Using Synchrotron X-Ray Scanning Tunneling Microscopy to Analyze the Elemental Composition of a Sample at the Nanoscale

A Nanoscale Approach to a Common Practice

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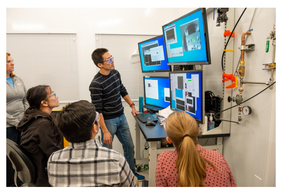
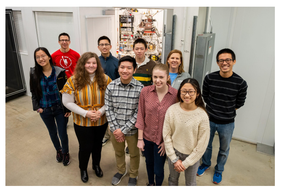
ABSTRACT

SX-STM (synchrotron x-ray scanning tunneling microscopy) combines high precision spatial imagery with the ability to gauge the magnetism and properties (elemental) of a particular sample. This experiment focuses on the x-ray side of the instrument, in which a sample of nickel and cobalt was elementally analyzed using the monochromatic x-rays of the beamline. By using x-rays with specific photon energies focused on a sample at the nanoscale, we could collect energy scans of the sample and compare it to energy scans of the elements of interest. Prior, calibration curves were created by analyzing iron and manganese using SX-STM. Based on this data, we could also determine the purity of the samples and analyze the "fingerprint" of each particle.





MOTIVATION

Although other technology exists that can examine the elemental composition of a substance at the nanoscale, these analyses provide an "average" of the composition of the specific sample of interest. We want to gauge the elemental composition of a sample with much higher precision. Since SX-STM uses a tip as the detector, the resolution is not limited by the beam size and we are able to examine the composition of a sample at the nanoscale.

METHODS

When calibrated to the matching energy level of the bonding energy of an element in the sample, the synchrotron x-ray excites electrons to tunnel through the tip of the SX-STM. Because we already had an idea of the sample's composition, we could target a specific energy range, and then analyze the peaks on the scans in this specific range.

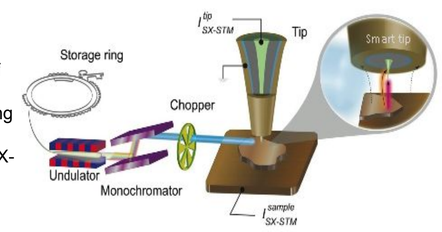
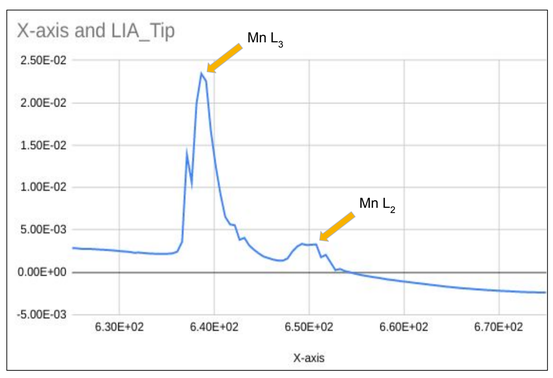
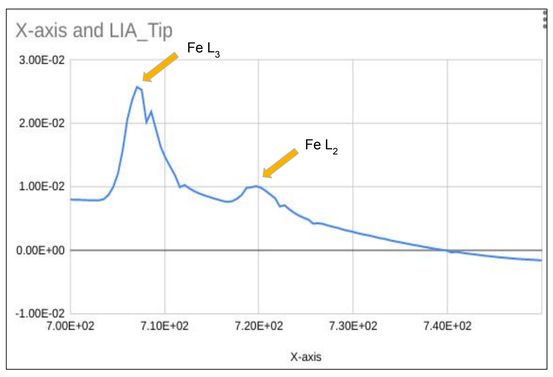


Figure 1: Schematic of Synchrotron X-Ray Scanning Tunneling Microscope (SX-STM)

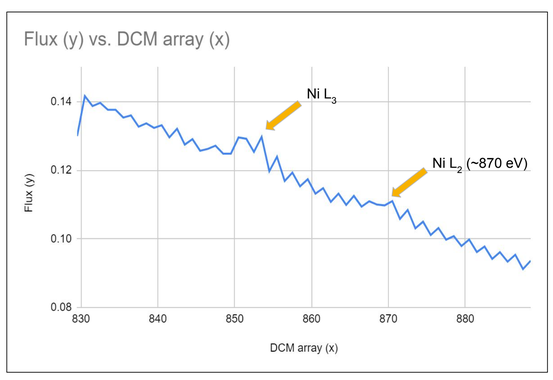
DATA



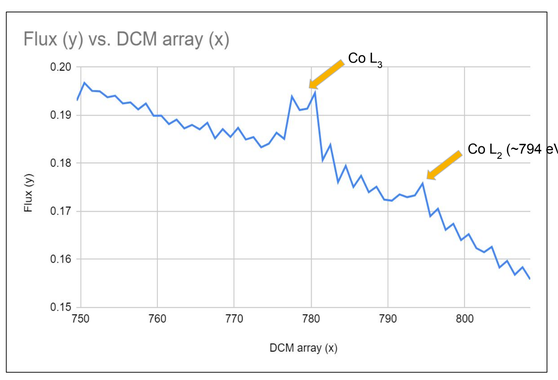
Graph 1:
Tip Current vs. Photon Energy of Mn Foil



Graph 2:
Tip Current vs. Photon Energy of Fe Foil



Graph 3:
Sample Current vs. Photon Energy of Nickel



Graph 4:
Sample Current vs. Photon Energy of Cobalt

DATA ANALYSIS

As seen in graphs 1 and 2, iron foil and manganese foil were used for calibration. Their smooth curves are attributed to the fact that there was ample material for the data to be collected from. We matched the observed peaks in the absorption spectra to known elements in order to identify the elements within each sample. The locations of the various peaks in each case correspond to the known peaks of iron, manganese, nickel, and cobalt. Because the nickel and cobalt samples are so small, there is more "noise" in the data. Therefore, the patterns for nickel and cobalt are less pronounced than the patterns for the iron and magnesium foil. However, even with noise in our measurements, peaks can be observed at corresponding binding energy levels.

			$L_2\ 2p_{1/2}$	$L_3\ 2p_{3/2}$			
25 Mn	6539	769.1†	649.9†	638.7†	82.3†	47.2†	47.2†
26 Fe	7112	844.6†	719.9†	706.8†	91.3†	52.7†	52.7†
27 Co	7709	925.1†	793.2†	778.1†	101.0†	58.9†	59.9†
28 Ni	8333	1008.6†	870.0†	852.7†	110.8†	68.0†	66.2†

Table 1. Mn, Fe, Co, and Ni Binding Energies

CONCLUSIONS

- Synchrotron X-ray Scanning Tunneling microscopy does have the power and resolution to identify the elemental composition of single particles at the nanoscale.
- However, as resolution increases, the size of the effect in the absorption spectra decreases, making it more difficult to identify elements at higher resolution.
- If more time was available to make measurements, the signal to noise ratio could be improved.

NEXT STEPS

In this experiment, only monochromatic x-rays with one polarization were utilized, providing little information regarding the particles' magnetic spin. By comparing the scans from left and right circular polarized x-rays, we could determine the sample's magnetism. When superimposed, identical graphs would indicate no magnetism while differences in peaks indicate the difference in energy an electron gives off when hit with a particular x-ray (magnetism). The differing excitation levels and intrinsic spins of electrons are characteristics of magnetism. Possible applications include any elemental analysis requiring high precision at a nanoscale. For example, analyzing substances in medical diagnostics or substances in exhaust.

REFERENCES

- Volker R., Nozomi S., Michael B., Alex D., Tolulope A., Daniel R., Saw-Wai H., Mike F., and Ruben R. 2020. XTIP - the world's first beamline dedicated to the synchrotron X-ray scanning tunneling microscopy technique. Journal of Synchrotron Radiation 27: 836-843.
- *Figure 1.* Rose, V., & Shirato, N. (n.d.). *Schematic of Synchrotron X-Ray Scanning Tunneling Microscope (Sx-Stm)*.
- *Table 1.* Thompson, A. et al. (2001). *Electron binding energies, in electron volts, for the elements in their natural forms.* From *X-Ray Data Booklet.* Berkeley: Lawrence Berkeley National Laboratory.

ACKNOWLEDGMENT

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