Chemical Analysis of MoS2 Nanoparticles formed by Ultrasonic Exfoliation

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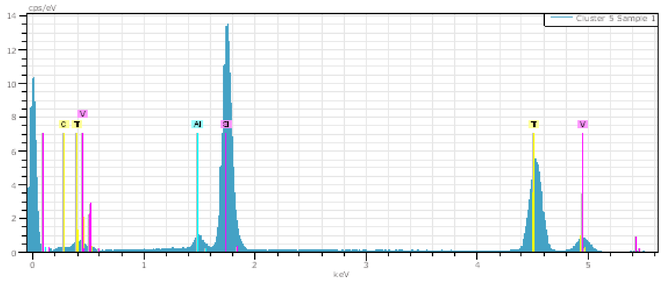
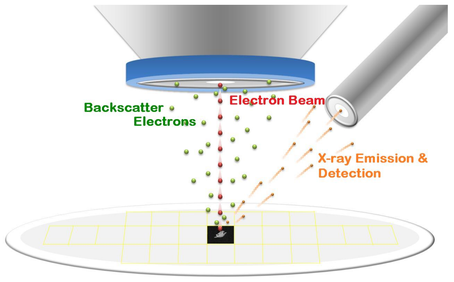
Purpose

MoS2 has been observed to be a compound with favorable electronic properties., with possible applications in small electronics. However, we wanted to analyze the chemical compositions of the samples that we've been producing because there have been electric inconsistencies observed. The chemical study, therefore, is to study exactly what the chemical compositions of these MoS2 nanostructures turn out to be after their manufacturing process, and the correlation of that to the observed electrical properties.



EDX

EDX means energy-dispersive x-ray spectroscopy. It analyzes the chemical composition of samples by detecting the various emission spectra of samples. First, the sample is bombarded with an electron beam. This excites the electrons in the atoms of the sample and cause them to jump to a higher energy state. When they fall back to their ground state, they emit radiation. The X-ray radiation that they emit is picked up by the X-ray detector part of the EDX setup. The X-rays will vary based on the chemical makeup of the sample, as each element has a unique emission spectrum. The detected X-rays are then analyzed, and the end result on the computer screen is a graph of the radiation as shown above. The peaks shown in the data will correlate to the known emission spectra of the elements, and from that data, the researcher can determine the chemical composition of the sample.



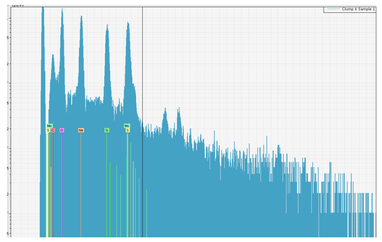
Graphical representation of EDX

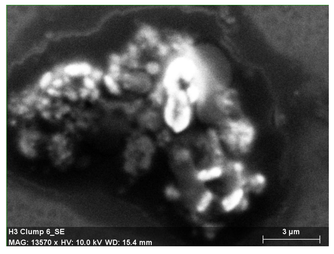
Ultrasonic Exfoliation

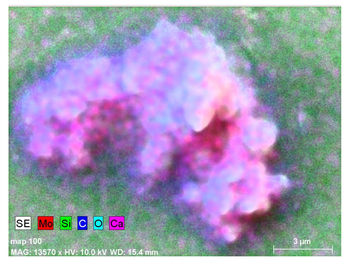
Ultrasonic exfoliation uses an ultrasonic probe to induce cavitation in a liquid. This is the process by which bubbles are formed inside a liquid. When the bubbles burst large amounts of energy are released that break down materials. MoS2 is a layered material so it most easily breaks apart the bonds holding the layers together. This separation of layers, or exfoliation, results in the formation of small scale MoS2 particles with a finite number of layers - perhaps even only 1 or two molecules thick. At such small scale thicknesses MoS2 is known to transform into a an optically active material with electronic properties of interest in nanoscale photovoltaics or transistors. Usually breaking down the material in this manner is a very labor intensive process. Using liquid exfoliation we hope to create large amounts of material so that the MoS2 nanoparticles could be used in a real device.

Typical Sample- MoS2 clump, slight impurities

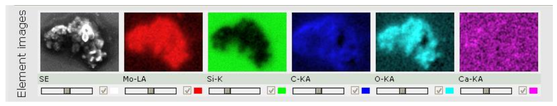
This sample is a good representative of how most of the MoS2 sample came out. We see a high concentration of the MoS2 in this clump to the right, as expected. However, there is also the impurity of carbon that is found everywhere throughout the batch of MoS2. The silicon shown in the scan is the surface onto which the sample was placed. The peaks seen above the scans , from left to right, are O, Na, Si, and Mo/S (their peaks overlap). Therefore, we can see that even though this object is mainly MoS2, impurities are also somewhat prominent in the sample, as they are throughout.







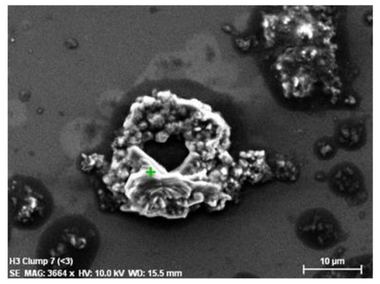
Mo Si C O Ca



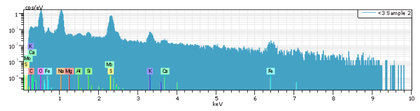
The grayscale image is the SEM image. The colored image is the result of an EDX function called mapping, where certain elements are being scanned for specifically, as to give an idea of exactly where each specific element can be found. Here, we scanned for Mo, Si, C, O, and Ca.

Chemical Impurities

Both of the MoS2 samples had many impurities ; there were were several ones, mostly carbon, sodium, and calcium, and oxygen. We believe the sodium to come from human sweat from the preparation process. Carbon can most likely be attributed to the process of synthesizing these nanoparticles, as isopropanol is used in the process. Oxygen is often a result of oxidation of the molybdenum. Some other impurities, though, like the potassium and iron found in the sample to the right, we're not sure as to the origins of. Mostly, they aren't present, but when they are, they tend to coincide with the appearance of sodium. This indicates that these could possibly be just human contaminants, and merely requires more careful handling of the samples.



This sample, shown above and analyzed to the right, was one of the most impurity laden samples that we had. Shown on a log scale, it clearly displays peaks for several elements not expected to be present, such as iron and potassium.

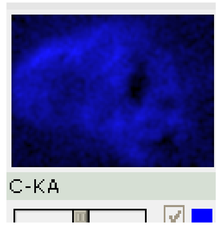


<3 Sample 2 Date:6/27/2013 2:05:51 PM
HV:10.0kV Puls th.:0.65kcps

El	AN	Series	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Error [%]
C	6	K-series	0.20	0.16	0.29	0.4
O	8	K-series	58.47	48.44	64.34	7.6
Na	11	K-series	27.88	23.10	21.35	1.7
Mg	12	K-series	0.39	0.32	0.28	0.1
Al	13	K-series	0.44	0.36	0.28	0.1
Si	14	K-series	0.84	0.70	0.53	0.1
S	16	K-series	13.69	11.34	7.52	1.4
K	19	K-series	2.49	2.06	1.12	0.1
Ca	20	K-series	0.58	0.48	0.25	0.1
Fe	26	K-series	8.73	7.24	2.75	0.7
Mo	42	L-series	7.00	5.80	1.28	2.6
Total:			120.71	100.00	100.00	

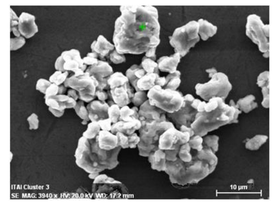
Unexpected Carbon

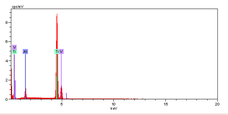
In a great number of the scans that we took of these MoS2 samples, there was a significant amount of carbon that was somewhat unexpected. One can see how the carbon is actually present throughout the whole area, not just in the crystal itself, although it is in higher concentration there. This carbon could be a byproduct of the manufacturing process of the MoS2 nanoparticles, as isopropanol is used in the preparation of the nanoparticles.The carbon, while unexpected, could be good or bad, which is yet to be determined. It could be that this carbon that we've found is benefiting the electronic properties of the MoS2 nanoparticles, but it could also very well be likely that the carbon is hindering those electronic properties.



Other impurities and contaminants

Occasionally, strange things make their way into the sample that are not immediately apparent. The image below is not actually an MoS2 sample, but a pure chunk of a Ti-V-Al alloy. This actually came from an AFM tip.





Peaks left to right: Ti&V, Al, Ti&V

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Photo source (EDX diagram): http://www.aspexcorp.com/solutions/omegamax/edxspectroscopy.aspx,