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Surface Elemental Analysis of Niobium Discs for Furnace Evaluation

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1) Introduction:

5 pure niobium discs were furnished to the MDTL lab for EDS (Energy Dispersive Spectroscopy) surface analysis. 5 discs were also submitted to SLAC for XPS analysis by Bob Kirby. The samples were approximately the size of a quarter with a small hole punched in one end with a pure niobium wire going through it. The purpose of the study was to evaluate 3 furnaces, including 1 furnace (Lab IV Large Oven) previously used to braze copper structures for the NLC project, for use with niobium for the ILC project.

The different samples were heat treated in the furnace previously used for copper ("IB4 Large"), the "IB4 Small Oven", and the Lab 7 Oven which has only been used on niobium. Samples disc F1 and F2 were not heat treated. The preparation history of each sample is listed below in Table 1. In Table 1 the grey rows represent samples analyzed at Fermilab by the MDTL department and the white columns represent samples analyzed at SLAC by Bob Kirby. Since the goal of this study was to investigate contamination of the samples in the new furnaces, special precautions were taken to avoid prolonged exposure of the samples to air. The samples as received were stored in containers filled with dry nitrogen. The samples were opened and placed into the SEM/EDS sample chamber as quickly as possible and with as little contact as possible. No sample preparation was done by the MDTL. The samples were run completely blind (It was unknown which sample number corresponded to which sample to avoid prejudice).

In this work Marco did the pre-cleaning and rinsing and had the overall responsibility for the sample logistics. Marco and Tug did the heat treatments in IB4 small and large furnaces. Eileen did the heat treatment in the Lab 7 oven. Donna did the BCP etchings. Tug also established the contact with Bob Kirby. Bob Kirby did XPS analysis.

FURNACE COMPARISON STUDY						
Sample	**Cold Work**	**De-grease**	**100 min Etch**	**750C Heat Treat**	**20 min Etch**	**Comment**
Disc F1	N	Y	BCP	N	N	Reference - XPS
Disc F2	N	Y	BCP	N	N	Reference – SEM, EDS
Disc F3	N	Y	BCP	Y	N	Heat treated in IB4 large oven - XPS
Disc F4	N	Y	BCP	Y	N	Heat treated in IB4 large oven - XPS
Disc F5	N	Y	BCP	Y	N	Heat treat in IB4 large oven - SEM/EDS
Disc F6	N	Y	BCP	Y	N	Heat treated in IB4 small oven - XPS
Disc F7	N	Y	BCP	Y	N	Heat treated in IB4 small oven SEM/EDS
Disc F8	N	Y	BCP	Y	N	Heat treated in Lab 7 oven - XPS
Disc F9	N	Y	BCP	Y	N	Heat treated in Lab 7 oven - SEM/EDS
Disc F10	N	Y	BCP	Y	Y	Heat treated in Lab 7 oven - SEM/EDS

Table 1: Summary of Samples Analyzed

Below a summary of results is listed. There is also a nearly complete listing of the results in appendixes A-F. Appendix G shows sample preparation data.

2) Results and Discussion:

The five samples listed in the grey rows of Table 1 were analyzed using EDS, for elemental analysis, and SEM. The results of these tests are presented in Section 1. Five samples, the white rows from Table 1, were also sent to Stanford University (SLAC) for XPS. XPS uses a X-ray source and is better for giving elemental analysis very close to the surface of the sample (4-5nm). The results from XPS analysis is seen in Section 2.

2.1) EDS Analysis (Energy Dispersive Spectroscopy):

The main purpose of this study was to determine if copper, or other contaminants, were being deposited on the surface of the niobium discs during heat treatment. Unfortunately EDS is not well suited for this. EDS gives an average elemental analysis of the top 2μm of a sample. The copper deposited from these ovens is thought to be in a very thin layer. XPS, an analysis method we do not have on site, is better suited for element analysis in thin films. To get an accurate amount of elements in a metal sample typically ICP is done. ICP is accurate to ppm levels but is a destructive test method and is not available at Fermilab. *No copper was reliably found using EDS on any samples even though it was confirmed to be present by XPS outside of Fermilab.* The samples were all still analyzed to look at surface defects and the results for individual samples are seen below.

Disc F2:

Disc F2 was not heat treated and the surface was filthy. There seemed to be a thin coating of carbon on the entire surface. Figure 1 shows the surface contamination on a macro and micro scale. There are large pieces of contamination (Fig 1A) and what appears to be a thin film of surface contamination (Fig 1B&C) across the entire sample.

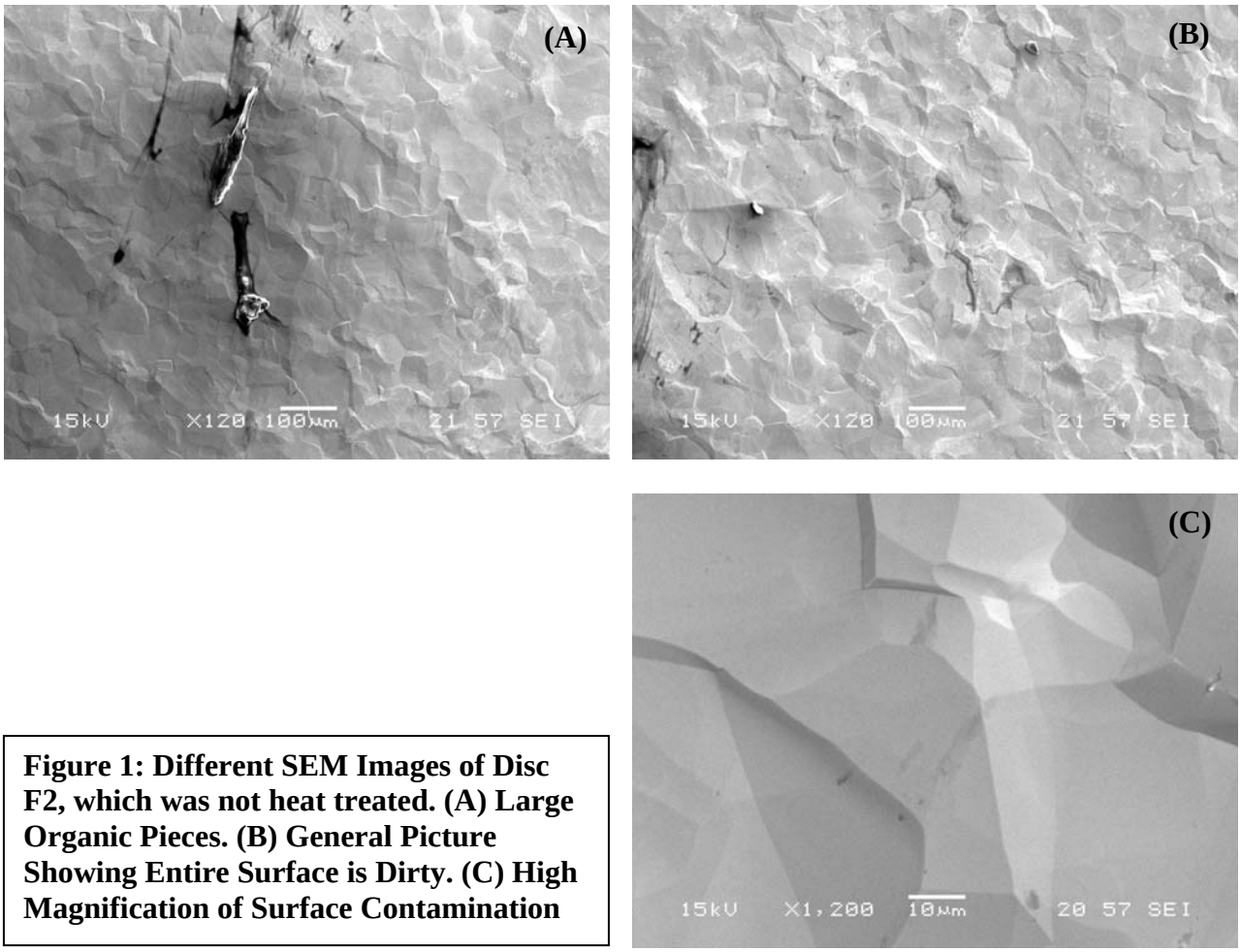
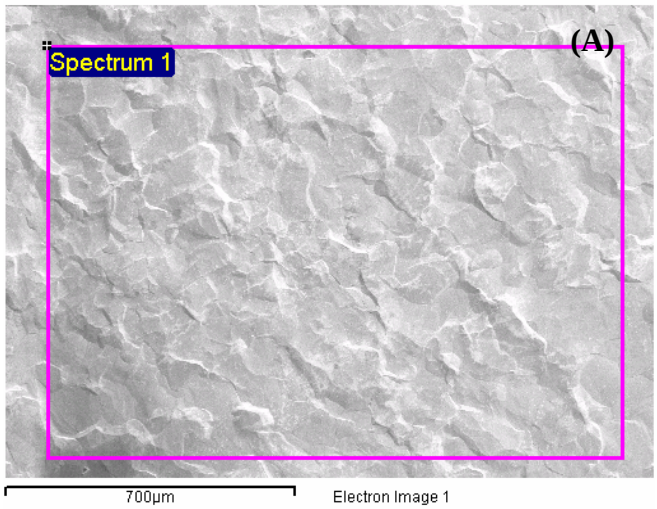


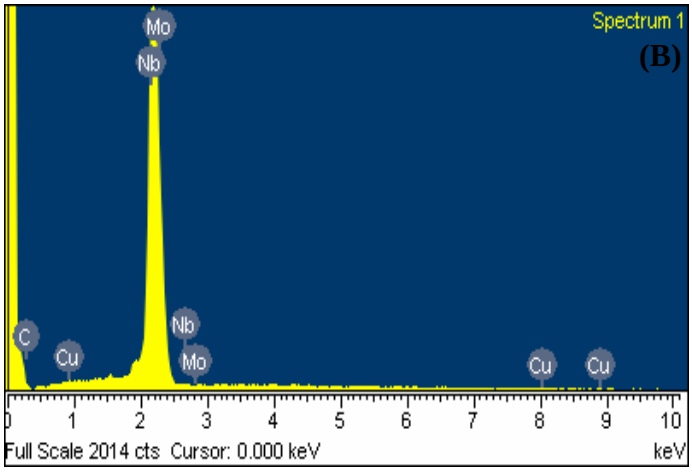
Figure 1: Different SEM Images of Disc F2, which was not heat treated. (A) Large Organic Pieces. (B) General Picture Showing Entire Surface is Dirty. (C) High Magnification of Surface Contamination

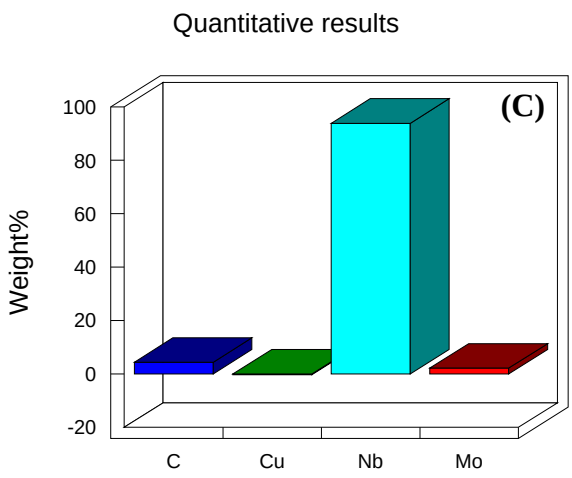
Several individual sites were examined on the niobium disc to determine elemental composition near the surface of the disc (≈2µm penetration depth). Figures 2 and Table 2 show the results of an EDS scan over a large surface of the disc which could be considered to be fairly representative of the entire sample. Table 2 shows that the EDS software predicts -0.40 wt.% copper. This is because the EDS did not detect copper but was manually forced to analyze for copper. There should not be copper on this sample since it did not go in the oven and the EDS results show no copper. From the EDS results there also appears to be molybdenum present. This is only a relatively small area of the sample that is being scanned, and considering that none was seen in other scans, the total amount of Mo in the sample is very small. Carbon was seen in this scan at 4.4 wt%. Since the carbon peak is partially in the noise peak (less than 0.3 keV), the

actual amount of carbon on the sample is probably much less. Carbon was seen in most all scans done on Disc 2.

Figure 3 shows a surface defect that initially was thought to be a deposition but is more likely a scratch since the composition on the area and around the area is the same. Table 3 shows the composition at the four points shown in Figure 3 and Table 4 shows the experimental error of the data point labeled line spectrum 4. The area is 20-25 wt% carbon which is quite high. Also of interest is that the two points in the middle of the scratch show oxygen content. It is possible that due to the increased surface area in the area of the scratch the EDS is better able to detect surface oxidation. The experimental error for line spectrum 4, shown in Table 4 is quite high at 2.23 wt%.







Element	Weight%	Atomic%
C K	4.33	26.00
Cu L	-0.40	-0.46
Nb L	93.89	72.82
Mo L	2.18	1.64
Totals	100.00	

Table 2: Elemental Analysis Disc 2

Figure 2: EDS Scan of a section of Disc 2. (A) SEM picture of area scanned. (B) Characteristic peaks obtained by EDS. (C) Weight % of elements found from EDS.

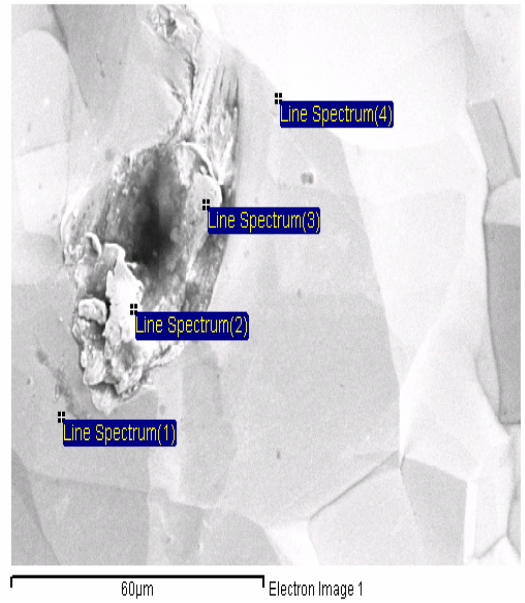


Figure 3: Line Scan of Surface Defect on Disc 2

Spectrum	C	O	Nb	Total
Line Spectrum(1)	23.68		76.32	100.00
Line Spectrum(2)	21.26	2.78	75.97	100.00
Line Spectrum(3)	20.69	2.50	76.81	100.00
Line Spectrum(4)	25.24		74.76	100.00
Max.	25.24	2.78	76.81	
Min.	20.69	0.00	74.76	
All results in weight%				

Table 3: Element Content from Figure 3

Spectrum Label: Line Spectrum(4)				
Element	App Conc.	Weight%	Weight% Sigma	Atomic%
C K	4.87	25.24	2.23	72.31
Nb L	51.07	74.76	2.23	27.69
Totals		100.00		

Table 4: Analysis Error from Figure 3

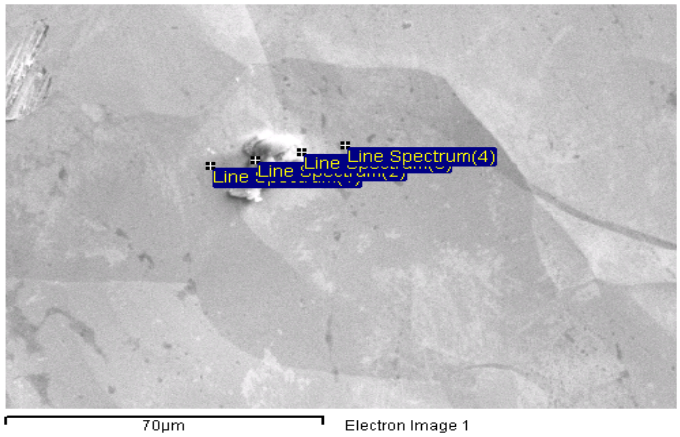


Figure 4: Depicts a Line Scan of a Surface Defect on Disc 2

Spectrum	**C**	**O**	**Na**	**Cl**	**K**	**Nb**	**Total**
Spectrum 1						100.00	100.00
Spectrum 2	57.56	18.14	1.97	1.39	0.64	20.31	100.00
Spectrum 3	48.86	14.76	1.83	0.88		33.66	100.00
Spectrum 4						100.00	100.00
All results in weight%							

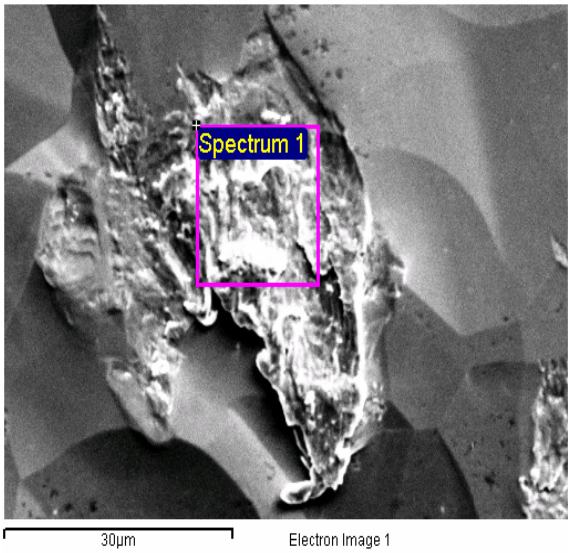
Table 5: Element Analysis for Line Scan Seen In Figure 4

Figure 4 shows a line scan across a piece of sample contamination. Points 1 and 4 are not on the piece of contamination and show 100% niobium. Table 5 shows that points 2 and 3, which are on the piece of contamination, contain salt (Na and Cl) and something organic (carbon and oxygen). This surface contamination is most likely from handling.

There are quite a few more EDS scans in Appendix A. In general Disc 2 was very dirty. The most commonly seen surface impurity was carbon. The carbon concentration was seen as high as 25 wt% on some sections of Disc 2. The carbon would be burned off during heat treatment. One of the EDS scans also showed evidence of molybdenum. There was only 2 wt% molybdenum in one scan. It is most likely that there is no molybdenum since the peak was very small and it may have been a misidentified overlapping niobium peak.

Disc F5:

Disc 5 was heated in the large oven in IB4 to 750°C. Because of this heat treating there was no coating of carbon across the surface of Disc 5 as seen in Disc 2. This is the oven that has been used for copper brazing in the past and has copper deposition on the walls. Figure 5, seen below, shows a common phenomenon seen on Disc 5 only. Small outcroppings, typically ranging in size from 10-50 microns, could be seen scattered periodically across the entire surface of Disc 5. Table 6 shows that the oxygen content of this area is 4.25 wt%. This is consistent with all the EDS data taken on other similar points on Disc 5 with oxygen contents typically ranging from 4.25 to 4.5 wt%. I do not know what caused this phenomenon, but it looks like these spots are not depositions but are part of the niobium disc. It maybe possible that during heating some surface defects reacted with the surface of the niobium disc. There are enough of these spots that it may cause the material to fail if not further etched considerably.



Element	Weight%	Atomic%
O K	4.25	20.51
Nb L	95.75	79.49
Totals	100.00	

Table 6: Elemental Analysis of One of the Typical defects seen on Disc 2

Figure 5: Area Scan of Small Defects Seen on Disc 5

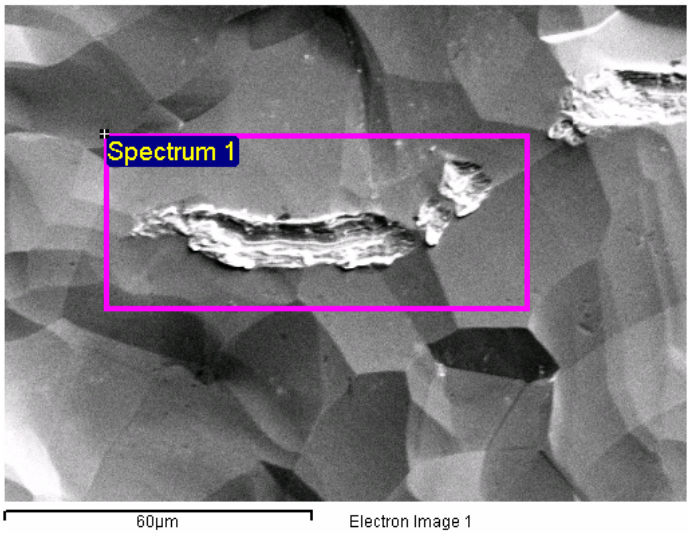


Figure 6: Area Scan of 60µm Scratch on Disc 5

There are many more results seen in Appendix B. There are some EDS scans of several small particles that are most likely post heat treating surface contamination. Some of these particles are possibly dust. There is one large area scan that gives an EDS result showing a large amount of mercury in the scan area. This is almost certainly a false positive due to the fact that the EDS spectrum line for mercury and niobium overlap. Any mercury would have been vaporized at 750°C. This is not uncommon to get false positives for elements due to overlapping spectrum. During EDS analysis of these samples I got false positives for a large range of materials including plutonium and rhodium. Figure 6, seen above, is a common picture of a surface scratch that is seen on several of the samples. The EDS analysis of the scratch showed that it was 100 wt% niobium.

Disc F7:

Disc 7 was heat treated to 750°C in the small oven in IB4. Disc 7 in general seemed to be cleaner than the other samples having little post heat treating surface contamination (ie microscopic dust or dirt). Several EDS scans were run on large and small areas of Disc 7 and all showed 100 wt% EDS. Some of these results are in Appendix C. The only unusual looking part of Disc 7 was seen around the edge of the hole that was cut in the discs to put the niobium wire through. Figure 7 shows the area where an EDS scan was done on one of the "flakes". The

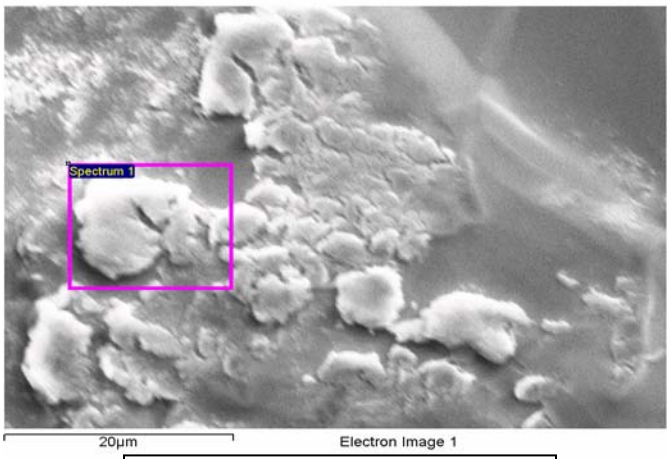


Figure 7: Area Scan of "Flakes" on Disc 7

results showed 100 wt% niobium. The stress on the area surrounding the hole, caused by the cutting of the whole, possibly caused the niobium flakes.

Disc F9:

Disc 9 was heat treated in the oven in Lab 7 to 750°C. Disc 9 had some large surface contamination that was barely visible with the naked eye as well as some smaller surface contamination. Figure 8 shows a 100 micron long particle. The EDS point analysis is given in Figure 9. The particle is primarily carbon and oxygen. This is a post heat treating contamination. Even though the EDS analysis shows that there is niobium, it is not actually in the particle seen in Figure 8. The EDS analysis is actually penetrating the surface particle and getting information from the niobium surface.

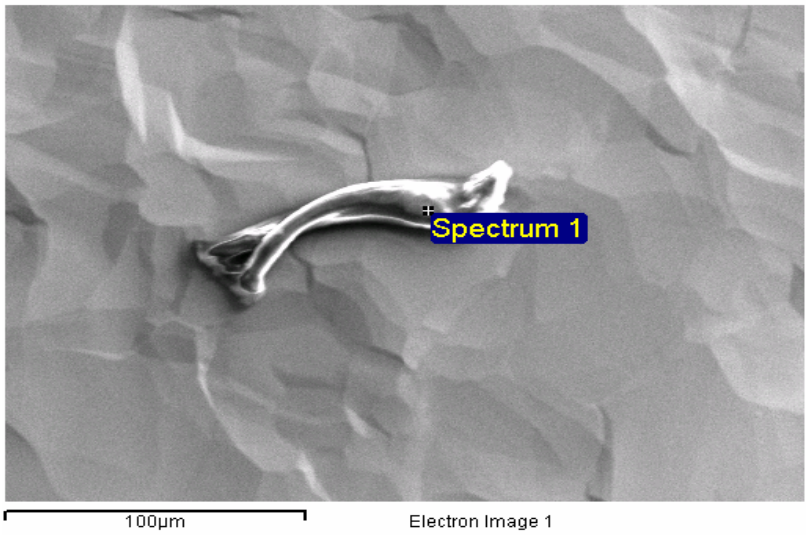


Figure 8: Point Scan of Surface Contamination on Disc 9

The penetration depth of the electron beam source into the sample is a direct function of the energy of the electron beam and the angle of incidence. Unfortunately for these experiments, done with EDS, the penetration depth is on the order of 2 µm. This makes it difficult to see the concentration of elements very near the surface. If an element is only found in the first 0.1 µm of the sample the signal from it will be overwhelmed by the signal of the elements in a majority of the sample.

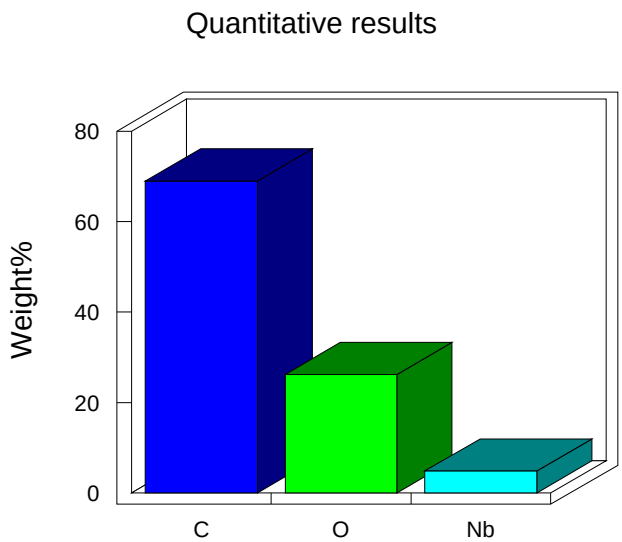


Figure 9: EDS Point Scan Results from Figure 8

Spectrum	Cu	Nb	Total
Line Spectrum(1)	1.97	98.03	100.00
Line Spectrum(2)	0.79	99.21	100.00
Line Spectrum(3)	-1.65	101.65	100.00
Line Spectrum(4)	0.76	99.24	100.00
Line Spectrum(5)	-0.94	100.94	100.00
All results in weight%			

Table 7: Elemental Analysis of Line Scan from Figure 10

Element	Weight%	Weight% Sigma	Atomic%
Cu L	1.97	1.27	2.85
Nb L	98.03	1.27	97.15
Totals	100.00		

Table 8: Error Analysis of Line Spectrum (1) from Table 7.

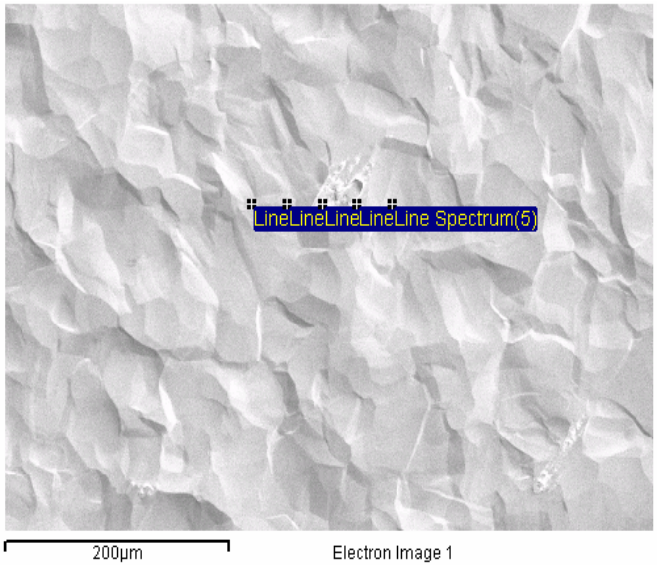


Figure 10: EDS Line Scan of Unusual Area on Disc 9

Since there was a concern that copper from the ovens may be vaporizing and depositing on the niobium samples, copper was looked at for all samples. Typically the EDS will automatically detect what elements are present and determine the weight percent for these elements. However, the software for the EDS can be forced to look at a particular element. In Figure 10 an EDS line scan was done over a large area containing a surface abnormality, specifically looking for copper. Table 7 shows the EDS results when the software was forced to look at copper concentration. Line Spectrum (1), or Point 1 in the scan, shows that there is almost 2 wt% copper in this sample. However this results is highly dubious. Firstly this EDS should not be used to look for elements that are of concentrations less than 1 wt%. This is a kind of practical limitation of the EDS. Also if you look at the experimental error of Line Spectrum(1), seen in Table 8, it is 1.27% for the copper. Additional scans of the area where the Line Spectrum(1) scan was done and copper presence could not be reconfirmed. It is most likely that this is a false positive for copper. *In fact copper was never reliably found in any of the samples analyzed at Fermilab.*

Disc F10:

Sample Disc 10 was heated to 750 C in the Lab 7 oven. Disc 10 had much less post heat treating surface contamination than Disc 9 did. Both Disc 9 and 10 were heat treated in the same oven. Disc 10 was acid etched after heat treating and all the other samples were not. Figure 11 shows an area of Disc 10 that had large scratches that were visible to the naked eye. These scratches were in the proximity of the hole for the niobium wire and were possible caused while making the hole or inserting the wire. Figure 11 shows another method that was attempted to find surface contamination. A line scan was done over a large area and if any contamination was found the area was looked at further. As can be seen from Table 9 the point labeled Line Spectrum (7)

showed carbon and oxygen. This point was analyzed closer (seen in Appendix D) and found to be a micron sized piece of dust or other surface contaminant.

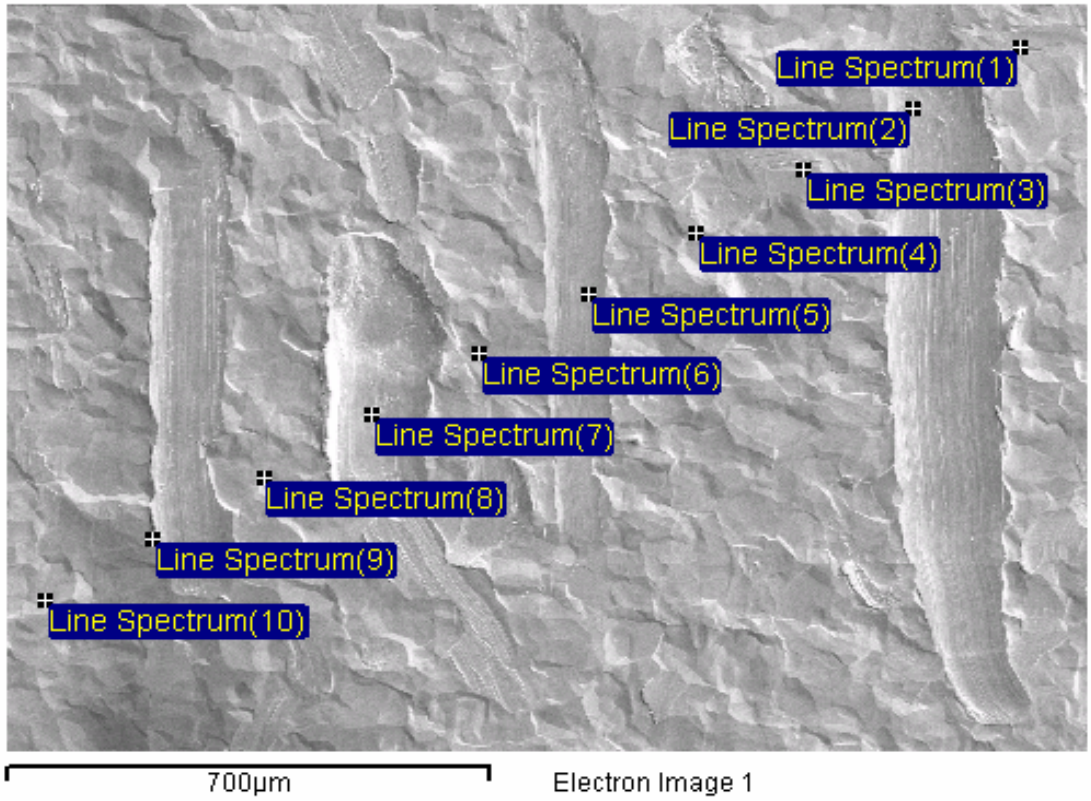


Figure 11: EDS Line Scan of Scratches on Disc 9

Spectrum	C	O	Nb	Total
Line Spectrum(1)			100.00	100.00
Line Spectrum(2)			100.00	100.00
Line Spectrum(3)			100.00	100.00
Line Spectrum(4)			100.00	100.00
Line Spectrum(5)			100.00	100.00
Line Spectrum(6)			100.00	100.00
Line Spectrum(7)	23.55	6.33	70.12	100.00
Line Spectrum(8)			100.00	100.00
Line Spectrum(9)			100.00	100.00
Line Spectrum(10)			100.00	100.00

All values are in weight %.

Table 9: Elemental Analysis of Line Scan from Figure 11

2.2) XPS Analysis (X-Ray Photoelectron Spectroscopy):

Samples similar to what were analyzed at Fermi using EDS were analyzed at SLAC using XPS by Robert Kirby. Table 10 below lists the samples that were analyzed and the major contaminants found. Appendix F shows all 5 XPS scans run on the various samples.

Details of Analysis

Use x-ray photoelectron spectroscopy (XPS) to measure surface condition.

Results of Analysis

Sample #	Furnace Name	Carbon Conc.(Atom%)	Other Contaminants
F1	Unbaked ref.	32	F(3.3%)
F3	AVS large vac.	21	
F4	"	32	
F6	IVI small vac.	21	
F8	Lab 7	19	Cu(0.5%), Sn(1.4%)

All of these coupons have relatively low carbon on them, given the extreme gettering ability of Nb. The Lab 7 furnace witness has Cu and Sn on it. Also, all witnesses have oxide on them. If the furnace has an air leak, Nb witnesses will not be the way to detect it. Finally, F1 has F (BCP HF acid residue?) on it, which may build up over time in the furnace atmosphere.

Table 10: List of Samples Analyzed Using XPS and Summary of Results

Figure 12 shows the XPS scan of the sample heat treated in the Lab 7 furnace. Five elements are identified, Cu, O, Sn, C, and Nb.

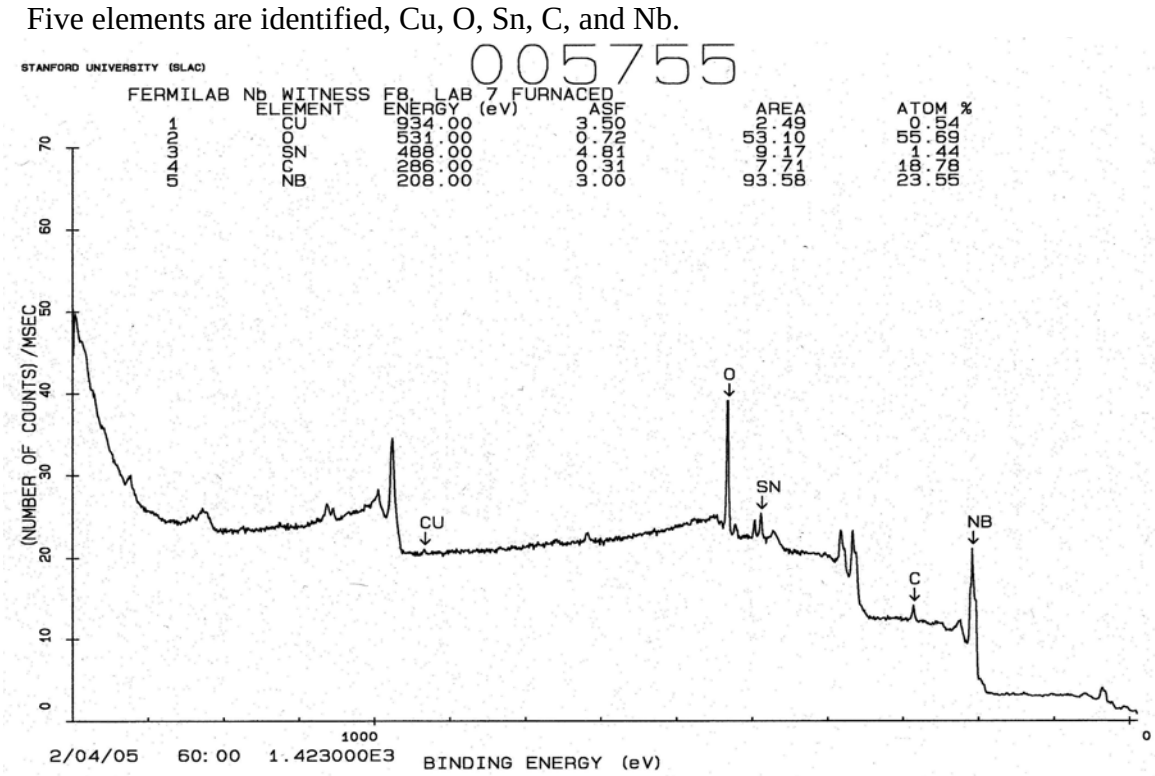


Figure 12: XPS Scan of Disc 8 (LAB 7 Furnace)

The area analyzed for surveys is 4 x 4 mm^2 at the sample using XPS. The x-ray penetration depth is approximately 2 μm, but the photoelectron escape depth, from which usable information is recovered, is about 3-4 nm for 932 eV.

As the summary suggests, and the data supports, all the samples were found to have carbon and oxygen on the top 3-4 nm of the niobium discs. This is not surprising since the samples are exposed to the air which contains oxygen and carbon from carbon dioxide. The sample that was not heat treated was found to have residual fluorine from the BCP etch. The sample heat treated in the Lab 7 furnace was found to have tin and possibly copper. It is interesting that the niobium sample from the Lab 7 oven, the oven which has only been used to heat pure niobium, would show tin and possibly copper contamination. It maybe possible that 2 of the samples got switched. It would be helpful to have a repeat XPS scan run for a longer time. Scans run for a longer time have more counts and therefore better resolution.

Summary:

The main objective of this work was to analyze the sample discs to determine if the ovens used were introducing surface contamination. It was feared that copper deposited on the walls of some of the ovens may vaporize and deposit on the niobium. No copper was ever reliably found using the EDS. Copper was found on Disc 9 (Lab 7 Furnace) but it was in such a small amount that it was within experimental error. This does not mean that copper is not present. It is possible that copper is deposited in a thin sub-micron layer that is not detectable using the EDS in IB3.

It was found that the samples do still have a lot of post heat treating contamination on the surface. Carbon and oxygen were found throughout the samples but only on pieces on the surface not as thin films on or in the surface.

Disc 2 (not heat treated) had a large amount of surface contamination and what looked like a coating of carbon on the surface. Disc 5 (IB4 large oven) had a lot of oxygen surface contamination that was found at unusual looking outcroppings throughout the sample.

XPS was used to find carbon and oxygen in all samples. It is not discussed in the results if the carbon and oxygen is uniformly deposited on the samples or is in small pieces like dust particles. Fluorine (residual from BCP) was found on the disc that was not heat treated. Copper and tin were found in the sample treated in the Lab 7 furnace. Copper was found at 0.5 atom %, but the Cu line is only 3-sigma above background which is close to being noise in the spectrum.

The source of the carbon and oxygen contamination is hard to determine. It is uncertain how much of the carbon and oxygen contamination occurs during and after heat treating. It is possible that the carbon and oxygen are only physical adsorbed on the surface from the air. If these samples are sent out for TGA analysis it would be possible to determine if the carbon and oxygen were chemi or physi-sorbed on the niobium surface. This would tell us if the carbon and oxygen contamination is occurring during or after heat treating. If it is found that the carbon and oxygen are chemically bound to the niobium surface the operating procedure will have to be modified to make sure the heating atmosphere is completely inert.

The Lab IV small and large ovens don't appear to introduce any other contaminants to the niobium than the carbon and oxygen previously mentioned. From the XPS results, the Lab 7 oven appears to be vaporizing tin and copper from the walls of the oven and depositing it on the niobium disc. This seems unlikely if that oven has only ever been used for niobium. Even if the samples are etched after the heat treatment it is possible that substantial surface depositions on the niobium could cause uneven etching or further contamination by dissolved copper or tin that could diffuse into the niobium surface during etching.

Appendix A – Not Heat Treated

Sample Disc #2
Analysis 1

Pure Nb Disc #2

4/11/2005 10:41:43 AM

Project: Pierre Nb Disc Purity
Owner: SEMUser
Site: Site of Interest 1

Sample: Nb Disc #2
Type: Default
ID: Nb Disc #2

Spectrum Label: Line Spectrum(4)
Livetime 180.0 s
Acquisition geometry (degrees):
Tilt = 0.0
Azimuth = 0.0
Elevation = 25.0
Accelerating voltage = 15.00 kV
Total spectrum counts = 115670

Sample data :	Energy (eV)	Resn. (eV)	Area
Strobe :	.0	70.90	342115

No quant optimization performed

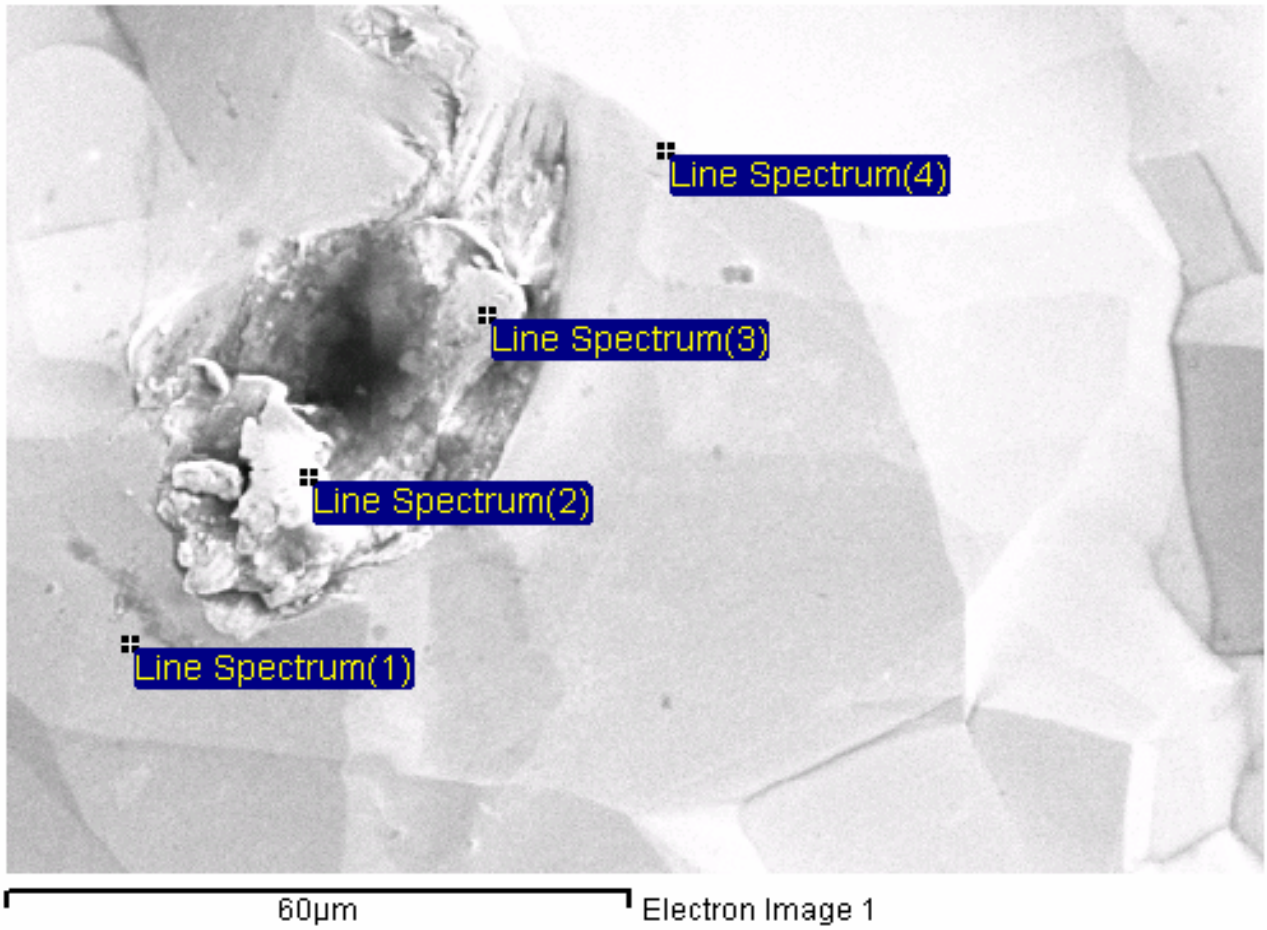
Sample is unpolished X-ray corrections may be approximate.
Sample is uncoated
No optimization has been performed.

Spectrum processing :
No peaks omitted

Processing option : All elements analyzed (Normalised)
Number of iterations = 3

Standard :
C CaCO3 1-Jun-1999 12:00 AM
Nb Nb 1-Jun-1999 12:00 AM

Element	App Conc.	Intensity Corrn.	Weight%	Weight% Sigma	Atomic%
C K	4.87	0.2669	25.24	2.23	72.31
Nb L	51.07	0.9460	74.76	2.23	27.69
Totals			100.00		

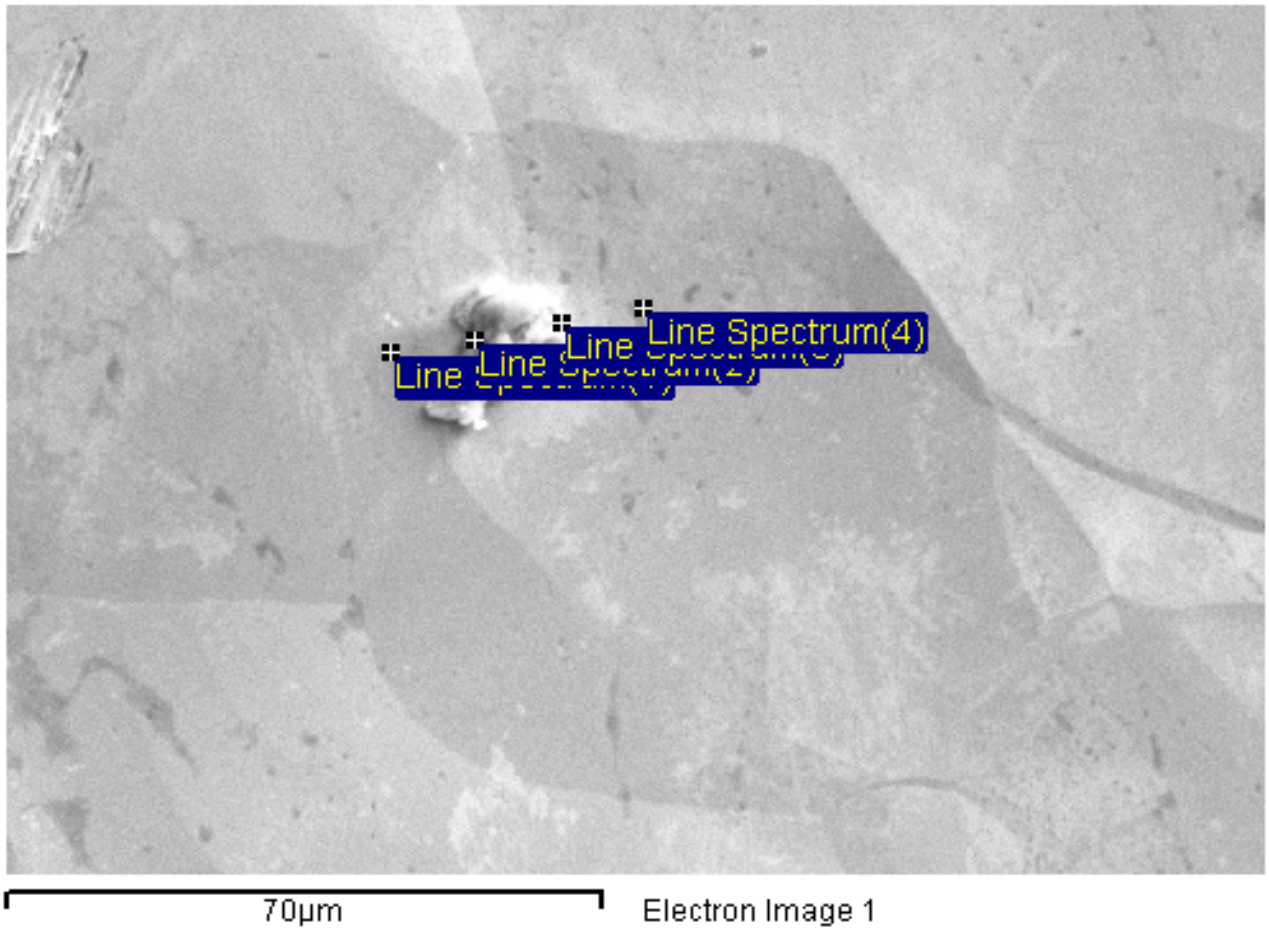


Processing option : All elements analysed (Normalised)

Spectrum	In stats.	C	O	Nb	Total
Line Spectrum(1)	Yes	23.68		76.32	100.00
Line Spectrum(2)	Yes	21.26	2.78	75.97	100.00
Line Spectrum(3)	Yes	20.69	2.50	76.81	100.00
Line Spectrum(4)	Yes	25.24		74.76	100.00
Max.		25.24	2.78	76.81	
Min.		20.69	0.00	74.76	

All results in weight%

Sample Disc #2
Analysis 2



Processing option : All elements analysed (Normalised)

Spectrum	In stats.	C	O	Na	Cl	K	Nb	Total
Line Spectrum(1)	Yes						100.00	100.00
Line Spectrum(2)	Yes	57.56	18.14	1.97	1.39	0.64	20.31	100.00
Line Spectrum(3)	Yes	48.86	14.76	1.83	0.88		33.66	100.00
Line Spectrum(4)	Yes						100.00	100.00
Max.		57.56	18.14	1.97	1.39	0.64	100.00	
Min.		0.00	0.00	0.00	0.00	0.00	20.31	

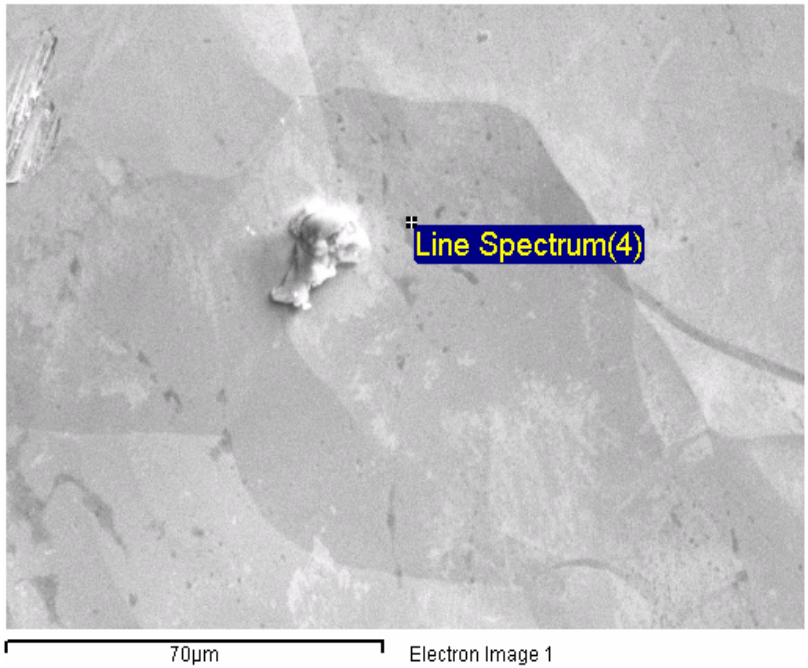
All results in weight%

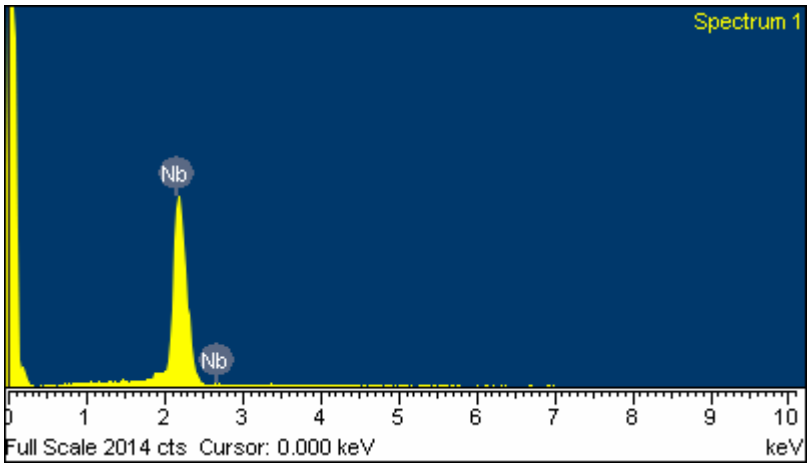
Spectrum processing :
No peaks omitted

Processing option : All elements analyzed (Normalised)
Number of iterations = 1

Standard :
Nb Nb 1-Jun-1999 12:00 AM

Element	Weight%	Atomic%
Nb L	100.00	100.00
Totals	100.00	



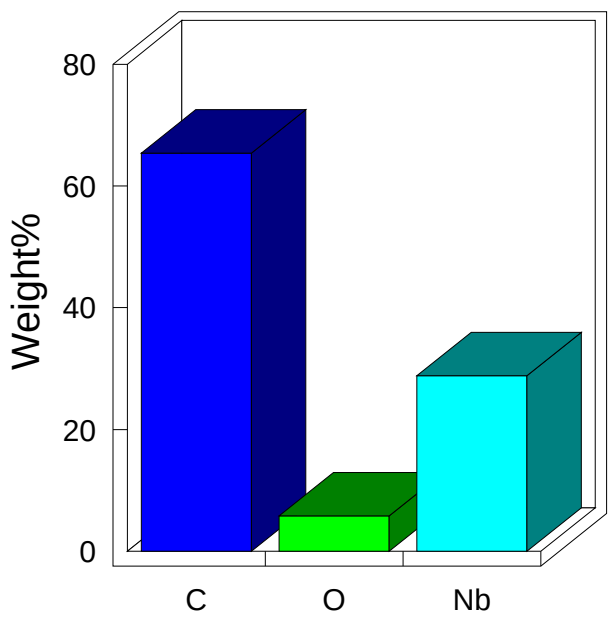


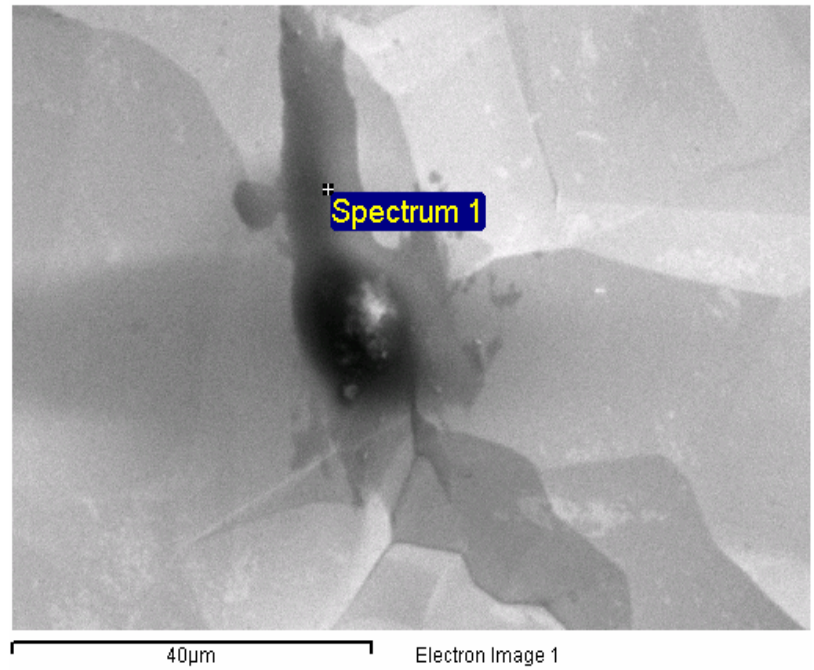
Sample Disc #2
Analysis 3

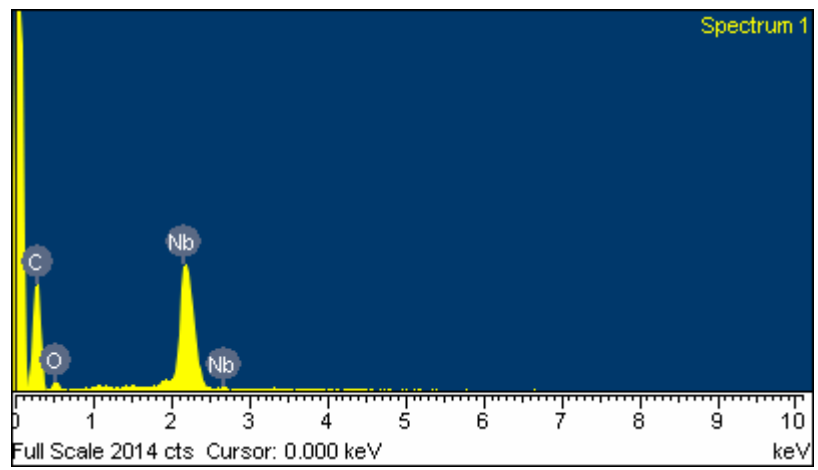
Spectrum processing :
Peak possibly omitted : 3.330 keV
Processing option : All elements analyzed (Normalised)
Number of iterations = 5
Standard :
C CaCO3 1-Jun-1999 12:00 AM
O SiO2 1-Jun-1999 12:00 AM
Nb Nb 1-Jun-1999 12:00 AM

Element	Weight%	Atomic%
C K	65.35	88.98
O K	5.81	5.94
Nb L	28.84	5.08
Totals	100.00	

Quantitative results







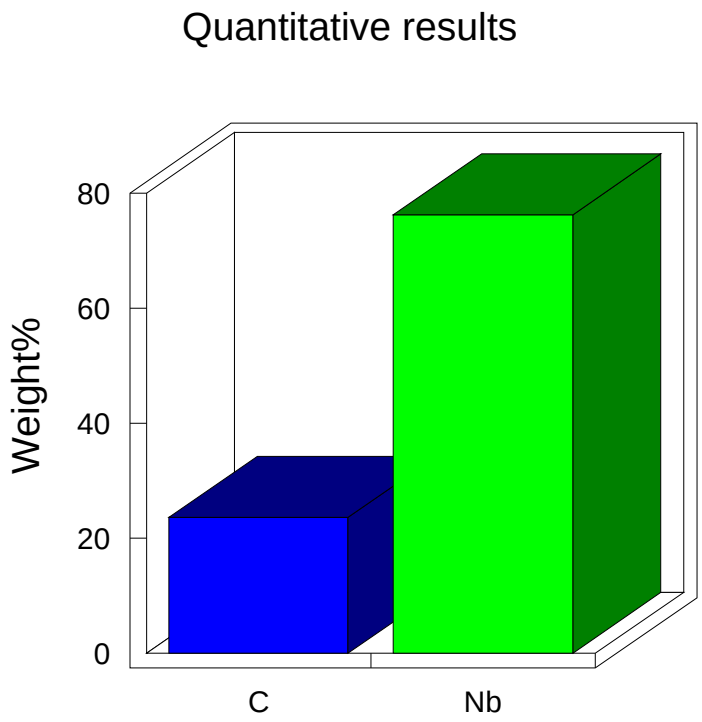
Sample Disc #2
Analysis 4

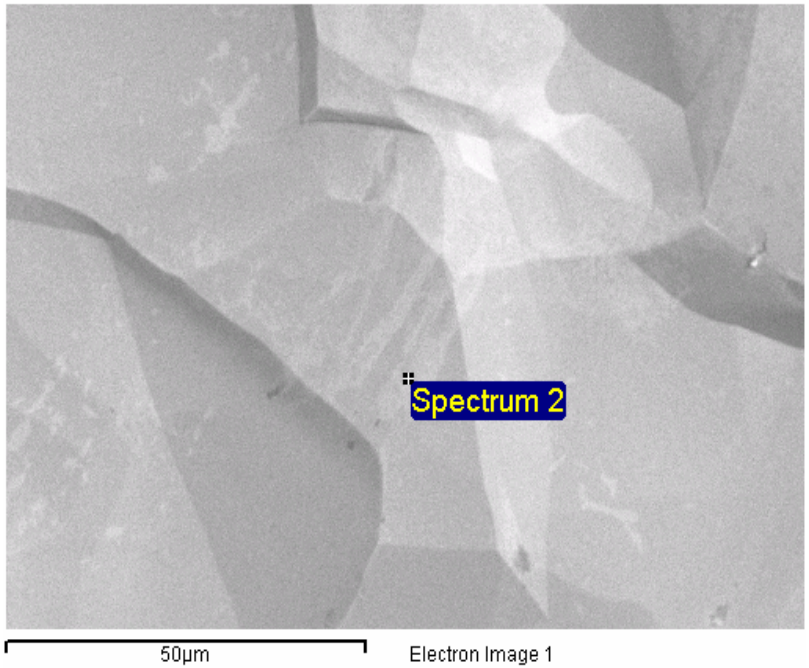
Spectrum processing :
No peaks omitted

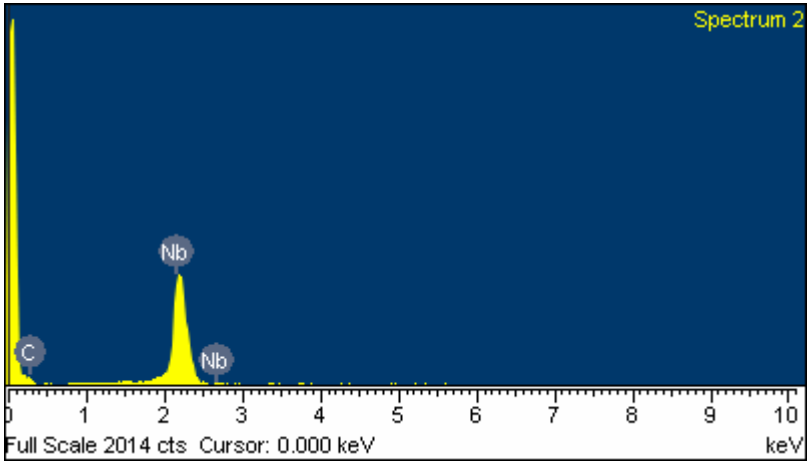
Processing option : All elements analyzed (Normalised)
Number of iterations = 3

Standard :
C CaCO3 1-Jun-1999 12:00 AM
Nb Nb 1-Jun-1999 12:00 AM

Element	Weight%	Atomic%
C K	23.68	70.59
Nb L	76.32	29.41
Totals	100.00	







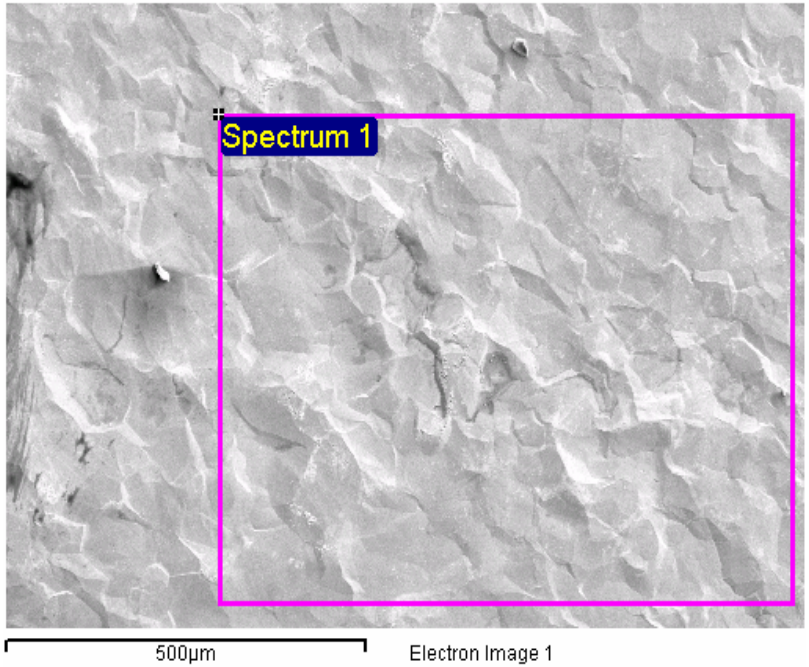
Sample Disc #2
Analysis 5

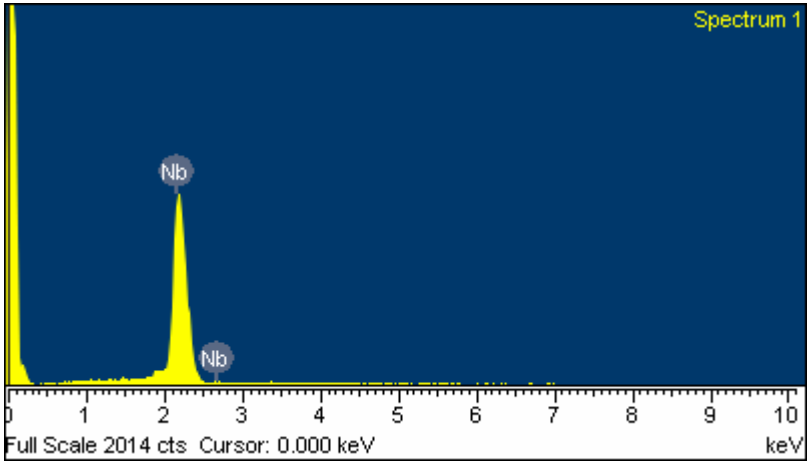
Spectrum processing :
No peaks omitted

Processing option : All elements analyzed (Normalised)
Number of iterations = 1

Standard :
Nb Nb 1-Jun-1999 12:00 AM

Element	Weight%	Atomic%
Nb L	100.00	100.00
Totals	100.00	

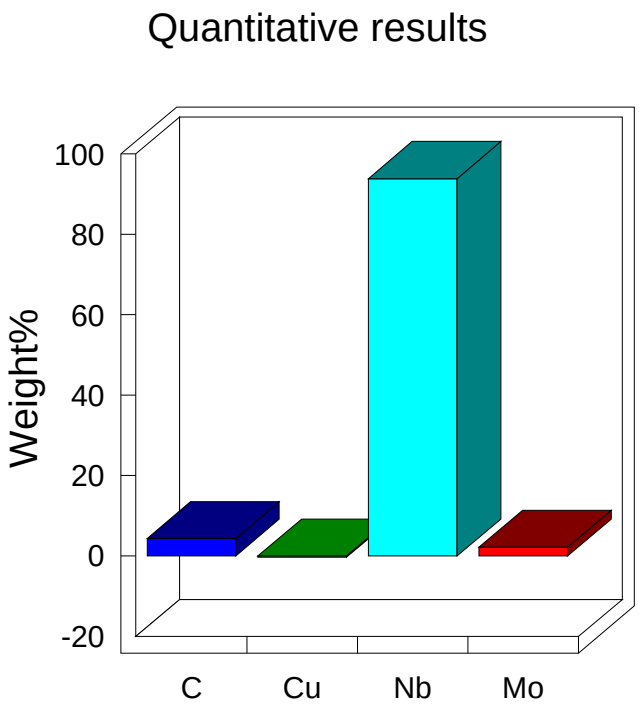


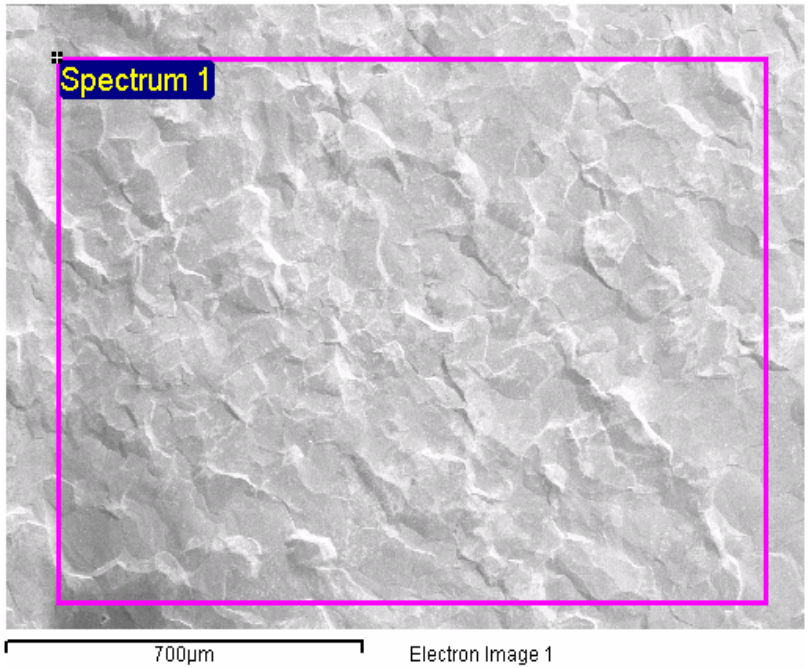


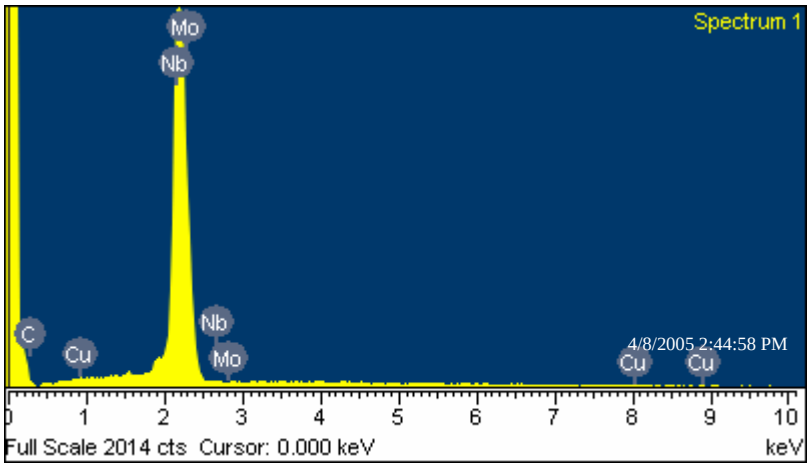
Sample Disc #2
Analysis 6

Spectrum processing :
No peaks omitted
Processing option : All elements analyzed (Normalised)
Number of iterations = 3
Standard :
C CaCO3 1-Jun-1999 12:00 AM
Cu Cu 1-Jun-1999 12:00 AM
Nb Nb 1-Jun-1999 12:00 AM
Mo Mo 1-Jun-1999 12:00 AM

Element	Weight%	Atomic%
C K	4.33	26.00
Cu L	-0.40	-0.46
Nb L	93.89	72.82
Mo L	2.18	1.64
Totals	100.00	







Spectrum Label: Spectrum 1

Livetime 179.9 s

Acquisition geometry (degrees):
Tilt = 0.0
Azimuth = 0.0
Elevation = 25.0

Accelerating voltage = 15.00 kV

Total spectrum counts = 131939

Sample data :	Energy (eV)	Resn. (eV)	Area
Strobe :	.0	71.80	344059

No quant optimization performed

Sample is unpolished X-ray corrections may be approximate.
Sample is uncoated
No optimization has been performed.

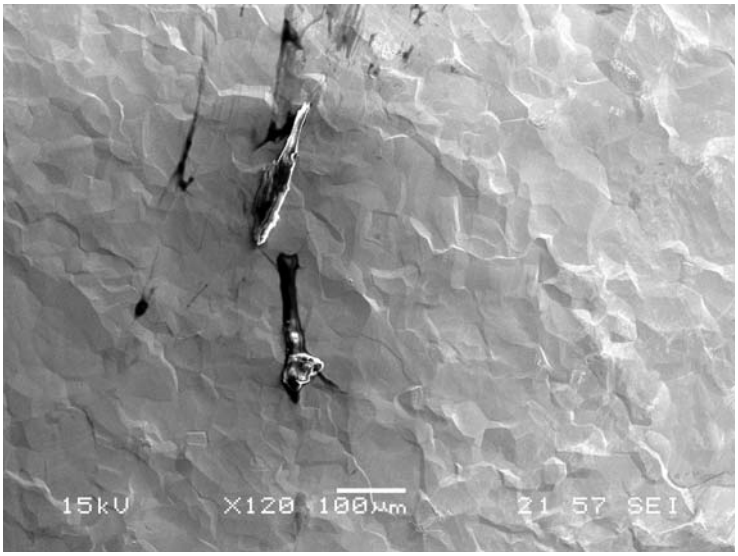
Comment: The Surface of this sample in general looked very bad. It looked like there was a light stain across the whole surface of the Nb disc.

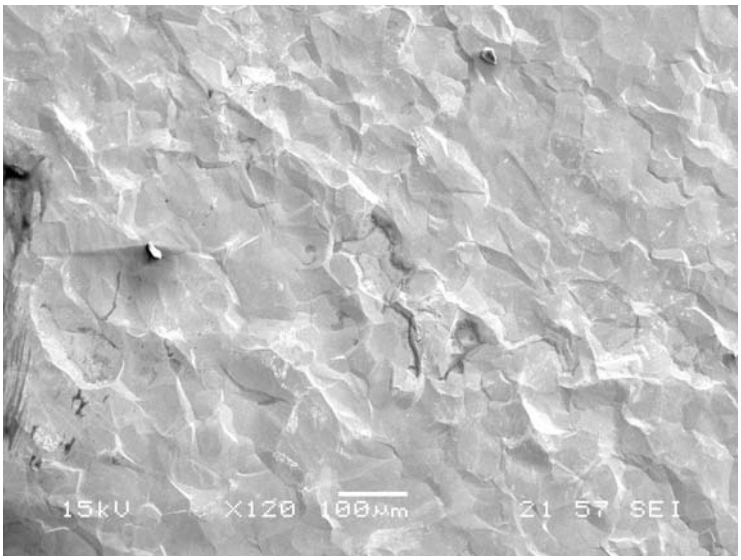
Spectrum processing :
No peaks omitted

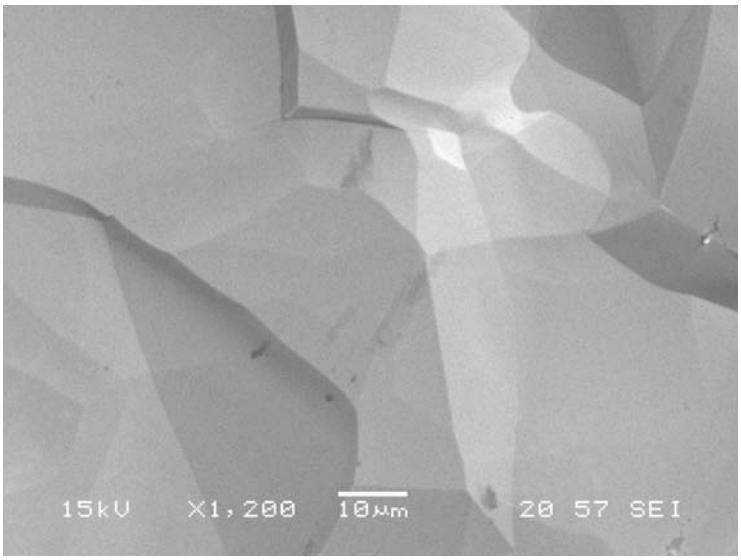
Processing option : All elements analyzed (Normalised)
Number of iterations = 3

Standard :
C CaCO3 1-Jun-1999 12:00 AM
Cu Cu 1-Jun-1999 12:00 AM
Nb Nb 1-Jun-1999 12:00 AM
Mo Mo 1-Jun-1999 12:00 AM

Element	App Conc.	Intensity Corrn.	Weight%	Weight% Sigma	Atomic%
C K	0.55	0.2257	4.33	2.79	26.00
Cu L	-0.10	0.4529	-0.40	0.58	-0.46
Nb L	52.65	0.9898	93.89	3.03	72.82
Mo L	1.23	0.9990	2.18	1.23	1.64
Totals			100.00		


15kV X120 100µm 21 57 SEI


15kV X120 100µm 21 57 SEI


15kV X1,200 10µm 20 57 SEI

Appendix B – Large IB4 Oven

Sample Disc #5
Analysis 1

Pure Nb Disc #5

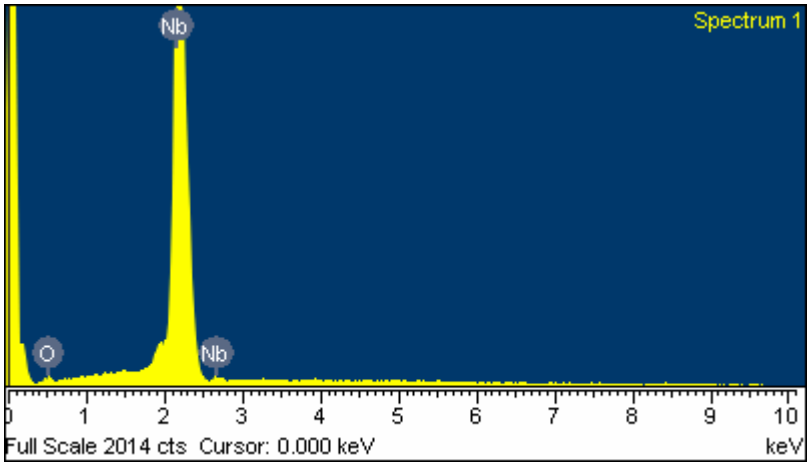
4/5/2005 2:50:09 PM

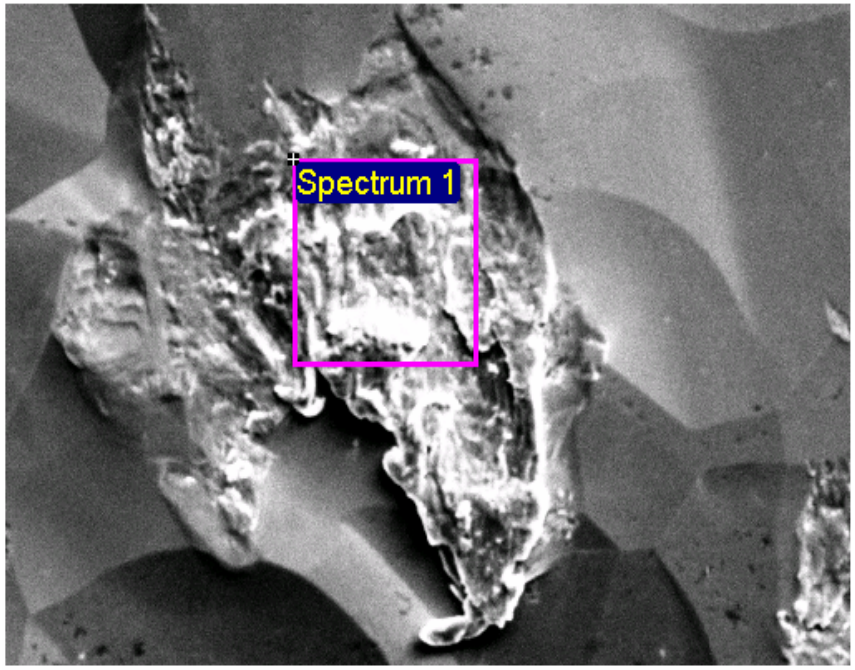
Spectrum processing :
No peaks omitted

Processing option : All elements analyzed (Normalised)
Number of iterations = 3

Standard :
O SiO2 1-Jun-1999 12:00 AM
Nb Nb 1-Jun-1999 12:00 AM

Element	Weight%	Atomic%
O K	4.25	20.51
Nb L	95.75	79.49
Totals	100.00	





30µm Electron Image 1

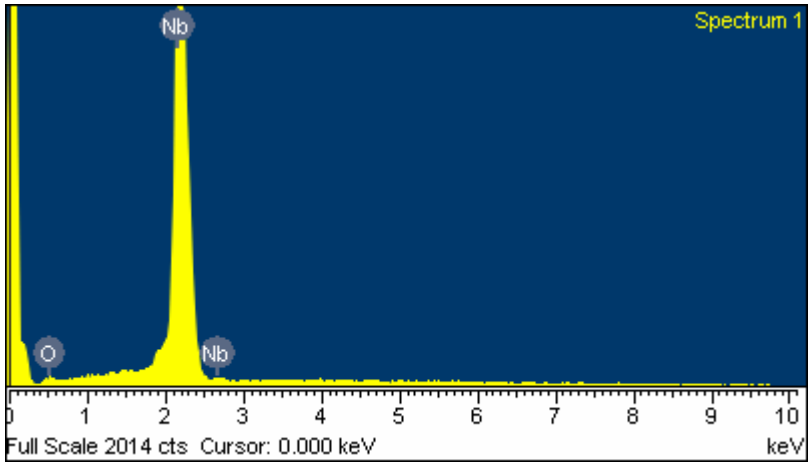
Sample Disc #5
Analysis 2

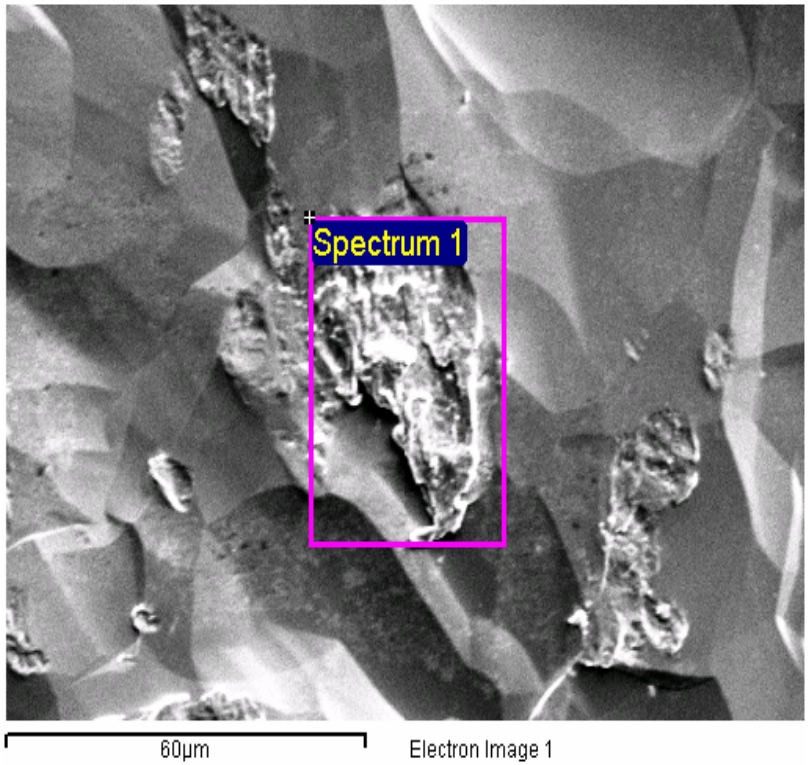
Spectrum processing :
Peak possibly omitted : 6.650 keV

Processing option : All elements analyzed (Normalised)
Number of iterations = 2

Standard :
O SiO2 1-Jun-1999 12:00 AM
Nb Nb 1-Jun-1999 12:00 AM

Element	Weight%	Atomic%
O K	2.71	13.94
Nb L	97.29	86.06
Totals	100.00	





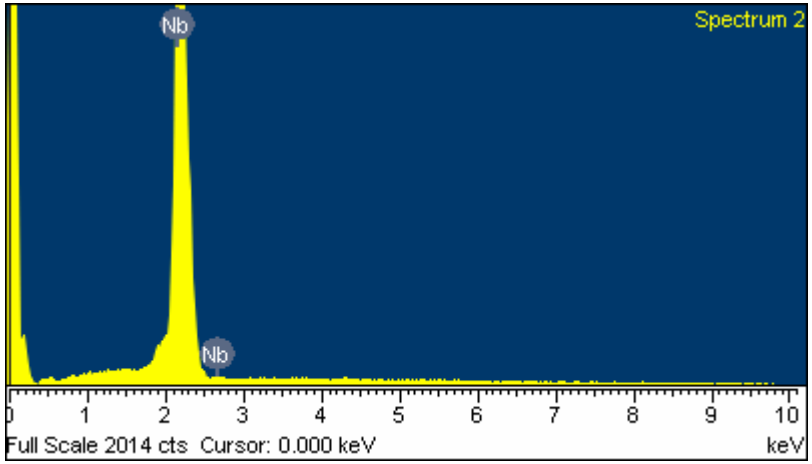
Sample Disc #5
Analysis 3

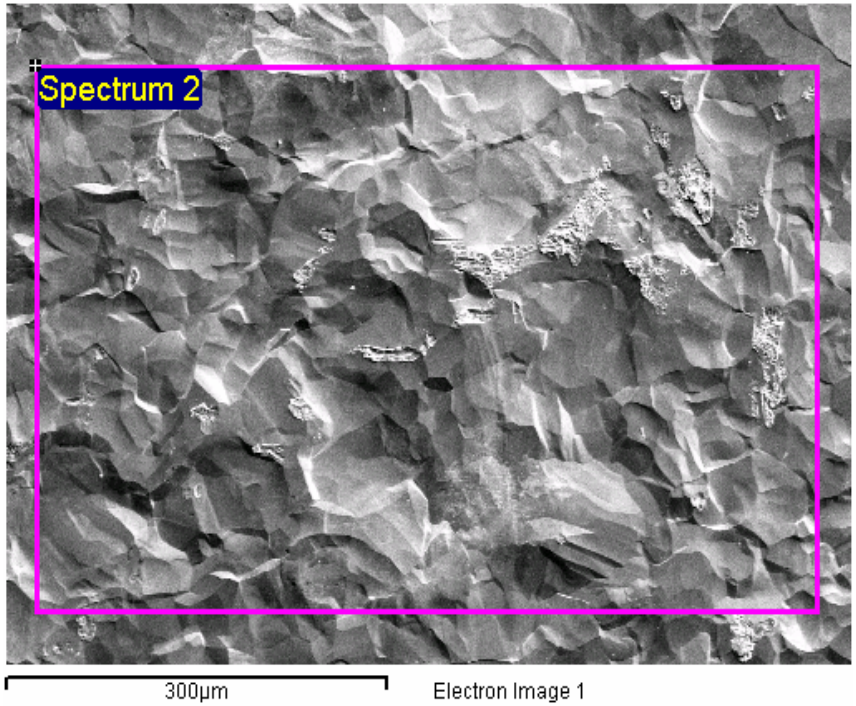
Spectrum processing :
No peaks omitted

Processing option : All elements analyzed (Normalised)
Number of iterations = 1

Standard :
Nb Nb 1-Jun-1999 12:00 AM

Element	Weight%	Atomic%
Nb L	100.00	100.00
Totals	100.00	





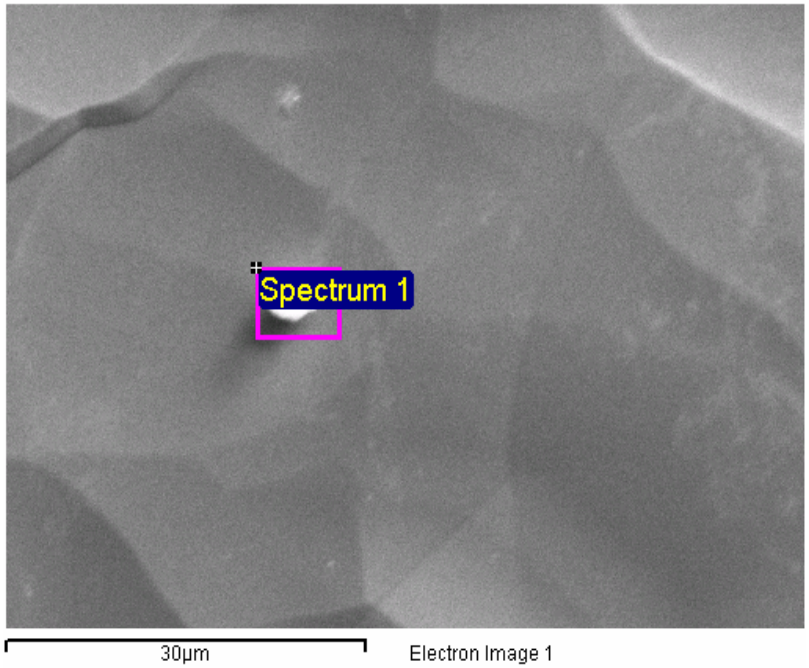
Sample Disc #5
Analysis 4

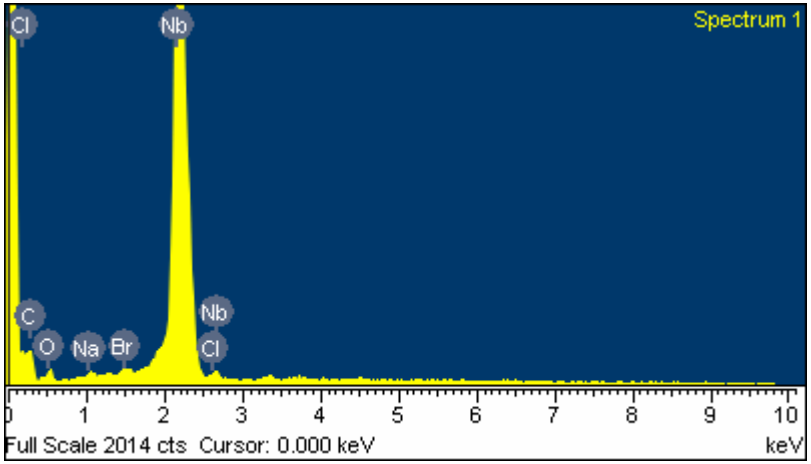
Spectrum processing :
Peak possibly omitted : 3.349 keV

Processing option : All elements analyzed (Normalised)
Number of iterations = 4

Standard :
C CaCO3 1-Jun-1999 12:00 AM
O SiO2 1-Jun-1999 12:00 AM
Na Albite 1-Jun-1999 12:00 AM
Cl KCl 1-Jun-1999 12:00 AM
Br KBr 1-Jun-1999 12:00 AM
Nb Nb 1-Jun-1999 12:00 AM

Element	Weight%	Atomic%
C K	32.52	70.03
O K	7.75	12.52
Na K	0.59	0.66
Cl K	0.65	0.48
Br L	0.69	0.22
Nb L	57.80	16.09
Totals	100.00	





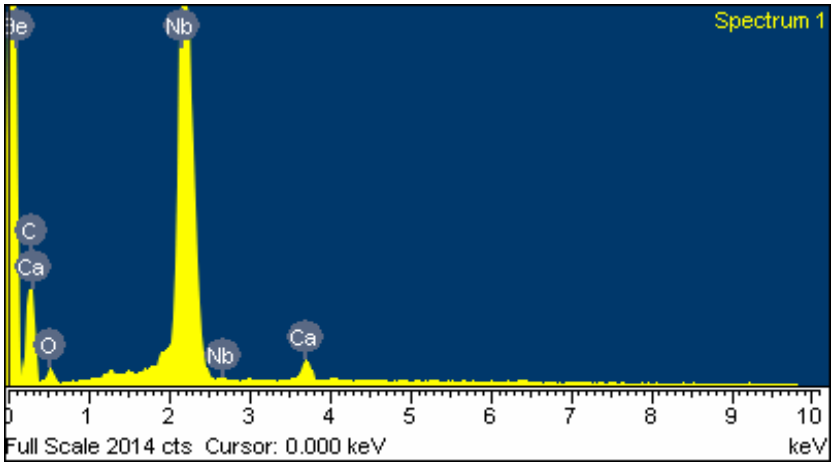
Sample Disc #5
Analysis 5

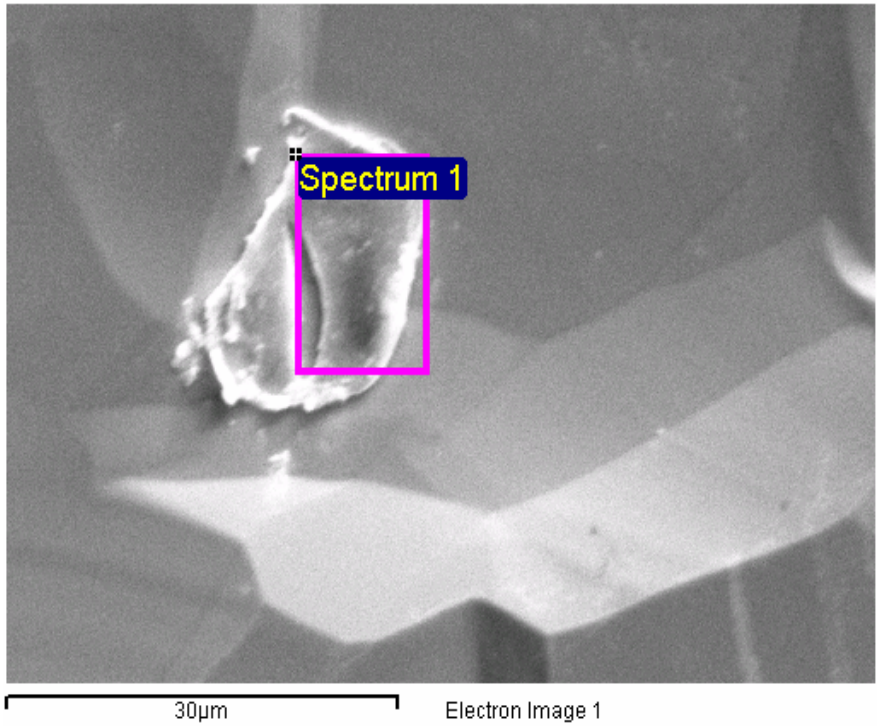
Spectrum processing :
No peaks omitted

Processing option : All elements analyzed (Normalised)
Number of iterations = 4

Standard :
C CaCO3 1-Jun-1999 12:00 AM
O SiO2 1-Jun-1999 12:00 AM
Ca Wollastonite 1-Jun-1999 12:00 AM
Nb Nb 1-Jun-1999 12:00 AM

Element	Weight%	Atomic%
C K	52.74	82.61
O K	7.70	9.06
Ca K	1.20	0.56
Nb L	38.36	7.77
Totals	100.00	





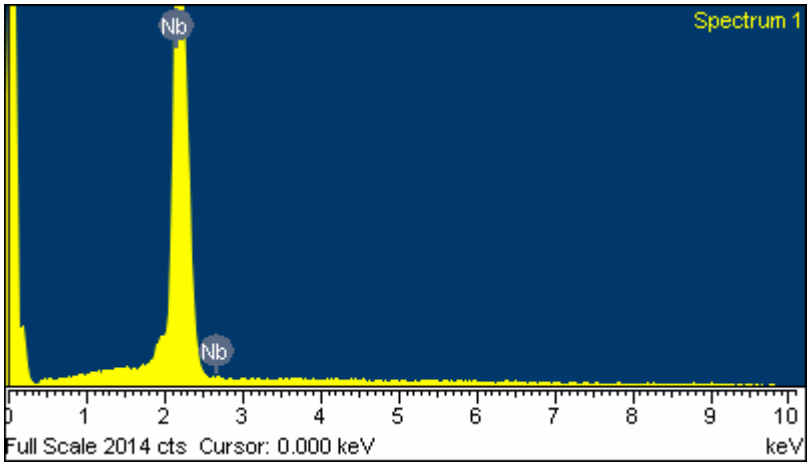
Sample Disc #5
Analysis 6

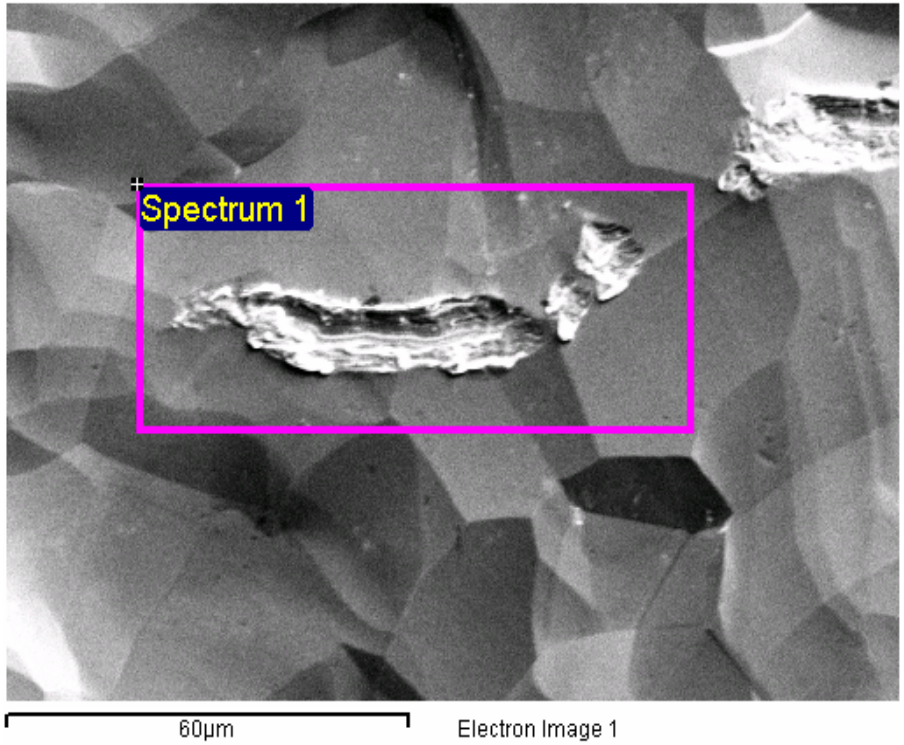
Spectrum processing :
No peaks omitted

Processing option : All elements analyzed (Normalised)
Number of iterations = 1

Standard :
Nb Nb 1-Jun-1999 12:00 AM

Element	Weight%	Atomic%
Nb L	100.00	100.00
Totals	100.00	





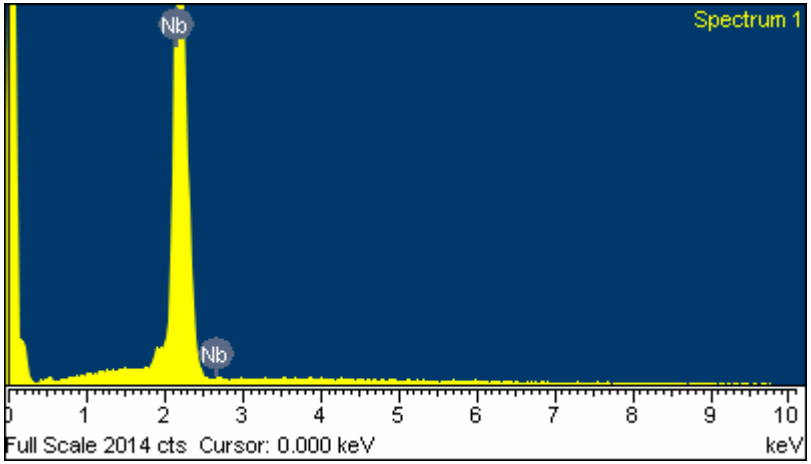
Sample Disc #5
Analysis 7

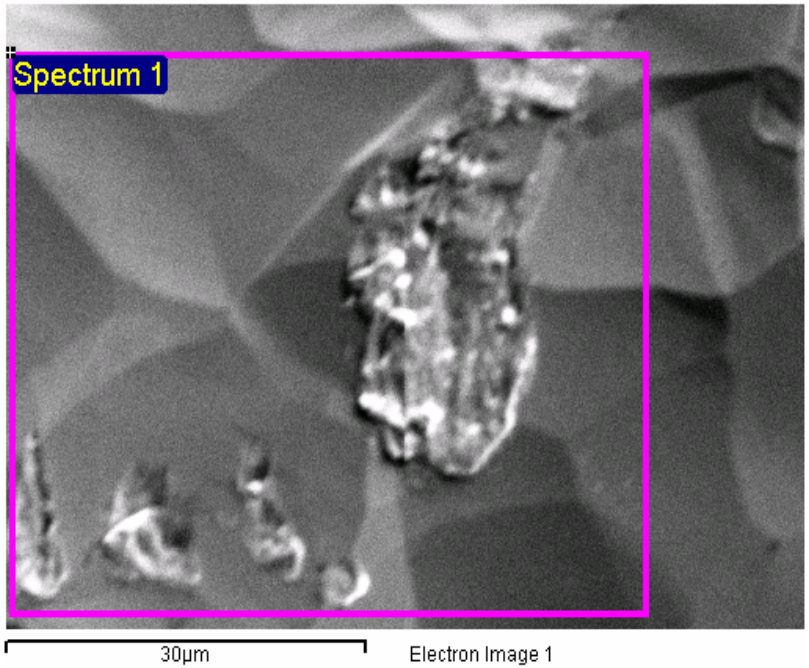
Spectrum processing :
No peaks omitted

Processing option : All elements analyzed (Normalised)
Number of iterations = 1

Standard :
Nb Nb 1-Jun-1999 12:00 AM

Element	Weight%	Atomic%
Nb L	100.00	100.00
Totals	100.00	





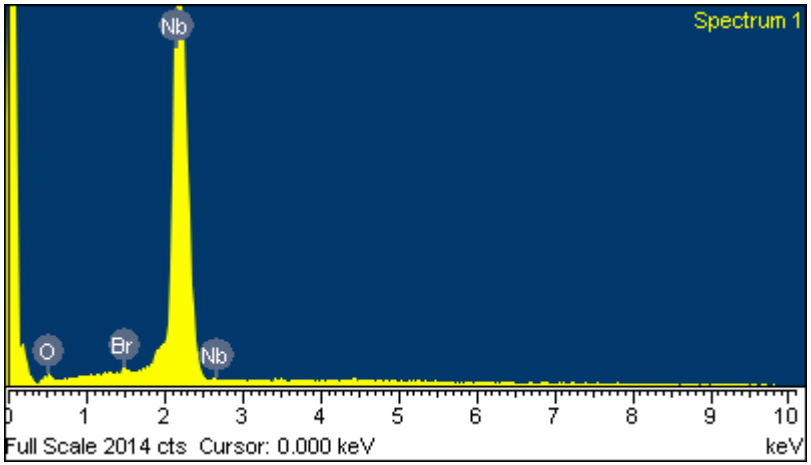
Sample Disc #5
Analysis 8

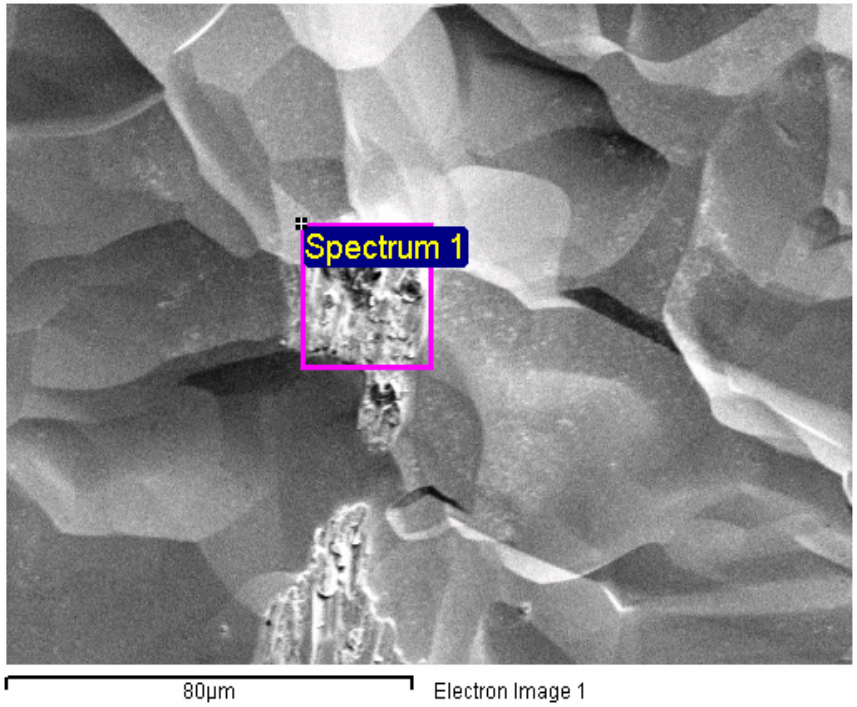
Spectrum processing :
Peak possibly omitted : 3.475 keV

Processing option : All elements analyzed (Normalised)
Number of iterations = 3

Standard :
O SiO2 1-Jun-1999 12:00 AM
Br KBr 1-Jun-1999 12:00 AM
Nb Nb 1-Jun-1999 12:00 AM

Element	Weight%	Atomic%
O K	4.45	21.27
Br L	1.03	0.98
Nb L	94.52	77.75
Totals	100.00	





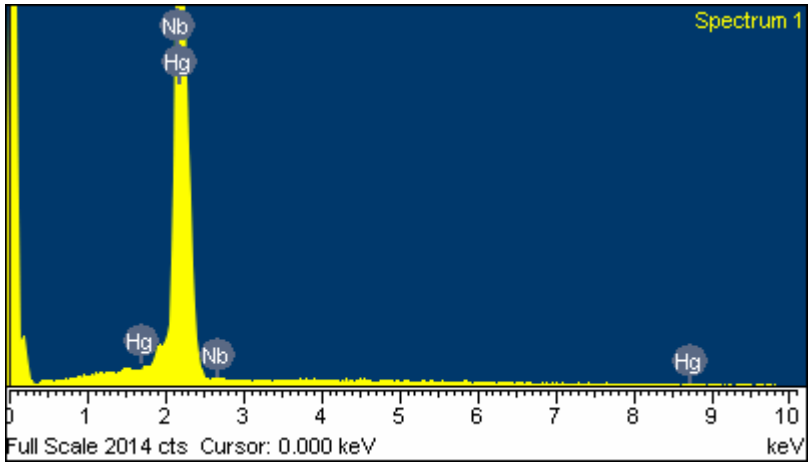
Sample Disc #5
Analysis 9

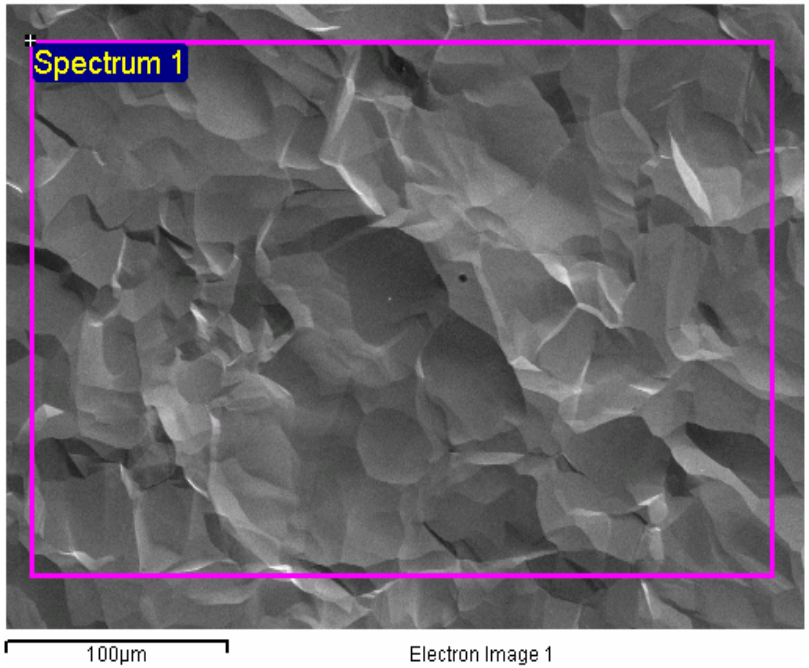
Spectrum processing :
No peaks omitted

Processing option : All elements analyzed (Normalised)
Number of iterations = 2

Standard :
Nb Nb 1-Jun-1999 12:00 AM
Hg HgTe 1-Jun-1999 12:00 AM

Element	Weight%	Atomic%
Nb L	86.60	93.31
Hg M	13.40	6.69
Totals	100.00	





Appendix C – Small IB4 Oven

Sample Disc #7
Analysis 1

Pure Nb Disc #7

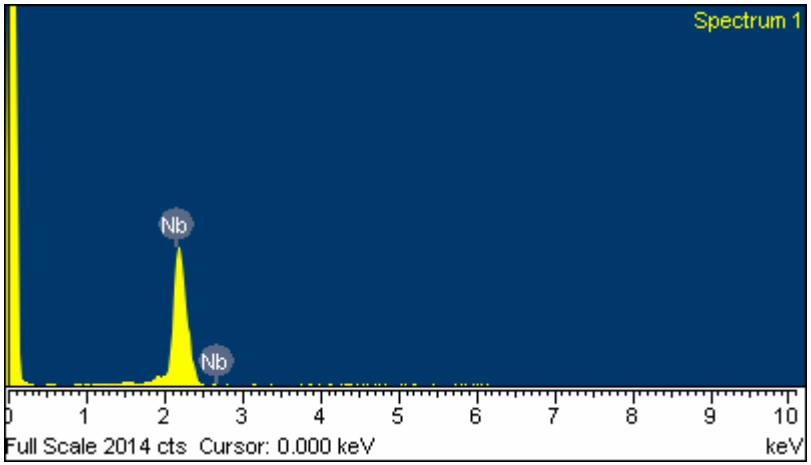
4/1/2005 11:55:32 AM

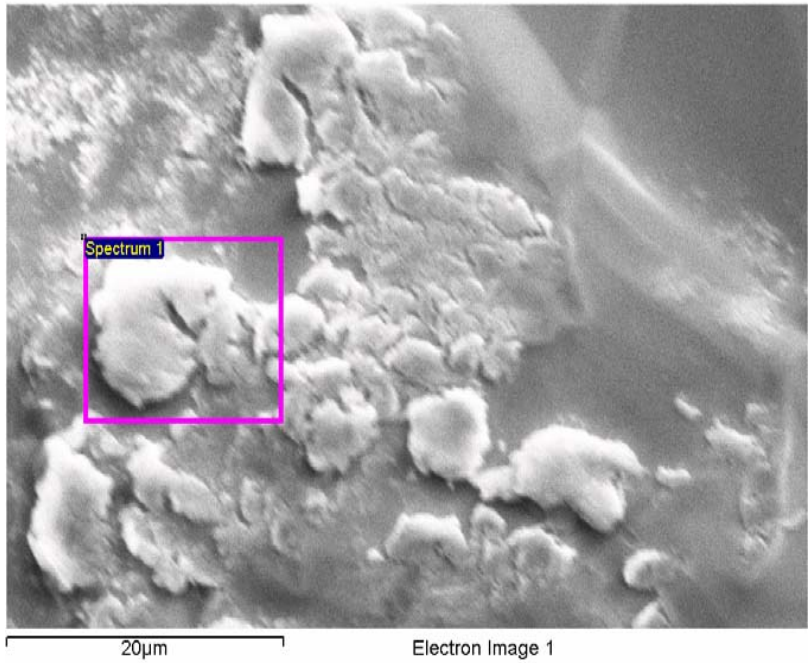
Spectrum processing :
No peaks omitted

Processing option : All elements analyzed (Normalised)
Number of iterations = 1

Standard :
Nb Nb 1-Jun-1999 12:00 AM

Element	Weight%	Atomic%
Nb L	100.00	100.00
Totals	100.00	





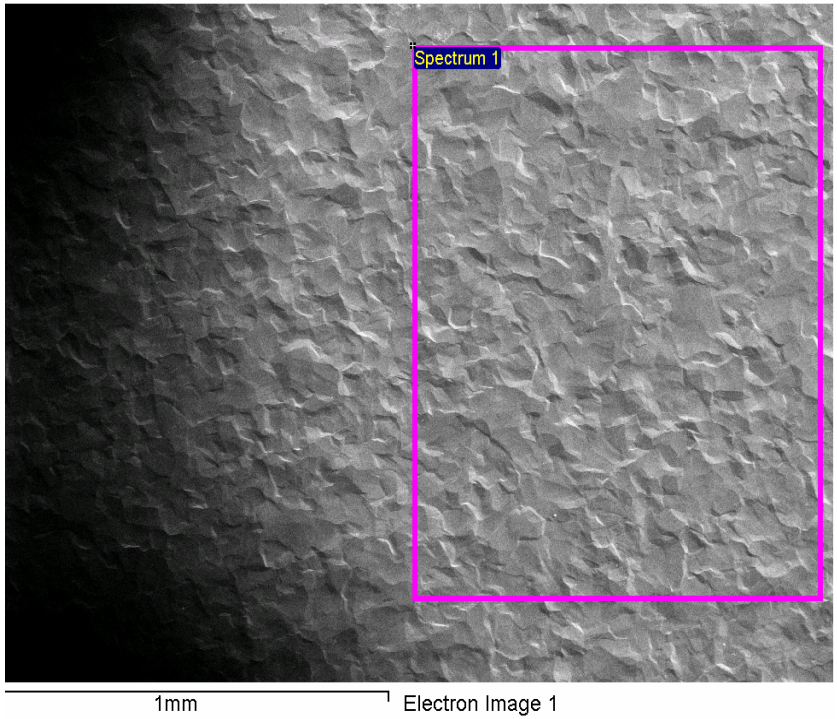
Sample Disc #7
Analysis 2

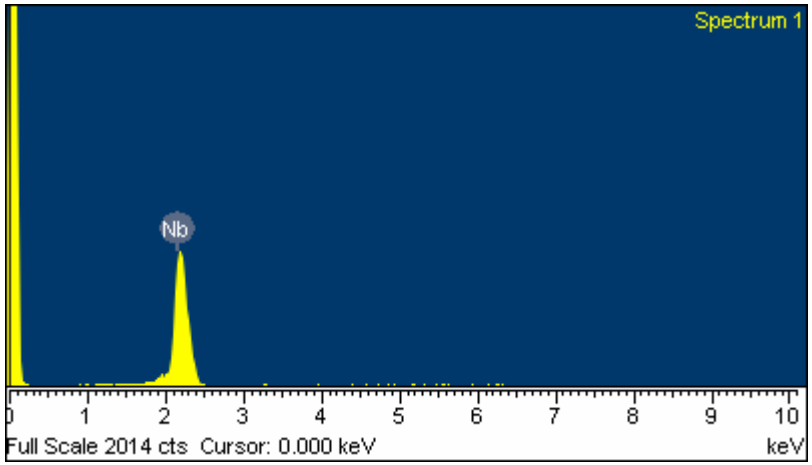
Spectrum processing :
No peaks omitted

Processing option : All elements analyzed (Normalised)
Number of iterations = 1

Standard :
Nb Nb 1-Jun-1999 12:00 AM

Element	Weight%	Atomic%
Nb L	100.00	100.00
Totals	100.00	





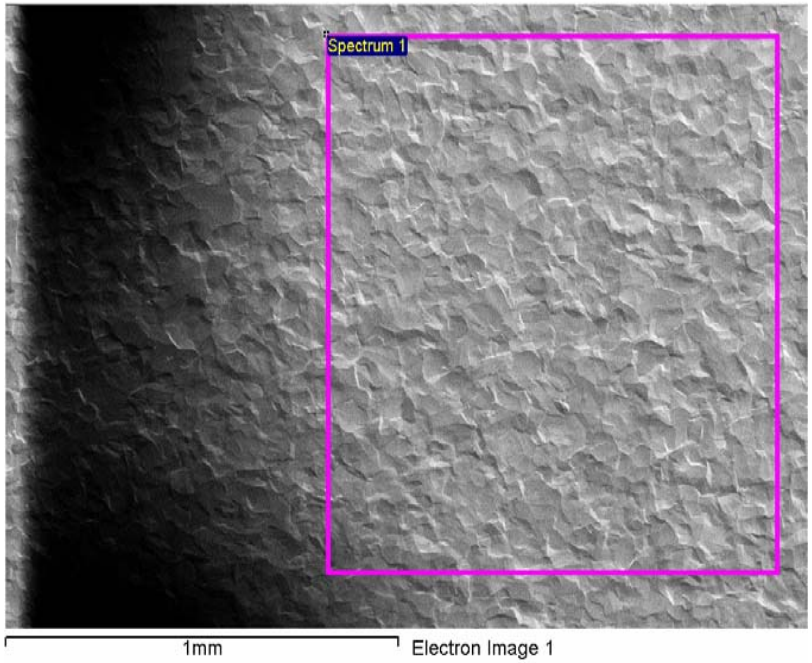
Sample Disc #7
Analysis 3

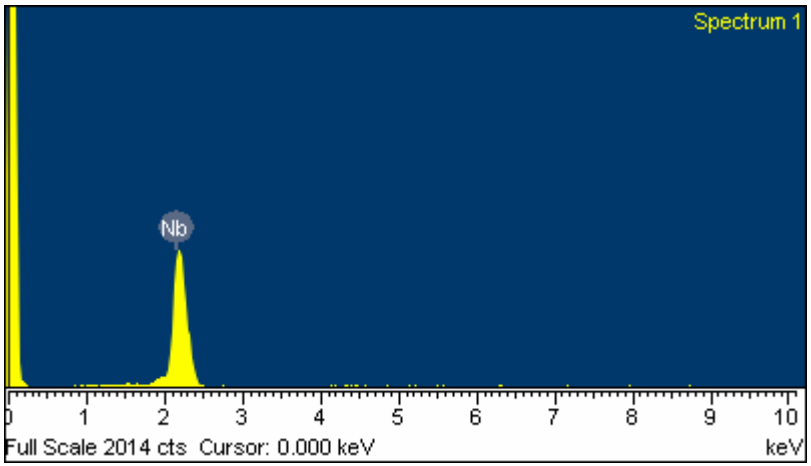
Spectrum processing :
No peaks omitted

Processing option : All elements analyzed (Normalised)
Number of iterations = 1

Standard :
Nb Nb 1-Jun-1999 12:00 AM

Element	Weight%	Atomic%
Nb L	100.00	100.00
Totals	100.00	





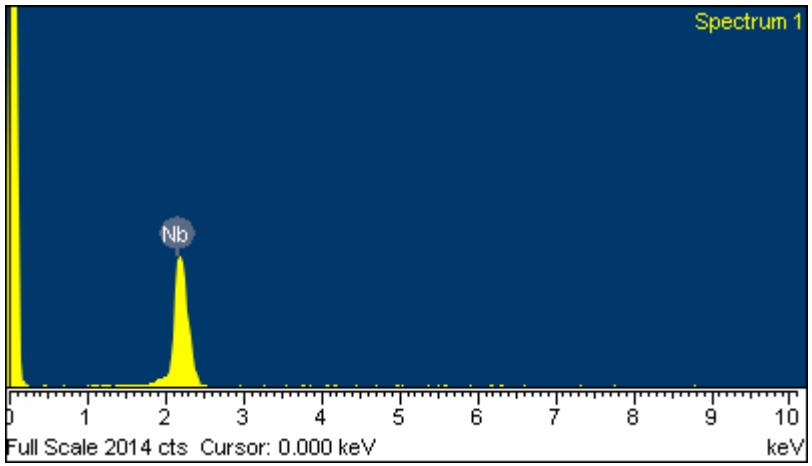
Sample Disc #7
Analysis 4

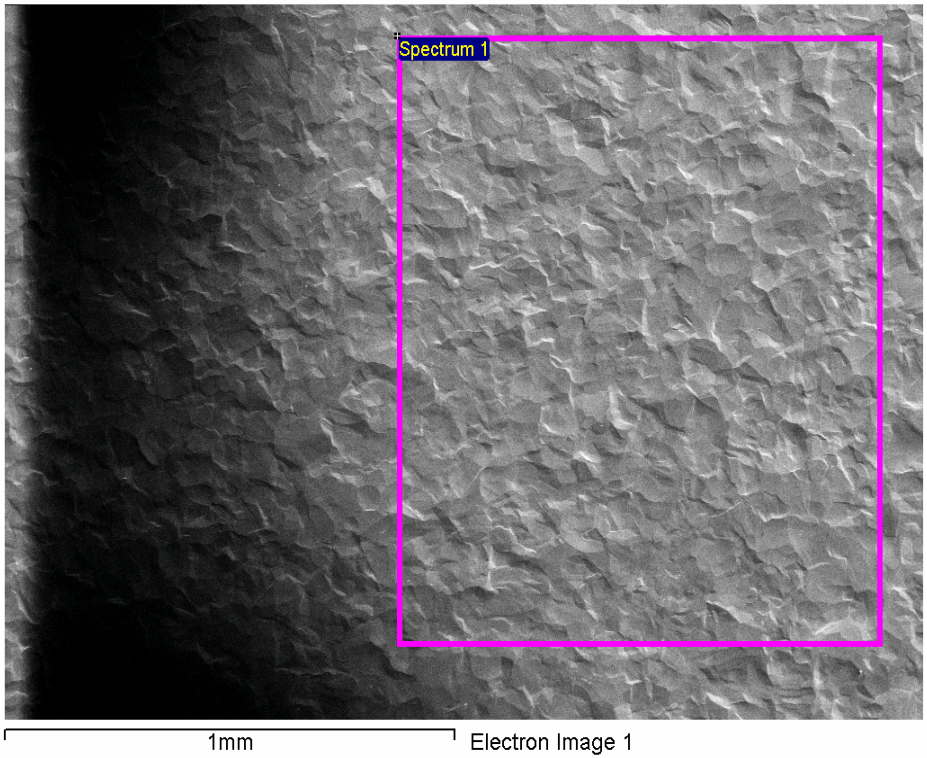
Spectrum processing :
No peaks omitted

Processing option : All elements analyzed (Normalised)
Number of iterations = 1

Standard :
Nb Nb 1-Jun-1999 12:00 AM

Element	Weight%	Atomic%
Nb L	100.00	100.00
Totals	100.00	





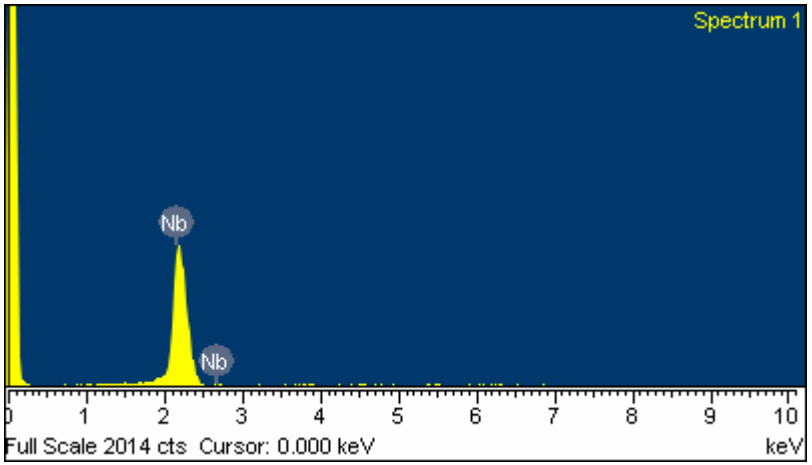
Sample Disc #7
Analysis 5

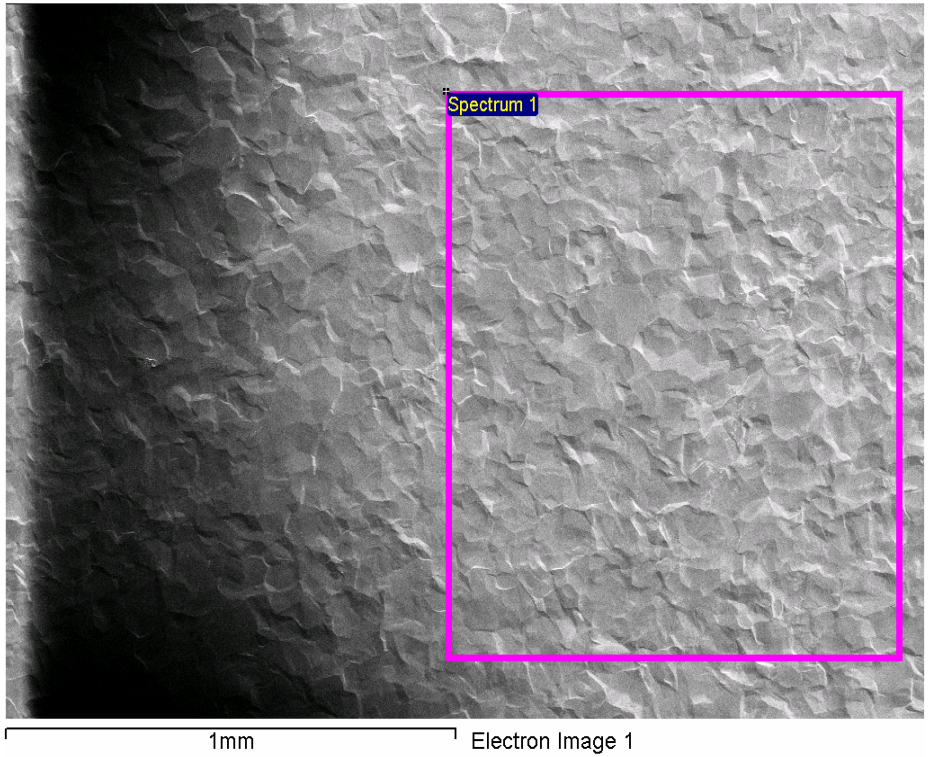
Spectrum processing :
Peak possibly omitted : 4.535 keV

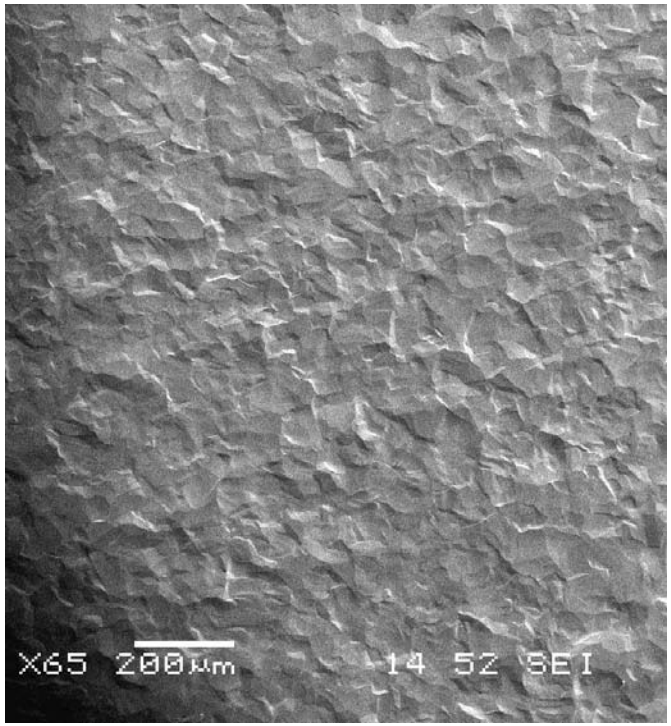
Processing option : All elements analyzed (Normalised)
Number of iterations = 1

Standard :
Nb Nb 1-Jun-1999 12:00 AM

Element	Weight%	Atomic%
Nb L	100.00	100.00
Totals	100.00	






X65 200µm 14 52 SEI

Appendix D – Lab 7 Oven

Sample Disc #9
Analysis 1

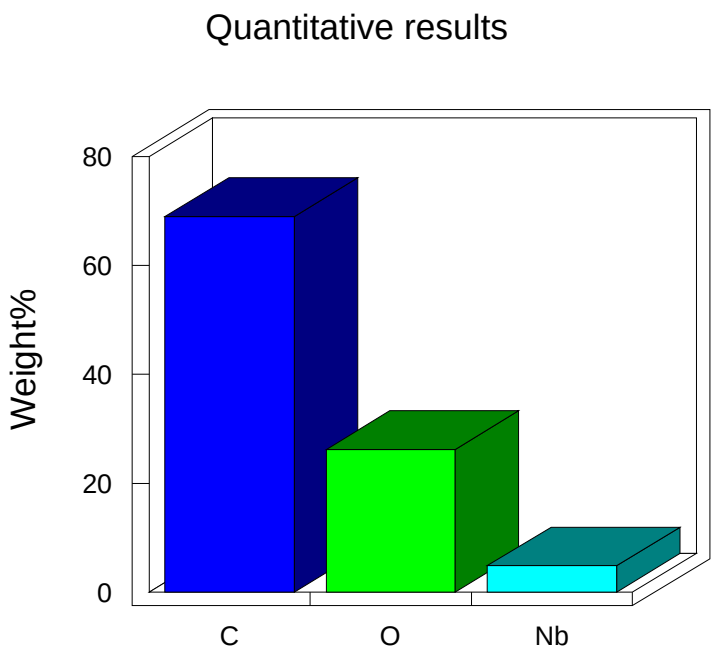
Pure Nb Disc #9

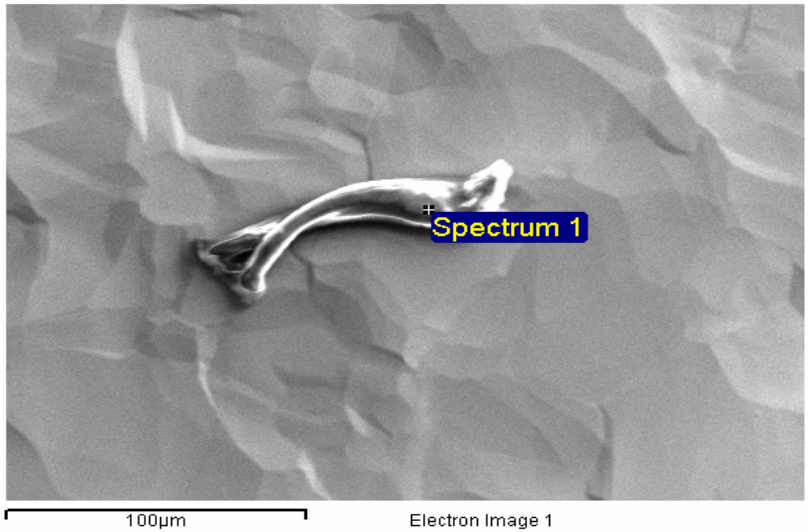
4/8/2005 1:05:16 PM

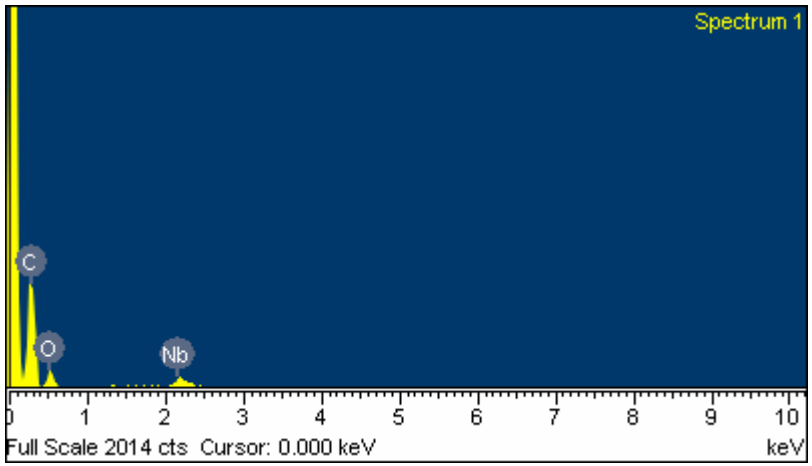
Spectrum processing :
No peaks omitted

Processing option : All elements analyzed (Normalised)
Number of iterations = 5

Standard :
C CaCO3 1-Jun-1999 12:00 AM







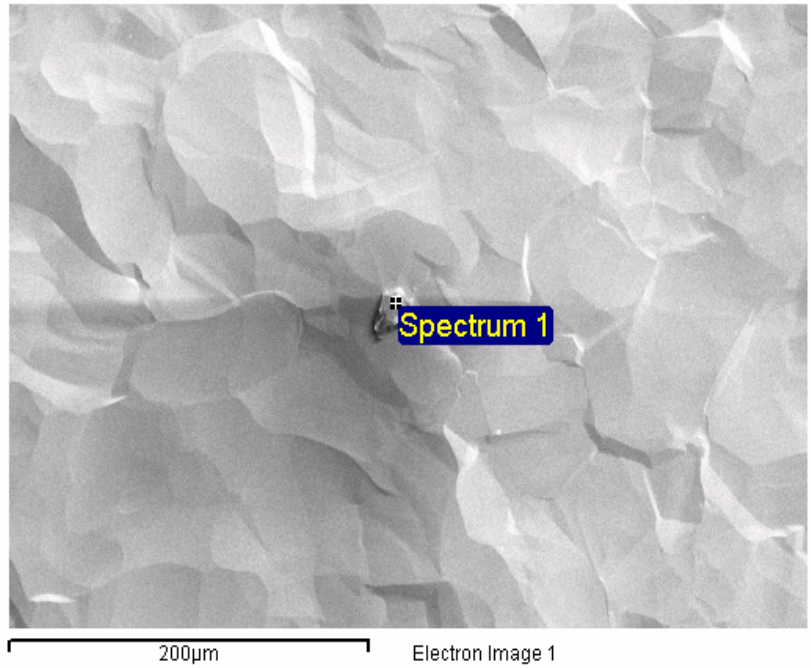
Sample Disc #9
Analysis 2

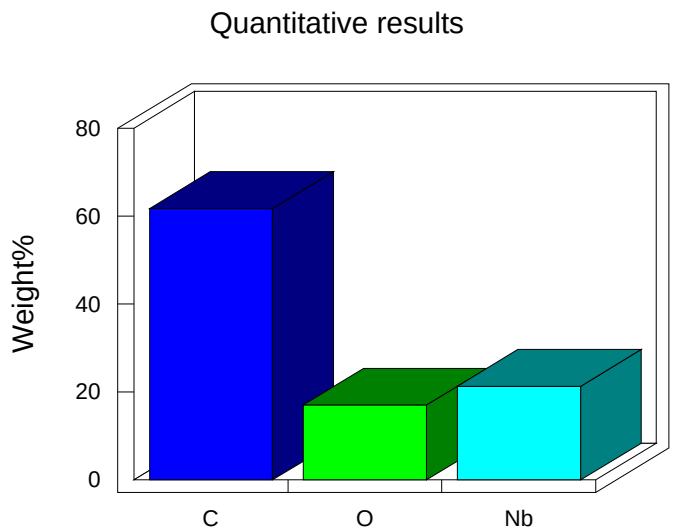
Spectrum processing :
No peaks omitted

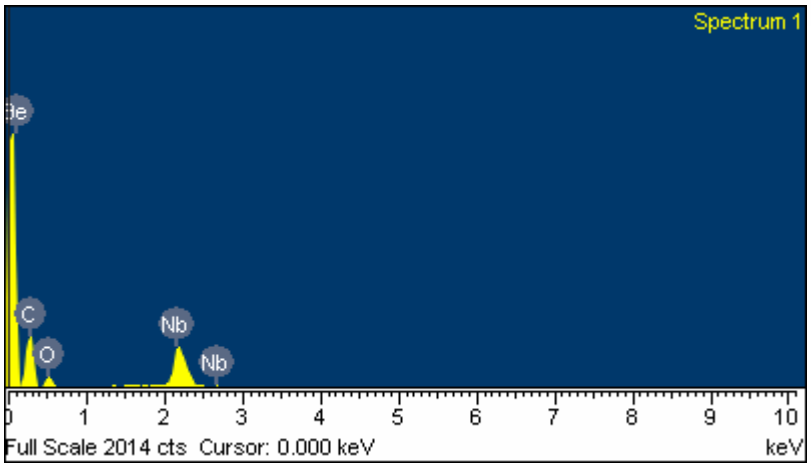
Processing option : All elements analyzed (Normalised)
Number of iterations = 5

Standard :
C CaCO3 1-Jun-1999 12:00 AM
O SiO2 1-Jun-1999 12:00 AM
Nb Nb 1-Jun-1999 12:00 AM

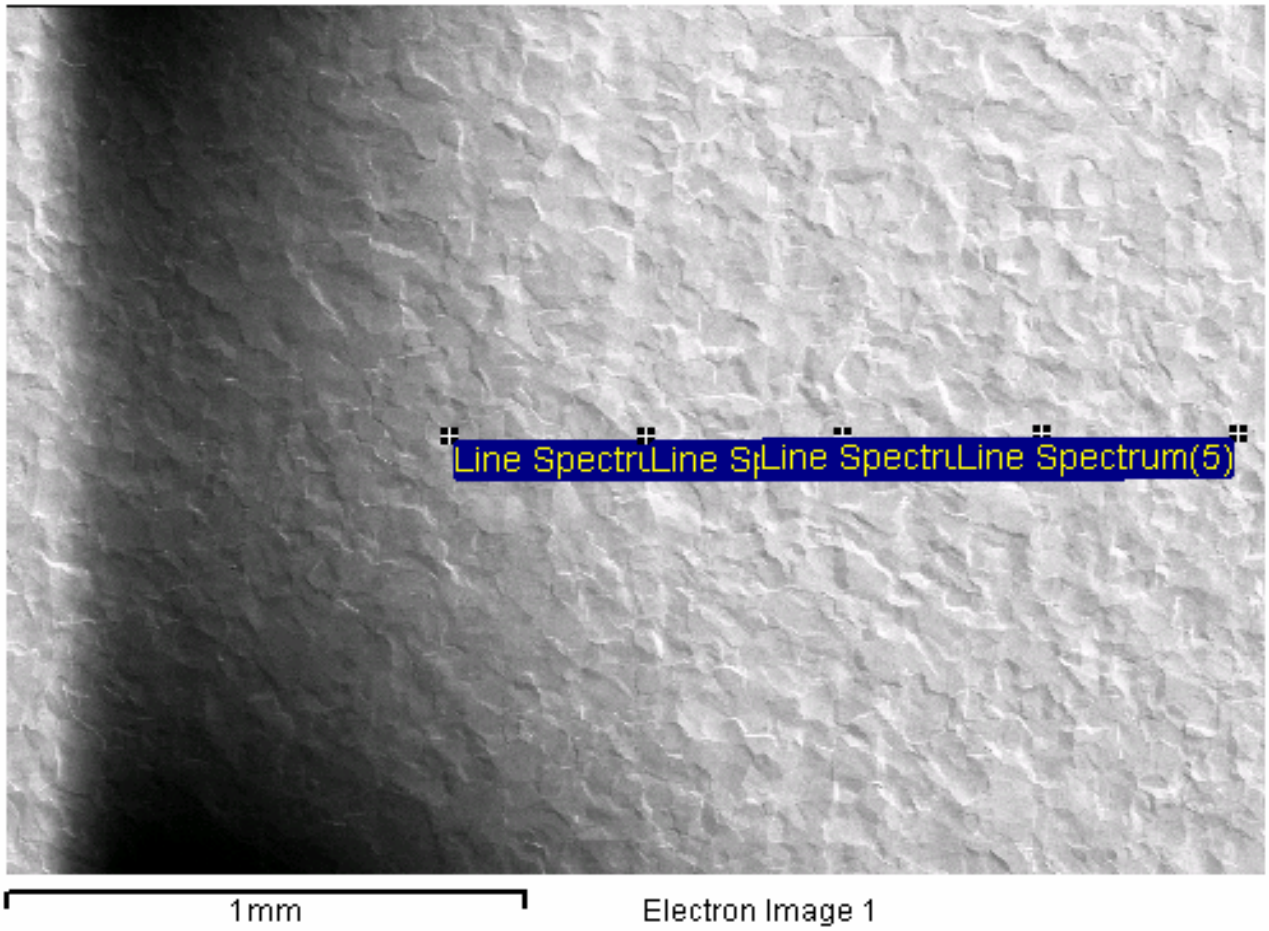
Element	Weight%	Atomic%
C K	61.72	79.93
O K	16.98	16.51
Nb L	21.30	3.57
Totals	100.00	







Sample Disc #9
Analysis 3

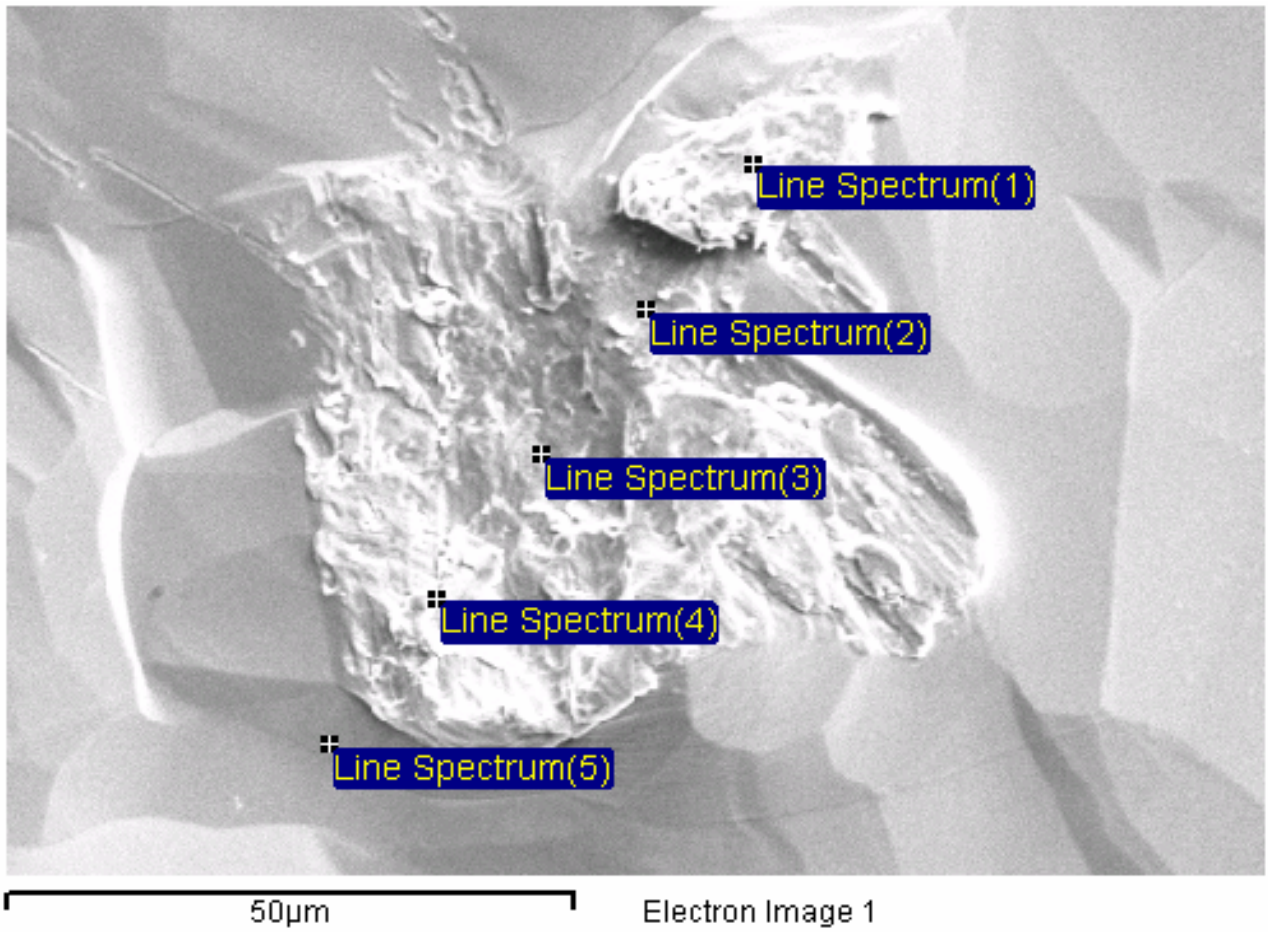


Processing option : All elements analysed (Normalised)

Spectrum	In stats.	Nb	Total
Line Spectrum(1)	Yes	100.00	100.00
Line Spectrum(2)	Yes	100.00	100.00
Line Spectrum(3)	Yes	100.00	100.00
Line Spectrum(4)	Yes	100.00	100.00
Line Spectrum(5)	Yes	100.00	100.00
Mean		100.00	100.00
Std. deviation		0.00	
Max.		100.00	
Min.		100.00	

All results in weight%

Sample Disc #9
Analysis 4

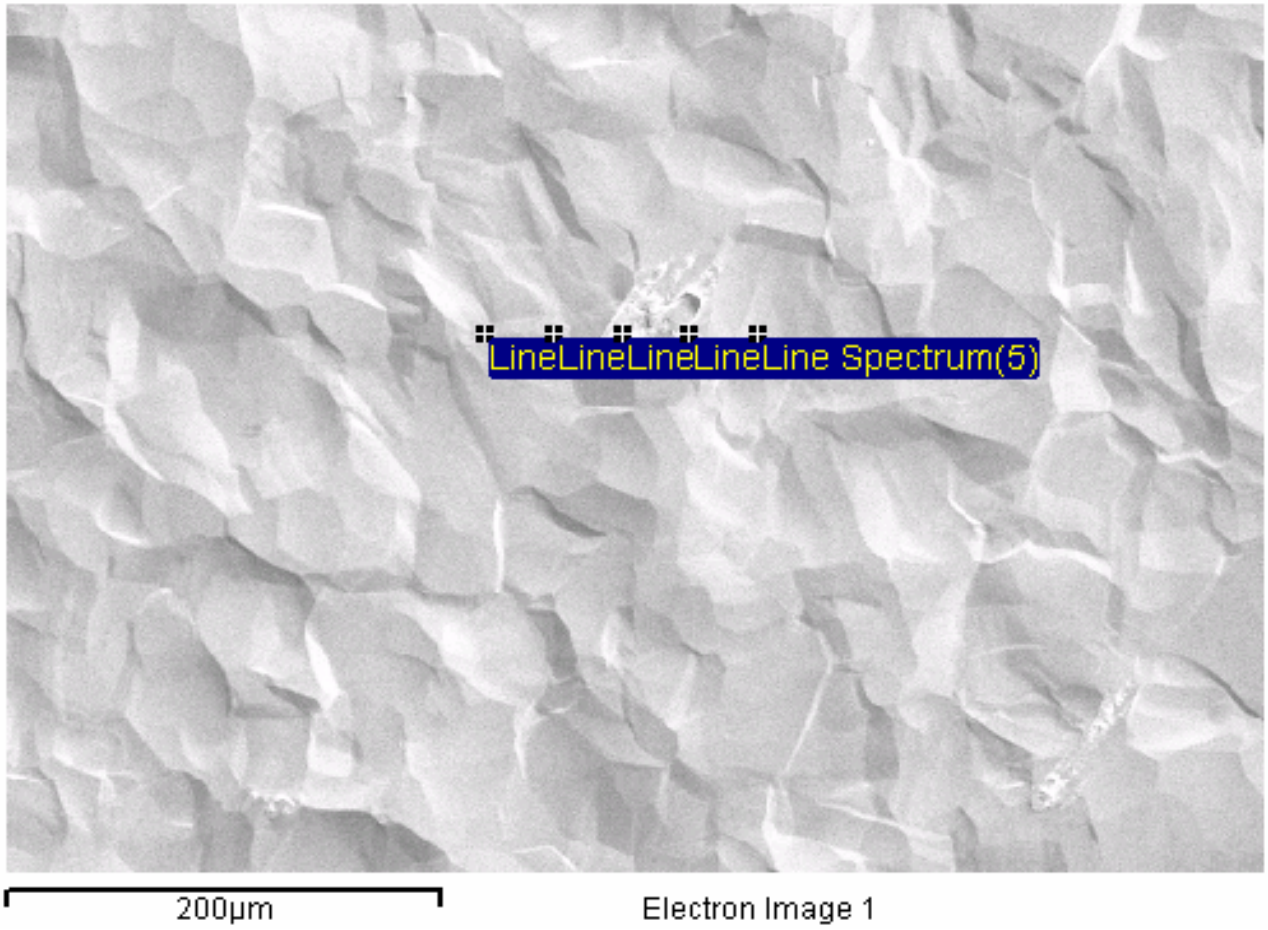


Processing option : All elements analysed (Normalised)

Spectrum	In stats.	Cu	Nb	Total
Line Spectrum(1)	Yes	-2.35	102.35	100.00
Line Spectrum(2)	Yes	0.45	99.55	100.00
Line Spectrum(3)	Yes	-0.25	100.25	100.00
Line Spectrum(4)	Yes	-0.92	100.92	100.00
Line Spectrum(5)	Yes	1.22	98.78	100.00
Mean		-0.37	100.37	100.00
Std. deviation		1.36	1.36	
Max.		1.22	102.35	
Min.		-2.35	98.78	

All results in weight%

Sample Disc #9
Analysis 5



Processing option : All elements analysed (Normalised)

Spectrum	In stats.	Cu	Nb	Total
Line Spectrum(1)	Yes	1.97	98.03	100.00
Line Spectrum(2)	Yes	0.79	99.21	100.00
Line Spectrum(3)	Yes	-1.65	101.65	100.00
Line Spectrum(4)	Yes	0.76	99.24	100.00
Line Spectrum(5)	Yes	-0.94	100.94	100.00
Mean		0.19	99.81	100.00
Std. deviation		1.46	1.46	
Max.		1.97	101.65	
Min.		-1.65	98.03	

All results in weight%

Label : Line Spectrum(1)
Collected : 8-Apr-2005 01:10 PM
Livetime (s) : 45.00
Real time (s) : 47.03
Detector : Silicon
Window: SATW

Tilt (deg) : 0.0
Elevation (deg) : 25.0
Azimuth (deg) : 0.0

Magnification: 230 X
Accelerating voltage (kV) : 15.00
Process time : 5

Spectrum processing:
No peaks omitted

Processing option : All elements analyzed (Normalized)
Number of iterations = 2

Standard :
Cu Cu 1-Jun-1999 12:00 AM
Nb Nb 1-Jun-1999 12:00 AM

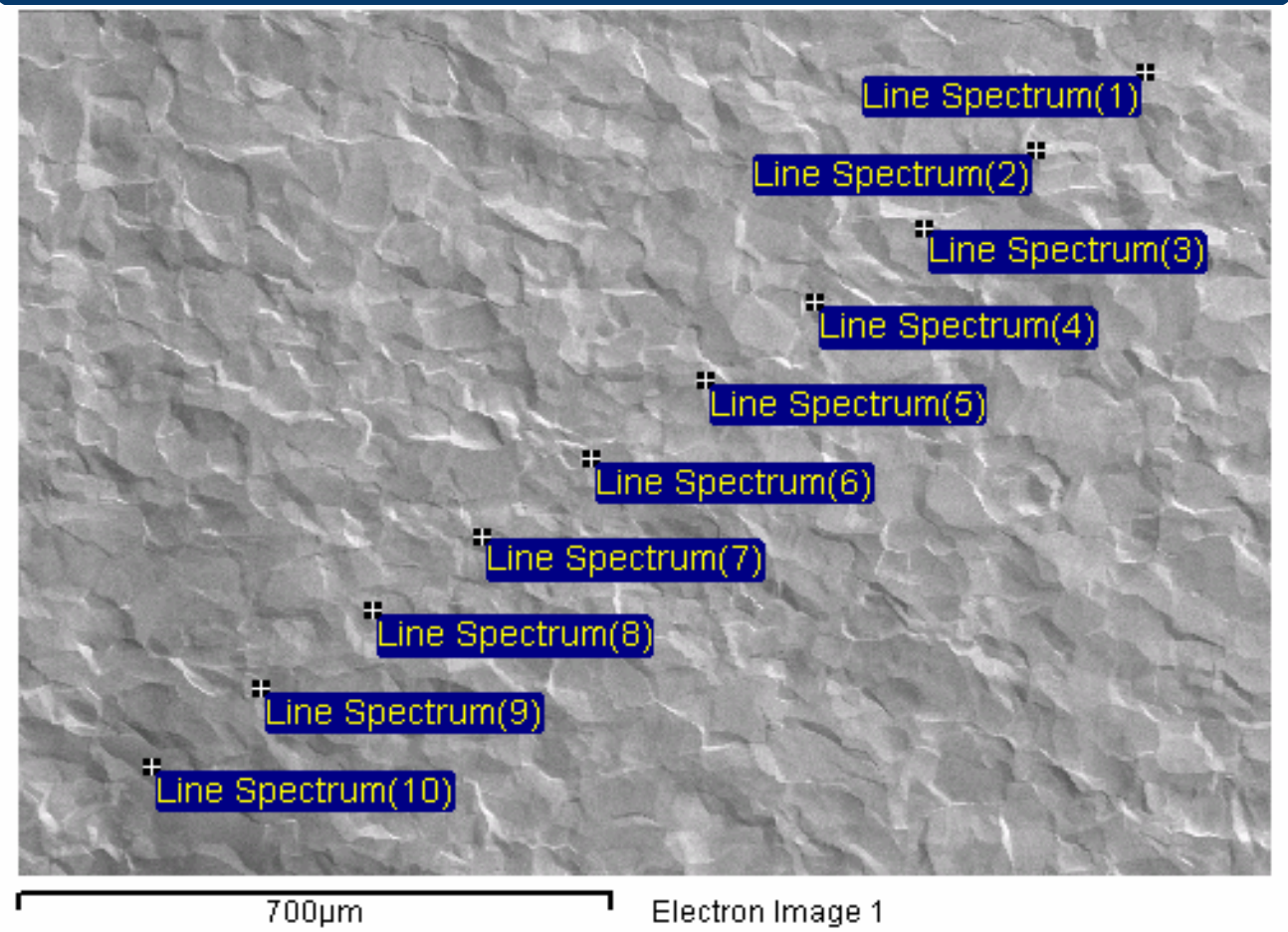
Element	App Conc.	Intensity Corrn.	Weight%	Weight% Sigma	Atomic%
Cu L	0.46	0.4549	1.97	1.27	2.85
Nb L	49.32	0.9899	98.03	1.27	97.15
Totals			100.00		

Appendix E – Lab 7 Oven

Sample Disc #10
Analysis 1

Pure Nb Disc #10

4/11/2005 1:21:53 PM

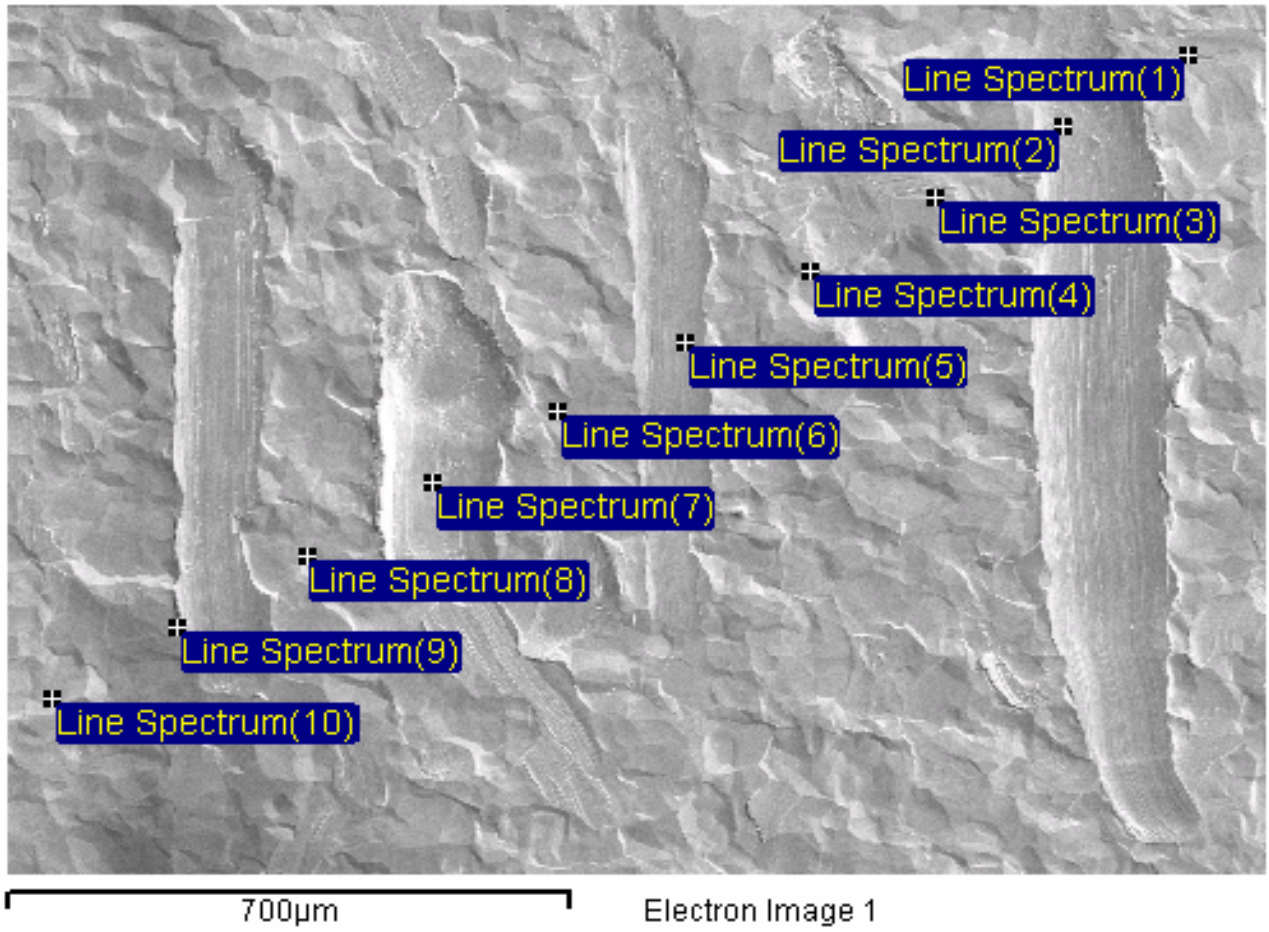


Processing option : All elements analysed (Normalised)

Spectrum	In stats.	B	Nb	Total
Line Spectrum(1)	Yes		100.00	100.00
Line Spectrum(2)	Yes	14.74	85.26	100.00
Line Spectrum(3)	Yes		100.00	100.00
Line Spectrum(4)	Yes		100.00	100.00
Line Spectrum(5)	Yes		100.00	100.00
Line Spectrum(6)	Yes		100.00	100.00
Line Spectrum(7)	Yes		100.00	100.00
Line Spectrum(8)	Yes		100.00	100.00
Line Spectrum(9)	Yes		100.00	100.00
Line Spectrum(10)	Yes		100.00	100.00
Max.		14.74	100.00	
Min.		0.00	85.26	

All results in weight%

Sample Disc #10
Analysis 2



Processing option : All elements analyzed (Normalized)

Spectrum	In stats.	C	O	Nb	Total
Line Spectrum(1)	Yes			100.00	100.00
Line Spectrum(2)	Yes			100.00	100.00
Line Spectrum(3)	Yes			100.00	100.00
Line Spectrum(4)	Yes			100.00	100.00
Line Spectrum(5)	Yes			100.00	100.00
Line Spectrum(6)	Yes			100.00	100.00
Line Spectrum(7)	Yes	23.55	6.33	70.12	100.00
Line Spectrum(8)	Yes			100.00	100.00
Line Spectrum(9)	Yes			100.00	100.00
Line Spectrum(10)	Yes			100.00	100.00
Max.		23.55	6.33	100.00	
Min.		0.00	0.00	70.12	

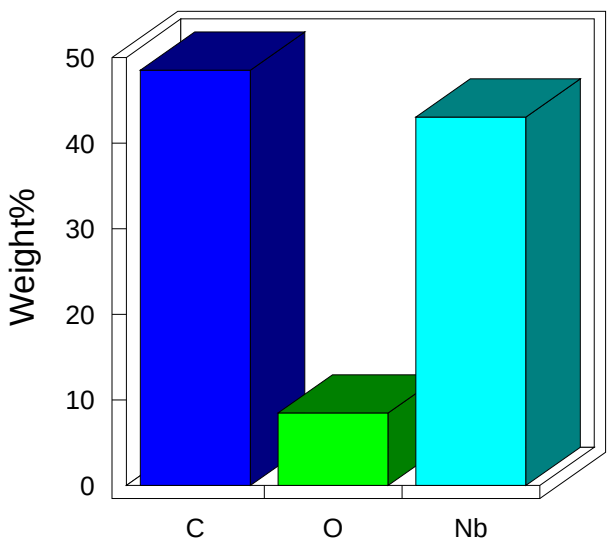
All results in weight%

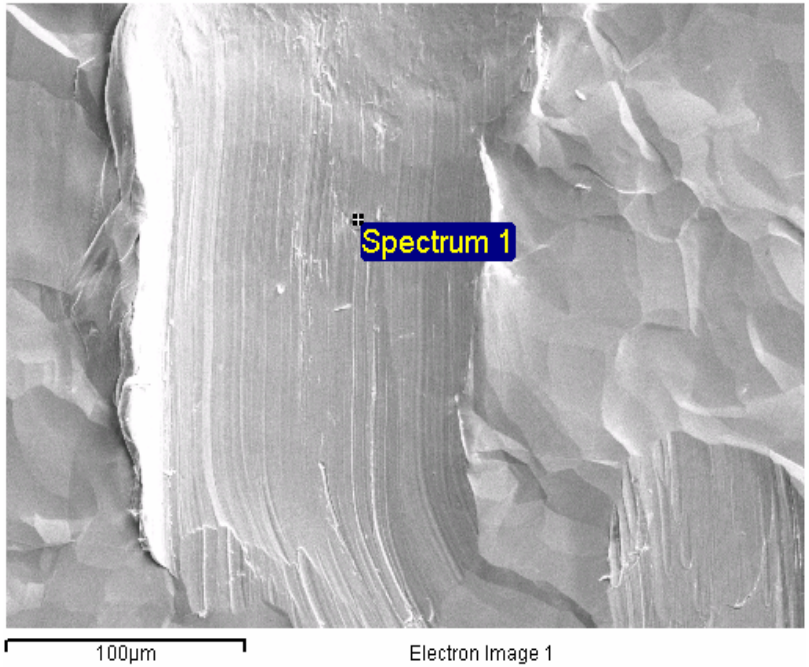
Sample Disc #10
Analysis 3

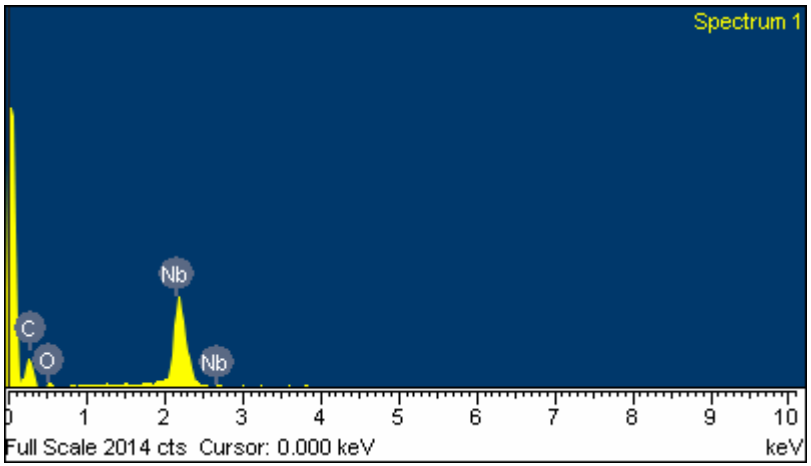
Spectrum processing :
No peaks omitted
Processing option : All elements analyzed (Normalised)
Number of iterations = 4
Standard :
C CaCO3 1-Jun-1999 12:00 AM
O SiO2 1-Jun-1999 12:00 AM
Nb Nb 1-Jun-1999 12:00 AM

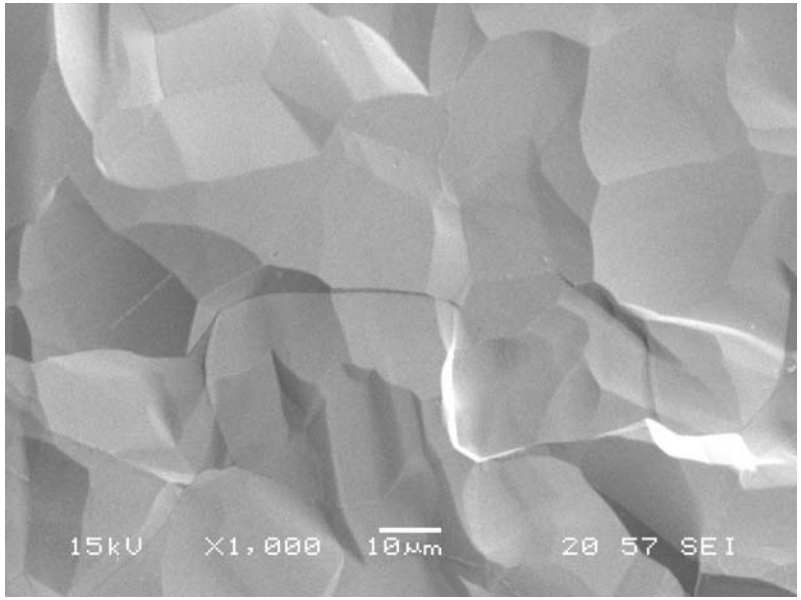
Element	Weight%	Atomic%
C K	48.52	80.29
O K	8.46	10.51
Nb L	43.02	9.20
Totals	100.00	

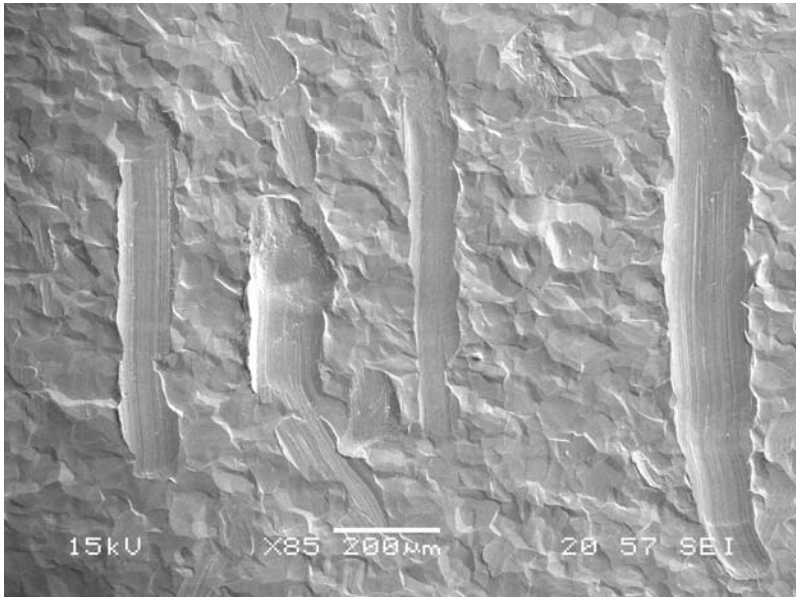
Quantitative results

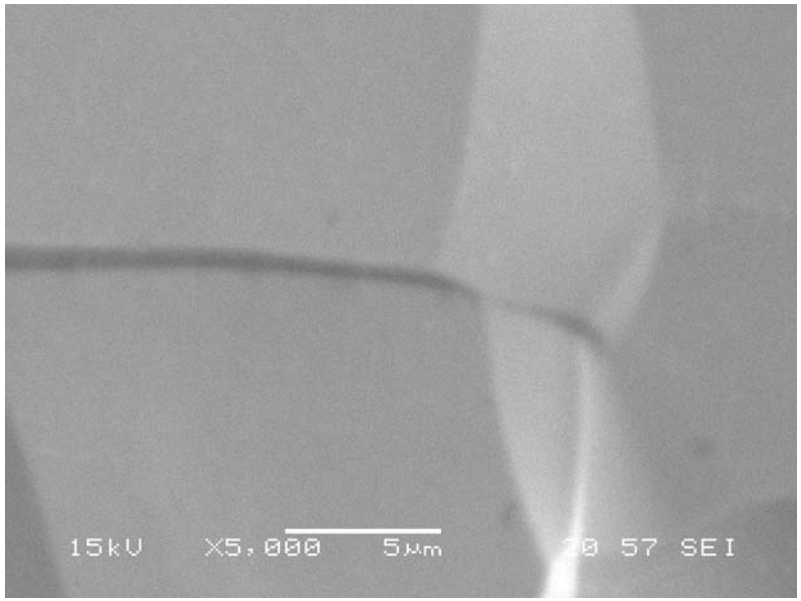


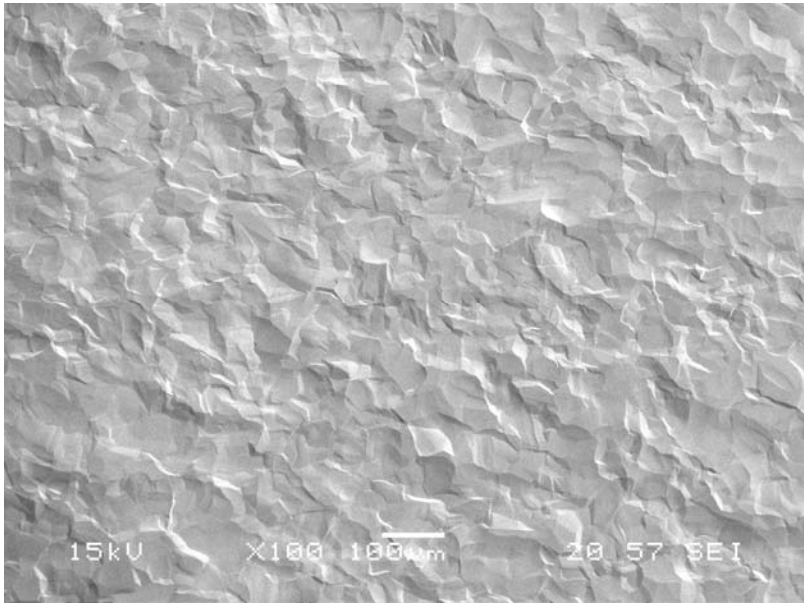


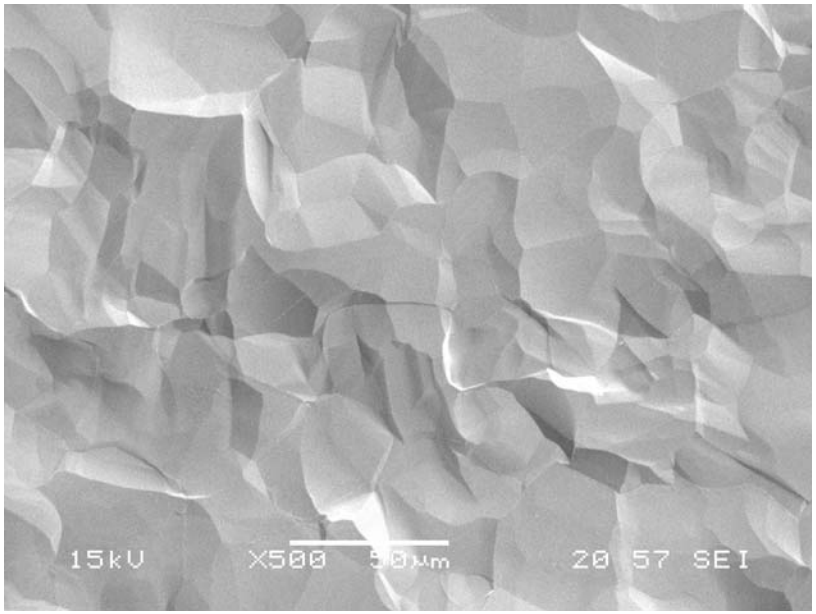


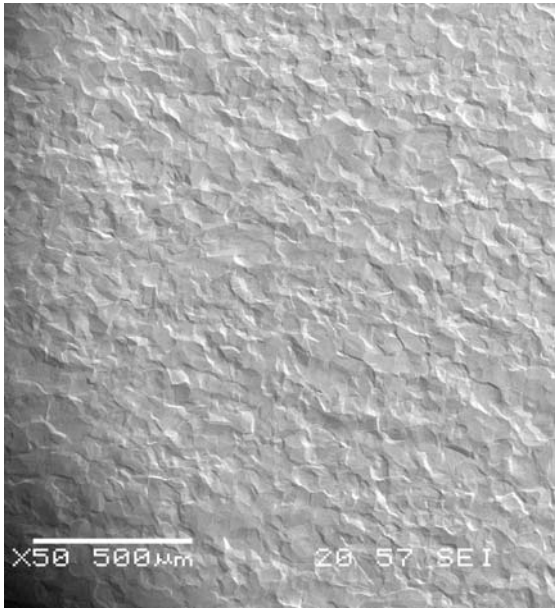

15kV X1,000 10µm 20 57 SEI


15kV X85 200µm 20 57 SEI


15kV X5,000 5µm 20 57 SEI


15kV X100 100µm 20 57 SEI


15kV X500 50µm 20 57 SEI


X50 500µm 20 57 SEI

Appendix F - XPS

Materials Characterization

Job ID	1031	Date In	1/24/2005
		Date Out	2/4/2005
Customer	T. Arkan, FermiLab	Phone	
Account#	1015346	M/S	
Analyst	R. Kirby		

Images

Chemistry XPS

Other

Submission

Material: Nb witness coupons, from vacuum furnaces. Coupons BCP-etched, then baked at 800C for 5 hrs under vacuum.

History: Check suitability of NLC for use with Nb cavity bakeout.

Purpose of: Surface analysis.
Analysis

Sample/Mount #	Spectrum #
	5753-57

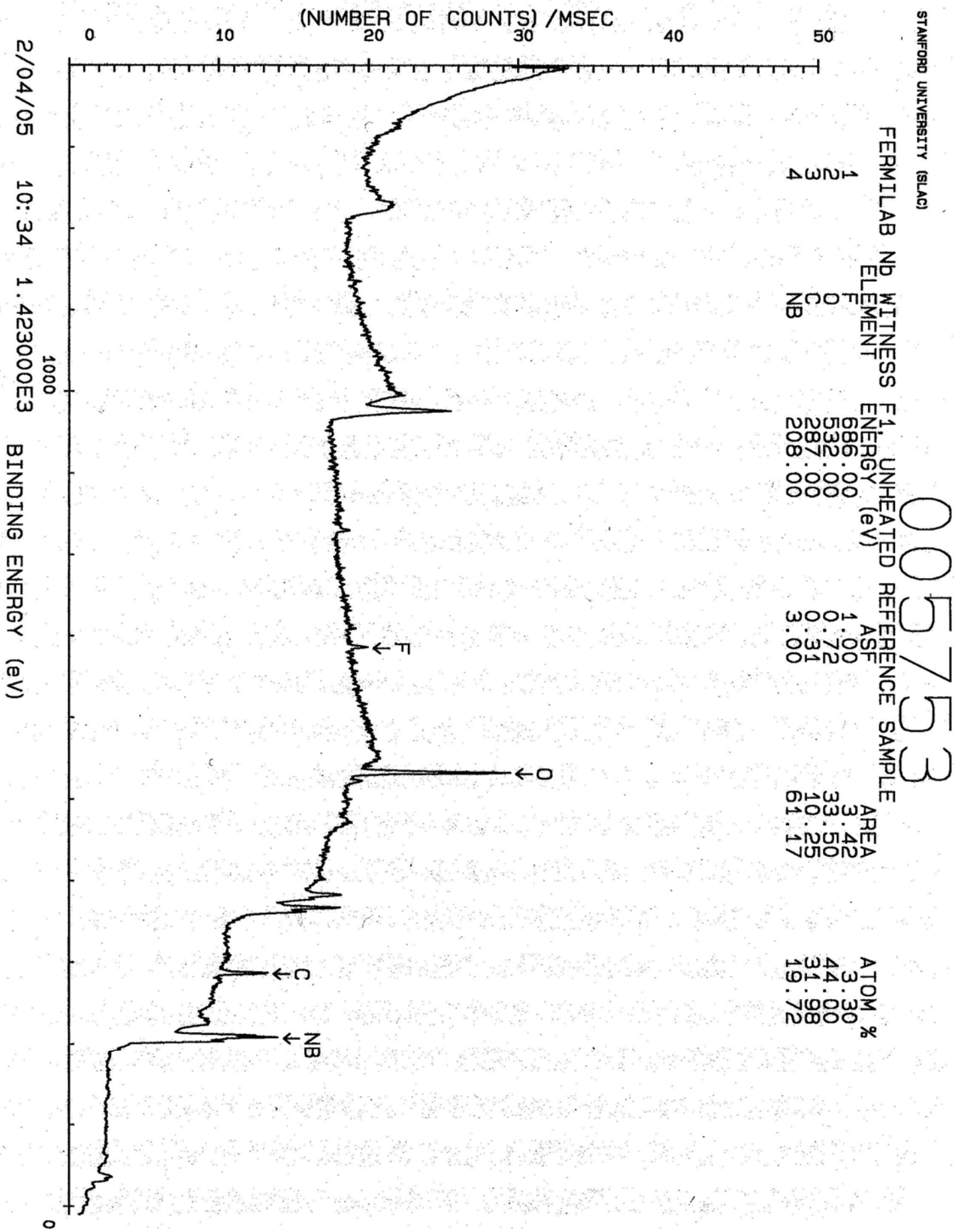
Details of Analysis

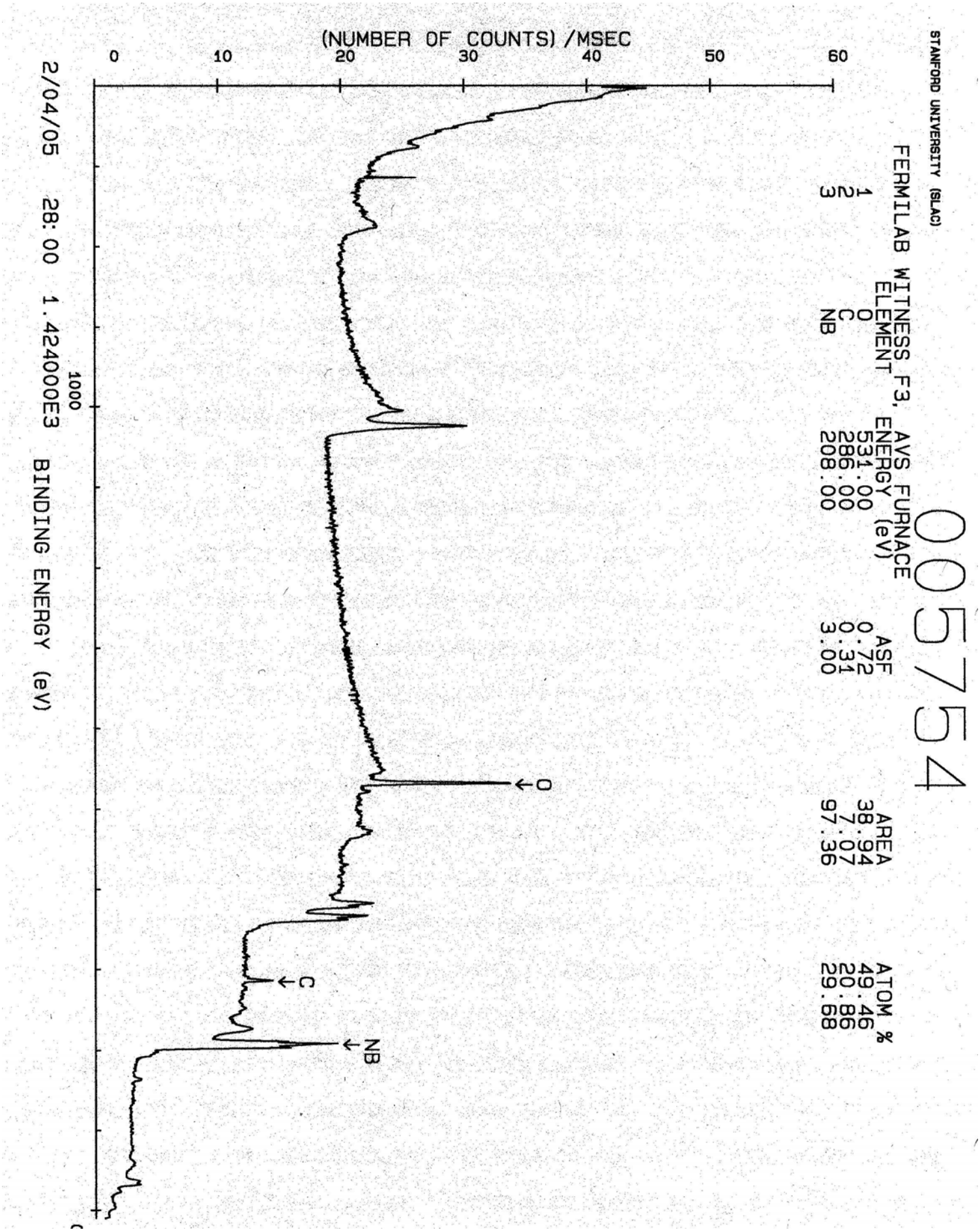
Use x-ray photoelectron spectroscopy (XPS) to measure surface condition.

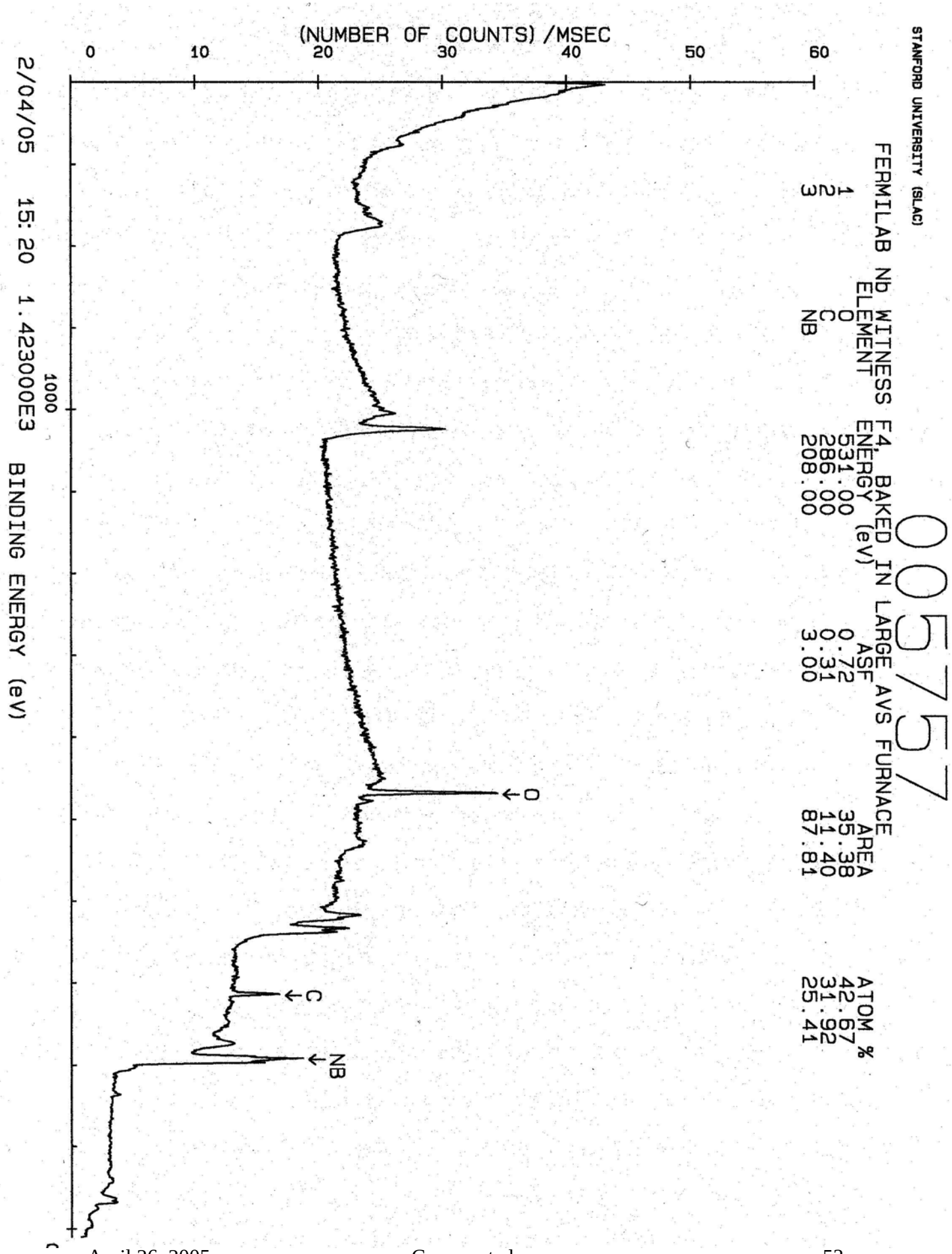
Results of Analysis

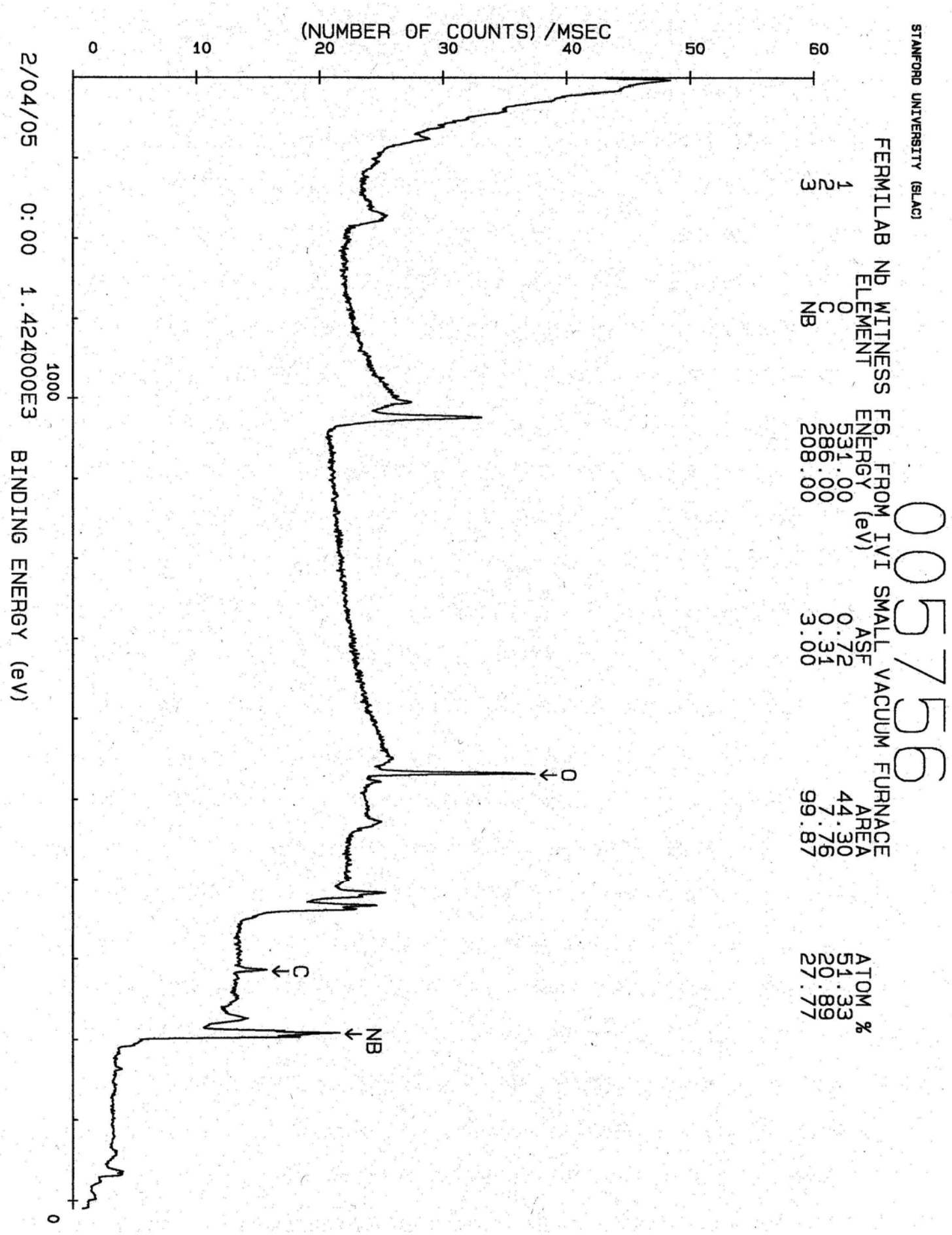
Sample #	Furnace Name	Carbon Conc .(Atom%)	Other Contaminants
F1	Unbaked ref.	32	F(3.3%)
F3	AVS large vac.	21	
F4	“	32	
F6	IVI small vac.	21	
F8	Lab 7	19	Cu(0.5%), Sn(1.4%)

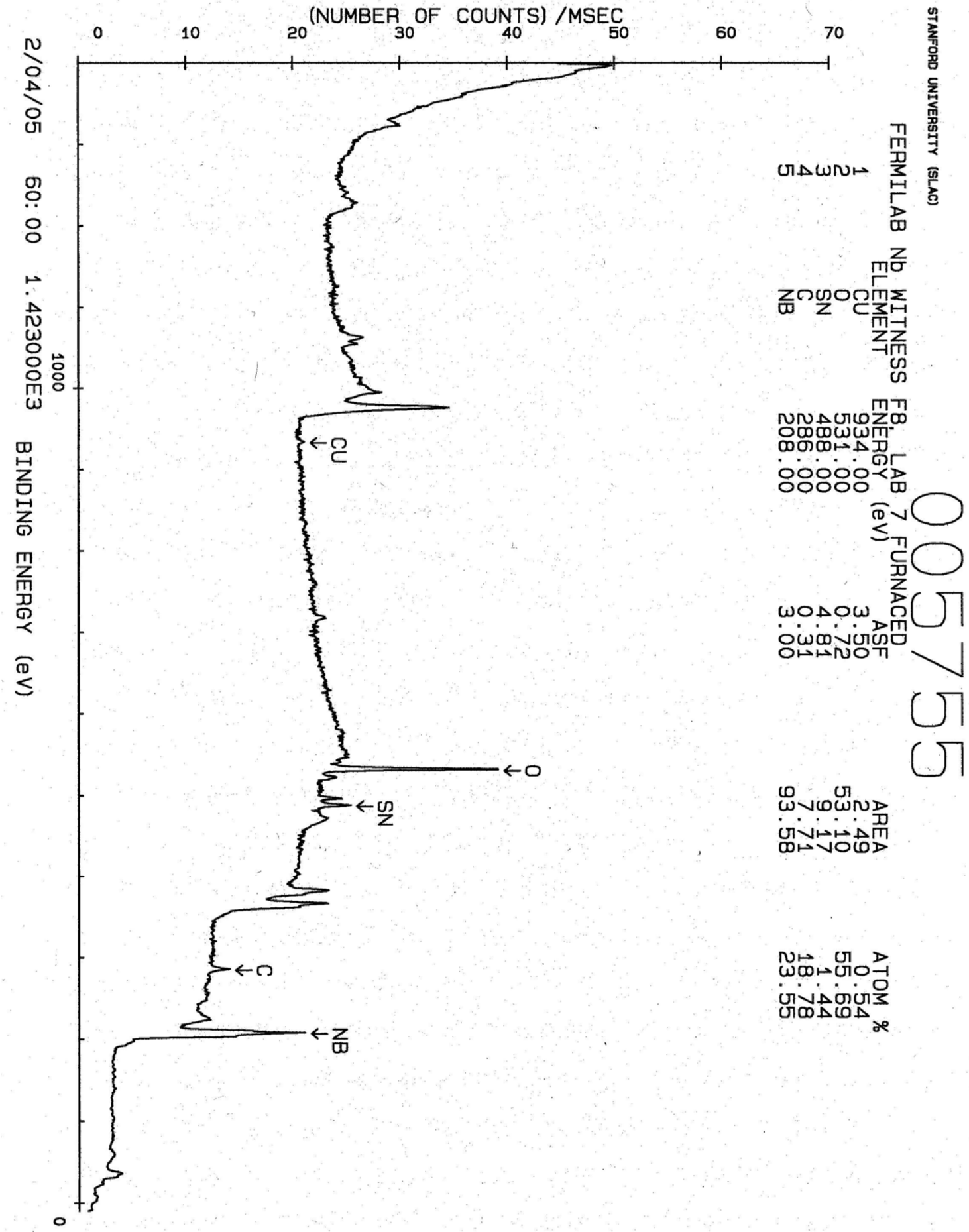
All of these coupons have relatively low carbon on them, given the extreme gettering ability of Nb. The Lab 7 furnace witness has Cu and Sn on it. Also, all witnesses have oxide on them. If the furnace has an air leak, Nb witnesses will not be the way to detect it. Finally, F1 has F (BCP HF acid residue?) on it, which may build up over time in the furnace atmosphere.


STANFORD UNIVERSITY (SLAC)
FERMILAB Nb WITNESS F1 UNHEATED REFERENCE SAMPLE
ELEMENT ENERGY (eV) ASF AREA ATOM %
1 F 686.00 1.00 3.42 3.30
2 O 532.00 0.72 33.50 44.00
3 C 287.00 0.31 10.25 31.98
4 NB 208.00 3.00 61.17 19.72
(NUMBER OF COUNTS) /MSEC
0
10
20
30
40
50
F
O
C
NB
1000
0
2/04/05 10:34 1.423000E3 BINDING ENERGY (eV)


STANFORD UNIVERSITY (SLAC)
FERMILAB WITNESS F3, AVS FURNACE
ELEMENT ENERGY (eV) ASF AREA ATOM %
1 O 531.00 0.72 38.94 49.46
2 C 286.00 0.31 7.07 20.86
3 NB 208.00 3.00 97.36 29.68
00574
(NUMBER OF COUNTS) /MSEC
0 10 20 30 40 50 60
O
C
NB
1000
2/04/05 28:00 1.424000E3 BINDING ENERGY (eV)


STANFORD UNIVERSITY (SLAC)
FERMILAB Nb WITNESS F4. BAKED IN LARGE AVS FURNACE
ELEMENT ENERGY (eV) ASF AREA ATOM %
1 O 531.00 0.72 35.38 42.67
2 C 286.00 0.31 11.40 31.92
3 NB 208.00 3.00 87.81 25.41
(NUMBER OF COUNTS) /MSEC
0 10 20 30 40 50 60
O
C
NB
1000
2/04/05 15:20 1.423000E3 BINDING ENERGY (eV)


STANFORD UNIVERSITY (SLAC)
FERMILAB Nb WITNESS F6 FROM IVI SMALL VACUUM FURNACE
ELEMENT ENERGY (eV) ASF AREA ATOM %
1 O 531.00 0.72 44.30 51.33
2 C 286.00 0.31 7.76 20.89
3 NB 208.00 3.00 99.87 27.77
(NUMBER OF COUNTS) /MSEC
0
10
20
30
40
50
60
O
C
NB
1000
0
2/04/05 0:00 1.424000E3 BINDING ENERGY (eV)


STANFORD UNIVERSITY (SLAC)
FERMILAB Nb WITNESS F8 LAB 7 FURNACED
ELEMENT ENERGY (eV) ASF AREA ATOM %
1 CU 934.00 3.50 2.49 0.54
2 O 531.00 0.72 53.10 55.69
3 SN 488.00 4.81 9.17 1.44
4 C 286.00 0.31 7.71 18.78
5 NB 208.00 3.00 93.58 23.55
(NUMBER OF COUNTS) /MSEC
0 10 20 30 40 50 60 70
CU
O
SN
C
NB
0 1000
2/04/05 60:00 1.423000E3 BINDING ENERGY (eV)

Appendix G – Sample Preparation

Sample	Cold Work	De grease	100 min Etch	750C Heat Treat	20 min Etch	Comment
FURNACE COMPARISON STUDY						
F1 - stick	N	Y	BCP	N	N	3R, reference for furnace study
F1 - disc	N	Y	BCP	N	N	SEM, reference for furnace study - SLAC
F2 - stick	N	Y	BCP	N	N	3R, reference for furnace study
F2 - disc	N	Y	BCP	N	N	SEM, reference for furnace study
F3 - stick	N	Y	BCP	Y	N	3R, heat treated in IB4 - large oven
F3 - disc	N	Y	BCP	Y	N	SEM, heat treated in IB4 - large oven - SLAC
F4 - stick	N	Y	BCP	Y	N	3R, heat treated in IB4 - large oven
F4 - disc	N	Y	BCP	Y	N	SEM, heat treated in IB4 - large oven - SLAC
F5 - stick	N	Y	BCP	Y	N	3R, heat treated in IB4 - large oven
F5 - disc	N	Y	BCP	Y	N	SEM, heat treated in IB4 - large oven
F6 - stick	N	Y	BCP	Y	N	3R, heat treated in IB4 - small oven
F6 - disc	N	Y	BCP	Y	N	SEM, heat treated in IB4 - small oven - SLAC
F7 - stick	N	Y	BCP	Y	N	3R, heat treated in IB4 - small oven
F7 - disc	N	Y	BCP	Y	N	SEM, heat treated in IB4 - small oven
F8 - stick	N	Y	BCP	Y	N	3R, heat treated in Lab 7 oven
F8 - disc	N	Y	BCP	Y	N	SEM, heat treated in Lab 7 oven - SLAC
F9 - stick	N	Y	BCP	Y	N	3R, heat treated in Lab 7 oven
F9 - disc	N	Y	BCP	Y	N	SEM, heat treated in Lab 7 oven
F10 - stick	N	Y	BCP	Y	Y	3R, heat treated in Lab 7 oven
F10 - disc	N	Y	BCP	Y	Y	SEM, heat treated in Lab 7 oven

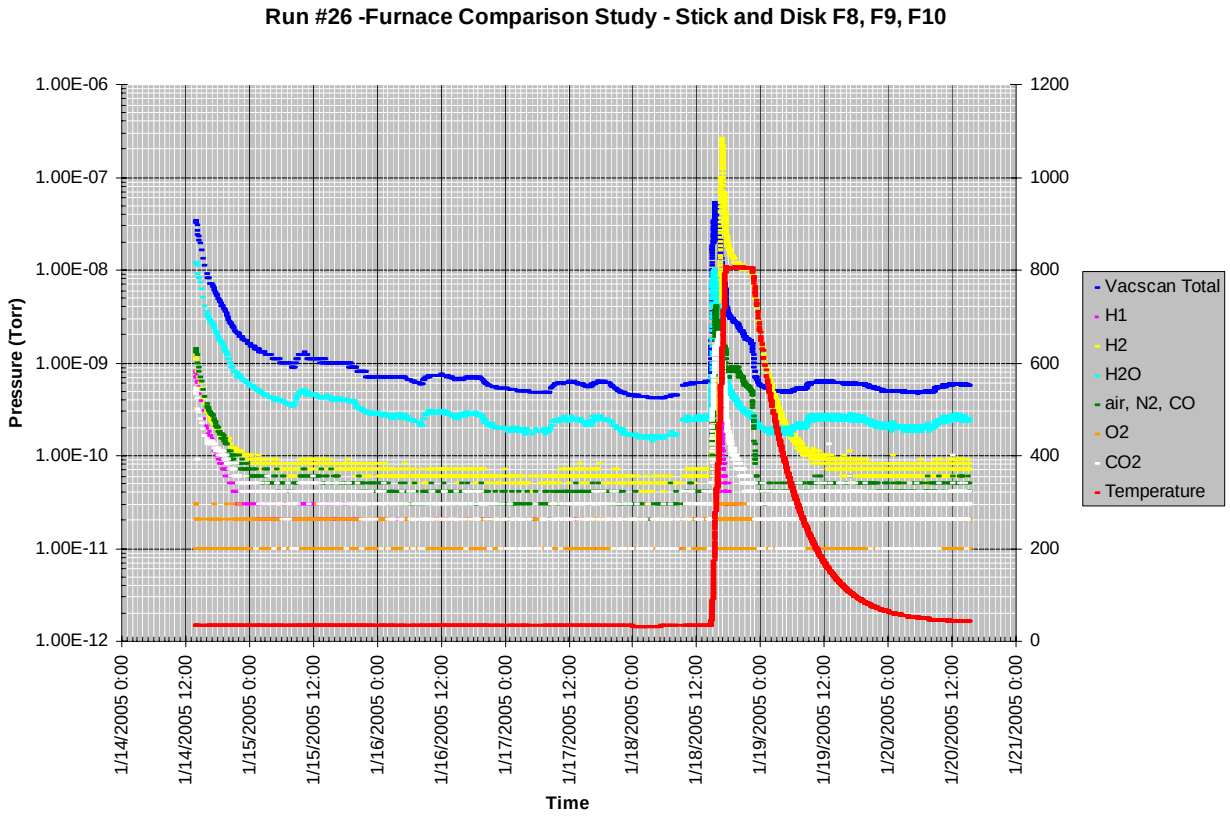
SRF SAMPLE ETCHING LOG

Date of entry	Person	Sample Type
01-19-05	Marco Battistoni	10 disks 1” diameter 10 sticks 3”,.01”,.01”
Etch type	**Operator**	**Material**
100 min	Donna Hicks	NB
Etch temp	**Location**	**Pre-and Post Cleaning**
Below 15 deg. C	MDL	IB-4 clean rooms

Sample #	Weight before etching (g)	Weight after etching (g)	Removed Thickness (calculated) (μm)	Etch-rate (μ/min)	Comment
Disk F1	12.8043	11.807	0.003659	0.9294622	
Disk F2	12.7406	11.6967	0.002829	0.9724644	
Disk F3	12.6758	11.673	0.00368	0.93472	
Disk F4	12.725	11.8	0.003397	0.8629142	
Disk F5	12.8213	11.8313	0.003633	0.922782	
Disk F6	12.8211	11.7768	0.00383	0.97282	
Disk F7	12.6879	11.6616	0.003765	0.95631	
Disk F8	10.134	9.1027	0.003783	0.9608566	
Disk F9	12.6868	11.6335	0.003863	0.9811258	
Disk F10	12.6899	11.6342	0.003871	0.983234	
Stick F1	3.538	2.778	0.004052	1.029208	
Stick F2	4.6212	3.684	0.004953	1.258062	
Stick F3	4.892	4.036	0.004542	1.153668	
Stick F4	4.6558	3.8132	0.004474	1.1364468	
Stick F5	4.6576	3.8159	0.004469	1.35126	
Stick F6	4.9006	3.9216	0.005164	1.311529	
Stick F7	4.6768	3.7872	0.004713	1.97102	
Stick F8	5.311	4.4172	0.004733	1.2022582	
Stick F9	4.8812	3.9648	0.004848	1.231392	
Stick F10	4.9125	3.9705	0.004978	1.264412	

Temperature chart for parts F-1through F-10 (disks & sticks) during etching

	100 MICRON ETCH						D. HICKS		
Sample	Date	Temp. C:	Start	30 min.	60 min.	100 min.	Comments:		
Disks									
F1	1/11/2005		13.2	10.4	13.4	11.6			
F2	1/11/2005		13.2	10.4	13.4	11.6			
F3	1/11/2005		13.2	10.4	13.4	11.6			
F5	1/11/2005		13.2	10.4	13.4	11.6			
F7	1/11/2005		13.2	10.4	13.4	11.6			
F4	1/11/2005		12.2	12	13.1	11.6			
F6	1/11/2005		12.2	12	13.1	11.6			
F8	1/11/2005		12.2	12	13.1	11.6			
F9	1/11/2005		12.2	12	13.1	11.6			
F10	1/11/2005		12.2	12	13.1	11.6			
Sticks									
F1	1/11/2005		11.6	11.8	12.2	12.9			
F2	1/11/2005		11.6	11.8	12.2	12.9			
F3	1/11/2005		11.6	11.8	12.2	12.9			
F4	1/11/2005		11.6	11.8	12.2	12.9			
F5	1/11/2005		11.6	11.8	12.2	12.9			
F6	1/11/2005		11.6	11.8	12.2	12.9			
F7	1/11/2005		11.6	11.8	12.2	12.9			
F8	1/11/2005		11.6	11.8	12.2	12.9			
F9	1/11/2005		11.6	11.8	12.2	12.9			
F10	1/11/2005		11.6	11.8	12.2	12.9			


Run #26 -Furnace Comparison Study - Stick and Disk F8, F9, F10
Pressure (Torr)
1.00E-06
1.00E-07
1.00E-08
1.00E-09
1.00E-10
1.00E-11
1.00E-12
1200
1000
800
600
400
200
0
1/14/2005 0:00
1/14/2005 12:00
1/15/2005 0:00
1/15/2005 12:00
1/16/2005 0:00
1/16/2005 12:00
1/17/2005 0:00
1/17/2005 12:00
1/18/2005 0:00
1/18/2005 12:00
1/19/2005 0:00
1/19/2005 12:00
1/20/2005 0:00
1/20/2005 12:00
1/21/2005 0:00
Time
Vacscan Total
H1
H2
H2O
air, N2, CO
O2
CO2
Temperature