

Aug 3rd, 9:00 AM - 12:00 PM

## Laser microraman study of reduced synthesized spinel powders

Alexandra J. Leandre  
*Bennett College for Women*

Michael M. Rodriguez  
*California Lutheran University*

Joseph Lussier  
*University of San Francisco, jelussier@usfca.edu*

Brittany Morgan  
*Seattle University*

Brian Hosterman  
*University of Nevada, Las Vegas, brian@physics.unlv.edu*

Follow this and additional works at: [https://digitalscholarship.unlv.edu/cs\\_urop](https://digitalscholarship.unlv.edu/cs_urop)  
See next page for additional authors

 Part of the [Biological and Chemical Physics Commons](#)

### Repository Citation

Leandre, Alexandra J.; Rodriguez, Michael M.; Lussier, Joseph; Morgan, Brittany; Hosterman, Brian; and Farley, John W., "Laser microraman study of reduced synthesized spinel powders" (2010). *Undergraduate Research Opportunities Program (UROP)*. 29.  
[https://digitalscholarship.unlv.edu/cs\\_urop/2010/aug3/29](https://digitalscholarship.unlv.edu/cs_urop/2010/aug3/29)

This Event is protected by copyright and/or related rights. It has been brought to you by Digital Scholarship@UNLV with permission from the rights-holder(s). You are free to use this Event in any way that is permitted by the copyright and related rights legislation that applies to your use. For other uses you need to obtain permission from the rights-holder(s) directly, unless additional rights are indicated by a Creative Commons license in the record and/or on the work itself.

This Event has been accepted for inclusion in Undergraduate Research Opportunities Program (UROP) by an authorized administrator of Digital Scholarship@UNLV. For more information, please contact [digitalscholarship@unlv.edu](mailto:digitalscholarship@unlv.edu).

---

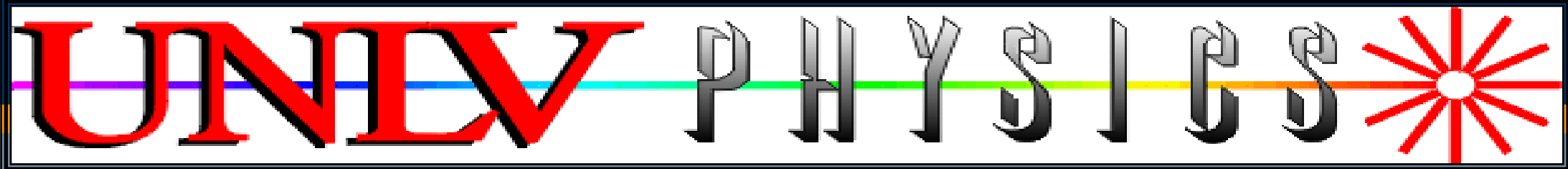
## **Presenters**

Alexandra J. Leandre, Michael M. Rodriguez, Joseph Lussier, Brittany Morgan, Brian Hosterman, and John W. Farley



# Laser Microraman Study of Reduced Synthesized Spinel Powders

Alexandra J. Léandre (Bennett College for Women), Michael M. Rodriguez (California Lutheran University), Joseph Lussier (University of San Francisco),  
Brittany Morgan (Seattle University), Brian D. Hosterman (University of Nevada, Las Vegas), John W. Farley (University of Nevada, Las Vegas)



Department of Physics and Astronomy, UNLV, Las Vegas, Nevada USA, 89154-4002

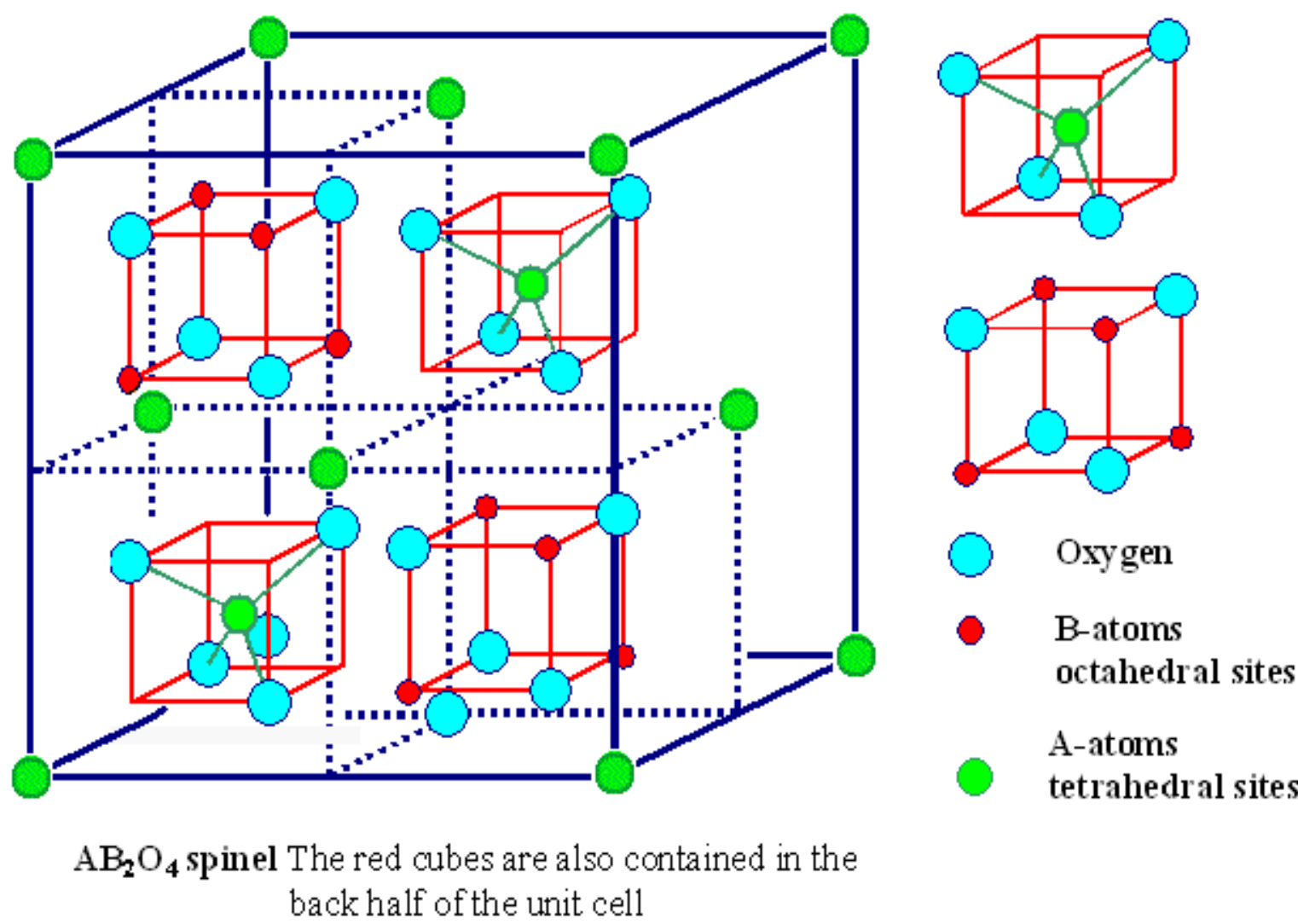
## Abstract

The Raman effect is the excitation or de-excitation of vibrational modes resulting from the inelastic scattering of light from a gas, liquid, or solid with a shift in energy from that of the usually incident radiation.

Raman microscopy was performed on synthesized spinel powders of solid solution  $\text{Fe}_x\text{Cr}_{3-x}\text{O}_4$  to determine the dependence of the vibrational modes upon the metal cations. The powders were synthesized in a combustion reaction using metal nitrates and urea. The oxide powders were reduced in a hydrogen/argon gas flow at high temperature.

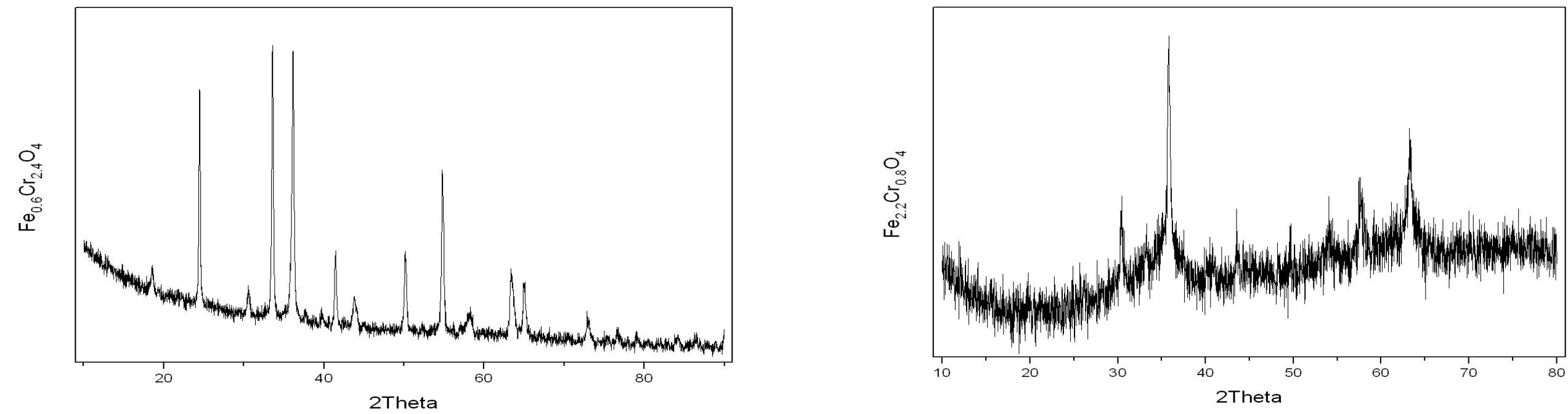
## Spinel Structure

The spinel structure is named after the mineral spinel ( $\text{MgAl}_2\text{O}_4$ ); the general composition is  $\text{AB}_2\text{O}_4$ . It is essentially cubic, with the O<sup>-</sup> ions forming a fcc lattice. The cations (usually metals) occupy 1/8 of the tetrahedral sites and 1/2 of the octahedral sites and there are 32 O<sup>-</sup>ions in the unit cell.

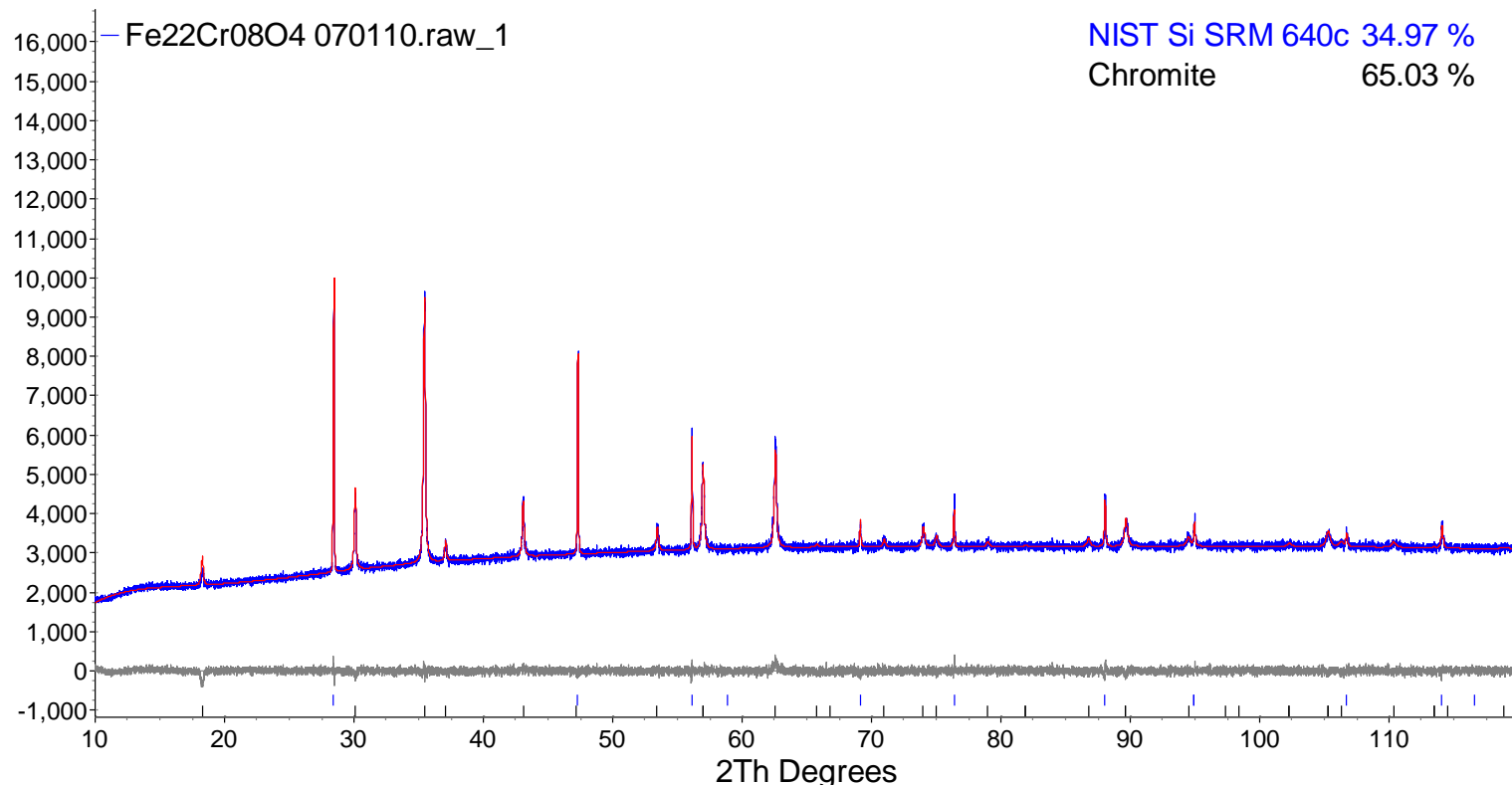


## Spinel Synthesis

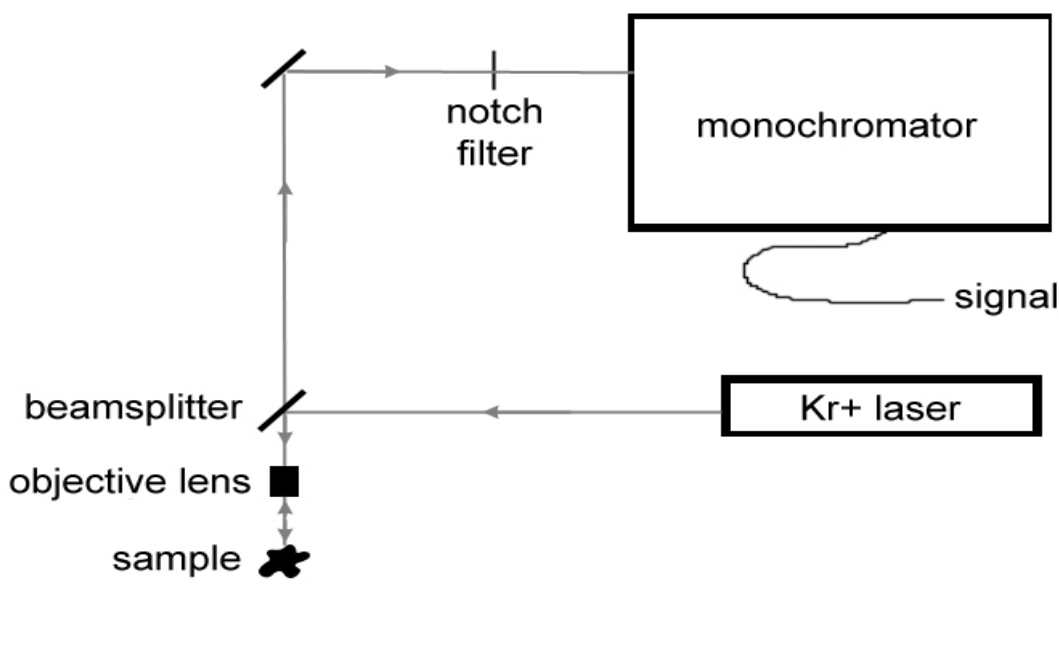
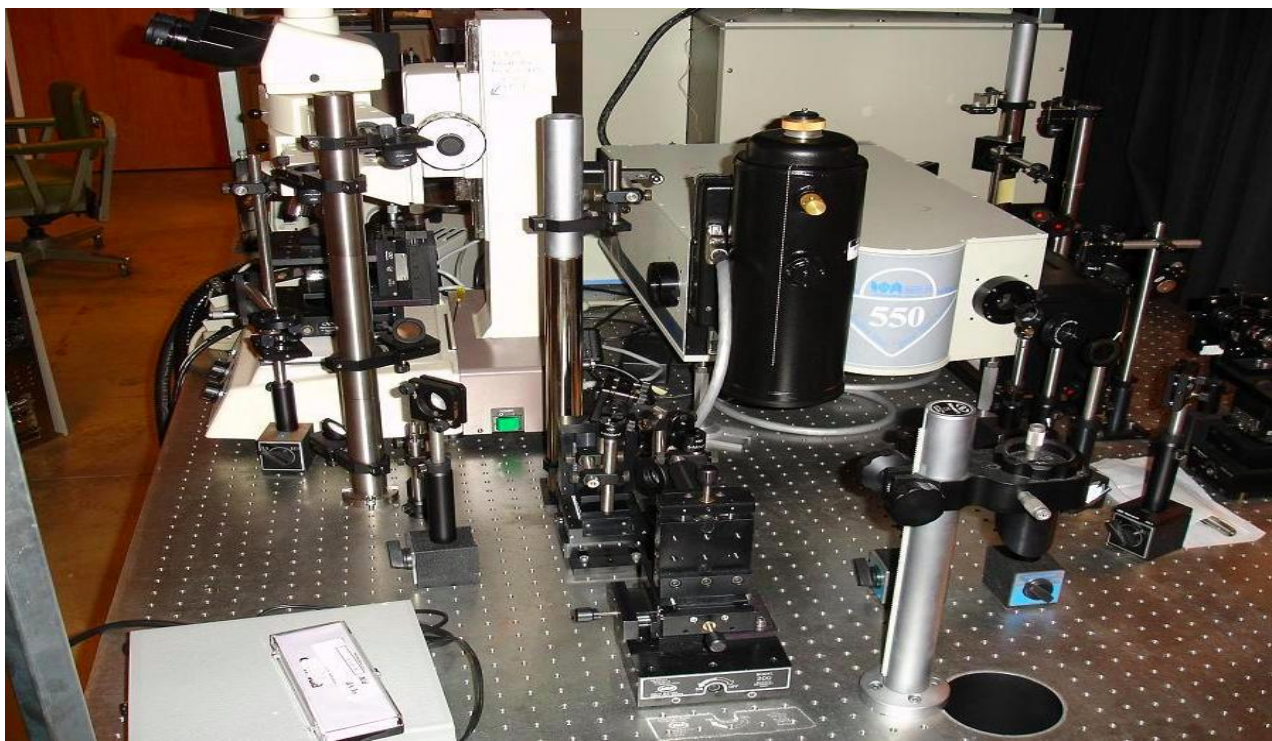
Iron Chromite spinel powders ( $\text{Fe}_x\text{Cr}_{3-x}\text{O}_4$ ) were synthesized in a combustion reaction. After the combustion reaction, XRD showed poor crystal structure, or even the corundum structure, instead of the desired spinel. Due to the high heat at which they were synthesized, the spinel ( $\text{Fe}_3\text{O}_4$ ) form tended to oxidize down to the corundum ( $\text{Fe}_2\text{O}_3$ ) structure.



To convert the samples to the spinel phase, the samples were heated to 700C in a reducing environment of 2% H<sub>2</sub> / 98% Argon gas flow.

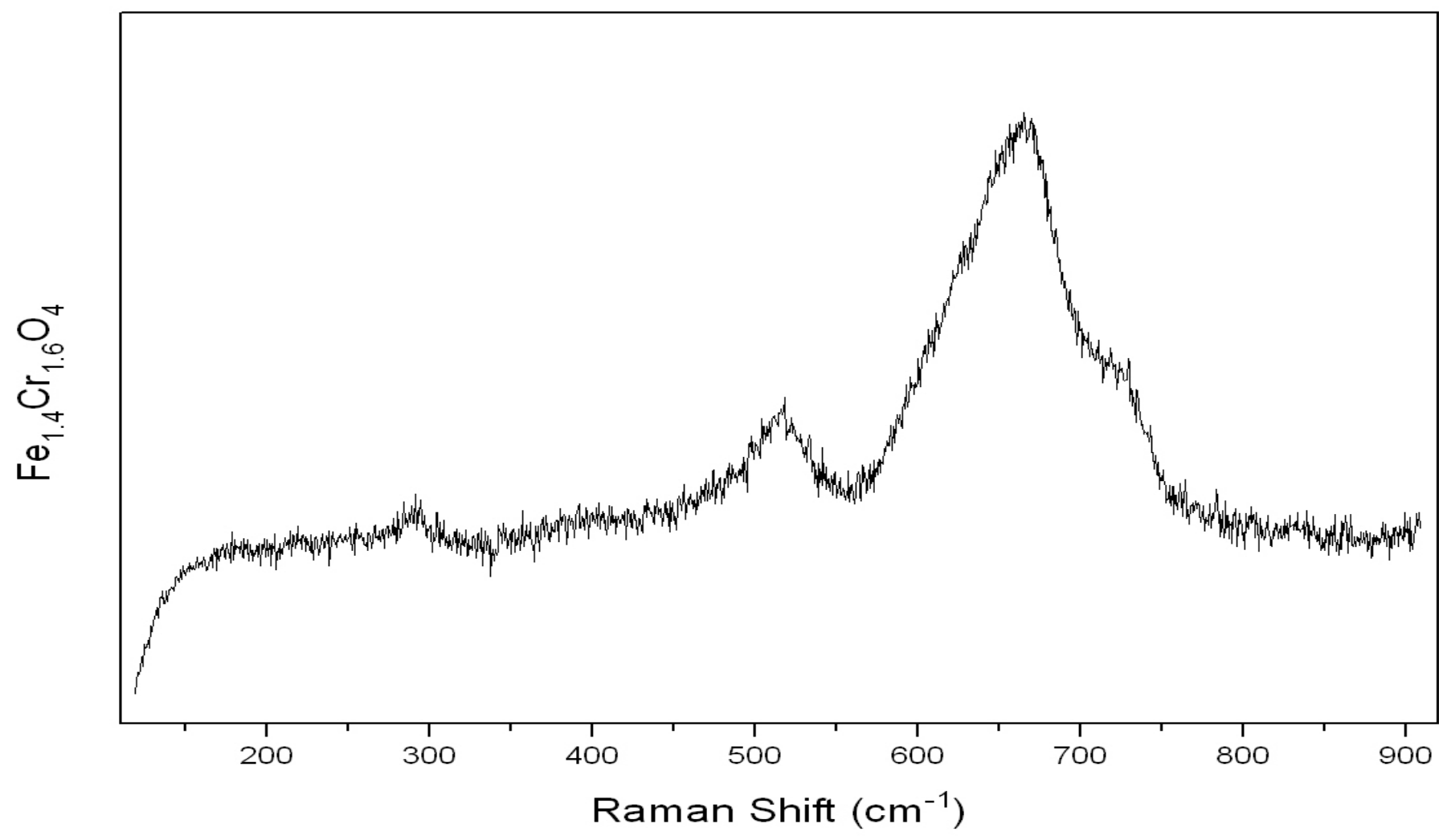


## Raman Microscope



This is the micro-Raman spectrometer used to collect all of our spectra.

## Raman Data



A typical Raman spectrum of an iron chromite

The peaks of the Raman spectra were then fit using PeakFit software to determine the positions of the Raman peaks. For many samples, only the A<sub>1g</sub> mode was visible.

Numerous Raman spectra were acquired on each sample at different spots and varying laser intensities to check uniformity of the powder. For the most part, all the spectra were consistent at similar laser powers and at different spots on the sample.

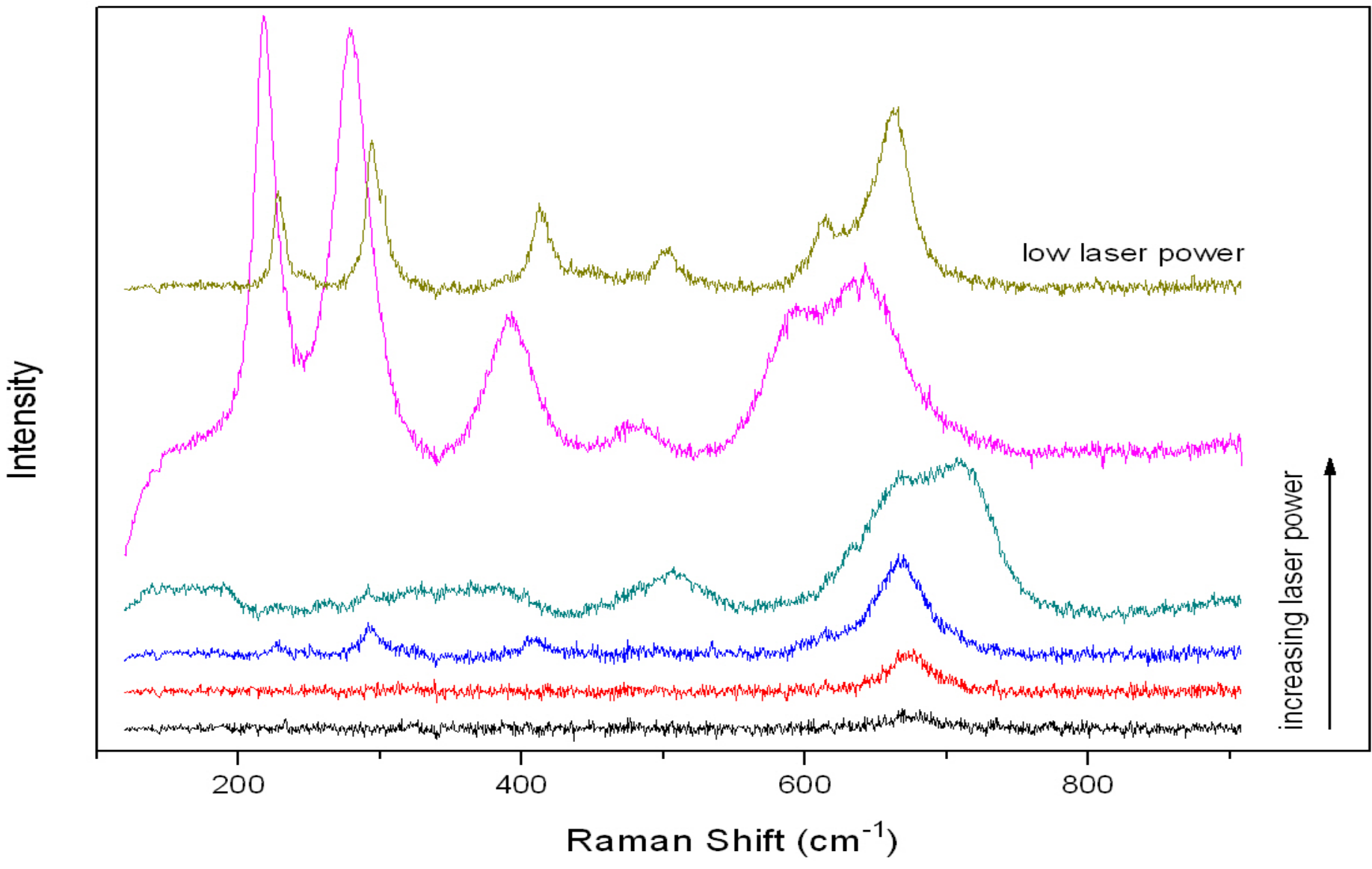
FeCr <sub>2</sub> O <sub>4</sub>	536	679
Fe <sub>1.2</sub> Cr <sub>1.8</sub> O <sub>4</sub>	---	678
Fe <sub>1.4</sub> Cr <sub>1.6</sub> O <sub>4</sub>	522	674
Fe <sub>1.6</sub> Cr <sub>1.4</sub> O <sub>4</sub>	---	672
Fe <sub>1.8</sub> Cr <sub>1.2</sub> O <sub>4</sub>	517	674
Fe <sub>2</sub> CrO <sub>4</sub>	536	671
Fe <sub>2.2</sub> Cr <sub>0.8</sub> O <sub>4</sub>	---	675
Fe <sub>2.4</sub> Cr <sub>0.6</sub> O <sub>4</sub>	---	673
Fe <sub>2.6</sub> Cr <sub>0.4</sub> O <sub>4</sub>	---	674

This table displays the Raman bands for Fe<sub>x</sub>Cr<sub>3-x</sub>O.

## Laser Intensity

A major problem we ran into when taking the data was that the power density of our laser spot, was too great and caused heating of the sample, which shifts the energy of the Raman modes to lower energy (wavenumber). For these iron chromites, it also converts the spinel phase to the corundum phase.

This conversion to corundum is permanent, remaining even after the intensity was reduced. We realized that it was necessary to use very low intensity and slowly remove a few strong neutral density filters to avoid oxidizing the sample.



This series of Raman spectra shows the conversion of the spinel phase to  $\gamma\text{-Fe}_2\text{O}_3$  and finally to corundum phase  $\alpha\text{-Fe}_2\text{O}_3$

## Conclusions

The reduction of the chromite powders was successful in producing high crystalline spinel phase samples which was confirmed using XRD.

The Raman position of the A<sub>1g</sub> mode shows no obvious trend on the amount of iron within the iron chromite. Literature shows that the lattice constant change is not linear for  $\text{Fe}_x\text{Cr}_{3-x}\text{O}_4$  and so the dependence upon cation exchange may not be obvious.

However, forms of iron oxides are common corrosion products of stainless steel in high temperature environments, and Raman spectroscopy provides a useful tool for identification of these products.

## Acknowledgements

Support from the REU program of the National Science Foundation under grant DMR-1005247 is gratefully acknowledged.

We also acknowledge Dr. Thomas Hartmann for assistance with XRD data.

## References

J. Preudhomme and P. Tarte, *Spectrochim. Acta* 27A, 845 (1971).  
Brian D. Hosterman. Micro-Raman Spectroscopic Study of the Corrosion of Stainless Steel by Lead-Bismuth Eutectic, 13 (2001).  
S.C. Abrahams and B.A. Calhoun, *Acta Cryst.* 6, 105 (1953).  
P.F. McMillian and A.M. Hofmeister. *Infrared and Raman Spectroscopy*. 109 .  
H.J. Levinstein, M. Robbins, and C. Capio, *Matter. Res. Bull.* 7, 27 (1972).