

## Fe Nanoclusters: Novel Solution Synthesis and Enhanced Magnetic Properties

by *D. L. Huber, E. L. Venturini, J. E. Martin, and P. P. Provencio*

**Motivation**—Structured superparamagnetic (i.e., monodomain) particle composites can produce susceptibility enhancements far superior to those achievable in micron scale, multi-domain particles. Micron-scale multidomain particles have a susceptibility that is limited by classical physics to a maximum of 3 (MKS units are used throughout this report) for spheres due to the effect of the demagnetizing field. This limitation does not exist for superparamagnetic particles, and they can have particle susceptibilities that are much greater than 3. Such high susceptibility particles have a very strong response to even weak magnetic fields, and represent a situation where nanoparticles have properties that are unachievable in coarser materials. Our goal was to synthesize magnetic nanoparticles that have a very strong magnetic response to be used in the formation of field-structured nanocomposites.

**Accomplishment**—Our synthetic approach is based on the widely used decomposition of iron pentacarbonyl. The novelty of the approach is the surfactant system used. Studies with a number of strongly bound surfactants have resulted in decreased magnetic response, due to surface oxidation, disturbing the electronic structure of the surface atoms, or some other mechanism. This typically results in iron nanoparticles that have a saturation magnetization of 40-50% that of bulk iron (for 5-6 nm particles), due to strong electronic interactions with the surfactants. With this in mind, we chose to work with a weak surfactant, a  $\beta$ -diketone.  $\beta$ -diketones do have a history as adhesion promoters in bonds between metals and polymers, but have never been used to

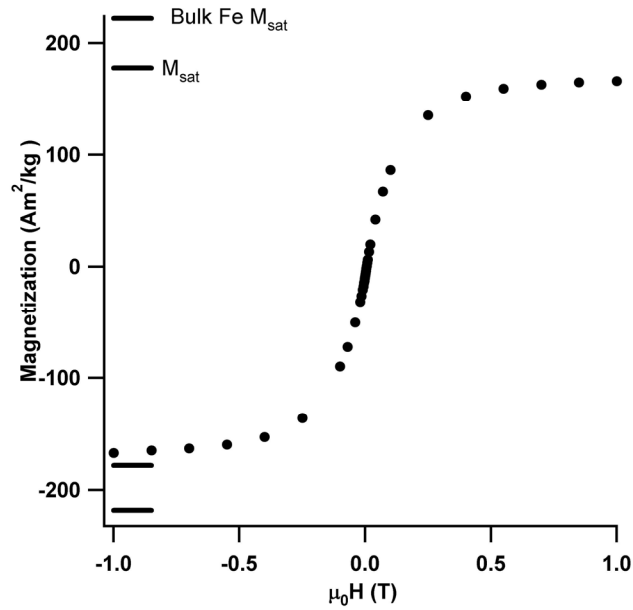
synthesize metallic nanoparticles. Synthesizing particles with this surfactant, we have achieved saturation magnetization values of greater than 80% that of bulk iron for approximately 5 nm particles (see Fig.1). Additionally we have observed very high susceptibilities, as high as 14 (at 150K), which far exceed the theoretical maximum of 3 for spherical multi-domain particles. This represents a sample of magnetic nanoparticles whose magnetic response is much higher than is possible in similar materials with larger dimensions. The magnetic susceptibility is so high that in the absence of an applied magnetic field the particles magnetically aggregate into coarse columnar structures (see Fig. 2). Although mechanical agitation can create a very fine dispersion, and both TEM and magnetic measurements confirm that the sample is made up of discrete nanoparticles, agglomerates quickly form in solution leading to a precipitate. The ease with which these particles magnetize and form structures in the absence of an applied magnetic field, implies that the application of an external field will readily structure the nanoparticles.

**Significance**—The synthetic procedure presented here represents a substantial step forward in the ability to synthesize highly magnetic iron nanoparticles. These particles are highly crystalline bcc iron, and are free of detectable oxide. This synthesis seems particularly useful in the 5-10 nm size range, which is an important size iron nanoparticles, as it represents the lower limit of sizes where iron particles are expected to strongly structure under an applied magnetic field at room temperature.

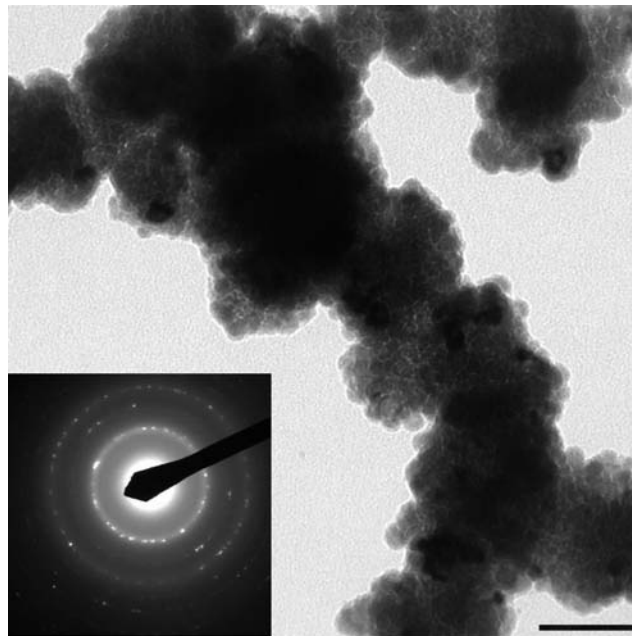
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**Sponsors for various phases of this work include:** DOE Office of Basic Energy Sciences and Laboratory Directed Research & Development

**Contact:** Dale L. Huber; Nanostructure & Semiconductor Physics, Dept. 1112  
Phone: (505) 844-9194, Fax: (505) 844-1197, E-mail: [dlhuber@sandia.gov](mailto:dlhuber@sandia.gov)



**Figure 1.** Magnetization versus field for an iron nanoparticle sample at 150 K. The sample is below its blocking temperature and therefore shows a weak but detectable coercivity (5.6 mT). Saturation magnetization is determined by extrapolating to infinite field and is 178 A m<sup>2</sup>/kg, or greater than 80% of the value of bulk iron.



**Figure 2.** TEM micrograph of approximately 6nm iron nanoparticles, showing magnetic agglomeration. The scale bar represents 60 nm. Inset diffraction pattern verifies that this sample is pure BCC Fe with diffraction rings at 0.8, 0.9, 1.0, 1.2, 1.4, and 2.0 Å.