

LAB 05

CHARACTERIZATION OF TEM GRIDS

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LAB SECTION No. 105 GROUP 2

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ABSTRACT

The goal of this experiment was to observe Energy Dispersive Spectroscopy (EDS) spectrums of four different transmission electron microscopy (TEM) grids. These analysis were used to determine purity, smoothness, and reaction of the grid to oxides and carbon. Copper, Molybdenum, and Nickel grids were imaged and processed. It was found that these grids were very pure, had various levels of smoothness with molybdenum grids the most smooth and nickel the least, and that molybdenum grids had the most oxygen content but still no more than 3%. Copper grids had the highest carbon content at 6.5%.

INTRODUCTION

Transmission Electron Microscopes (TEM) image a sample by accelerating electrons *through* a very thin sample and recording the location and intensity of transmitted beam on the detector². The magnification of these microscopes can range from 40x all the way to 20-50,000x. TEM presents many opportunities to visualize a sample in suspension or even sample that has been grown on the imaging surface. However, the quality of these images can be limited by the holder on which the sample is placed.

TEM grids are traditionally 3 mm diameter perforated disks with anywhere from 50 to 400 holes (called the mesh of the grid). These disks oven have a film of carbon placed on them in order to provide a surface on which the sample (usually smaller than the hole width) can sit. Unlike the thicker metal of the grid, which absorbs most of the electrons that strike it, a thin carbon film allows electrons to be transmitted and recorded. Other sample holding mediums can be used such as Formvar® or amorphous ice.

The composition of the grid is extremely important for the integrity and quality of the sample.

Copper grids are most commonly used for general work, but impure copper will more quickly oxidize (a process exacerbated by the presence of liquid, which is usually the transfer medium for the sample). Copper can also poison cells if grown directly on the grid, and can react with the sample during immunogold labeling. If working at high temperatures or if grid shrinkage is a concern, a molybdenum grid can be used. These grids have improved resistance to deformation. A nickel grid is often a good alternative when doing immunostaining, because it will not react. However, nickel grids are prone to sticking to tweezer tips due to becoming magnetized.

Contamination and oxidation are some of the most important considerations when selecting and using grids, and as a result batches of grids should be checked for impurities, surface smoothness, and deformability.

The Scanning Electron Microscope (SEM) is a powerful tool not only for imaging the surface of various samples but also for Energy Dispersive Spectroscopy (EDS).

Scanning electron microscopes take advantage of the dispersive properties of electrons, which do not penetrate deeply into the sample. Compared to other electron microscopes, like transmission electron microscopes, SEMs can image bulk samples because the machine takes advantage of backscattered beam.

The basic parts of a scanning electron microscope are an electron source, a high voltage across the electron source and the sample to accelerate electrons toward the sample, electromagnetic lenses to focus electrons, temperature control aspects, and a data collection system or detector. Standard SEMs collect backscattered electrons as they diffract off the sample at high angles of incidence. This is the most common detector found. Unlike diffractometers, the SEM does not rely on a 2θ angle in the user interface, but rather acts more like a light microscope.

Some SEMs are also equipped with the ability to detect, count, and analyze x-rays that have been scattered off a sample. With this type of detector, it is possible to determine the elemental composition of samples. The detector used in this lab is similar to Si(Li) type detectors but does not use Lithium and therefore does not need extremely low temperatures to prevent lithium from diffusing out. These detectors often need some sort of cooling (liquid nitrogen for Si(Li) or simply peltier for this ultra-pure Si with novel geometry).

The SEM can display information as either an image or as spectrum as described above. The resolution of the image is dependent on probe size and volume of the tested sample. The range of EDS that can be achieved is also dependent on the voltage across the electrodes, which in this case is 15kV.

The types of information determined by EDS are described more fully in the *Methods* section, however the main types are point analysis, line profile analysis, and digital x-ray maps.

EXPERIMENTAL PROCEDURES

For this experiment, materials were a Hitachi TM-3000 SEM with EDS software and Bruker Nano X-Flash 430-H detectors, three types of TEM grids (SPI Copper Finder, Ted Pella Molybdenum 300 Mesh, SPI Nickel Finder), and conductive sample tape.

Before beginning both sections of the lab, we initialized the Hitachi TM-3000 SEM and the Bruker Nano X-Flash 430-H detector. The voltage for this SEM was preset to 15kV.

SEM IMAGE COLLECTION

Each of the grid samples was first loaded into the SEM, and exploratory images to determine surface quality and any interesting surface features were taken between 100x and 1000x after focusing the sample. Then, depending on the area of interest, multiple sample magnifications were collected.

EDS DATA COLLECTION

After taking the initial SEM images from the viewer, *Quantax 70* software was used to calibrate and modify spectral output in the form of point, line, and elemental distribution maps. These types of data were each specifically collected for different analytical reasons.

The point map compared integrated elemental contents in a particular area which was determined by the user. Numerical data was collected specifically comparing Nickel to Copper only and once

comparing Nickel, Copper, Oxygen and Carbon. The purpose of this second data quantification was to determine what was formed on the surface of unpolished samples. In addition to numerical data, a spectrum was also collected from each sample.

The line scan profile was used to determine compositions of defects or particulates on the surface of the sample. A line over which analysis would be done was drawn over pure sample and partially over a defect. The relative elemental compositions of each region on the line were displayed and saved, which would elucidate the elemental composition in terms of the indicated elements, Cu, Ni, C, and O. The analysis profile and the location within the image along which the line scan was taken were both recorded for each sample.

Finally, the elemental distribution map showed the topographic distribution of elements. First images were collected which only quantified the grid's expected composition then to quantify the amount of oxygen and carbon. Both the area on the surface was collected as well as peak distributions, and then the same was repeated including carbon and oxygen.

EXPERIMENTAL RESULTS

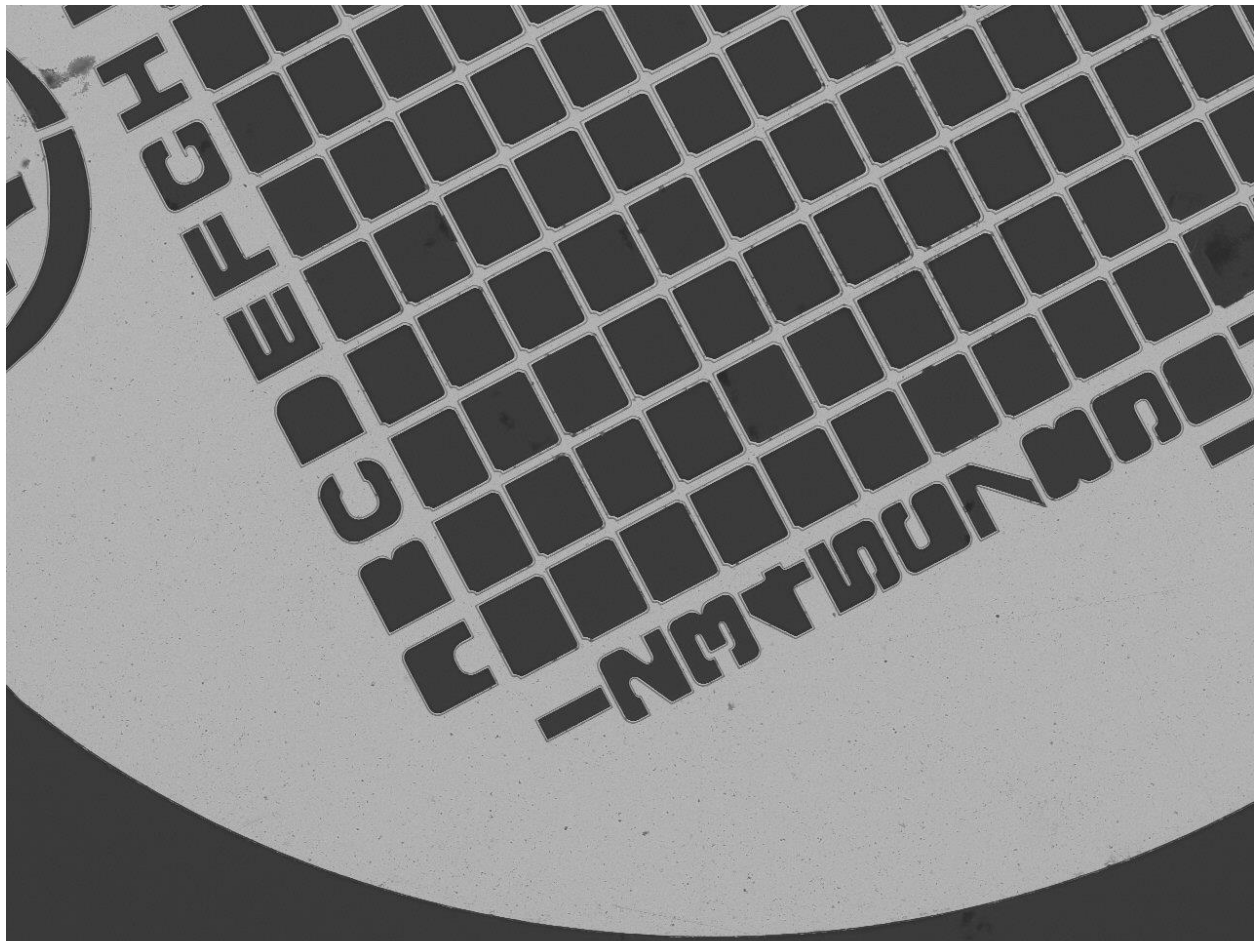
In all x-ray experiments, the background radiation was found to be 0.02 mR/hr using a Geiger counter during and before data collection. The data for each was collected slightly differently for each sample based on which properties needed to be investigated.

SAMPLE 1: COPPER FINDER

EXPLORATORY SCANS

The copper grids were first examined in order to determine smoothness, purity, and zones of interest.

Figure 1: At 100x magnification, the copper grid appears to have a smooth surface and the grid squares do not have rough edges. Already, some discoloration can be seen which may be contamination or carbon/oxygen byproducts.



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x100

1 mm

Figure 2: An increased magnification of 300x emphasizes the smoothness of the grid squares. It can also be seen that the grid squares have a slight bevel. Dark spots indicate that contaminants may be present.

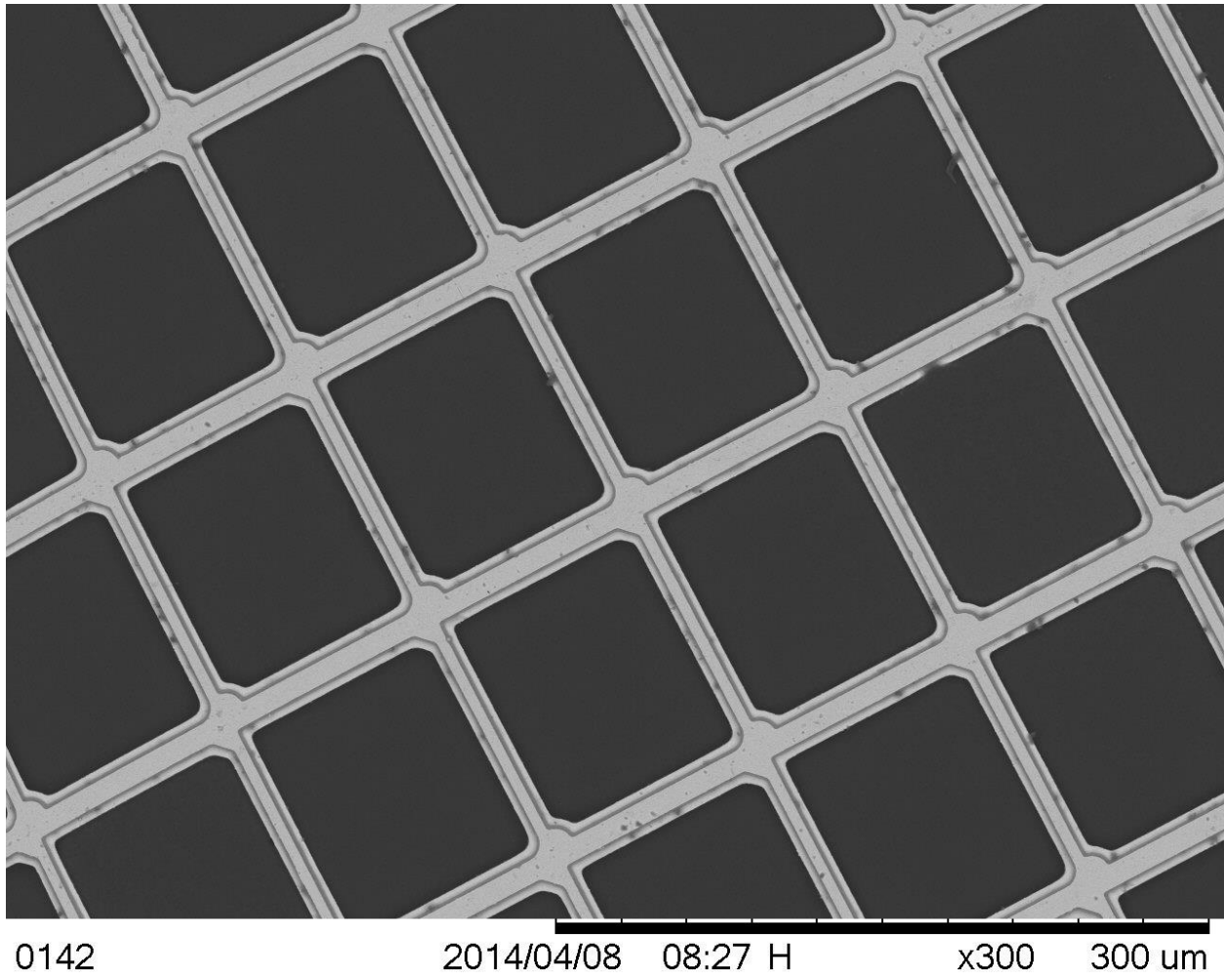


Figure 3: In order to examine the surface characteristics of the copper, 1000x magnification was used. There appear to be either bumps or divets in the surface of the sample. The long lines may be raised or inscribed, but it is difficult to tell from the angle.

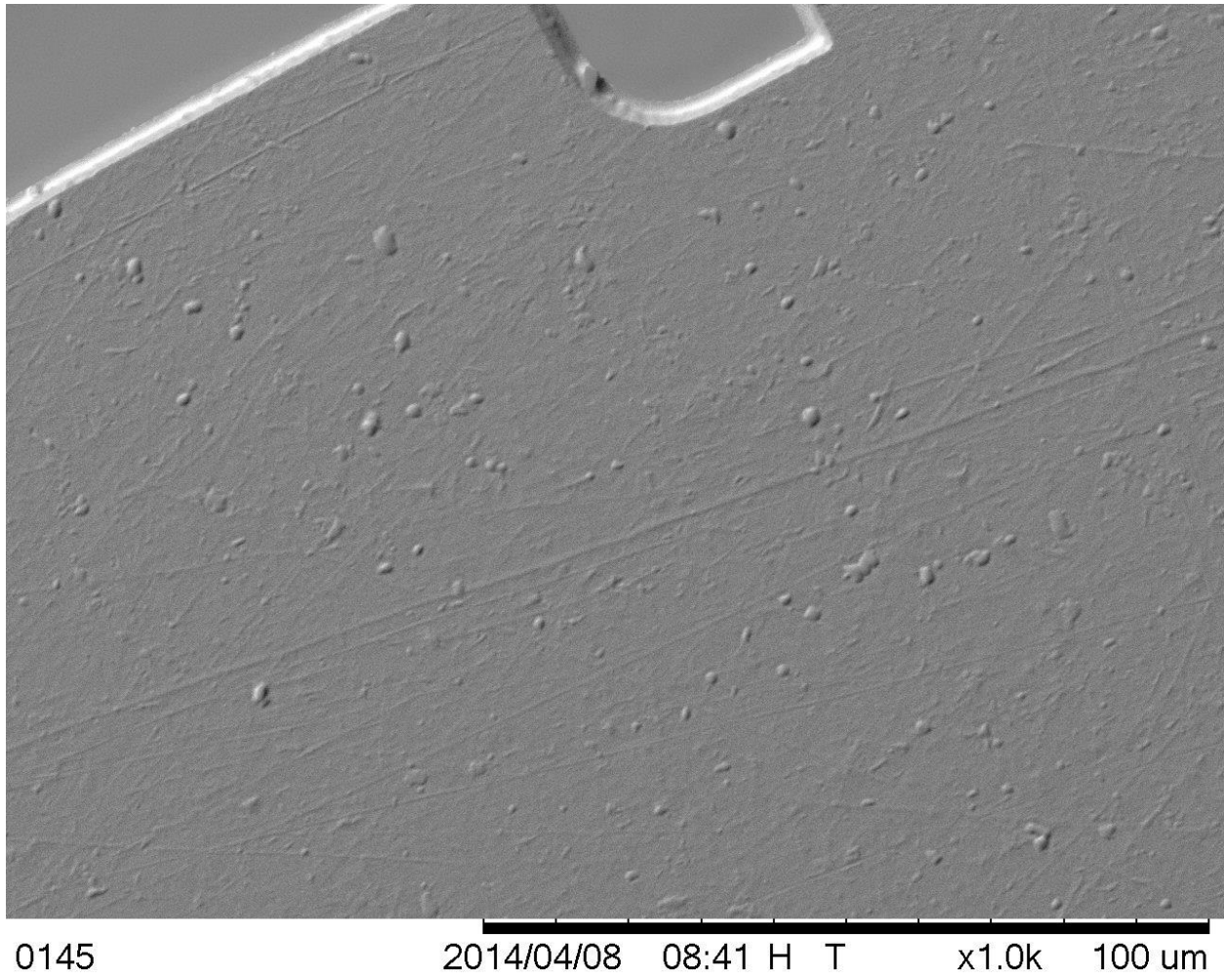
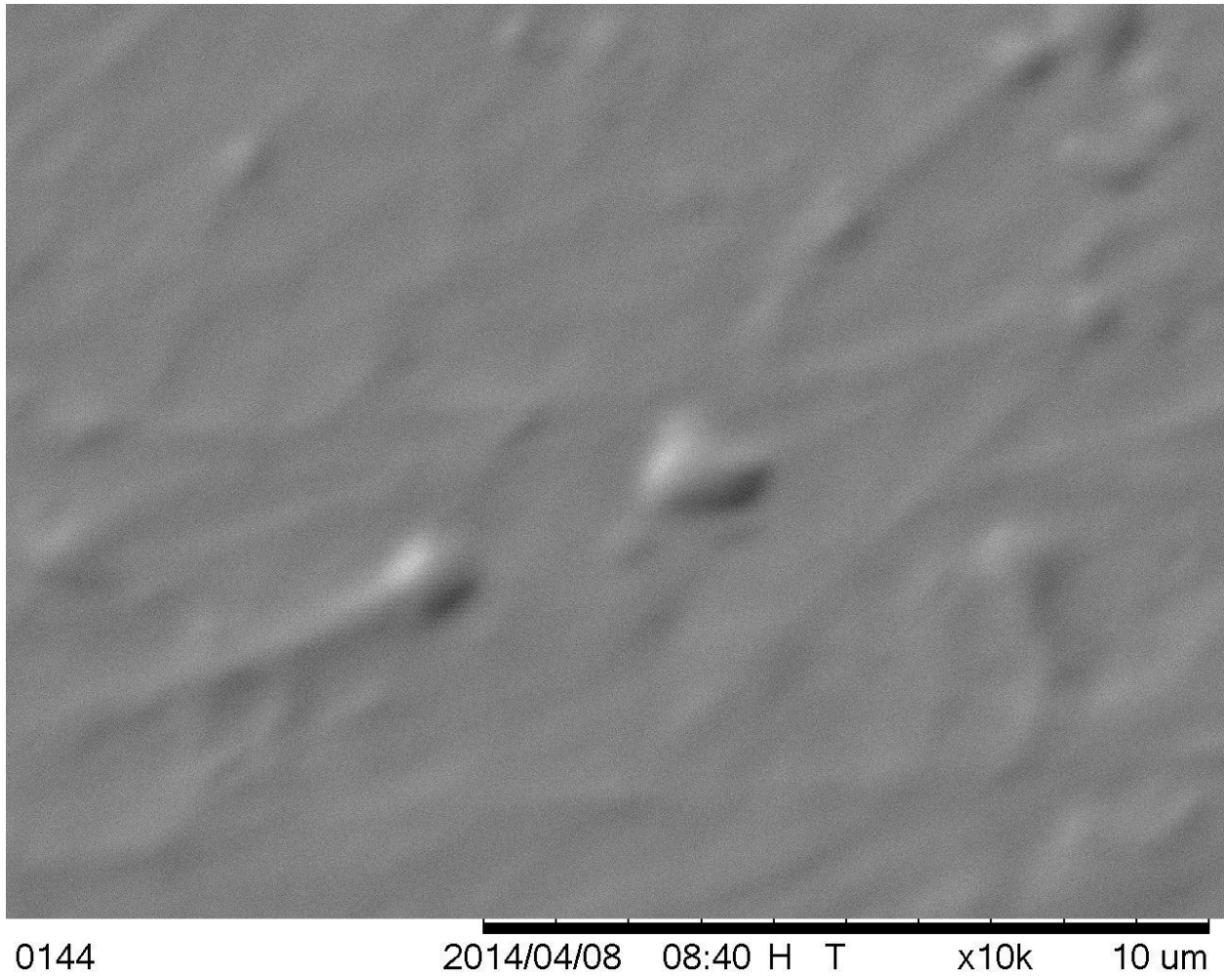


Figure 4: An increased magnification of 10,000x shows the uneven surface, which may be a natural result of not polishing the sample. This image better shows that the round spots are most likely bumps in the surface rather than dips.



POINT ANALYSIS

Table 1: Tabulated data from point analysis of the copper grid. **A** In order to test for contaminants, Ni, Mo, Ti, and Au concentrations were compared. None of these elements displayed a significant contribution, however. **B** Analysis of sample including both carbon and oxygen. Carbon was the only element to show a significant contribution, at 6.7 wt. %. Oxygen was a close second, but the low concentration indicates that the grid was not yet subject to excessive oxidation.

A

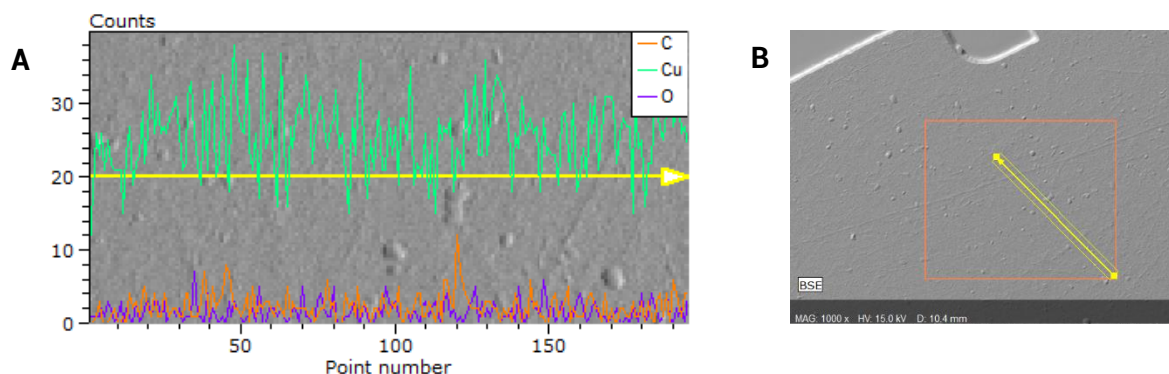
Element	AN	Series	Net	[wt.%]	[norm. wt.%]	[norm. at.%]	Error in %
Copper	29	K-series	93944	100.5514	99.85726	99.87392	3.180556
Nickel	28	K-series	115	0.074708	0.074192	0.080339	0.027271
Molybdenum	42	L-series	243	0.067029	0.066566	0.044098	0.027339
Titanium	22	K-series	0	0.001	0.000993	0.001318	8.75E-06
Gold	79	M-series	0	0.001	0.000993	0.00032	1.73E-05
			Sum:	100.6952	100	100	

B

Element	AN	Series	Net	[wt.%]	[norm. wt.%]	[norm. at.%]	Error in %
Copper	29	K-series	93945	95.73514	93.03586	72.04075	3.029408
Carbon	6	K-series	5177	6.898682	6.704172	27.46515	0.906619
Oxygen	8	K-series	314	0.13574	0.131912	0.405694	0.053897
Nickel	28	K-series	115	0.071061	0.069057	0.057894	0.027161
Molybdenum	42	L-series	223	0.058716	0.05706	0.029265	0.027049
Titanium	22	K-series	0	0.001	0.000972	0.000999	8.75E-06
Gold	79	M-series	0	0.001	0.000972	0.000243	1.73E-05
			Sum:	102.9013	100	100	

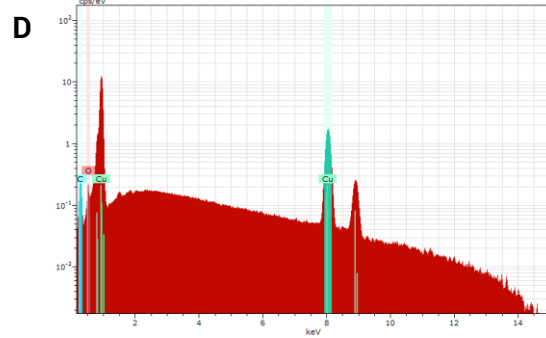
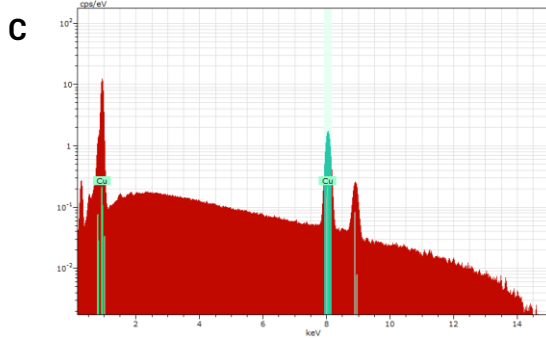
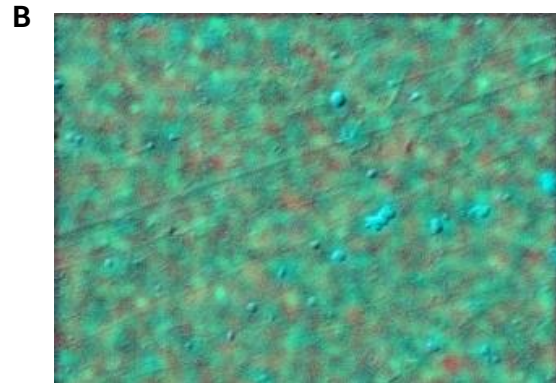
LINE ANALYSIS

Figure 5: Line analysis data was used to characterize the “bumps” **A** Line spectrum indicating relative concentrations of various elements (Cu, C, and O). Behind the spectrum is the image which is being analyzed. **B** Region over which line spectrum data was captured relative to the SEM image which was analyzed. The bumps appeared to have higher carbon concentration but this is likely due to increased exposed surface.



AREA ANALYSIS

Figure 6: Area sample data. **A** Elemental distribution over the sample surface with only Cu taken into account. From the image, it appears the distribution of copper (green) in the sample is roughly uniform, but not high in the region of the “bumps”. **B** The addition of carbon (light blue) and oxygen (red) considerations. The overall color of the image become slightly redder, indicating a possible oxide layer on the sample. However, this change in hue is much more directly a result of carbon on surface of the sample. Blue spots obviously indicate higher carbon content in those regions. **C** Elemental absorption spectrum corresponding to **A**. **D** Elemental absorption spectrum corresponding to **B**.

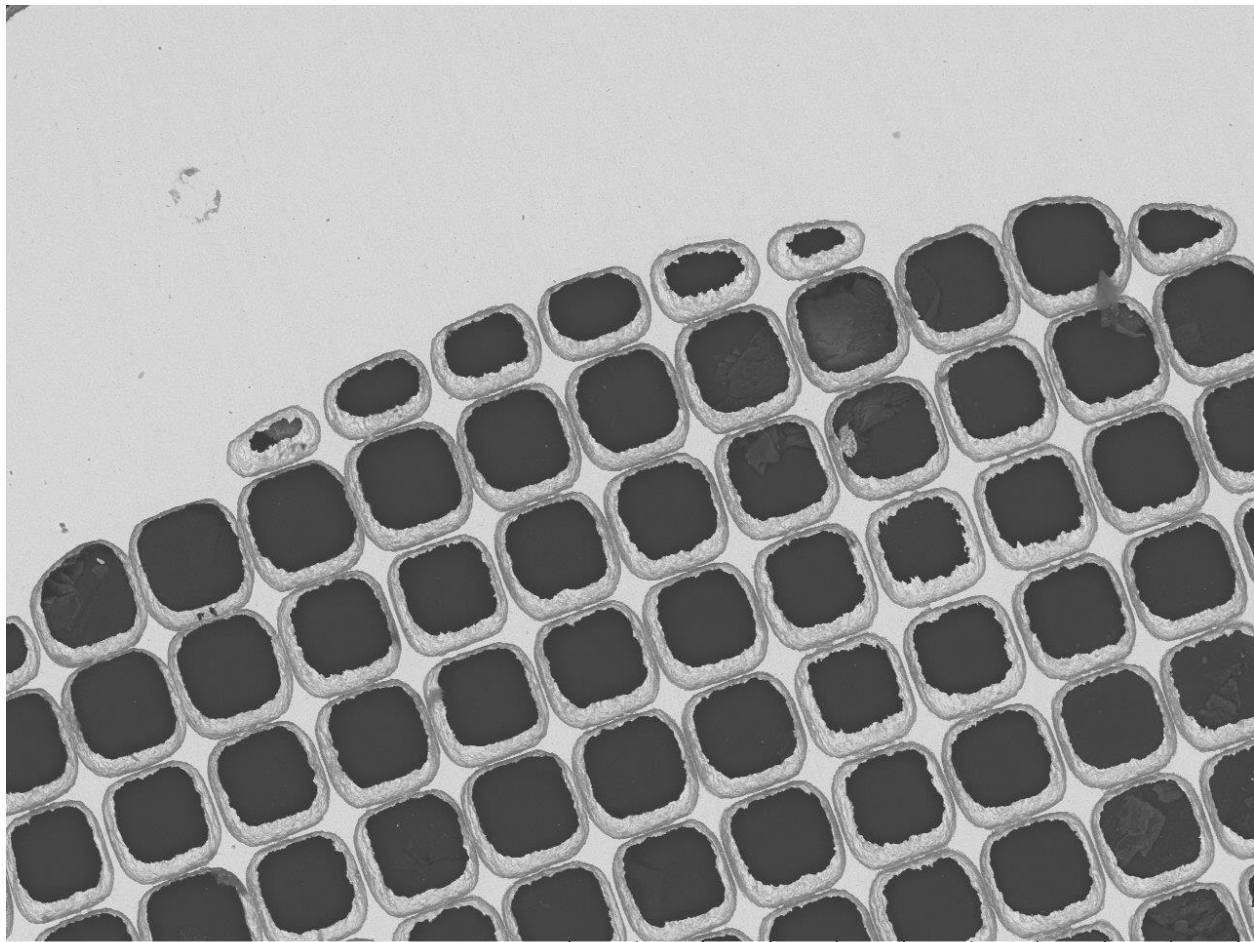


SAMPLE 2: MOLYBDENUM 300 MESH

EXPLORATORY SCANS

Molybdenum grids were first examined in order to determine smoothness, purity, and zones of interest.

Figure 7: At 100x magnification, it is clear that there is a difference between the machining of the copper and molybdenum grids. Molybdenum grid bars have only an apex at the top and appear to have been etched in order to make the grid shape.

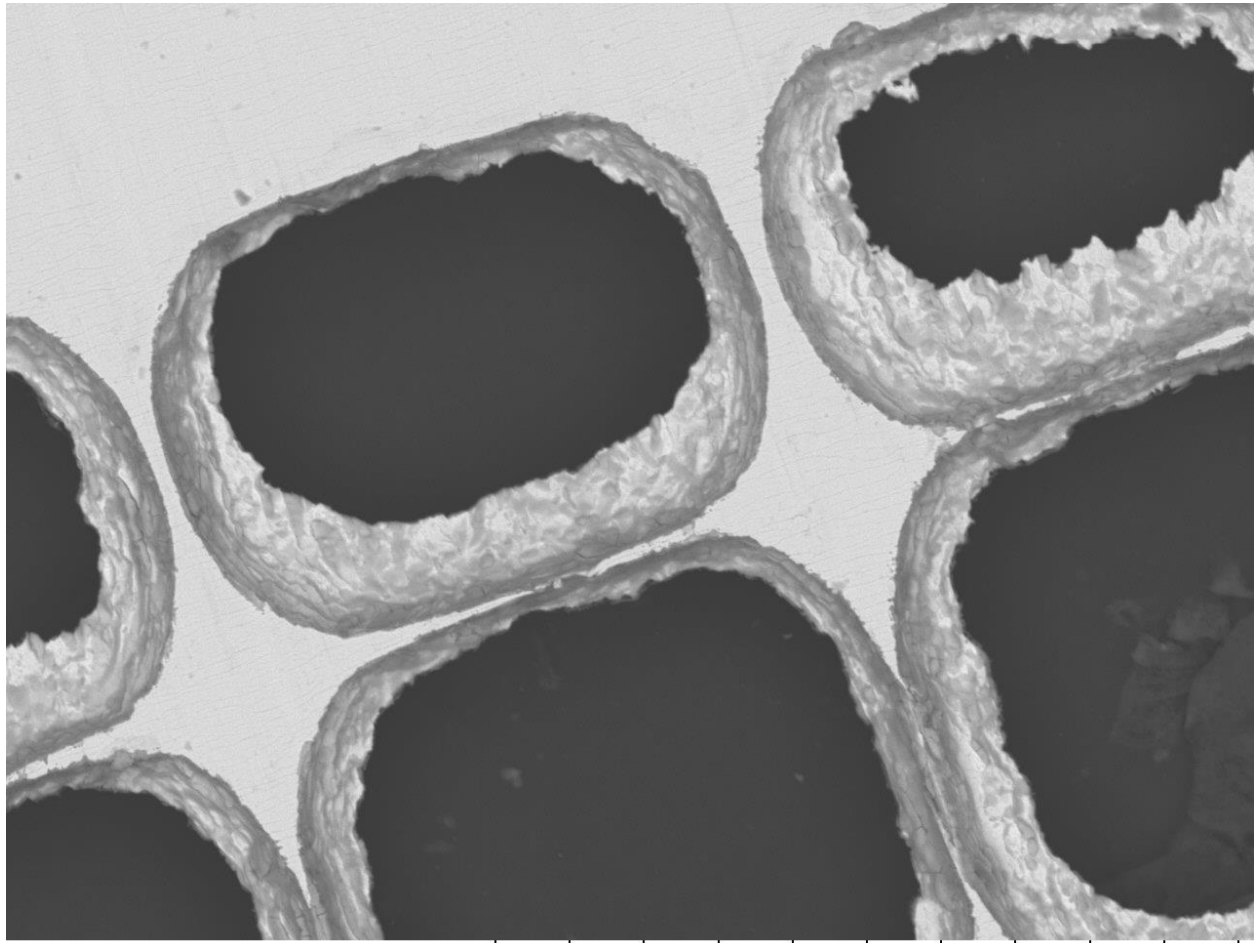


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x180 500 um

Figure 8: An increased magnification of 1000x shows that the molybdenum grid would have a very smooth surface, but where the grid has holes etched the grid is jagged and wrinkled..

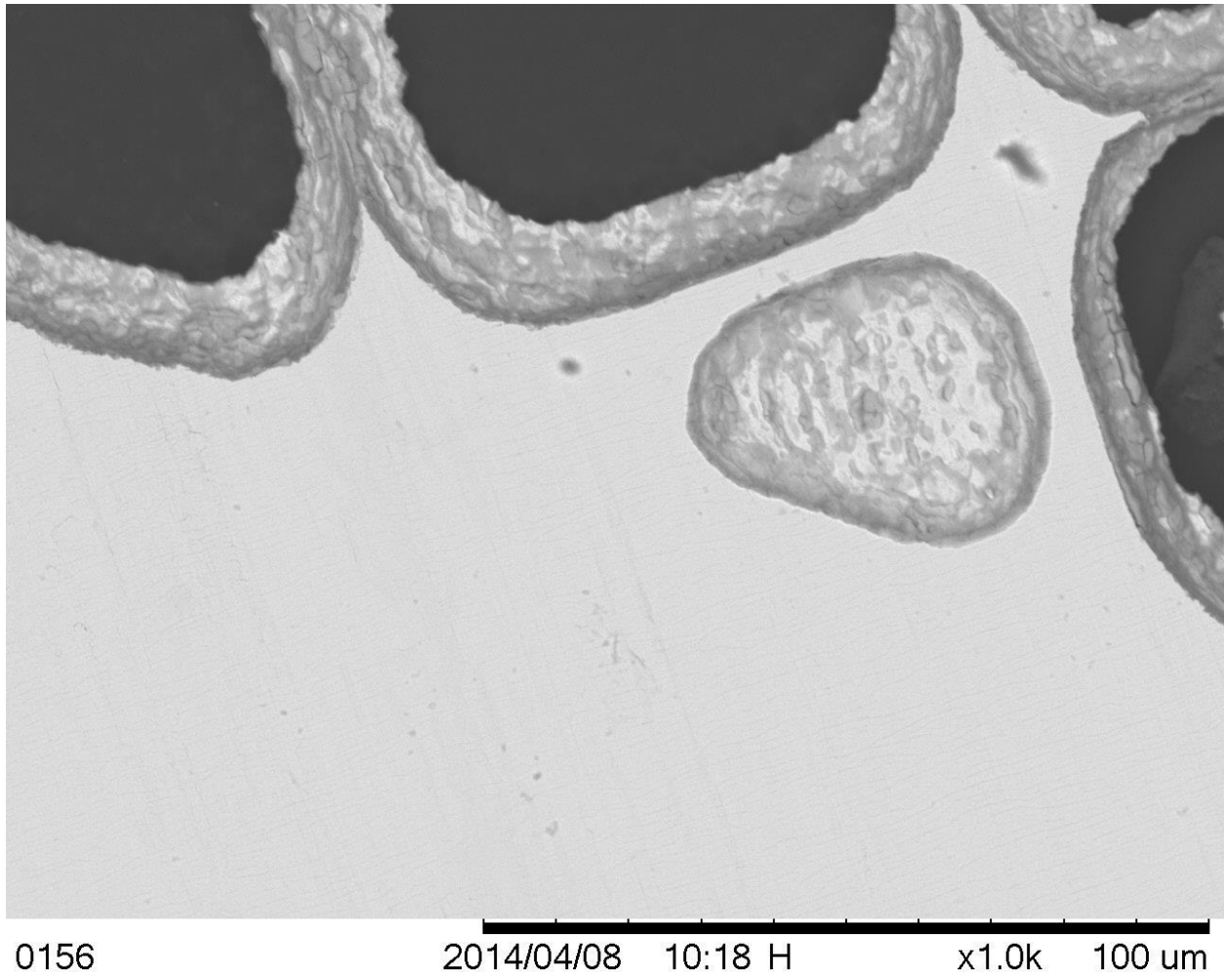


0155

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x1.0k 100 um

Figure 9: Another 1000x image is the one used for EDS because a larger continuous molybdenum area was available. The goal of the analysis was to determine whether organic matter or oxide layers built up in the jagged regions.



POINT ANALYSIS

Table 2: Tabulated data from point analysis of the molybdenum grid. **A** In order to test for contaminants, Pb, Zn, Au, Ni and Cu concentrations were compared. None of these elements displayed a significant contribution, except Pb appeared to have a higher concentration. Note, however, that on the spectrum Pb peaks are remarkably close to molybdenum, so the software may have been generous when fitting the data. **B** Analysis of sample including both carbon and oxygen. Carbon and oxygen both had a small but significant contribution.

A

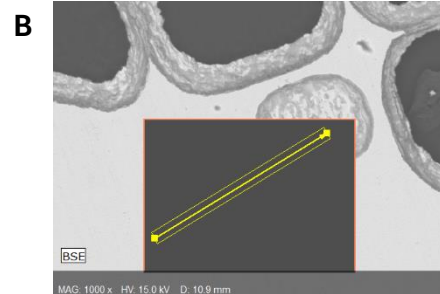
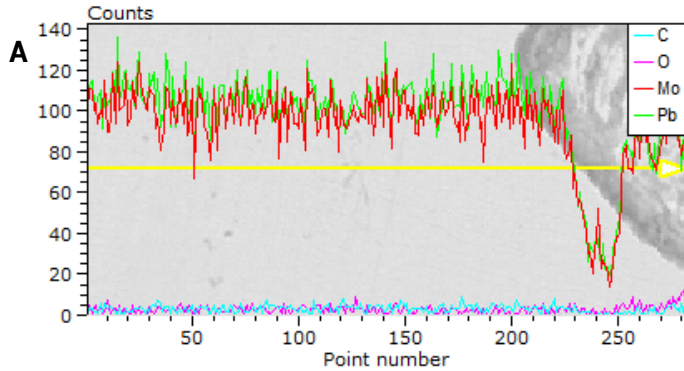
Element	AN	series	Net	[wt.%]	[norm. wt.%]	[norm. at.%]	Error in %
Molybdenum	42	L-series	242569	89.68055	85.7479	92.65902	3.154068
Lead	82	M-series	32118	14.57865	13.93935	6.974559	0.537145
Zinc	30	K-series	74	0.179086	0.171233	0.271481	0.03086
Gold	79	M-series	314	0.12984	0.124146	0.065344	0.029844
Nickel	28	K-series	7	0.009766	0.009338	0.016494	0.000297
Copper	29	K-series	5	0.008399	0.008031	0.013101	0.000264
			Sum:	104.5863	100	100	

B

Element	AN	series	Net	[wt.%]	[norm. wt.%]	[norm. at.%]	Error in %
Molybdenum	42	L-series	242605	85.83582	79.7582	58.42015	3.01992
Carbon	6	K-series	2106	4.77046	4.432687	25.93432	0.70994
Oxygen	8	K-series	1298	2.698435	2.507372	11.01291	0.44464
Lead	82	M-series	32181	13.99235	13.00162	4.409555	0.516548
Zinc	30	K-series	73	0.168439	0.156512	0.168199	0.030512
Gold	79	M-series	377	0.149513	0.138927	0.049566	0.030578
Copper	29	K-series	2	0.003636	0.003379	0.003737	0.000114
Nickel	28	K-series	1	0.001406	0.001307	0.001565	4.28E-05
			Sum:	107.6201	100	100	

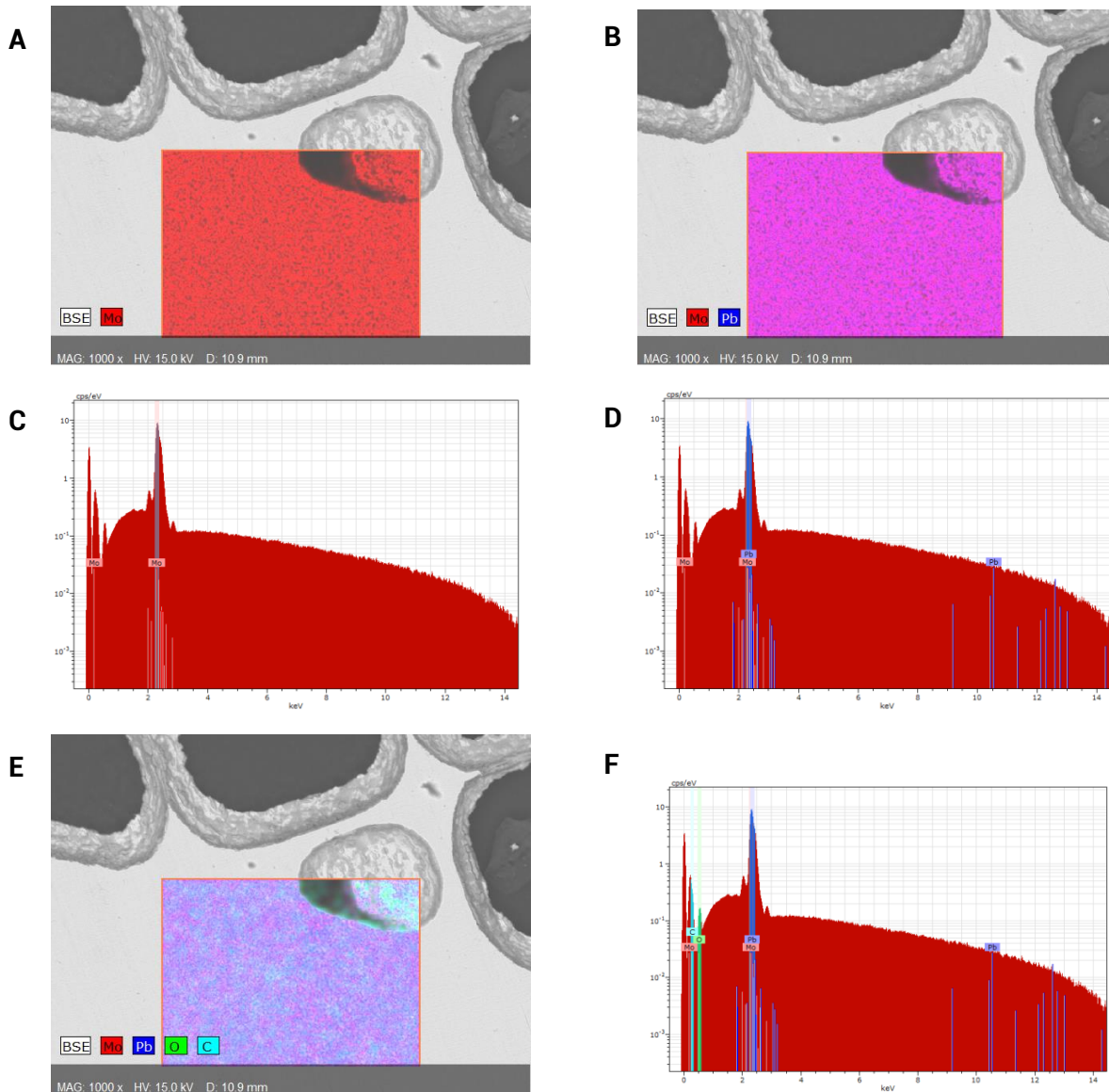
LINE ANALYSIS

Figure 10: Line analysis data **A** Line spectrum indicating relative concentrations of various elements (Mo, Pb, C, and O). Behind the spectrum is the image which is being analyzed. **B** Region over which line spectrum data was captured relative to the SEM image which was analyzed. There is an expected drop in Mo and Pb where the grid hole is etched but not a significant increase in carbon or oxygen, indicating that the layer of two is not particularly thick. Also, noticing how the Pb line almost exactly rides on the Mo line supports the previous assertion that the existence of Pb indicated in the sample may be a software flaw.



AREA ANALYSIS

Figure 11: Area sample data for Molybdenum grid. **A** Elemental distribution over the sample surface with Molybdenum. From the image, it appears the distribution of molybdenum in the sample is roughly uniform. **B** Addition of Pb considerations almost exactly overlaps the Molybdenum, once again indicating that the Pb might be an error and not actually present. **C** Elemental absorption spectrum corresponding to **A**. **D** Elemental absorption spectrum corresponding to **B** where we can see Pb and Mo overlap clearly. **E** Carbon and oxygen elements are added to the area map and the distribution is less uniform with patches of carbon and oxygen on the surface. **F** Spectrum corresponding to **E**.



SAMPLE 3: NICKEL FINDER

EXPLORATORY SCANS

The nickel grids were first examined in order to determine smoothness, purity, and zones of interest.

Figure 12: At 100x magnification, the nickel grid already looks more uneven than the other two grids.

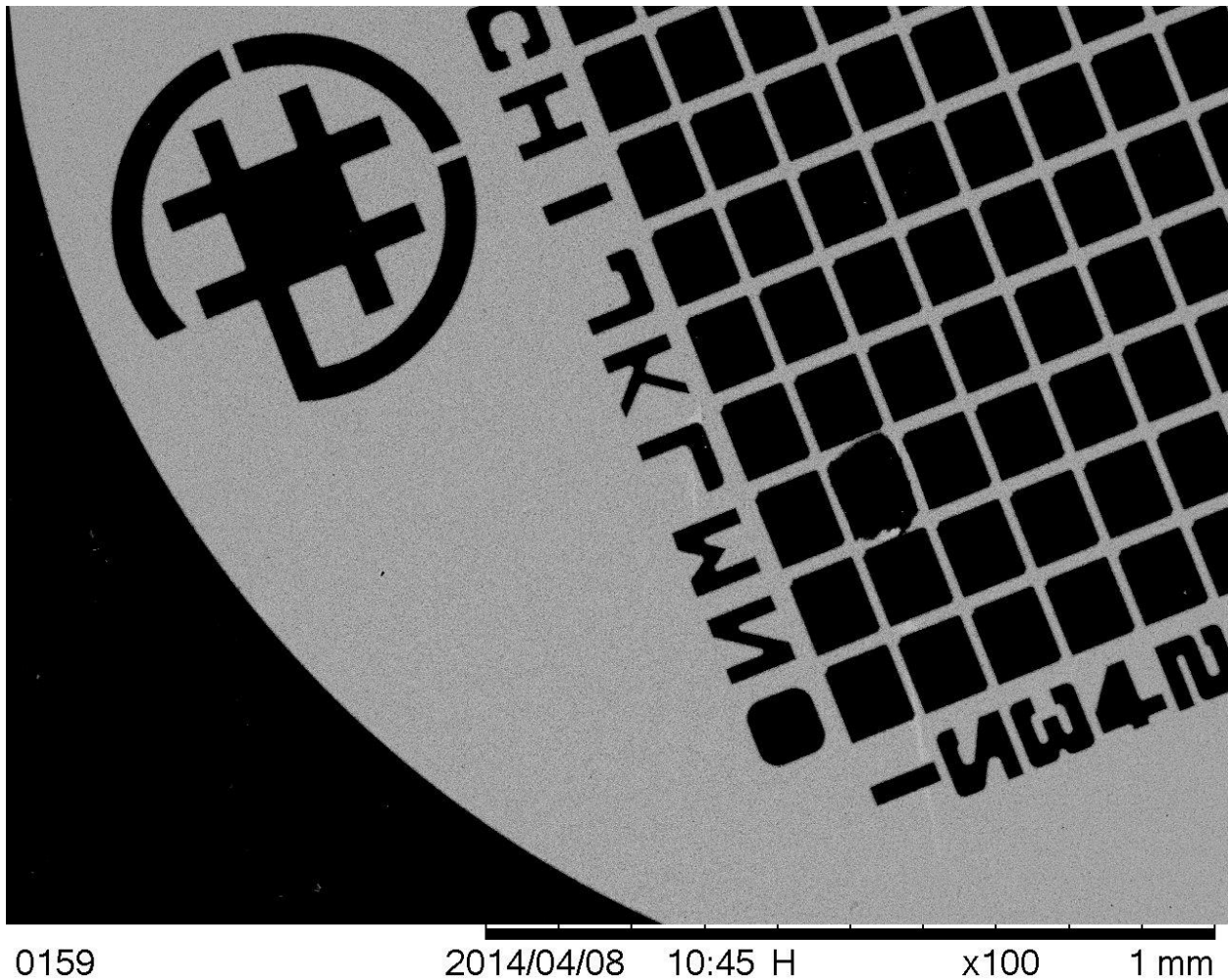


Figure 13: An increased magnification of 1000x shows that the surface is definitely not smooth, and there are pointed bumps all along the surface of the sample. The composition of these bumps will be of interest.

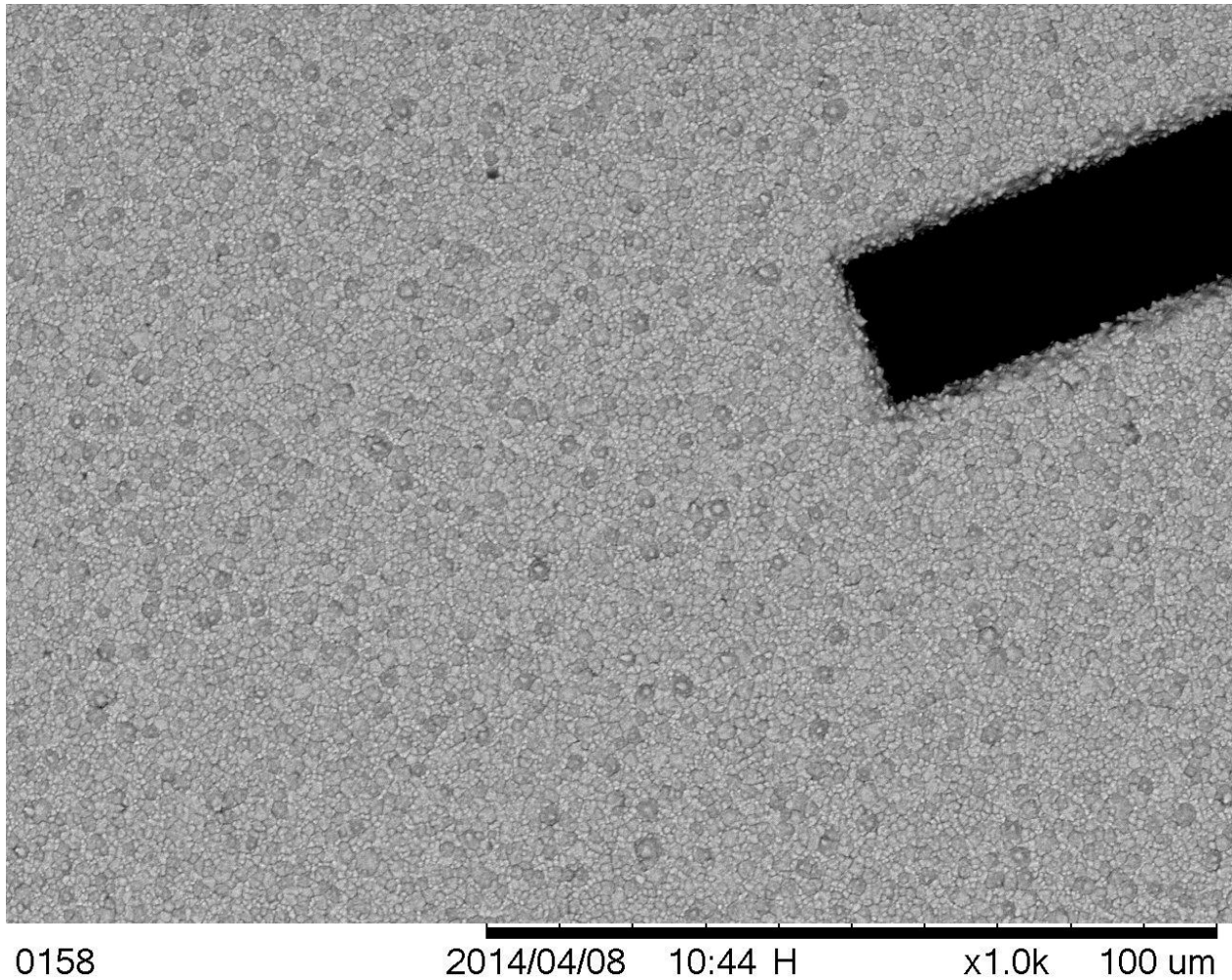
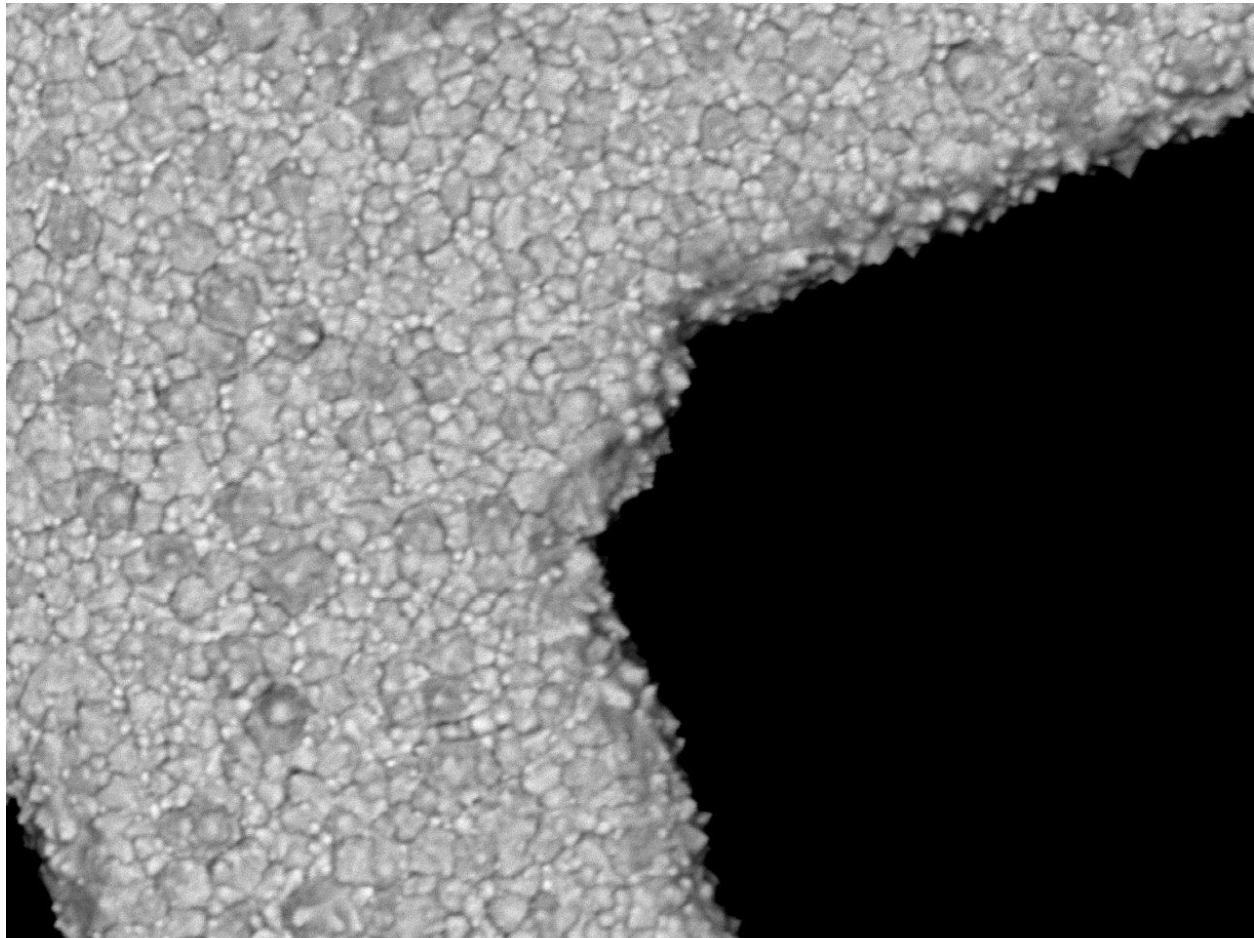


Figure 14: 3000x confirms that the bumps are pointed, and the bumps appear to be variegated. It is unclear if this is a result of different composition or different surface smoothness.



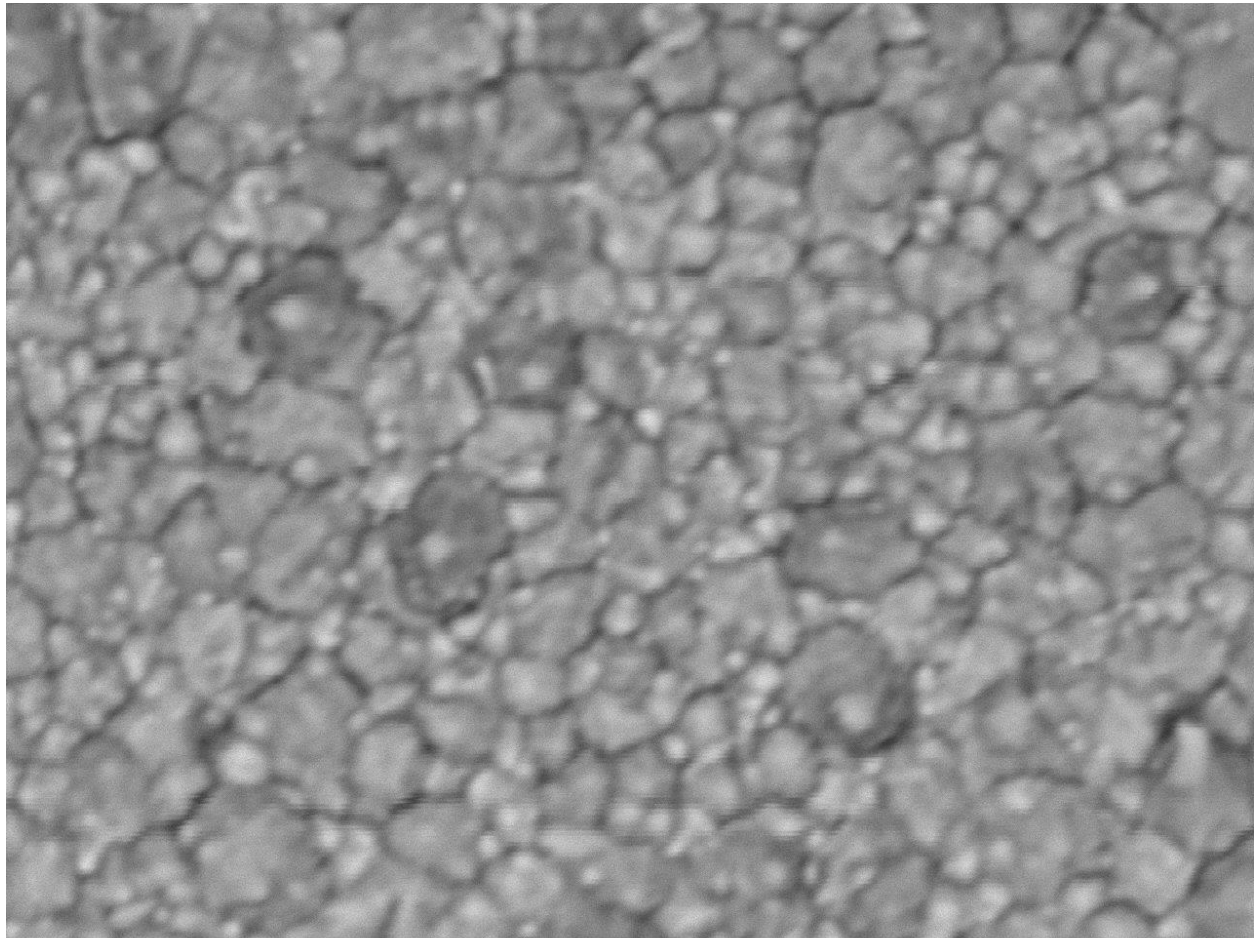
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x3.0k

30 um

Figure 15: The magnification was increased to 7000x to image the pointed spikes, which appear to be nonuniform in size.



0157

2014/04/08

10:43 H

x7.0k

10 um

POINT ANALYSIS

Table 5: Tabulated data from point analysis. **A** First Cu, Fe, and Ti were compared to concentrations of Ni in order to determine contaminants, and only copper showed any comparable value (even then, very small). **B** Analysis of sample including both carbon and oxygen but excluding Fe and Ti. This analysis resulted in a much higher incidence of carbon on the surface of the sample compared to the amount of oxygen found (5% vs 0.1%). There was more copper in the sample than oxygen, indicating there was likely not a thick oxide layer.

A

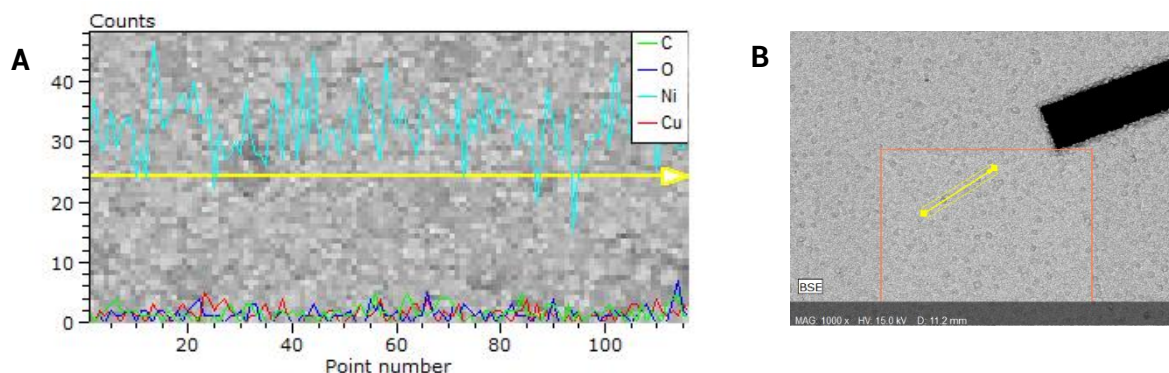
Element	AN	series	Net	[wt.%]	[norm. wt.%]	[norm. at.%]	Error in %
Nickel	28	K-series	73334	95.81331	98.92751	98.97042	2.938114
Copper	29	K-series	442	0.84121	0.868551	0.802574	0.051399
Iron	26	K-series	193	0.127908	0.132065	0.138857	0.028729
Titanium	22	K-series	144	0.069613	0.071875	0.088146	0.027002
			Sum:	96.85204	100	100	

B

Element	AN	series	Net	[wt.%]	[norm. wt.%]	[norm. at.%]	Error in %
Nickel	28	K-series	73412	95.73055	94.13837	79.06857	2.935598
Carbon	6	K-series	2047	4.870253	4.789252	19.6569	0.727318
Copper	29	K-series	478	0.895289	0.880399	0.682995	0.053096
Oxygen	8	K-series	264	0.195226	0.191979	0.59153	0.068539
			Sum:	101.6913	100	100	

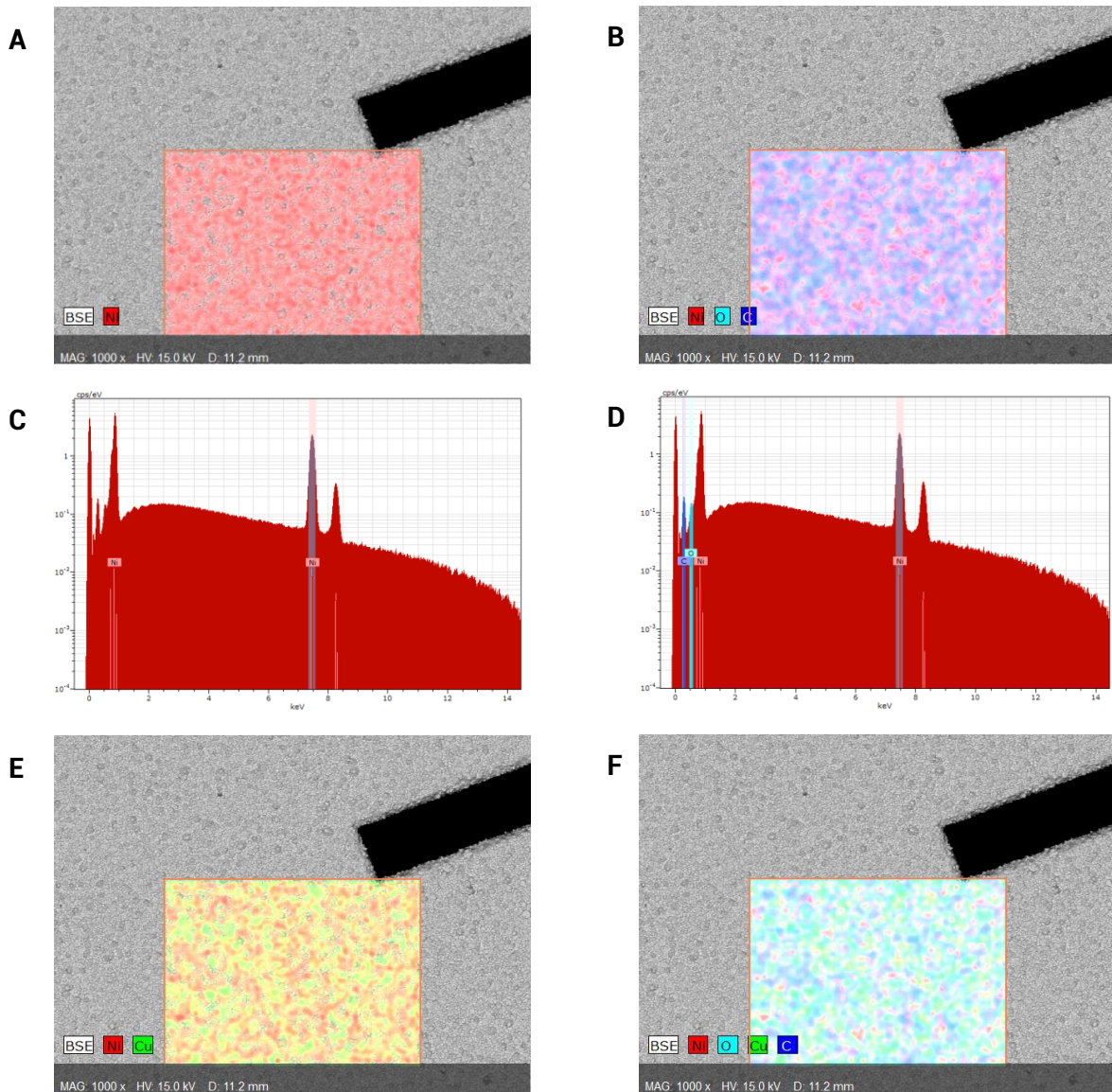
LINE ANALYSIS

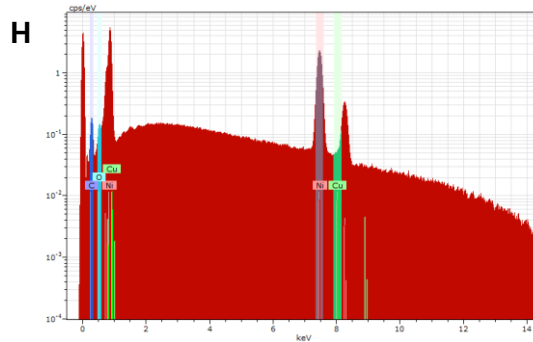
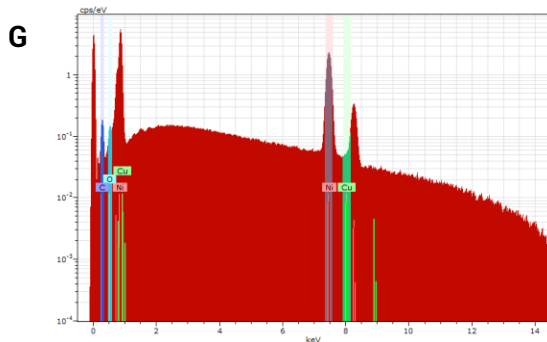
Figure 14: Line analysis data. **A** Line spectrum indicating relative concentrations of various elements (Ni, Cu, C, and O). Behind the spectrum is the image which is being analyzed. **B** Region over which line spectrum data was captured relative to the SEM image which was analyzed. The jagged shape of the Ni line makes it appear as though the bumps are largely nickel, also the changes in the other elements is so small they likely don't make up the majority of the bumps.



AREA ANALYSIS

Figure 16: Area sample data for Nickel grid. **A** Elemental distribution over the sample surface with nickel. From the image, it appears the distribution of nickel in the sample is roughly uniform. **B** Analysis of carbon and oxygen with nickel only. The carbon and oxygen appear to be patchy. **C** Elemental absorption spectrum corresponding to **A**. **D** Elemental absorption spectrum corresponding to **B** where we can see Pb and Mo overlap clearly. **E** Copper is added to the spectrum and seems to be uniformly mixed in with the Ni. **F** Carbon and Oxygen with Cu and Ni are once again patchy. **G** Elemental absorption spectrum corresponding to **E**. **H** Elemental absorption spectrum corresponding to **F**.





DISCUSSION

The data from these samples elucidated the purity, surface smoothness, and grid features that can affect the quality of TEM data.

DATA ANALYSIS

SAMPLE 1: COPPER FINDER GRID

Figure 1 through Figure 4 show that the machining of the copper grid created smooth grid bars, but left a bumpy surface (only visible above 1000x). 100x emphasized machining and grid size, 300x revealed smoothness/ dark spots, 1000x exposed surface smoothness (bumpy) and 10,000x was a little out of focus but revealed scratching on the surface. It was sometimes unclear if the features were protrusions or intrusions on the sample.

Table 1 explored possible contaminants in the grid, but no metal was found in large quantities. Carbon was the main contaminant.

Figure 5 shows the homogeneity of the sample. The only increases in carbon were around the "bumps" and the oxygen concentration change was not significant.

Figure 6 show the uniformity of the distribution of copper. Oxygen was uniformly distributed but carbon was concentrated at the bumps even in the area view.

SAMPLE 2: MOLYBDENUM 300 MESH GRID

Figure 7 through Figure 9 show that the molybdenum grid has a smoother surface than copper but the process for machining the grid leaves the holes jagged and the grid bars very thin. It is probably an etching process with a resist that leaves the grid like that. There are potential issues with placing a continuous or noncontinuous carbon film in grid bars of that shape.

Table 3 shows that there might have been a significant amount of lead in the sample with molybdenum, however this is likely an error (expanded from Figure 10 analysis).

Figure 10 shows that lead and molybdenum almost exactly overlap in the line spectrum, indicating that they are both being extrapolated from the same peak region.

Figure 11 confirms that Pb and Mo take up the same spatial and concentrational niche, and therefore the Pb signal is likely an error. There was spatial homogeneity for carbon and oxygen over the sample surface.

SAMPLE 3: NICKEL FINDER GRID

Figure 12 to Figure 15 reveal an incredibly bumpy surface region visible at magnifications as low as 1000x.

Table 3 does not show any metallic contaminants other than trace amounts of copper. Carbon is more prevalent than copper but there is less oxygen by weight percent than copper.

Figure 16 shows that the mysterious pointed bumps are composed of nickel and not contaminants or strange oxidation.

Figure 17 confirms the uniformity of the sample is nickel with carbon, oxygen and trace copper uniformly distributed on the surface.

DATA REDUCTION

PRE-COLLECTION

During the data collection process of the images collected there were some that required consideration to optimize results.

The point analysis option allowed the used to select a working area, over which the point integration would be run. Similarly, the area elemental analysis was completed over a user defined area. When selecting these areas, results were made most relevant by choosing the same area for both quantifications and by choosing an area with fewer defects. This allowed for proper quantification of relative copper-nickel values. Alternatively, if a defect was more interesting, then selecting an area with a large defect can elucidate in the "area" viewer what the spatial composition of the sample was.

The line analysis method was intended to investigate particulates that may be contaminating the surface of the sample or for nonuniformity in the sample surface. Therefore, it was best to draw the line through a particle of interest to determine the composition of that particle.

PURITY

Most of the samples were not alloys. The maximum impurity percentage found was lead in molybdenum (14%), but this is likely to have been an error. Table 4 shows the relative impurity of each type of grid. Molybdenum is the most contaminated, however we found that the percentage of lead in the sample may have been an error, leaving the actual highest concentration as zinc in molybdenum (ratio is 0.002, which is just under the nickel grid contamination).

Table 4: Highest metallic contaminant values for copper, molybdenum, and nickel samples.

Cu Finder	[norm wt %]	Mo 300 Mesh	[norm wt %]	Ni Finder	[norm wt %]
Copper	99.85726	Molybdenum	85.7479	Nickel	98.92751
Nickel	0.074192	Lead	13.93935	Copper	0.868551
Ratio:	0.00074298		0.162562		0.00878

CARBON AND OXYGEN CONTENT

The highest relative carbon content was found in the copper finder grid, and the highest oxygen content was found in the molybdenum grid (2.5%). Values are tabulated in **Table 5**.

Table 5: Weight percent calculations for each grid indicating relative carbon and oxygen concentrations. The highest carbon content was in the copper finder grids and the highest oxygen content was in the molybdenum 300 mesh grid.

Cu Finder	[norm wt %]	Mo 300 Mesh	[norm wt %]	Ni Finder	[norm wt %]
Copper	93.03586	Molybdenum	79.7582	Nickel	94.13837
Carbon	6.704172	Carbon	4.432687	Carbon	4.789252
Ratio:	0.07206009		0.05558		0.05087

Cu Finder	[norm wt %]	Mo 300 Mesh	[norm wt %]	Ni Finder	[norm wt %]
Copper	93.03586	Molybdenum	79.7582	Nickel	94.13837
Oxygen	0.131912	Oxygen	2.507372	Oxygen	0.191979
Ratio:	0.00141786		0.03144		0.00204

COMPOSITIONAL HOMOGENEITY

All of the tested samples were homogenous and did not display noticeable signs of contamination. Even samples with higher oxygen content did not show obvious oxidation regions.

This is important because grid manufacturers should make pure grids of only the element which they are advertising. Otherwise the grid may contaminate or react with the sample which is placed on it. The only grid which was concerning may have the nickel grid which showed trace copper in the sample.

Nickel grids are traditionally used for their lack of reactivity, however copper does not have the same properties. This contamination could damage cells or effect immunogold¹ tagged samples because electrochemical reactions can occur with copper.

SURFACE SMOOTHNESS

Surface smoothness plays an important role when placing carbon films on TEM grids to support the sample. A jagged or rough surface can tear the film, which is only 100µm thick.

The surface smoothness of the three grids were very different. Molybdenum grids had exceptional surface smoothness, but then had jagged grid bars without flat surfaces to reduce stress on the carbon. This fulcrum-like support would concentrate stress on the carbon and may compromise the film if large, heavy sample was placed on it. Molybdenum grids tend to be used with cryo-TEM, however, because of their exceptional resistance to shrinkage. Cryo-TEM does not use carbon films, but rather amorphous ice, which can be stabilized by these sorts of edges.

Copper had the second smoothest surface, with small bumps that appeared to be extrusions. These bumps had higher carbon content (likely due to increased surface area) and would have little effect on the carbon film other than added friction to help the film adhere to the grid.

Finally, the nickel grid had a pointed bumps in high concentration. From **Figure 15** a rough estimate is 40 points per $100 \mu\text{m}^2$. Compare this to copper in **Figure 4** which has less than 3 bumps per $100 \mu\text{m}^2$. The increased roughness of the grid could compromise the carbon film with heavy samples in regions but may also improve adhesion of the carbon film for lighter samples. Nickel grids are sometimes used with a Formvar® layer and sections of resin embedded tissue are placed on top. Formvar® is a plastic which is much stronger than carbon film, and which may have an adhesive benefit when used with a nickel grid that has these pointed bumps.

ERROR SOURCES

The main notable source of error was the expression of lead in the molybdenum grid. The EDS software has an algorithm that bases the fit of the different expected peaks on a user-defined predicted set of elements. Because the possible contaminants were unknown, choosing lead as an element to check was an error. Lead peaks are very close to molybdenum peaks as seen in **Figure 11 D** which can lead to error. Users must be very careful to both know the elements which they are looking for and to check if expected elements are too close to other expected elements. Otherwise erroneous data may be collected, as was accidentally found in this report.

CONCLUSIONS

From this lab we concluded that the EDS was useful for spatial compositional analysis, and copper, molybdenum, and nickel grids have different smoothness and impurity attributes. It was also found that the manufacturers produced pure grids with little contamination (less than 1%).

REFERENCES

¹ SPI Supplies. *Introduction to TEM grids*. <http://www.2spi.com/catalog/grids/selection-of-grids.html>. Accessed April 2014.

² B.D. Cullity and S.R. Stock, *Elements of X-Ray Diffraction*, 3rd Edition, Prentice-Hall, New York, (2001), p. 118.

APPENDIX

Overflow materials should appear here. Examples include extra data runs, problematic data (such as interrupted data runs), copies of pertinent literature or other documentation, computer source code listings, or derivations of equations.

Additional Information provided when exporting point integral files (only saved for one sample, 100Cu unpolished)

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[SemImageFile]
Version=02-01
InstructName=TM3000
SerialNumber=103102-03
DataNumber=0067
SampleName=
Format=JPG
ImageName=100CuunpolishedSEM.jpg
Directory=C:\Users\MSE104\Desktop\Store      Data      in      this
folder\MSE104\MSE104 sp 2014\TUESB\100CuUnpolished\
SaveMode=2
Date=3/18/2014
Time=09:24:43
DateCheck=Yes
TimeCheck=Yes
NumberCheck=Yes
CommentCheck=No
DigitalZoom=No
Media=Drive_C
DataSize=1280x1100
DPI=193.52
PixelSize=13.13
SignalName=BSE COMPO
AcceleratingVoltage=15000 Volt
DecelerationVoltage=
Magnification=10000
WorkingDistance=12600 um
EmissionCurrent=67400 nA
FilamentCurrent=1750 mA
ObservationCondition=Analy
Brightness=2213
Contrast=3000
Rotation=0
LensMode=
PhotoSize=
Vacuum=High
MicronMarker=10000
SubMagnification=
SubSignalName=
SpecimenBias=
Condencer2=1937
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ScanSpeed=Slow3
CalibrationScanSpeed=
ColorMode=Grayscale
ColorPalette=
ScreenMode=
Comment=[whoa this is awesome]
KeyWord1=
KeyWord2=
Condition=Vacc=15.0kV Mag=x10.0k WD=12.60mm
DataDisplayCombine=
StageType=0
StagePositionX=0
StagePositionY=0
StagePositionR=
StagePositionZ=
StagePositionT=