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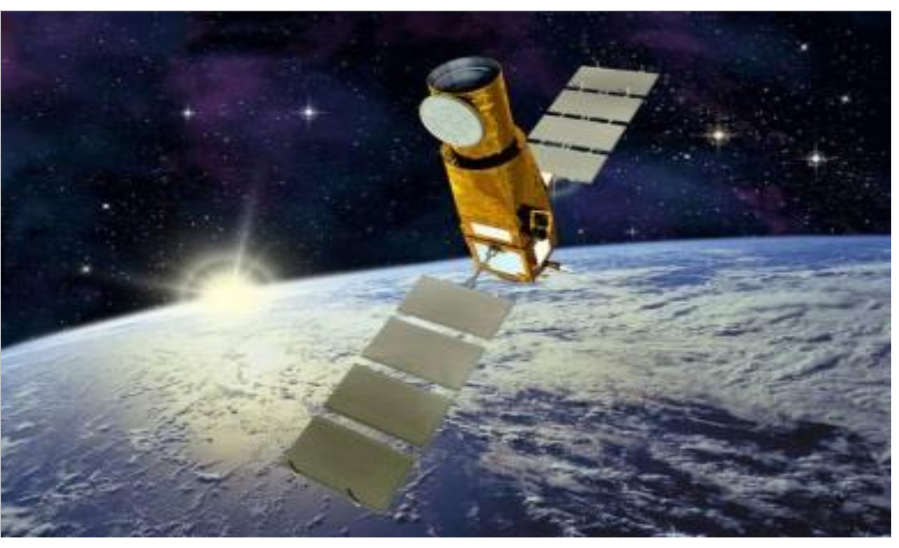

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Synthesis of γ'-Fe_4N, a new soft magnetic material for inductors and transformers

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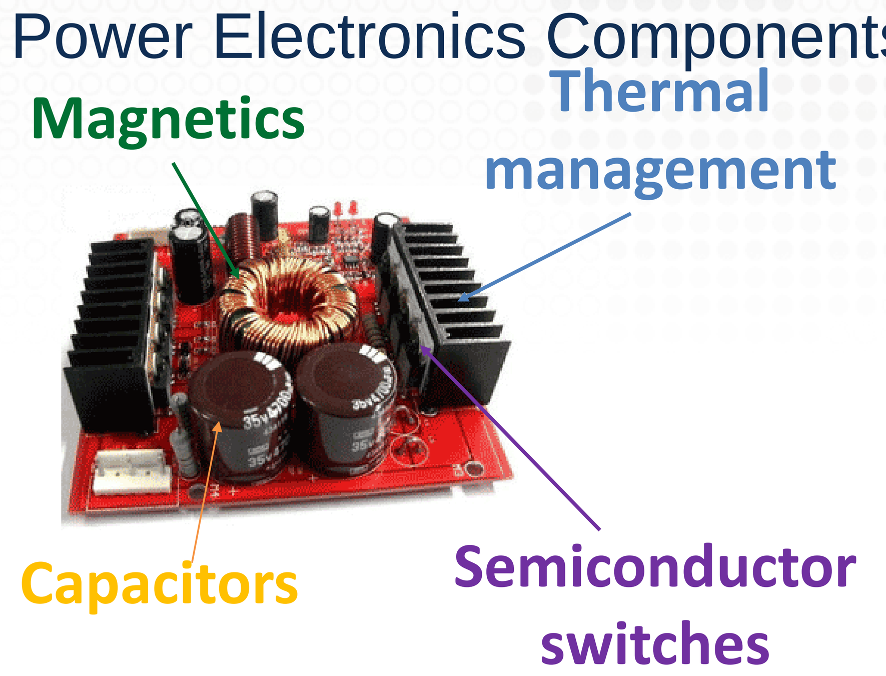
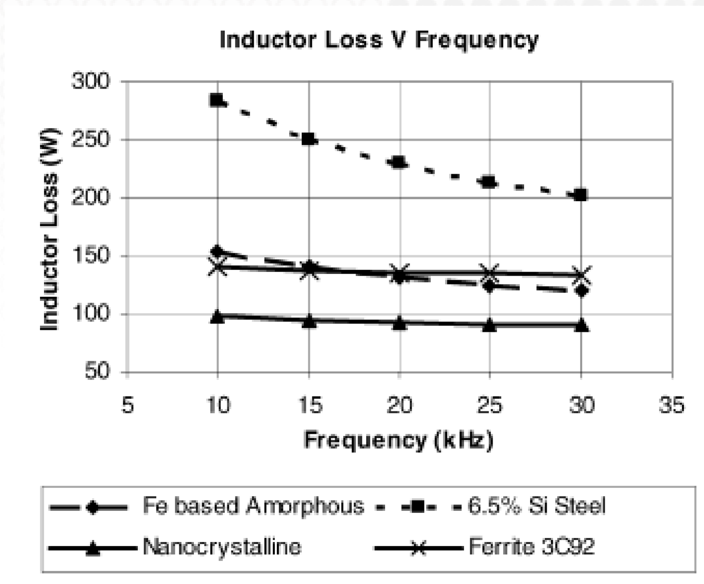
Motivation

Magnetic materials comprise a significant portion of power electronics. To decrease the size requirements, new materials are required to work in conjunction with improved semiconductor switches. Specifically, magnetic materials capable of operating at higher switching frequency are needed.

Power Electronics Components

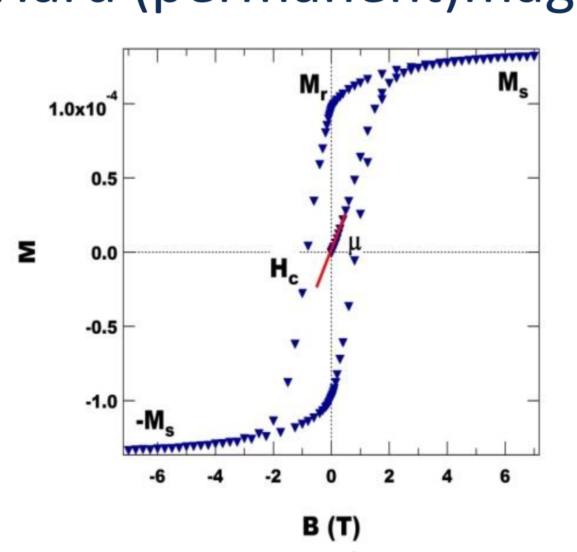




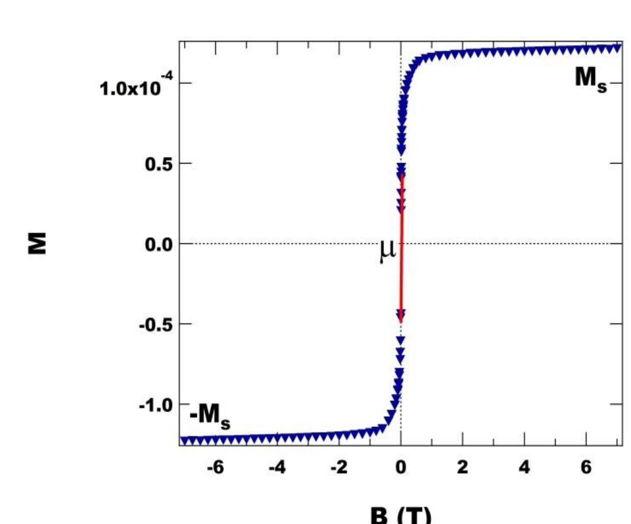
B.J. Lyons, J.G. Hayes, M.G. Egan, Magnetic Material Comparisons for High-Current Inductors in Low-Medium Frequency DC-DC Converters, IEEE, 71 (2007).

Requirements for a new magnetic material: Low loss in 10-200 kHz frequency range, high permeability (low coercivity), high saturation magnetizations, low magnetostriction, high temperature performance, and scalable & affordable.

Hard (permanent)magnet

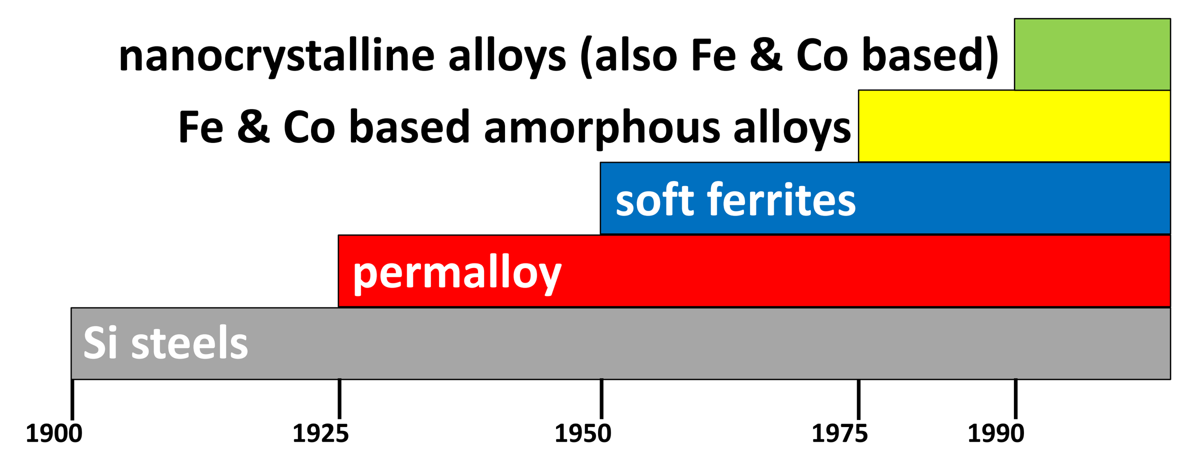


Soft magnet



M_s = saturation magnetization, M_r = magnetic remnance
H_c = coercivity, μ = permeability

Development of soft magnetic materials

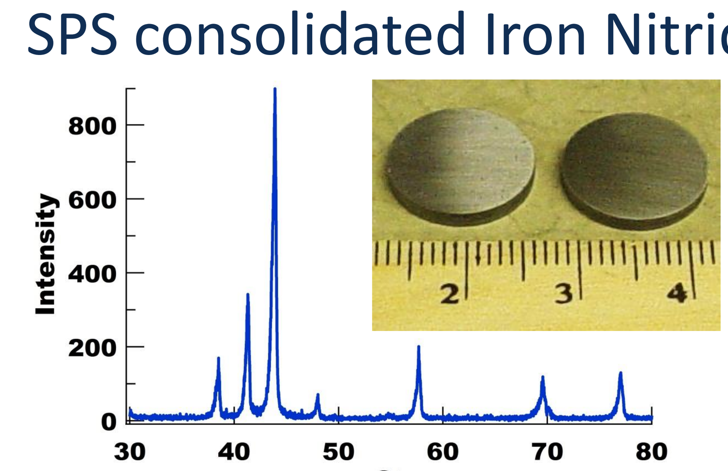


Adapted from: L.A. Dobrzański, M. Drak, B. Ziębowicz, Materials with specific magnetic properties, Journal of Achievements in Materials and Manufacturing Eng., 17, 37 (2006).

γ'-Fe_4N meets all of the requirements

Magnetic Material	J_s (T)	ρ(μΩ·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

SPS consolidated Iron Nitride



Grain sizes 200 nm – 1 μm → fine grain size = low loss, γ'-Fe_4N primary phase, Fe_3N secondary phase from mixed phase starting material. Monson, U.S. Patent Application # 15/002,220

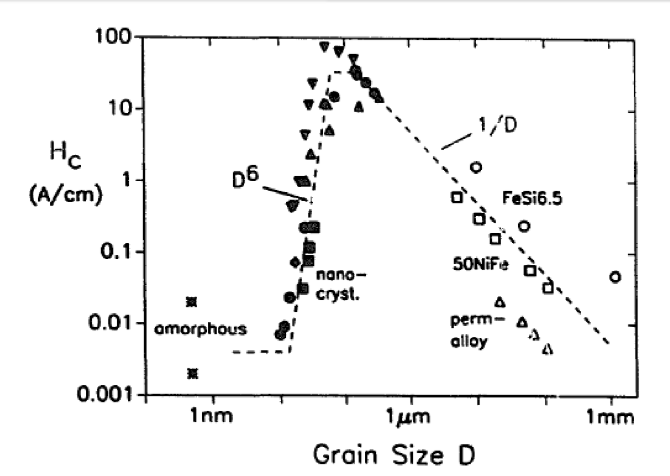


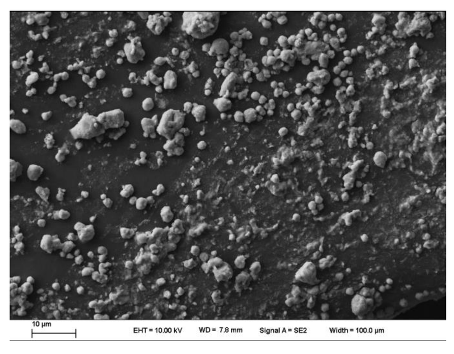
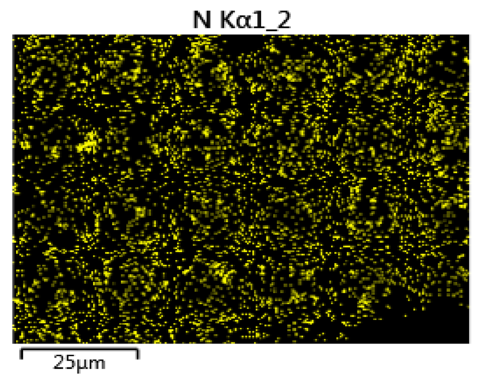
Fig. 2. Coercivity H_c vs. grain size for various soft magnetic metallic alloys. The data of the nanocrystalline material refer to (▲) FeNbSiB [14], (●) FeCuNbSiB [14], (◆) FeCuVSiB [15], (■) FeZrB [4] and (▼) FeCoZr [16].

G. Herzer, Nanocrystalline Soft Magnetic Materials, J. Magn. Mag. Mat., 112, 258 (1992).

Synthesis from Fe(0)

- Current methods enable large-scale iron nanoparticle synthesis (Huber, 2011 *US Patent* #7,972,410)
  - Size control by reversible magnetic agglomeration

Fe nanoparticles —NH_3 (*l*)→ Fe_xN

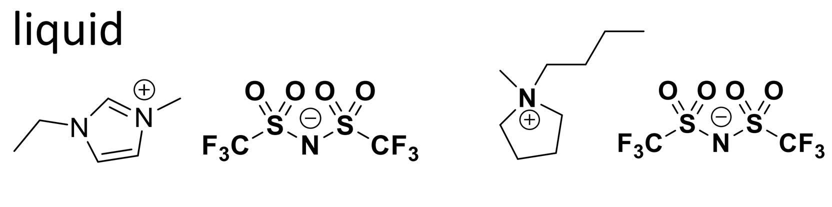



- SEM-EDS confirms nitrogen content
- Powder XRD- mixed phase + iron oxides
- Currently working to optimize conditions to give phase pure material

Electrochemical nitridation of iron wire

- Growth of γ'-Fe_4N demonstrated in LiCl-KCl eutectic melt, γ'-Fe_4N Formed at surface of Fe(0) electrode, Li_3N nitride source (Ito *Electrochimica Acta* **2000**, *45*, 3367
- *Requires > 700 K, Multiple phases present (XRD)*

Goal: Development of a low temperature approach using a room temperature ionic liquid



- Even with the use of crown ethers, we have yet to observe solubility of Li_3N in the RTILs
- Currently exploring other avenues

Salt Metathesis with Li_3N

- Effective for early transition metals

Group	MX_n	Product
4	$TiCl_4$	TiN
	$ZrCl_4$	ZrN
	$HfCl_4$	HfN
5	VCl_3	VN, V_2N
6	$CrCl_3$	Cr, Cr_2N
7	MnI_2	Mn_4N, Mn
8	$FeCl_3$	Fe
10	$NiCl_2$	Ni

Adapted from Parkin *J. Chem. Soc., Dalton Trans.* **1993**, 2435.

- Reactions with late metals generate too much heat
  - Decomposition to elemental metal

$FeCl_3$ + Li_3N → FeN + 3 LiCl

- When reaction is performed in dilute solution at high temp, iron nitride is formed
- Confirmed by powder XRD
- Optimization to form pure phase iron nitride is underway

Acknowledgements

- γ'-Fe_4N R&D supported by Dr. Imre Gyuk and the Energy Storage Program in the Office of Electricity Delivery and Energy Reliability at the US Department of Energy
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