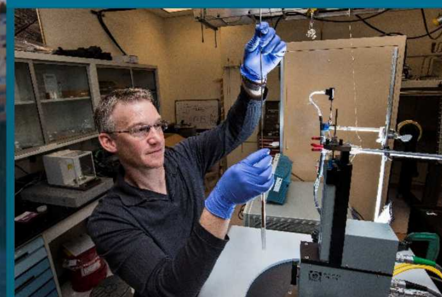
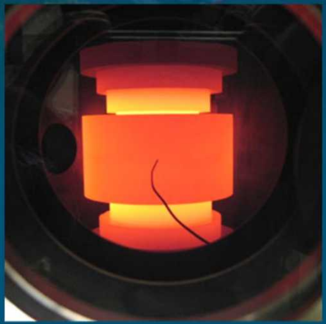
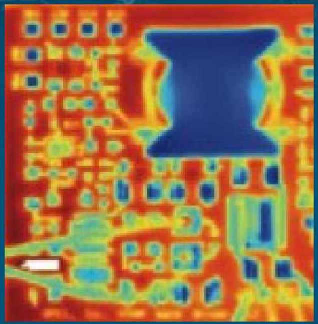
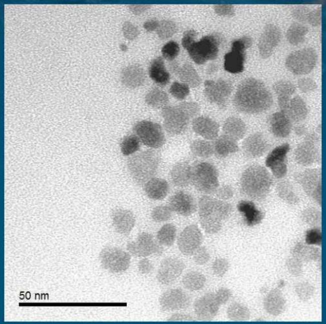
SAND2020-8708PE

Magnetic materials and ferroelectric nanoparticle research at Sandia









Sandia – UPRM Research Discussion

Todd C. Monson, Sandia National Labs,
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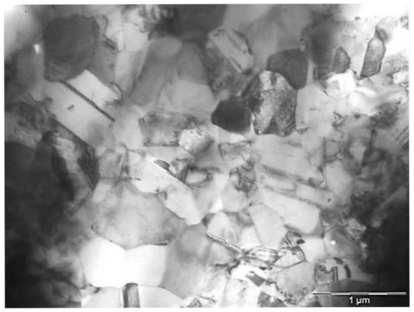
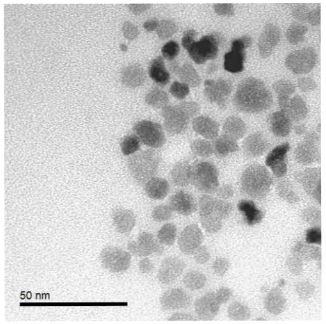
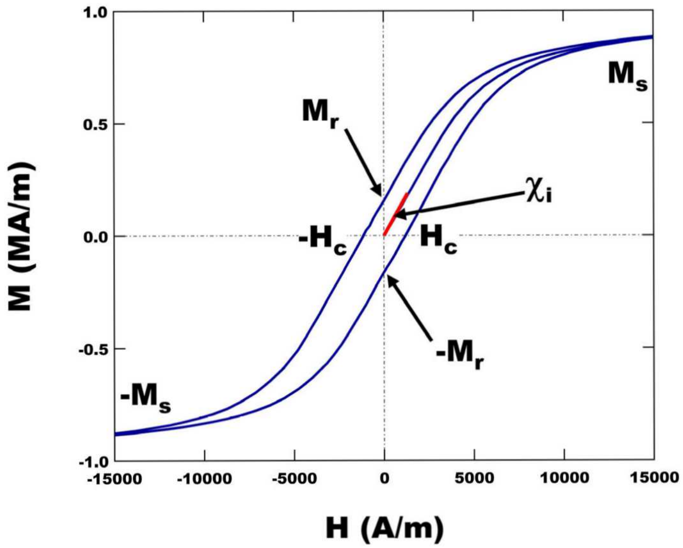
Activities in Monson Labs

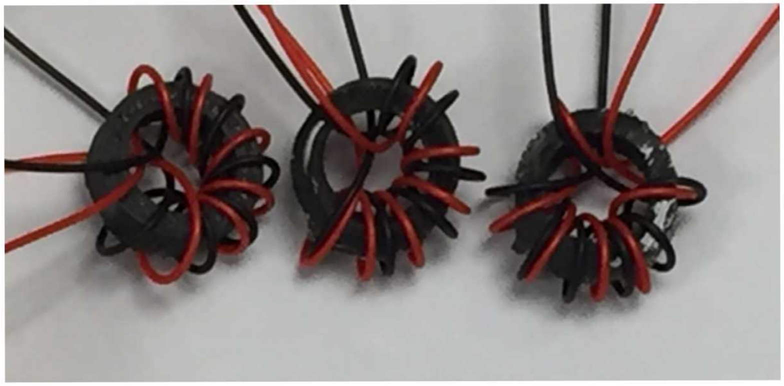




Synthesis and characterization of magnetic and ferroelectric nanoparticles, composites, and bulk materials



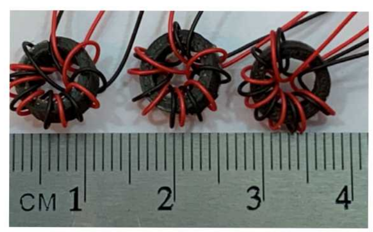
Magnetic characterization of materials and devices



Research Capabilities – Magnetic Characterization

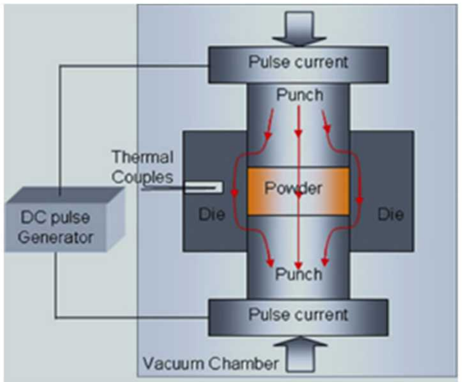
Quantum Design Magnetic Property Measurement System (MPMS)

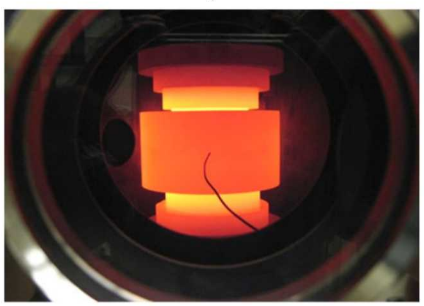
Iwatsu B-H analyzer (measurements up to 10 MHz and 150 °C)



- LCR meter
- Impedance analyzer

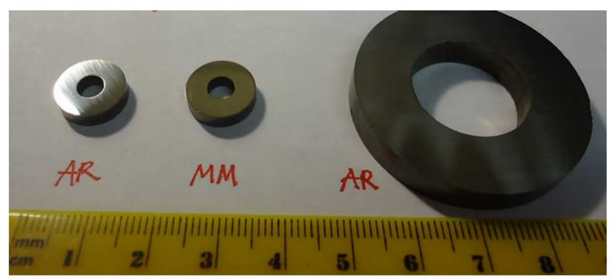
Research Capabilities – Ferroic Material Synthesis





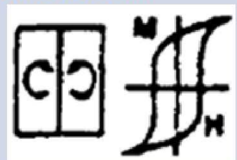
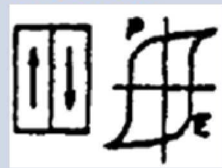
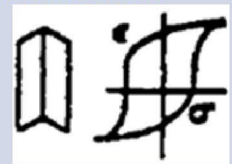
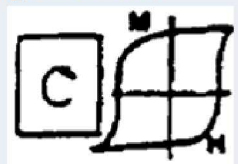
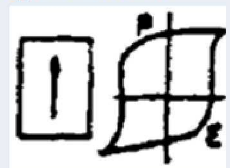
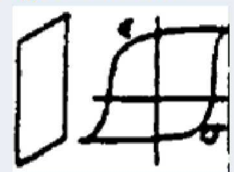
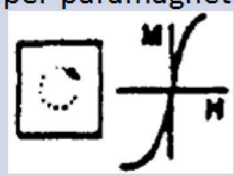
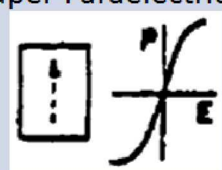
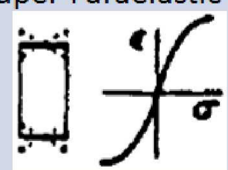
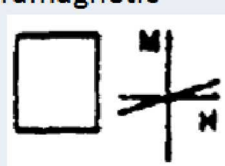
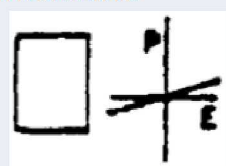
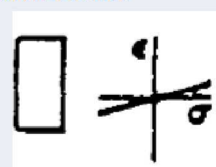
Spark Plasma Sintering (SPS)
(in collaboration with UC Irvine)

- Inert gas gloveboxes
- Fume hoods equipped with Schlenk lines for air free chemistry
- Solvothermal reaction vessels
- Electron microscope for raw powder characterization
- Hot press



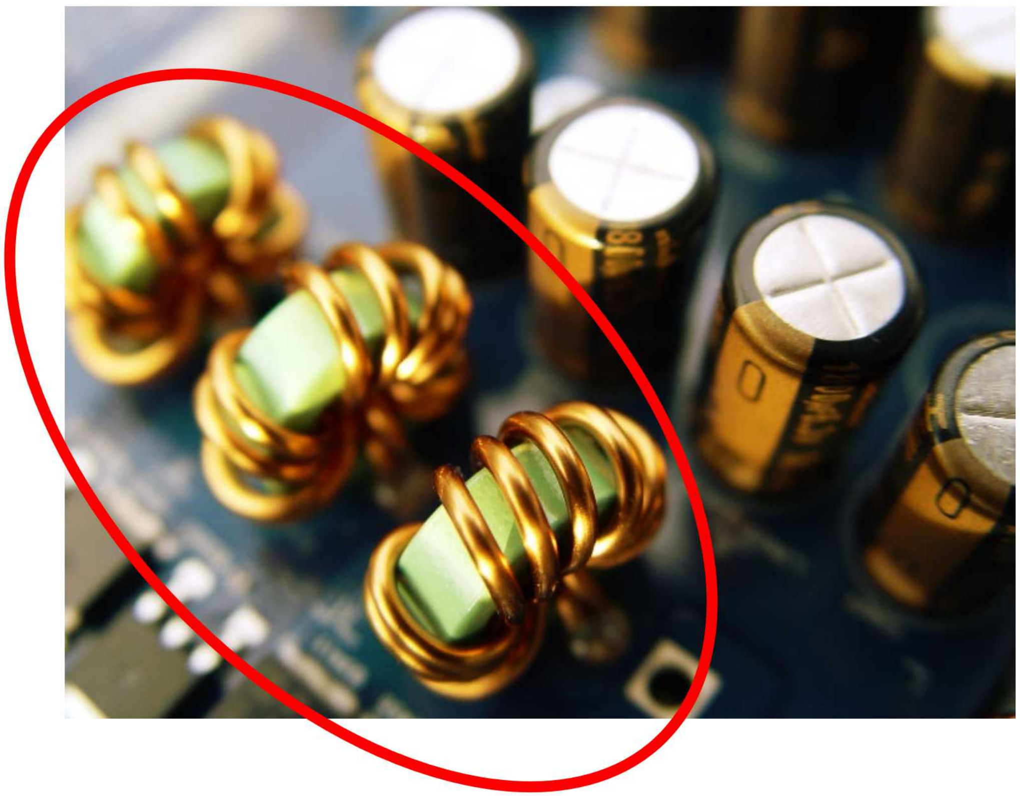
γ'-Fe_4N toroidal inductive cores

Ferroics

Size	Ferromagnetic	Ferroelectric	Ferroelastic
≈1µm-10^3 nm	Multidomain 	Multidomain 	Multidomain 
≈0.1µm-10^2 nm	Single Domain 	Single Domain 	Single Domain 
≈10nm-10^3 Å	Super-paramagnetic 	Super-Paraelectric 	Super-Paraelastic 
≈1nm-10 Å	Paramagnetic 	Paraelectric 	Paraelastic 

Newnham, R.E. "Size Effects in Ferroics", *Integrated Ferroelectrics*, 1998, Vol.20, pp. 1-13

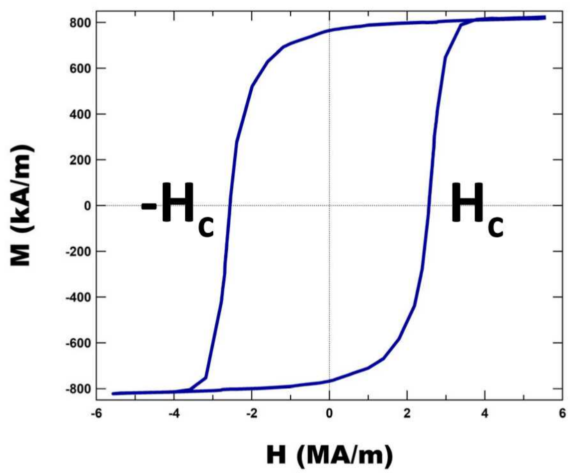
Soft Magnetics

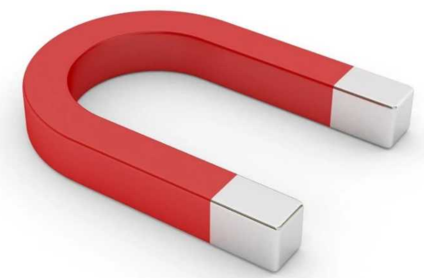


Hard vs. Soft Magnets

Hard (permanent) magnet

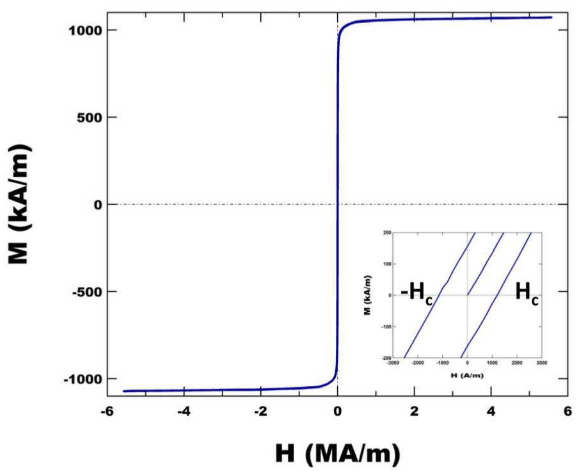
H_c > 1000 A/m

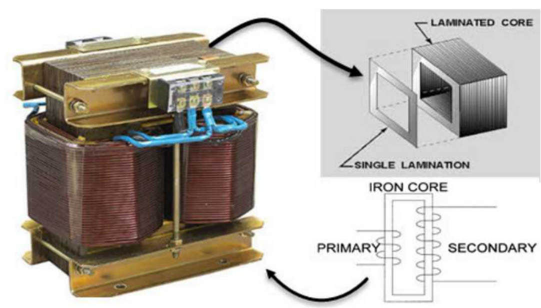


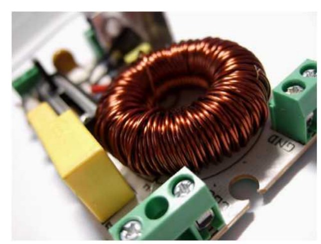


Soft magnet

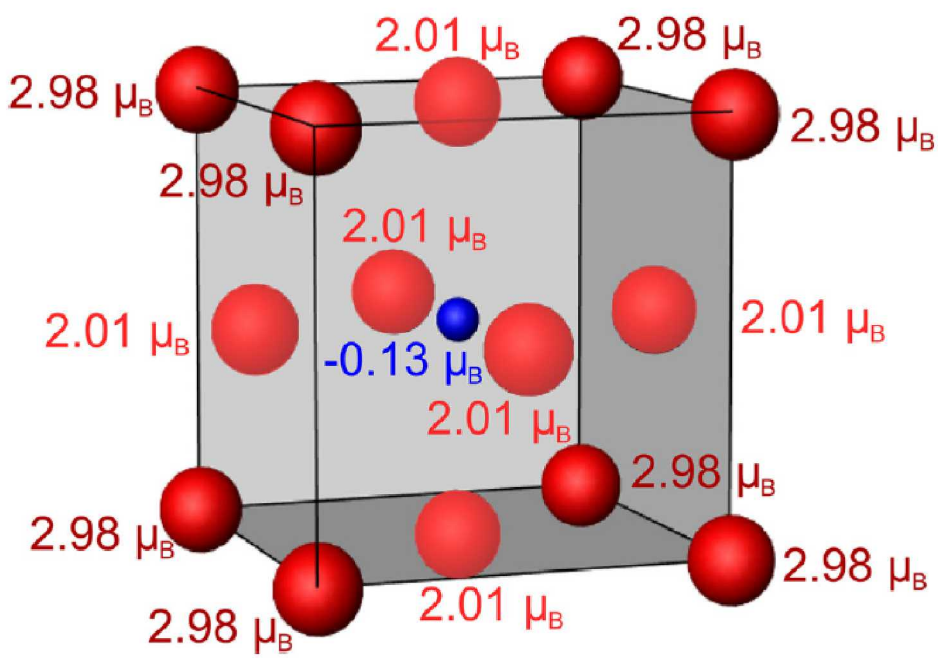
H_c < 1000 A/m



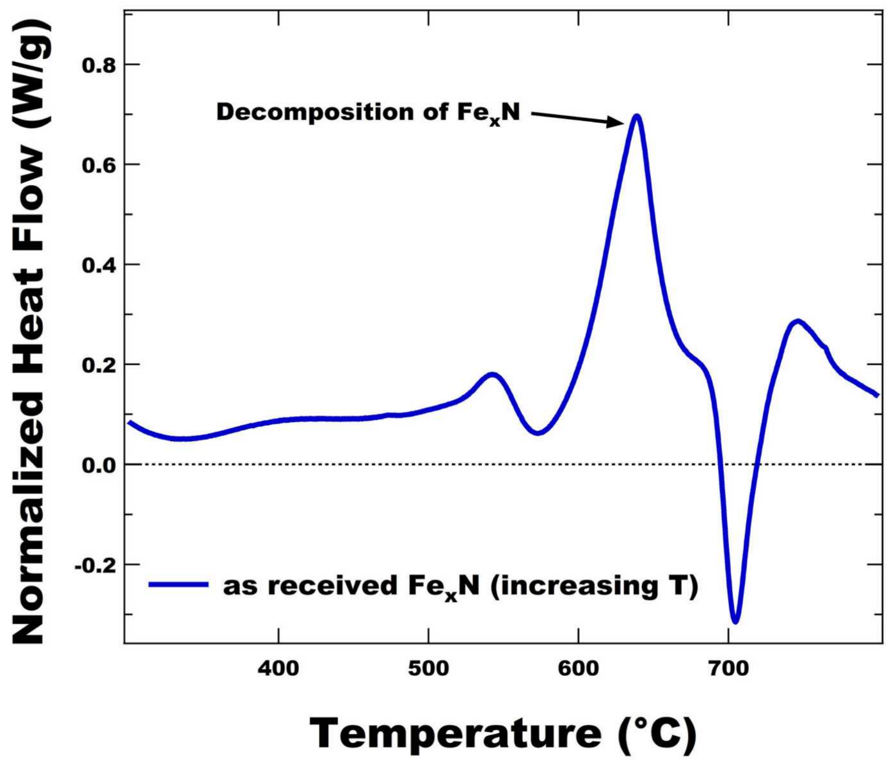




γ'-Fe_4N



fcc γFe structure stabilized by interstitial nitrogen in the body center

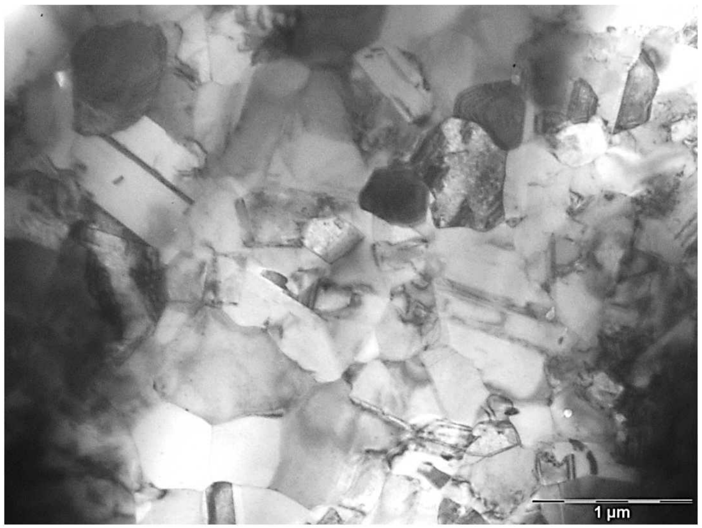


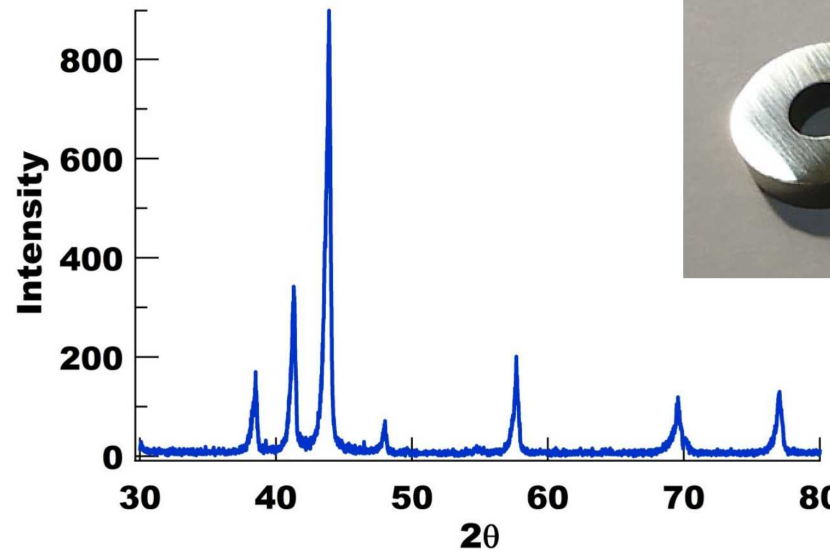
G. Scheunert, et al., A review of high magnetic moment thin films for microscale and nanotechnology
Applications, Appl. Phys. Rev., 3, 011301 (2016).
J.M.D. Coey, *Magnetism and Magnetic Materials* (Cambridge University Press, Cambridge, UK, 2012).

Relatively low thermal decomposition limits consolidation/fabrication methods

SPS consolidated Iron Nitride

First ever bulk γ'-Fe_4N! **U.S. Patent #9,963,344**



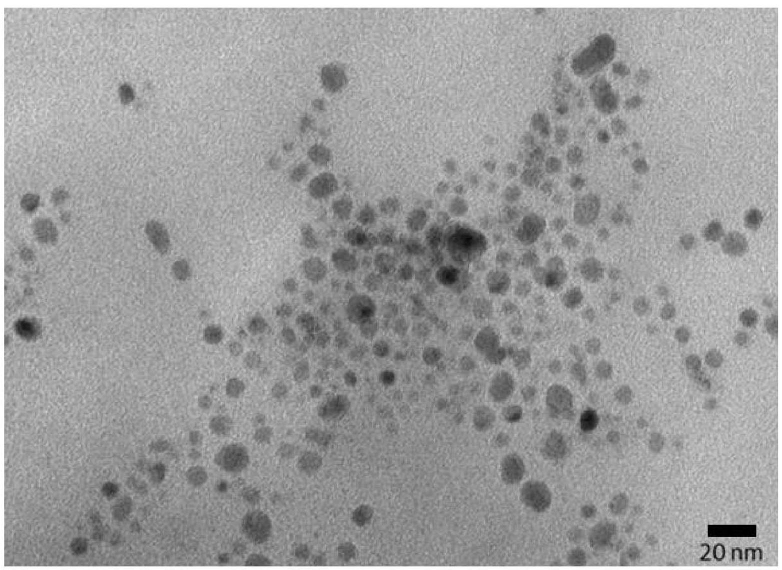


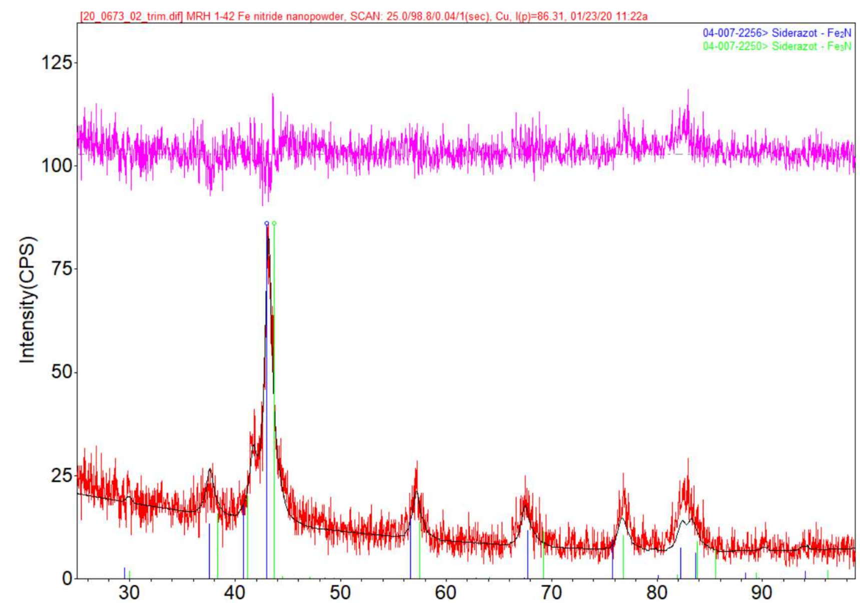
Net-shaped toroid (no machining required)

- **Fe nitride powders well consolidated with little porosity**
- **Grain sizes 200 nm – 1 µm → fine grain size = low H_c**
- **γ'-Fe_4N primary phase**
- **Fe_3N secondary phase from mixed phase starting material**

Routes to Nanoparticle Iron Nitride

$$FeCl_3 + Li_3N \xrightarrow[\text{oleylamine, -LiCl}]{350\ °C} Fe_2N + Fe_3N$$

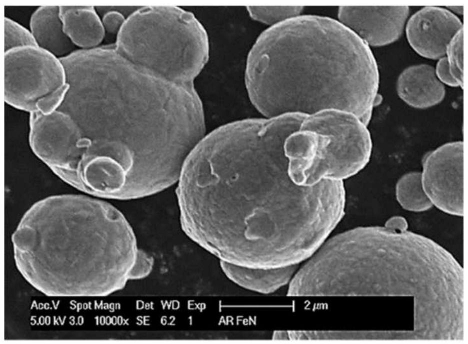


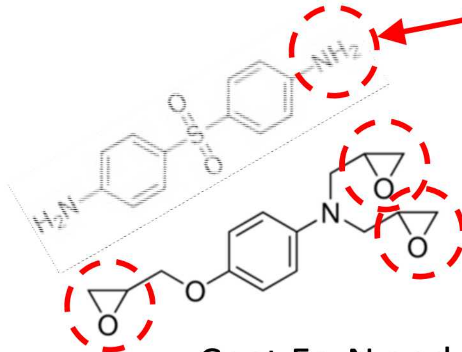


Magnetic Composites

Diamines will bond directly to Fe_4N surface and epoxy matrix for enhanced mechanical robustness and particle electrical isolation

Convert commercial Fe_xN powder to phase pure Fe_4N

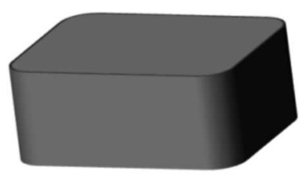


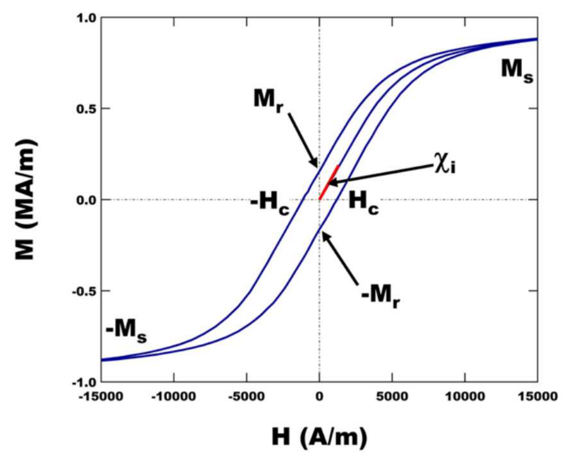


4-aminophenyl sulfone
&
N,N-diglycidyl-4-glycidyloxyaniline (NND)

Coat Fe_4N and mix with epoxy monomers

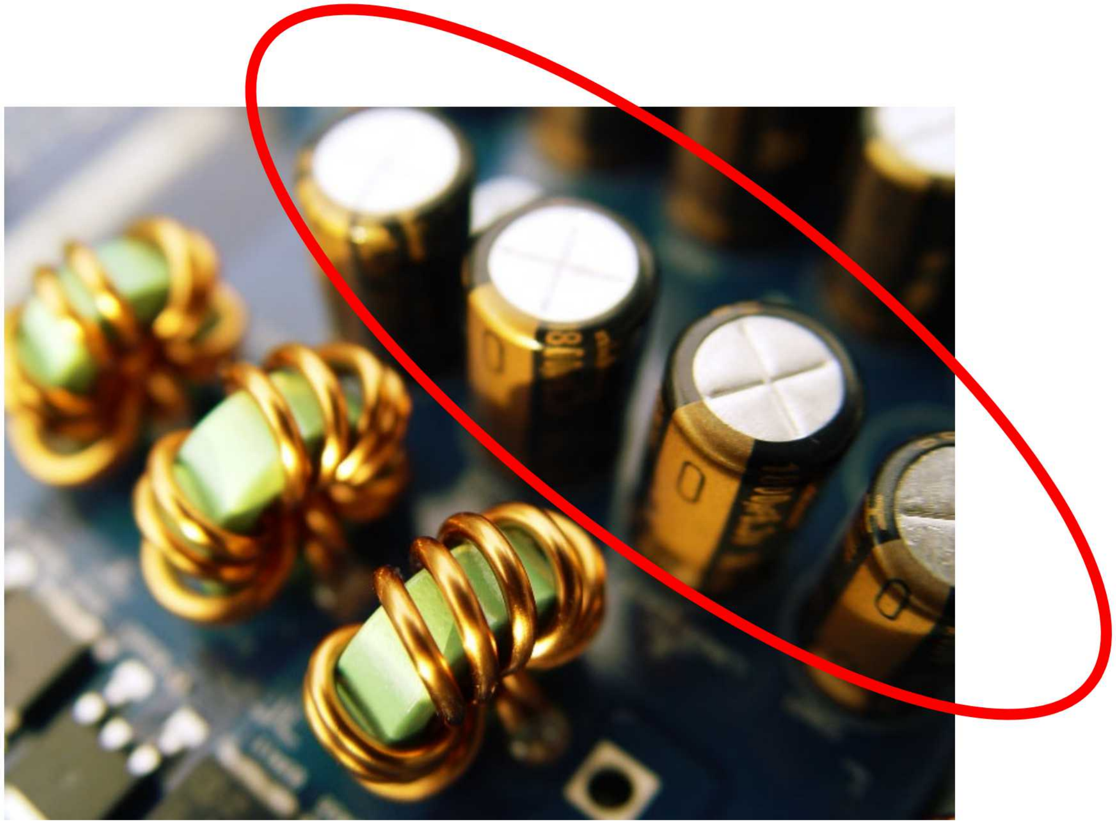
- Pour into 3D printed mold and cure into inductor/motor part
- Hot press to increase density and loading factor
- Results in a net-shaped part (no machining required)



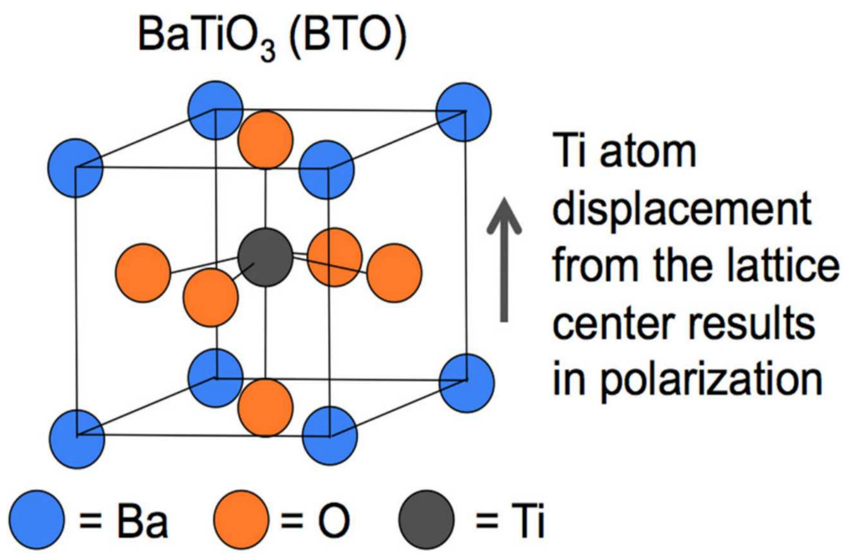


Evaluate and test

Ferroelectrics



Barium Titanate ($BaTiO_3$ or BTO)

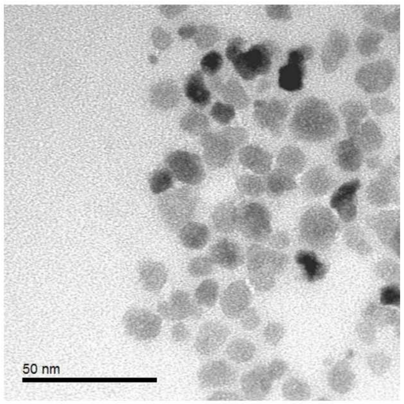


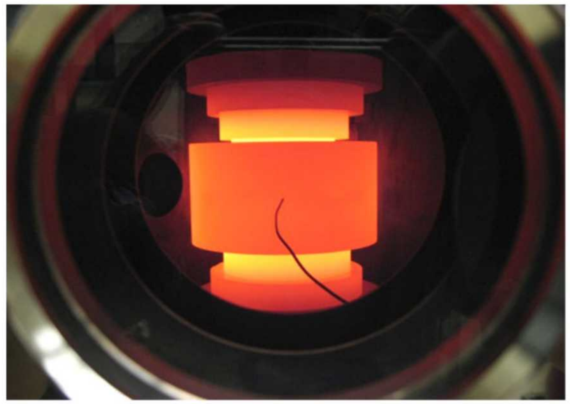
(Perovskite Structure)

- **Ferroelectric in bulk at room temperature**
- **Curie temp. (T_c) of 120 °C**
- **High dielectric constant**
- **Wide variation in reported nanoparticle dielectric constants**

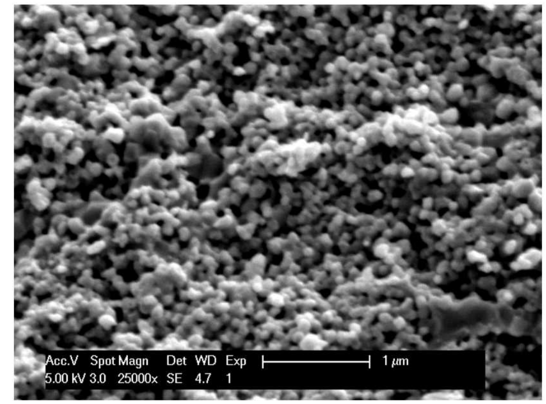
Device Fabrication

Sintering



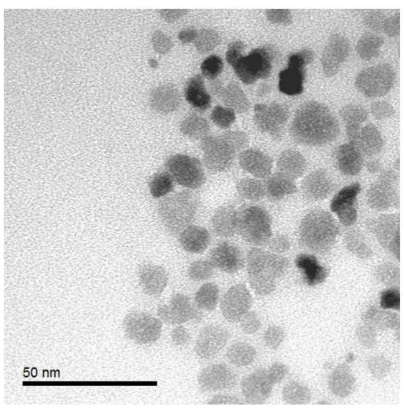


Spark Plasma Sintering die



SNL BTO, grain size 100 – 200 nm

Polymer composite



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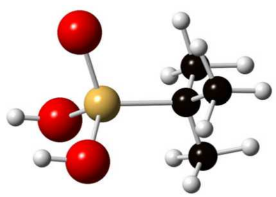
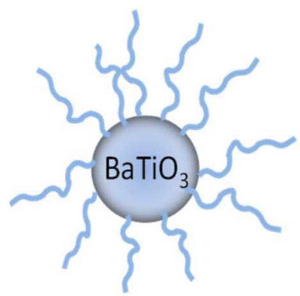
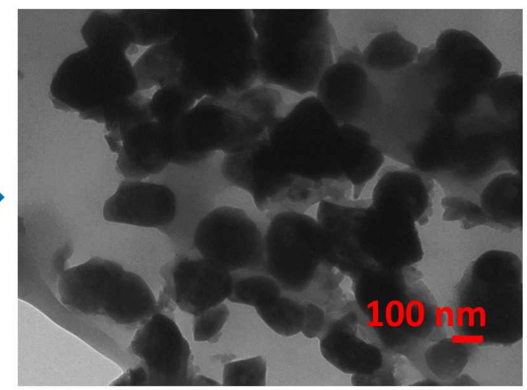
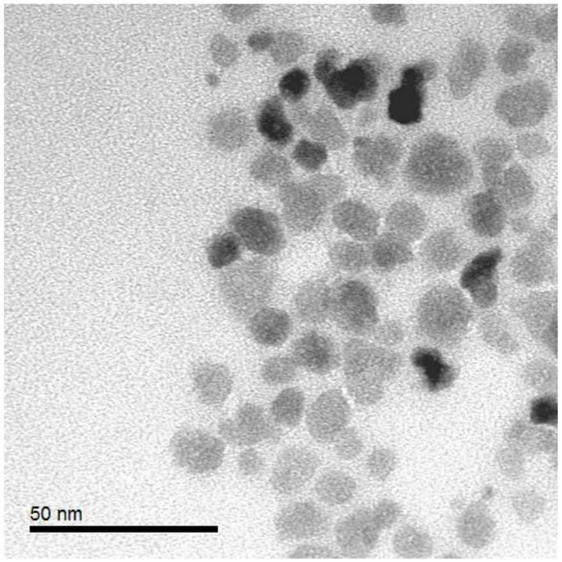


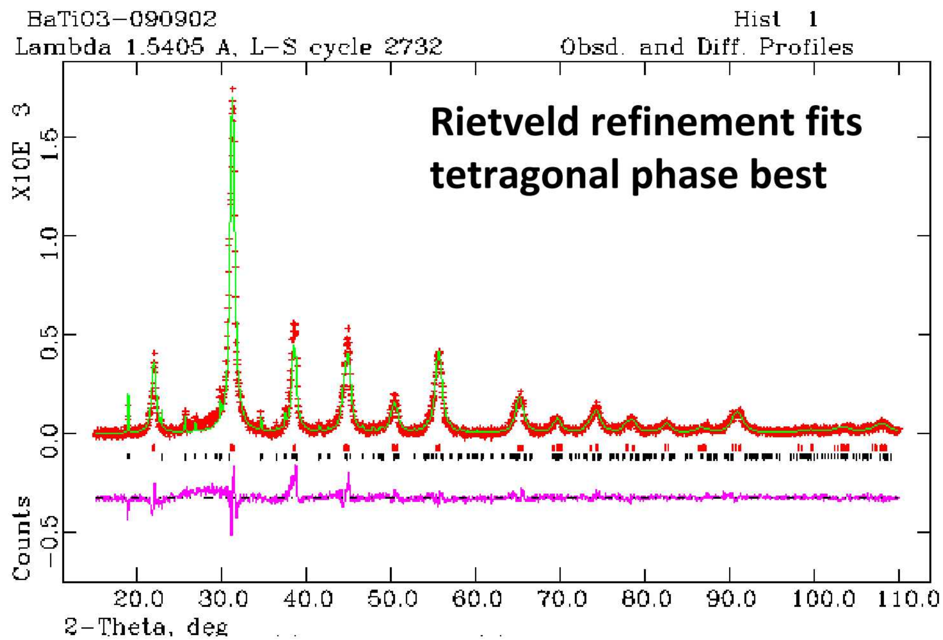




SNL BTO Nanoparticle Synthesis



~ 10 nm diameter

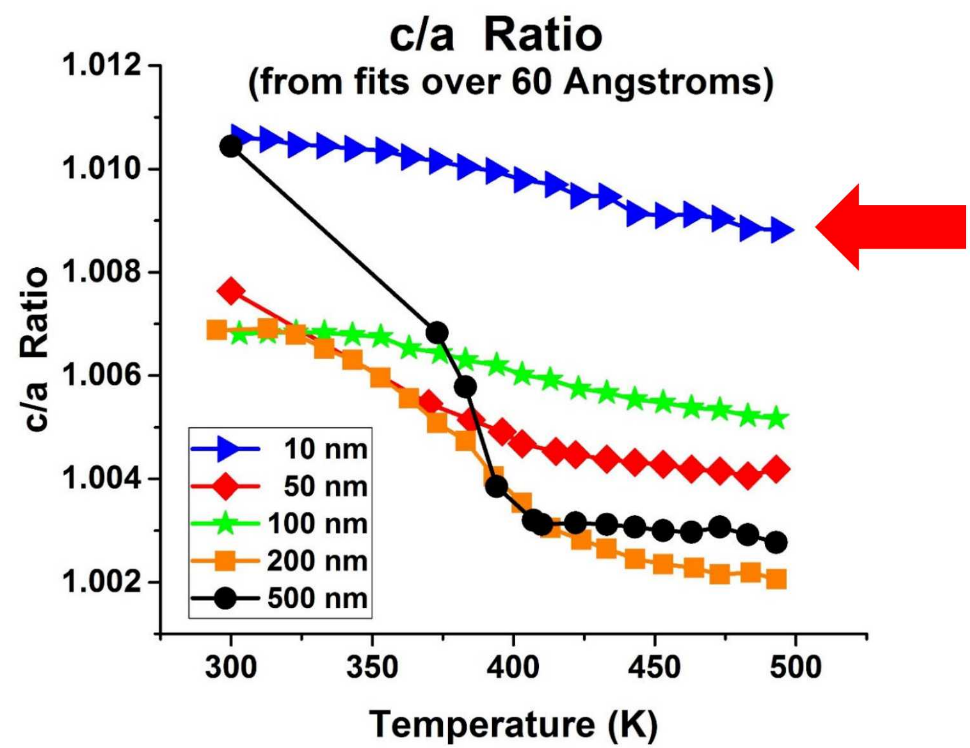


- $Ba(OH)_2 \cdot 8H_2O$ and $Ti(OPr)_4$ precursors at 80 ˚C
- Redesigned synthesis with air-free chemistry and improved control over water addition
- Rietveld analysis fits tetragonal phase when particles synthesized with 0.5 and 0.6 mol H_2O

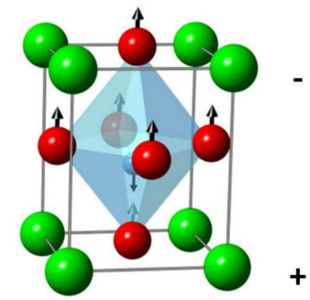
Yoon et. al., *J. Am. Ceram. Soc.* 90 311 (2007)

Particle Size Dependent Behavior

Synchrotron aPDF (atomic pair distribution function) Analysis

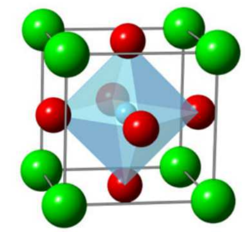


< 120 °C (393.15 K)



Tetragonal (Ferroelectric)

> 120 °C (393.15 K)



Cubic (Paraelectric)

Synchrotron X-ray data collected at NSLS at Brookhaven National Lab

Shi, et al., "Barium titanate nanoparticles: Short-range lattice distortions with long-range cubic order," Phys. Rev. B., vol. 00, pp. 005400, 2018.

Questions/Discussion