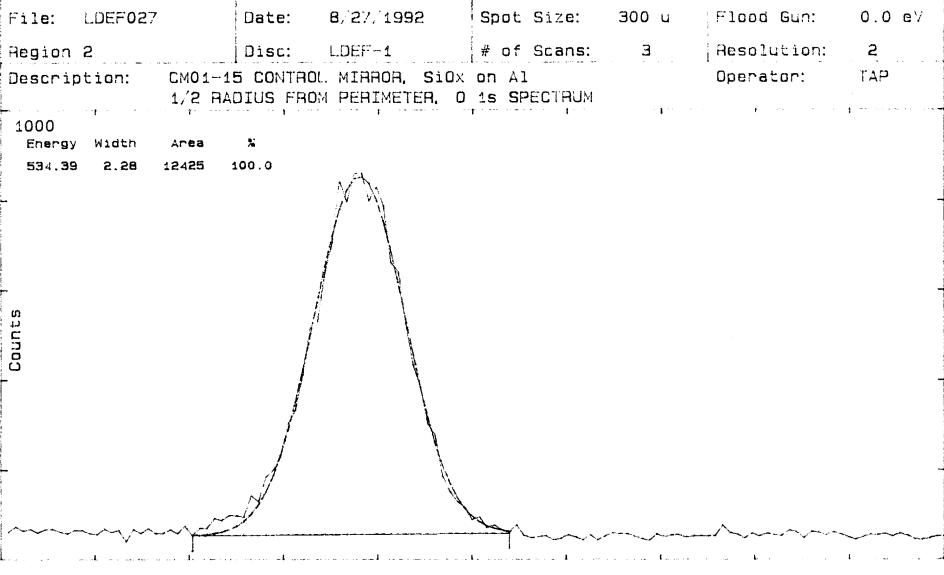




163.0 Binding Energy (eV) 143.0

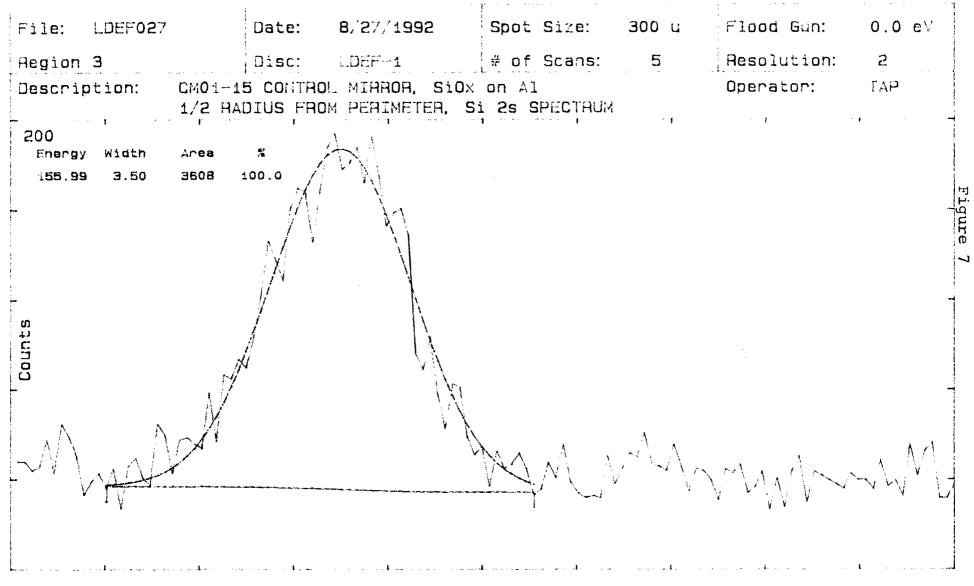




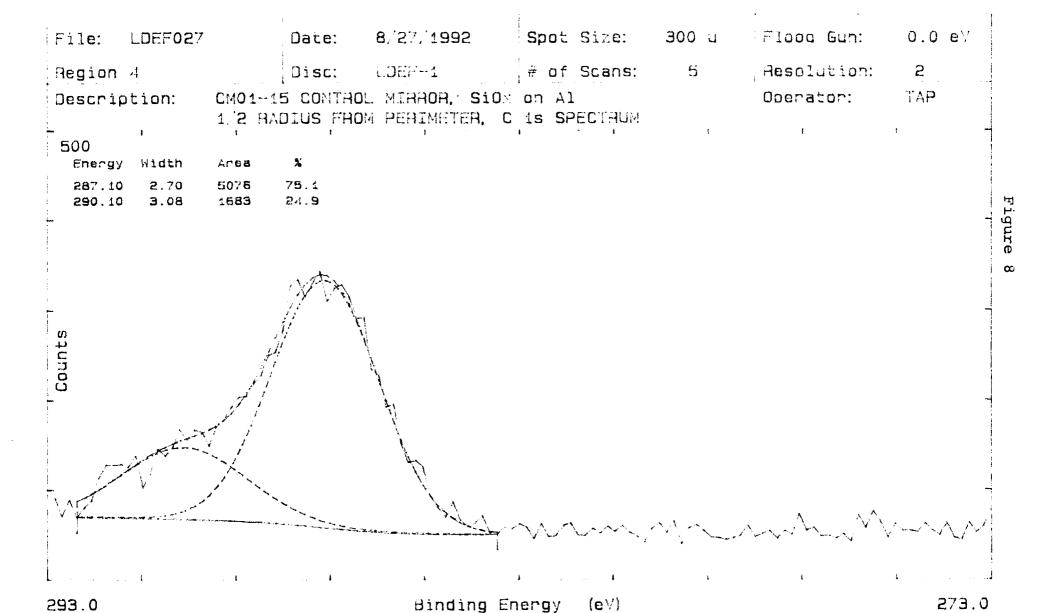


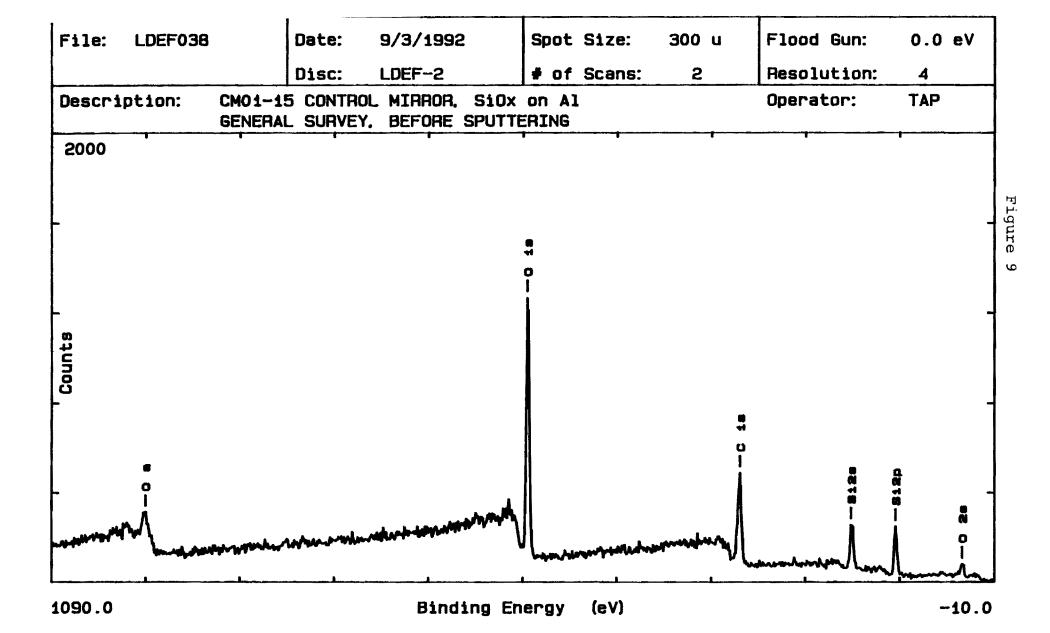
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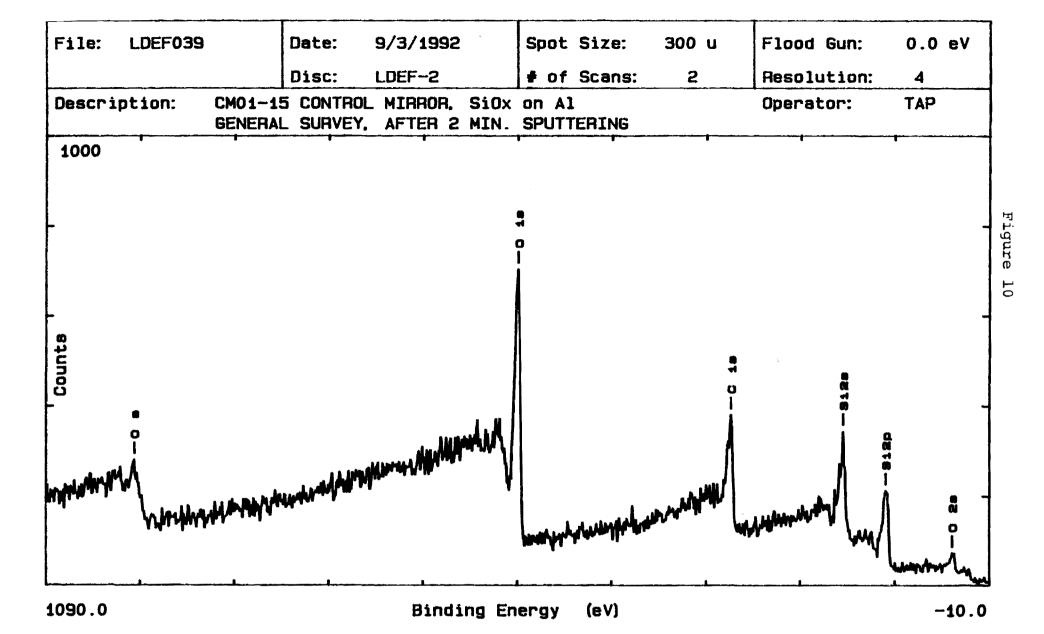
Binding Energy (eV) 522.0



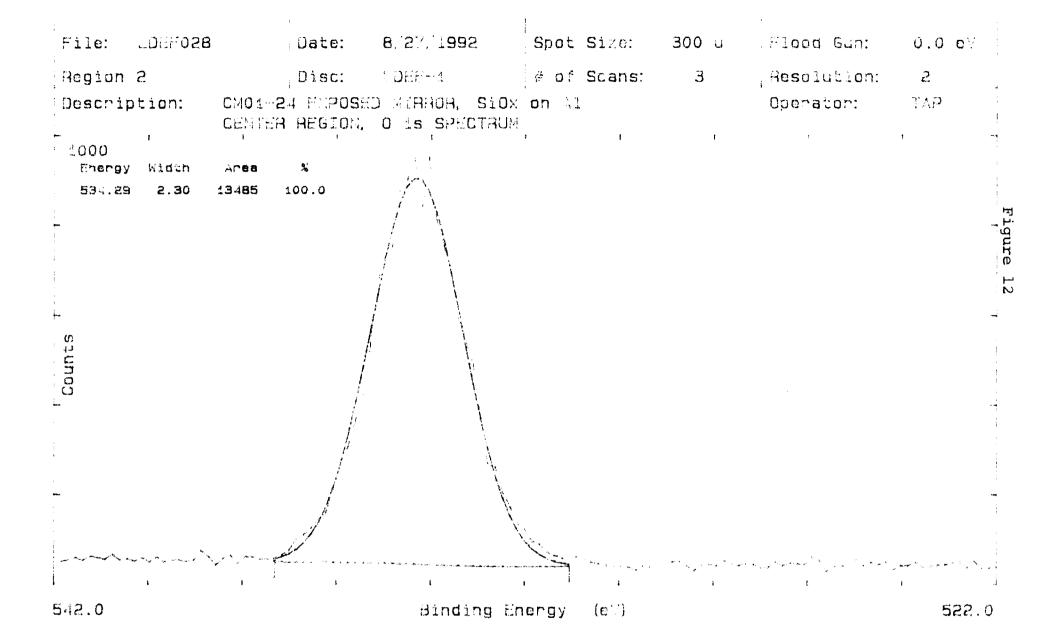
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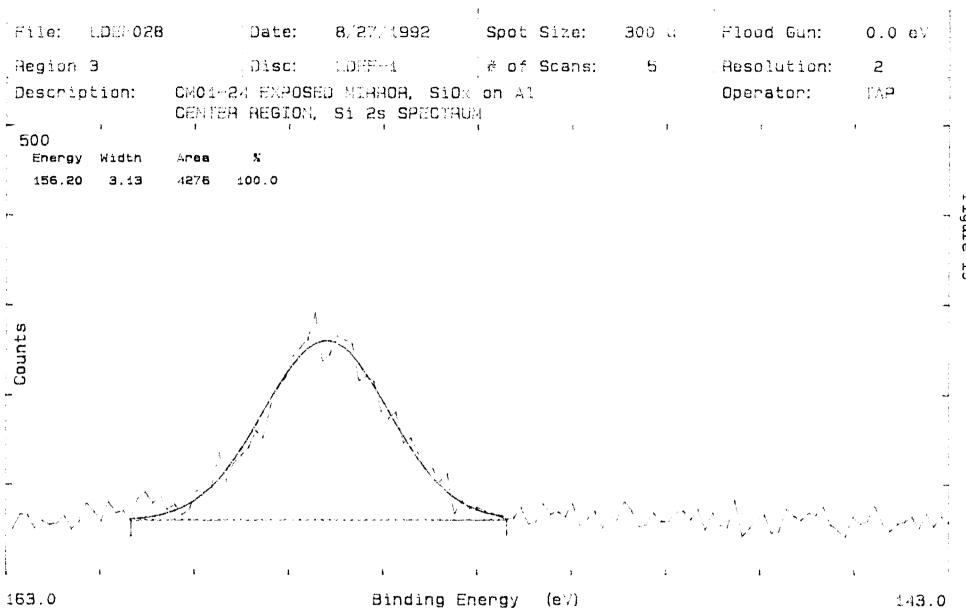




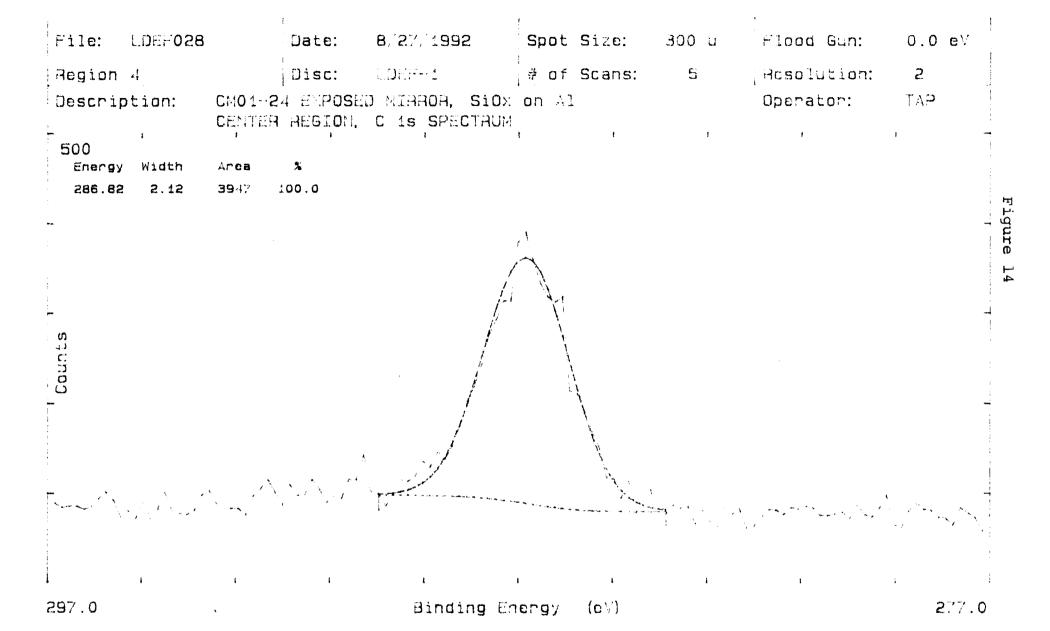


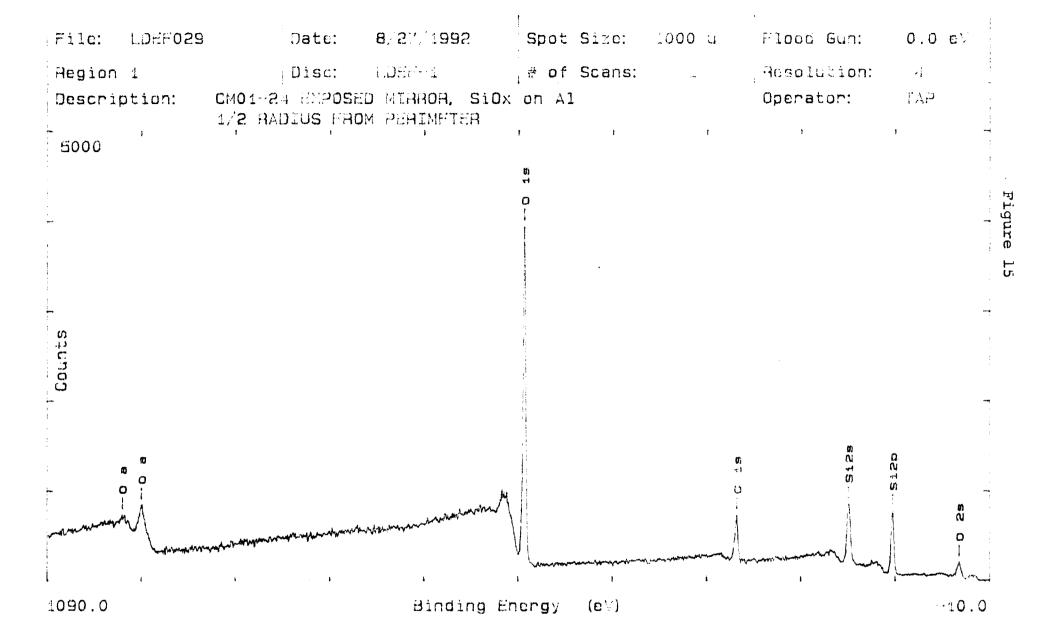
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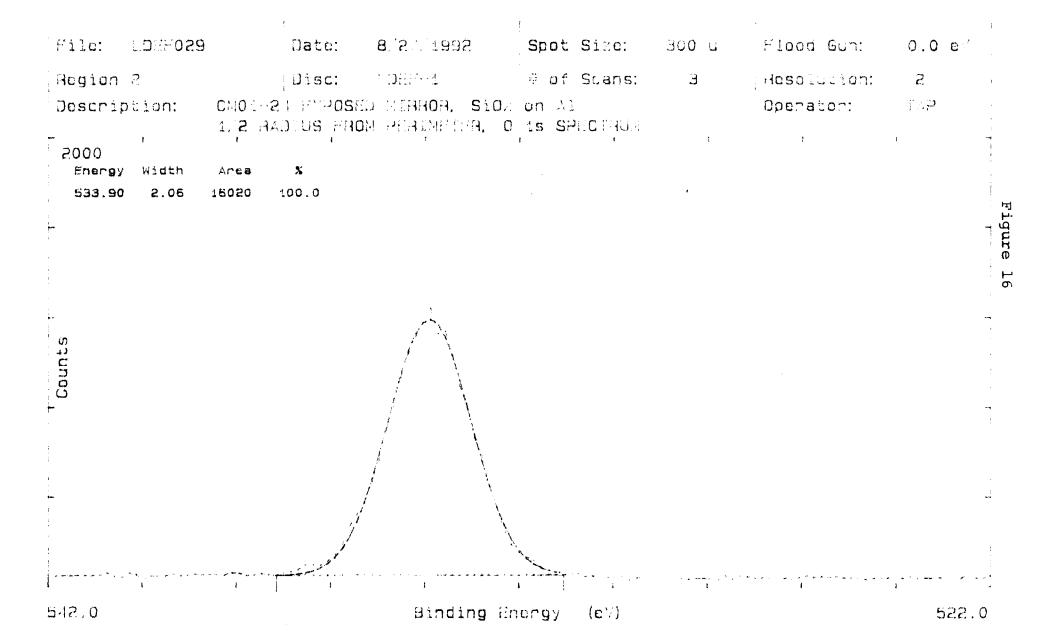




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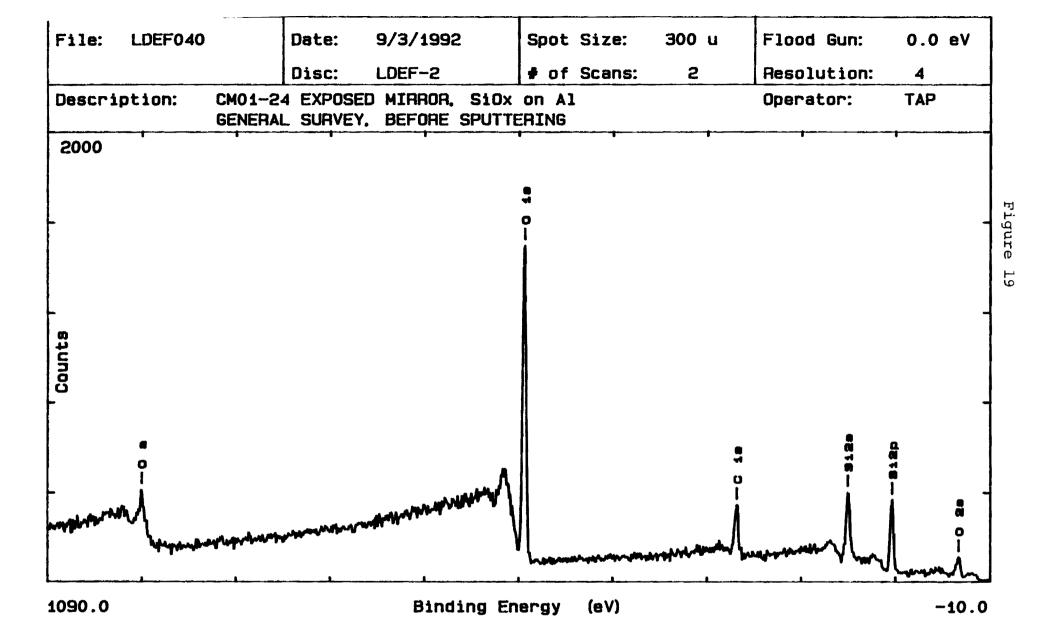


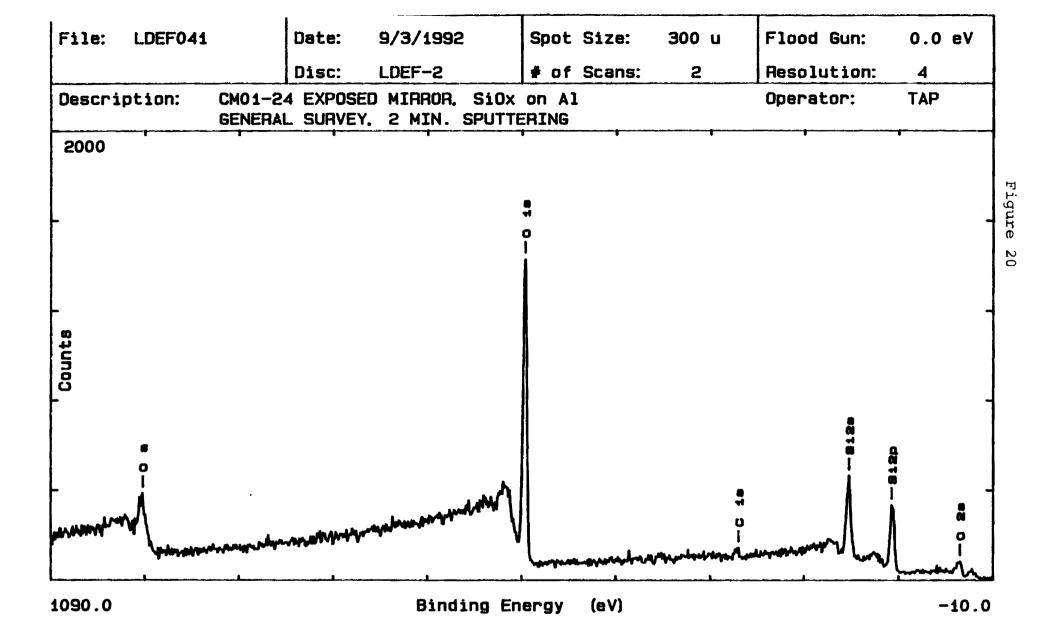
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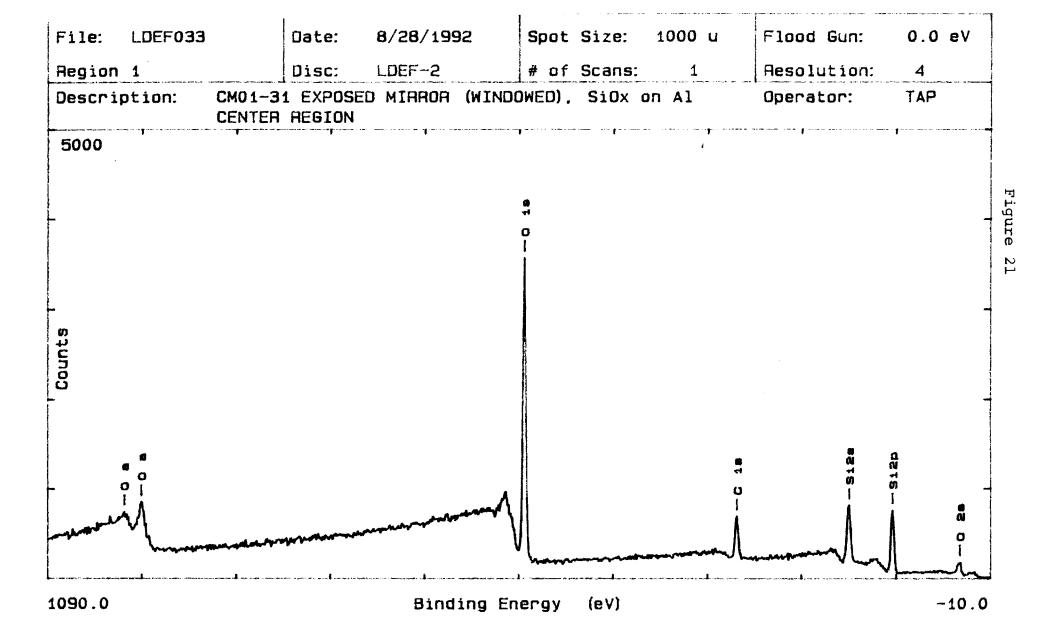
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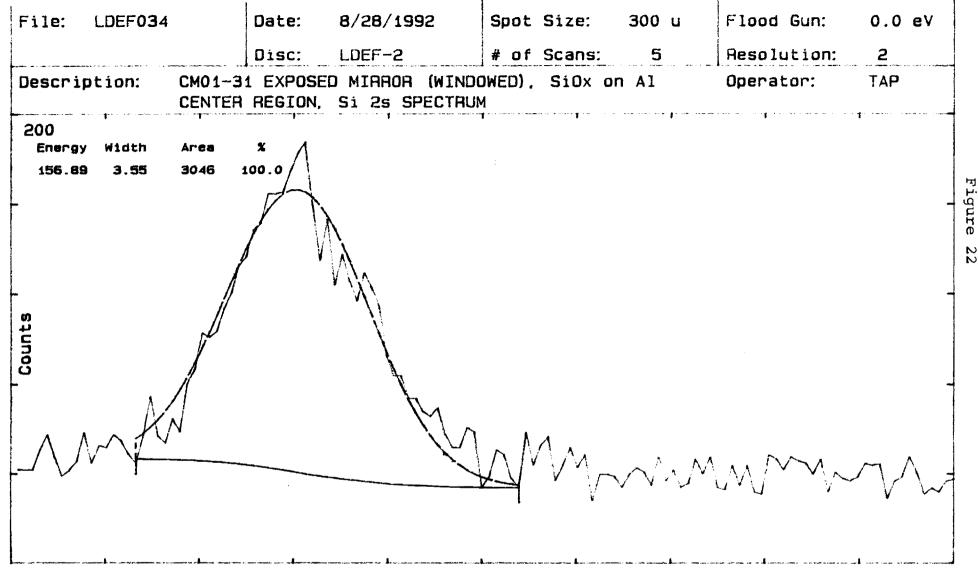
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297.0 Binding Energy (eV) 277.0



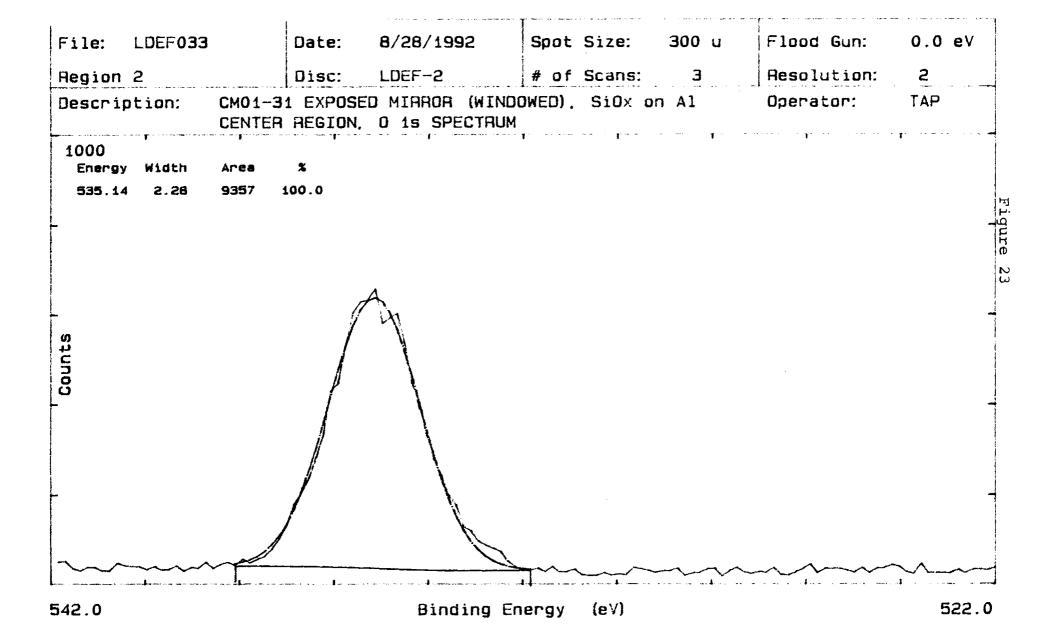


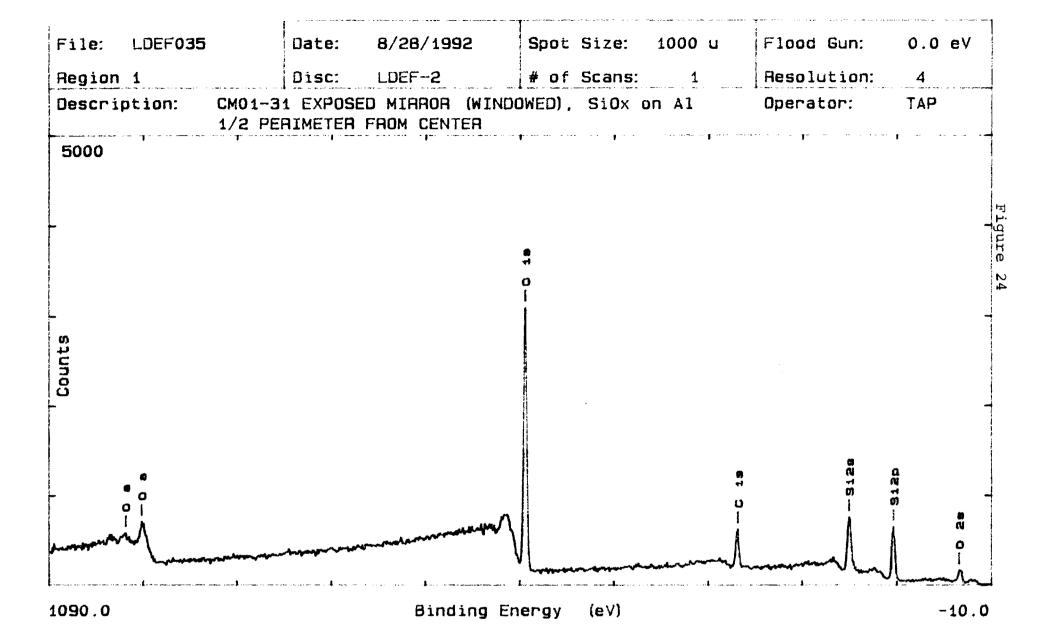


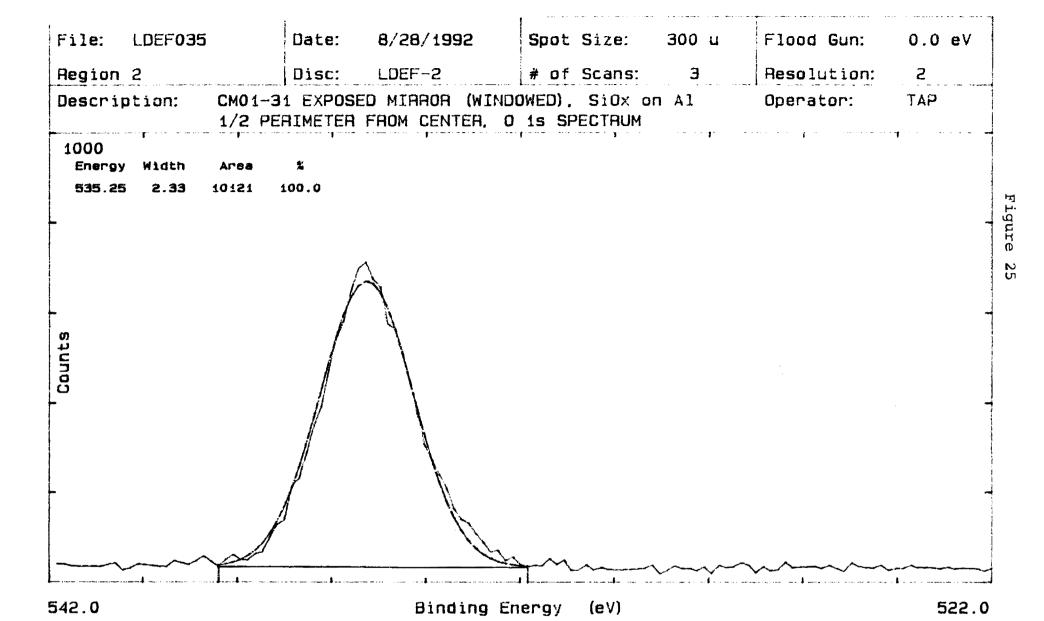


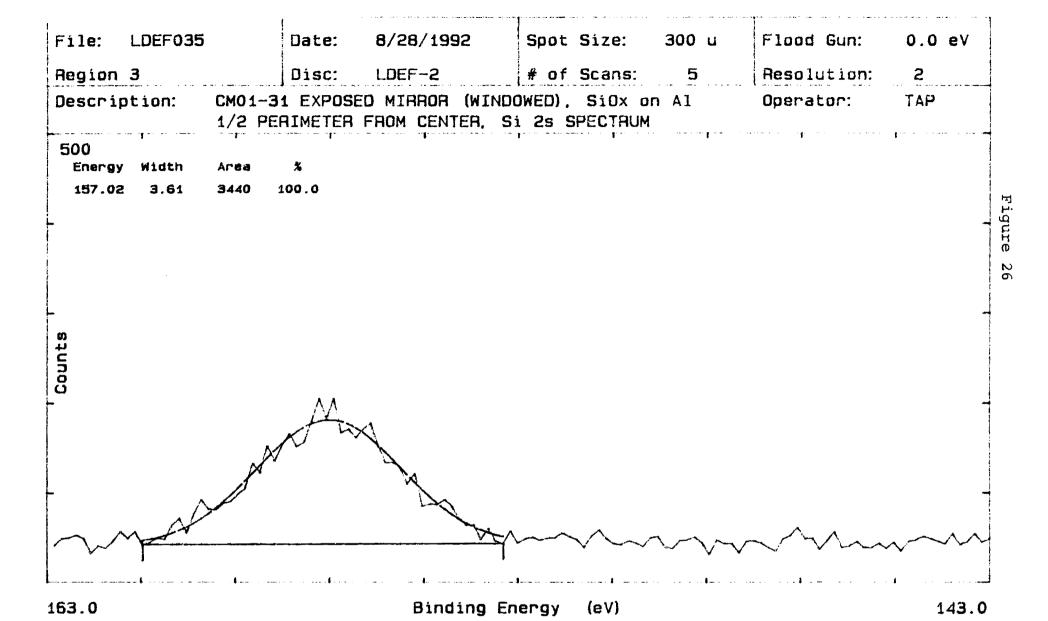
Binding Energy (eV)

143.0

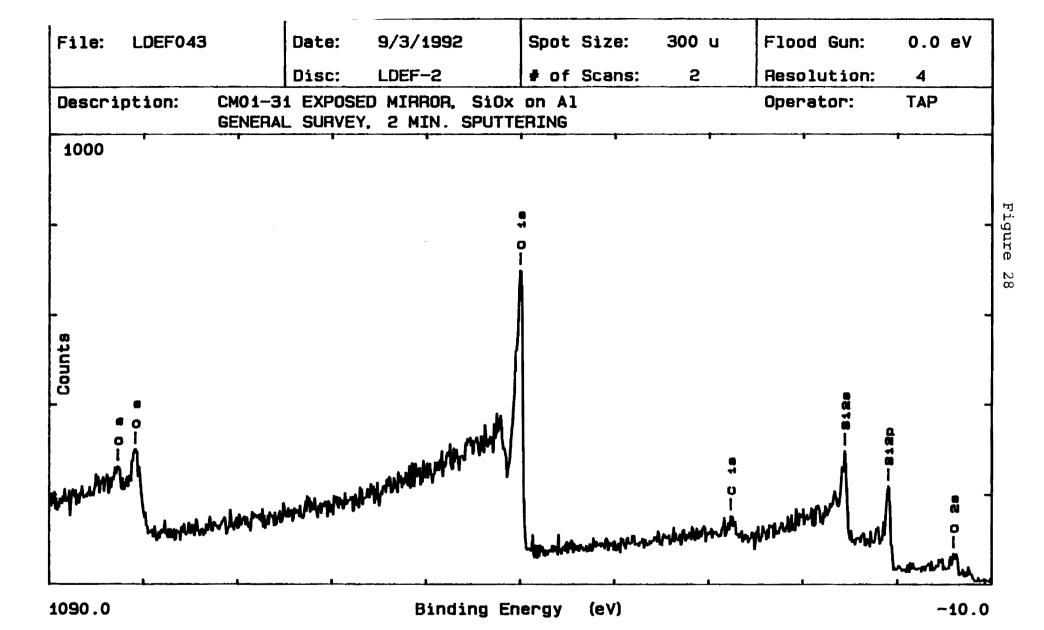


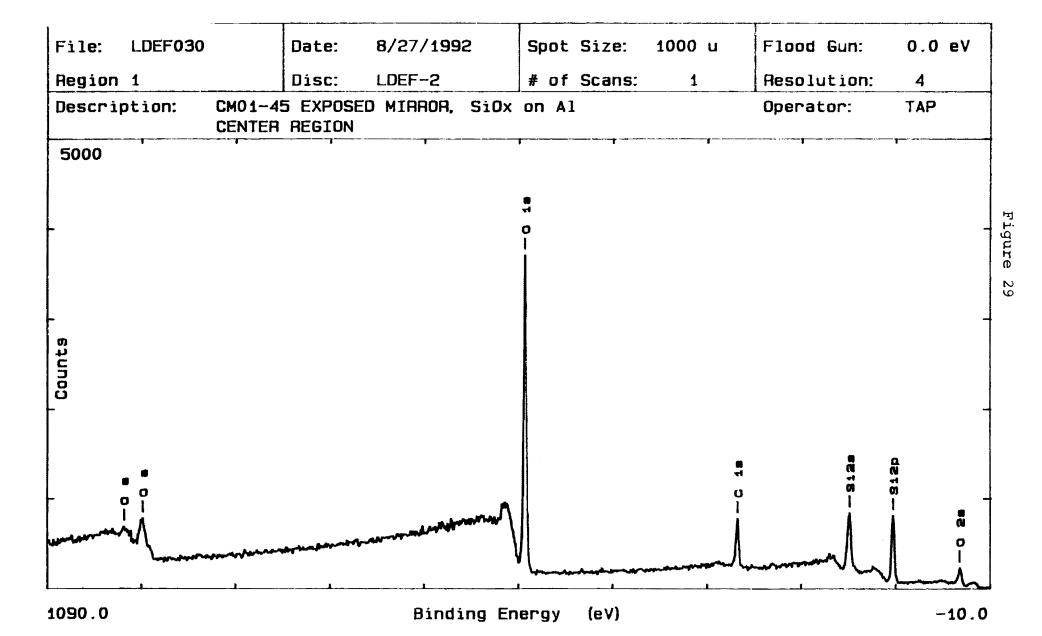


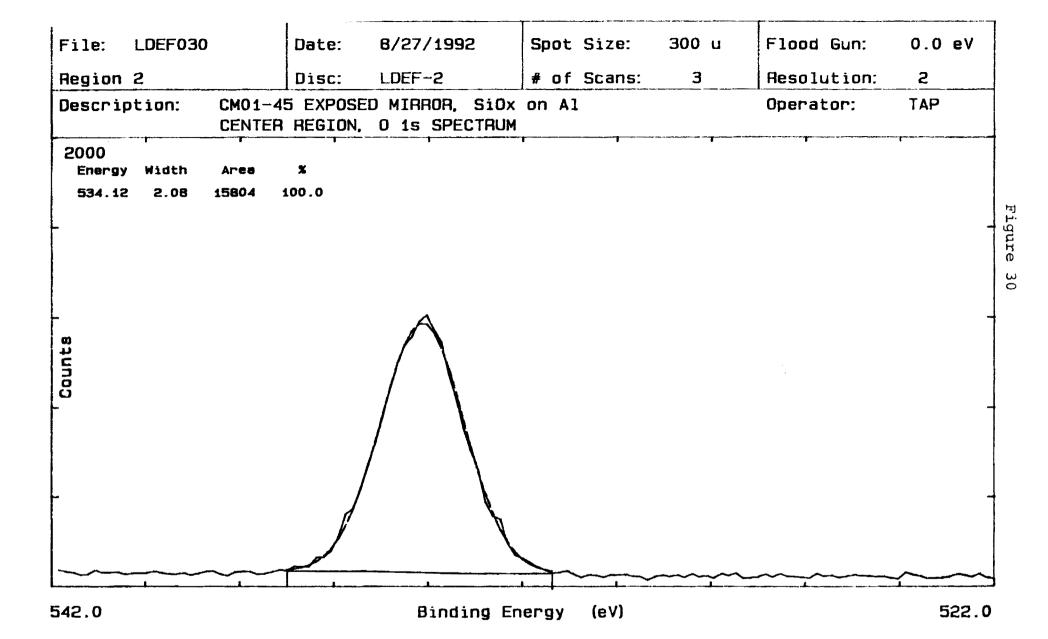


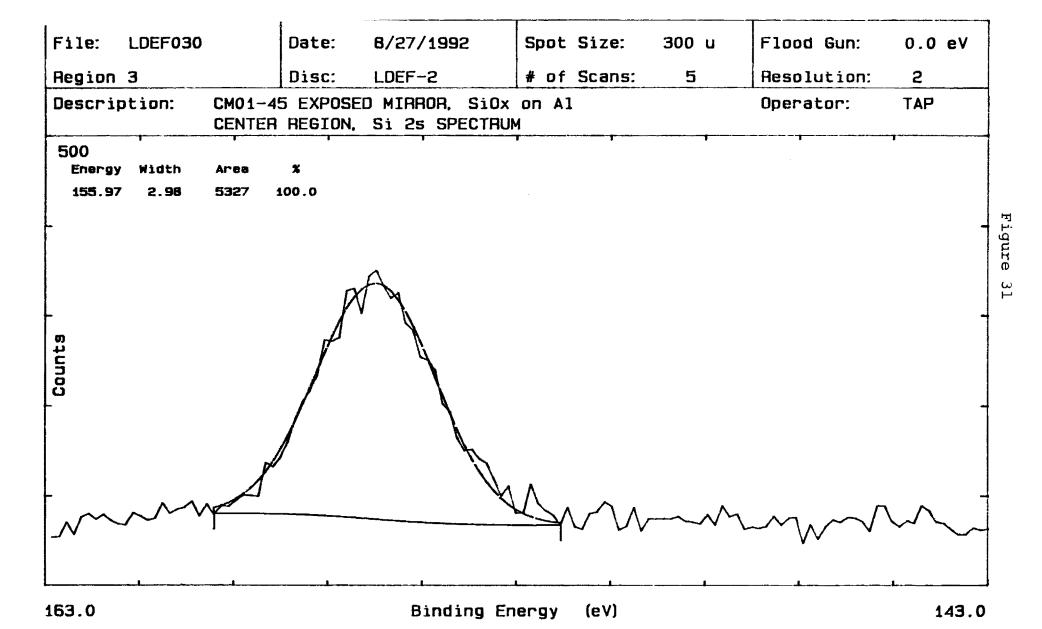


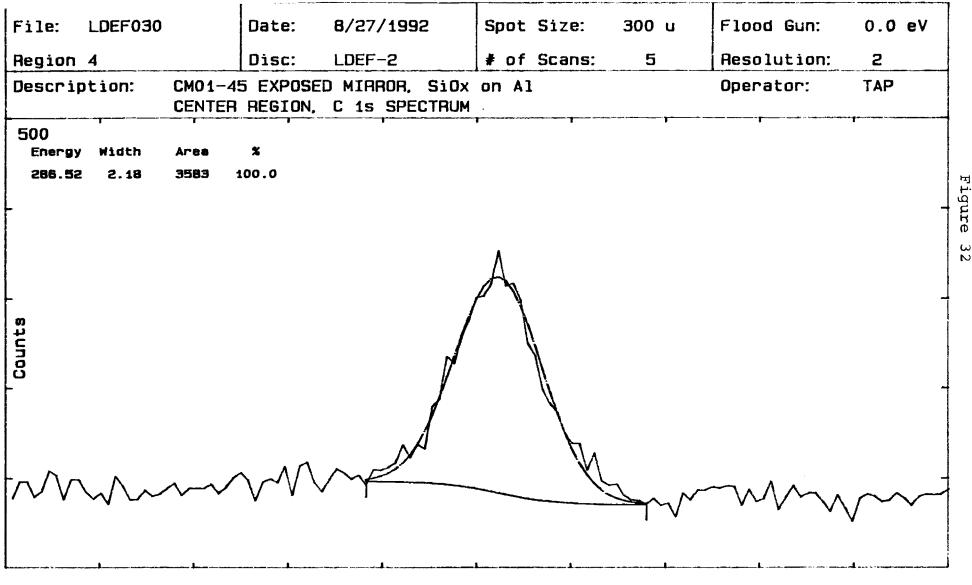
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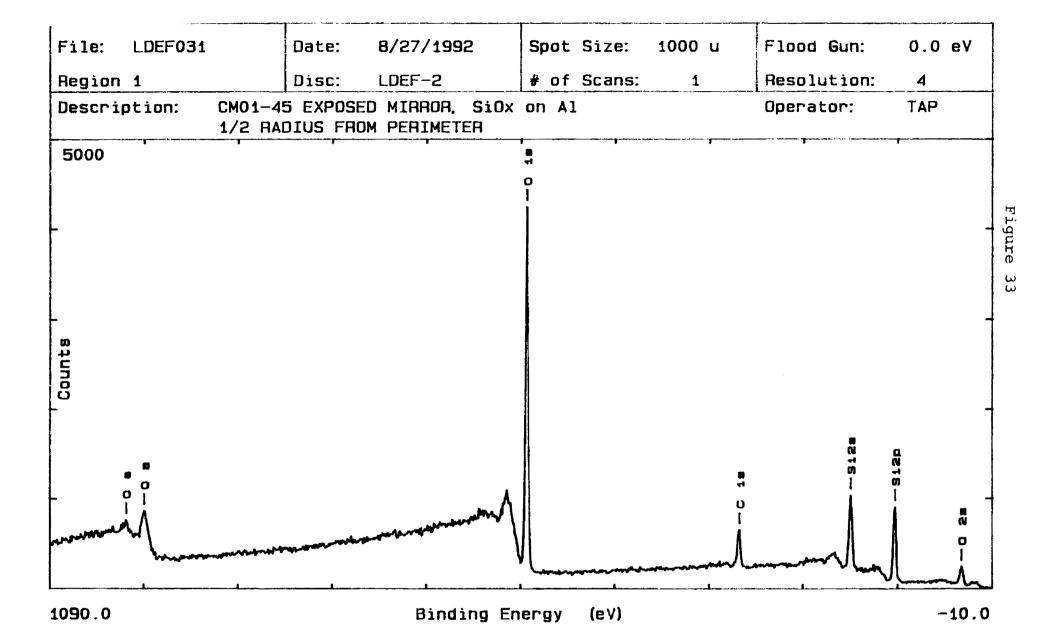


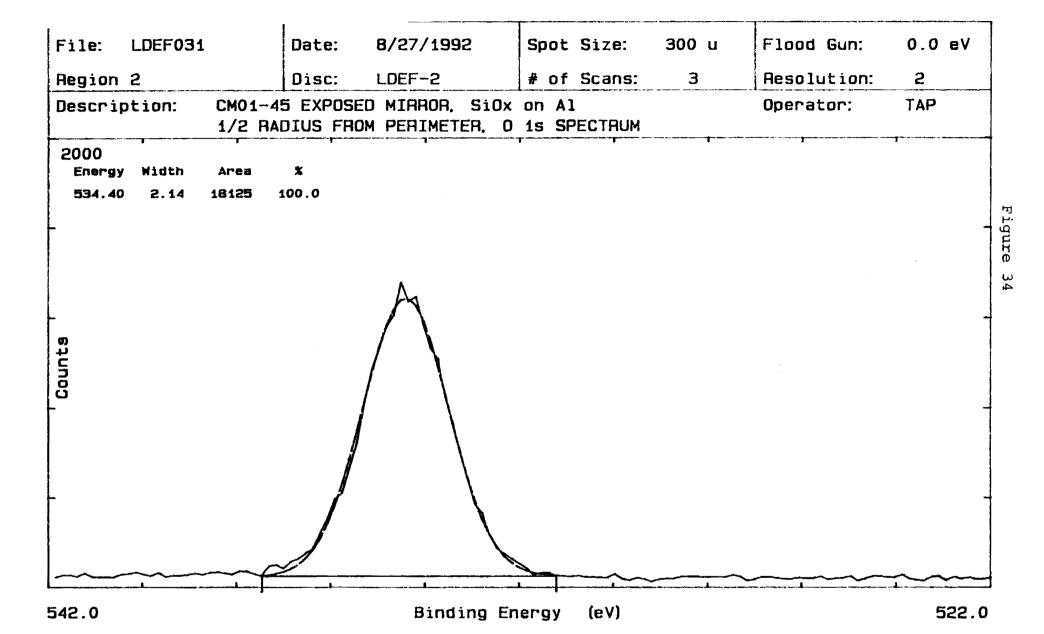


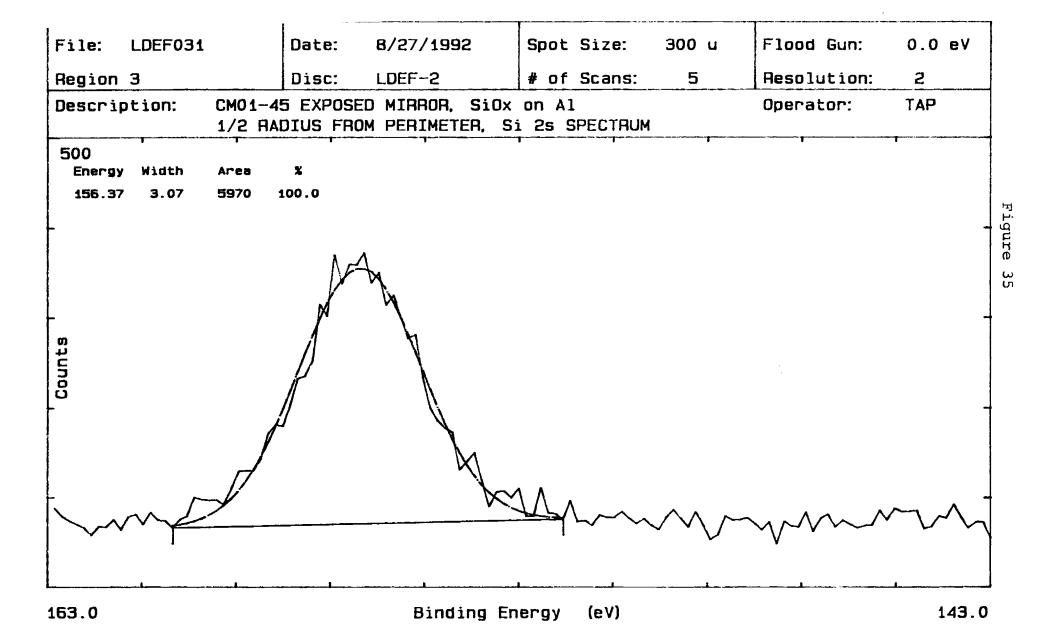
297.0

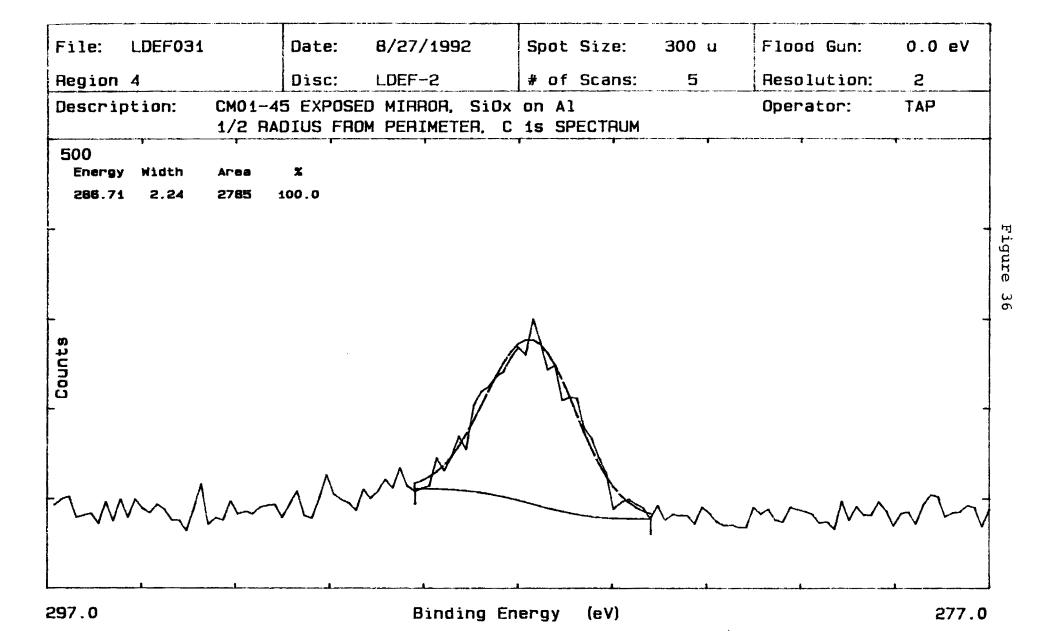
Binding Energy (eV) 277.0

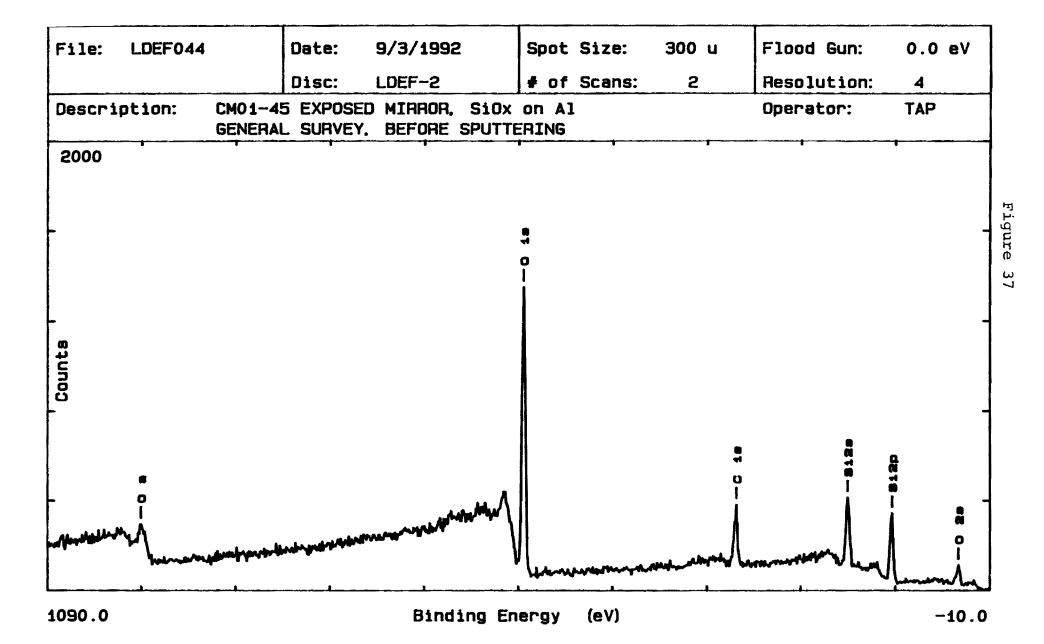
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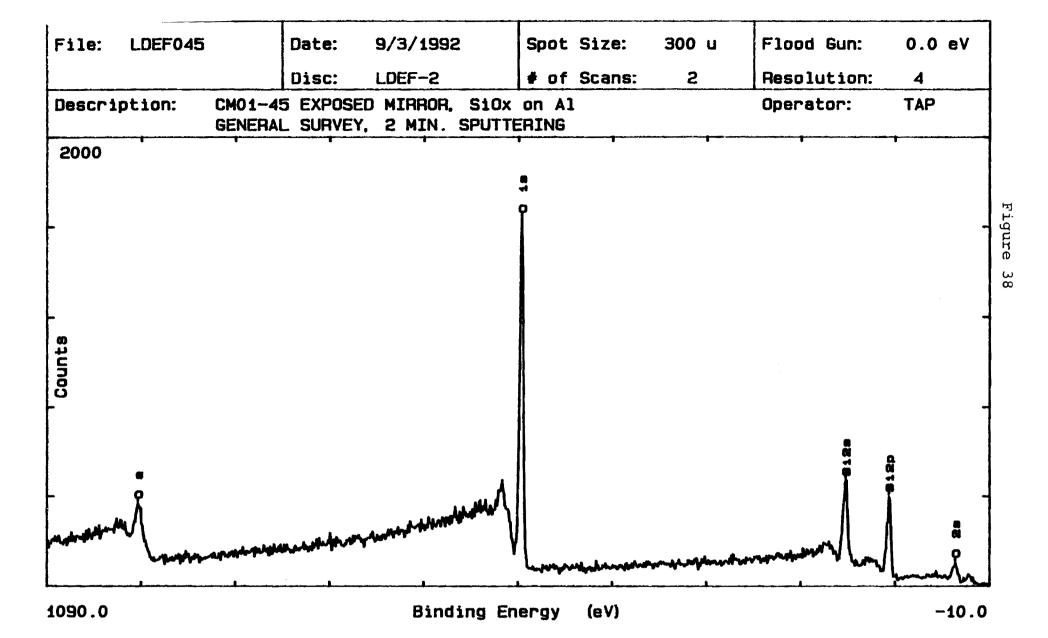


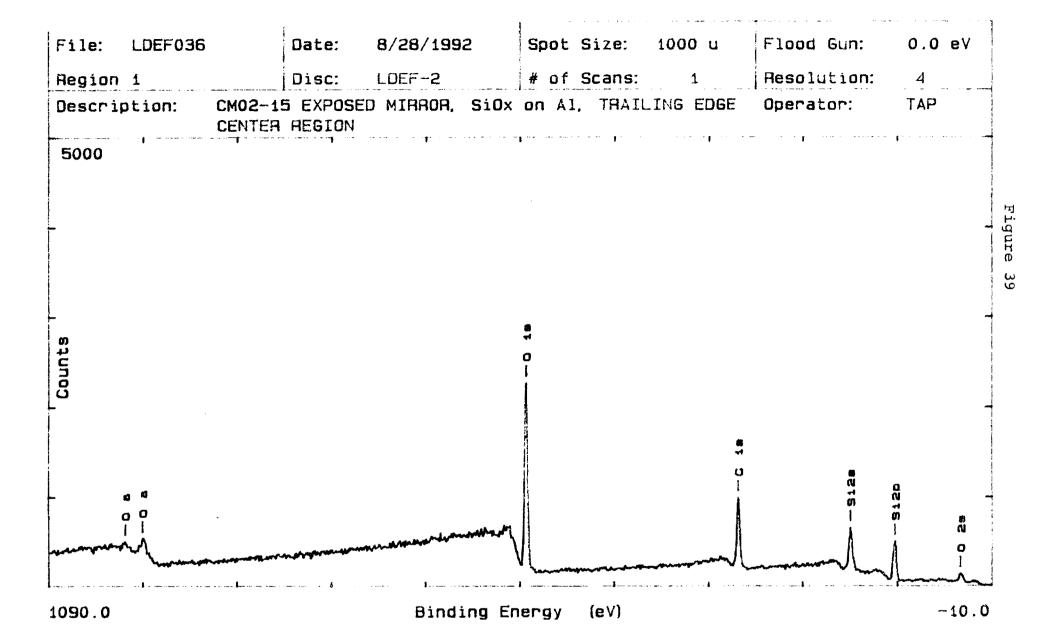


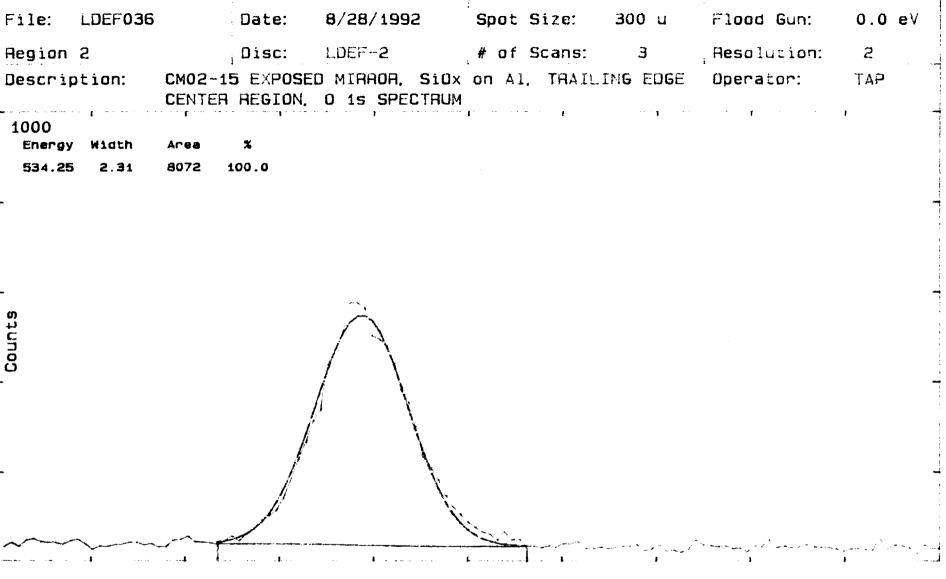




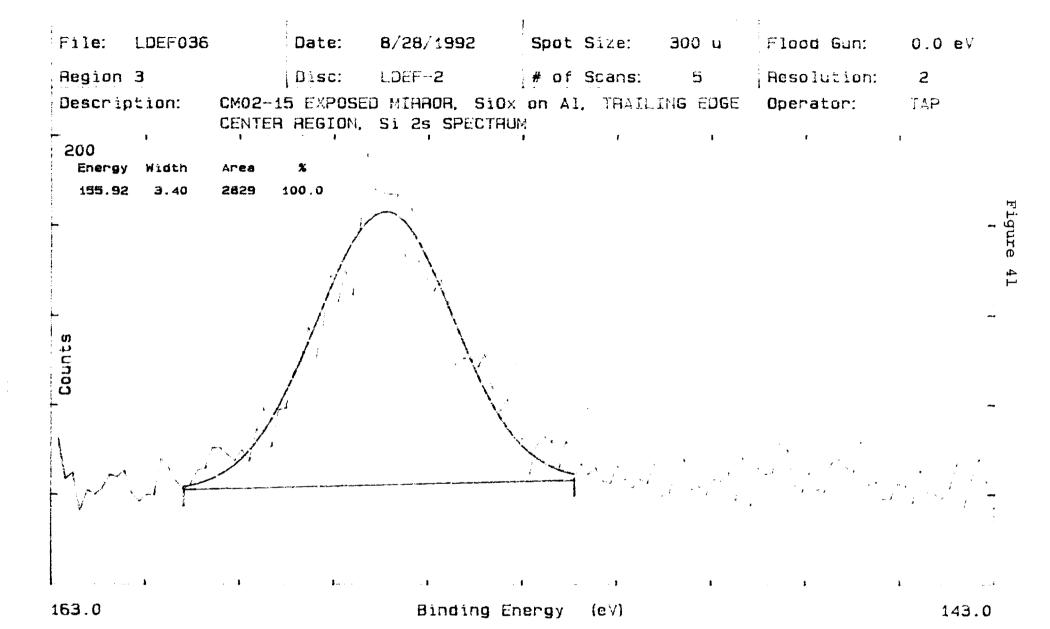


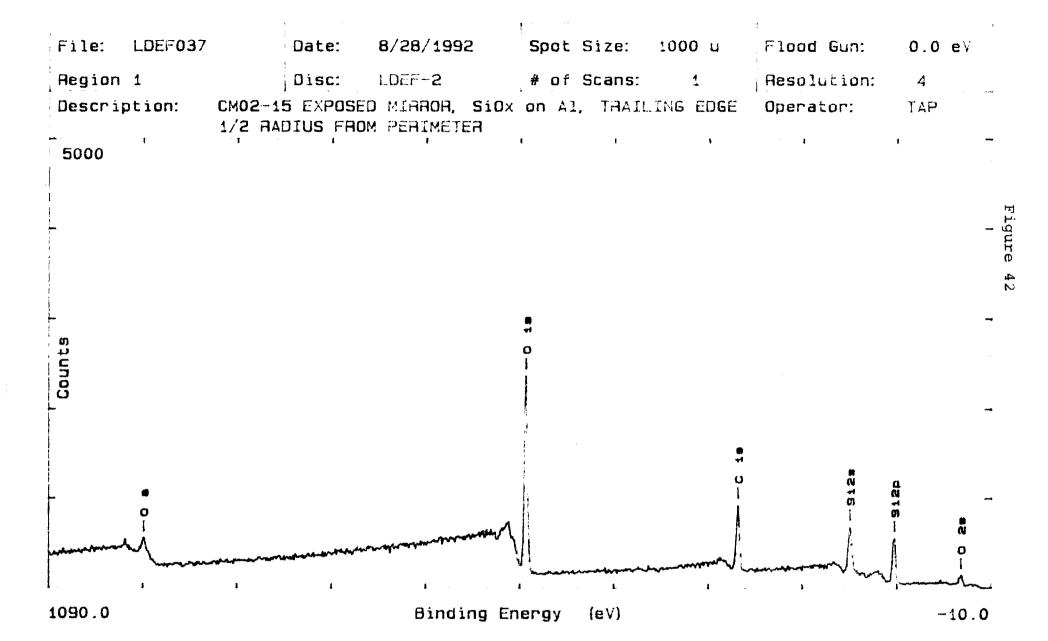


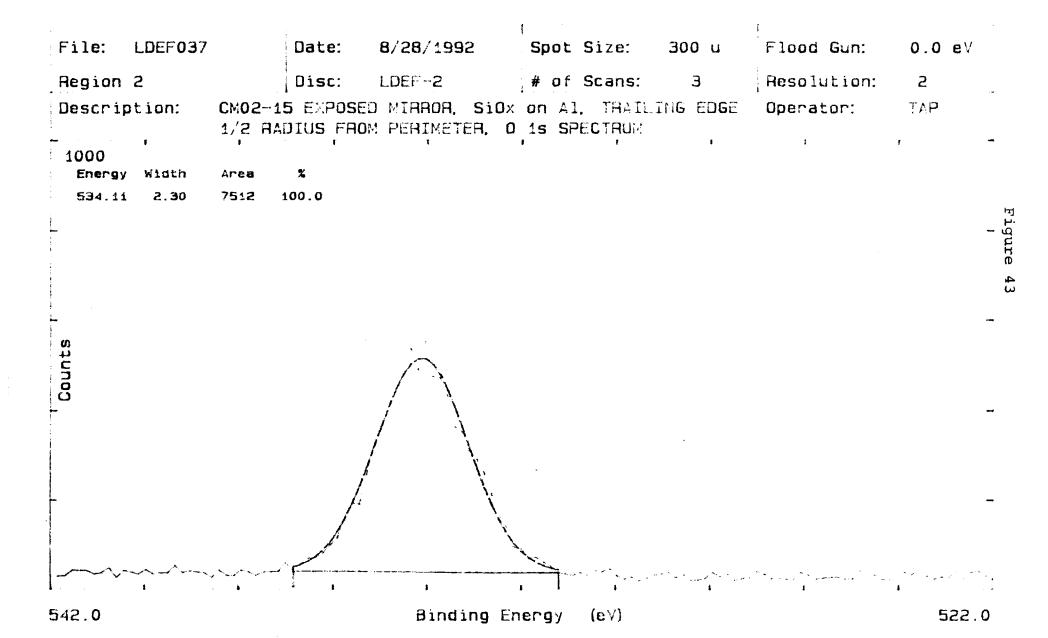


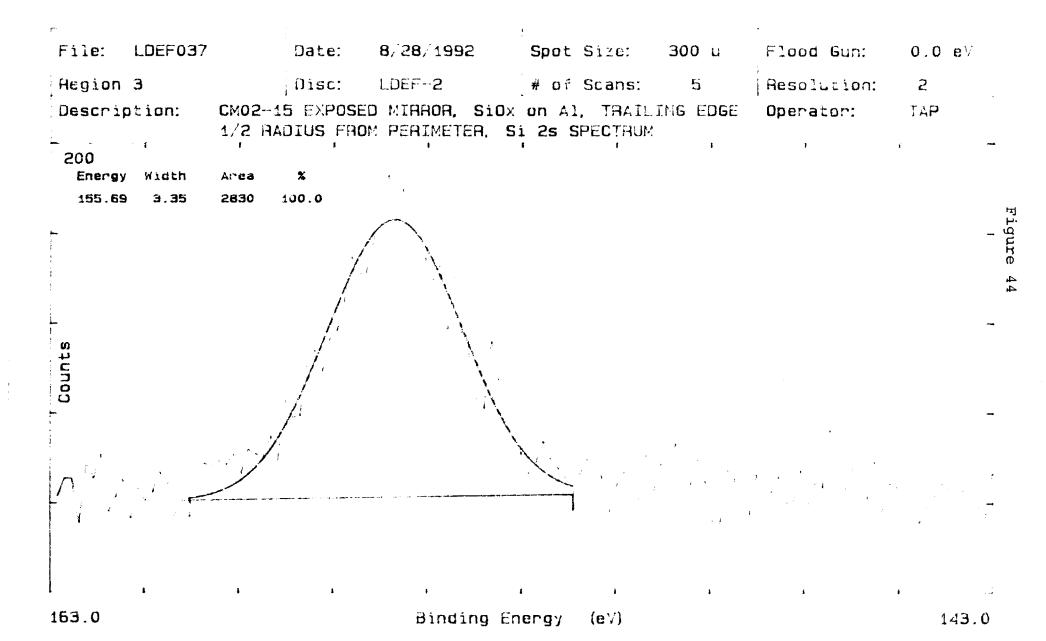


542.0 Binding Energy (eV) 522.0

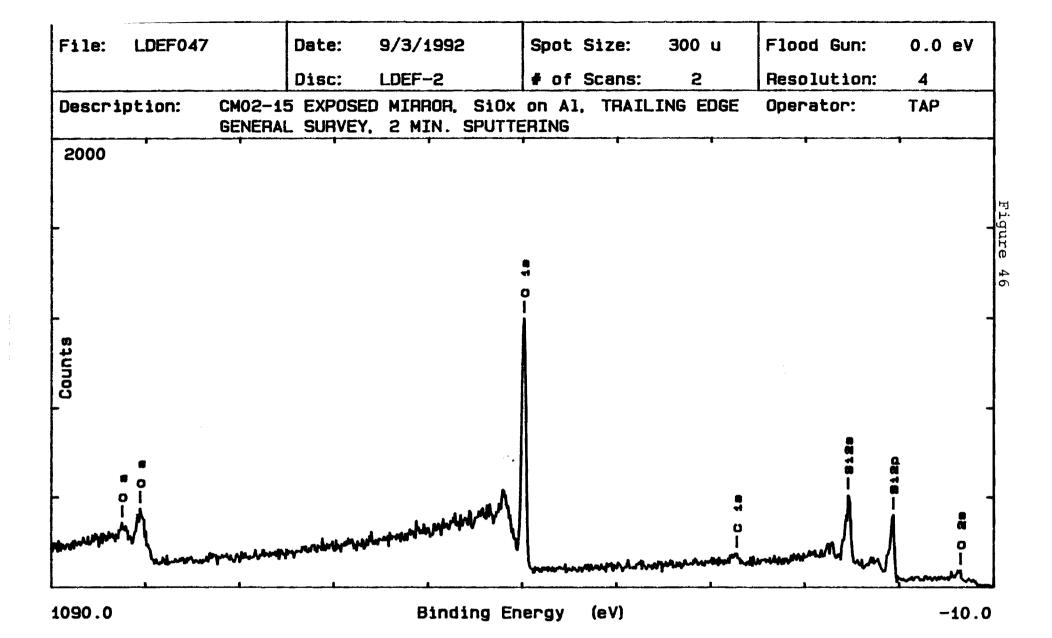


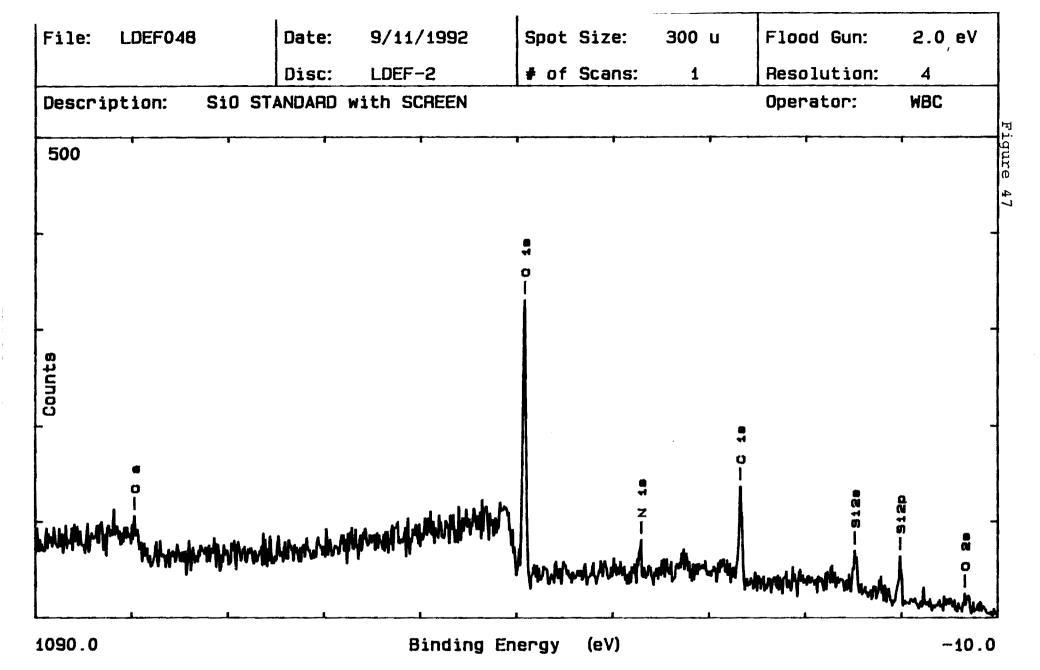




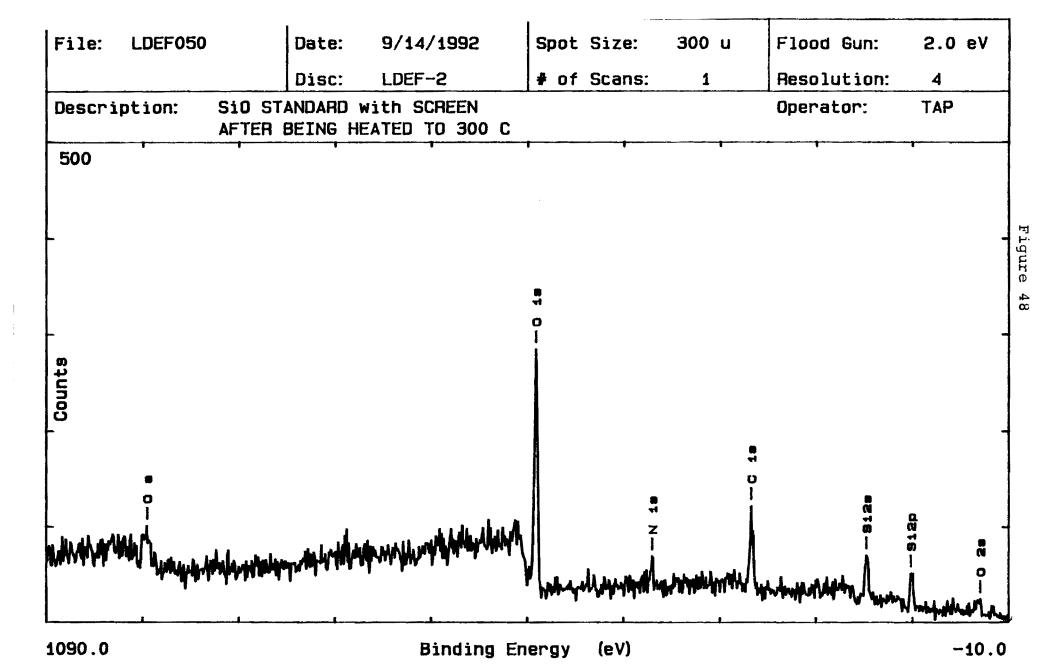


File: LDEF046  Date: 9/3/1992  Disc: LDEF-2  of Scans: 2  Resolution: 4  Description: CM02-15 EXPOSED MIRROR, SiOx on Al, TRAILING EDGE  GENERAL SURVEY, BEFORE SPUTTERING  2000												
Description: CM02-15 EXPOSED MIRROR, SiOx on Al. TRAILING EDGE Operator: TAP GENERAL SURVEY, BEFORE SPUTTERING  2000	File:	LDEF046		Date:	9/3/1992		Spot	Size:	300 u	Flood Gun:	0.0	eV
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GEORGIA TECH MICROELECTRONICS RESEARCH CENTER



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# ELECTRON SPECTROSCOPY FOR CHEMICAL ANALYSIS -- SAMPLE ANALYSIS

116-25

REPORT # LDEF-03

Prepared by:

W.B. Carter School of Materials Science and Engineering Georgia Institute of Technology Atlanta, GA 30332-0245

for:

NASA/MSFC Marshall Space Flight Center, AL 35812

under:

PO # H-13010D

October 12, 1992

This document reports the results of ESCA analyses performed on the following samples:

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CM01-14, CM01-25, CM02-25, CM02-34, IV-10, IV-16, IV-22, IV-28, IV-38, IV-47, IV-80, EOIM-3 04-34
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A general description of the ESCA data is contained in previous reports. Sputter depth profiles were performed on the CM0x-xx samples in at least two spots. These were carried out under the same conditions used for sputtering previous samples. A sputter rate calibration performed on sample CM01-14 indicated that the sputter rate was about 0.1 Å/s. No sputtering was performed on the IV-xx samples or sample # EOIM-3 04-34. Details of the sample analyses follow.

## Reports on ESCA Analyses

#### CMOx-xx Samples:

Two spots were examined on each sample (with the single exception that three were examined on sample # CM01-14). Each spot was examined as follows: first a general survey was taken, second a depth profile was performed and finally another general survey was made (with the exception that a second general survey was not performed on the first spot examined on sample # CM01-14).

# CM01-14: Exposed under window (SiO_x on Al)

Three spots were examined on this specimen. The depth profile on spot 1 was made by alternately collecting data and sputtering every 50 s ( $\Delta t = 50$  s) for a total of 2450 s (after an initial sputter of 100 s). Spot 2 was profiled for a total time of 2400 s using a  $\Delta t = 100$ s. Spot 3 was profiled for a total time of 190 s using a  $\Delta t = 10$ s. Data were collected for the 0 1s and the Si 2s photoemission peaks during profiling.

Figure 1 is the general survey taken of spot 1 prior to depth profiling. The results of a semiquantitative composition analysis taken from this survey are presented in Table 1. The small N 1s photoemission peak evident on figure 1 was not included in the semiquantitative analysis. The depth profile of spot 1 is displayed in figure 2. Note that spot 1 was sputtered for 100 s prior to profiling. A general survey was not performed after this profile.

Figure 3 is the general survey taken of spot 2 prior to depth profiling. The results of a semiquantitative analysis taken from this survey are presented in Table 2. The small N 1s photoemission peak evident on figure 1 was not included in the

semiquantitative analysis. The depth profile of this spot is displayed in figure 4. Figure 5 displays the general survey obtained after profiling spot 2. No N is evident after profiling. The results of a semiquantitative analysis taken from this survey are presented in Table 3.

Figure 6 is the general survey taken of spot 3 prior to depth profiling. The results of a semiquantitative analysis taken from this survey are presented in Table 4. N was not observed on spot 3. The depth profile of this spot is displayed in figure 7. Figure 8 displays the general survey obtained after profiling spot 3. The results of a semiquantitative analysis taken from this survey are presented in Table 5.

Table 1: Semiquantitative Composition of Spot 1 on Sample CM01-14 from Figure 1 Prior to Sputtering

Element	Approximate Atomic Percent
0	30
Si	10
С	60

Table 2: Semiquantitative Composition of Spot 2 on Sample CM01-14 from Figure 3 Prior to Sputtering

Element	Approximate Atomic Percent
0	31
Si	10
С	59

Table 3: Semiquantitative Composition of Spot 2 on Sample CM01-14 from Figure 5 After Profiling

Approximate Atomic Percent
58
40
2

Table 4: Semiquantitative Composition of Spot 3 on Sample CM01-14 from Figure 6 Prior to Sputtering

Approximate Atomic Percent
30
10 60

Table 5: Semiquantitative Composition of Spot 3 on Sample CM01-14 from Figure 8 After Profiling

Element	Approximate Atomic Percent
0	45
Si	37
С	18

These data indicate that there is more 0 on the surface than in the bulk and that the 0 to Si ratio decreases for the first 100 s of sputtering after which it gradually increases. Note that since 100 s of sputtering is about 10 Å in  $\mathrm{SiO}_x$ , the initial decrease in the 0 to Si ratio may be due to 0 containing surface contamination. The long term, gradual increase in the 0 to Si ratio may be an artifact produced by the preferential sputtering of Si during depth profiling. Thus no clear, unambiguous evidence of a change in the 0 content of the  $\mathrm{SiO}_x$  coating with depth was observed. A maximum depth of about 250 Å was reached in profiling this sample.

There is substantial C on the surface of this sample. Its distribution with depth was not followed during depth profiling.

## CM01-25: Exposed, SiO, on Aluminum

Two spots were examined on this specimen. The depth profile on spot 1 was made with  $\Delta t = 10$  s for a total sputtering time of 490 s, or a total depth of about 49 Å. Spot 2 was profiled for a total time of 762 s (total depth ~76 Å) using a  $\Delta t = 20$  s. Data were collected for the 0 ls, Si 2s and the C ls photoemission peaks during profiling.

Figure 9 is the general survey taken of spot 1 prior to depth profiling. The results of a semiquantitative composition analysis taken from this survey are presented in Table 6. The depth profile of spot 1 is displayed in figure 10. Figure 11 is the general survey taken after profiling spot 1. The results of a semiquantitative composition analysis taken from this survey are presented in Table 7.

Figure 12 is the general survey taken of spot 2 prior to depth profiling. The results of a semiquantitative composition analysis taken from this survey are presented in Table 8. The first 320 s of the depth profile of spot 1 is displayed in figure 13. This profile was extended for 442 s without significant change. Figure 14 is the general survey taken after profiling spot 2. The results of a semiquantitative composition analysis taken from this survey are presented in Table 9.

Table 6: Semiquantitative Composition of Spot 1 on Sample CM01-25 from Figure 9 Prior to Sputtering

Element	Approximate Atomic Percent
0	51
Si	33
С	14
Na	2

Table 7: Semiquantitative Composition of Spot 1 on Sample CM01-25 from Figure 11 After Profiling

Element	Approximate Atomic Percent
0	60
Si	40

Table 8: Semiquantitative Composition of Spot 2 on Sample CM01-25 from Figure 12 Prior to Sputtering

Element	Approximate Atomic Percent
o	52
si	36
c	12

Table 9: Semiquantitative Composition of Spot 2 on Sample CM01-25 from Figure 14 After Profiling

Element	Approximate Atomic Percent
o	58
si	42

During profiling, both O and Si appear to increase in concentration initially. This is due to the removal of a C layer that appears to be about a monolayer thick. The initial decrease in the O to Si ratio observed on CM01-14 is not evident on this sample and the O to Si ratio appears constant throughout the depth profiles. No clear, unambiguous evidence of a change in the O content of the  ${\rm SiO}_{\rm x}$  coating with depth was observed. A maximum depth of about 76 Å was reached in profiling this sample.

### CM02-25: Exposed (Trailing Edge), SiO, on Al

Two spots were examined on this specimen. The depth profile on spot 1 was made with  $\Delta t = 20$  s for a total sputtering time of 680 s, or a total depth of about 68 Å. Spot 2 was profiled for a total time of 500 s (total depth ~50 Å) also using  $\Delta t = 20$  s. Data were collected for the 0 1s, Si 2s and the C 1s photoemission peaks during profiling.

Figure 15 is the general survey taken of spot 1 prior to depth profiling. The results of a semiquantitative composition analysis taken from this survey are presented in Table 10. The first 380 s of the depth profile of spot 1 is displayed in figure 16. This profile was extended for 300 s with a slight increase in the 0 to Si ratio occurring with depth (the C 1s peak was not scanned during the profile extension). Figure 17 is the general survey taken after profiling spot 1. The results of a semiquantitative composition analysis taken from this survey are presented in Table 11.

Figure 18 is the general survey taken of spot 2 prior to depth profiling. The results of a semiquantitative composition analysis taken from this survey are presented in Table 12. The first 400 s of the depth profile of spot 2 is displayed in figure 19. This profile was extended for 100 s without significant change (the C 1s peak was not scanned during the profile extension). Figure 20 is the general survey taken after profiling spot 2. The results of a semiquantitative composition analysis taken from this survey are presented in Table 13.

Table 10: Semiquantitative Composition of Spot 1 on Sample CM02-25 from Figure 15 Prior to Sputtering

Element	Approximate Atomic Percent
0	35
Si	19
С	46

Table 11: Semiquantitative Composition of Spot 1 on Sample CM02-25 from Figure 17 After Profiling

Element	Approximate Atomic Percent
O	50
Si	43
C	7

Table 12: Semiquantitative Composition of Spot 2 on Sample CM02-25 from Figure 18 Prior to Sputtering

Element	Approximate Atomic Percent
O	35
Si	19
C	46

Table 13: Semiquantitative Composition of Spot 2 on Sample CM02-25 from Figure 20 After Profiling

Element	Approximate Atomic Percent
O	50
Si	44
C	6

There appears to be a substantial C layer on this sample about 30 to 40 Å thick. Similarly to CM01-14, which also is covered with much surface C, the O to Si ratio decreases for the first about 100 s of sputtering after which it slowly increases. The small thickness sputtered through in 100 s (~10 Å), precludes a conclusion of an O enhanced surface layer of  $\mathrm{SiO}_x$ . Thus the depth profiles on this sample do not present clear, unambiguous evidence of a change in the O content of the  $\mathrm{SiO}_x$  coating with depth. A maximum depth of about 68 Å was reached in profiling this sample.

### CM02-34: Exposed (Trailing Edge), SiO, on Al

Two spots were examined on this specimen. The depth profile on spot 1 was made with  $\Delta t = 20$  s for a total sputtering time of 600 s, or a total depth of about 60 Å. Spot 2 was profiled for a total time of 720 s (total depth ~72 Å) also using  $\Delta t = 20$  s. Data were collected for the 0 1s, Si 2s and the C 1s photoemission peaks during profiling.

Figure 21 is the general survey taken of spot 1 prior to depth profiling. The results of a semiquantitative composition analysis taken from this survey are presented in Table 14. The depth profile of spot 1 is displayed in figure 22. Figure 23 is the general survey taken after profiling spot 1. The results of a semiquantitative composition analysis taken from this survey are presented in Table 15.

Figure 24 is the general survey taken of spot 2 prior to depth profiling. The results of a semiquantitative composition analysis taken from this survey are presented in Table 16. The depth profile of spot 2 is displayed in figure 25. Figure 26 is the general survey taken after profiling spot 2. The results of a semiquantitative composition analysis taken from this survey are presented in Table 17.

Table 14: Semiquantitative Composition of Spot 1 on Sample CM02-34 from Figure 21 Prior to Sputtering

Element	Approximate Atomic Percent
0	39
Si	24
С	37

Table 15: Semiquantitative Composition of Spot 1 on Sample CM02-25 from Figure 23 After Profiling

Element	Approximate Atomic Percent
0	36
Si	36
С	28

Table 16: Semiquantitative Composition of Spot 2 on Sample CM02-34 from Figure 24 Prior to Sputtering

Element	Approximate Atomic Percent
0	36
Si	23
С	41

Table 17: Semiquantitative Composition of Spot 2 on Sample CM02-34 from Figure 26 After Profiling

Element	Approximate Atomic Percent
o	47
si	41
c	12

The depth profiles collected on this sample are qualitatively similar to those from CM02-25 with the most evident quantitative difference being that the C layer is substantially thicker on CM02-34. Indeed, the C layer on this sample is thicker than that on any of the other three CM0x-xx samples reported on here. This C layer was not entirely penetrated by 720 s of sputtering. As with both CM01-14 and CM02-25, which are also covered with much surface C, the O to Si ratio decreases for the first about 100 s of sputtering after which it slowly increases. The small thickness sputtered through in 100 s (~10 Å), precludes a conclusion of an O enhanced surface layer of SiO_x. Thus the depth profiles on this sample do not present clear, unambiguous evidence of a change in the O content of the SiO_x coating with depth. A maximum depth of about 72 Å was reached in profiling this sample.

### IV-xx Samples:

Samples IV-10, IV-16, IV-22, IV-28 and IV-28 were fully exposed during the LDEF mission. General surveys were taken of two spots on each of these samples. High resolution spectra were taken of the major photoemission peaks displayed on the general surveys and fit with gaussians.

Samples IV-47 and IV-80 were half exposed during flight. General surveys were taken of four spots on each of these: two spots in the unexposed region and two in the exposed region.

#### IV-10: Al + overcoat

Figure 27 is the general survey taken from spot 1. Table 18 displays the semiquantitative composition determined from this spectrum. Figures 28 - 30 are high resolution spectra of the 0 1s, Si 2s and C 1s photoemission peaks, respectively.

Figure 31 is the general survey taken from spot 2. Table 19 displays the semiquantitative composition determined from this spectrum. Figures 32 - 34 are high resolution spectra of the 0 1s, Si 2s and C 1s photoemission peaks, respectively.

Table 18: Semiquantitative Composition of Spot 1 on Sample IV-10 from Figure 27

Element	Approximate Atomic Percent
0	43
Si	32
С	25

Table 19: Semiquantitative Composition of Spot 2 on Sample IV-10 from Figure 31

Element	Approximate Atomic Percent
0	47
Si	31
С	22

### IV-16: Aq + overcoat

Figure 35 is the general survey taken from spot 1. Table 20 displays the semiquantitative composition determined from this spectrum. Figures 36 - 40 are high resolution spectra of the 0 1s, Si 2s, C 1s, Al 2p and Ag 3d photoemission peaks, respectively.

Figure 41 is the general survey taken from spot 2. Table 21 displays the semiquantitative composition determined from this spectrum. Figures 42 - 46 are high resolution spectra of the 0

1s, Si 2s, C 1s, Al 2p and Ag 3d photoemission peaks, respectively.

Table 20: Semiquantitative Composition of Spot 1 on Sample IV-16 from Figure 35

50 15 20 12

Table 21: Semiquantitative Composition of Spot 2 on Sample IV-16 from Figure 41

Element	Approximate Atomic Percent
0	41
Si	14
С	22
Al	16
Ag	7

# IV-22: Enhanced Al + overcoat

Figure 47 is the general survey taken from spot 1. Table 22 displays the semiquantitative composition determined from this spectrum. Figures 48 - 50 are high resolution spectra of the 0 1s, Si 2s and C 1s photoemission peaks, respectively.

Figure 51 is the general survey taken from spot 2. Table 23 displays the semiquantitative composition determined from this spectrum. Figures 52 - 54 are high resolution spectra of the 0 1s, Si 2s and C 1s photoemission peaks, respectively.

Table 22: Semiquantitative Composition of Spot 1 on Sample IV-22 from Figure 47

Element	Approximate Atomic Percent
0	53
Si	33
C	14

Table 23: Semiquantitative Composition of Spot 2 on Sample IV-22 from Figure 51

Element	Approximate Atomic Percent
0	57
Si	34
С	9

## IV-28: A1 + ??

Figure 55 is the general survey taken from spot 1. Table 24 displays the semiquantitative composition determined from this spectrum. Figures 56 - 58 are high resolution spectra of the 0 1s, Si 2s and C 1s photoemission peaks, respectively.

Figure 59 is the general survey taken from spot 2. Table 25 displays the semiquantitative composition determined from this spectrum. Figures 60 - 62 are high resolution spectra of the 0 1s, Si 2s and C 1s photoemission peaks, respectively.

Table 24: Semiquantitative Composition of Spot 1 on Sample IV-28 from Figure 55

Element	Approximate Atomic Percent
0	54
Si	29
С	17

Table 25: Semiquantitative Composition of Spot 2 on Sample IV-28 from Figure 59

Element	Approximate Atomic Percent
0	51
Si	36
С	13

#### IV-38: Enhanced Al + overcoat

Figure 63 is the general survey taken from spot 1. Table 26 displays the semiquantitative composition determined from this spectrum. Figures 64 - 66 are high resolution spectra of the 0 1s, Si 2s and C 1s photoemission peaks, respectively.

Figure 67 is the general survey taken from spot 2. Table 27 displays the semiquantitative composition determined from this spectrum. Figures 68 - 70 are high resolution spectra of the 0 1s, Si 2s and C 1s photoemission peaks, respectively.

Table 26: Semiquantitative Composition of Spot 1 on Sample IV-38 from Figure 63

Element	Approximate Atomic Percent
0	53
Si	33
С	14

Table 27: Semiquantitative Composition of Spot 2 on Sample IV-38 from Figure 67

Element	Approximate Atomic Percent
0	53
Si	35
С	12

#### IV-47: Niobium

Figure 71 is the general survey taken from spot 1 in the unexposed region of the sample. Table 28 presents the results of the semiquantitative analysis made from this spectrum. Figures 72 - 75 are high resolution spectra of the O 1s, Si 2s, C 1s and Nb 3d photoemission peaks, respectively.

Figure 76 is the general survey taken from spot 2 in the unexposed region of the sample. Table 29 presents the results of the semiquantitative analysis made from this spectrum. Figures 77 - 80 are high resolution spectra of the 0 1s, Si 2s, C 1s and Nb 3d photoemission peaks, respectively.

Figure 81 is the general survey taken from spot 3 in the exposed region of the sample. Table 30 presents the results of the semiquantitative analysis made from this spectrum. Figures 82 - 85 are high resolution spectra of the 0 1s, C 1s, Nb 3d and Pb 4f photoemission peaks, respectively.

Figure 86 is the general survey taken from spot 4 in the exposed region of the sample. Table 31 presents the results of the semiquantitative analysis made from this spectrum. Figures 87 - 90 are high resolution spectra of the 0 1s, C 1s, Nb 3d and Pb 4f photoemission peaks, respectively.

Table 28: Semiquantitative Composition of Spot 1 from the Unexposed Region of Sample IV-47 from Figure 71

Element	Approximate Atomic Percent
0	54
Si	16
C	22
Nb	8

Table 29: Semiquantitative Composition of Spot 2 from the Unexposed Region of Sample IV-47 from Figure 76

Element	Approximate Atomic Percent
0	51
Si	16
C	25
Nb	8

Table 30: Semiquantitative Composition of Spot 3 from the Exposed Region of Sample IV-47 from Figure 81

Element	Approximate Atomic Percent
0	39
С	49
Nb	11
Pb	1

Table 31: Semiquantitative Composition of Spot 4 from the Exposed Region of Sample IV-47 from Figure 86

Element	Approximate Atomic Percent
0	43
С	44
Nb	11
Pb	2

Significant differences exist between the unexposed and exposed regions of this sample. Si was only observed on the unexposed region (both spots) while Pb was only observed on the exposed region (both spots). There also is more C on the exposed region than the unexposed region.

The binding energies of the Nb  $3d_{5/2}$  photoemission peaks in the unexposed region (207.3 eV calibrated to C 1s of adventitious C at 284.8 eV) and the exposed region (207.1 eV) are similar to

those of  $Nb_2O_5$  and  $NbO_2.^{1,2}$  Pure Nb has a  $3d_{5/2}$  B.E. of 201.5 eV.^{1,3}

Likewise, the Pb on the exposed region of the sample has a  $4f_{7/2}$  B.E. (138.8 eV calibrated to C 1s of adventitious C at 284.8 eV) similar to that of oxidized forms of Pb (Pb₃O₄ at 137.4 eV⁴, PbO at 137.7 eV⁵, and PbO₂ at 136.8 eV⁴.) Pure Pb has a  $4f_{7/2}$  B.E. of 136.6 eV.⁶

### IV-80: Ag Alloy + overcoat

Figure 91 is the general survey taken from spot 1 in the unexposed region of the sample. Table 32 presents the results of the semiquantitative analysis made from this spectrum. Figures 92 and 93 are high resolution spectra of the 0 1s and C 1s photoemission peaks, respectively.

Figure 94 is the general survey taken from spot 2 in the unexposed region of the sample. Table 33 presents the results of the semiquantitative analysis made from this spectrum. Figures 95 - 98 are high resolution spectra of the 0 1s, Si 2s, C 1s and Ag 3d photoemission peaks, respectively.

Figure 99 is the general survey taken from spot 3 in the exposed region of the sample. Table 34 presents the results of the semiquantitative analysis made from this spectrum. Figures 100 - 103 are high resolution spectra of the 0 ls, Si 2s, C ls and Ag 3d photoemission peaks, respectively.

Figure 104 is the general survey taken from spot 4 in the exposed region of the sample. Table 35 presents the results of the semiquantitative analysis made from this spectrum. Figures 105 - 108 are high resolution spectra of the 0 1s, Si 2s, C 1s and Ag 3d photoemission peaks, respectively.

Table 32: Semiquantitative Composition of Spot 1 from the Unexposed Region of Sample IV-80 from Figure 91

Element	Approximate Atomic Percent
0	29
С	52
Al	19

Table 33: Semiquantitative Composition of Spot 2 from the Unexposed Region of Sample IV-80 from Figure 94

Element	Approximate Atomic Percent
0	48
Si	23
С	23
Ag	6

Table 34: Semiquantitative Composition of Spot 3 from the Exposed Region of Sample IV-80 from Figure 99

Element	Approximate Atomic Percent
0	48
Si	29
С	19
Aq	4

Table 35: Semiquantitative Composition of Spot 4 from the Exposed Region of Sample IV-80 from Figure 104

Element	Approximate Atomic Percer
0	55
Si	30
С	14
Ag	1

The most striking observation regarding this sample is that spot 1 in the unexposed region displayed Al and no Si and Ag while the other spots displayed Si and Ag but no Al. All spots displayed O. The composition of spot 2 in the unexposed region was thus similar to both spots 3 and 4 in the exposed region.

# EOIM-3 04-34: Al + MgF₂ overcoat

Figures 109 and 110 are general surveys taken from the unexposed region of this sample. The results of semiquantitative analyses from these spectra are presented in tables 36 and 37, respectively. Figures 111 and 112 are general surveys taken from the exposed region of this sample. The results of semiquantitative analyses from these spectra are presented in tables 38 and 39, respectively.

Table 36: Semiquantitative Composition of Spot 1 from the Unexposed Region of Sample EOIM-3 04-34 from Figure 109

Element	Approximate Atomic Percent
F	35
0	11
C	21
Mg	32
Mg Ar	1

Table 37: Semiquantitative Composition of Spot 2 from the Unexposed Region of Sample EOIM-3 04-34 from Figure 110

Element	Approximate Atomic Percent
F	36
0	11
С	22
Mg	30
Ar	1

Table 38: Semiquantitative Composition of Spot 3 from the Exposed Region of Sample EOIM-3 04-34 from Figure 111

Element	Approximate Atomic Percent
F	20
0	31
Si	10
С	14
Mg	25
Ar	<1

Table 39: Semiquantitative Composition of Spot 4 from the Exposed Region of Sample EOIM-3 04-34 from Figure 112

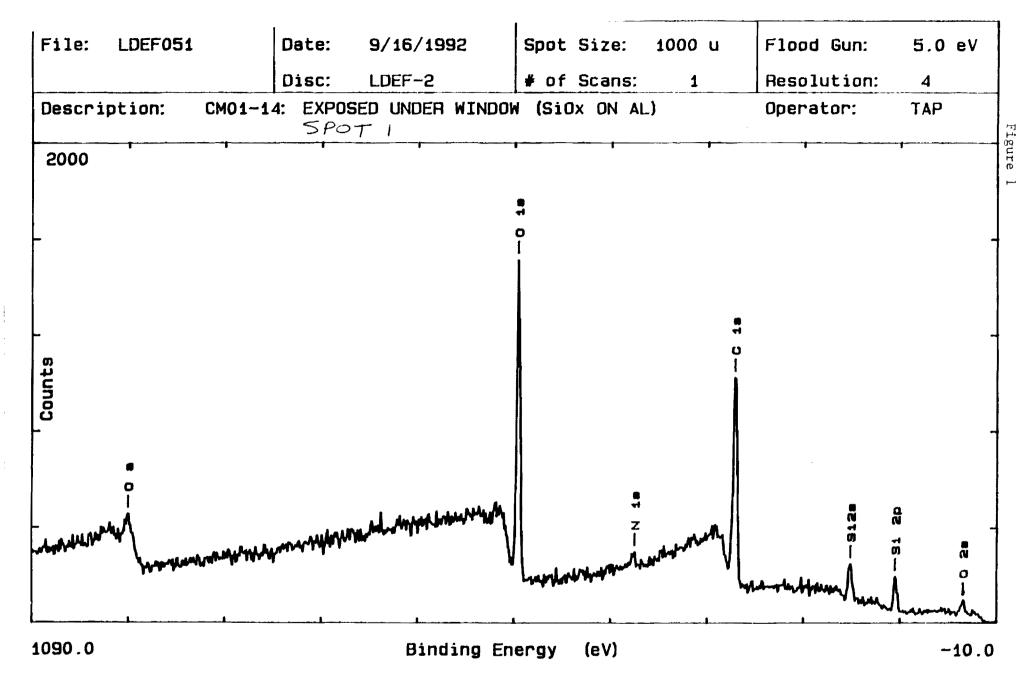
Element	Approximate Atomic Percent
F	21
Ö	27
Si	13
С	17
Mg	21
Ar	1

The unexposed and exposed regions of this sample differed in that 1) the unexposed region displayed no Si while the exposed region did and 2) there is about 2.5 times more 0 on the exposed region than on the unexposed region. There is also a small amount of Ar present over the sample. The sample surface charged about 6 volts under x-ray bombardment which is indicative of an electrical insulator.

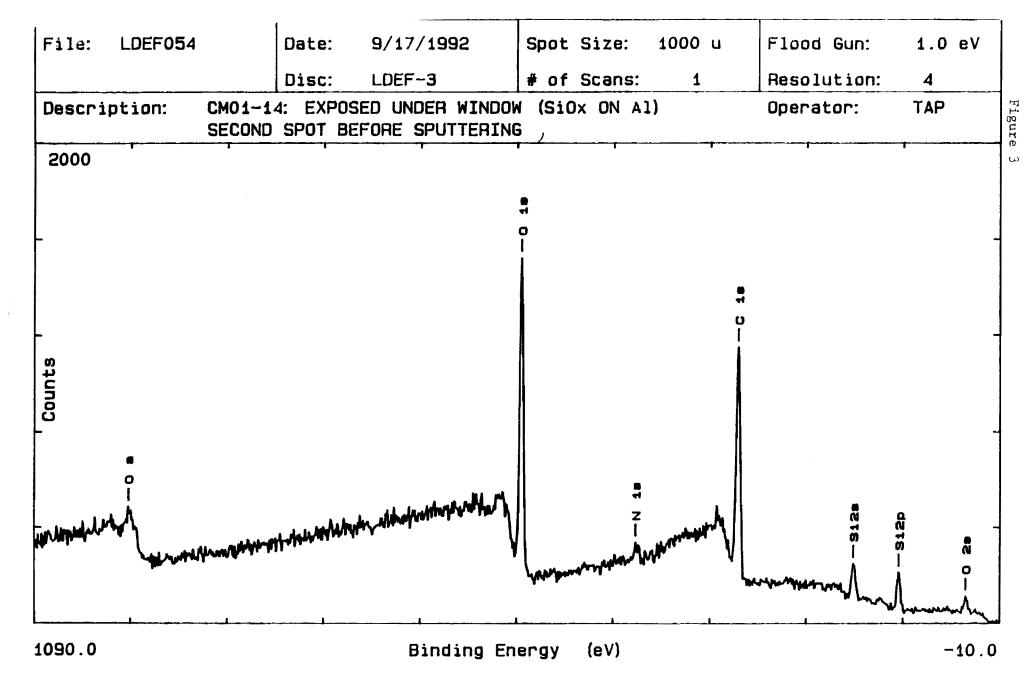
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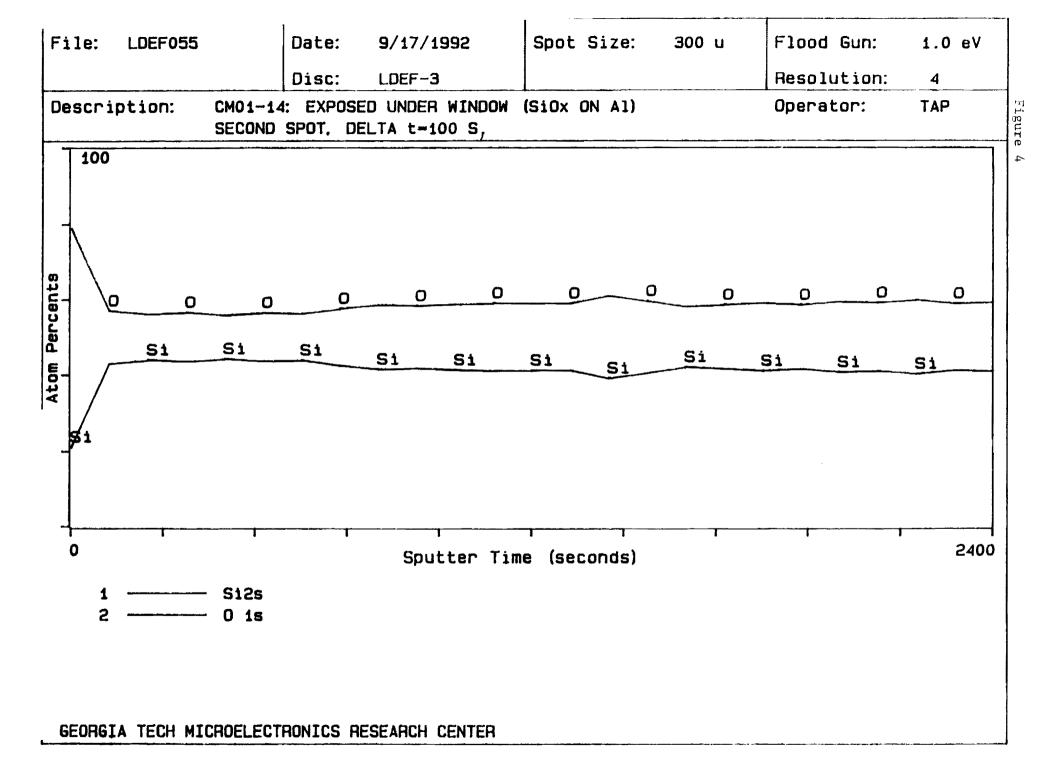
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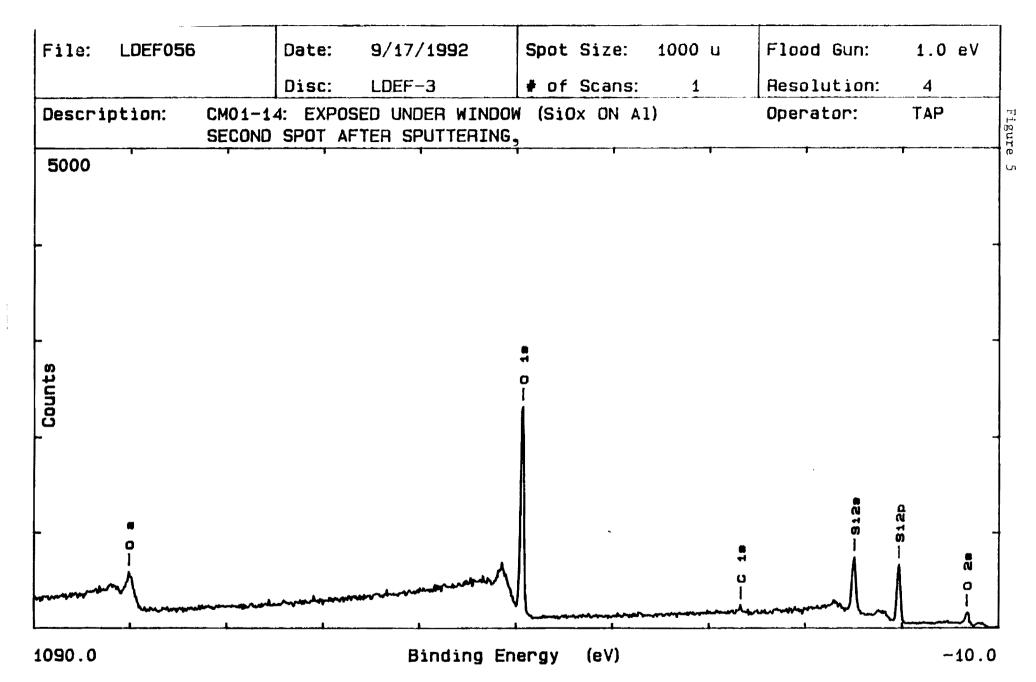


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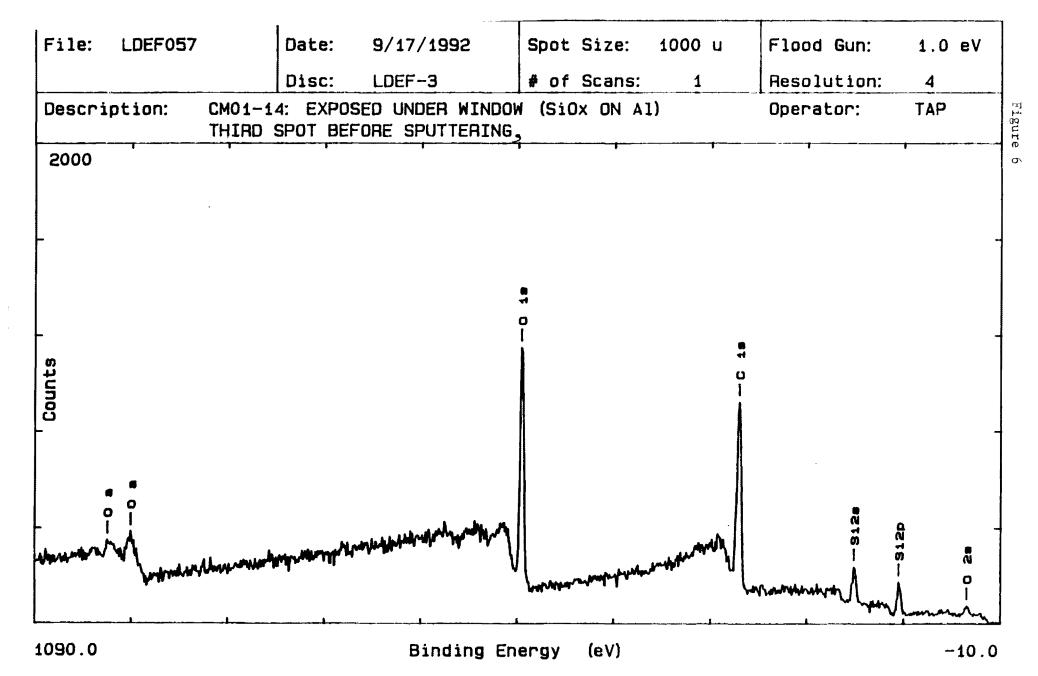


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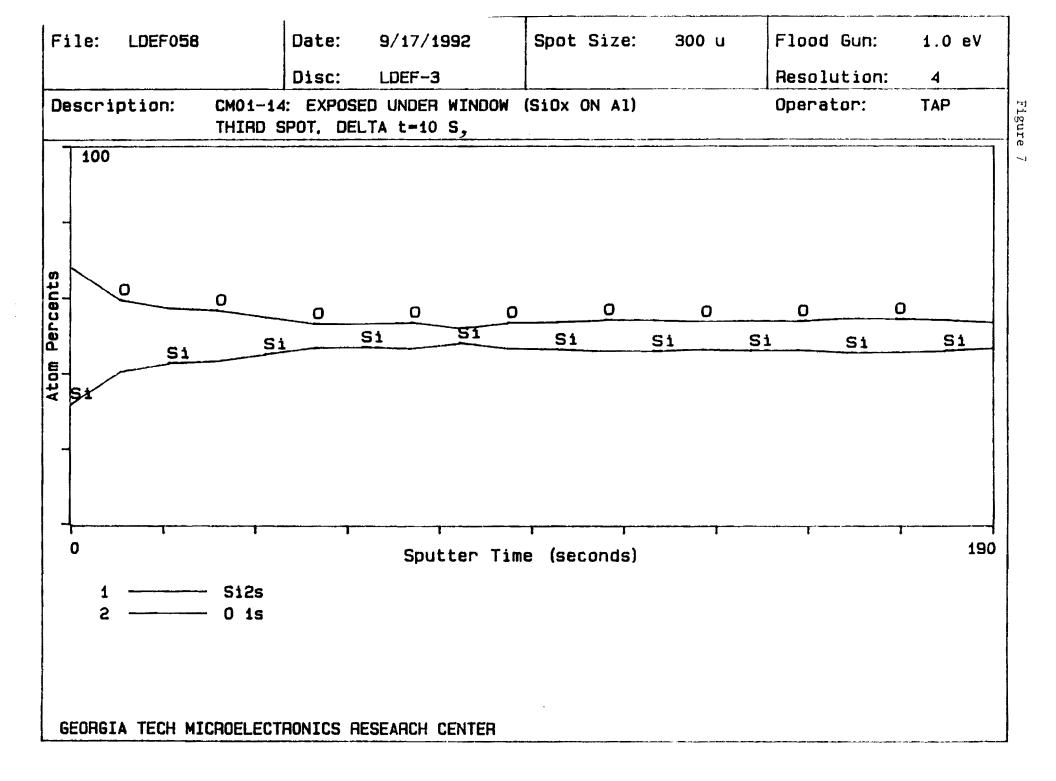


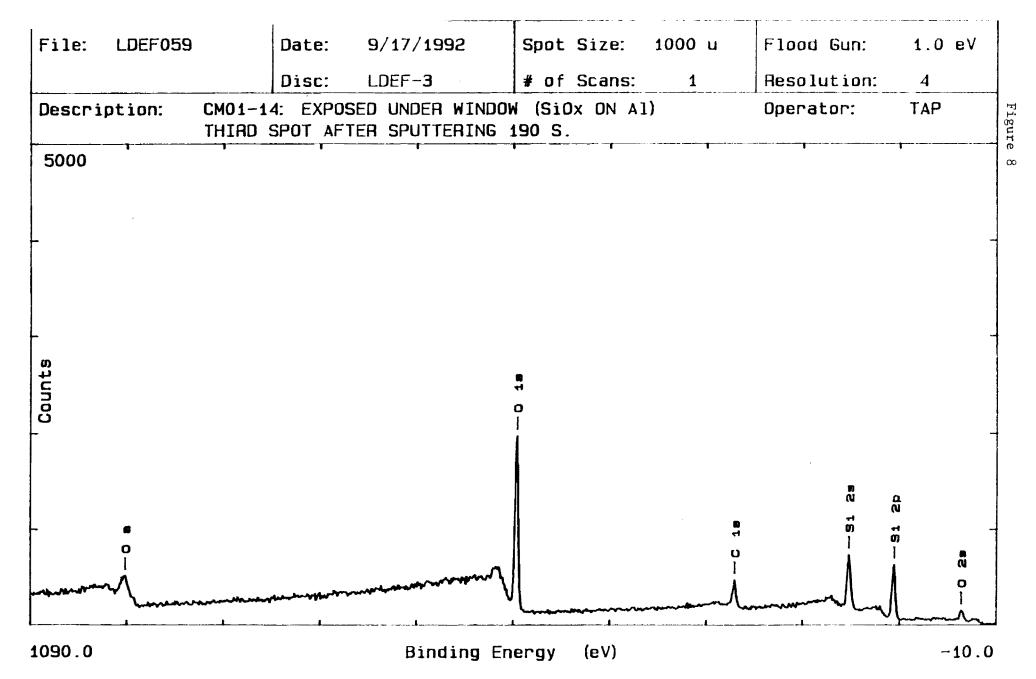


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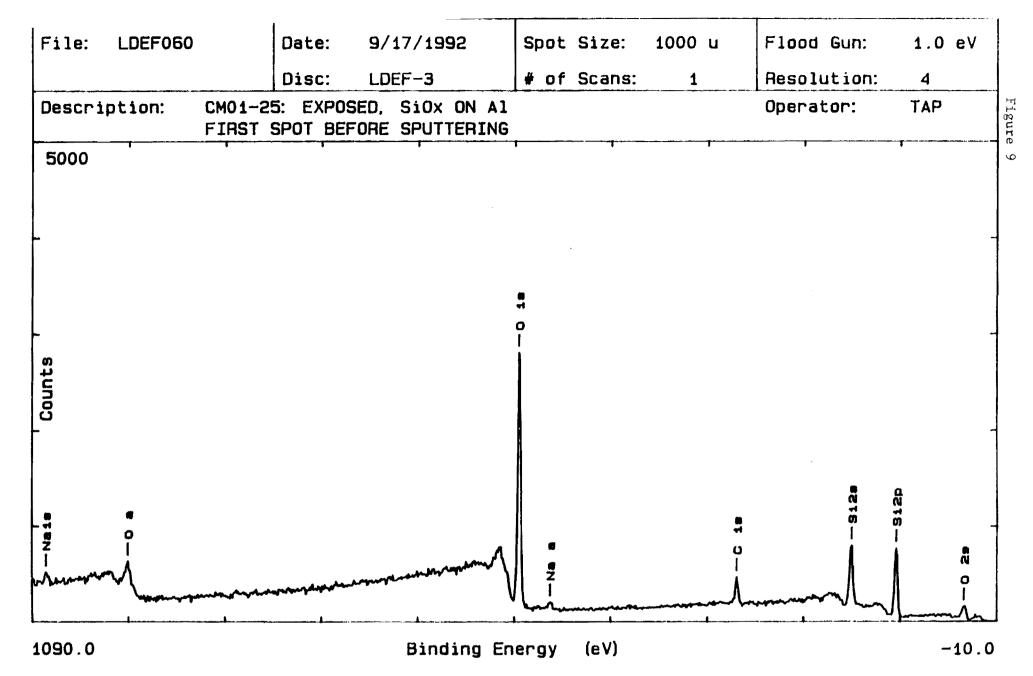


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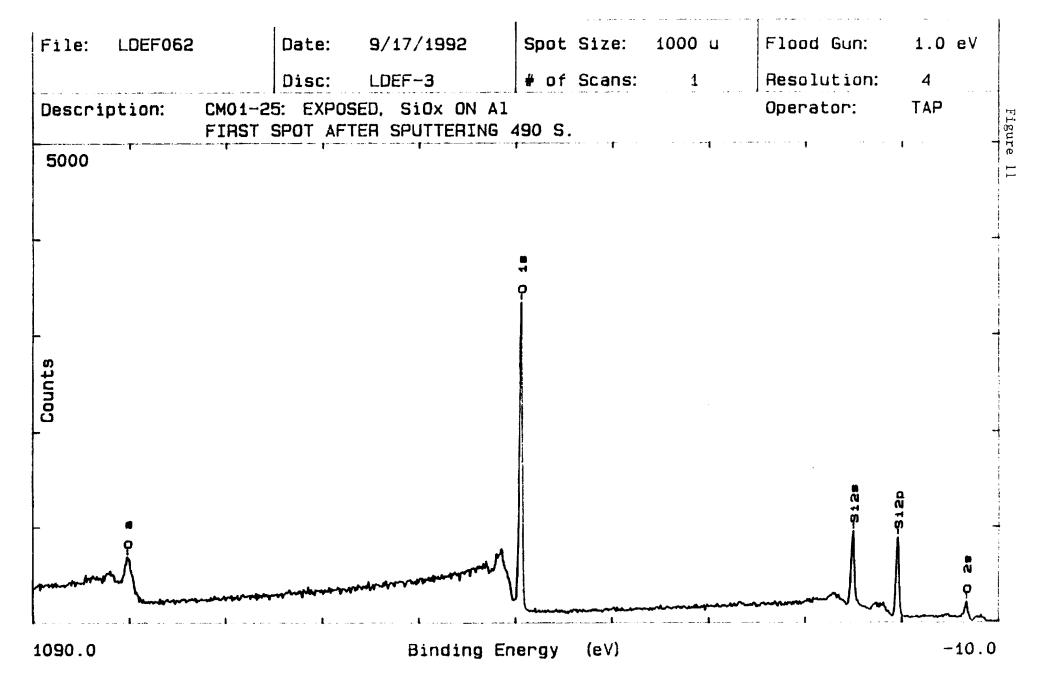




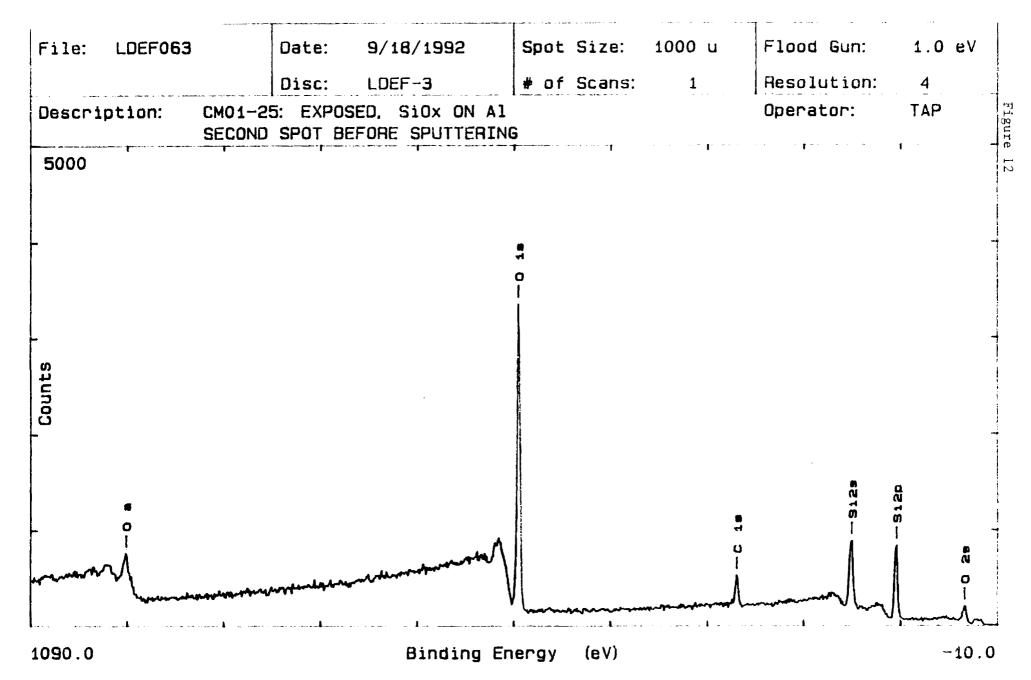
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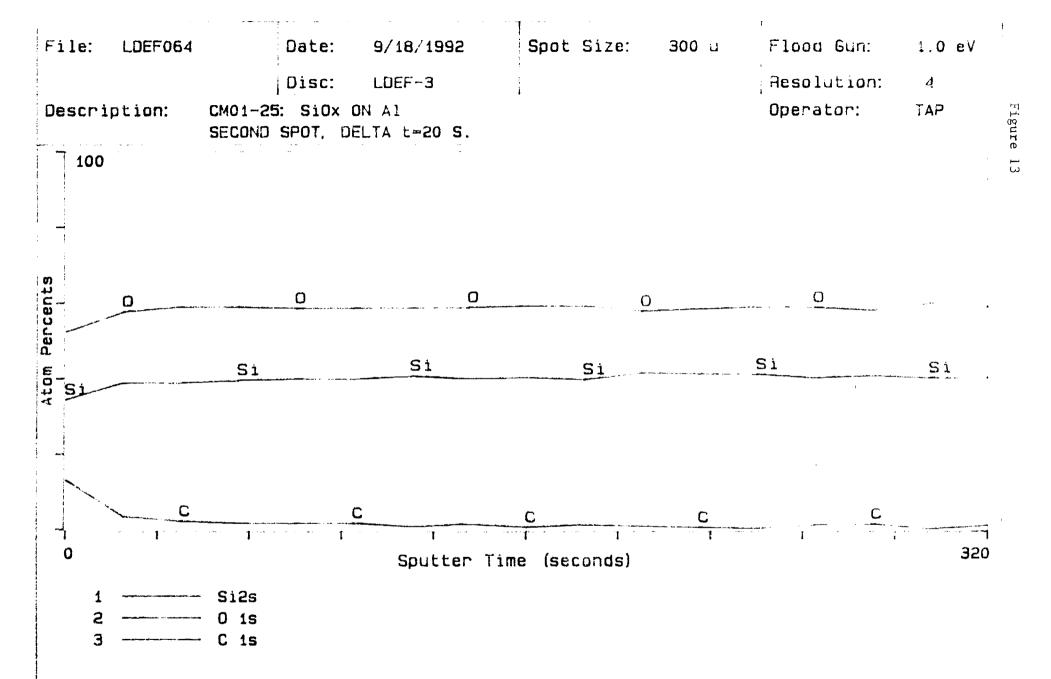
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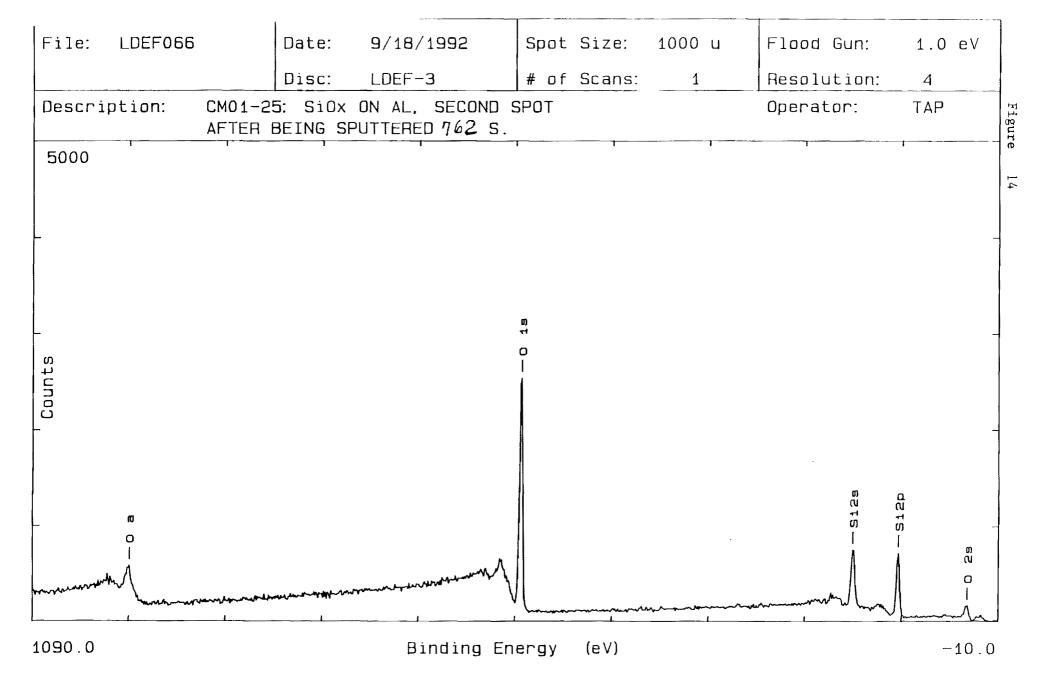


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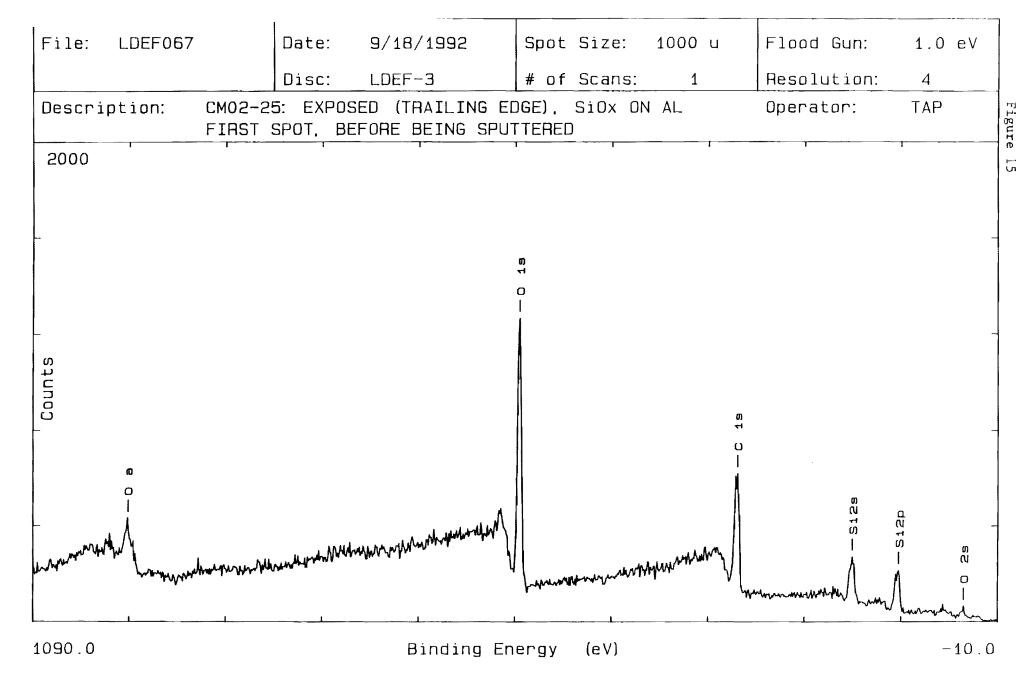


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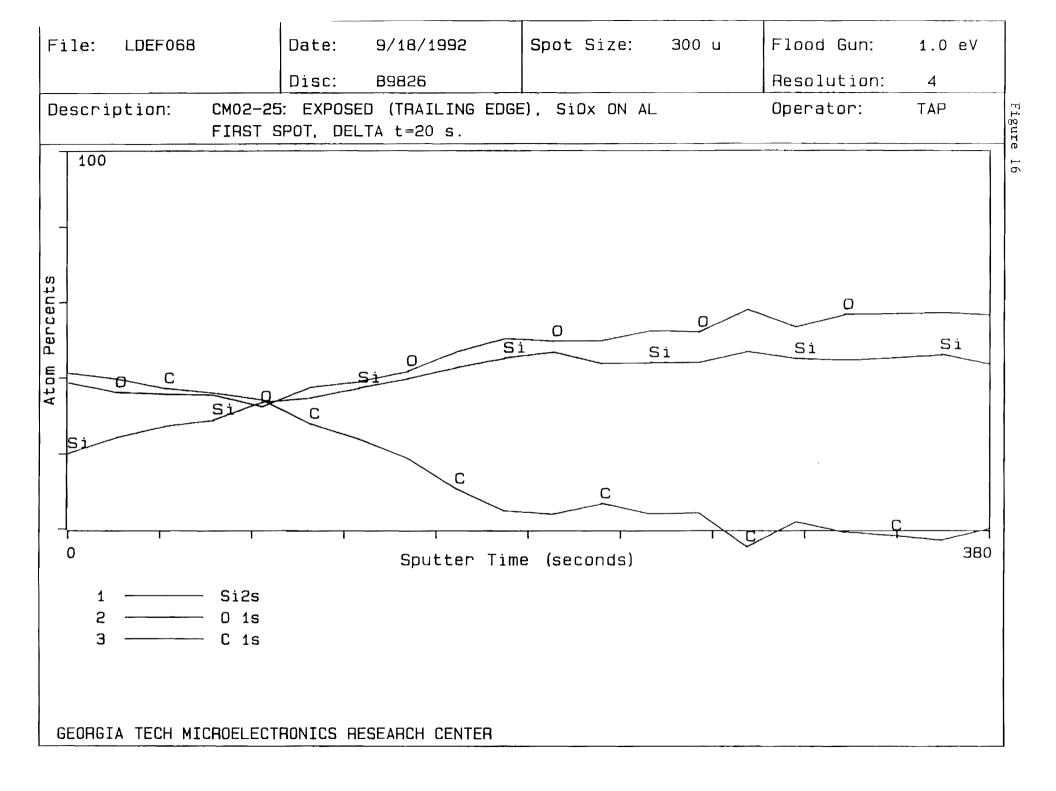


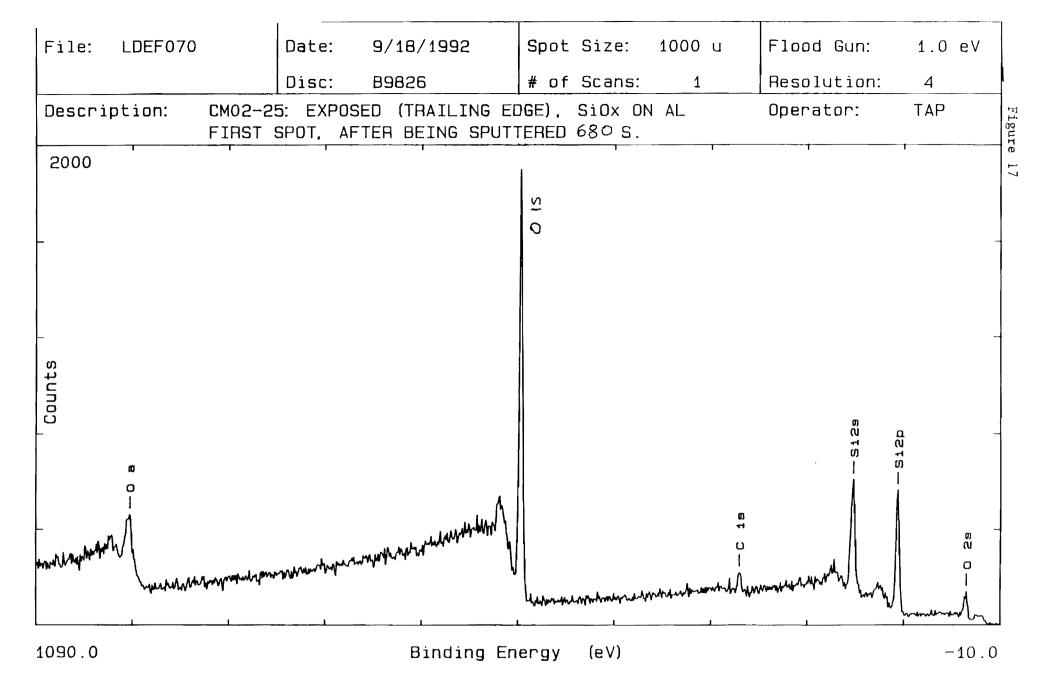


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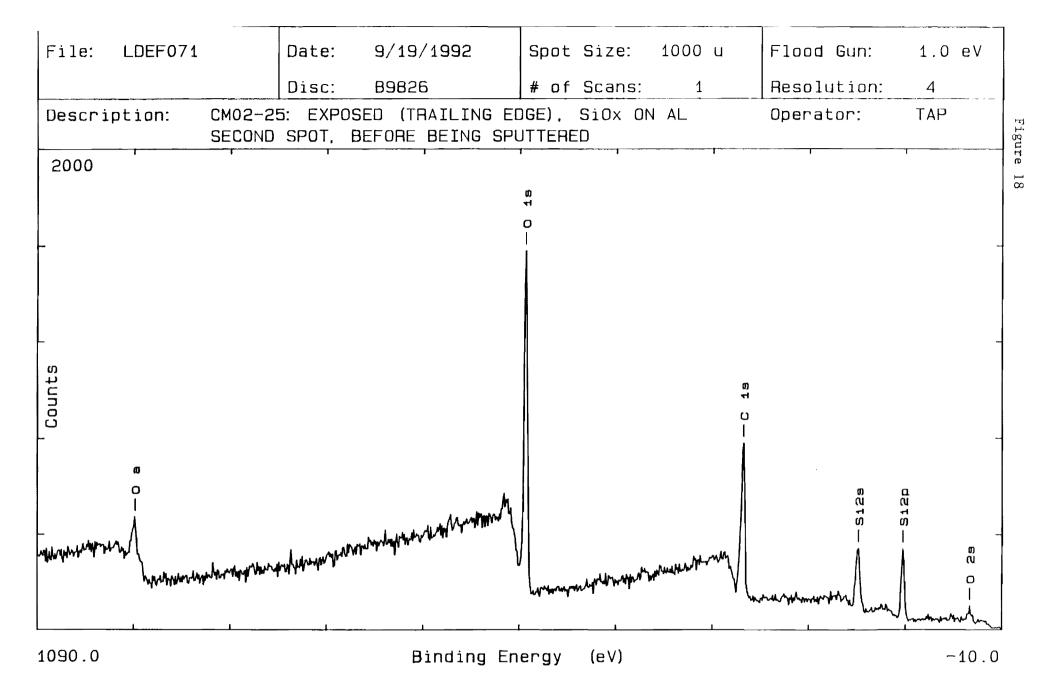


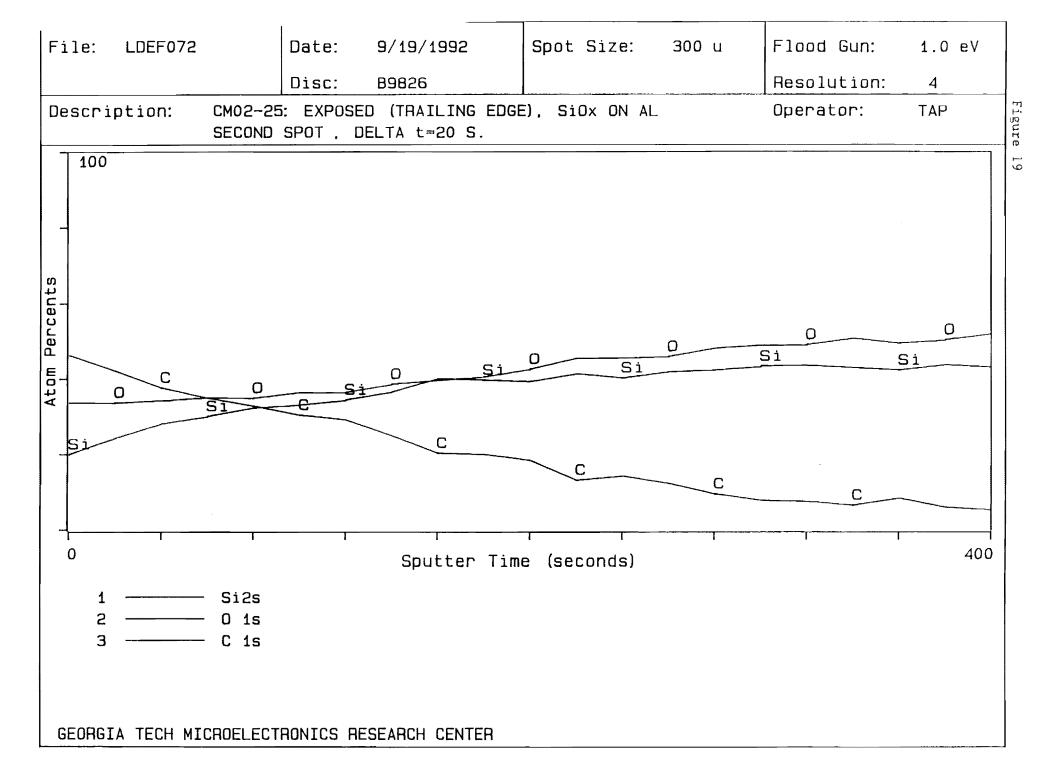
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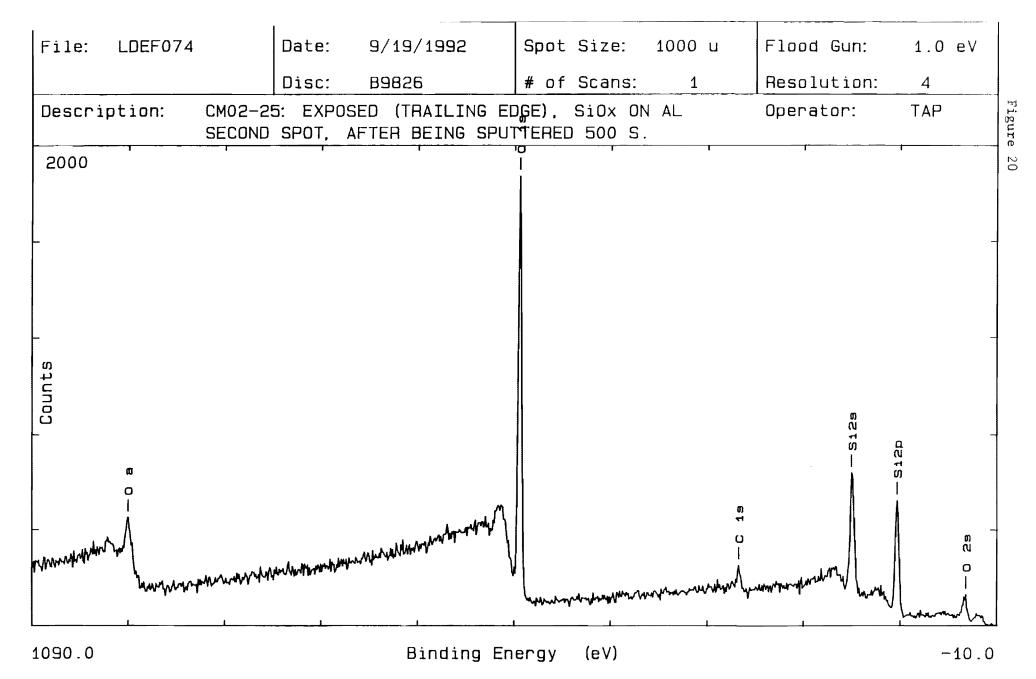


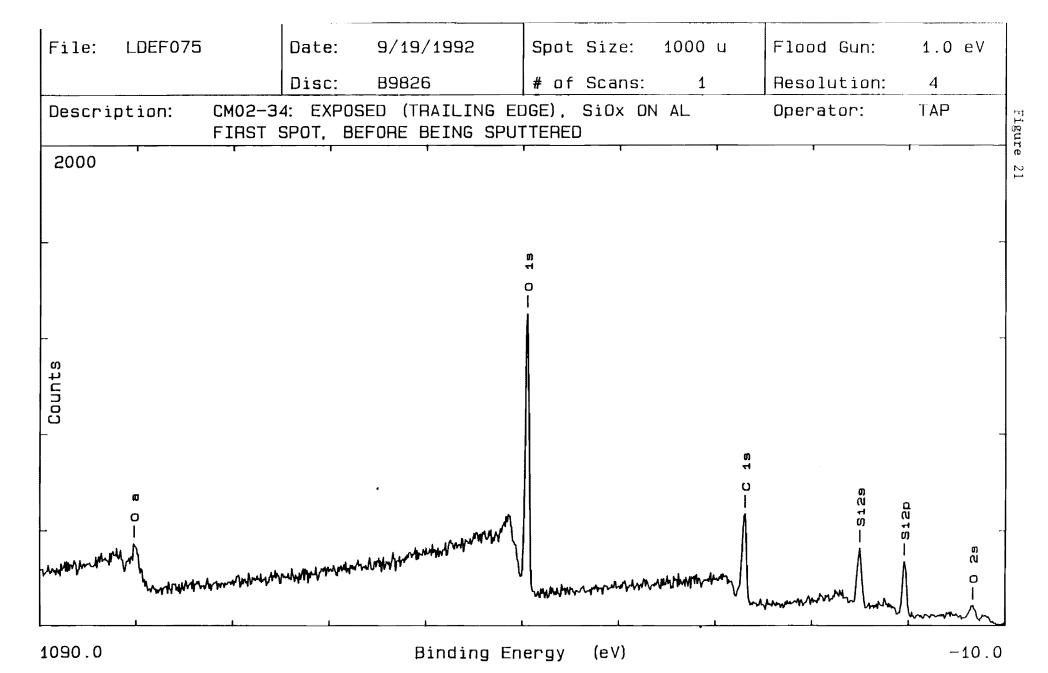


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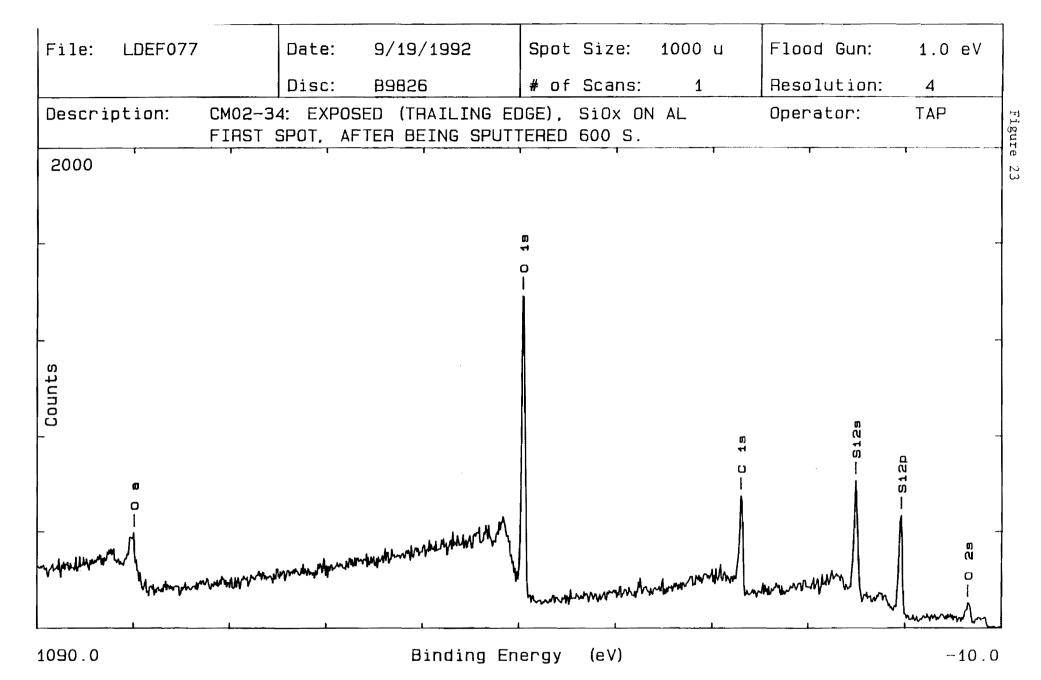


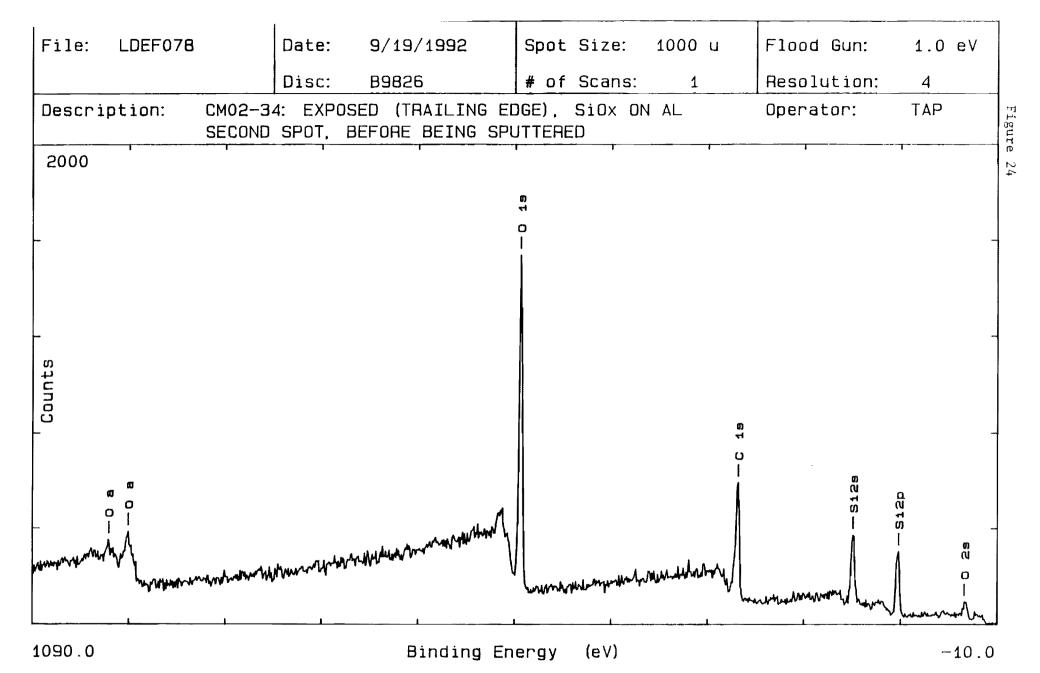




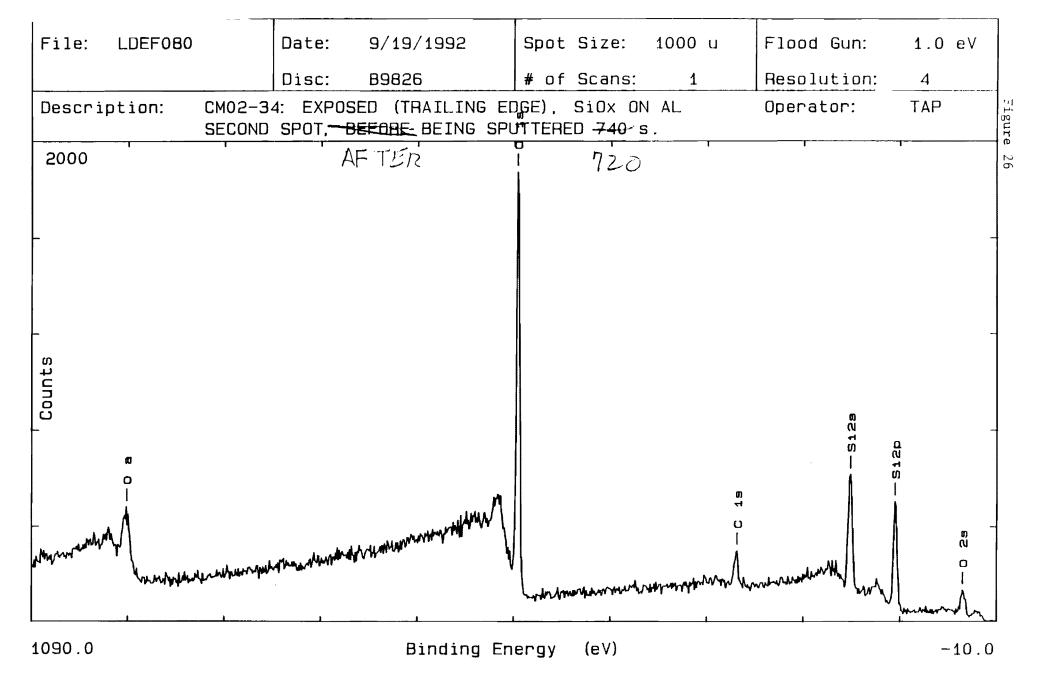


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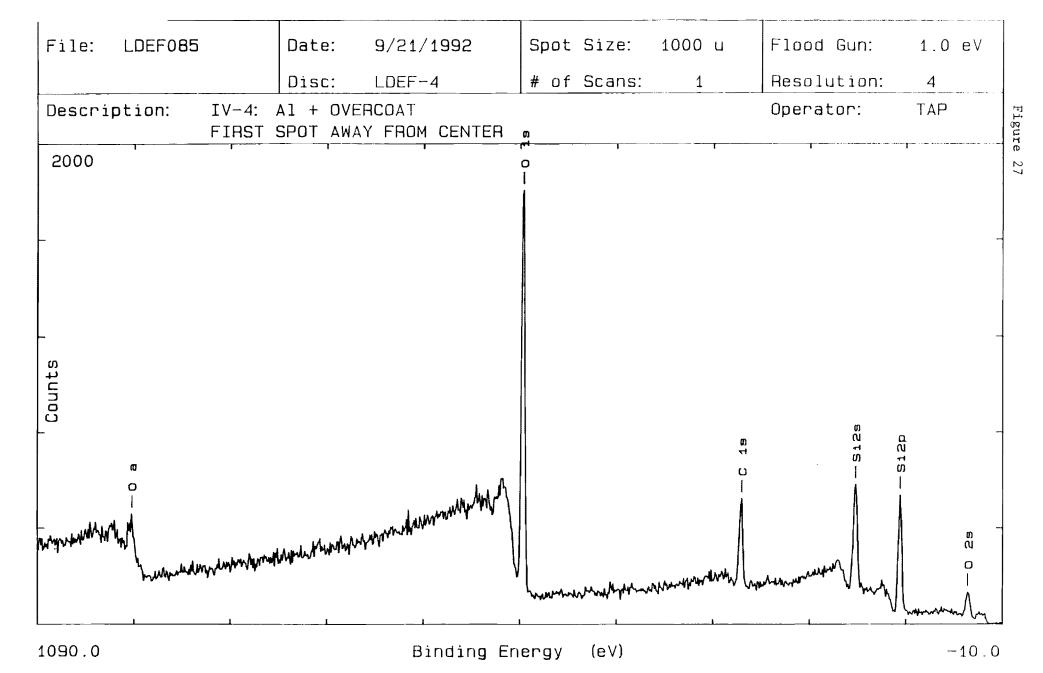




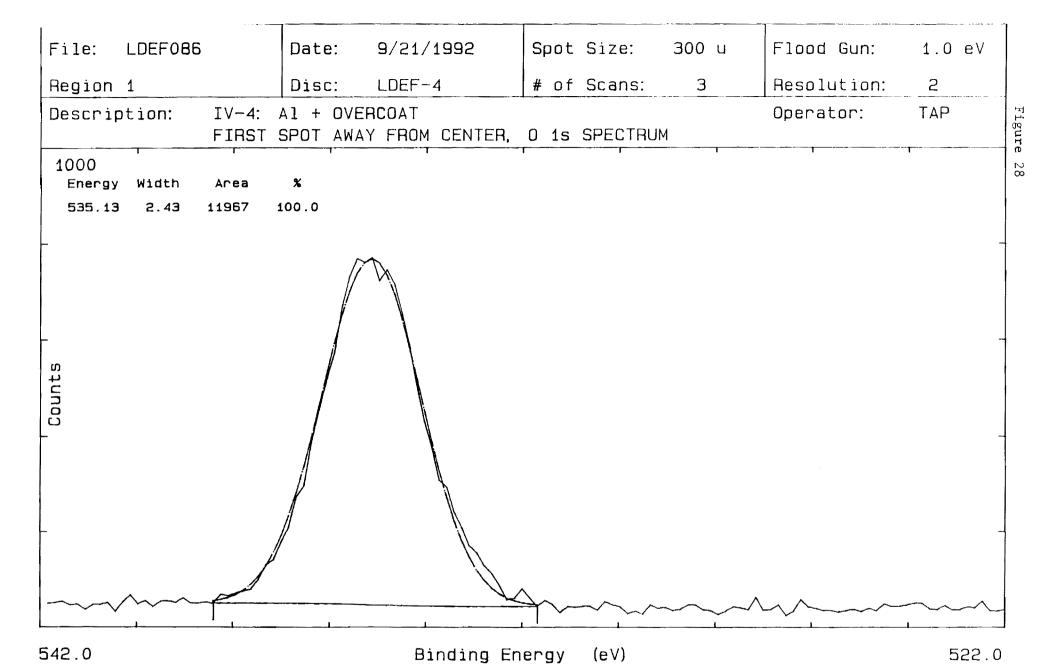
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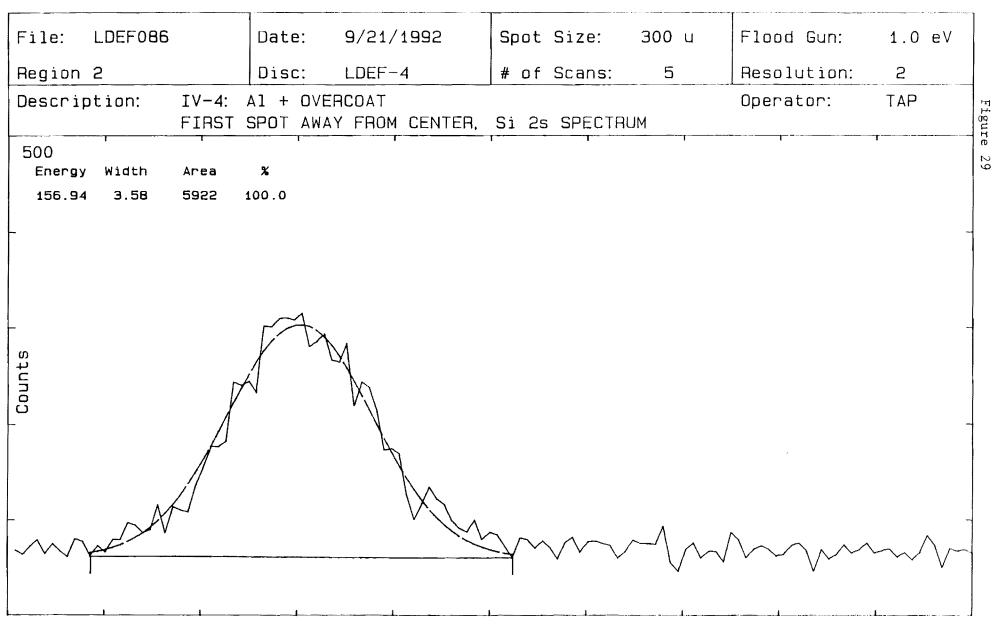
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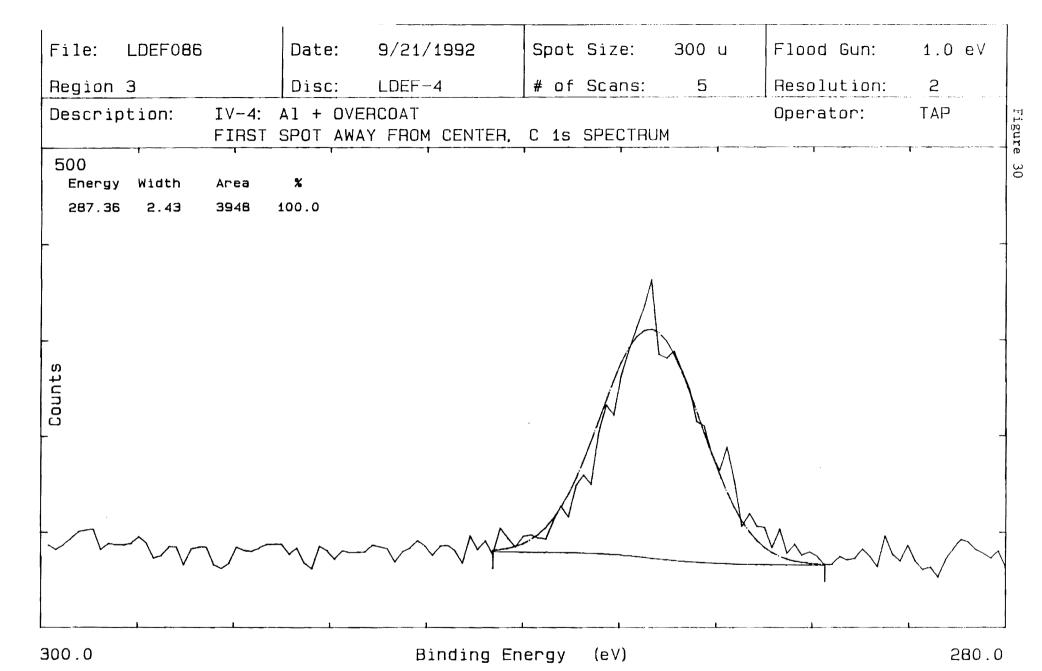
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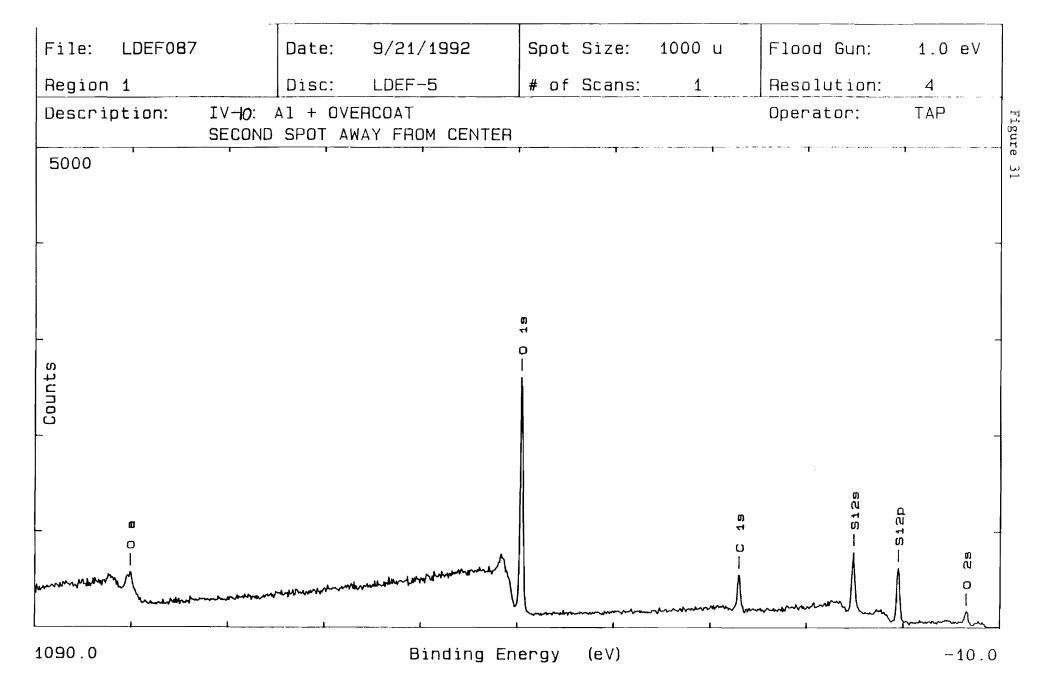


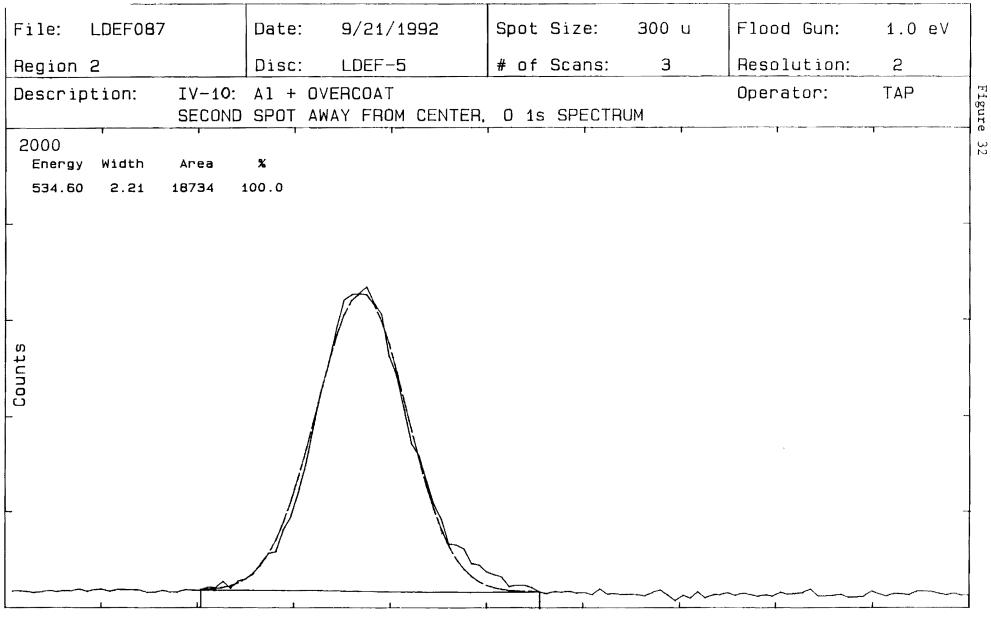
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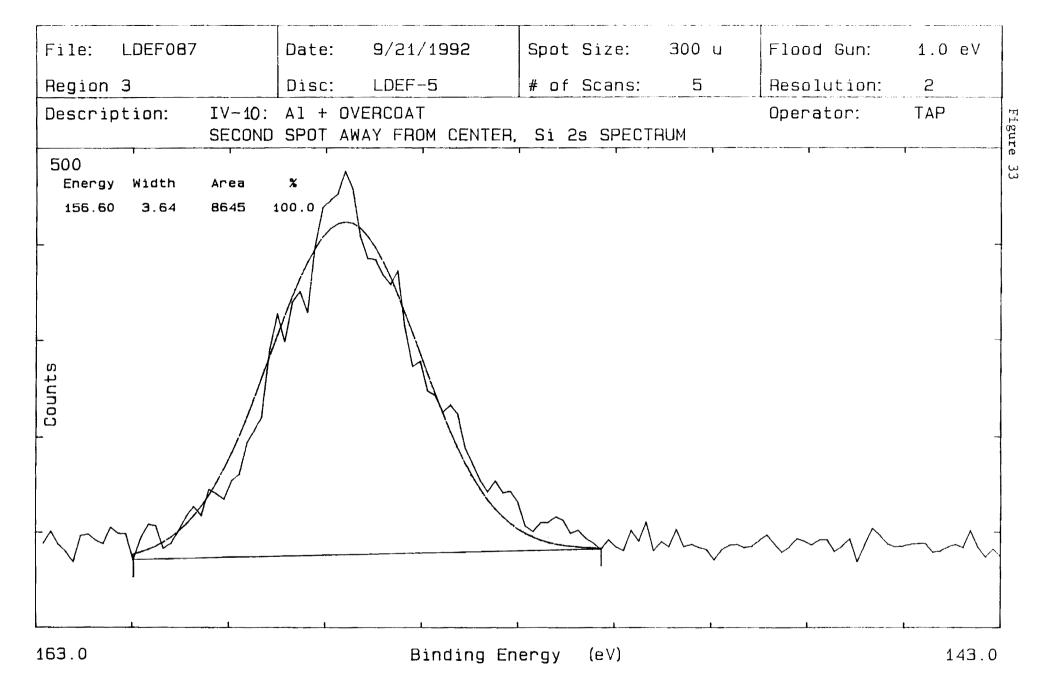
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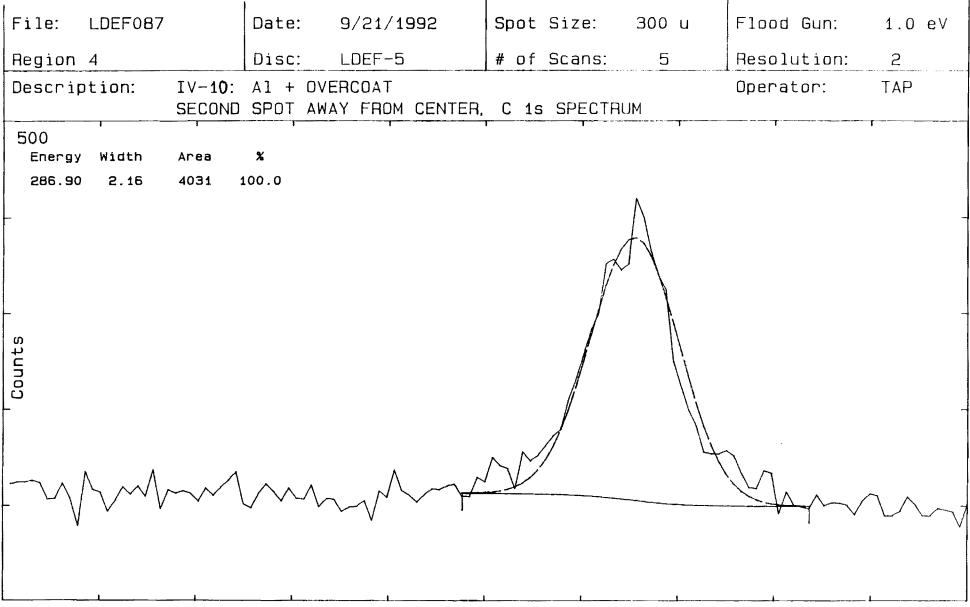




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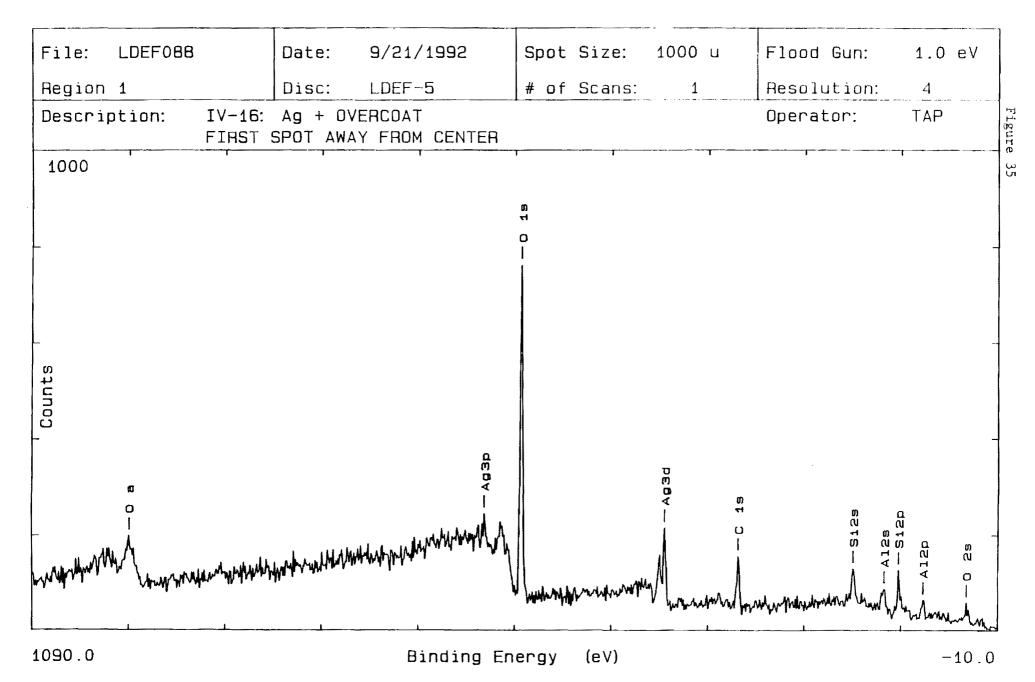


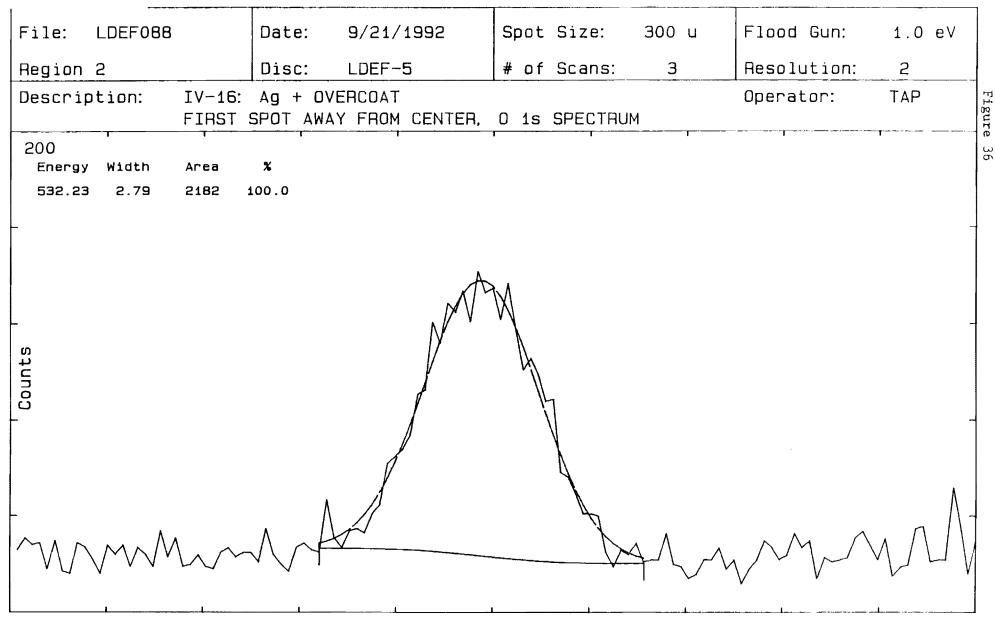
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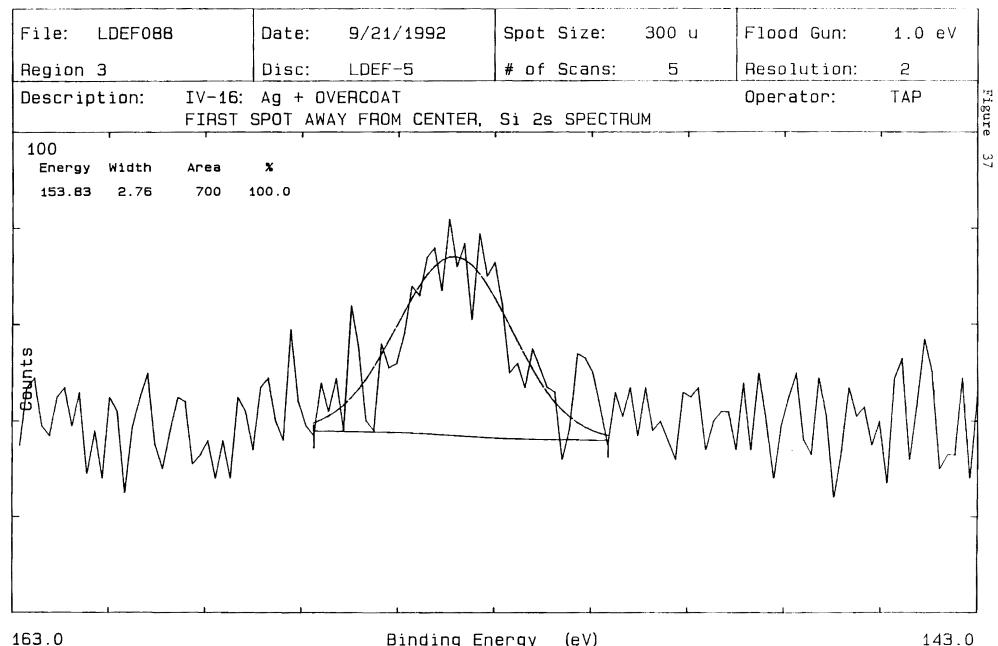
300.0 Binding Energy (eV) 280.0

Figure



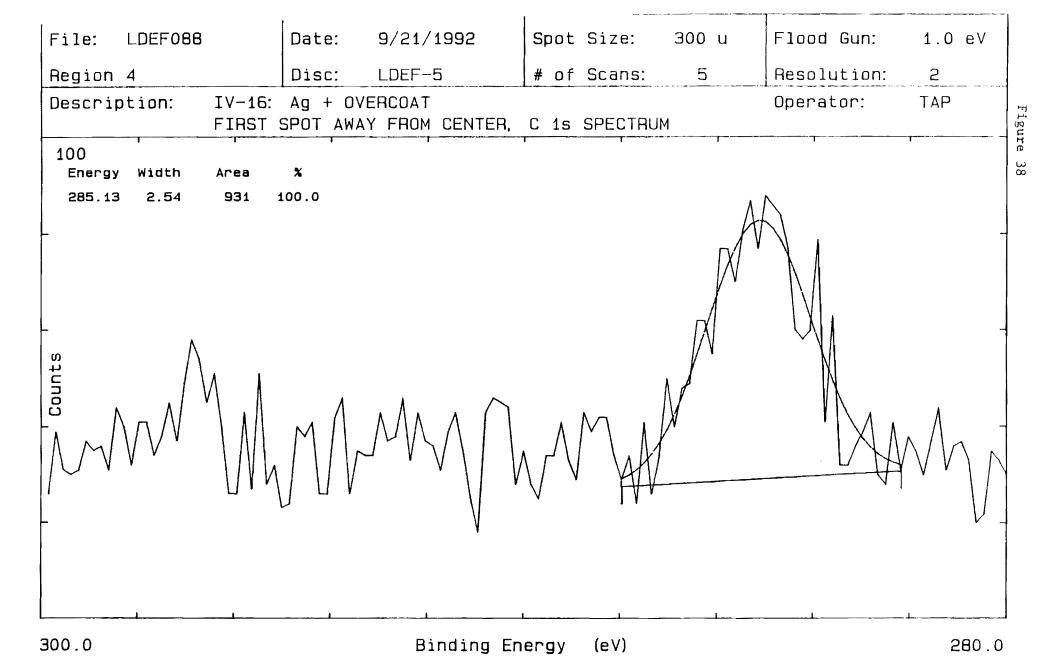


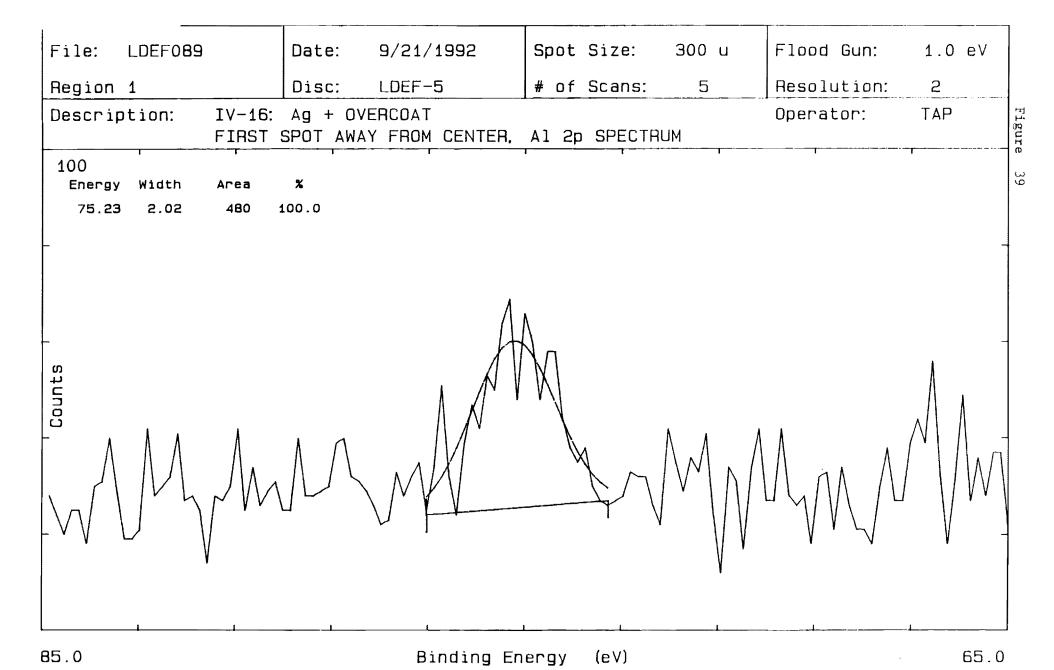
542.0 Binding Energy (eV) 522.0

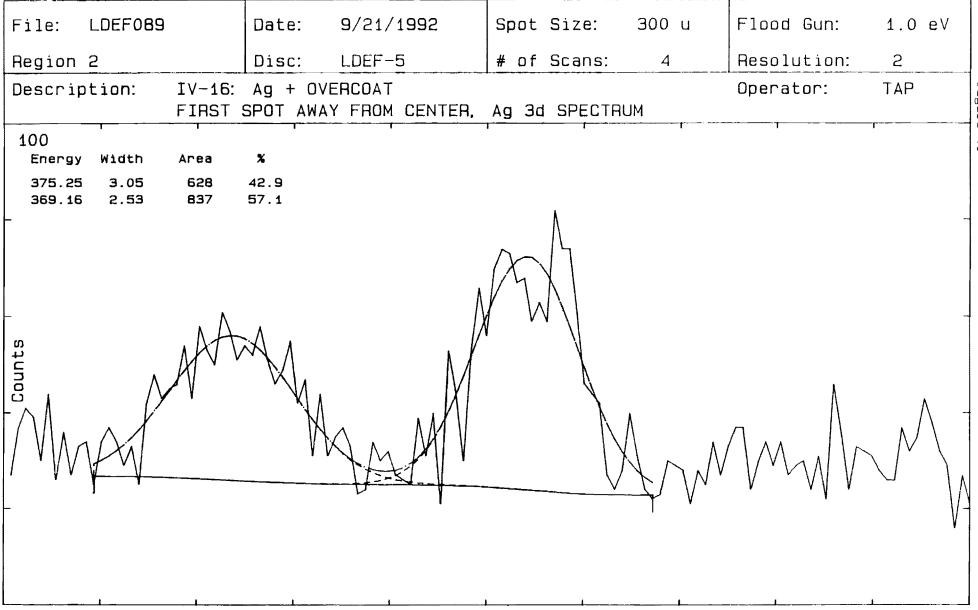


Binding Energy (eV)

143.0

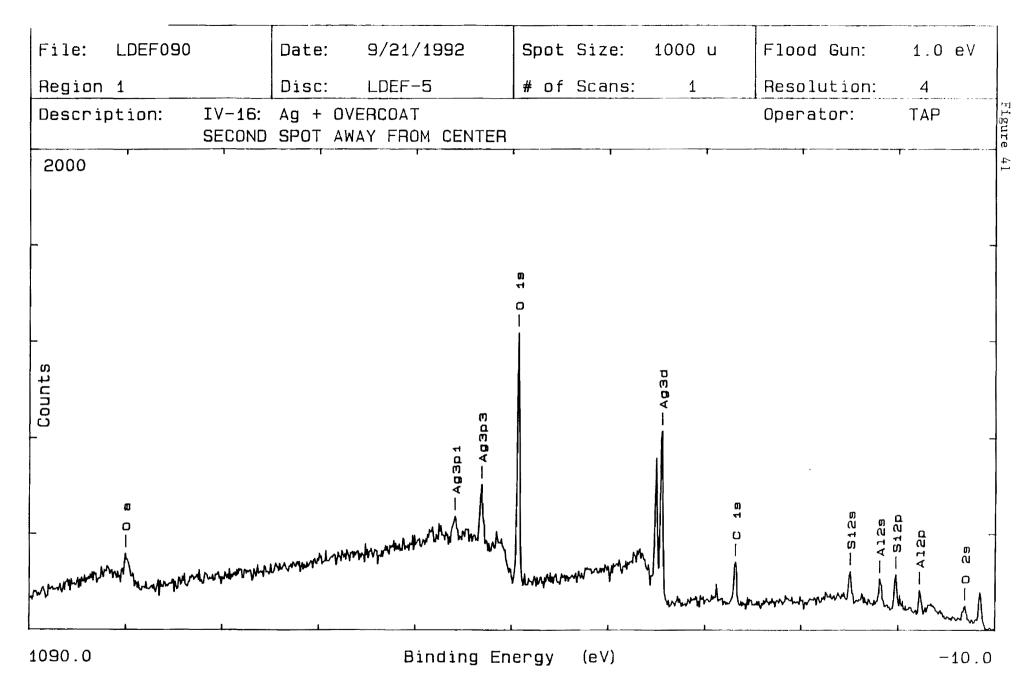


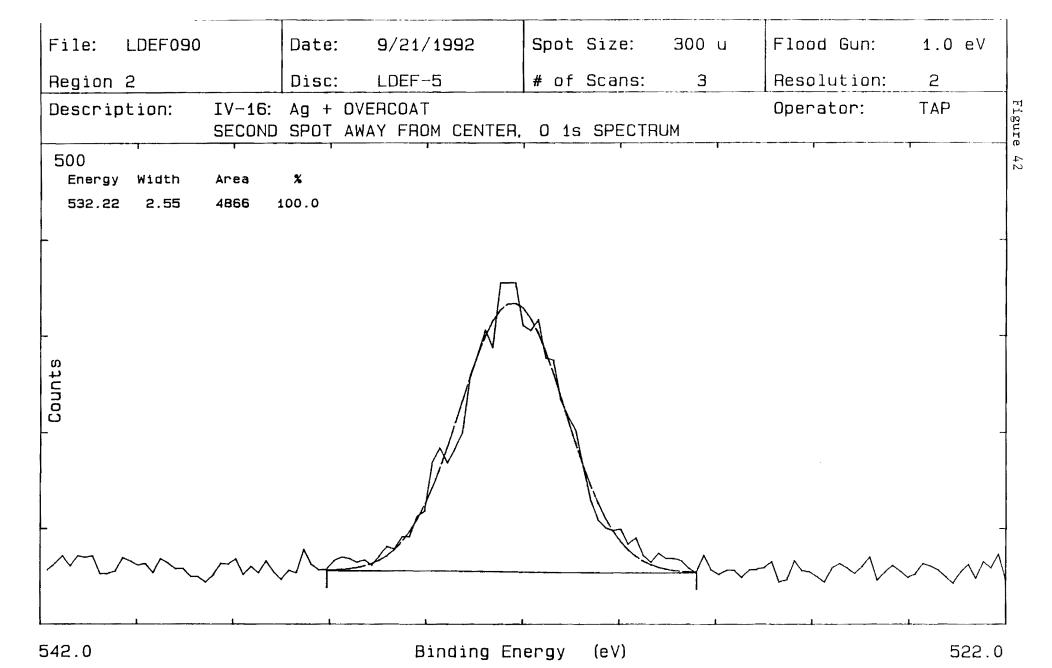




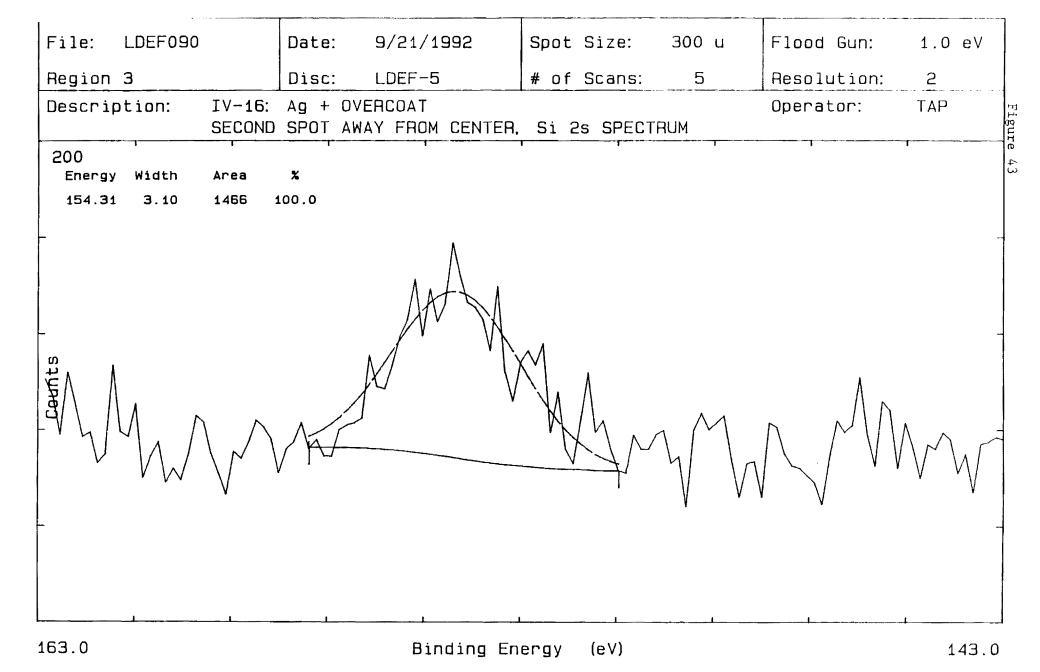
380.0 Binding Energy (eV) 360.0

Figure 40

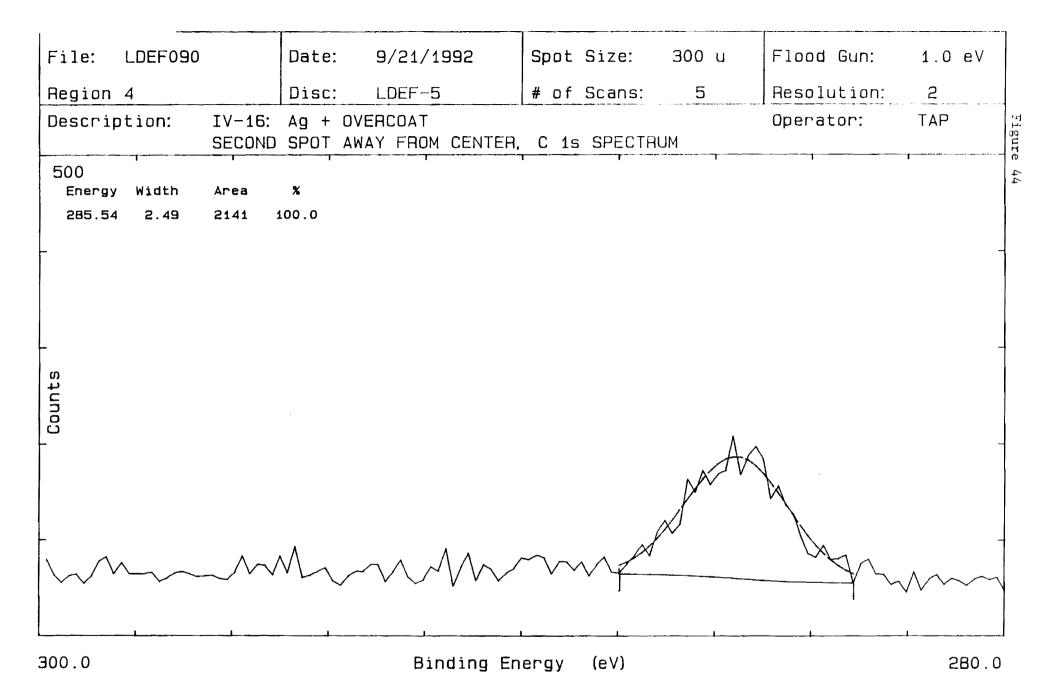


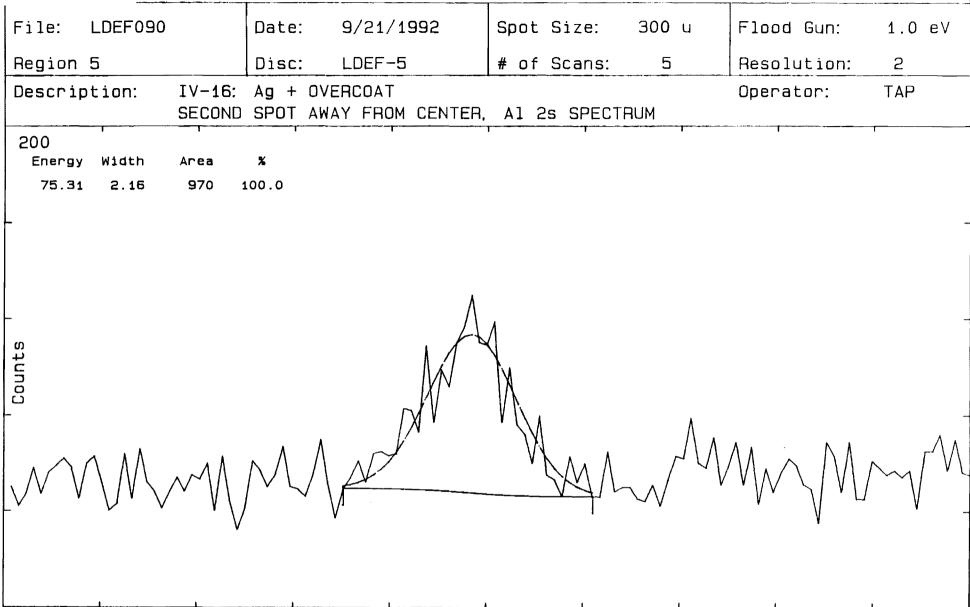


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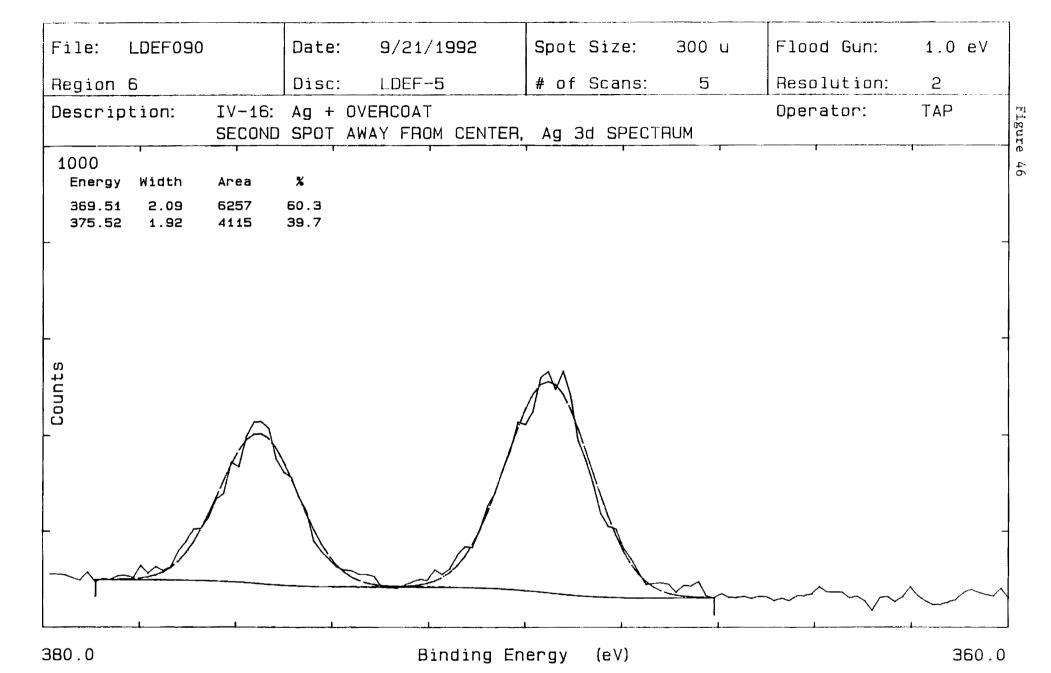
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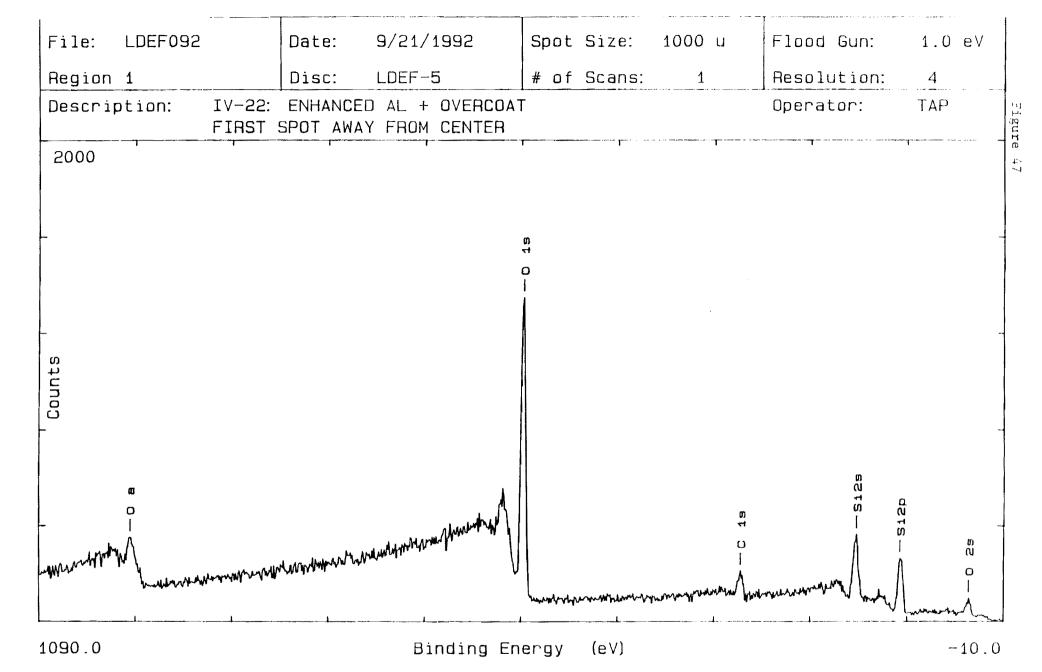




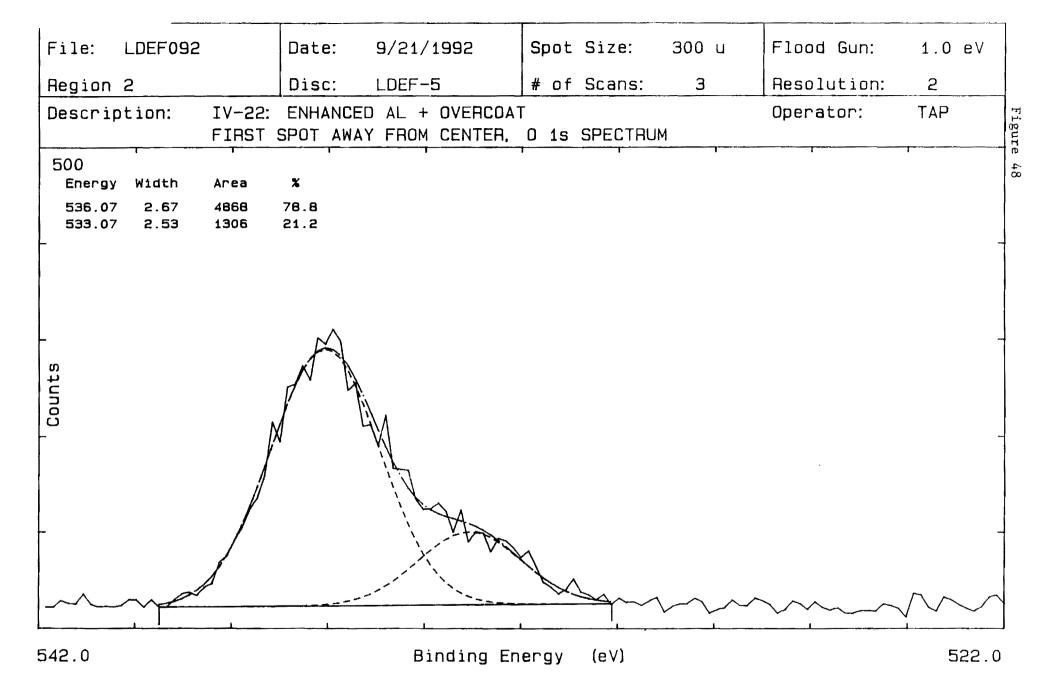
85.0 Binding Energy (eV) 65.0

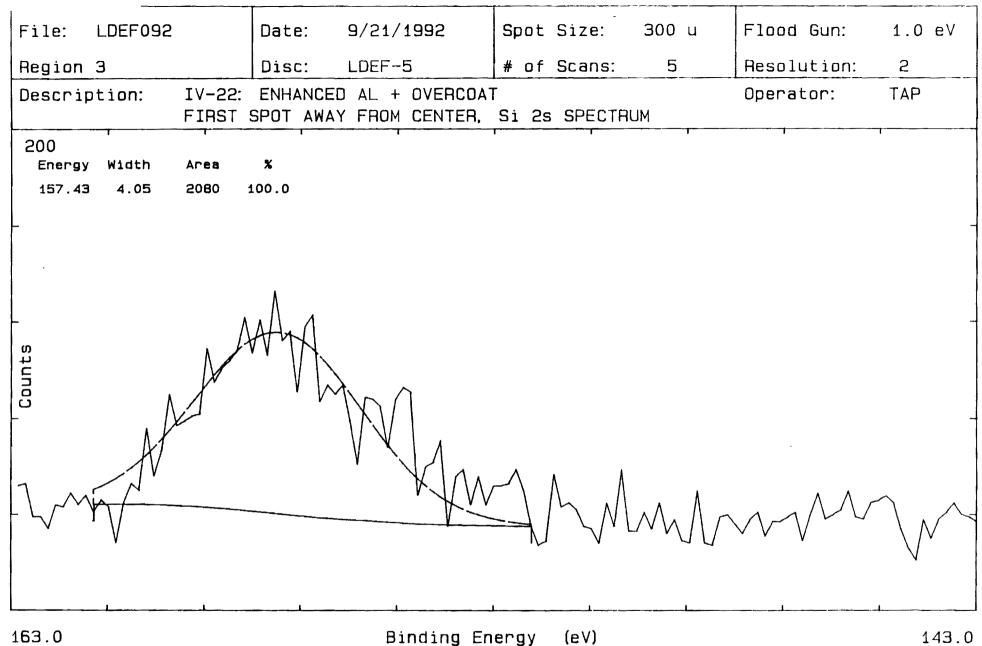
Figure 45

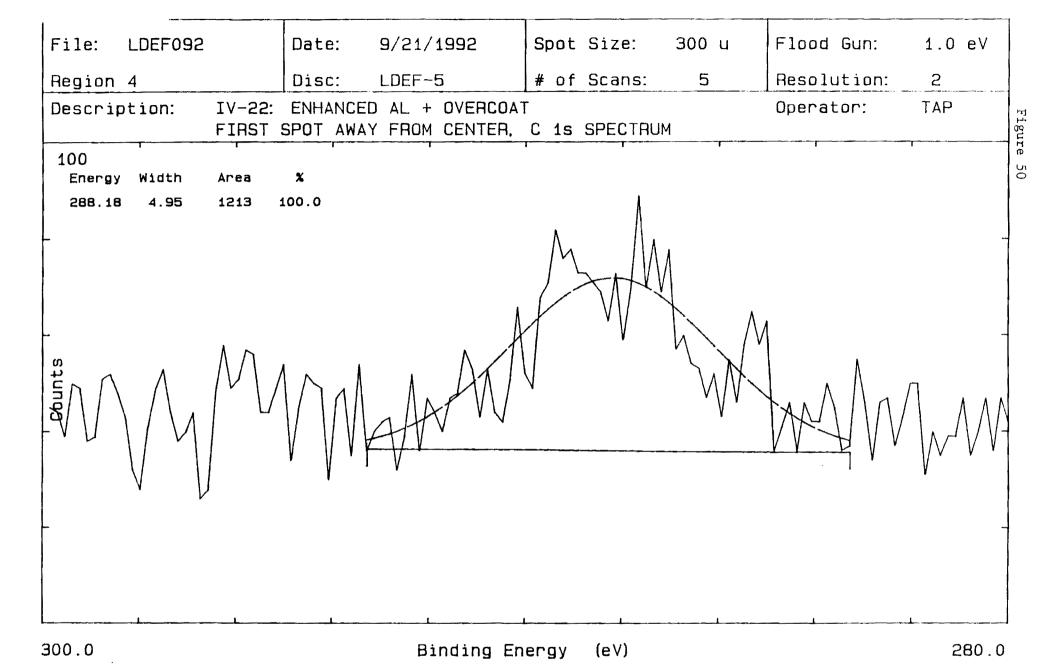


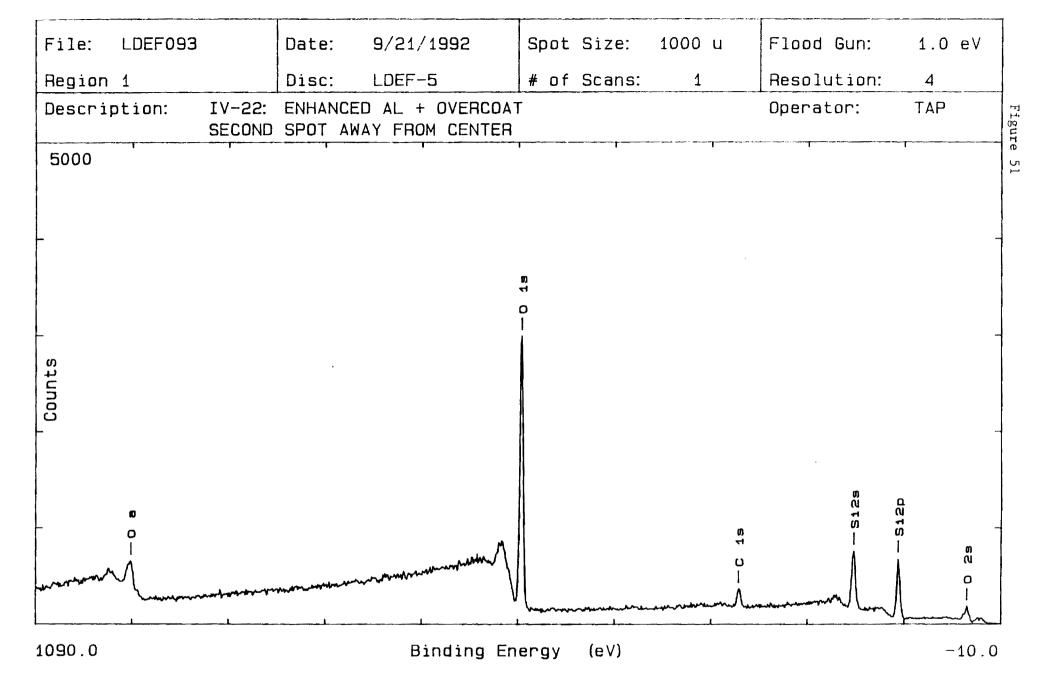


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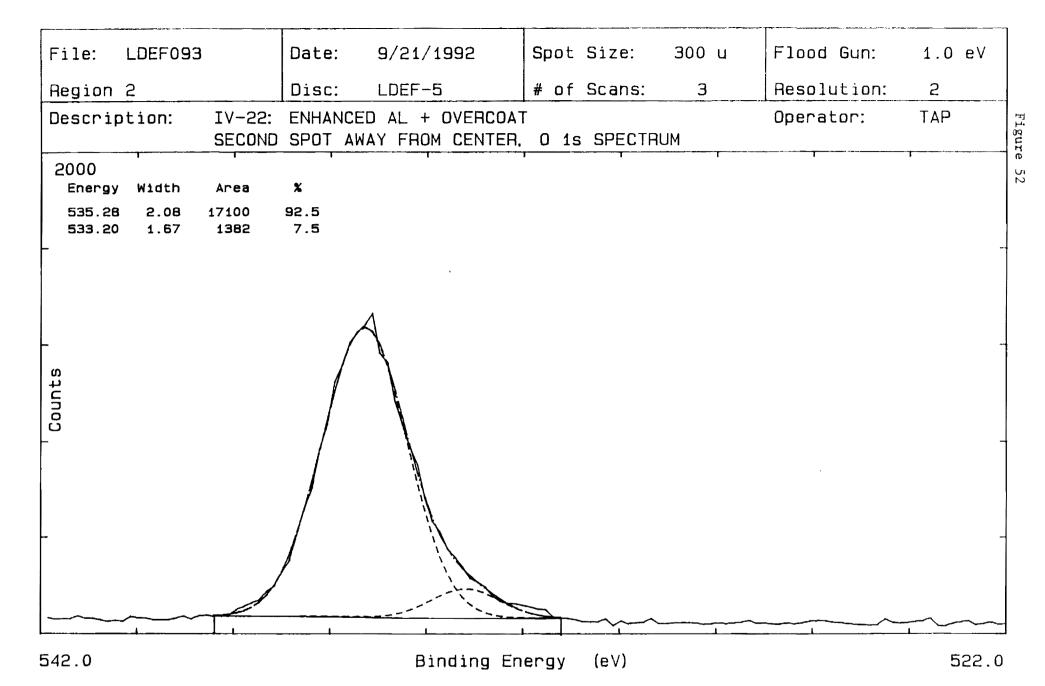


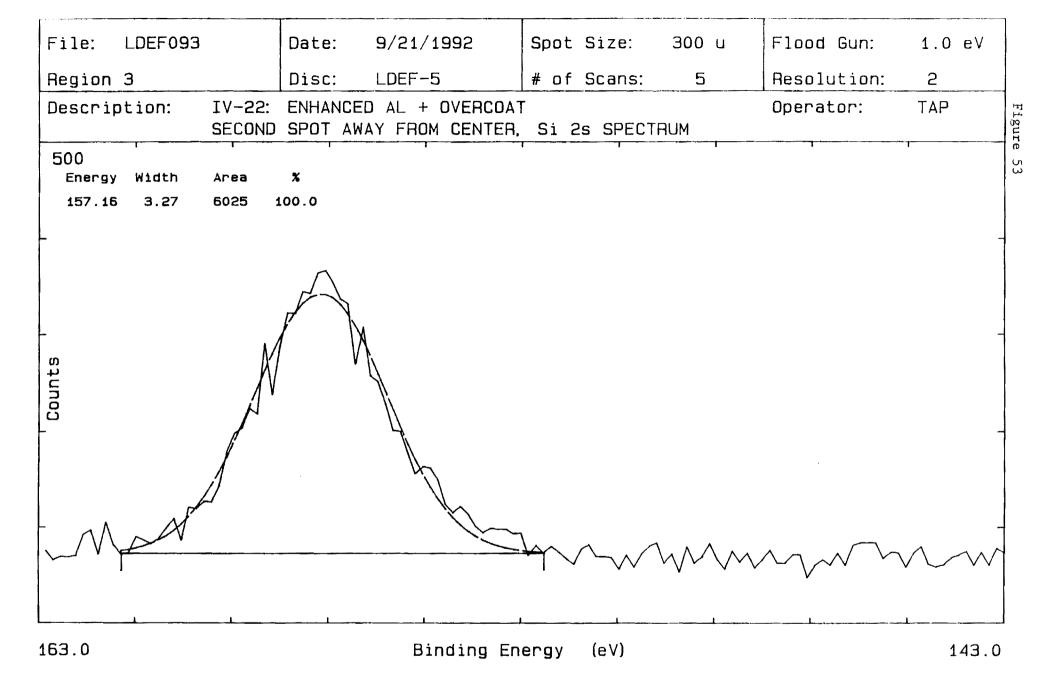


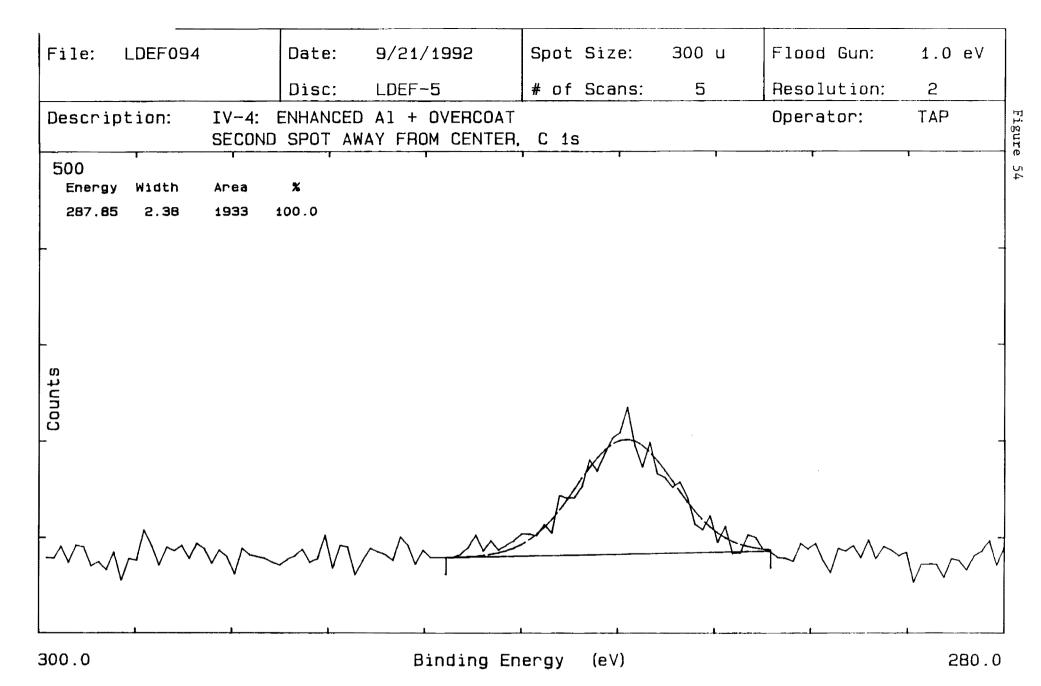


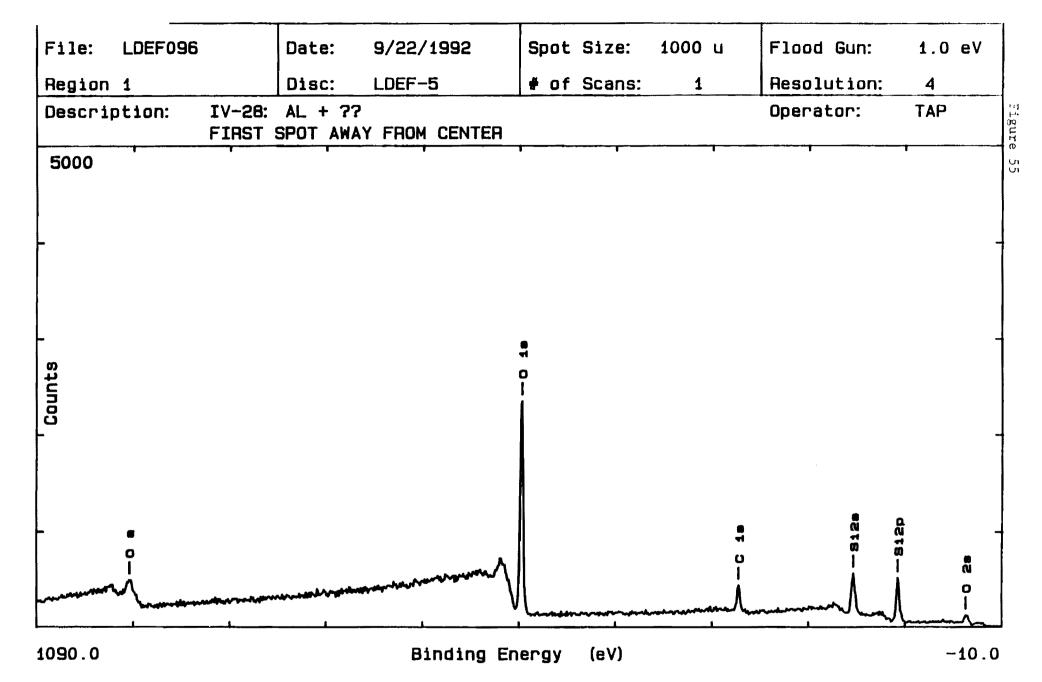


GEORGIA TECH MICROELECTRONICS RESEARCH CENTER

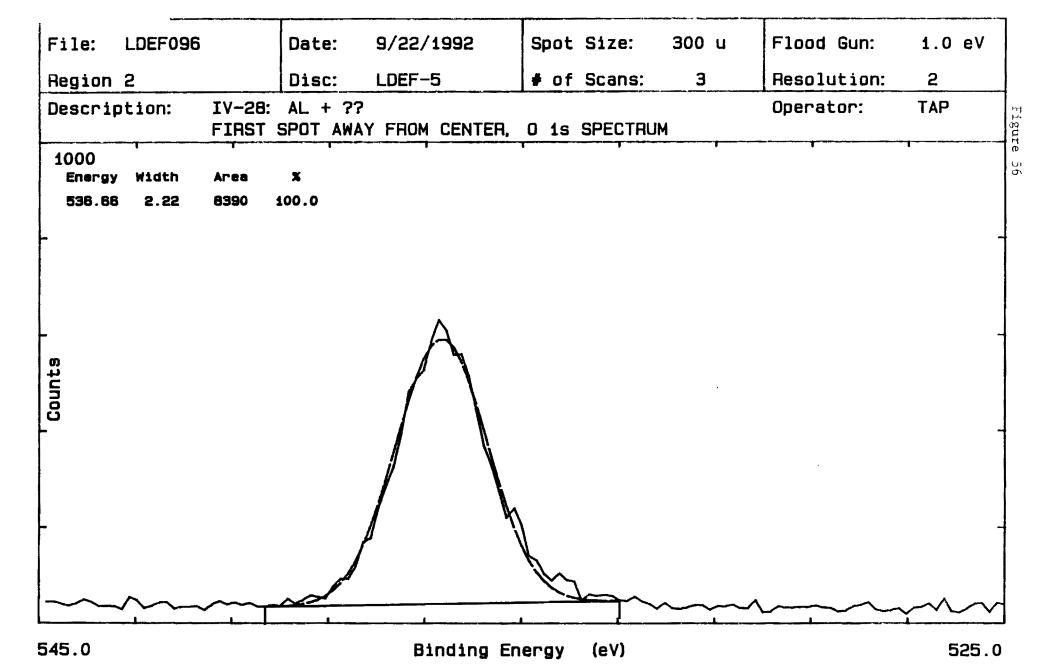


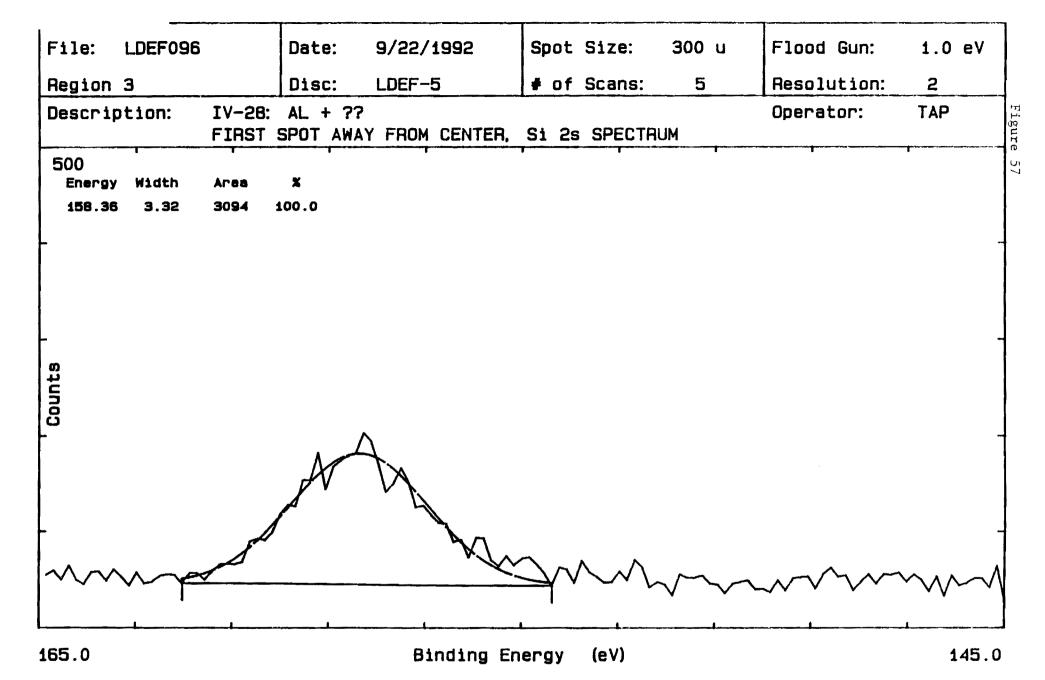


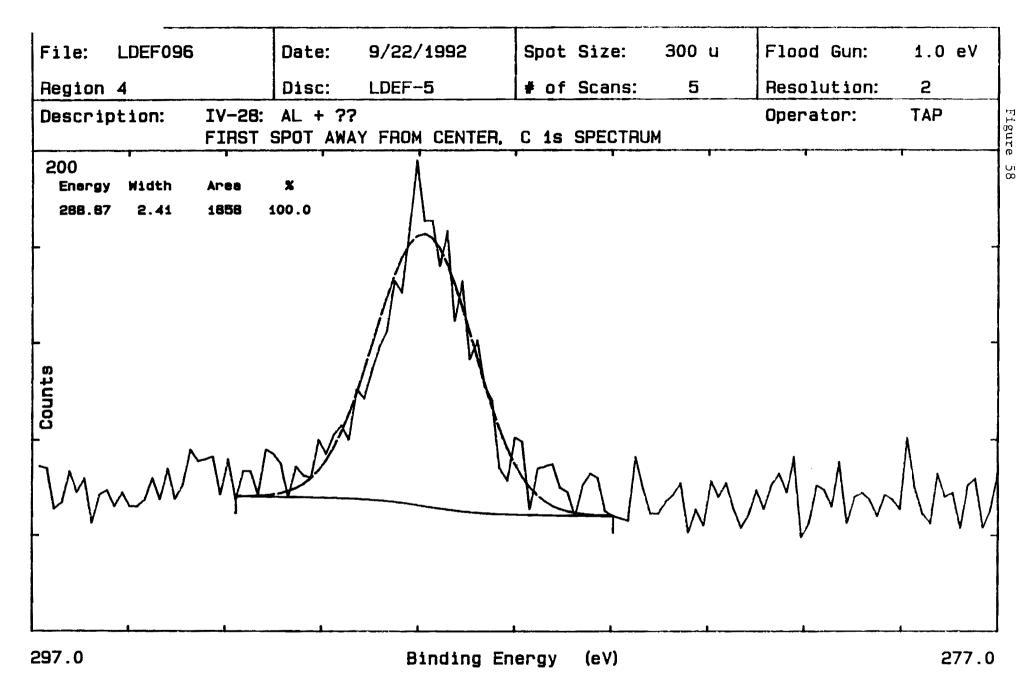




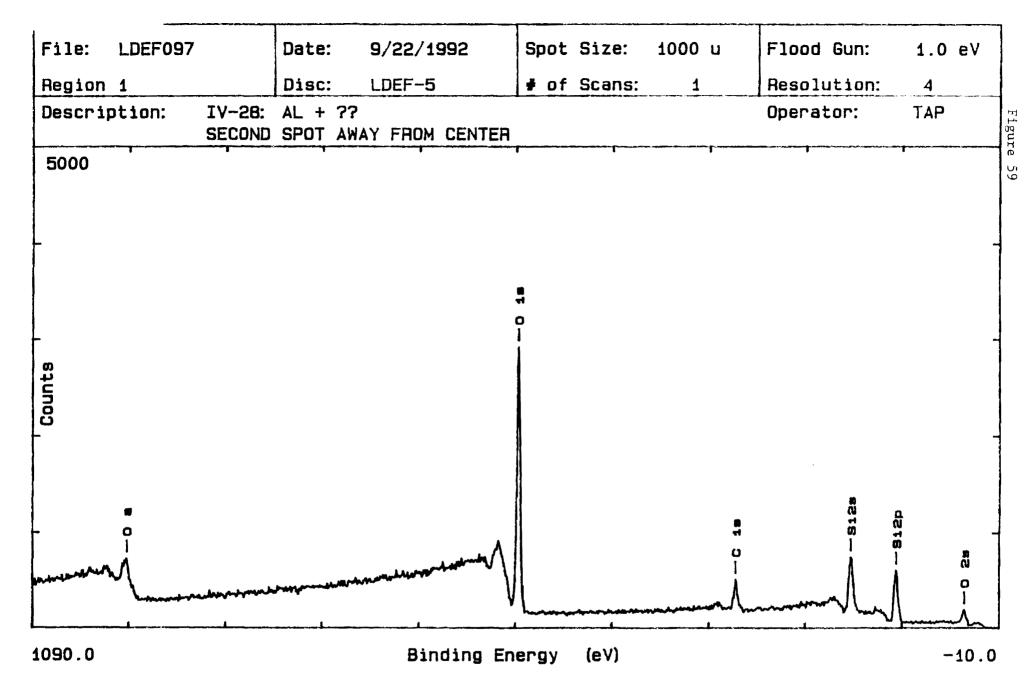
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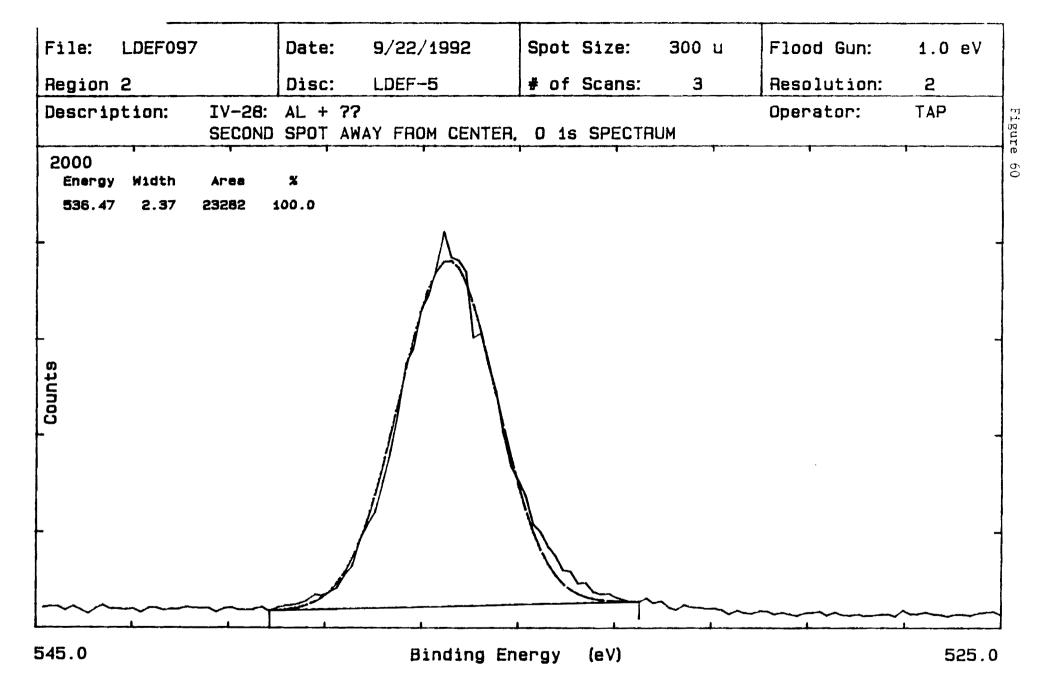


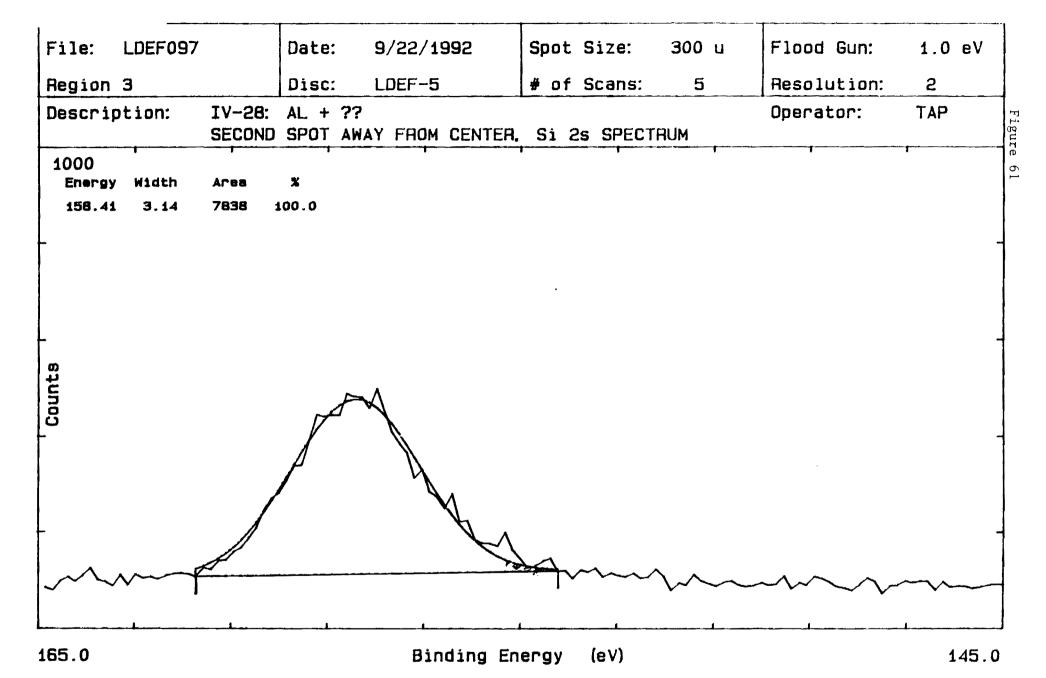


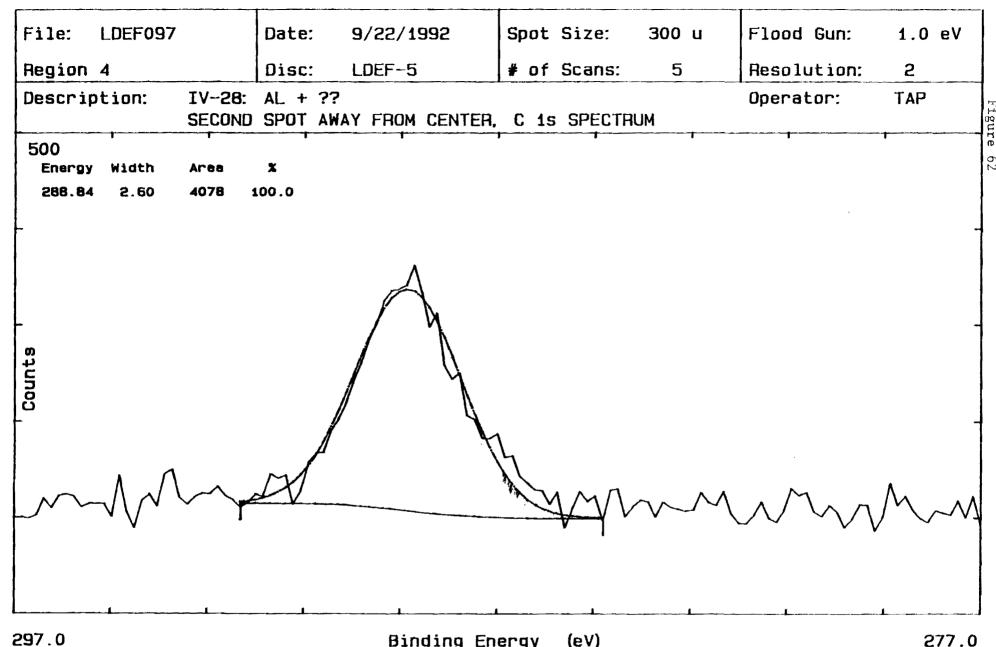


GEORGIA TECH MICROELECTRONICS RESEARCH CENTER







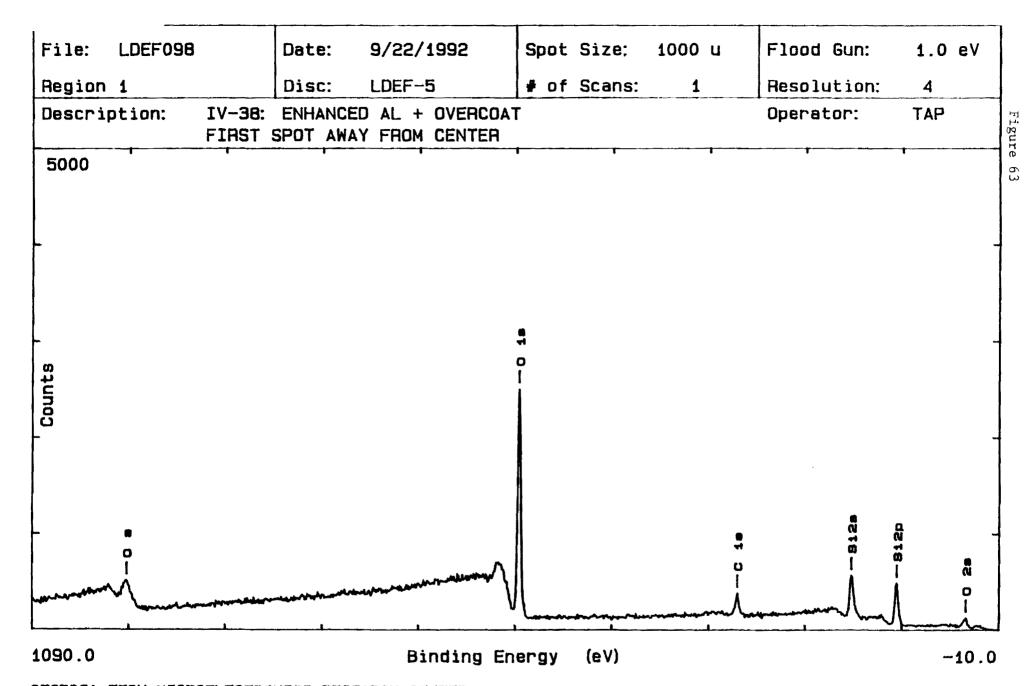


Binding Energy

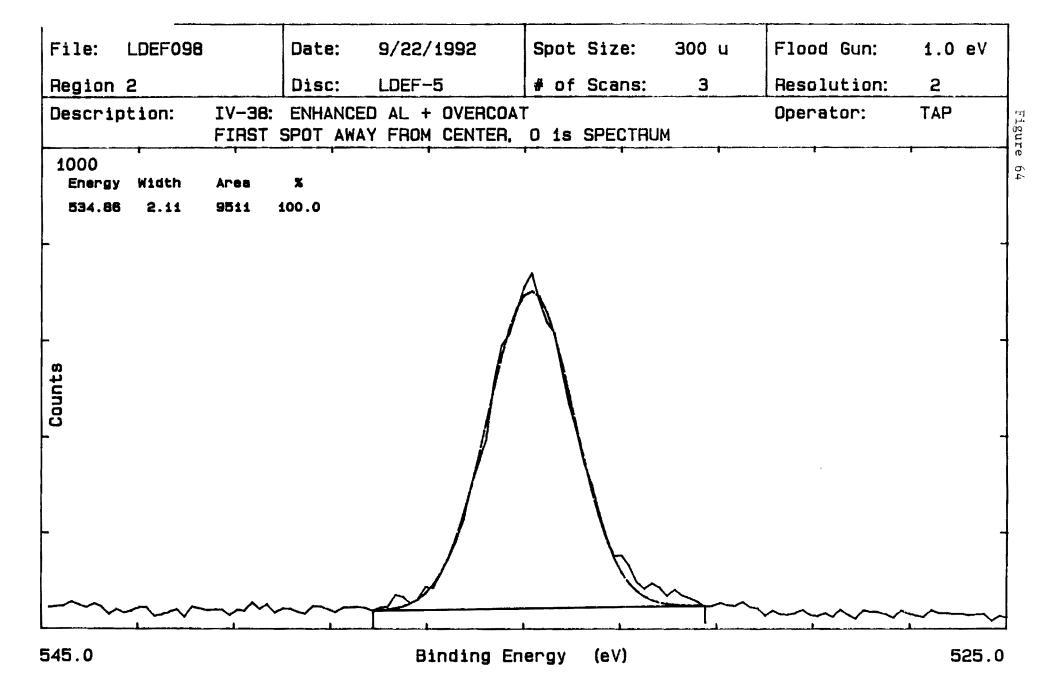
(eV)

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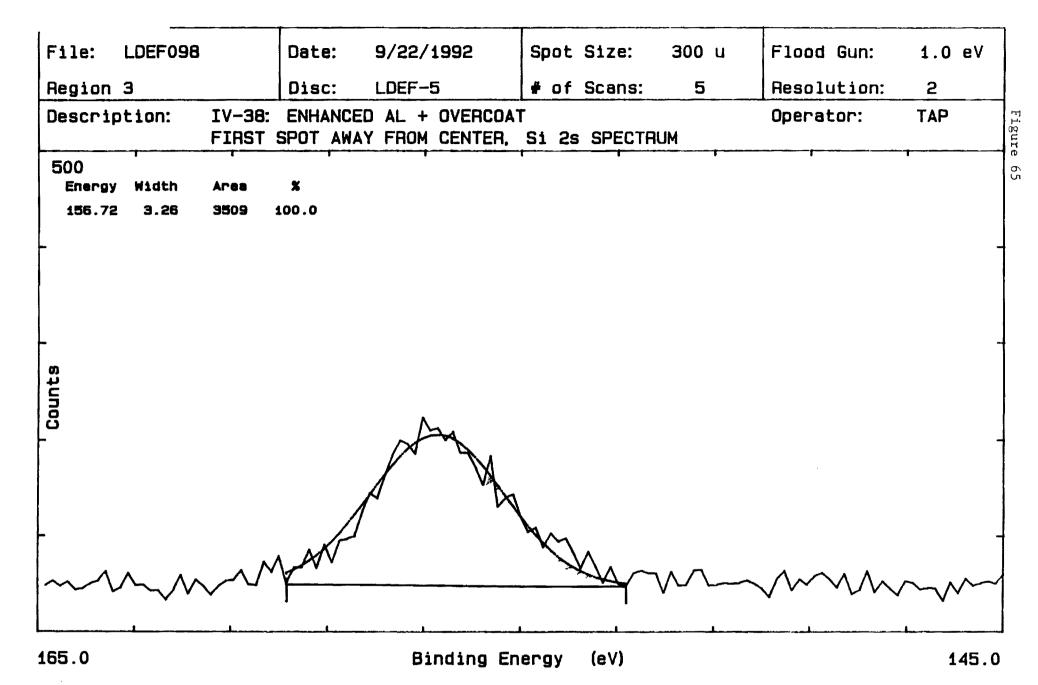
277.0

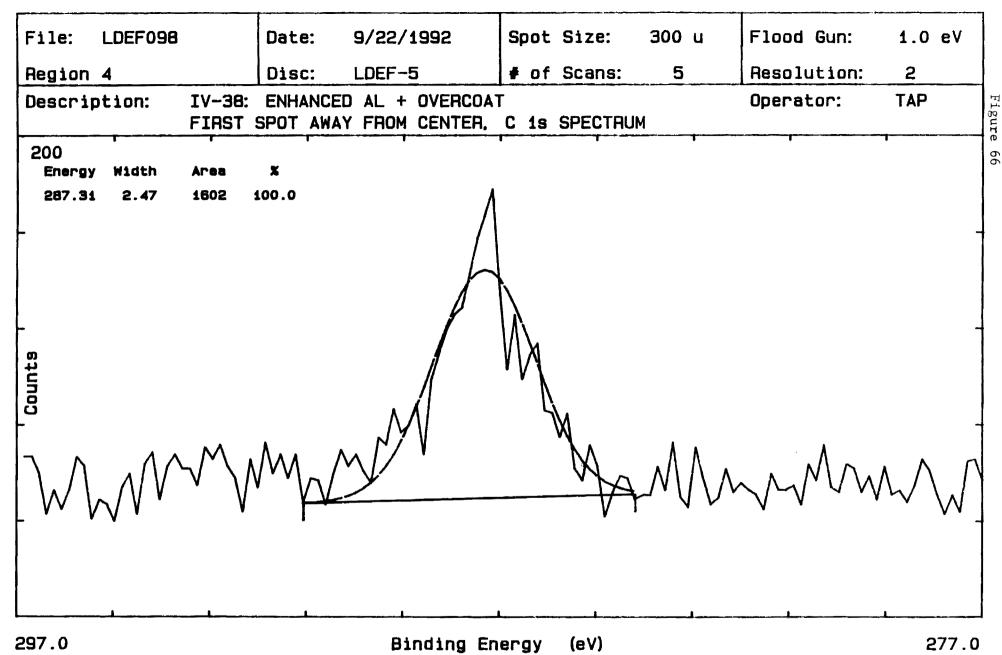


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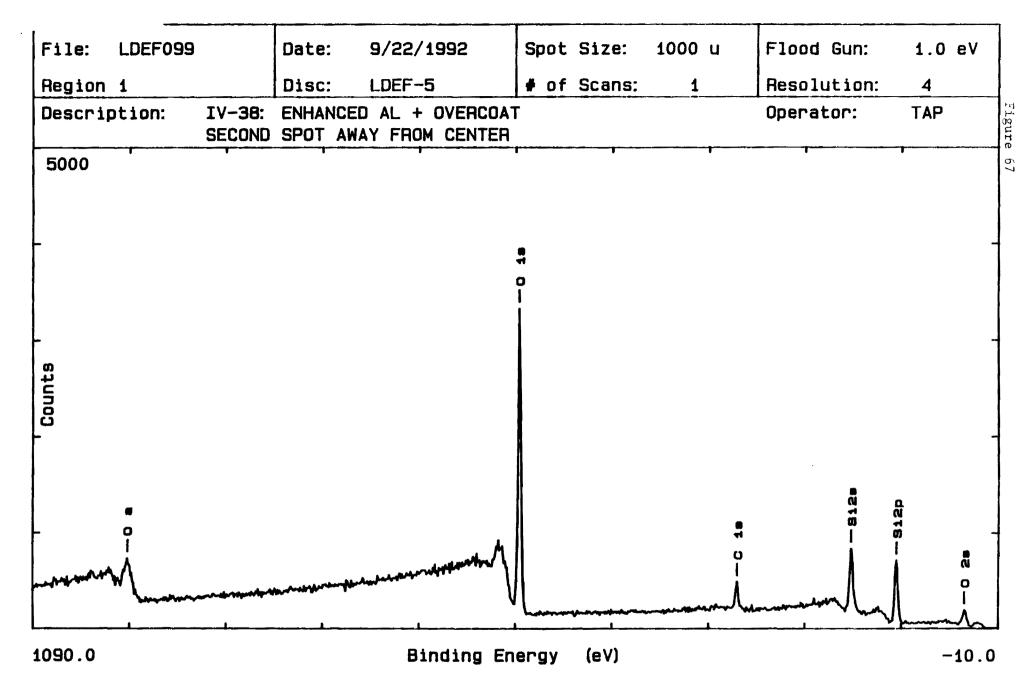


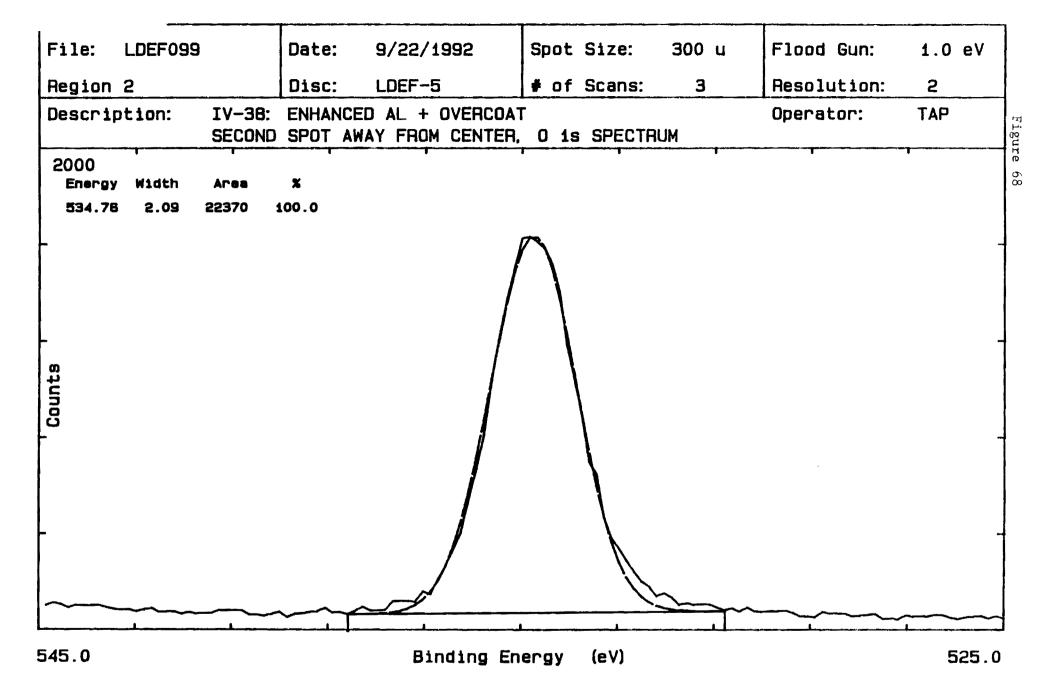


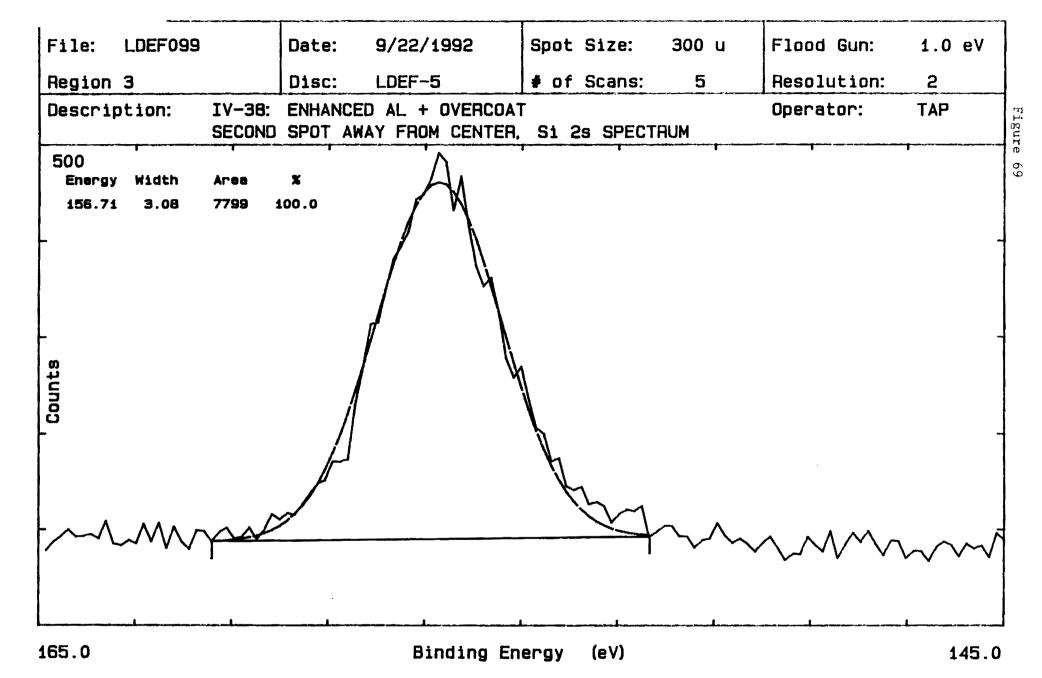
(eV)

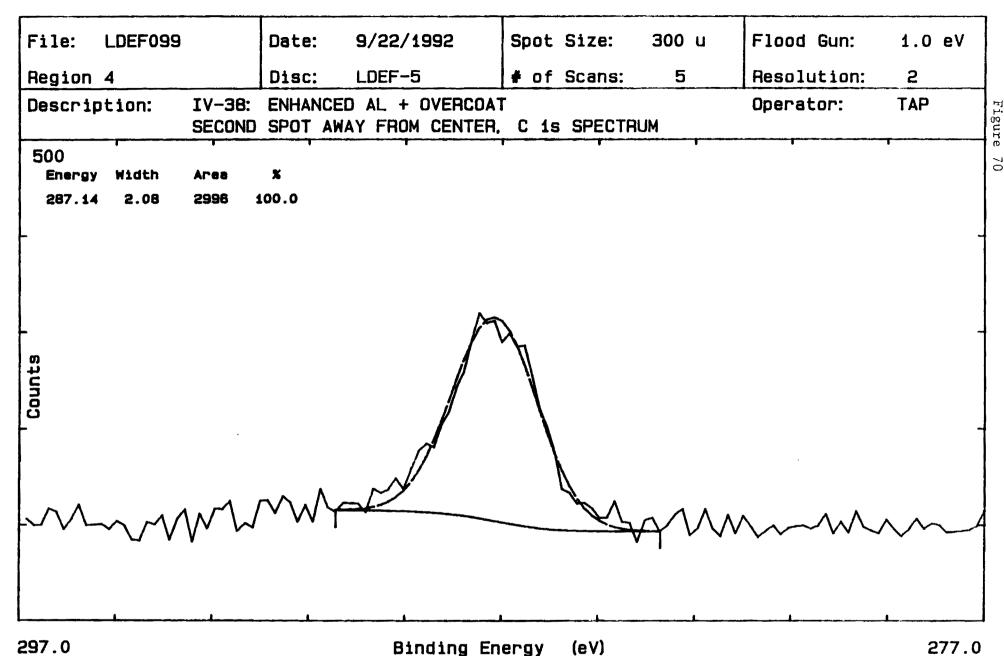
GEORGIA TECH MICROELECTRONICS RESEARCH CENTER

277.0

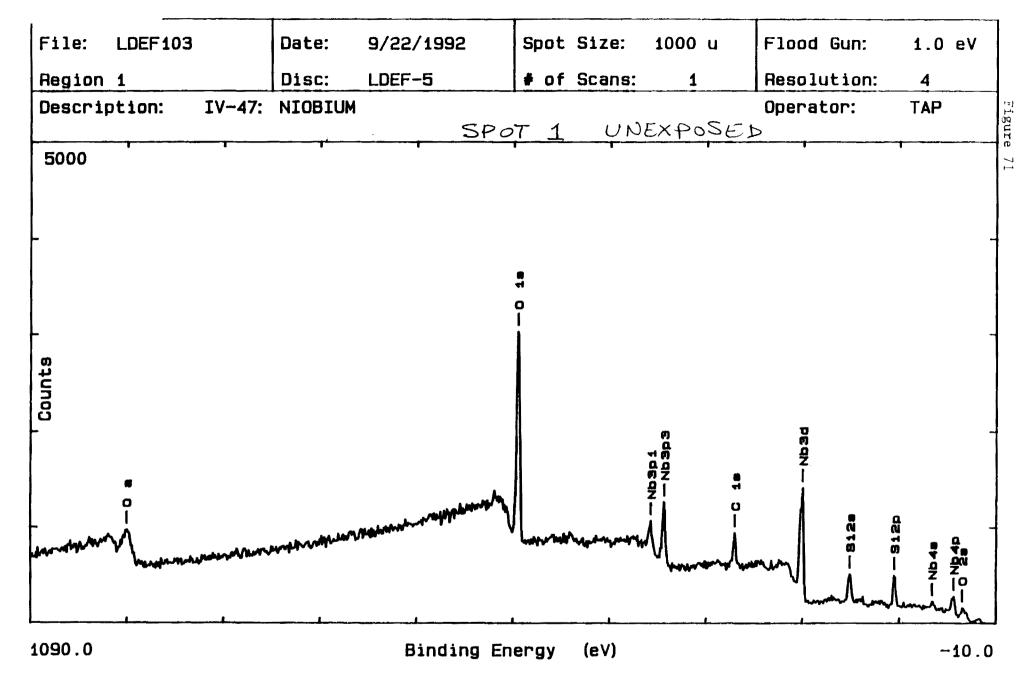




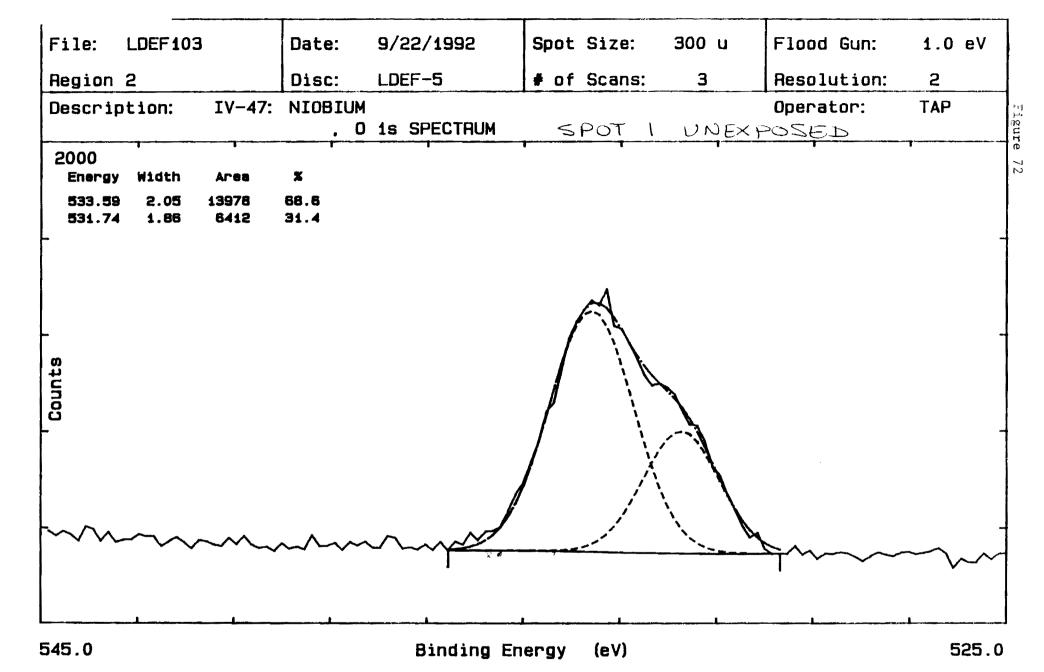


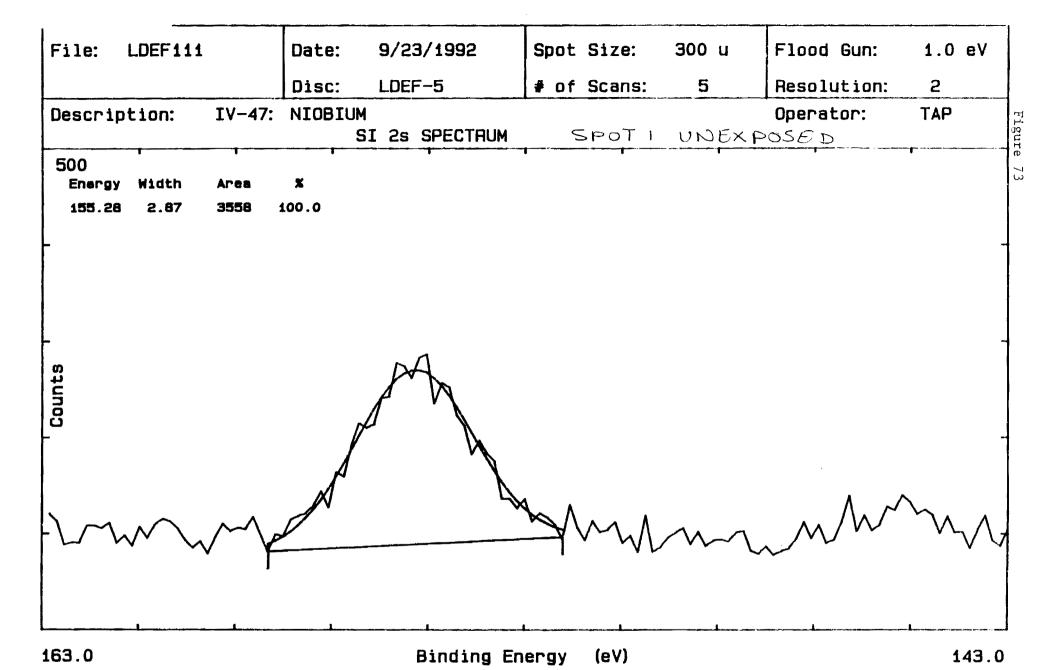


277.0

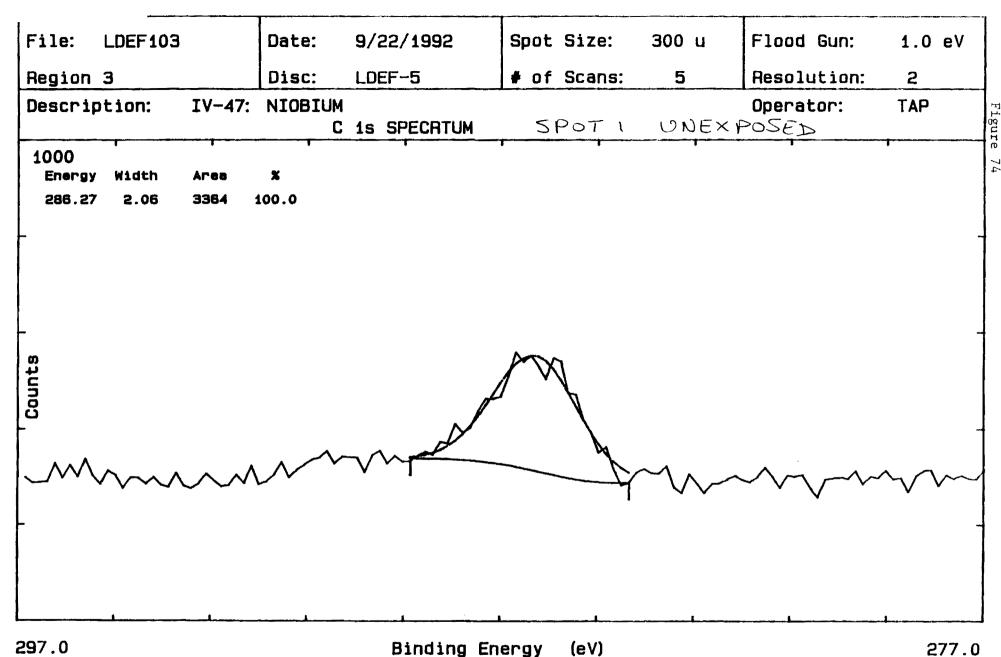


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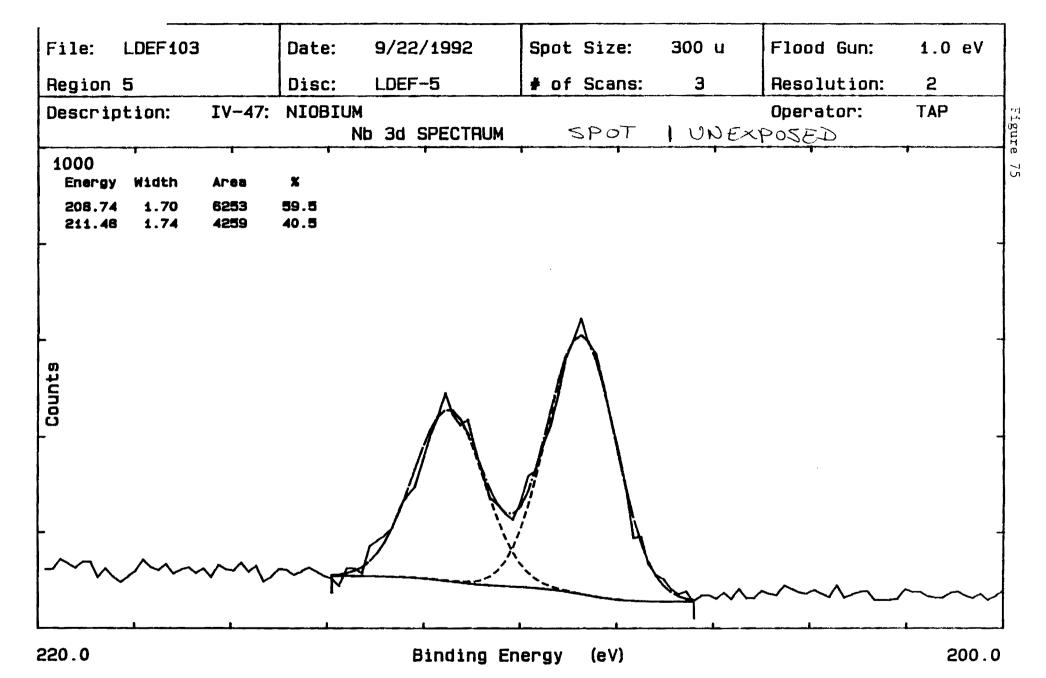


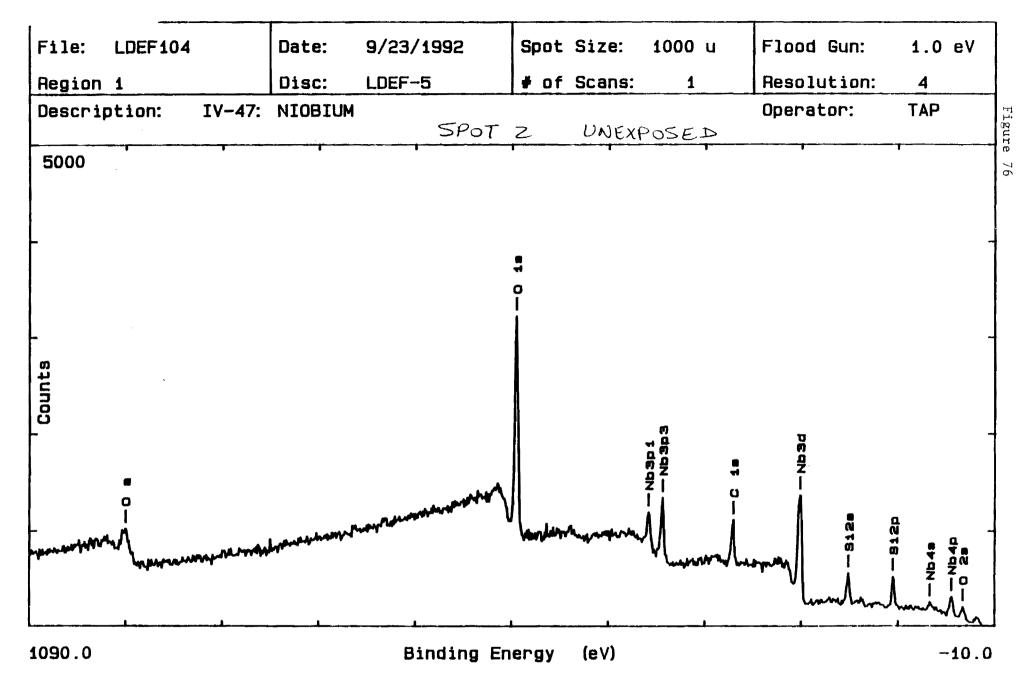


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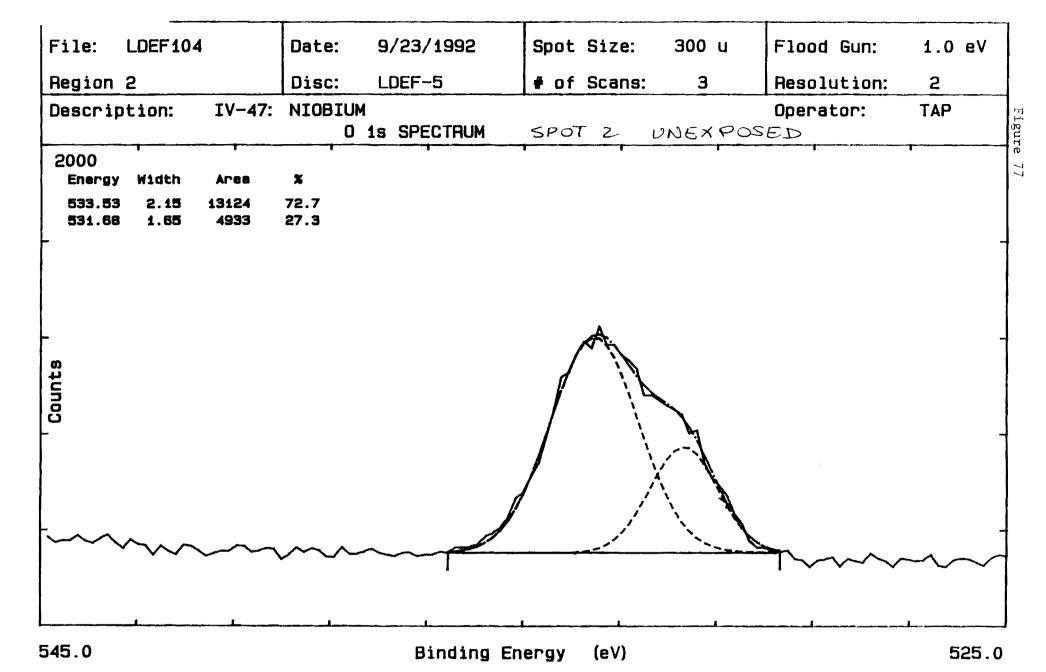


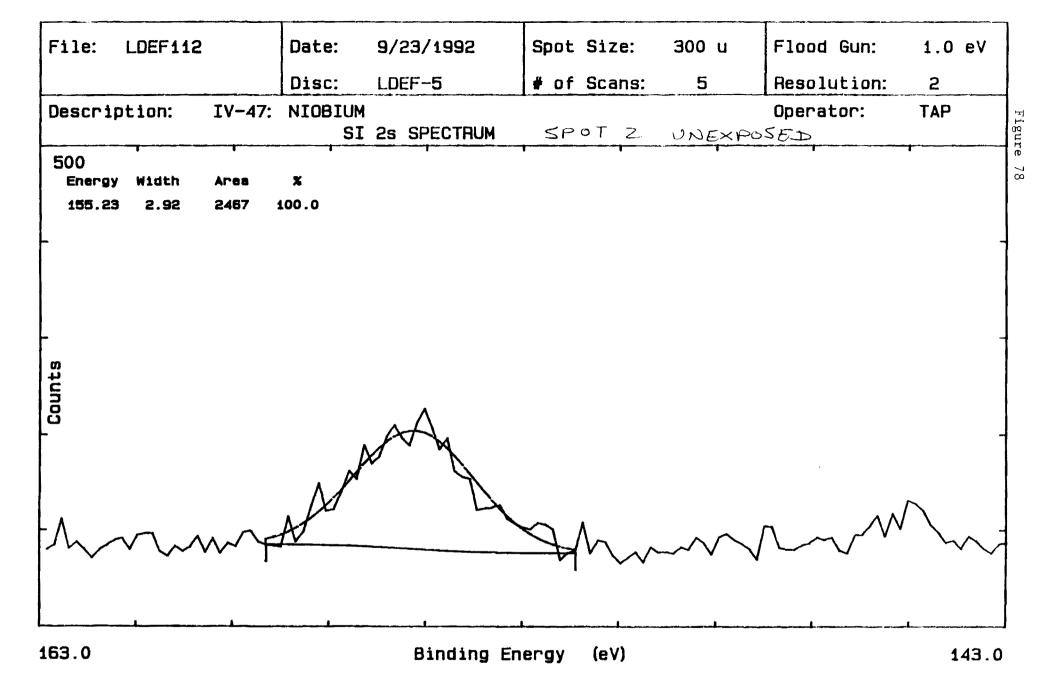
277.0

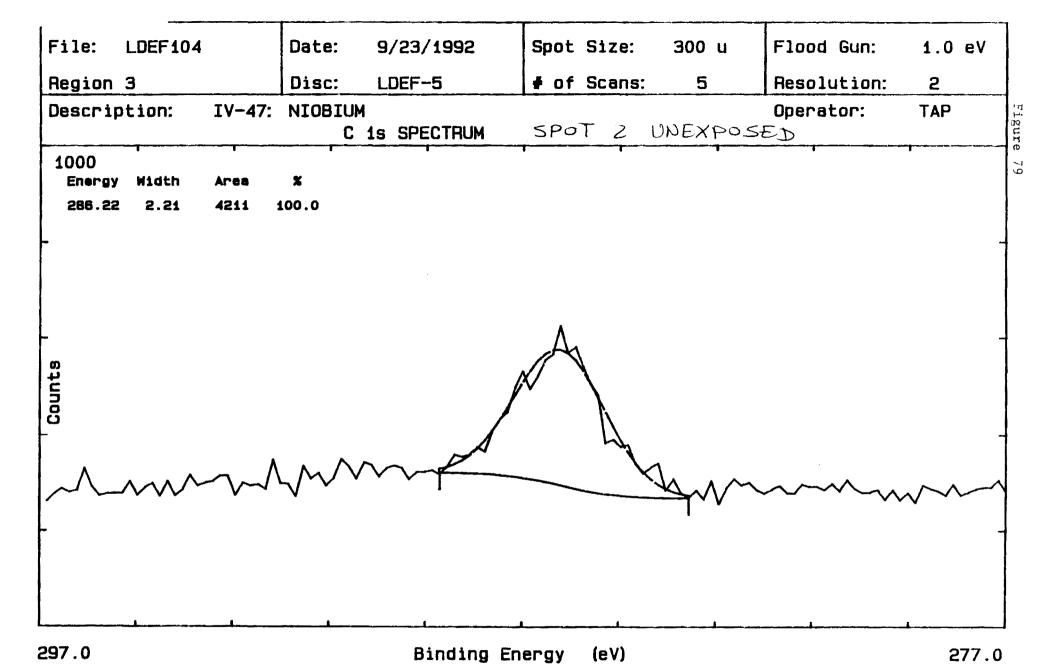


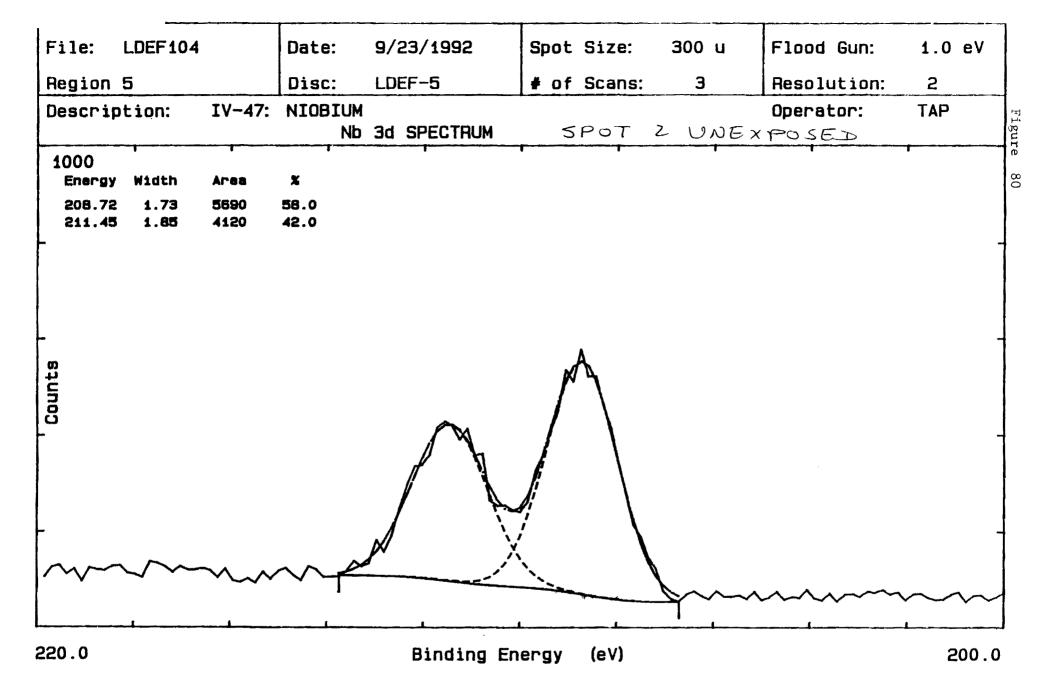


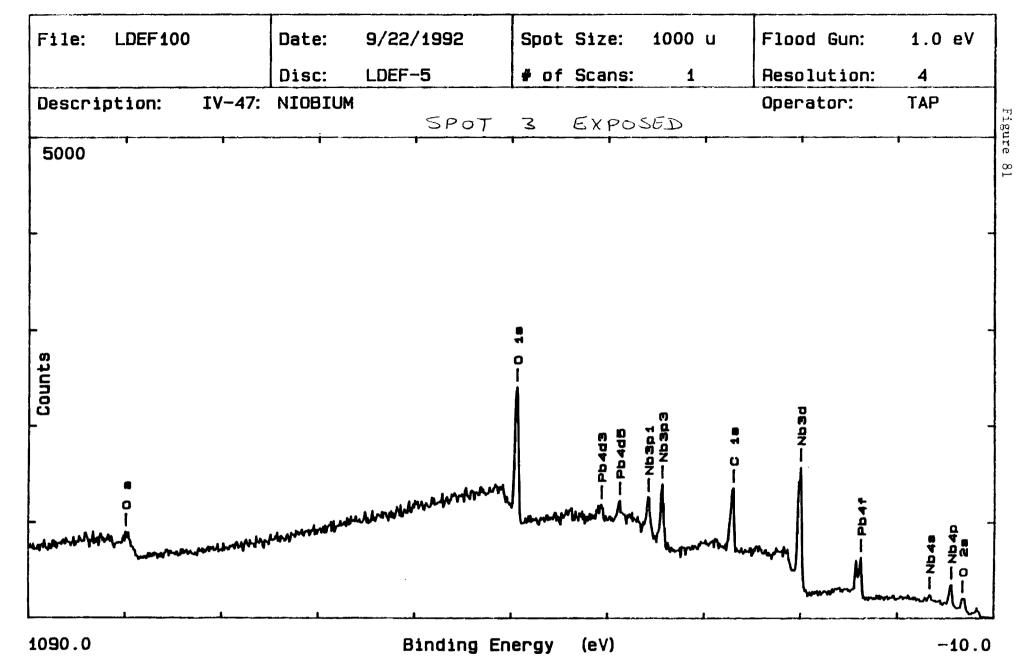
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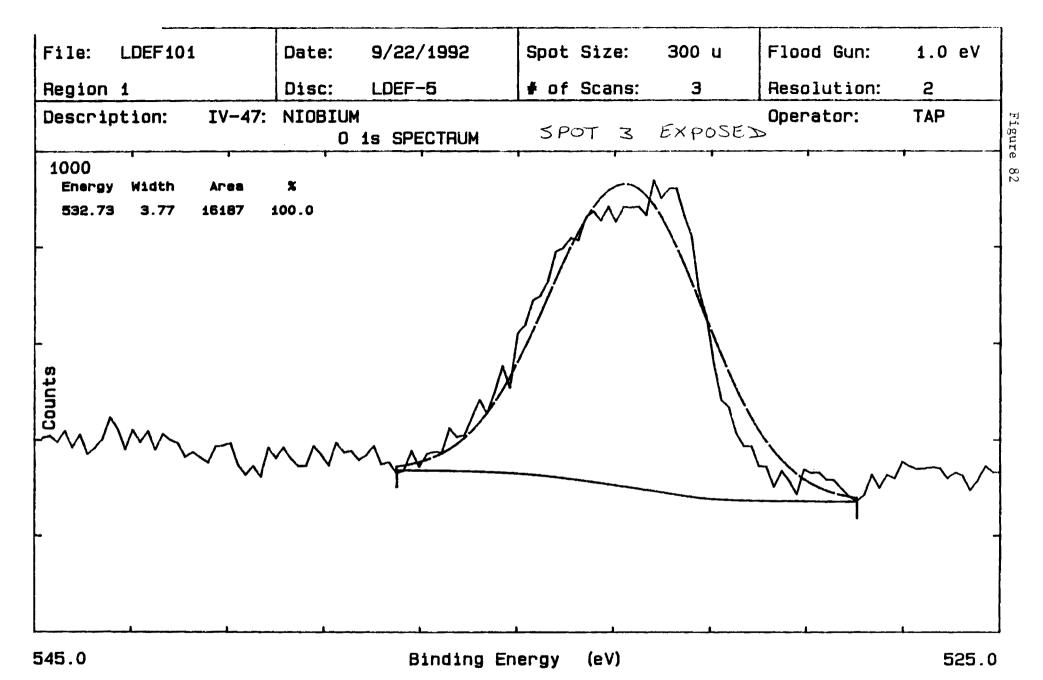




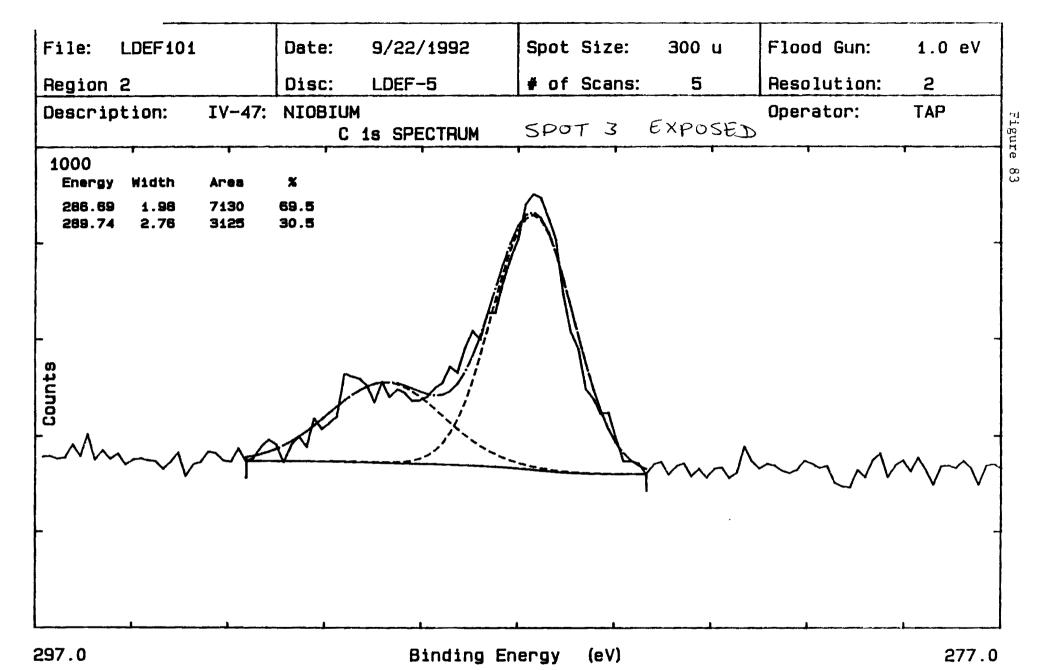




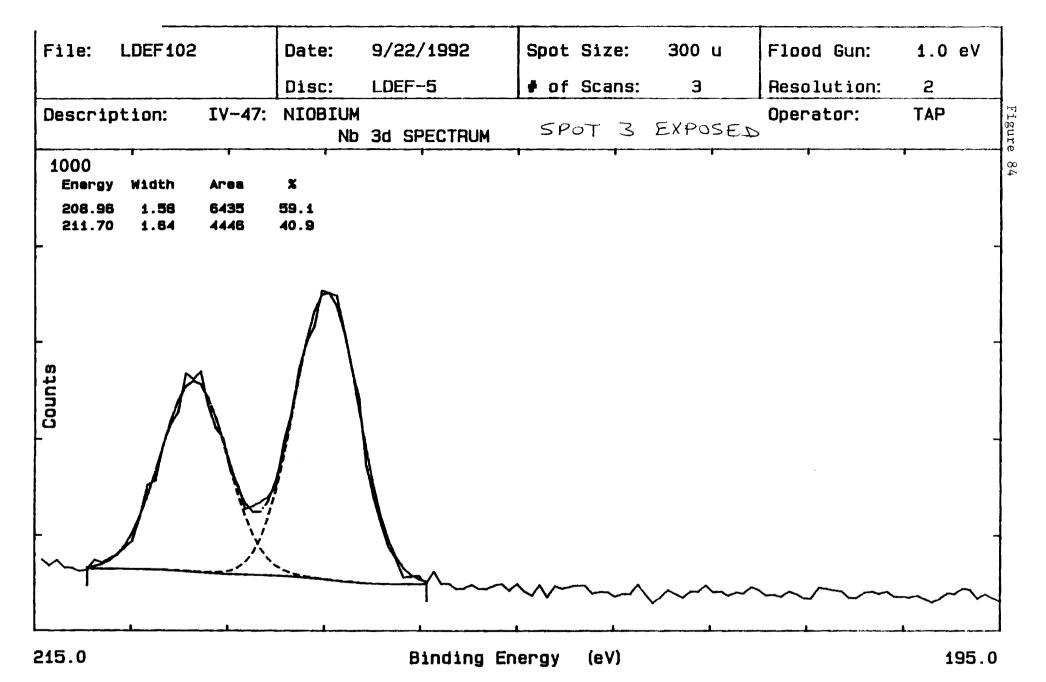


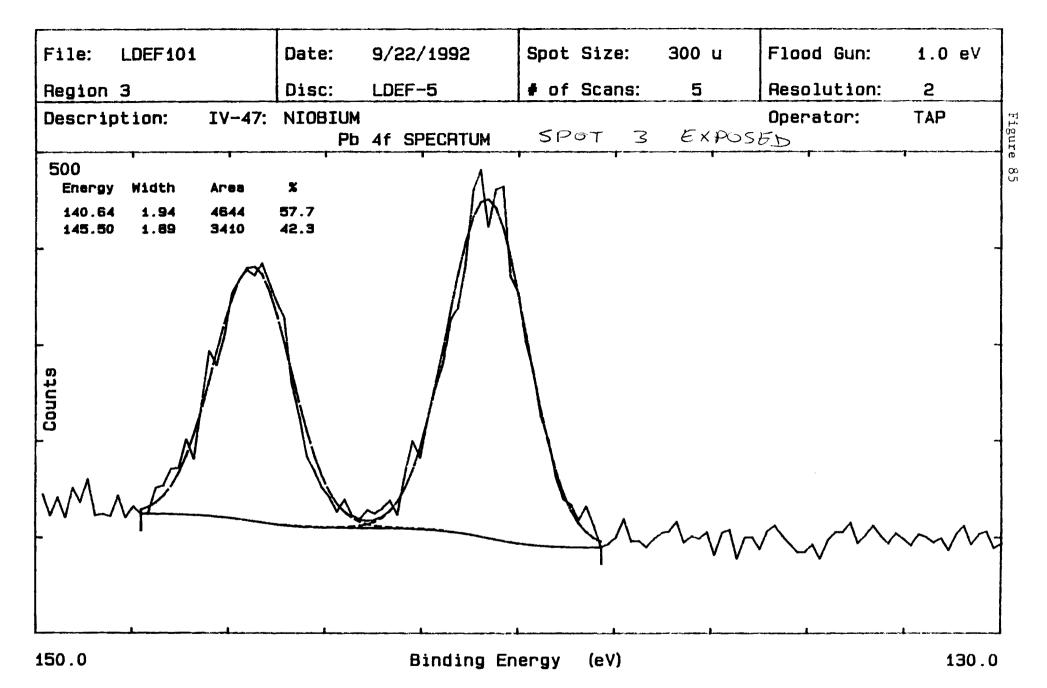


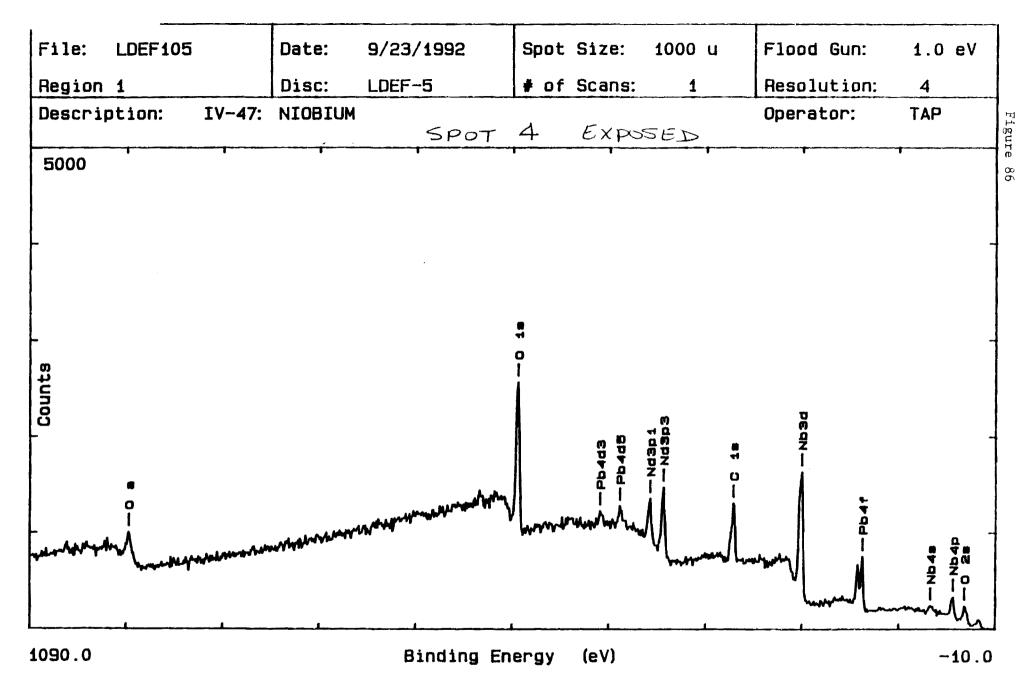
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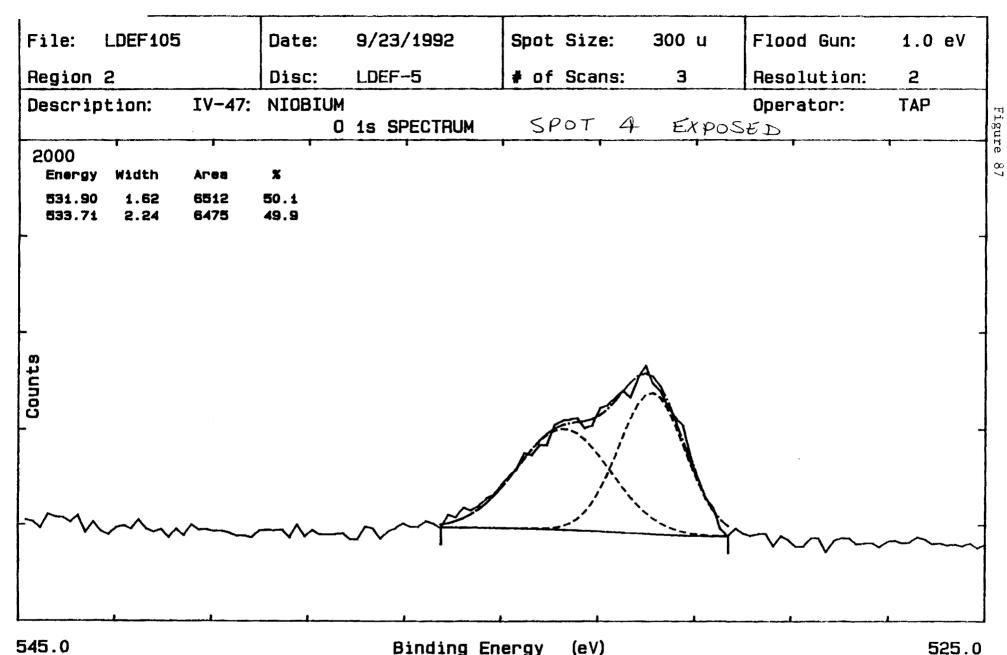
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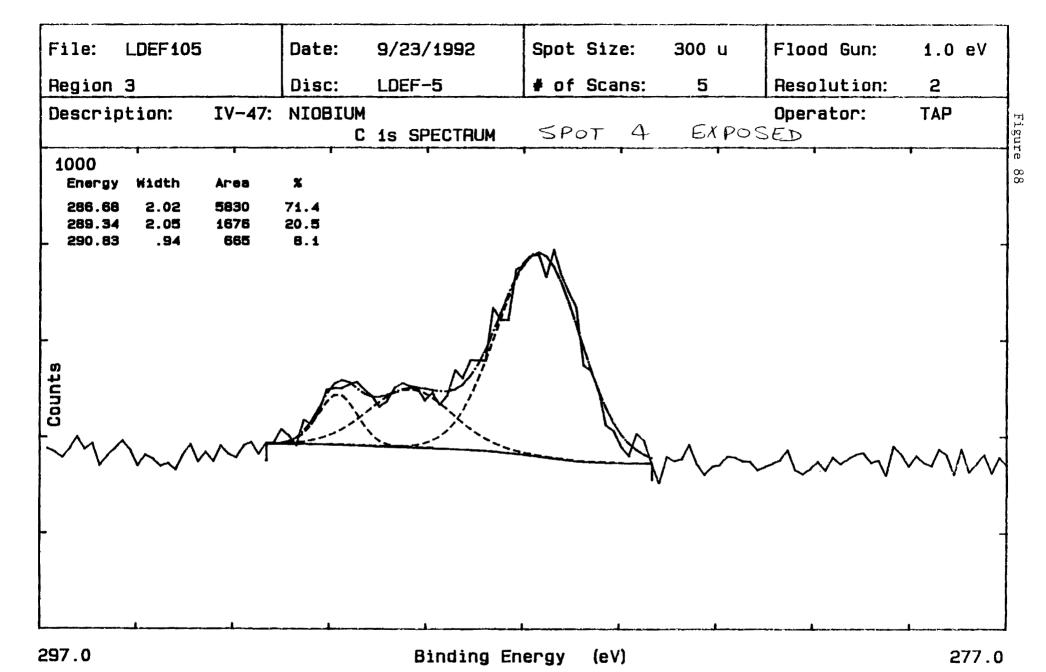




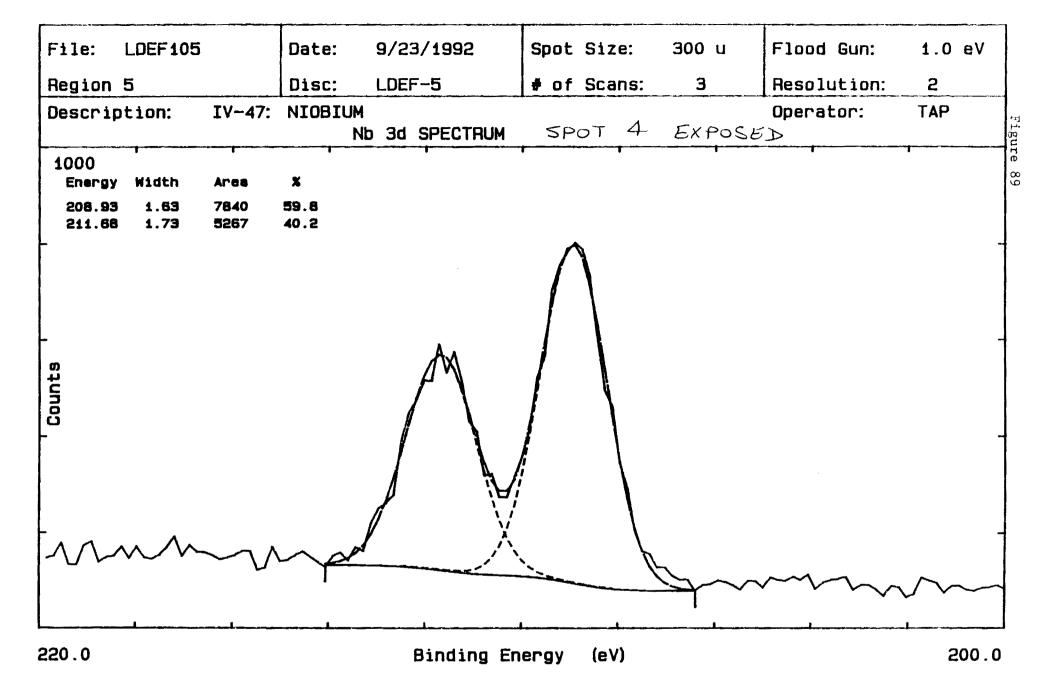
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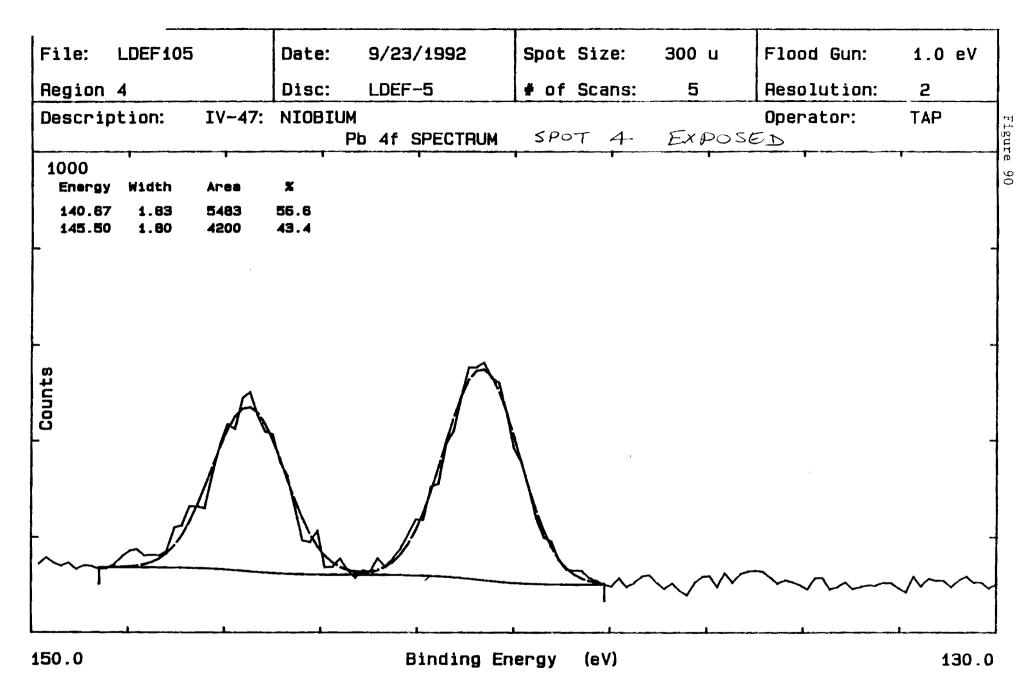


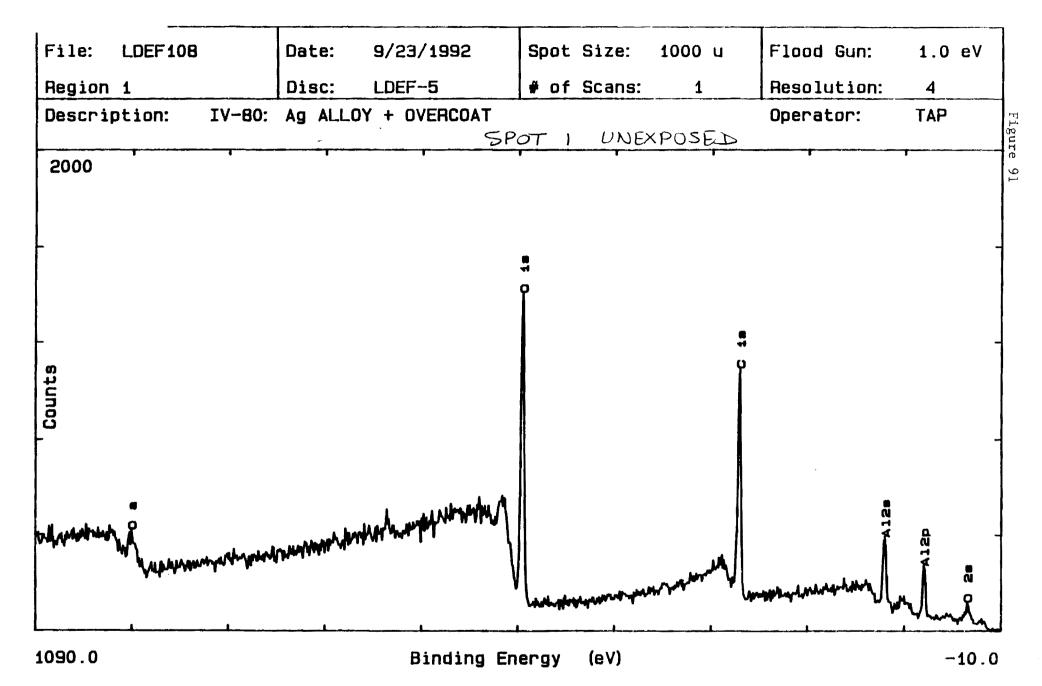
Binding Energy

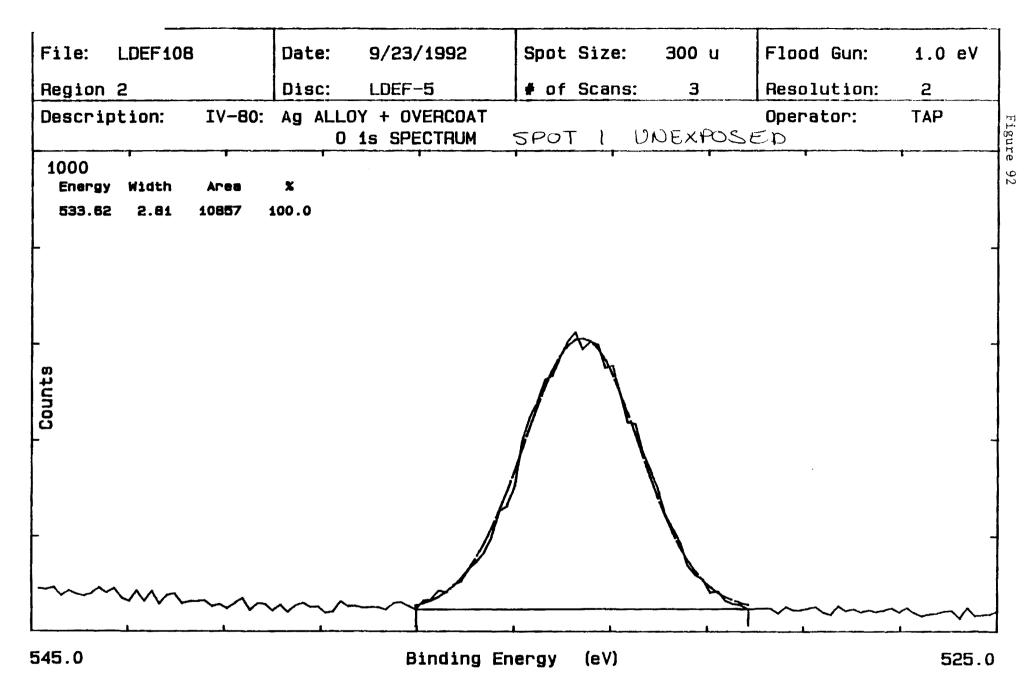


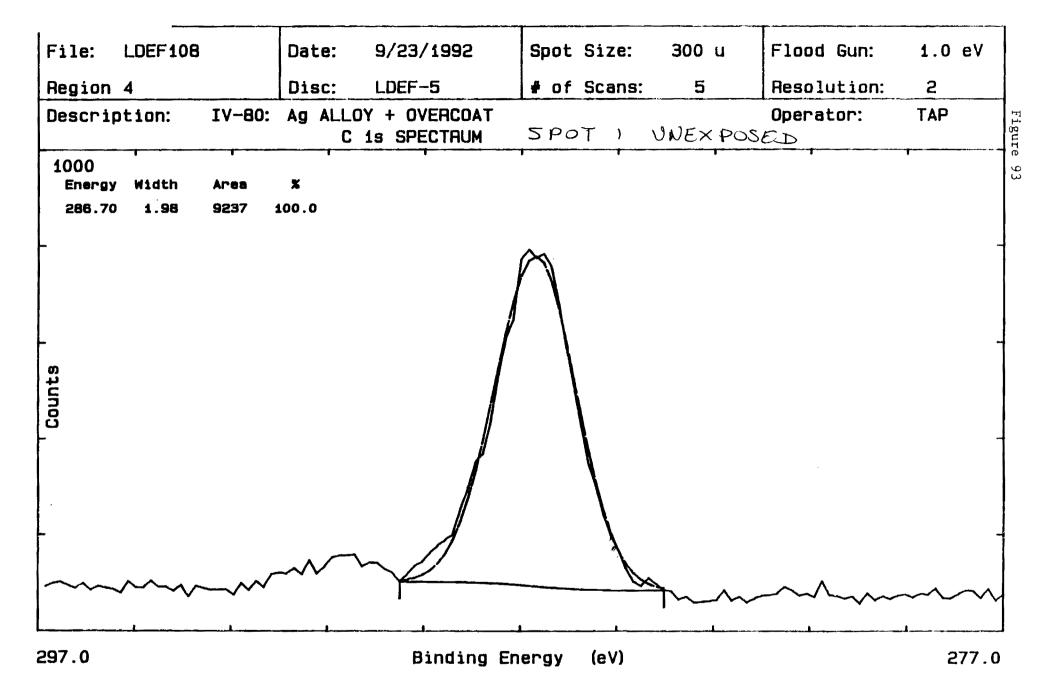
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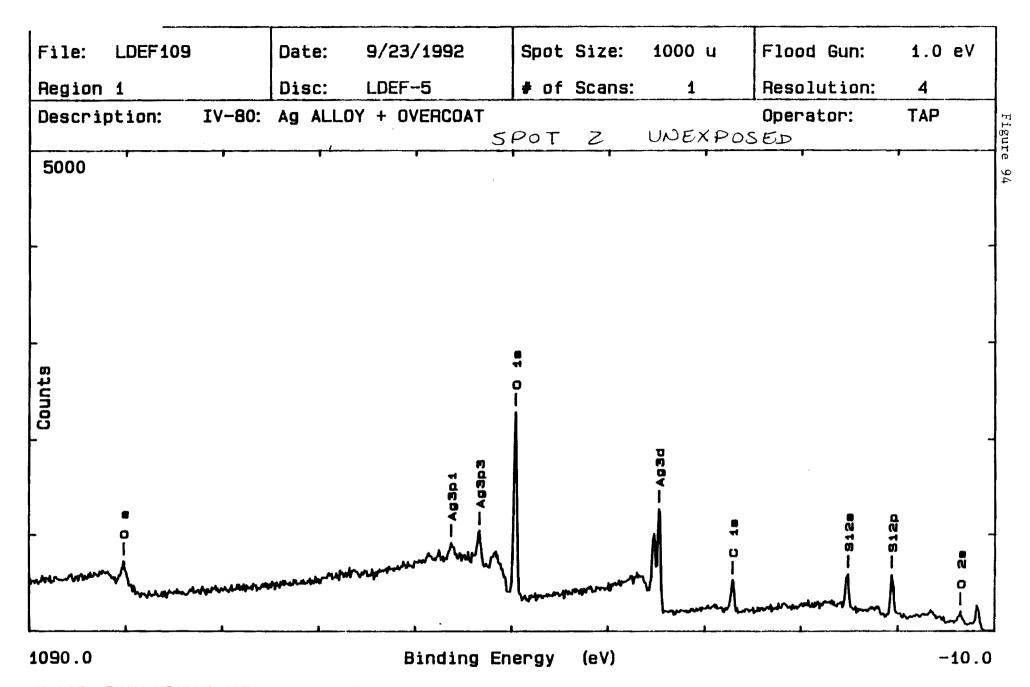




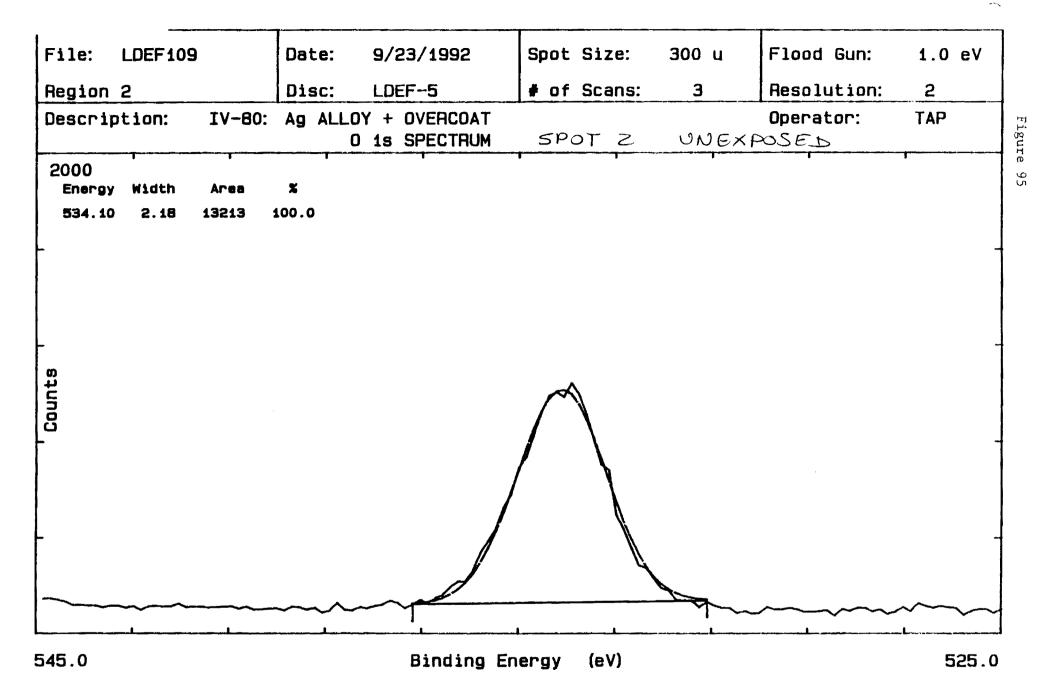




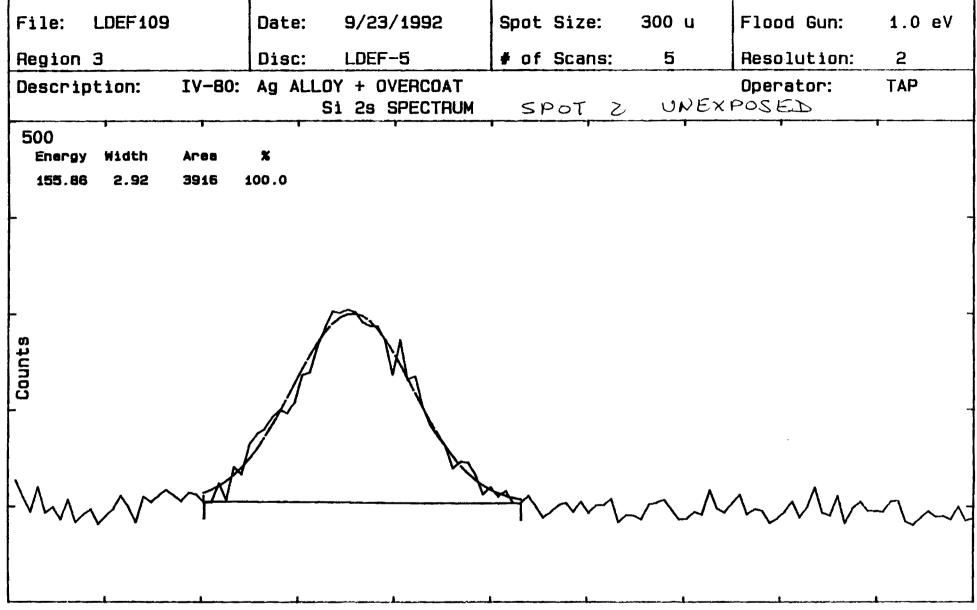




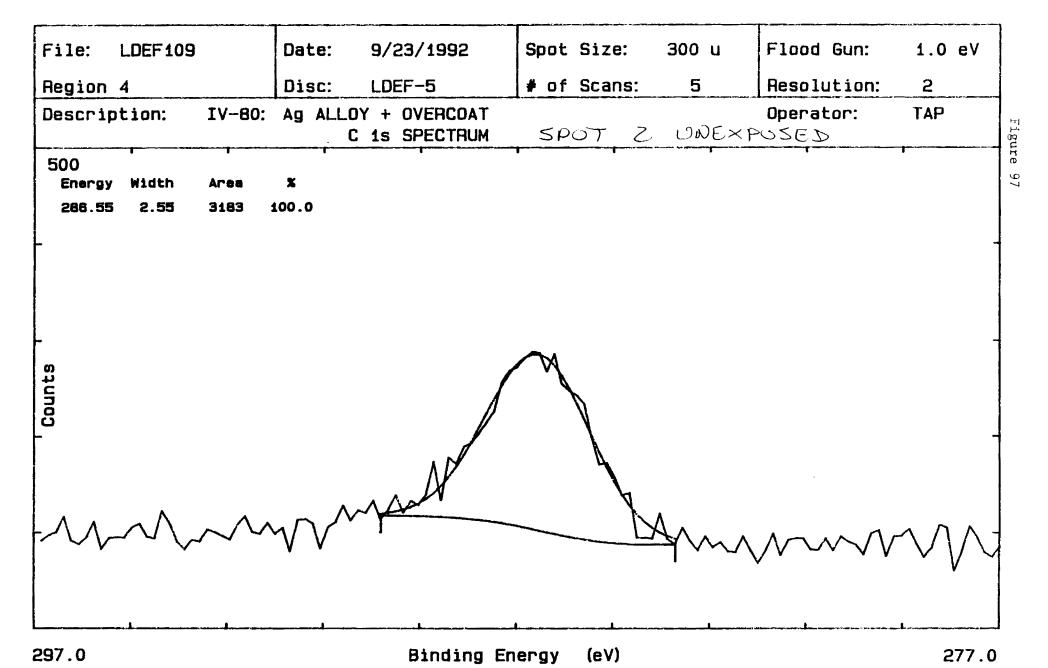
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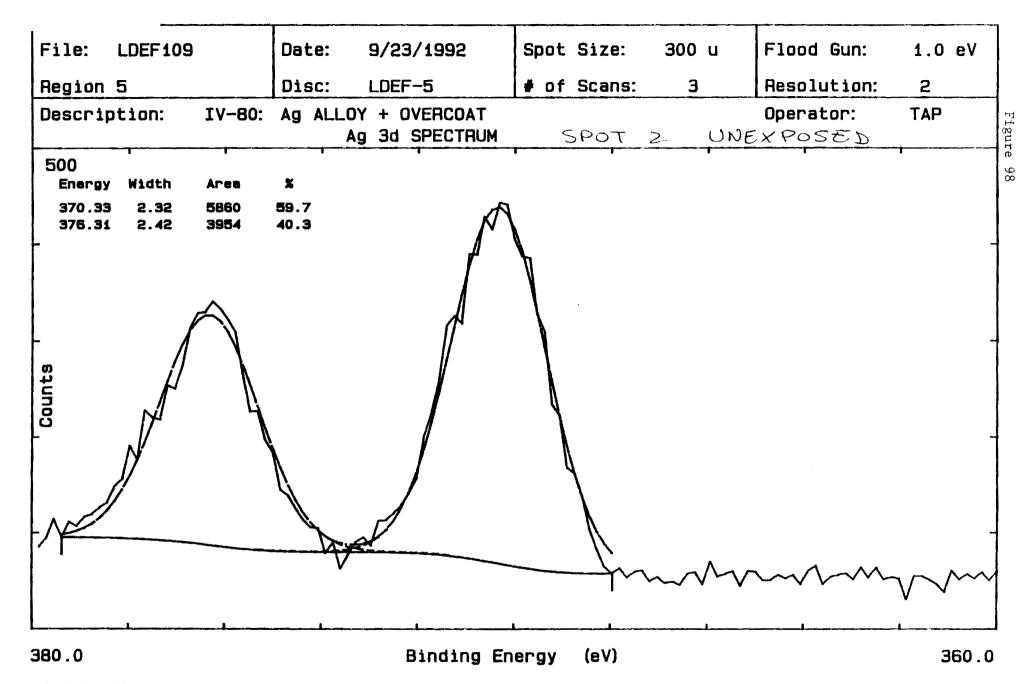




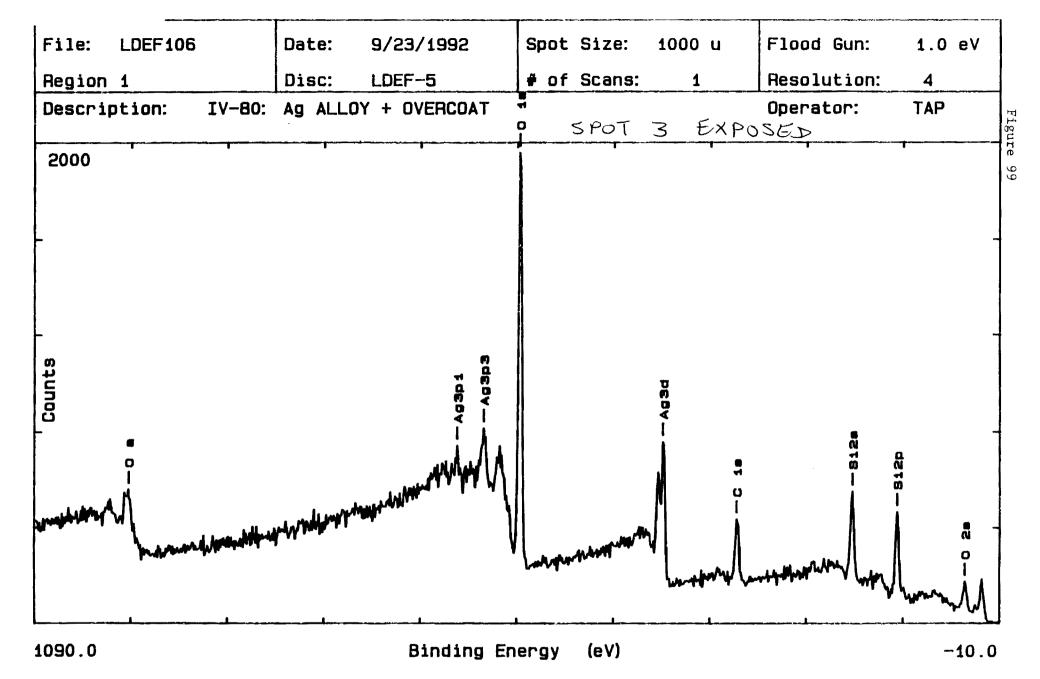


163.0 Binding Energy (eV) 143.0

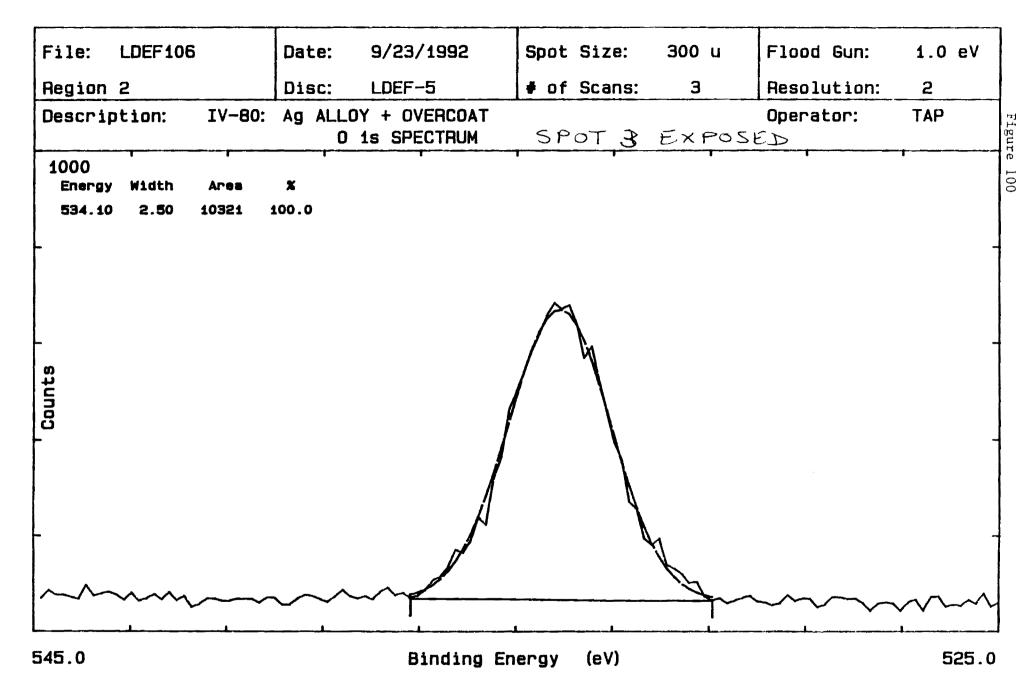


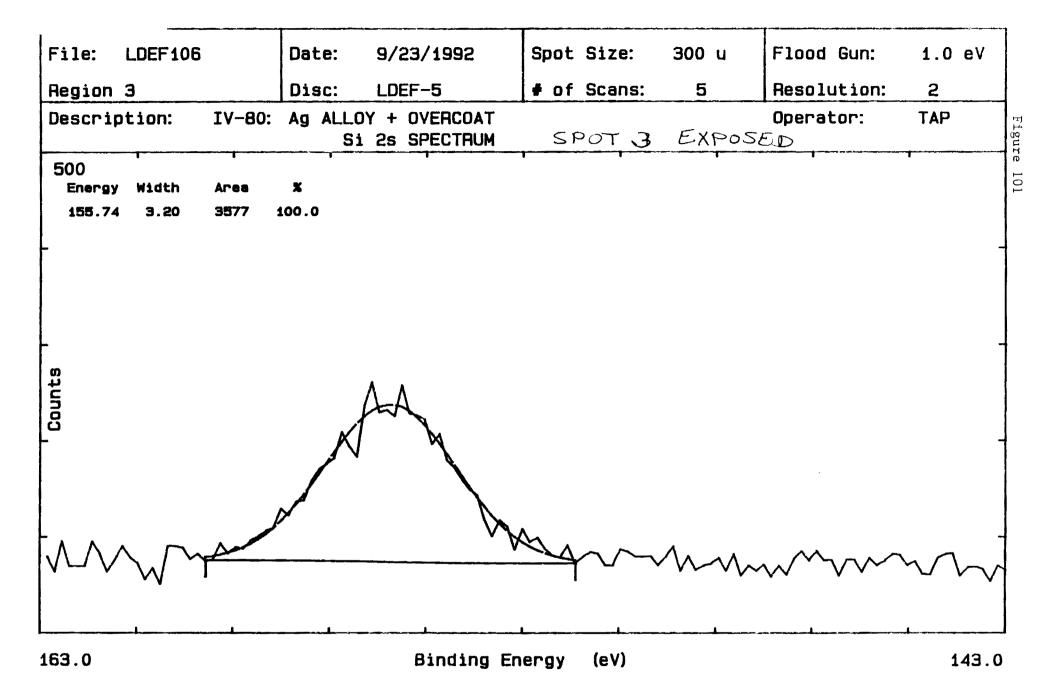


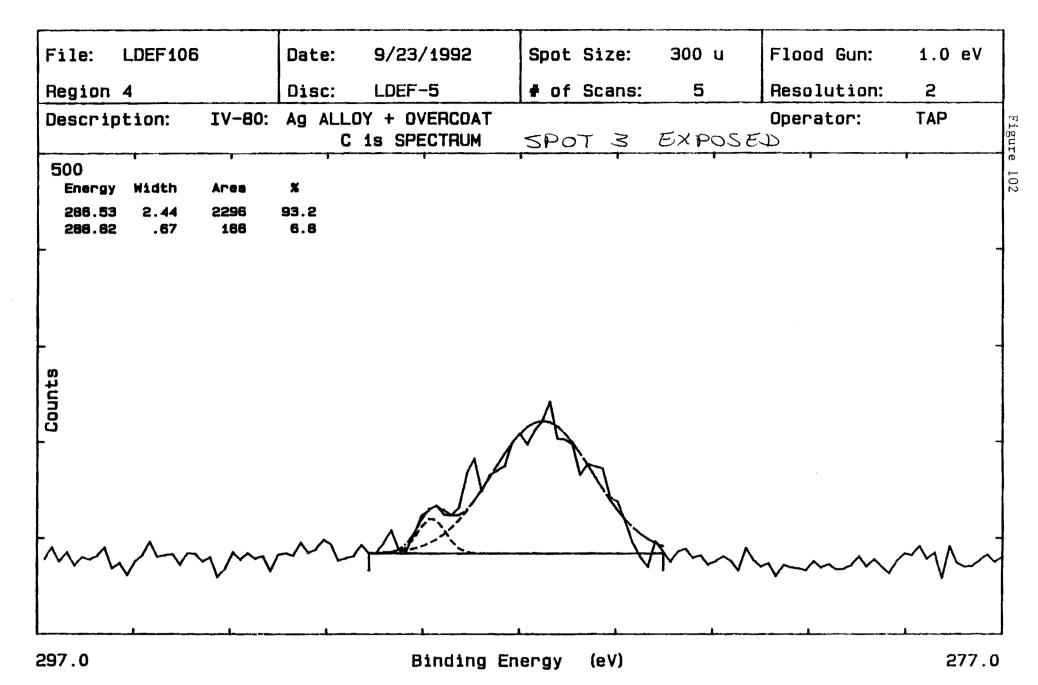
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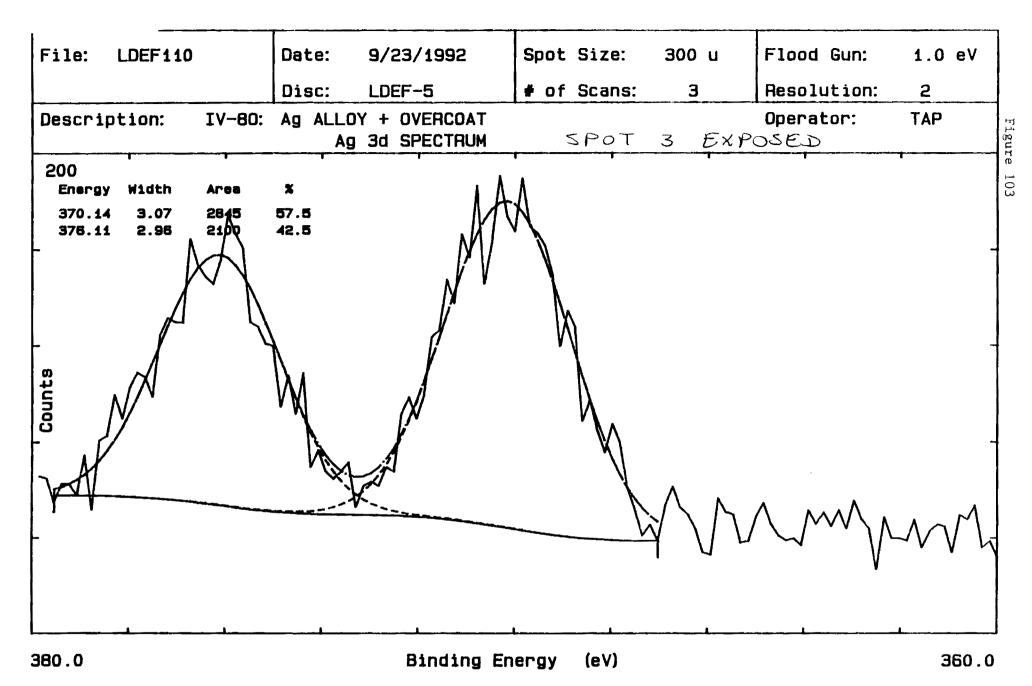


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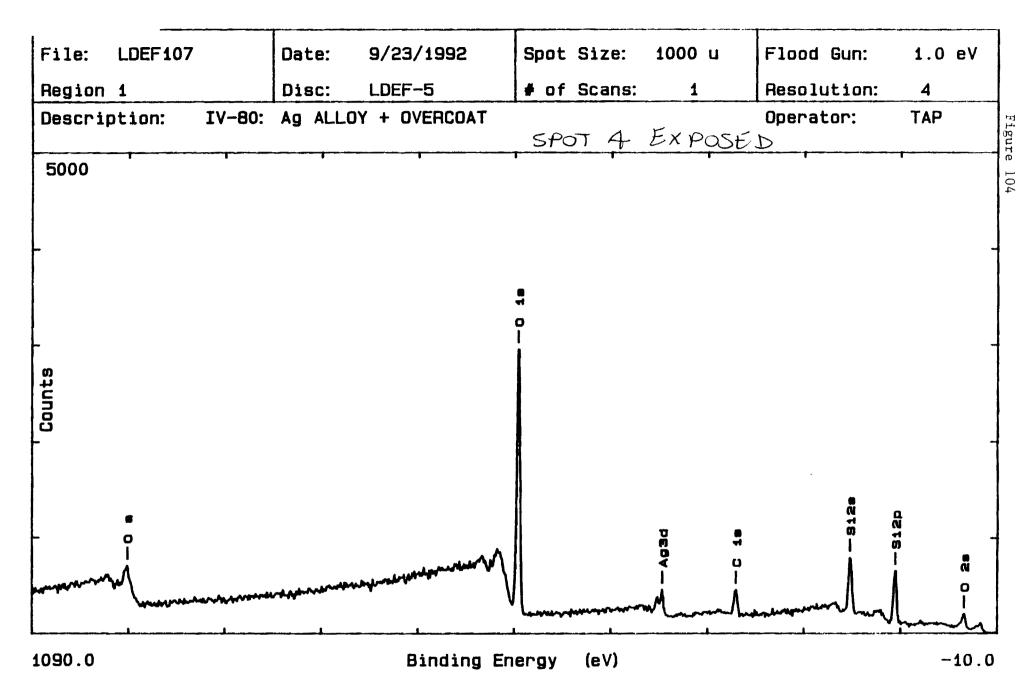




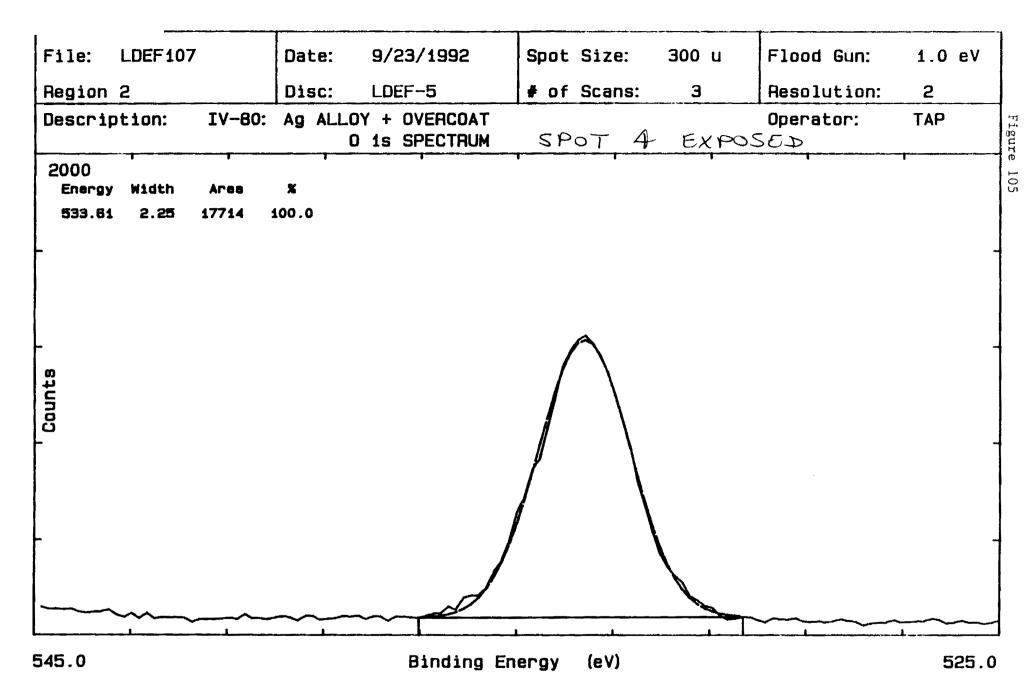




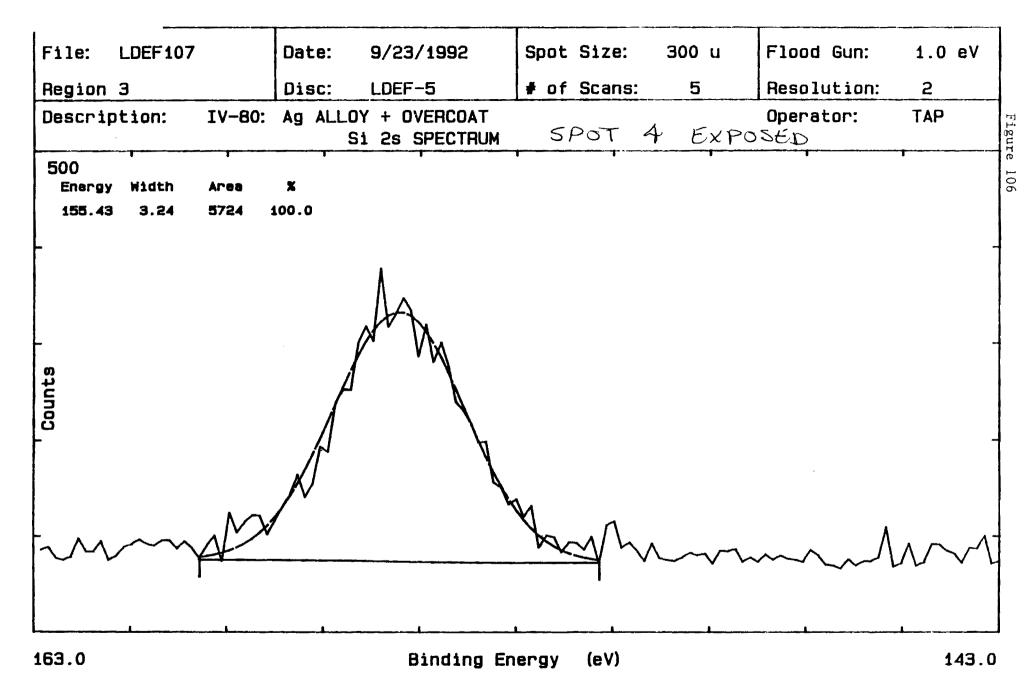
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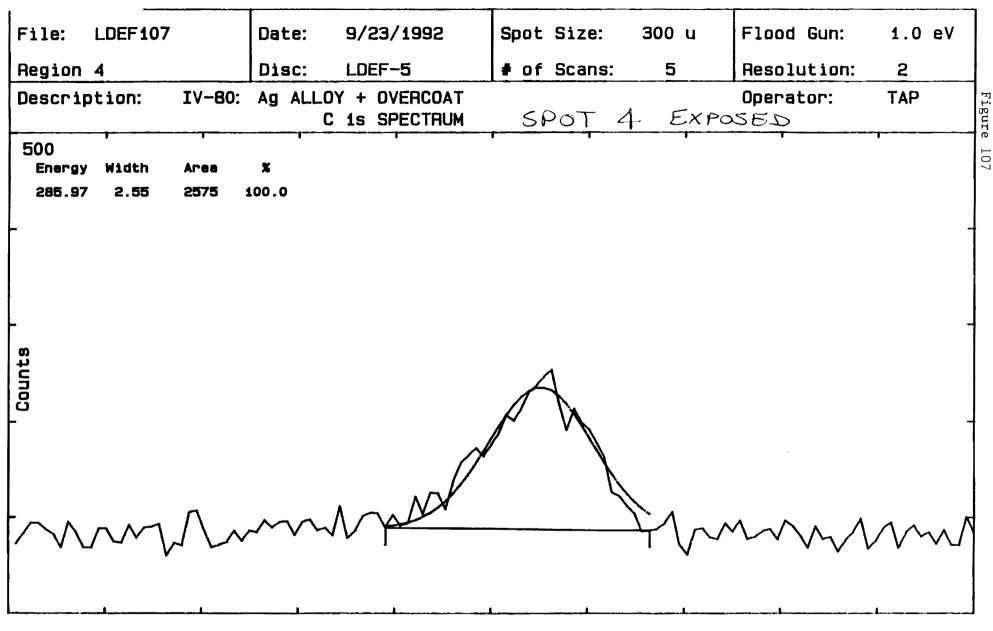


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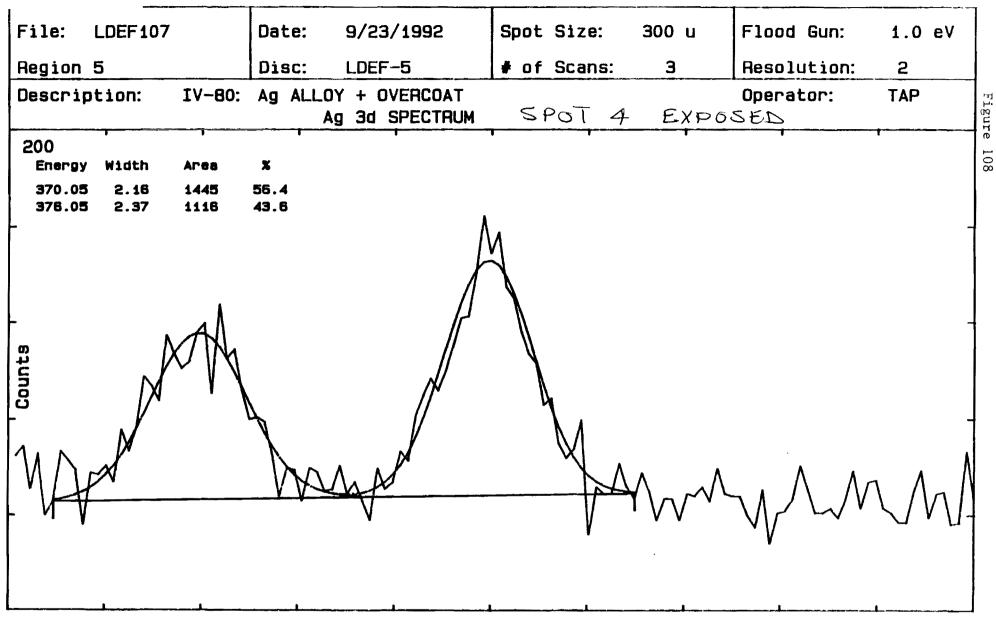


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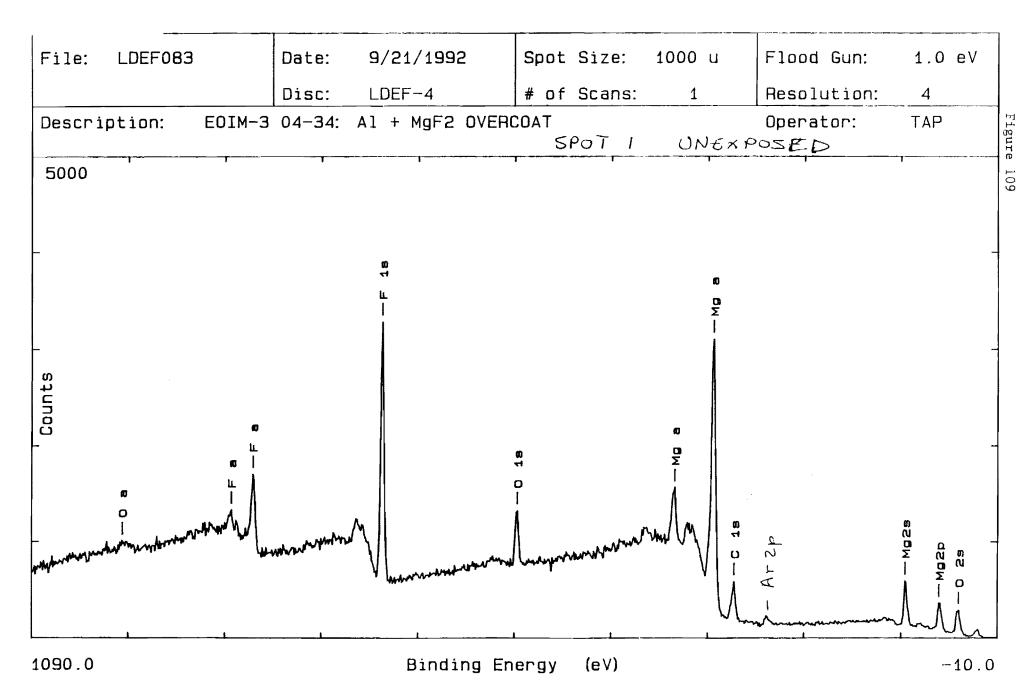




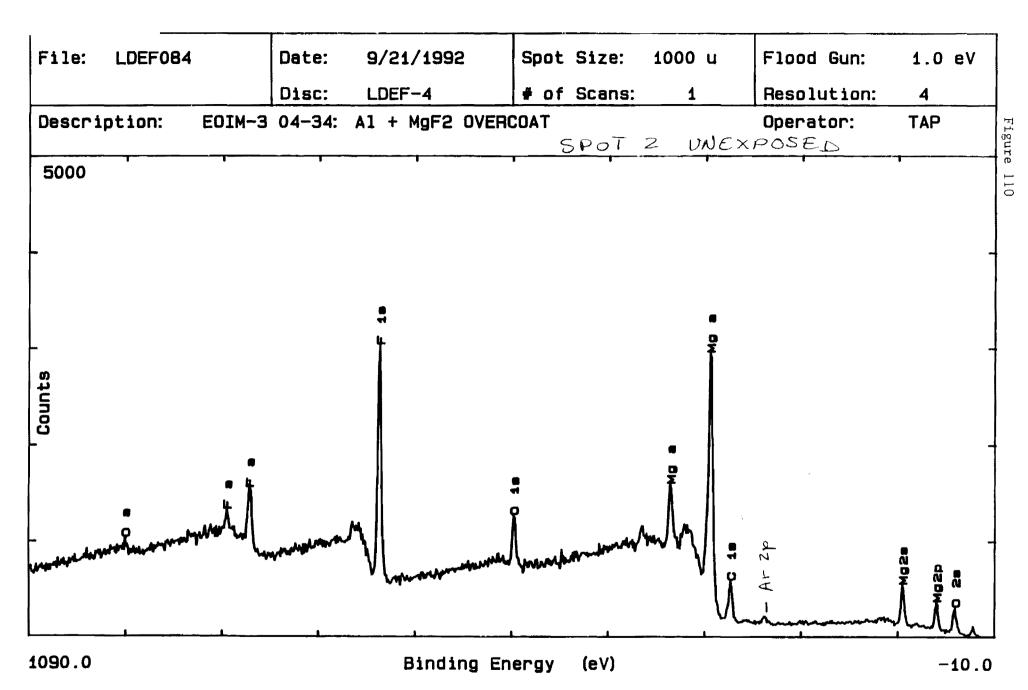
297.0 Binding Energy (eV) 277.0

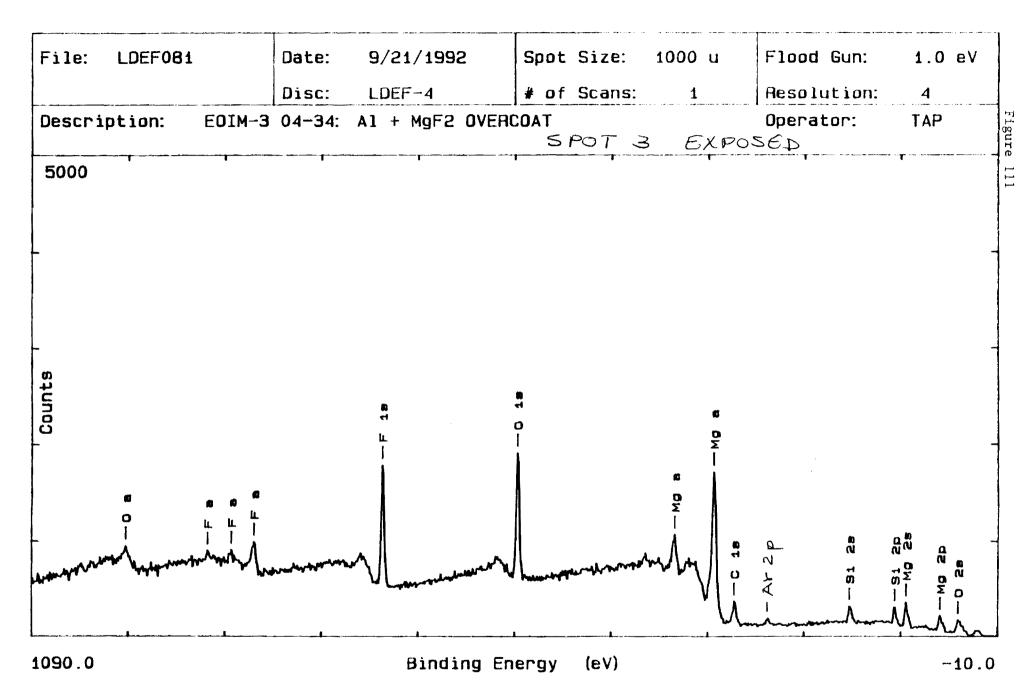


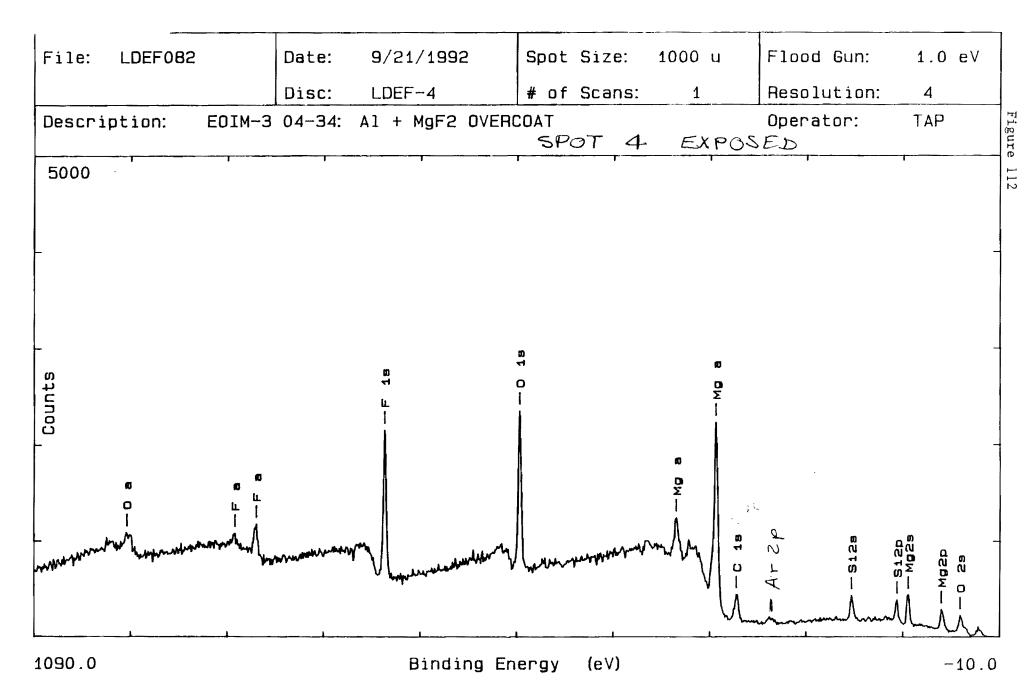
380.0 Binding Energy (eV) 360.0



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