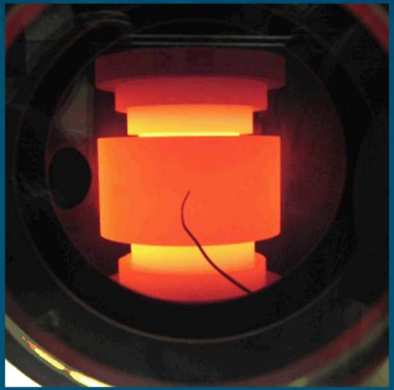
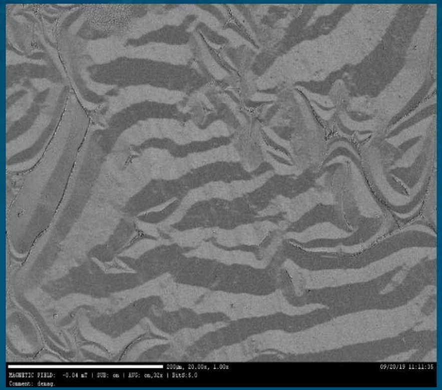
SAND2020-11225C

New pathways to iron nitride soft magnets





Todd C. Monson, Tyler E. Stevens, Charles J. Pearce, Melinda Hoyt, Erika Vreeland, Robert Delaney, Stan Atcitty

Sandia National Labs, tmonson@sandia.gov

Baolong Zheng, Calvin H. Belcher, Yizhang Zhou, Enrique J. Lavernia, Tim Rupert

University of California, Irvine

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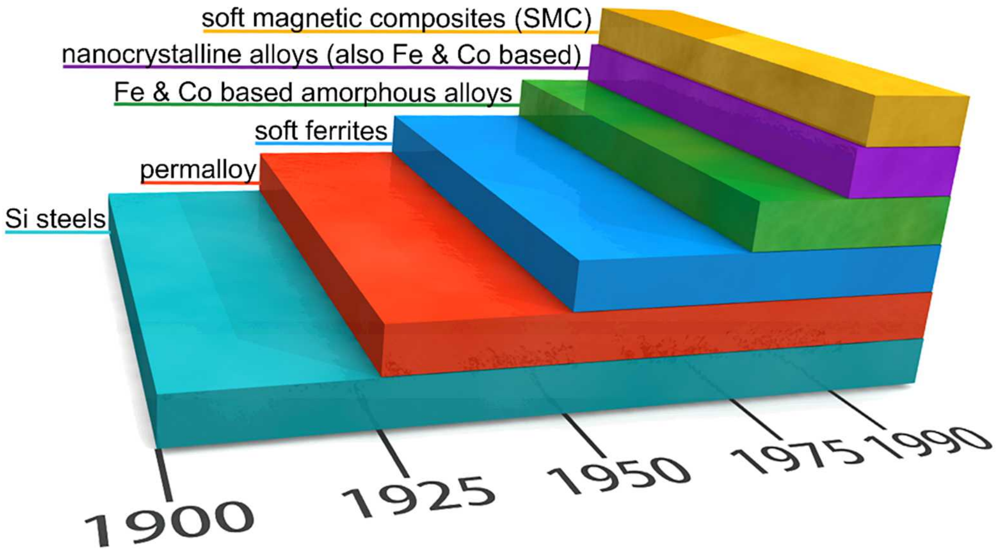
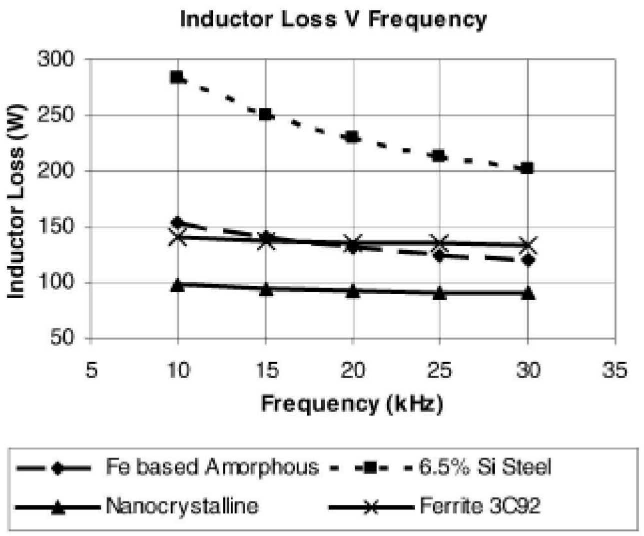
Soft Magnetic Material Development



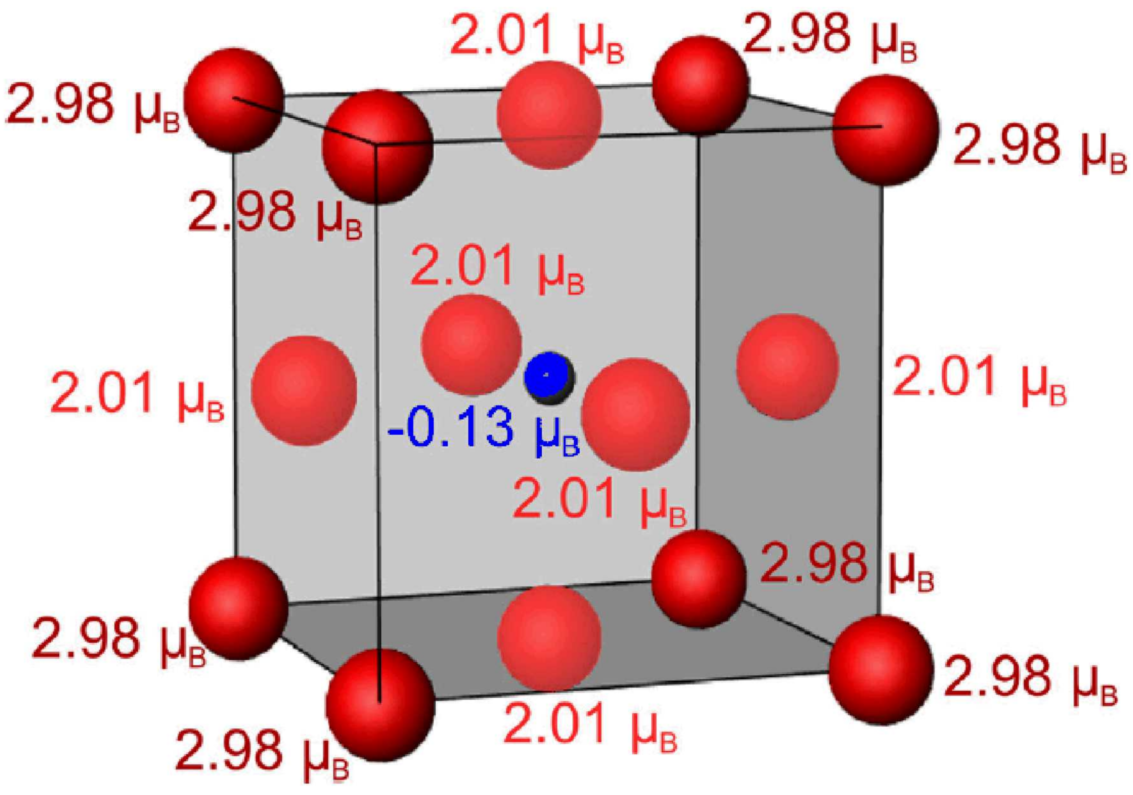
Adapted from: L.A. Dobrzański, M. Drak, B. Ziębowicz, Materials with specific magnetic properties, Journal of Achievements in Materials and Manufacturing Eng., 17, 37 (2006).



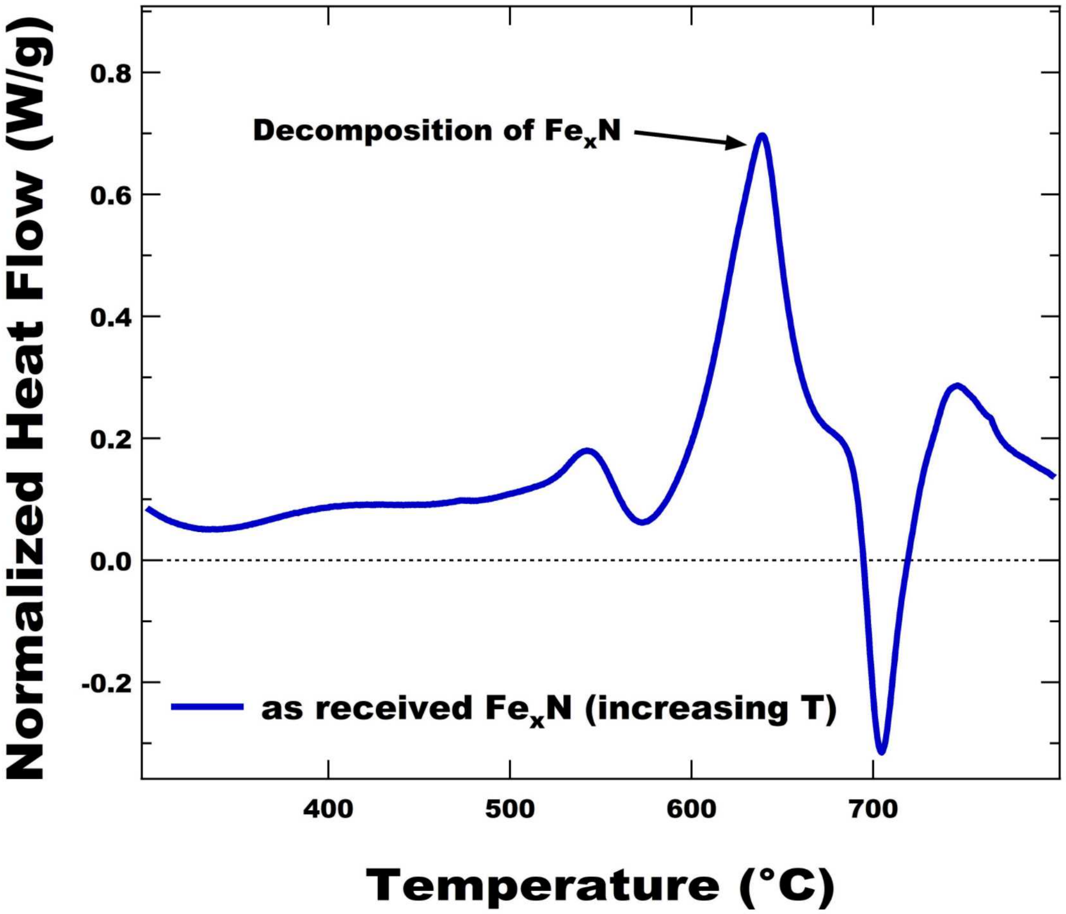
B.J. Lyons, J.G. Hayes, M.G. Egan, Magnetic Material Comparisons for High-Current Inductors in Low-Medium Frequency DC-DC Converters, IEEE, 71 (2007).

Magnetic Material	J_s (T)	$\rho(\mu\Omega\cdot m)$	Cost
VITROPERM (Vacuumschmelze)	1.20	1.15	High
Metglas 2605SC	1.60	1.37	High
Ferrite (Ferroxcube)	0.52	$5x10^6$	Low
Si steel	1.87	0.05	Low
γ'-Fe_4N	1.89	> 200	Low

γ'-Fe_4N



fcc γFe structure stabilized by interstitial nitrogen in the body center



Relatively low thermal decomposition limits consolidation/fabrication methods

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).

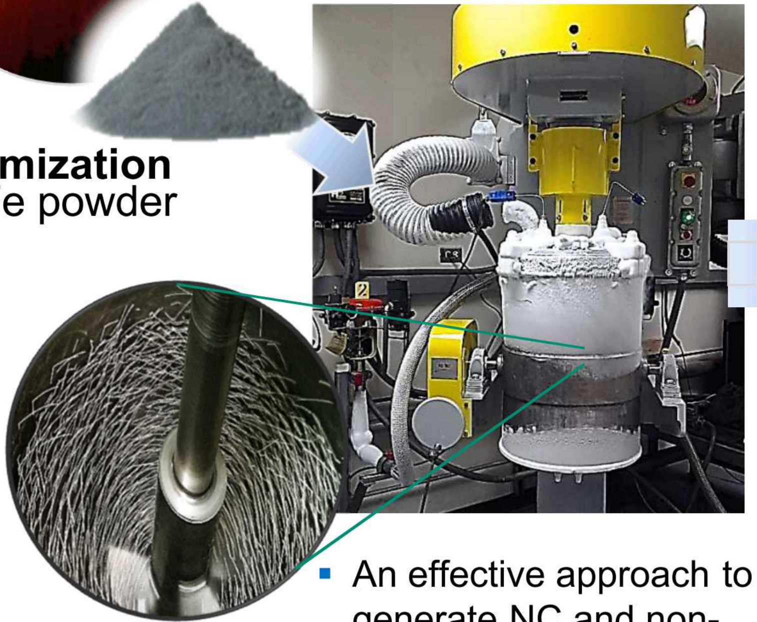
Mechanochemical Synthesis of γ'-Fe_4N

U.S. Patent Issued (#9,963,344)

Atomization of Fe powder

Cryomilling

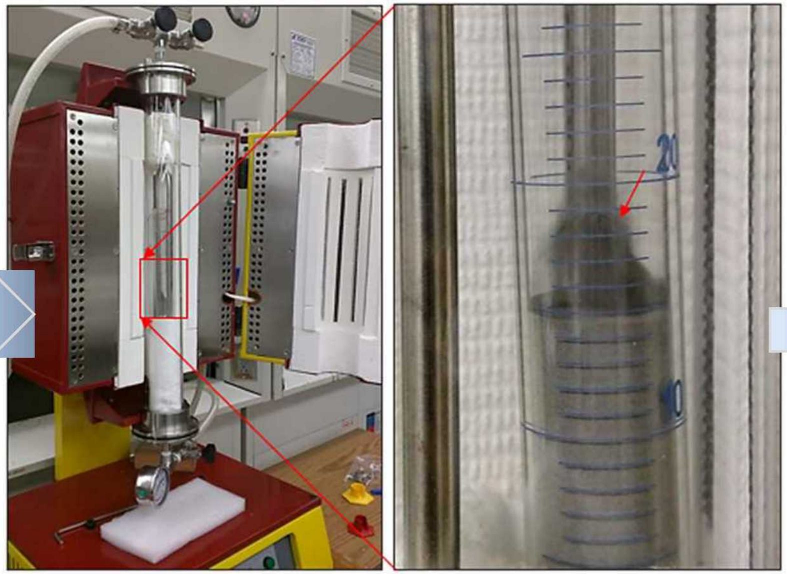
Severe Plastic deformation in cryogenic temperature



- An effective approach to generate NC and non-equilibrium structures in large quantities;

Fluidized Bed Reactor (FBR)

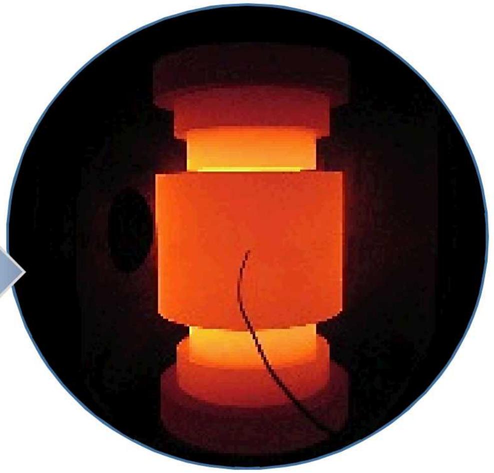
Reactor for multiphase chemical reactions



- Synthesizing FeN powder with NH_3+H_2 forming gas and vertical fluidized-bed furnace;
- Provides uniform distribution of the gas blast over the tube chamber;
- Particles are suspended by an upward gas flow.

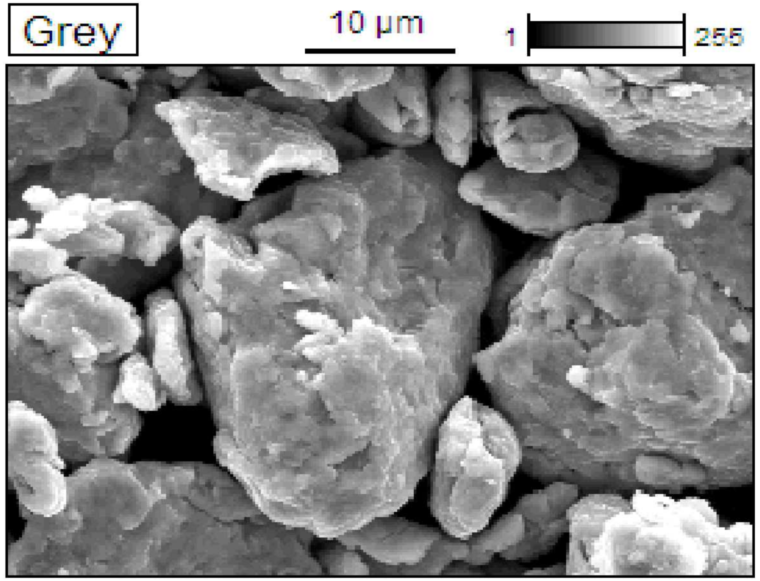
Spark Plasma Sintering (SPS)

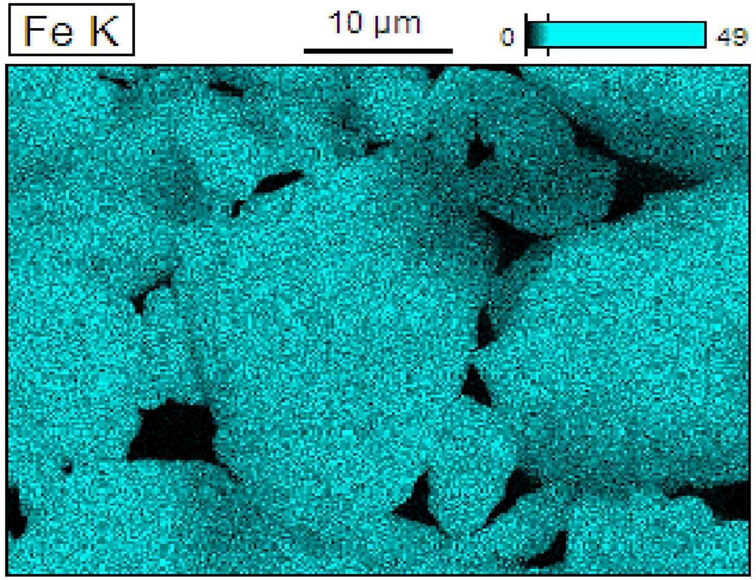
Field Assisted Sintering Technology (FAST)

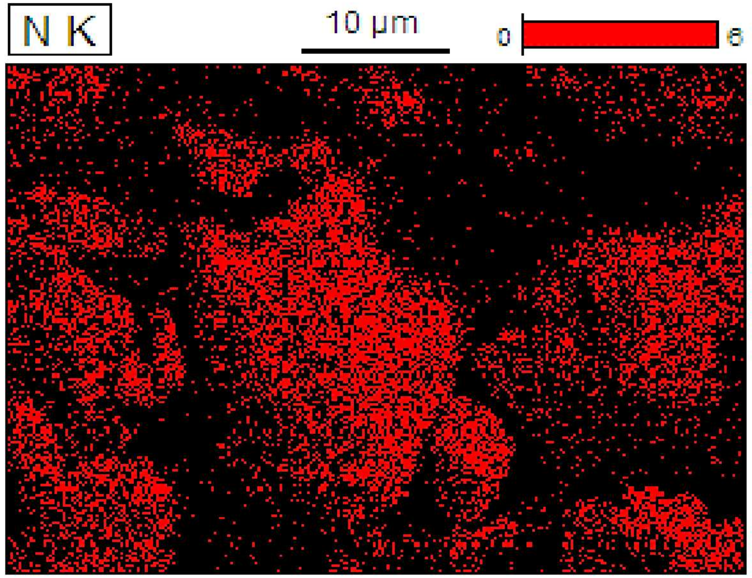


- Pressure and pulsed current assisted sintering process;
- Restrict grain growth;
- Precision control heat and pressure;
- Full density materials.

EDS Mapping of Synthesized FeN Powder Surface



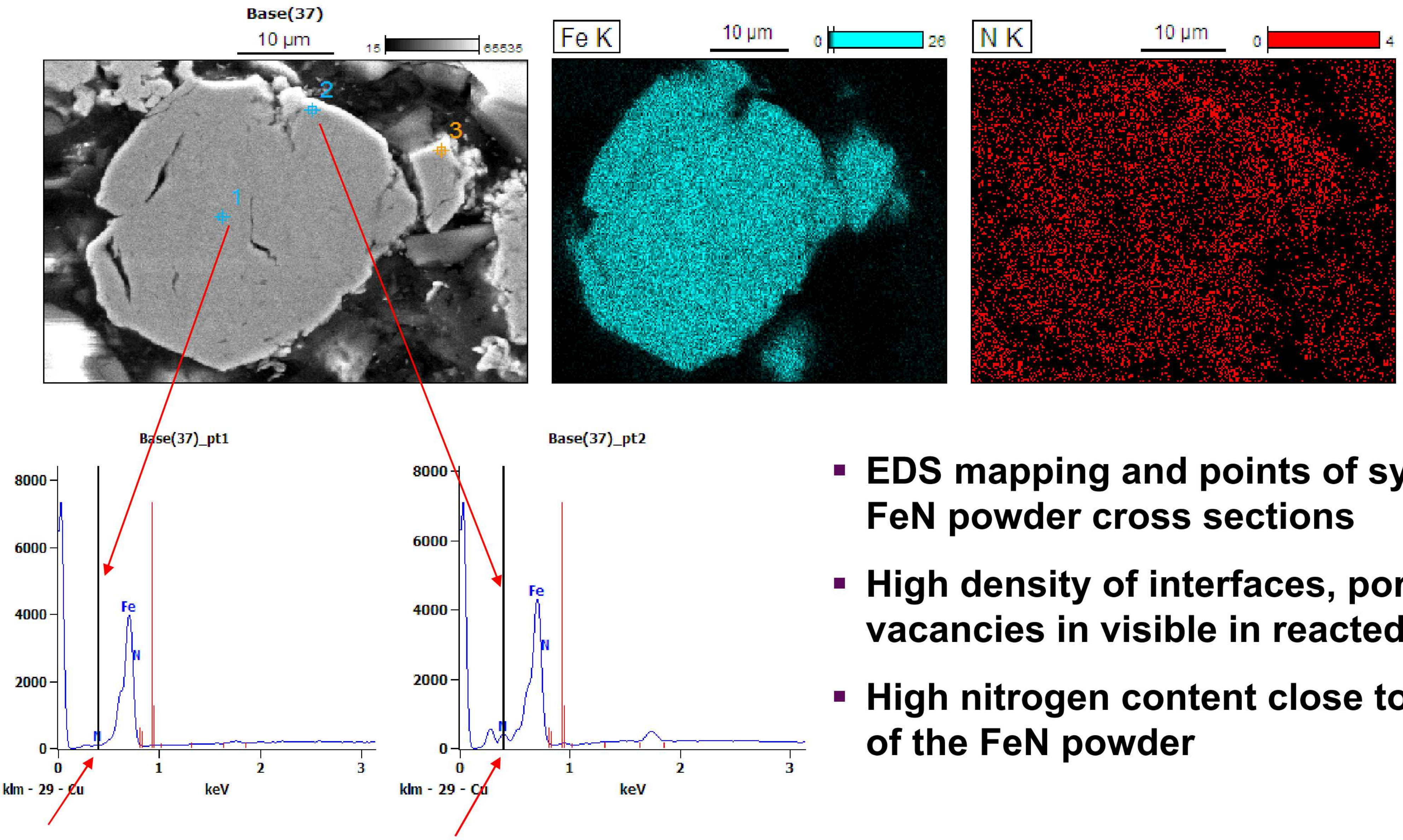




- **EDS mapping of synthesized FeN powder**
- **High nitrogen content uniformly distributed on the particle surface in the powder**
- **The measured element fraction indicated Fe_4N was mostly formed during the reaction**

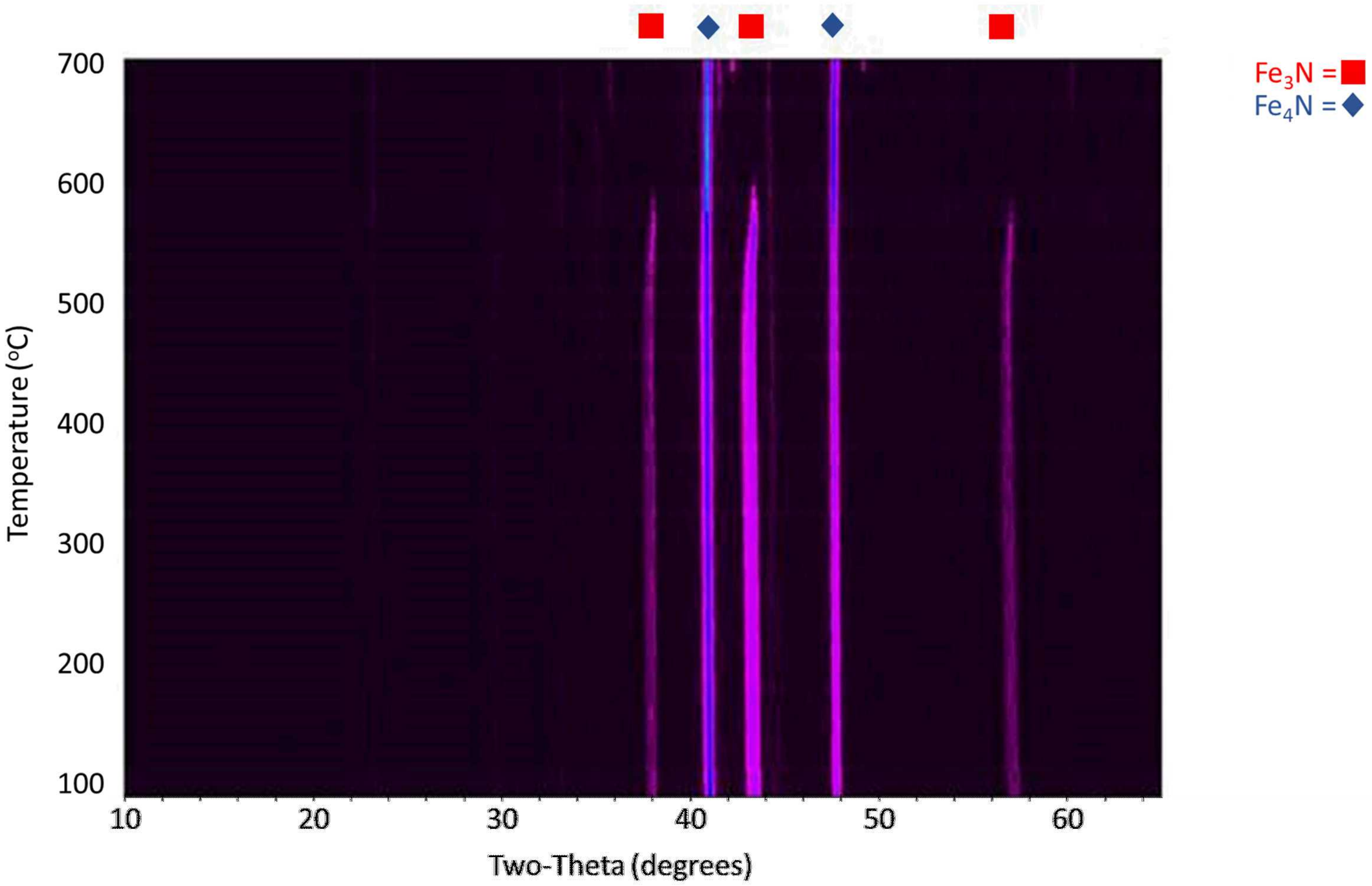
Element Line	Net Counts	Weight %	Weight % Error	Atom %	Formula	Compnd %
N K	8937	21.2	± 0.4	51.8	N	21.2
Fe K	269857	78.8	± 0.3	48.2	Fe	78.8
Total		100.0		100.0		100.0

EDS Mapping of Cross Sectioned FeN Powder



- **EDS mapping and points of synthesized FeN powder cross sections**
- **High density of interfaces, porosity, and vacancies in visible in reacted Fe powder**
- **High nitrogen content close to the surface of the FeN powder**

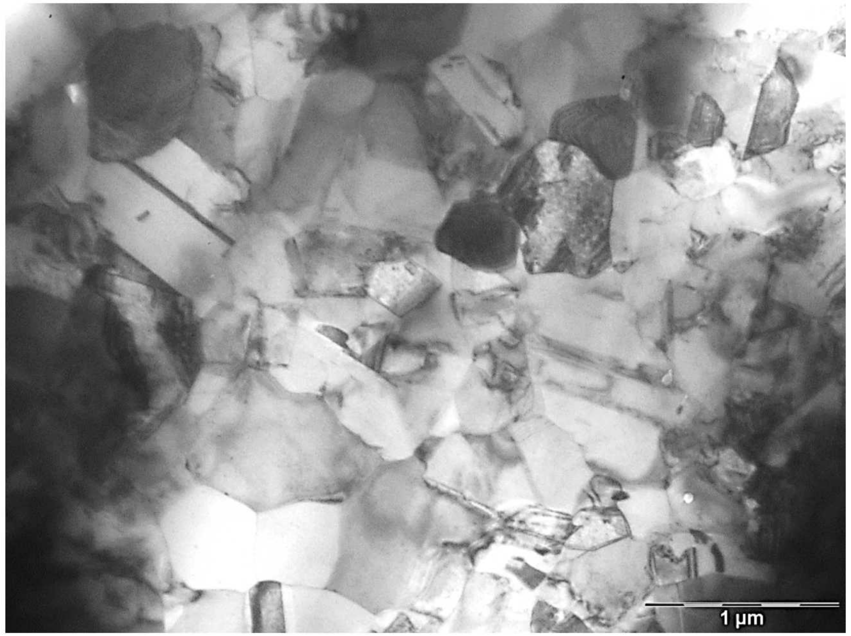
Production of phase pure γ’-Fe4N powder

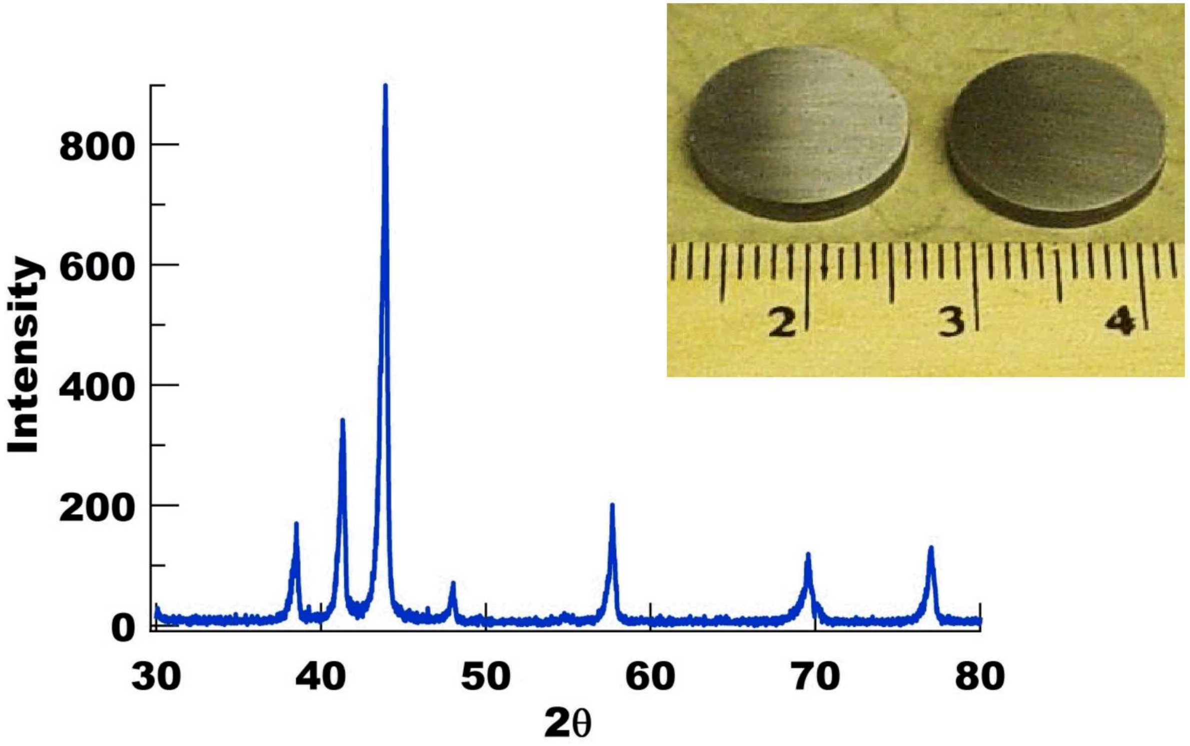


- **Simple heat treatment converts mixed phase iron nitride powders**
- **Only phase pure γ’-Fe_4N remains**

SPS consolidated Iron Nitride

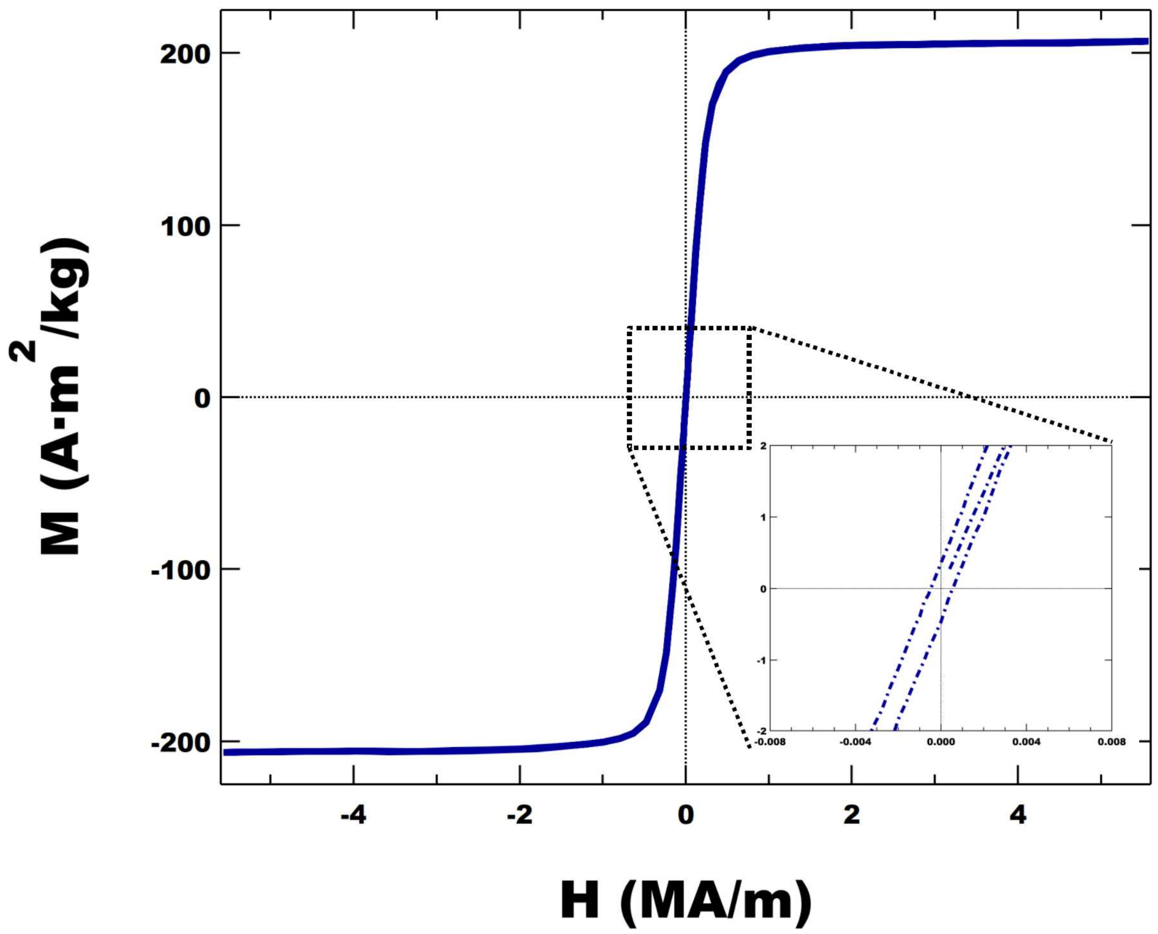
First ever bulk γ’-Fe_4N! **U.S. Patent #9,963,344**





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

SPSed iron nitride (γ'-Fe_4N) Magnetic Properties



- SPSed at 530°C and 200 MPa
- M_s = 207 Am^2/kg
- Theoretical M_s = 209 Am^2/kg
  - α-Fe is 217 Am^2/kg @ room temp.
- H_c < 500 A/m

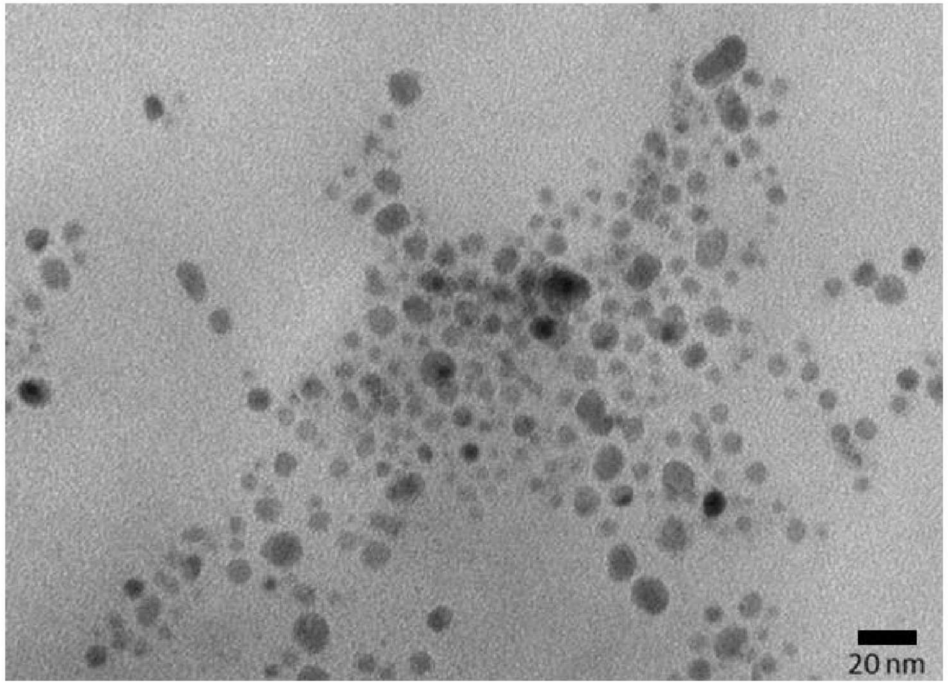
Net-shaped toroid
(no machining required)

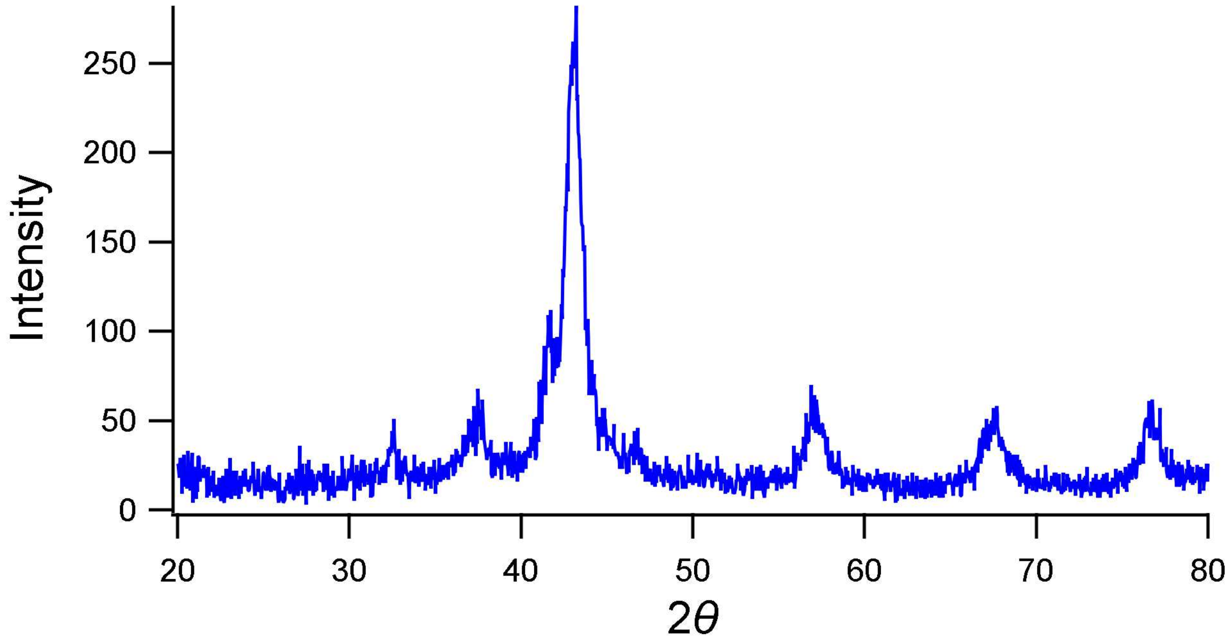
T. C. Monson, B. Zheng, R. E. Delany, C. J. Pearce, E. D. Langlois, S. M. Lepkowski, T. E. Stevens, Y. Zhou, S. Atcitty, E. J. Lavernia, "Soft magnetic multi-layered FeSiCrB-Fe_xN metallic glass composites fabricated via spark plasma sintering," IEEE Magnetics Letters **10**, 1-5 (2019).

Metathesis Route to Nanoparticle Iron Nitride

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

-LiCl





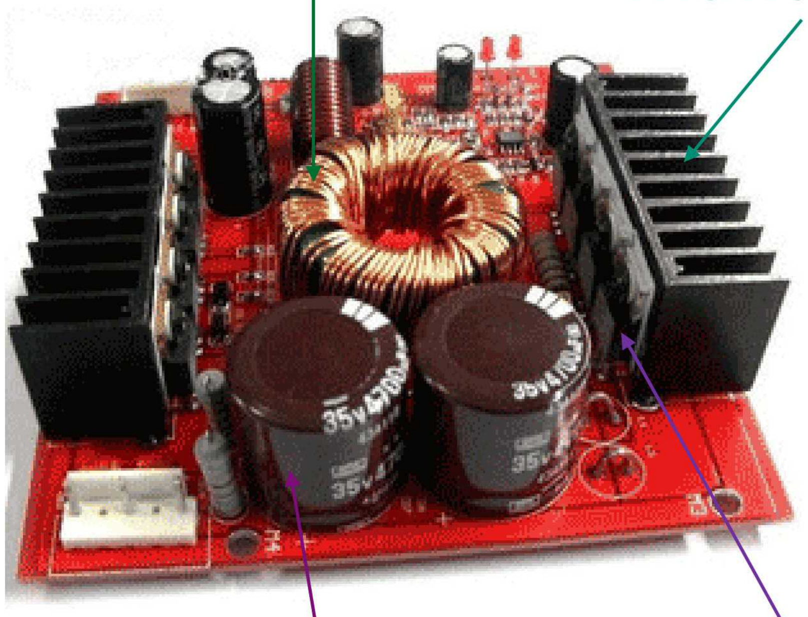
Questions/Discussion

Backup Slides

Magnetics Impact on Power Electronics Systems

Passive elements and thermal management comprise the bulk of the volume and mass of a power converter

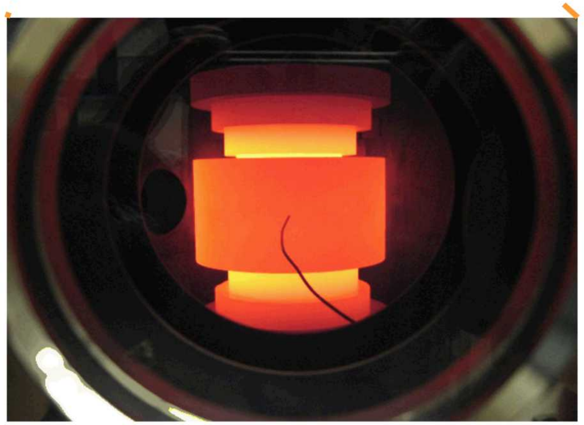
WBG/UWBG materials enable higher switching frequency and better thermal management



J. Neely, J. Flicker, B. Kaplar (SNL)

Spark Plasma Sintering (SPS)

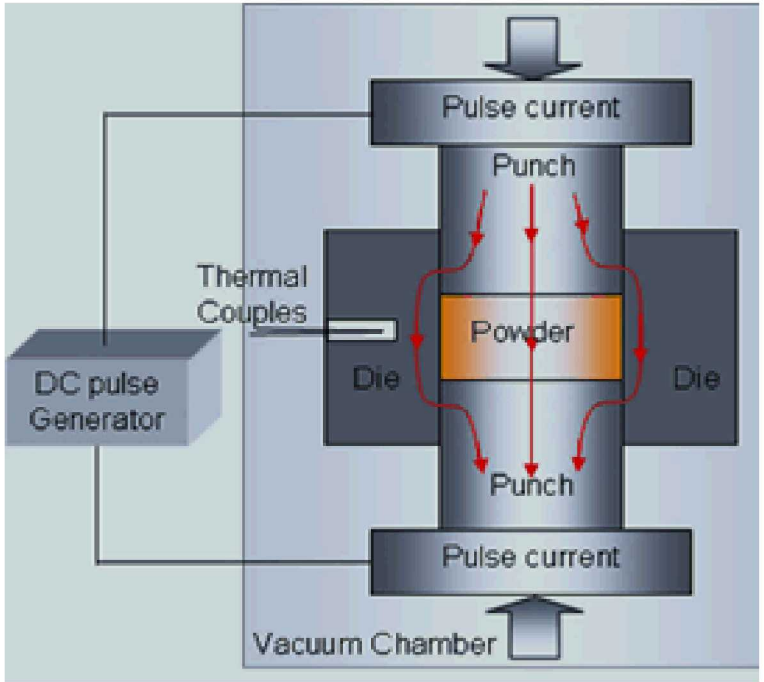
SPS Model: SPS-825S Dr. Sinter® at UC Irvine



SPS Chamber

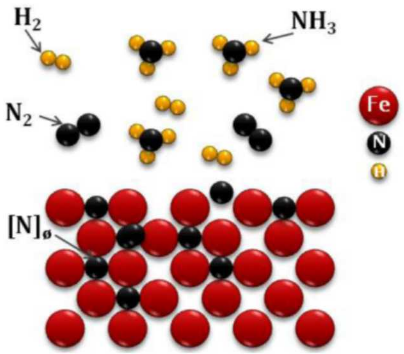
Starting Powder in Die

End Product

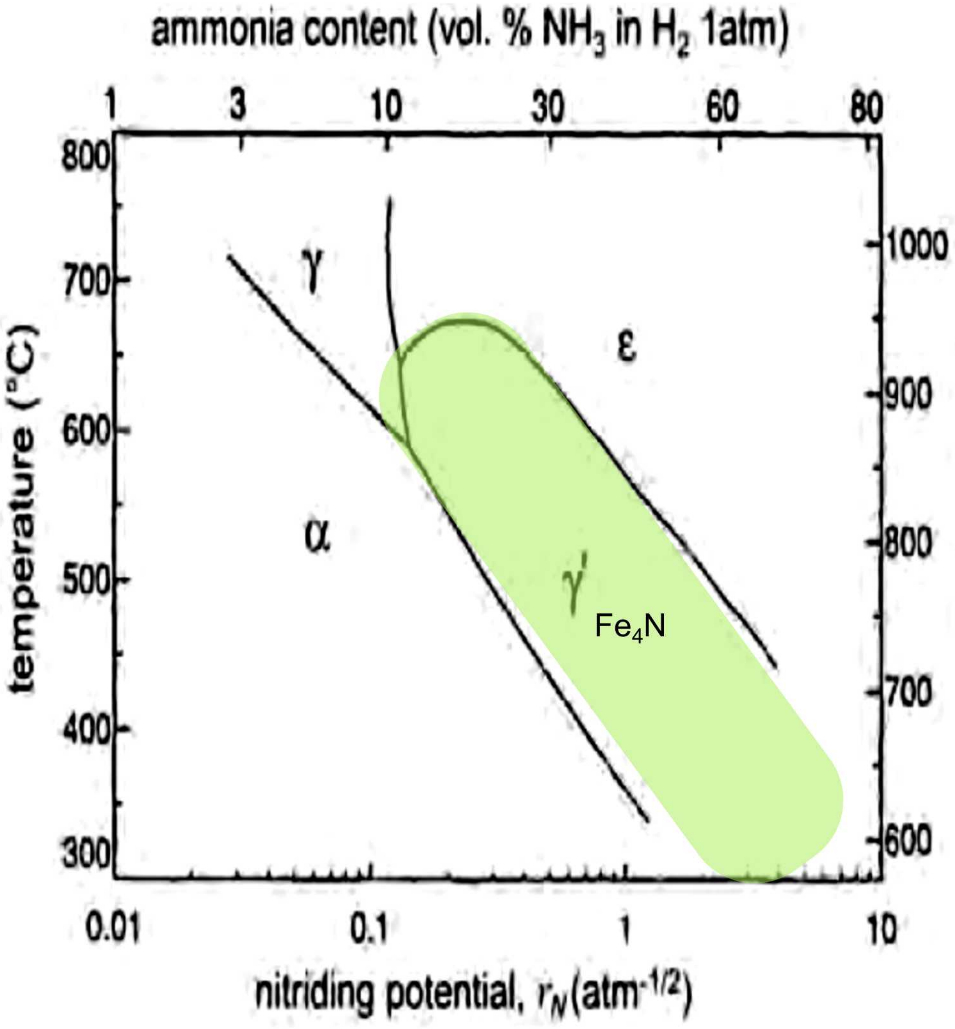


Thermodynamics of Nitriding

Schematic of nitriding



Dissolution of nitrogen in Fe occurs via the dissociation of ammonia at the surface



[ASM Handbook, Volume 4A,2013]

$$4Fe + NH_3 \rightarrow Fe_4N + (3/2)H_2$$

Relatively higher chemical potential of nitrogen in NH_3 compared to nitrogen gas, N_2

$$NH_3 = [N] + \tfrac{3}{2} H_2$$

[N] represents nitrogen which is dissolved on the steel surface

N in the Fe surface, the activity of nitrogen, a_N:

$$a_N = K \frac{p_{NH_3}}{p_{H_2}^{3/2}} p_0^{1/2}$$

where K is the equilibrium constant of reaction, P_0 is the total pressure, and P_{NH3} and P_{H2} are the partial pressures of the ammonia and hydrogen gases respectively. $p_{N2}{}^o$ is the partial pressure of nitrogen at the standard state.

Magnetic Properties of some Iron Nitrides

Material	M_s (Am^2/kg)
α-Fe	218
Magnetite	80 - 103
FeN	209
Fe_2N	~70
Fe_3N	144
Fe_4N	209