Santa Rosa Junior College

Solution Synthesis of Magnetic Nanoparticles

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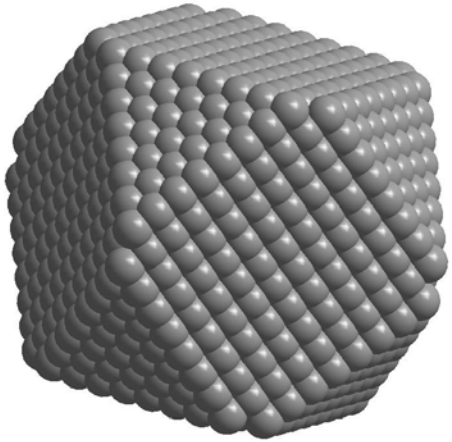

CALIFORNIA NANOSYSTEMS INSTITUTE

INSET Internships in Nanosystems Science, Engineering and Technology

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Solution Synthesis of Magnetic Nanoparticles

Project Overview:

- A project of the Cheetham and Seshadri groups at the Materials Research Laboratory, under the mentorship of Katharine Page.
- Research focuses on the synthesis and characterization of magnetic nanoparticles.
- At a fundamental level, the research examines how the size of nanoparticles affects their properties.
- A number of potential applications for magnetic nanoparticles exist, such as those in medical imaging, data storage, and catalysis. Synthesis occurs at relatively low temperatures in solution, thus allowing a highly scalable method of production.
- Funding provided through the following NSF programs: The Chemical Bonding Center, Graduate Student Fellowship, and the Faculty Career Award.

Solution Synthesis of Magnetic Nanoparticles

- We are currently looking at what parameters affect the size and morphology of the synthesized nanoparticles. Some of the parameters include the reaction time, reaction temperature, and the type of capping agent used.

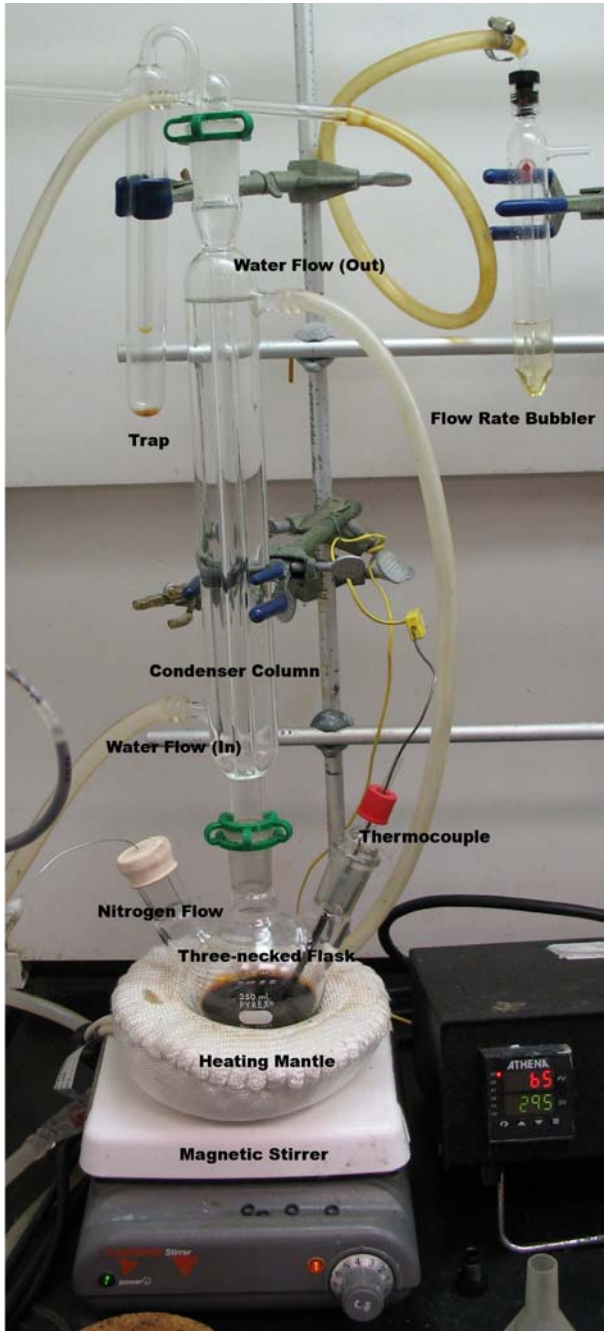
- Many metals, including nickel, are most stable in the face-centered cubic (fcc) phase. One of the systems we are working with, cobalt oxide, is most stable in the rock-salt phase.

- Powder X-ray diffraction (XRD) is the principle means of characterization. A superconducting quantum interference device (SQUID) magnetometer will be used for collecting magnetic data, and electron microscopy will be used for imaging.

- Our group has previously prepared wurtzite cobalt oxide. Further effort will be directed at preparing additional energetically trapped, meta-stable materials, such as hexagonally close-packed nickel.

Solution Synthesis of Magnetic Nanoparticles

- A glovebox is used for handling the starting materials.
  1) The precursor consists of 1 g (3.9 mmol) of cobalt(II) acetylacetonate OR $Ni(acac)_2$, added to a three-necked flask.
  2) For the solvent, 40 mL (210 mmol) of dibenzyl ether is added.
- The solution is then allowed to reflux for a specific amount of time.
- Next, the nanoparticles are washed several times in ethanol.
- Upon drying, the sample is then ground using a mortar and pestle for later characterization.



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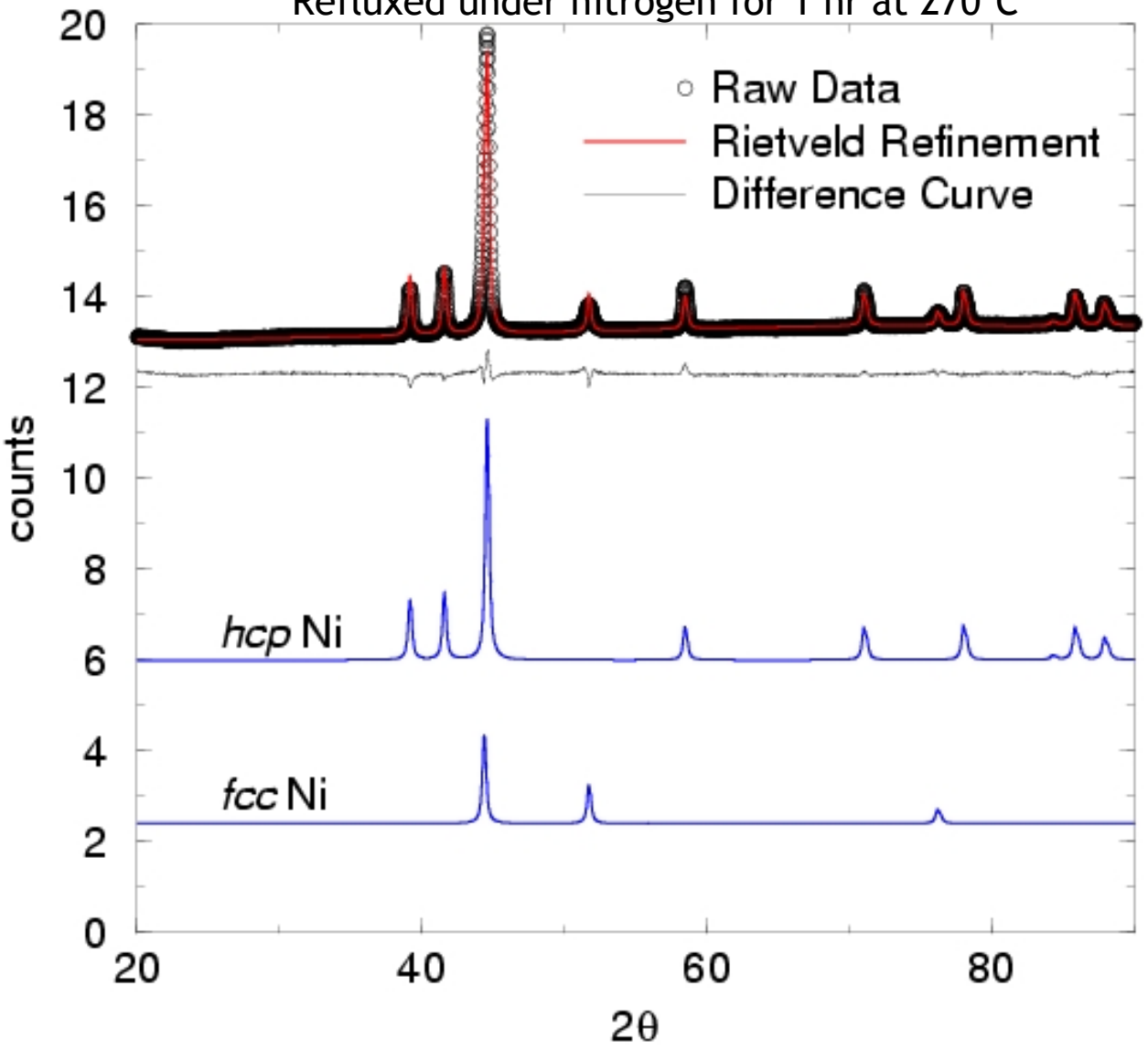
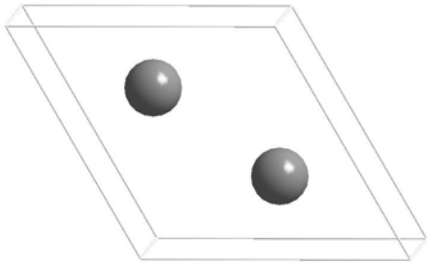
Powder X-Ray Diffraction Data for Ni Nanoparticles

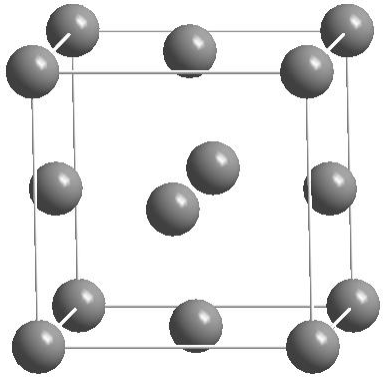
Refluxed under nitrogen for 1 hr at 270°C



A particle size of **~30 nm** was calculated via Scherrer broadening



hcp Ni: *$P6_3/mmc$*



fcc Ni: *Fm-3m*

We are forming both the fcc and hcp Ni phase with our solution synthesis.

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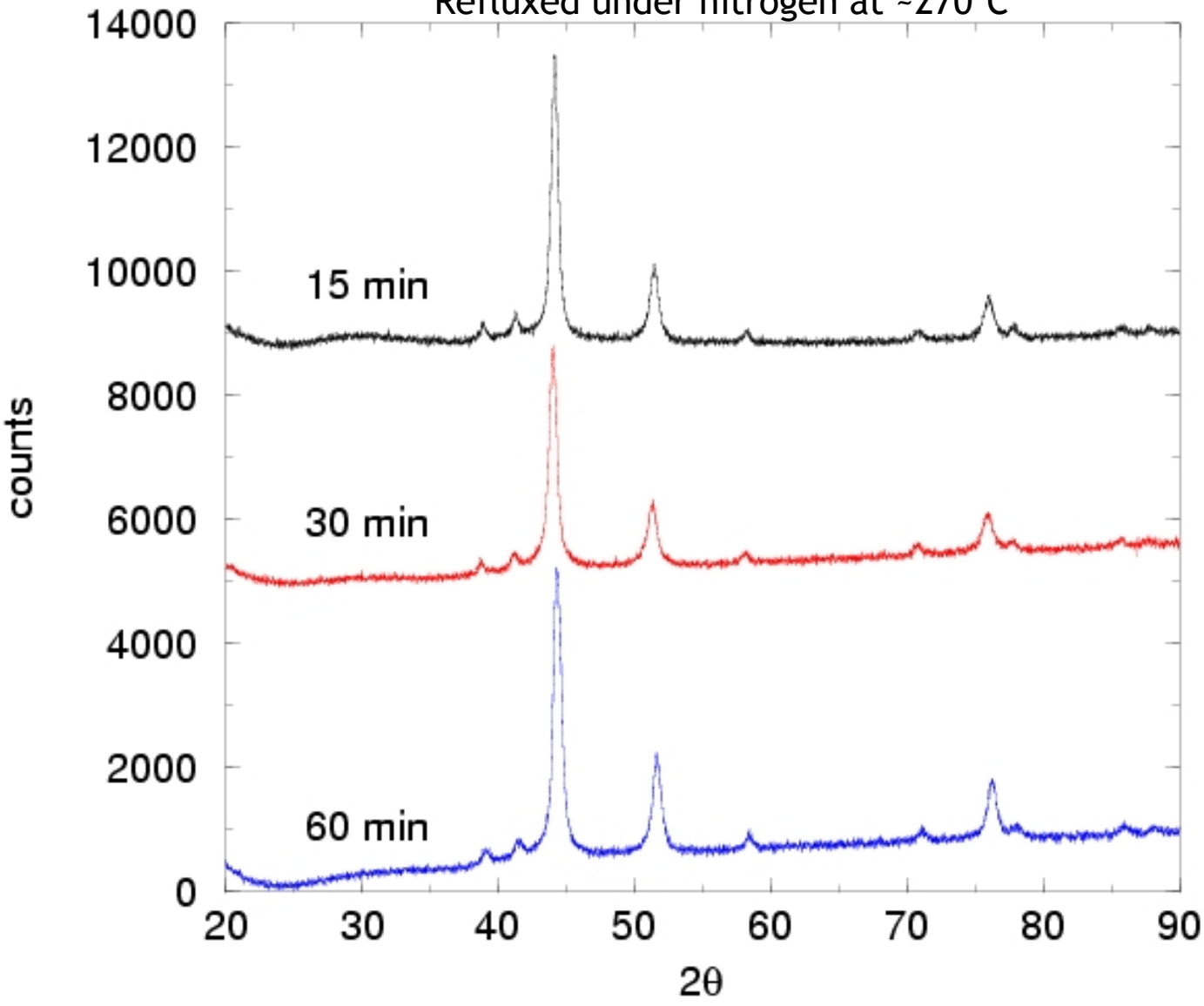
Effect of Reflux Time on Morphology for Ni Nanoparticles

Refluxed under nitrogen at ~270°C



The broadening of the peaks and the relative phase amounts are not affected by reflux time.

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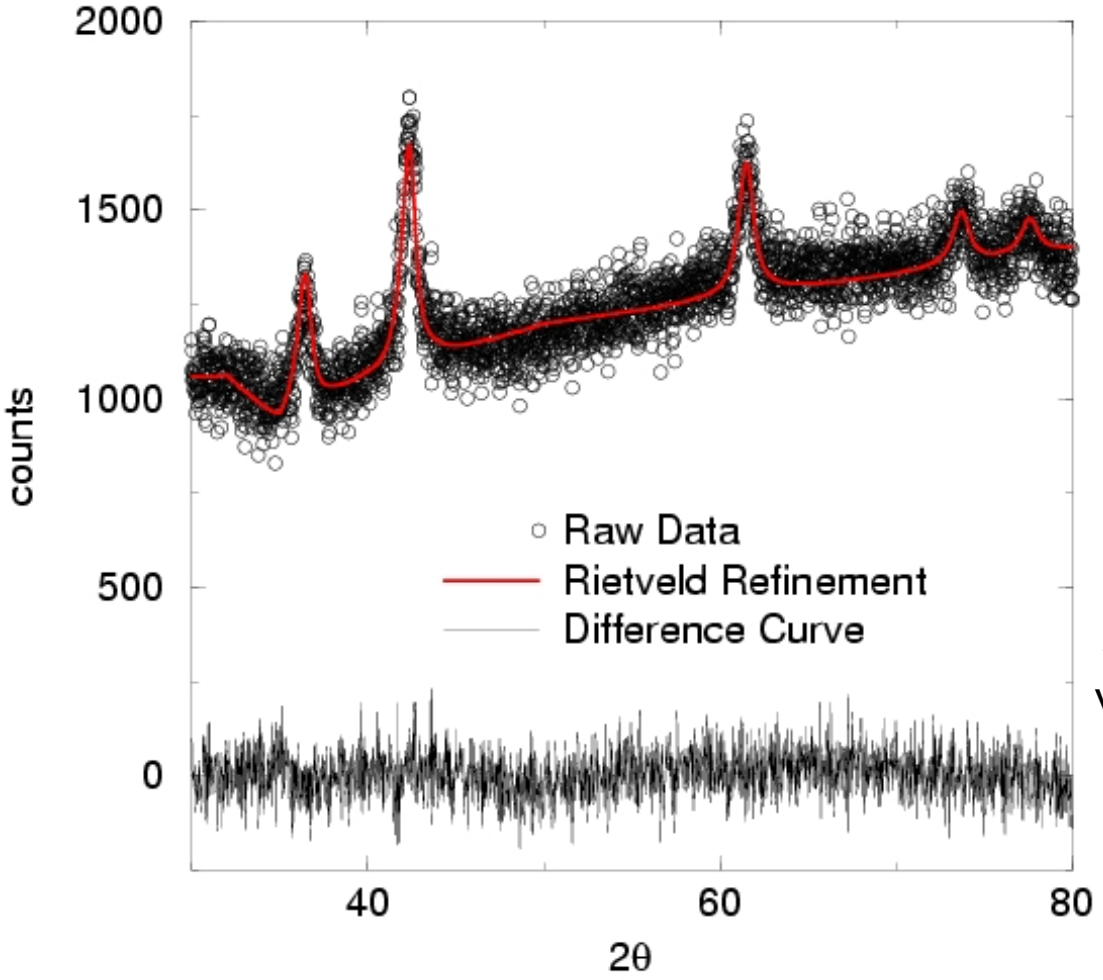
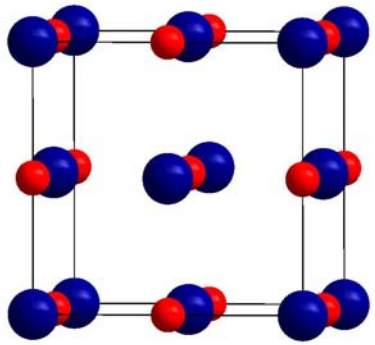
Powder X-Ray Diffraction Data for CoO Nanoparticles

Refluxed under nitrogen for 1 hr at 273ºC
(with capping agent)





rock-salt CoO

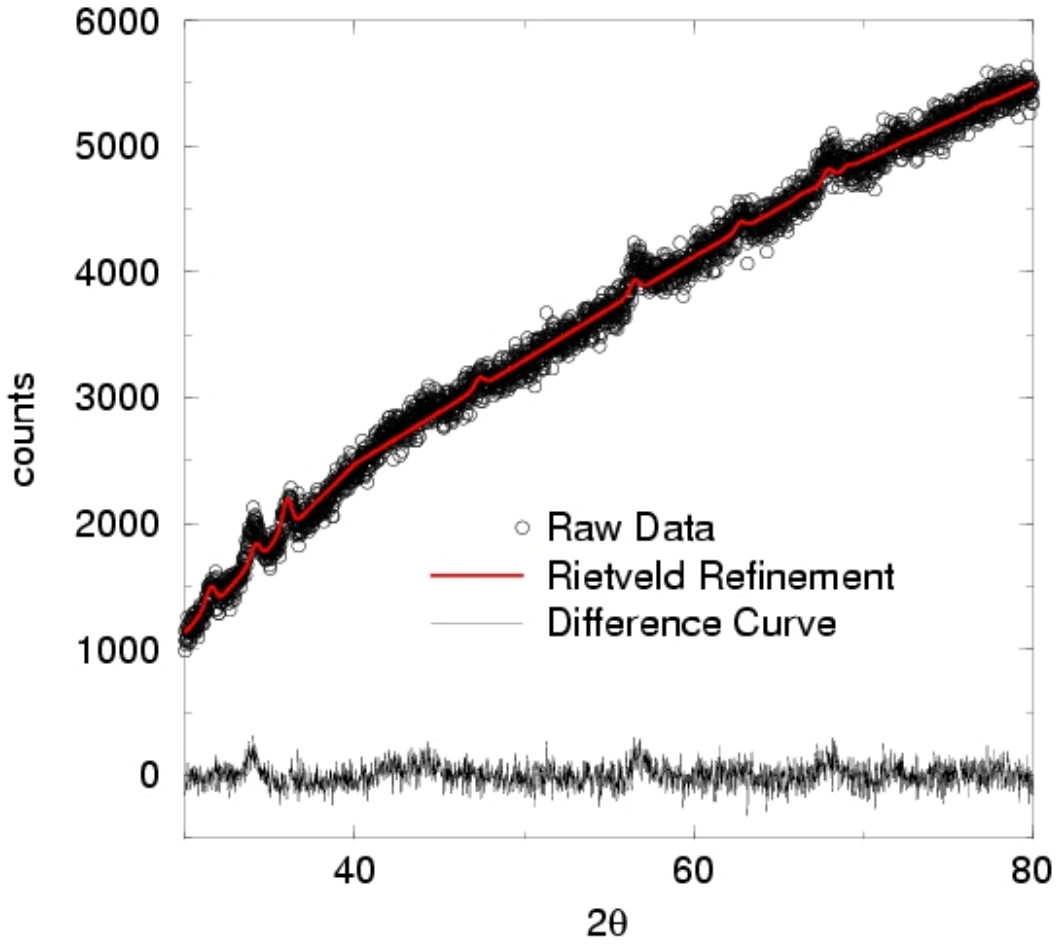
A particle size of
~10 nm was calculated
via Scherrer broadening

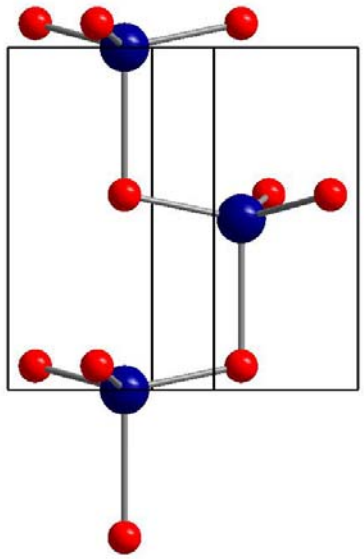
These experimental parameters allow us to prepare clean, rock-salt phase CoO nanoparticles.

Solution Synthesis of Magnetic Nanoparticles

Powder X-Ray Diffraction Data for CoO Nanoparticles

Refluxed under nitrogen for 1 hr at 271°C
(without capping agent)





***wurtzite* CoO**

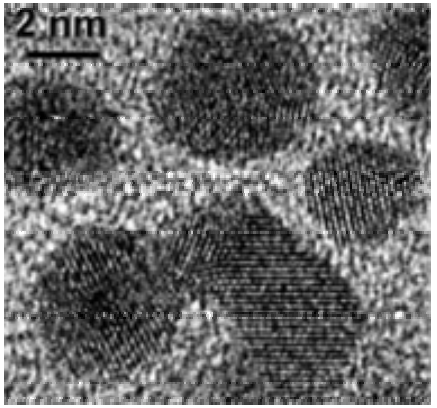
A particle size of
~10 nm was calculated
via Scherrer broadening

We can selectively form CoO nanoparticles in either the rock-salt or *wurtzite* modification by introducing a capping agent during the reflux.

Solution Synthesis of Magnetic Nanoparticles

Future Plans

- Samples will be taken to a synchrotron x-ray source for further characterization.
- Novel diffraction techniques will be explored.
- Transmission electron microscopy (TEM) will allow us to compare the actual sizes of the nanoparticles to our calculated values.



Katharine Page

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Solution Synthesis of Magnetic Nanoparticles

Acknowledgements

- Funding provided through the following NSF programs:
  Chemical Bonding Center
  Graduate Student Fellowship
  Faculty Career Award

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- The University of California, Santa Barbara and the staff of the Materials Research Laboratory (MRL).

And…

a big thanks goes to the Cheetham and Seshadri Groups, and my mentor, Katharine Page!