A. Title Page

Growth and Characterization of Magnetic Nanoparticles
Final Report

Dr. Daniel Bullock
Department of Physical Science
McEver Room 03
Ph: 479-968-0230
daniel.bullock@atu.edu

B. Restatement of problem researched or creative activity

Quantum dots are nanometer (10^{-9} meter) scale particles that are neither small molecules nor bulk solids. Their composition and small size (a few hundred to a few thousand atoms) give these dots extraordinary optical, chemical, electrical, and magnetic properties that cannot be achieved by their bulk counterparts. Most of the work in this area has been focused upon the II – VI semiconductors (so called because they are composed of elements from columns, II and VI on the periodic table) due to their potential as biomedical sensors. Unfortunately little work has been conducted upon the fabrication of uniform oxide nanoparticles despite their many important technological applications. The fabrication of patterned media arrays of discrete single domain magnetic nanoparticles is very important for their potential applications in multi-terabit/in^2 magnetic memory devices. Such magnetic nanoparticles could also find applications in ferrofluids, refrigeration systems, medical imaging, drug targeting, and catalysis. The syntheses of several uniform-sized magnetic metal nanoparticles has been reported. However, relatively little work has been done on the fabrication of monodispersed (i.e. single size distribution) and crystalline magnetic oxide nanoparticles. Several magnetic oxide nanoparticles have been synthesized by using micro-emulsion and other methods. However, particle size uniformity and crystallinity of these nanoparticles are poor. Although the syntheses of relatively uniform maghemite and magnetite nanoparticles have been reported, exhaustive size selection procedures were necessary. What is desired is a novel process to synthesize these particles which demonstrate both a high degree of crystalline quality as well as a monodispersion. Our proposal is a detailed study of the effect that different heating rates have on these crystals.

C. Brief review of the research procedure utilized

An important component of our project was the design and construction of a high precision reactor vessel. Figure 1 shows a picture of the reactor vessel that was constructed. The reactor vessel is where all the necessary chemical reactions take place in order to produce the nanocrystals. In order to produce a highly uniform sample the

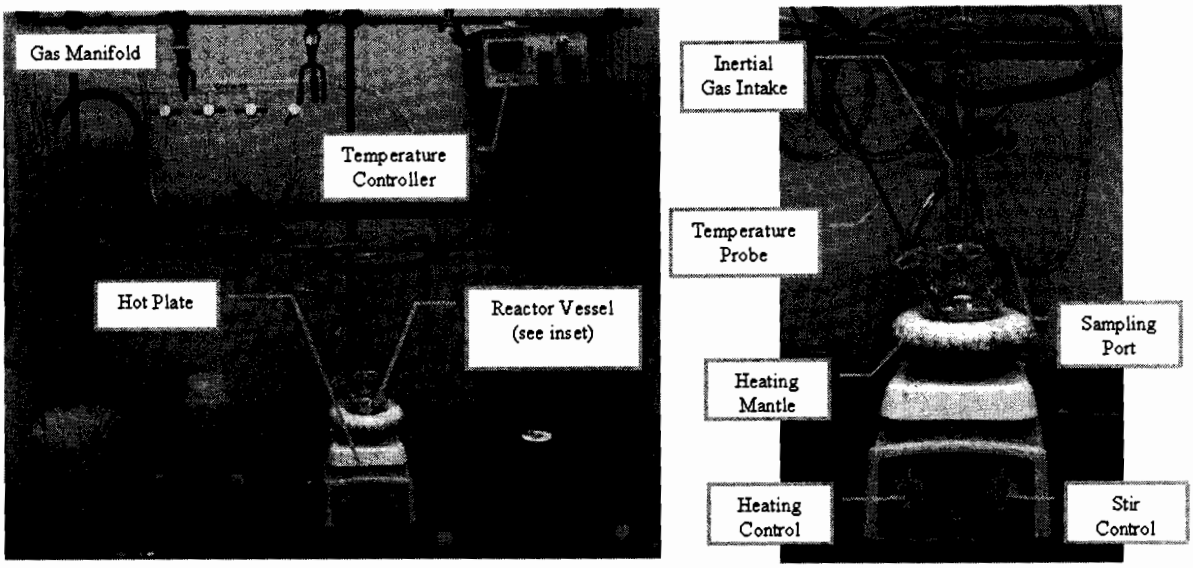

Figure 1 Experimental setup for nanocrystal synthesis.

reactor vessel must (1) have a high degree of temperature uniformity during the sample synthesis process, (2) not be reactive with any of the chemicals, and finally (3) be accessible for sample withdrawal during the reaction. The temperature uniformity was achieved by encasing the reactor vessel with thermal isolating foam (not shown). In addition to keeping the temperature uniform it also allowed us for more precise temperature control during the reaction.

The needed chemicals for this study included Stearic Acid (95%), 1-Octadecene (90%), Iron (II) Stearate (Fe 9%) and n-tetracosane.

The Fe_3O_4 nanocrsytals are generally prepared by the decomposition of iron stearate however, when this project began we had trouble finding a vendor that sold iron stearate in the proper form. We contacted ten chemical vendors and all of them had discontinued carrying the crystalline form of iron stearate that we needed for this experiment and special ordering this material was well outside the budget of this project. In an attempt to complete the project we spent a significant amount of time trying to synthesize the iron stearate "in house" but we were unsuccessful. Finally, we contacted a research group at the University of Arkansas – Fayetteville and they supplied us with a small amount of the crystalline form of iron stearate. Unfortunately, the number of samples we were able to produce were limited by our supply of the crystalline iron stearate.

The Fe_3O_4 nanocrsytals were then prepared by the decomposition of iron stearate in octadecene (ODE) with stearic acid ligand at 300 °C. 1.87gr iron stearate, 0.57gr stearic acid and 5gr ODE or tetracosane were combined in a 25mL 3-neck reaction flask under argon atmosphere and vigorous stirring conditions. The iron stearate dissolved at 60 – 70 °C along with the stearic acid. Formation of the first particles was observed at 250 °C.

D. Summary of findings

Unfortunately we were not able to run the experiment with the desired number of samples due to the limited amount of iron stearate we had. However we were able to take data for two different heating rates (10 °C/min and 20 °C/min). We found that the size distribution of the nanocrystals were more uniform with the lower heating rates as compared to the higher heating rates. This limited data is consistent with a heat-transport limiting process. With the fast heating rate the system could not reach thermal equilibrium as the temperature increased thereby causing an uneven heat distribution.

This nonuniformity caused some crystals to nucleate at different times throughout the reaction which explains the multiple size distributions that were observed.

E. Conclusions and recommendations

In conclusion the differing heating rates significantly effect the size distributions. We would like to continue this work by looking at a larger sample set but we would need to either find a suitable distributor for the crystalline iron stearate or perfect the synthesis of it on house.