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An Overview of Magnetic Materials R&D at Sandia National Labs

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Sandia National Laboratories develops advanced technology needed to ensure global peace. Focused R&D is a requirement.

SNL is an FFRDC: Federally funded Research and Development Center

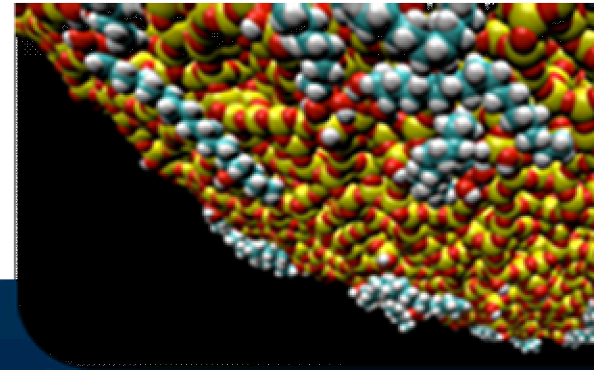
SNL is GOCO: Government owned, contractor operated

SNL GOCO Management:
- AT&T: 1949–1993
- Martin Marietta: 1993–1995
- Lockheed Martin: 1995–2017
- Honeywell and Northrop Grumman (NTESS): 2017 - present

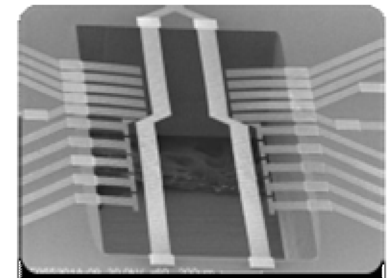
SNL's Science and Engineering Foundations

Computing and information science

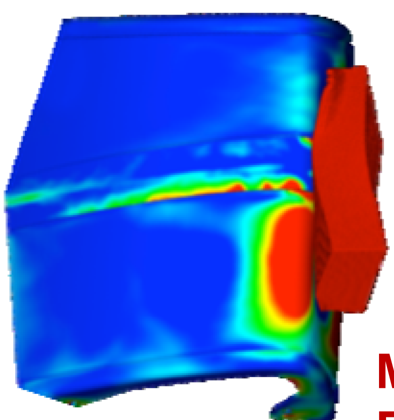
Materials science



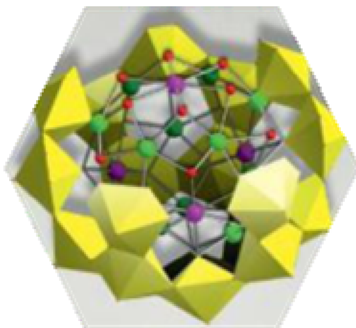
Nanodevices and microsystems



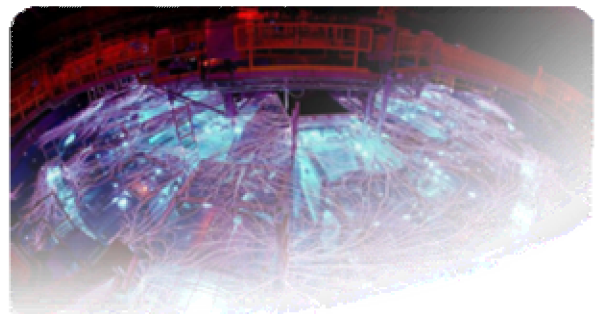
Engineering sciences



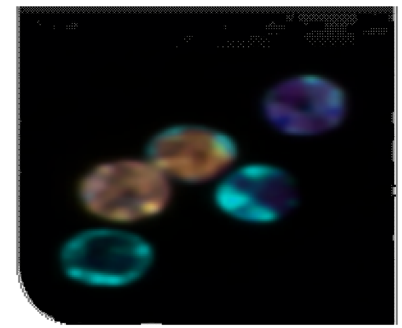
Geoscience



Radiation effects and high-energy density science



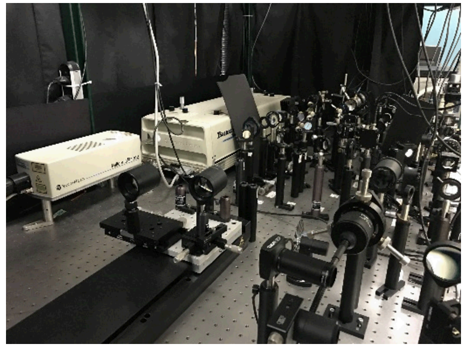
Bioscience



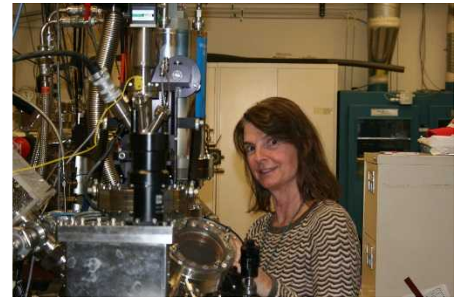
Missions: NW, Defense/Intelligence/Homeland Security, Energy/Climate

SNL Nanoscale Sciences Department R&D Activities

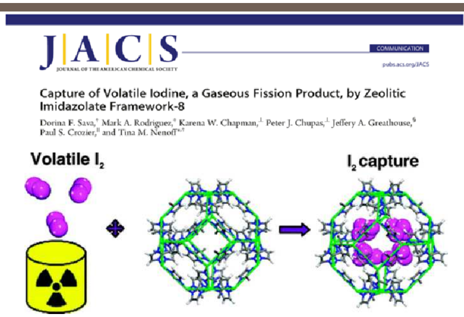
Sandia National Laboratories

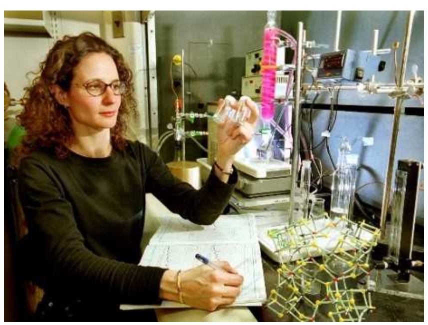


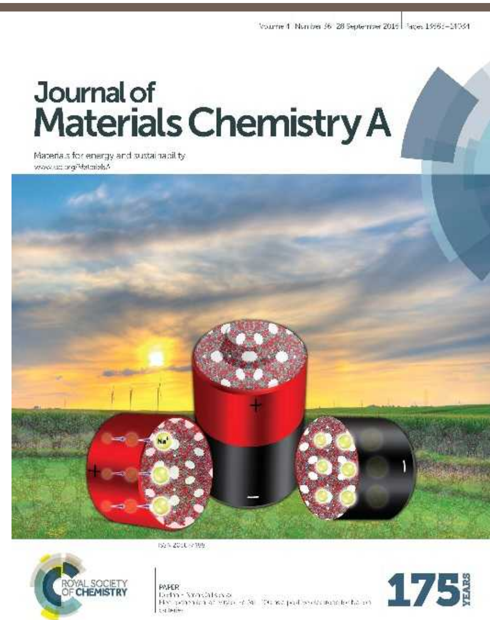
Thermal Imaging, Characterization & Spectroscopy



Thin Film and Nanotemplate Materials

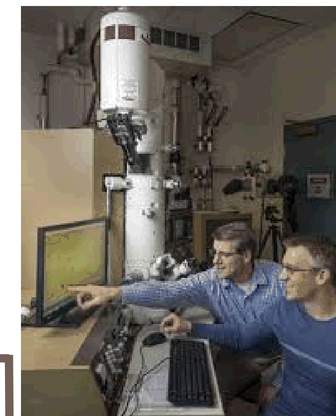


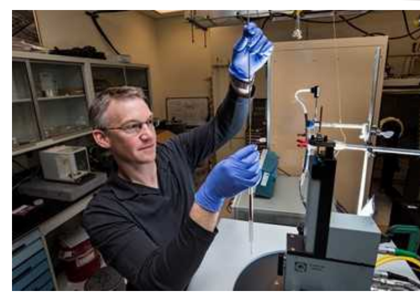


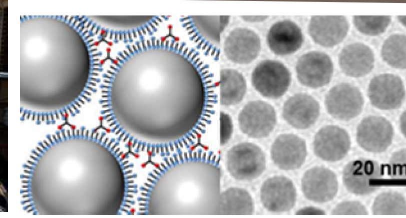


Materials and Electro Chemistry Synthesis and Characterization

Electron & Scanning Probe Microscopy







Magnetic Materials and Characterization

Magnetic Device Materials impact many National Security Needs

Applications:

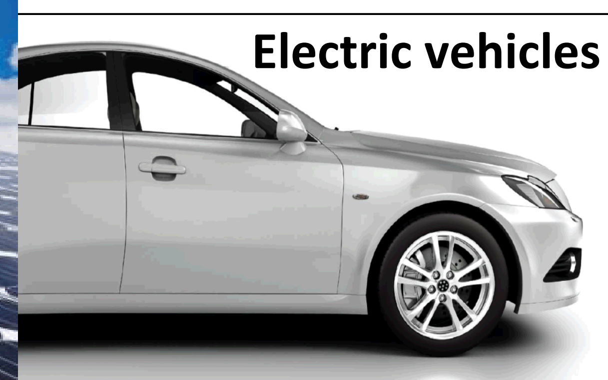
- Permanent Magnet based Actuators
- Passive Components for Power Electronics (i.e., inductors)
- Advanced Electronics (including Beyond Moore and Quantum Computing)
- Magnetic Sensors and Tags
- RF Applications

Typical Performance Needs:

- High frequency (into GHz)
- Reduced losses during operation (i.e., due to eddy currents)
- Small form factor (i.e., high saturation magnetization materials)
- Environmental stability in demanding environments
- Manufacturable

Key Challenge: Technology Readiness Level (TRL) Maturation, need academic and other partners

Power Electronics (and Therefore Inductive Elements) are Ubiquitous



Courtesy of Bob Kaplar

Soft Magnetic Devices Impact Power System Volume and Weight

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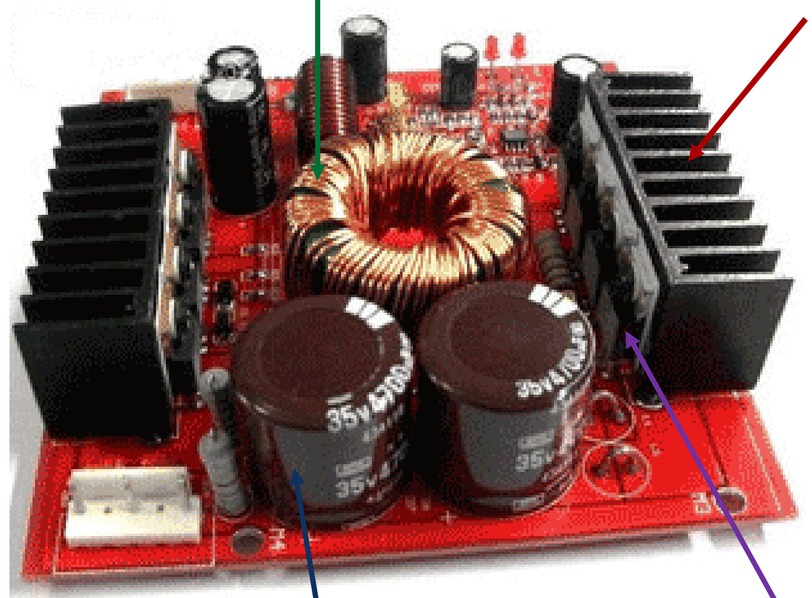
Passive elements and thermal management comprise the bulk of the volume and mass of a power converter

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

Magnetics

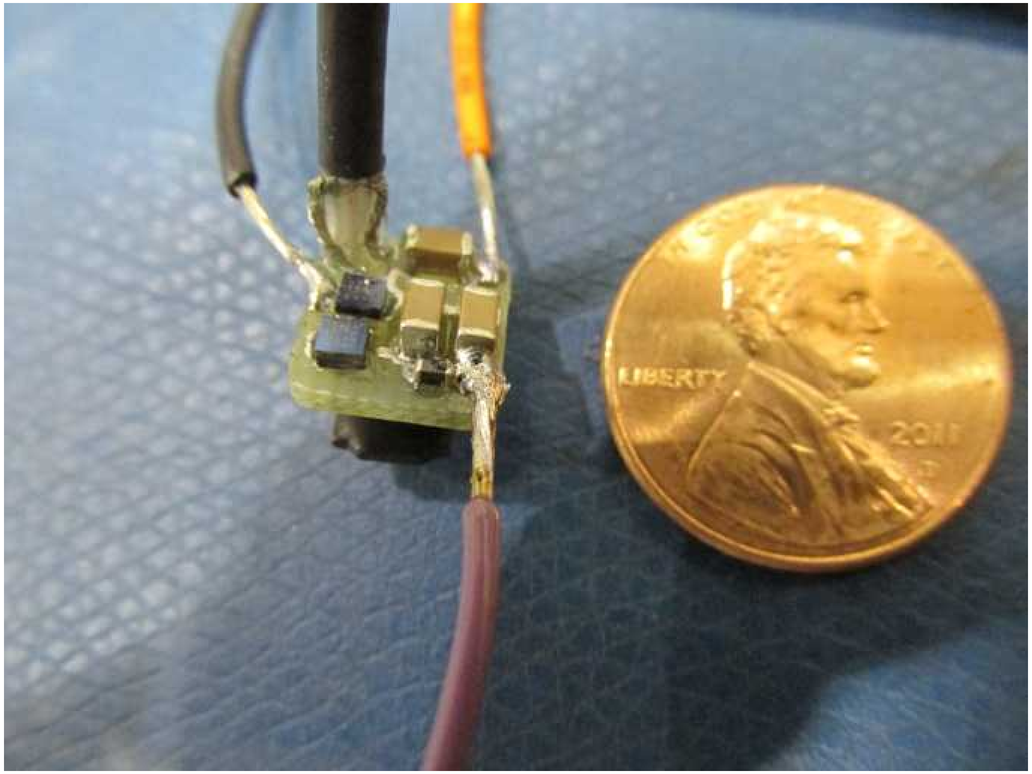
Thermal management



Capacitors

Semiconductor switches

Concept: Miniaturized power electronics

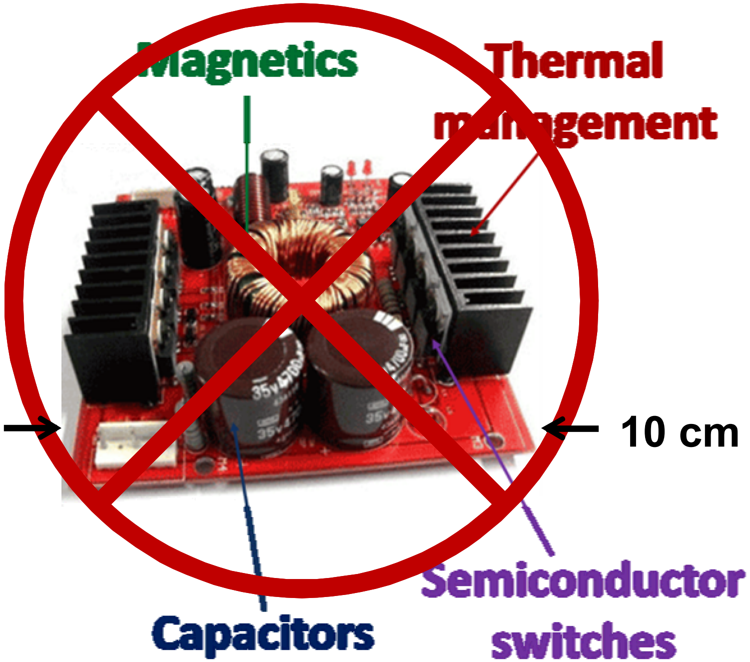


Courtesy of Jack Flicker

- Achieved: GaN ⇒ 215 W/in^3
- 92 V, ~92 mA ⇒ 8.5 W, 215 W/in^3, 1 MHz

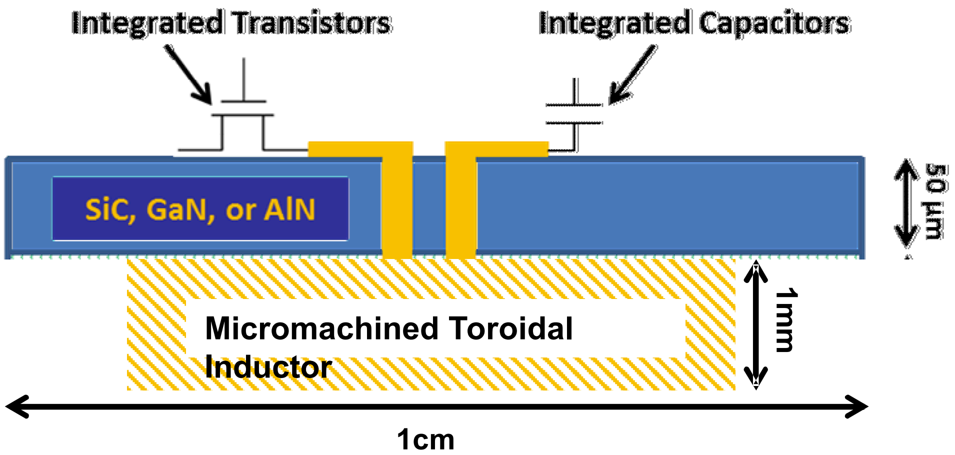
Status Quo and Future State

Status Quo



Discrete components on PCB

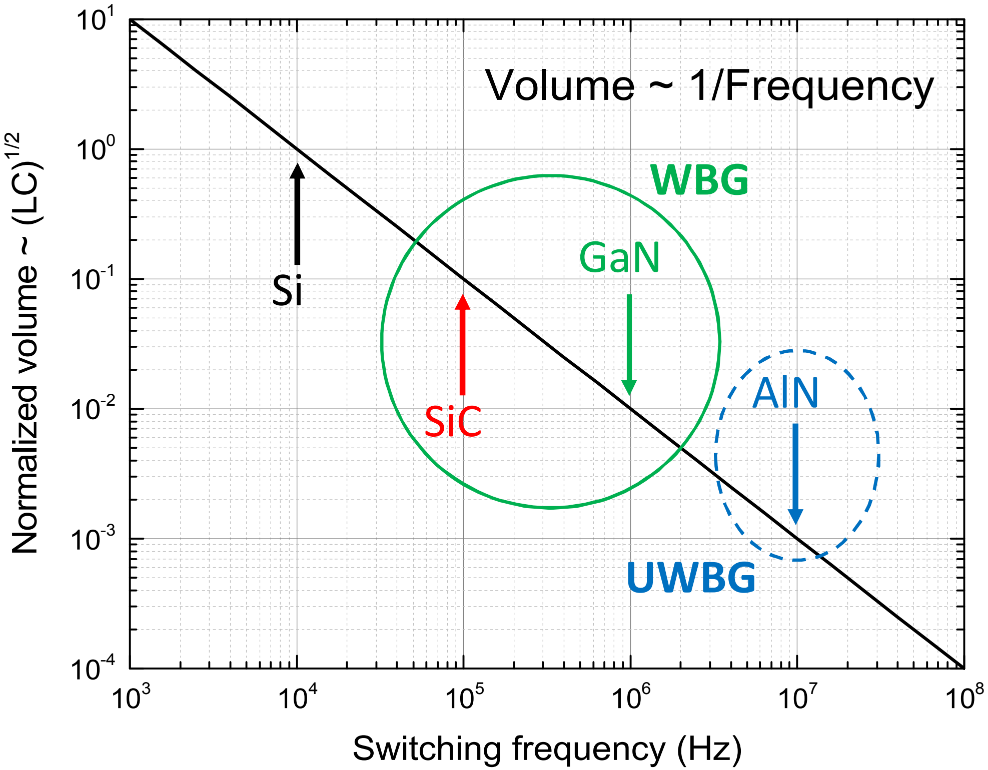
Future State



Power Supply on Chip (PSoC)

#1 figure of merit: reduced form factor!

Higher Frequencies Decrease Inductance Requirements, However…



- **Higher switching frequency is enabled by scaling properties of WBG/UWBG materials**
- **Ideal 1/f SWaP dependence (true dependence likely weaker $1/f^n$ with n < 1 due to nonidealities)**
- **Other benefits exist, e.g. higher voltage without series stacking of devices, and higher temperature operation**
- ***UWBGs may be required for specialized applications such as pulsed power***

Inductive core materials have essentially been an afterthought and new magnetic materials are needed

Adapted from Bob Kaplar

Transformer EMP Vulnerability

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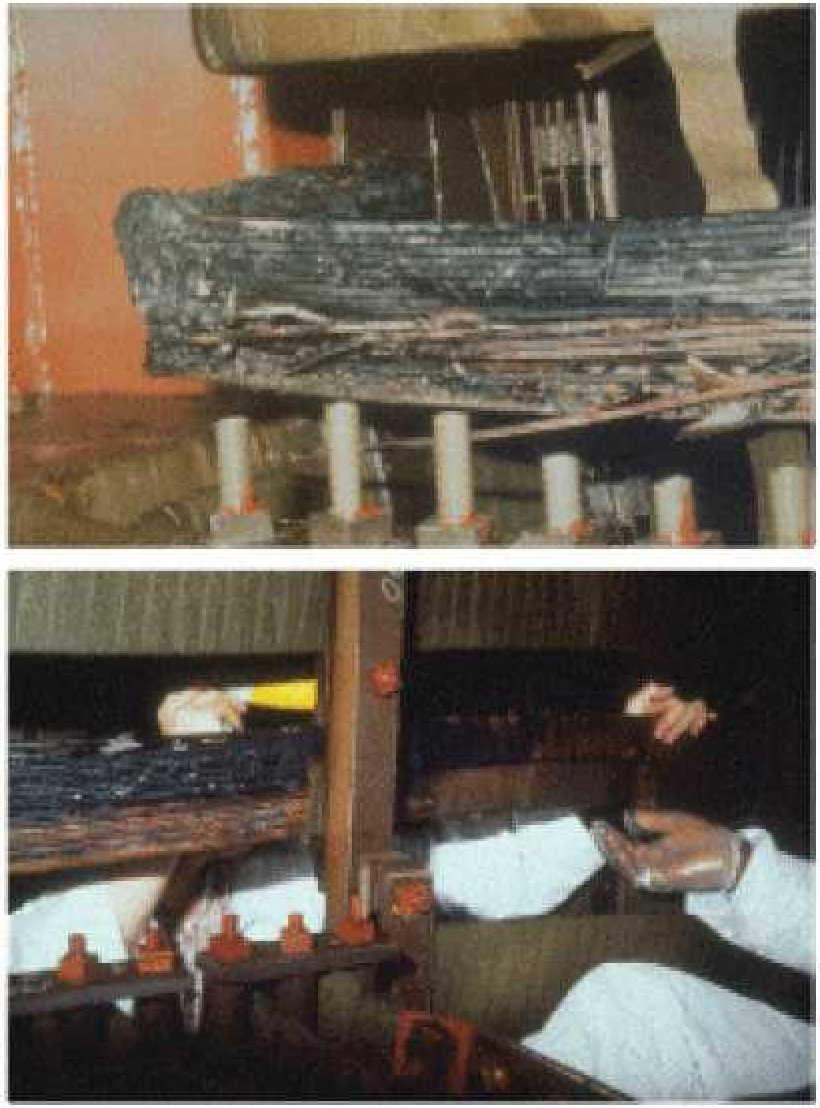
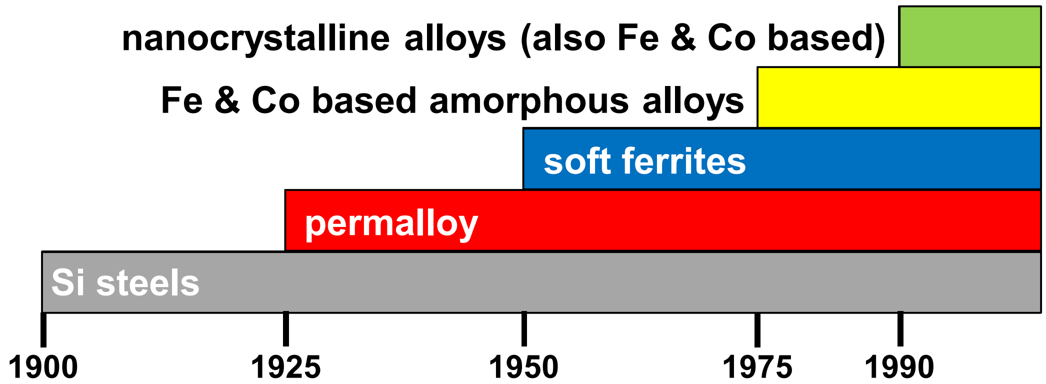


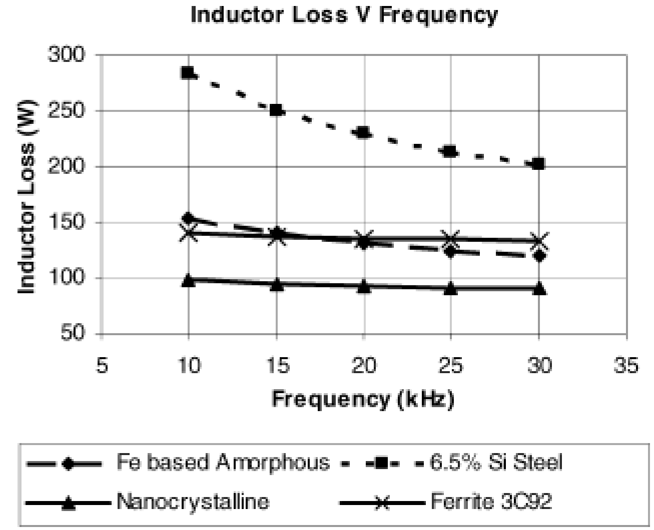
- **Geomagnetic storms couple very efficiently to long transmission lines**
- **Transformer can be driven to saturation, creating harmonics and reactive power**
- **Enough heat generated to melt copper windings**
- **Transformers the grid component that is hardest to replace**

Permanent damage to the Salem New Jersey Nuclear Plant GSU Transformer caused by the March 13, 1989 geomagnetic storm. Photos courtesy of PSE&G.

Soft Magnetic Material Development



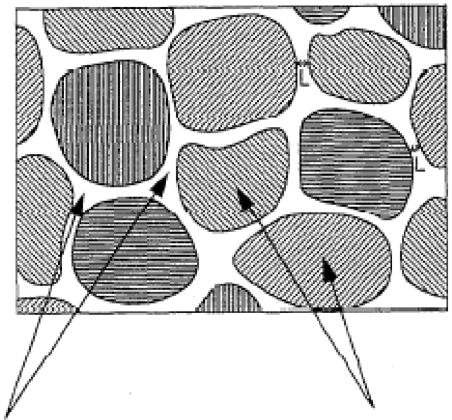
Adapted from: L.A. Dobrzański, M. Drak, B. Ziębowicz, Materials with specific magnetic properties, Journal of Achievements in Materials and Manufacturing Eng., 17, 37 (2006).



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 (Fexxocube)	0.52	$5x10^6$	Low
Si steel	1.87	0.05	Low
γ'-Fe_4N	1.89	~200	Low

Nanocrystalline Alloys & Manufacturing

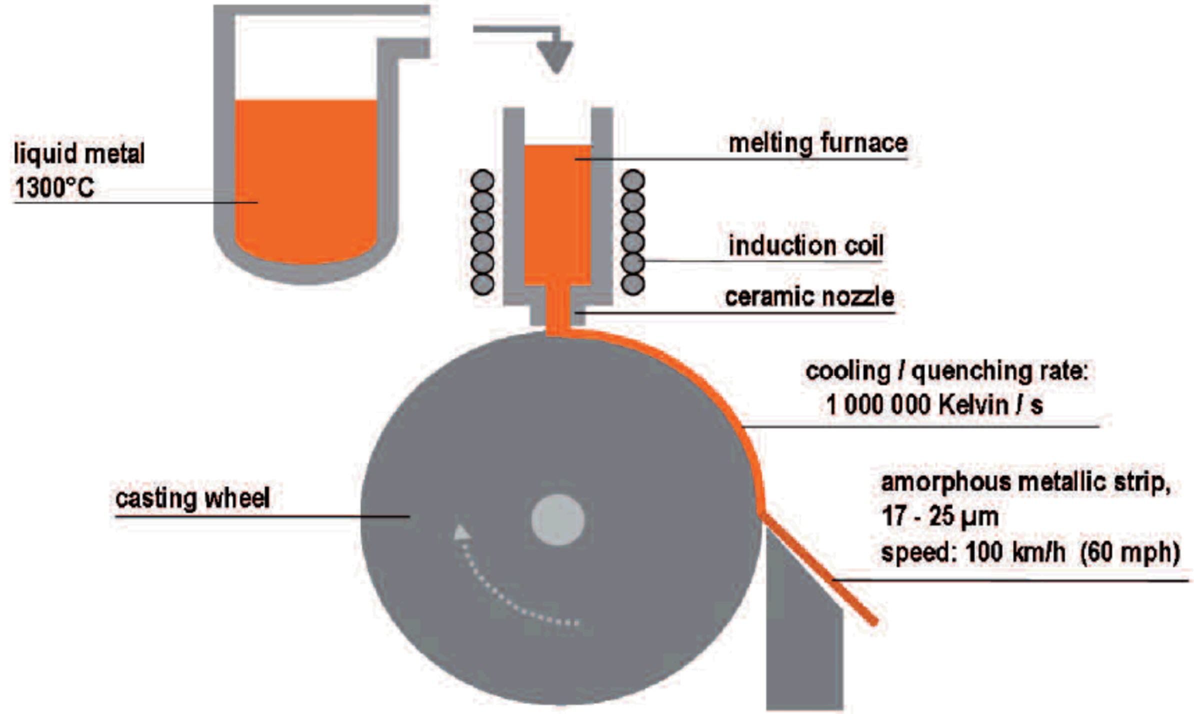


Intergranular amorphous phase with high Tc and high thermal stability due to large amounts of M and B elements.

Nano-scale α-Fe grains with small λ due to small amounts of M and B elements.

"NANOPERM"

A. Makino, et. al., Nanocrystalline Soft Magnetic Fe-M-B (M = Zr, Hf, Nb) alloys and their applications, Mat. Sci, and Eng., A226-228, 594 (1997).



VITROPERM
(Vacuumschmelze)

- **Complex stoichiometries including Fe, Co, and other inactive elements such as B, Zr, Hf, Nb, Cu, Mo, Si, C**
- **Time consuming and high temperature processing → costly!**
- **Substantial inactive material to form a low loss nanocrystalline structure**
- **Material produced in tapes and often combined with plastic laminations**

Coercivity as a Function of Particle Size

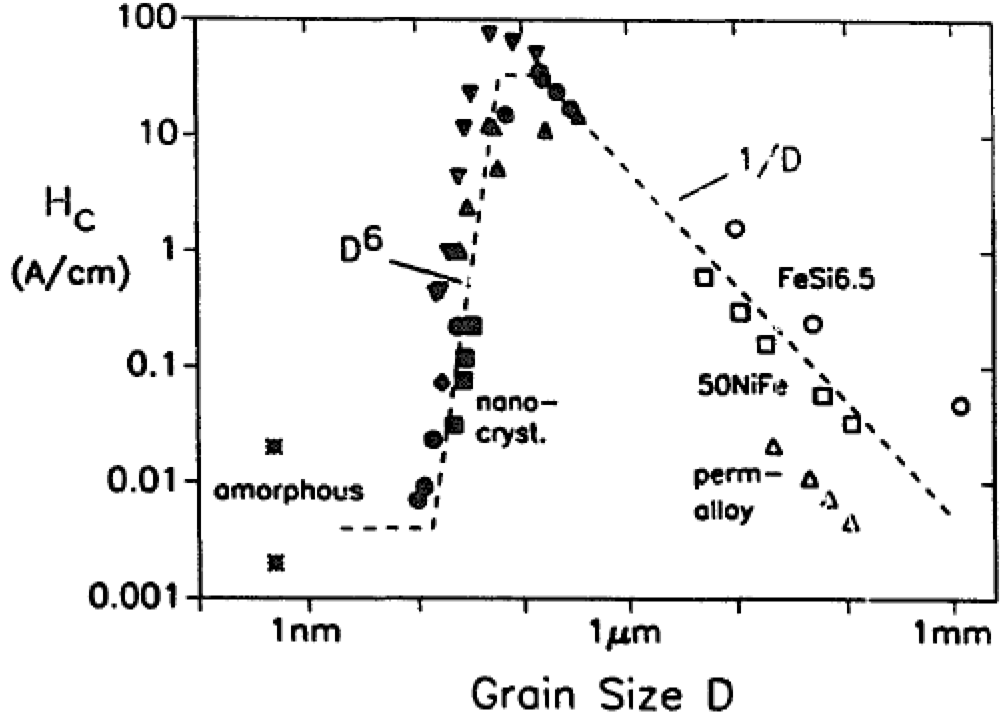


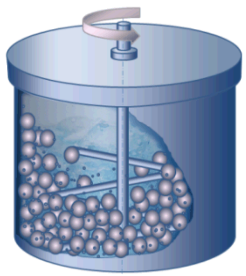
Fig. 2. Coercivity H_c vs. grain size for various soft magnetic metallic alloys. The data of the nanocrystalline material refer to (▲) FeNbSiB and (●) FeCuNbSiB [14], (◆) FeCuVSiB [15], (■) FeZrB [4] and (▼) FeCoZr [16].

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

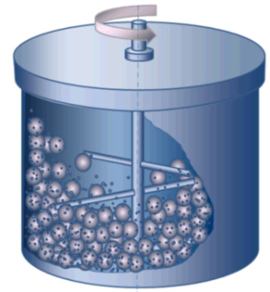
γ'-Fe_4N Synthesis and Processing

U.S. Patent Filed January 2015 (#62/105,918)

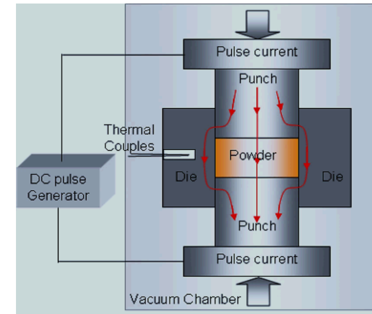
Synthesis of dense nanocrystalline iron nitrides using a two-step reactive milling and high pressure spark plasma sintering (SPS).



Liquid N_2



NH_3

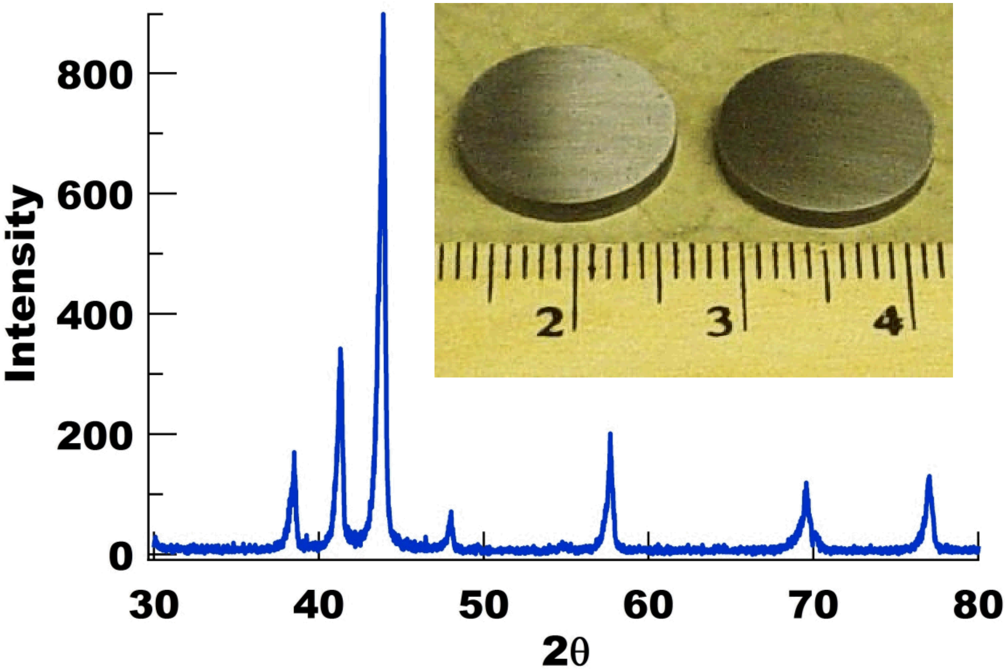


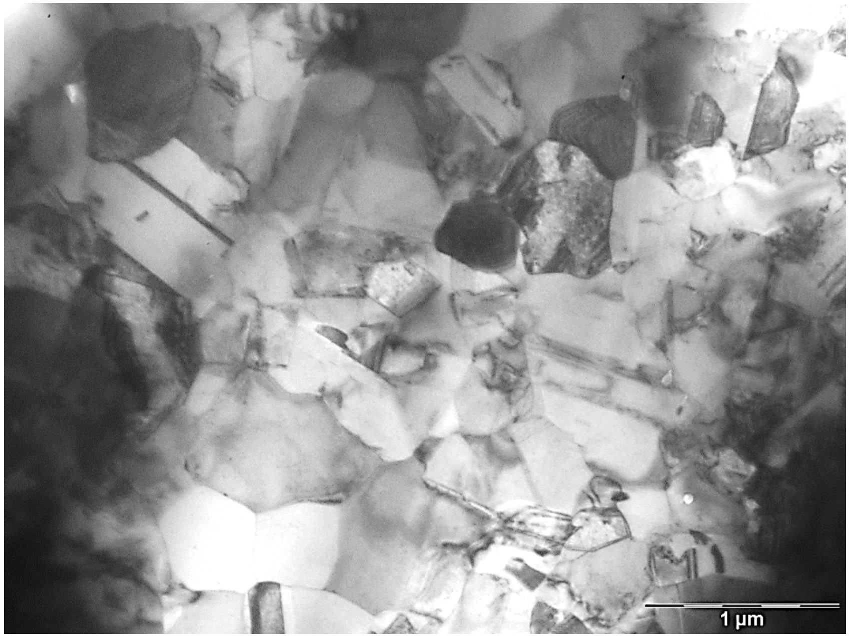
Spark Plasma Sintering (SPS)

- Cryomilling creates nanocrystalline Fe powder with large amounts of vacancies, grain boundaries, and dislocations
- Defects serve as fast diffusion pathways for nitrogen atoms from NH_3
- SPS quickly consolidates raw powders with a low sintering temperature
  - Excellent control over grain growth
  - Result: Improved magnetic properties

SPS consolidated Iron Nitride

First ever bulk γ'-Fe_4N!

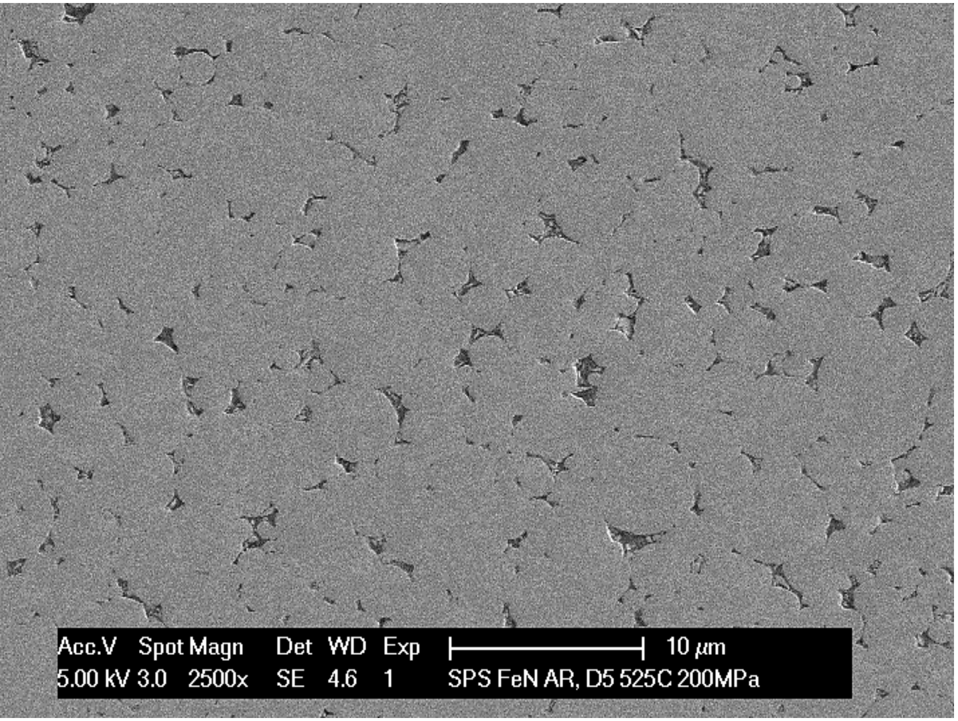




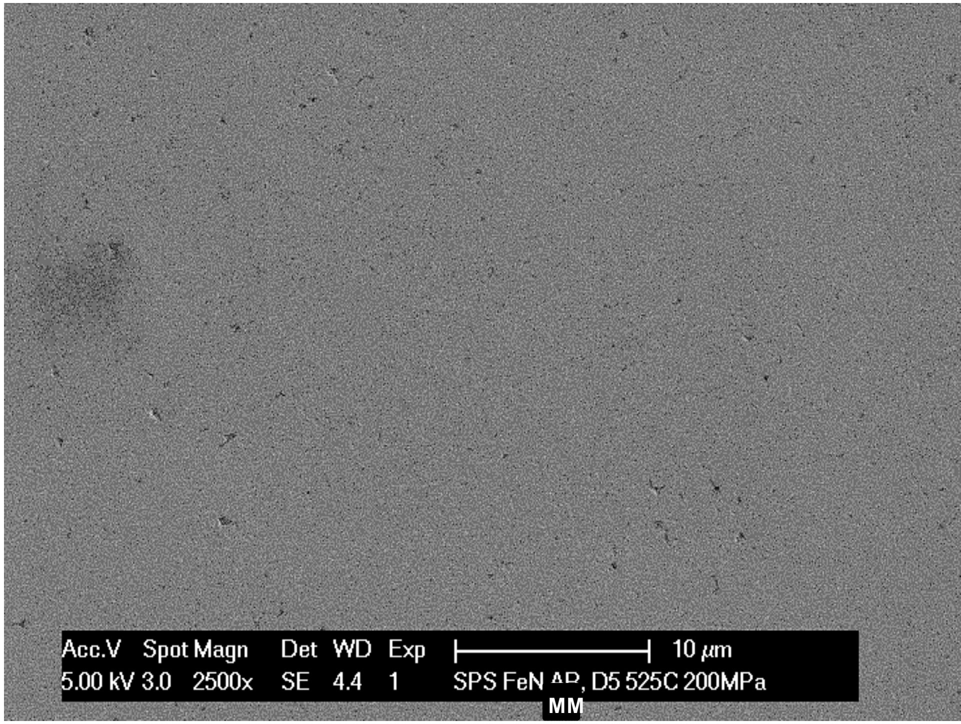
- Fe nitride powders well consolidated with little porosity
- Grain sizes 200 nm – 1 µm → fine grain size = low loss
- γ'-Fe_4N primary phase
- Fe_3N secondary phase from mixed phase starting material

SEM of SPSed FeN samples, (Ø5mm, 525°C, 200MPa)

W/ as-received FeN powder

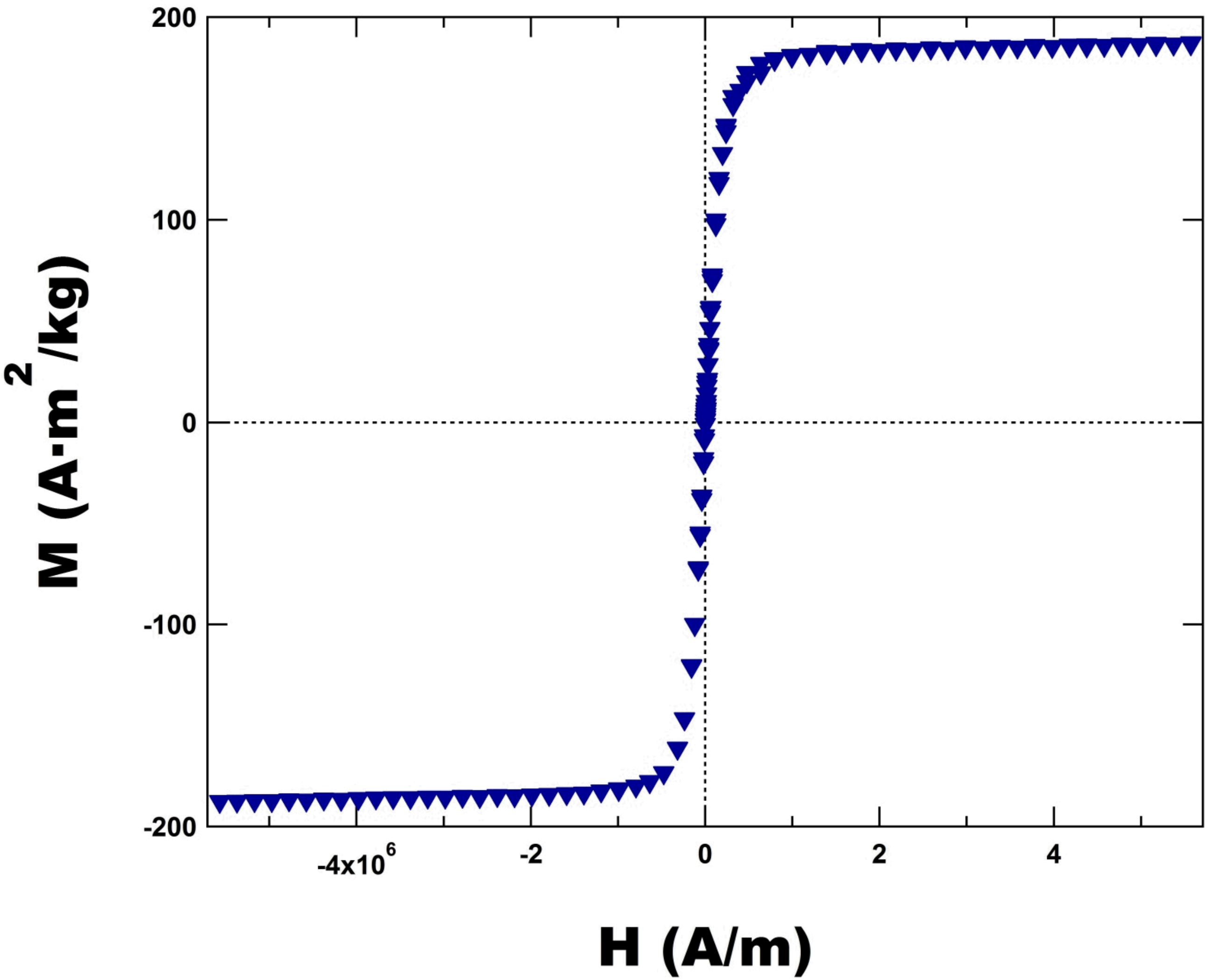


W/ milled FeN powder



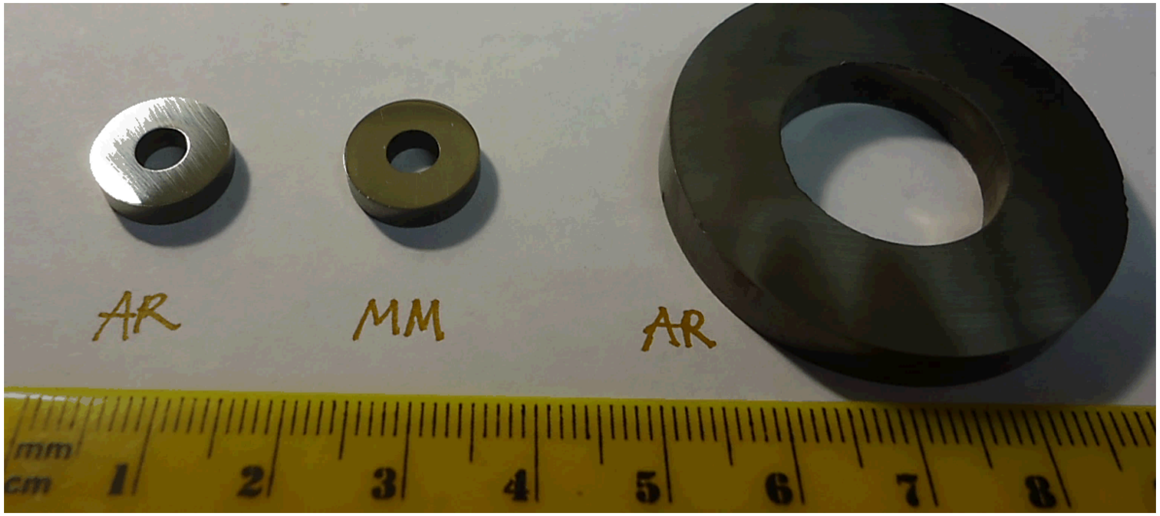
- **Milled FeN powder produces more uniform and dense SPSed billets**
  - **Higher packing density with smaller particle size**
  - **Increased diffusion ability with smaller grain size of milled powder**

Magnetic Characterization



- Fe_4N SPSed at 550°C and 100 MPa achieved an M_{sat} of 188 $A{\cdot}m^2/kg$.
- Predicted M_{sat} of bulk γ'-Fe_4N is 209 $A{\cdot}m^2/kg$ (Fe is 217 $A{\cdot}m^2/kg$)
- Negligible coercivity

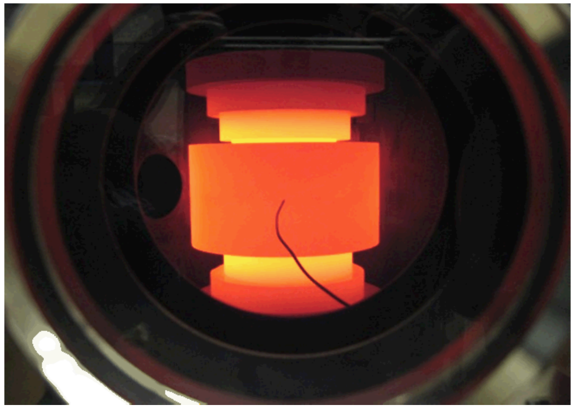
Net-Shaping of Transformer Cores



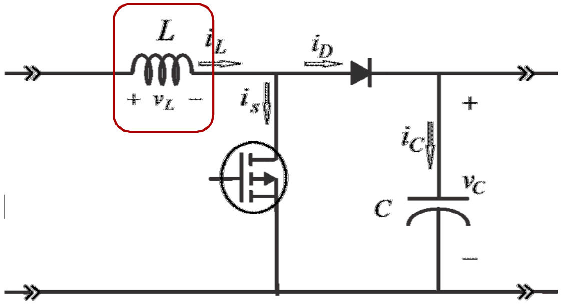
- Can sinter toroids and other shapes directly (net-shaping)
- Eliminates the need for machining
- Toroids being wound and tested

γ'-Fe_4N Conclusions

- γ'-Fe_4N has the potential to serve as a new low cost, high performance transformer core material
  - M_{sat} > Si steel
  - Increased current and field (and therefore power) carrying capability
  - Resistivity 200X greater than nanocrystalline and amorphous alloys
  - Only requires low cost and abundant elements (Fe & N)
  - High temperature (T) operation complementing Sandia development of high T capacitors and WBG semiconductors
- The fabrication of bulk γ'-Fe_4N using SPS has been demonstrated
  - SPS can consolidate iron nitrides without material decomposition
  - Parts can be fabricated directly using net-shaping



Another interesting approach for soft magnetic composites

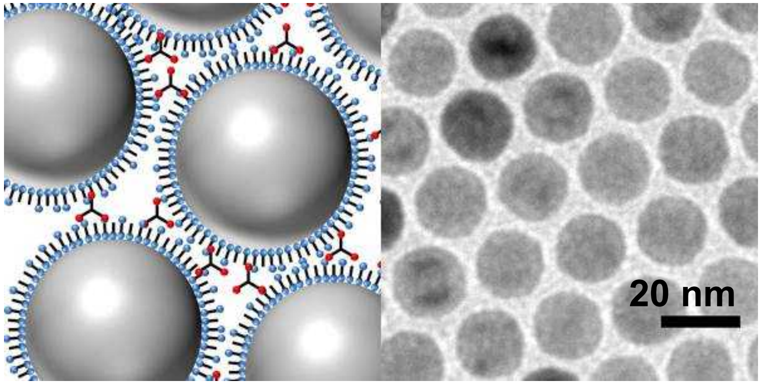


Develop new inductor materials made from superparamagnetic nanoparticle composites

- Eliminates main loss mechanisms: hysteresis and eddy cur
- Inductors can be "tailor made" (i.e. 3D printed)
- Achieve B_{max} >1.0 T at 10 MHz

The S&T Challenges

- Magnetic nanoparticle size will need to be tightly controlled to eliminate hysteresis
- Material will need to be optimized for flux density/rad-hardness



All particles magnetize at the same rate, allowing maximization of magnetic saturation with zero magnetic hysteresis.

Su

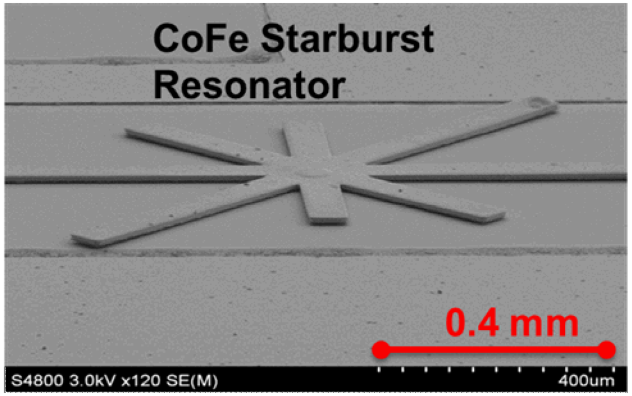
- New Synthesis and Characterization tools offer a fresh perspective towards generating new magnetic device materials for a wide variety of emerging needs

- R&D and Technological Maturation needs create an opportunity for creative partnerships between National Labs like SNL with academic, government lab and industry partnerships

Future:

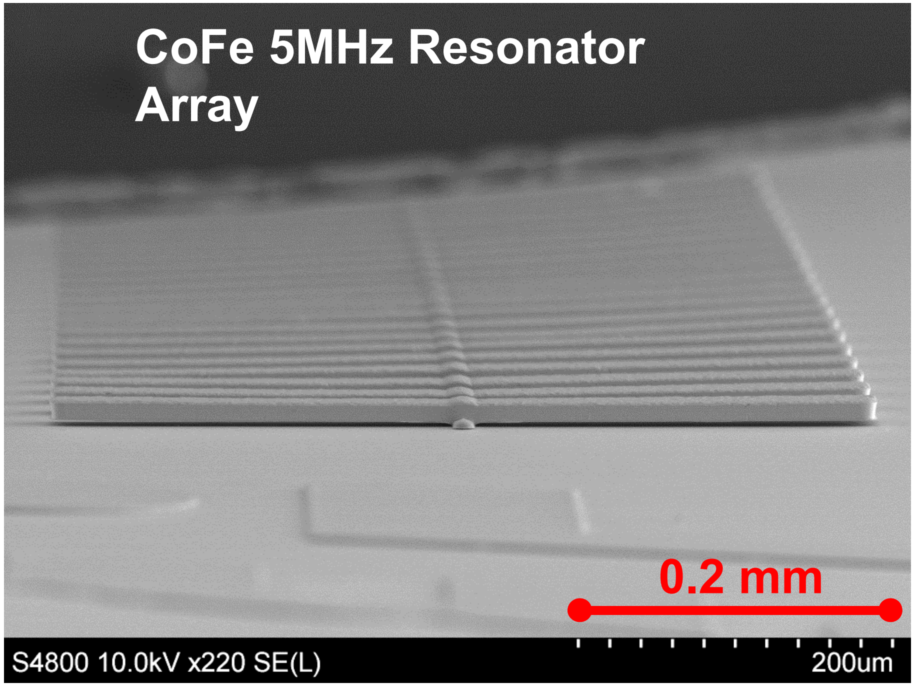
- Advances in Electrodeposited Magnetic Materials for Compact MEMS Magnetoelastic Resonators and Magnetostrictive Sensors
- Magnetic Josephson Junction Device Materials for enabling superconducting electronics (for Beyond Moore's Law)

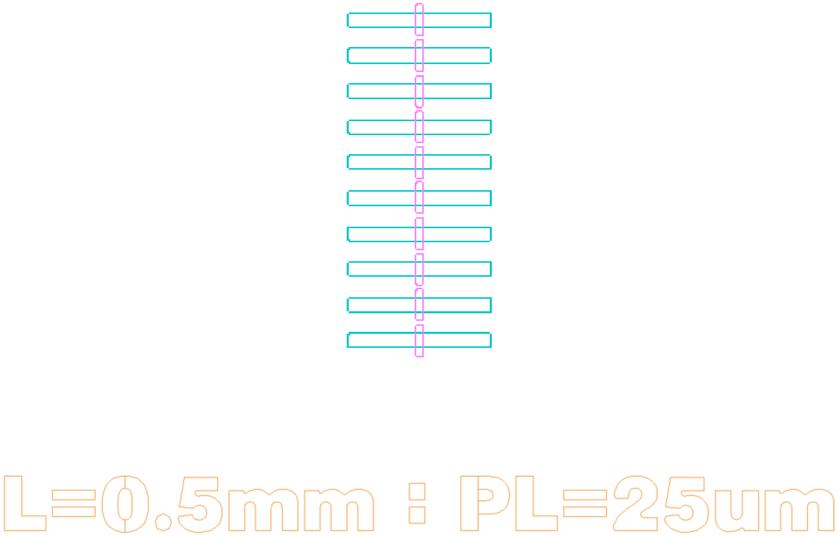
2.4 mm long freestanding resonator



Multi-Frequency Micro Resonator, "Starburst Pattern"

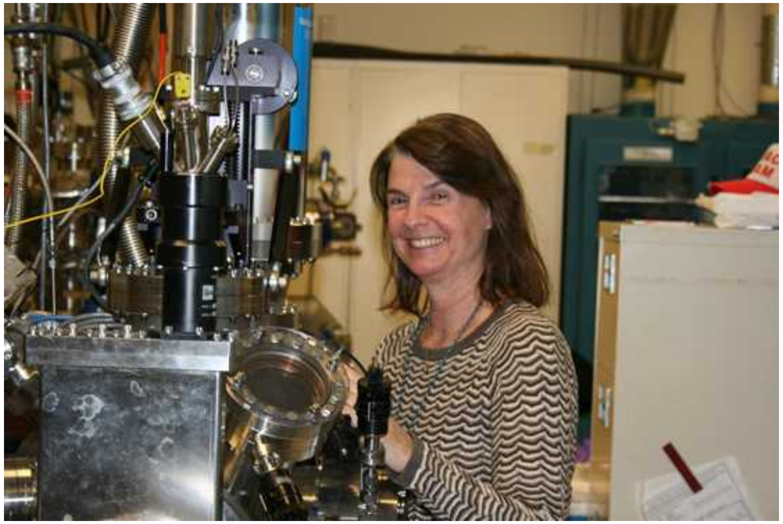
First ever electroplated CoFe micro-resonators

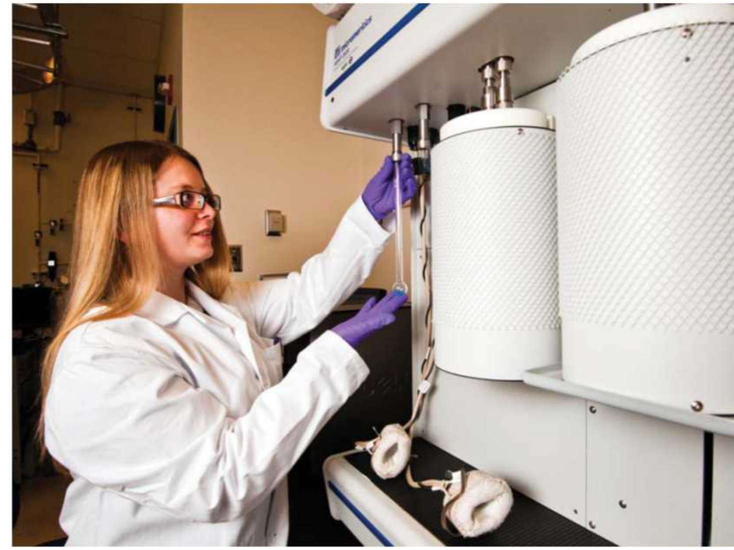


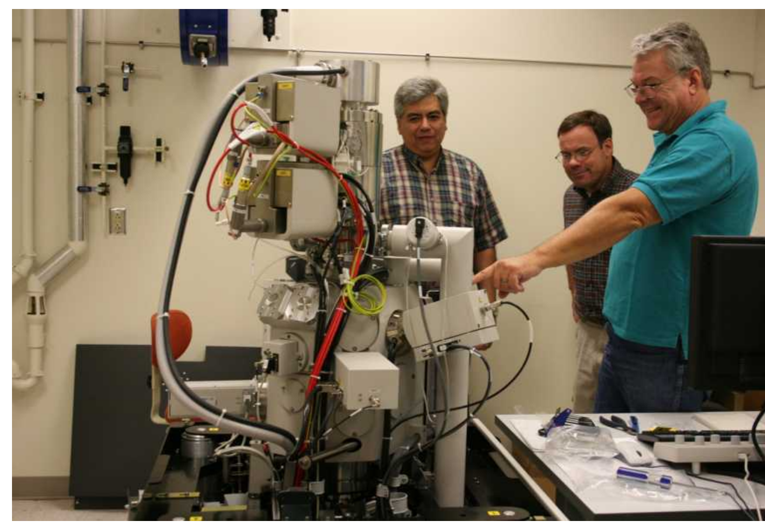


- Resonator arrays increase device signal amplitude
- Via proper design resonators can measure a host of environmental parameters
- Temperature, pressure, current, gases, stress/strain, angular position, and many more!

Questions?







Back Up Slides

Sandia's Governance History & Structure

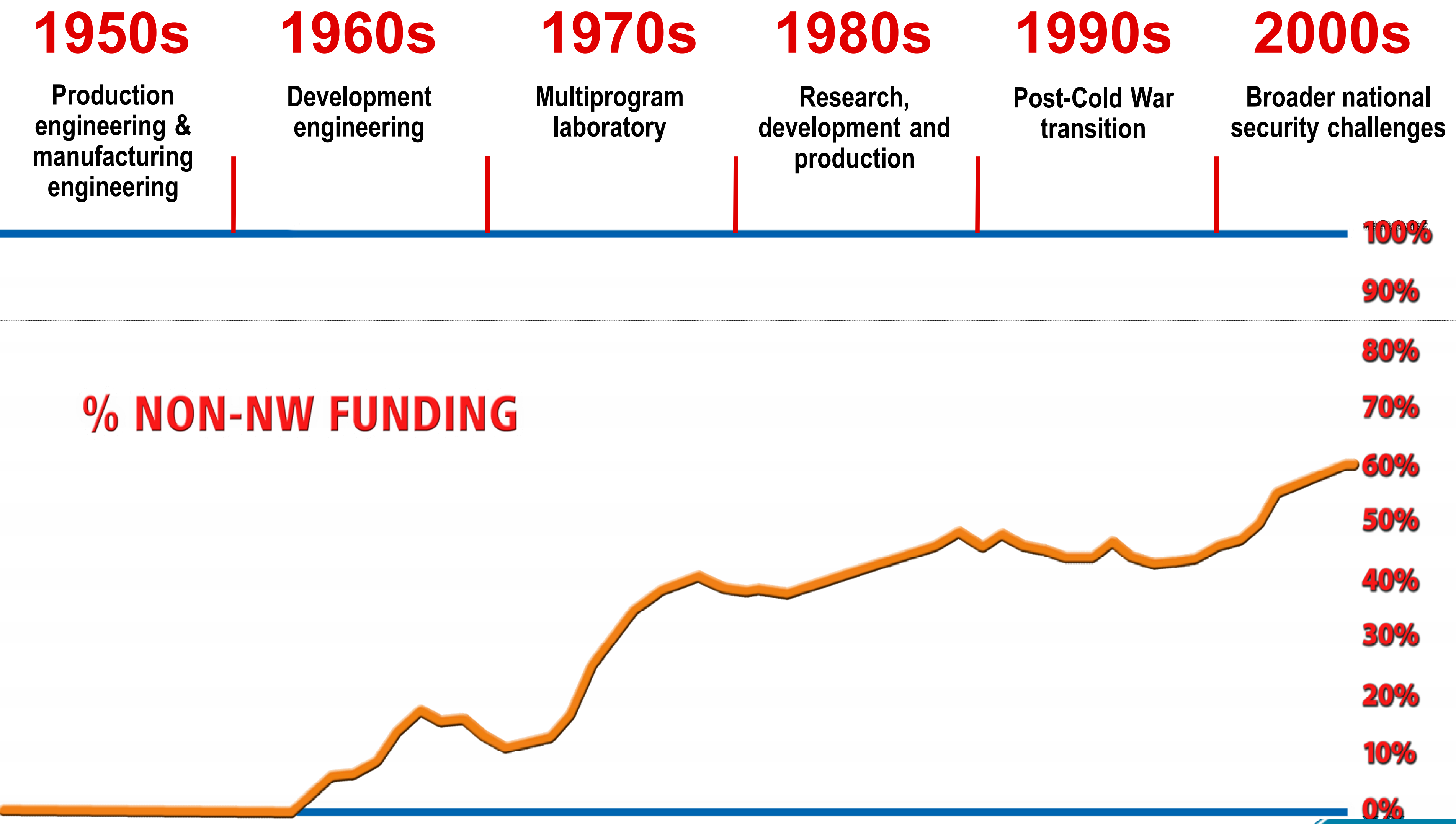
Sandia Corporation

- AT&T: 1949–1993
- Martin Marietta: 1993–1995
- Lockheed Martin: 1995–2017
- NTESS (Honeywell): 2017-present

GOCO: Government owned, contracto
operated

research and development center

Sandia's Mission Has Evolved

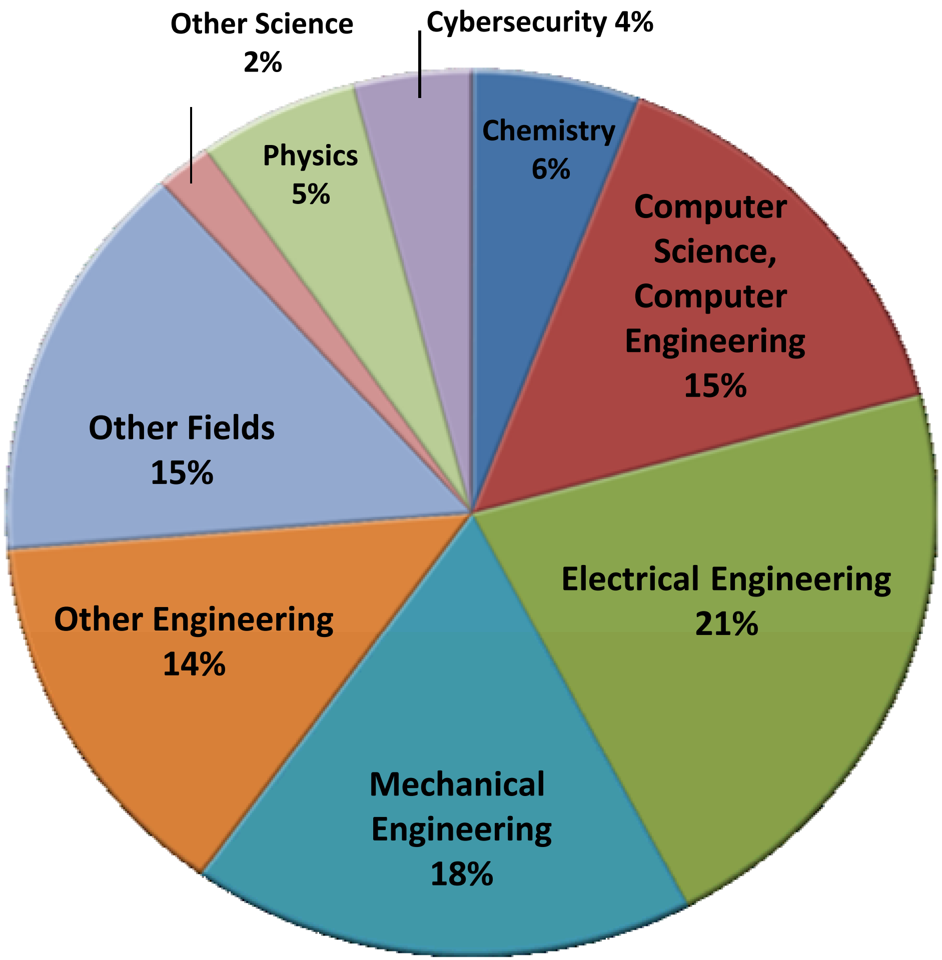


Sandia's Workforce

- On-site workforce: 11,711
- Regular employees: 9,494
- Advanced degrees: 5,330

Data as of April 22, 2014

R&D staff (5,000) by discipline



General SNL Career Categories Engaged in Science and Engineering

Researchers:

- Technical Staff – generally PhD (sometimes MS in mission application areas)) Scientists and Engineers who take leadership roles in defining R&D
- Technologists – HS, associates degree, bachelors degrees and masters degree educated people in technical areas who support Technical Staff
- Postdocs – temporary (2-3 years), sometimes are not US citizens
- Undergraduate and Graduate Student Interns (summer and year round, US citizens)
- Visiting Faculty

Research Managers – usually PhD educated, often promoted from experienced technical staff; responsible for enabling the technical staff and setting coherent "big picture" research directions

Sandia targets the "best and the brightest" for technical staff, i.e. the top 10% of all college graduates. GPA's must be ≥ 3.0/4.0. Scientific leadership and initiative is desired as much as teamwork and good human interaction skills.

Benefits of a High Frequency Transformer

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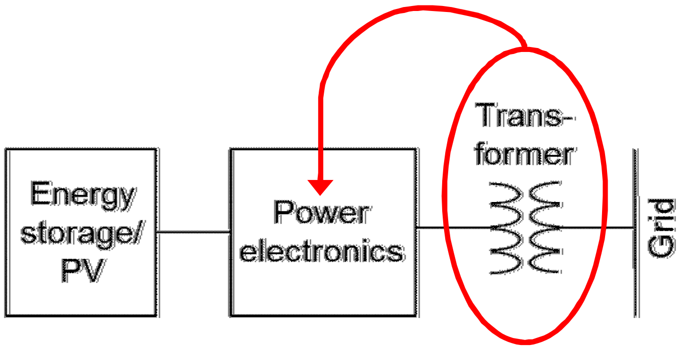
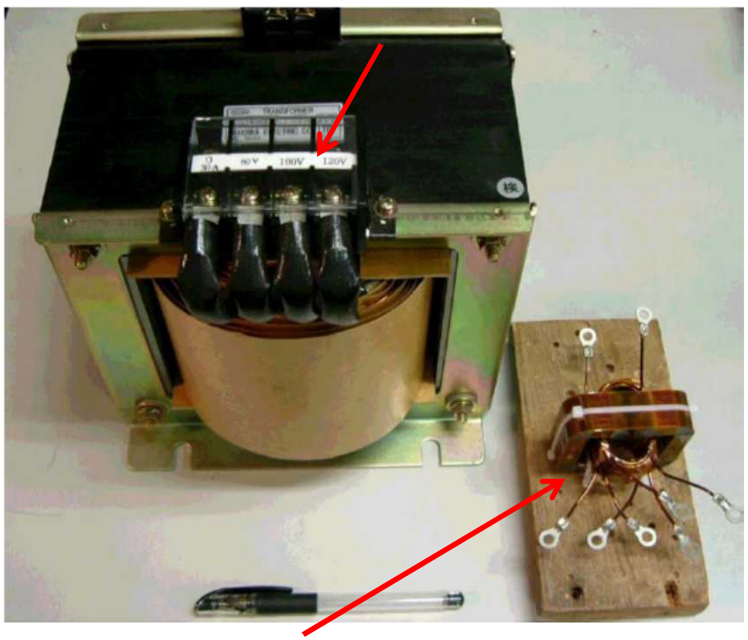


Line frequency (50 Hz) transformer



High frequency (20 kHz) transformer

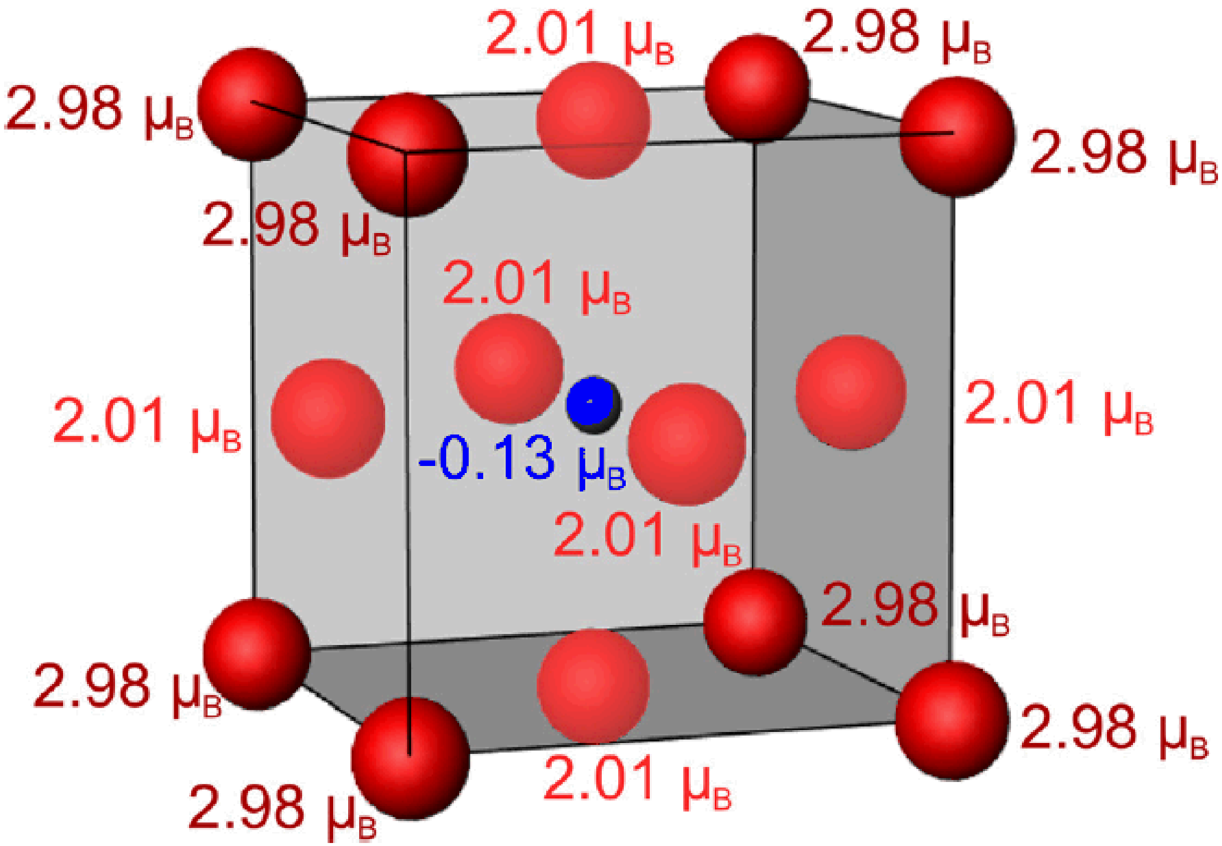
S. Krishnamurthy, Half Bridge AC-AC Electronic Transformer, IEEE, 1414 (2012).

- Integrate output transformer within power conversion electronics
- Leverage high switching speed, voltage, and temperature performance of WBG semiconductors
- Transformer core materials for high frequency transformers have been an afterthought (no current material meets all needs)

Material requirements:

- Low loss in 10-200 kHz frequency range
- High permeability (low coercivity)
- High saturation magnetizations
- Low magnetostriction
- High temperature performance
- Scalable & affordable

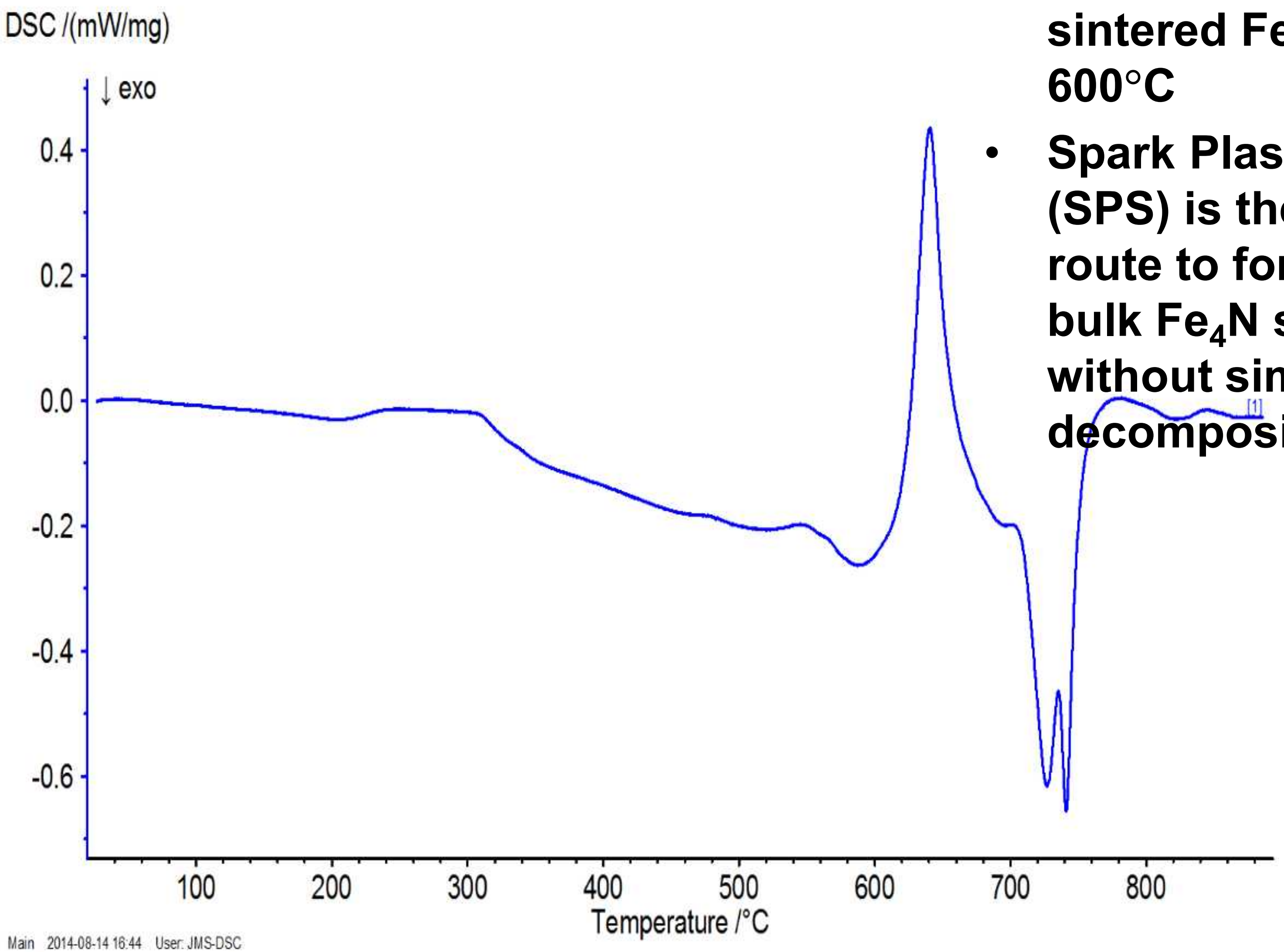
γ'-Fe_4N Unit Cell



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).

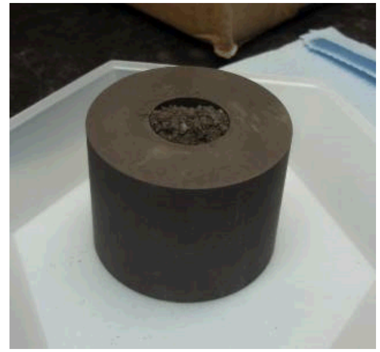
Differential Scanning Calorimetry (DSC) of Fe_4N



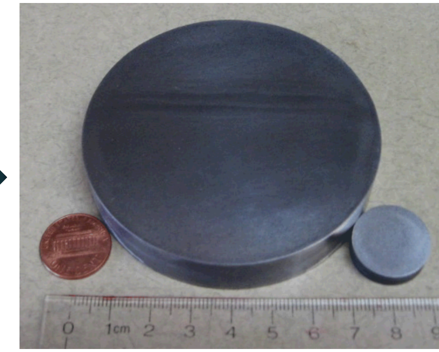
- **Decomposition of sintered FeN begins ≈ 600°C**
- **Spark Plasma Sintering (SPS) is the only viable route to form fully dense bulk Fe_4N samples without simultaneous decomposition**

Spark Plasma Sintering (SPS)

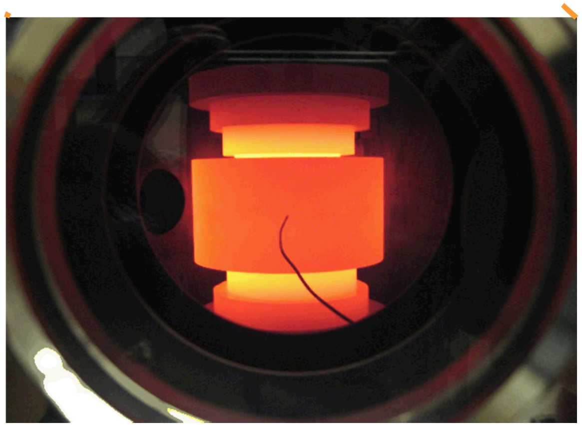
SPS Model: SPS-825S Dr. Sinter® at UC Irvine



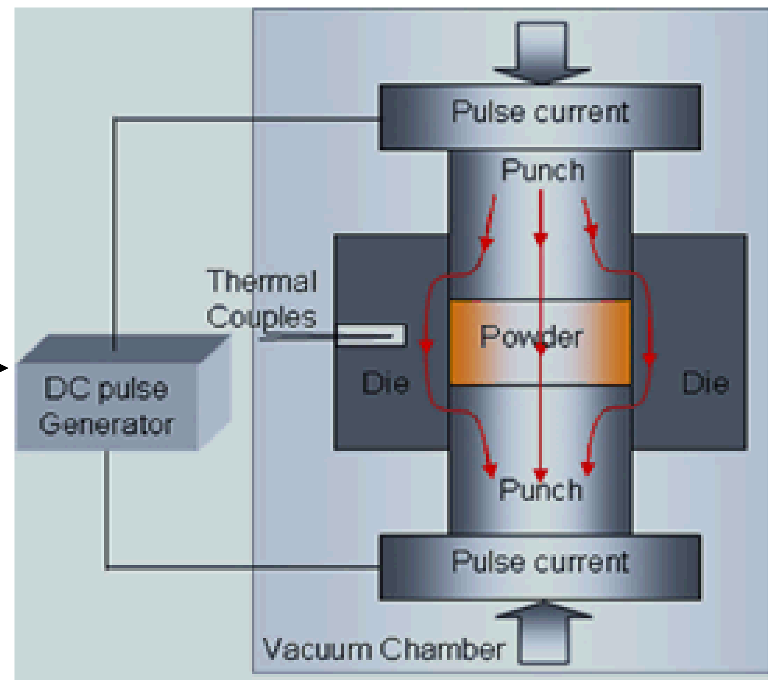
Starting Powder in Die



End Product

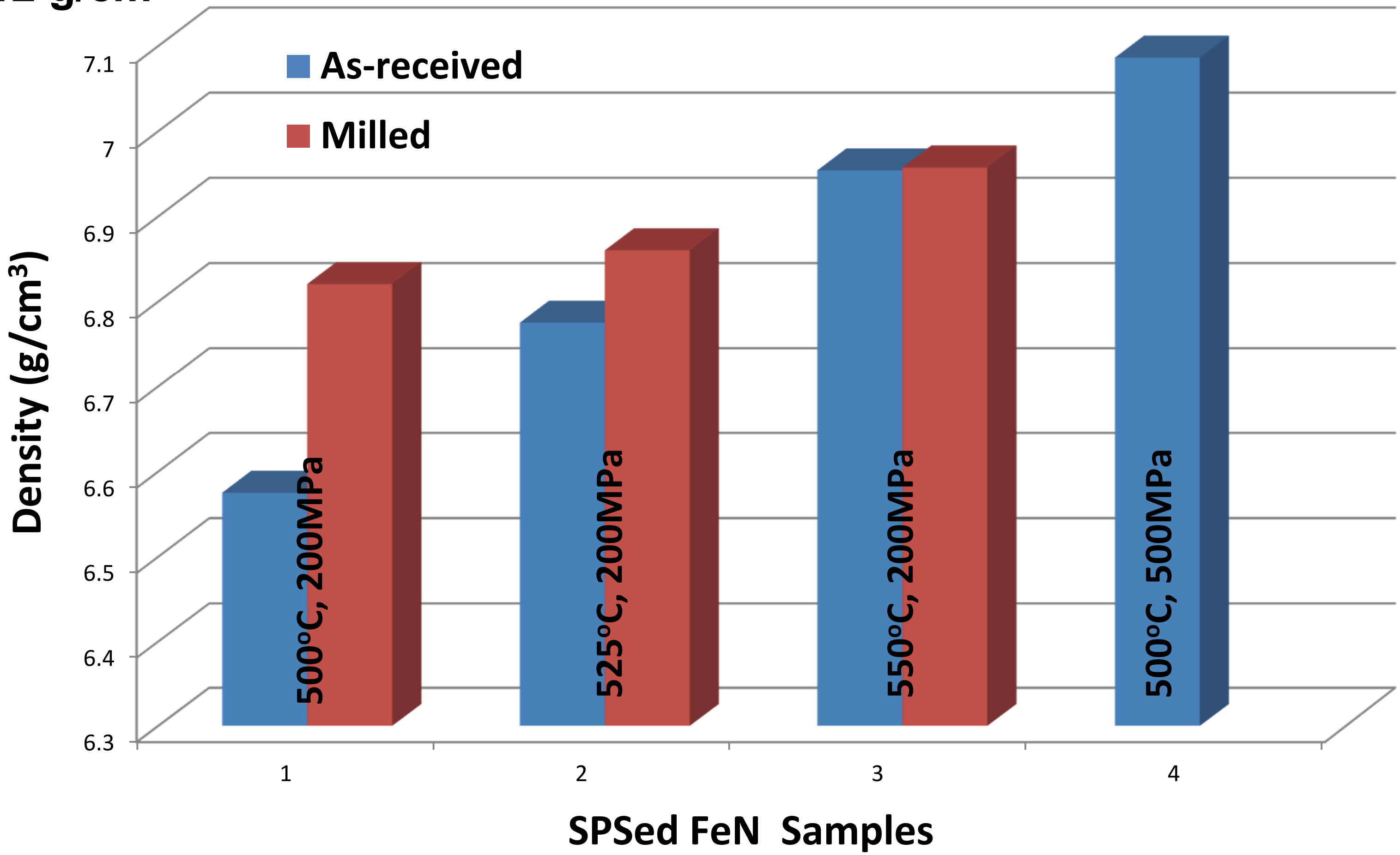


SPS
Chamber



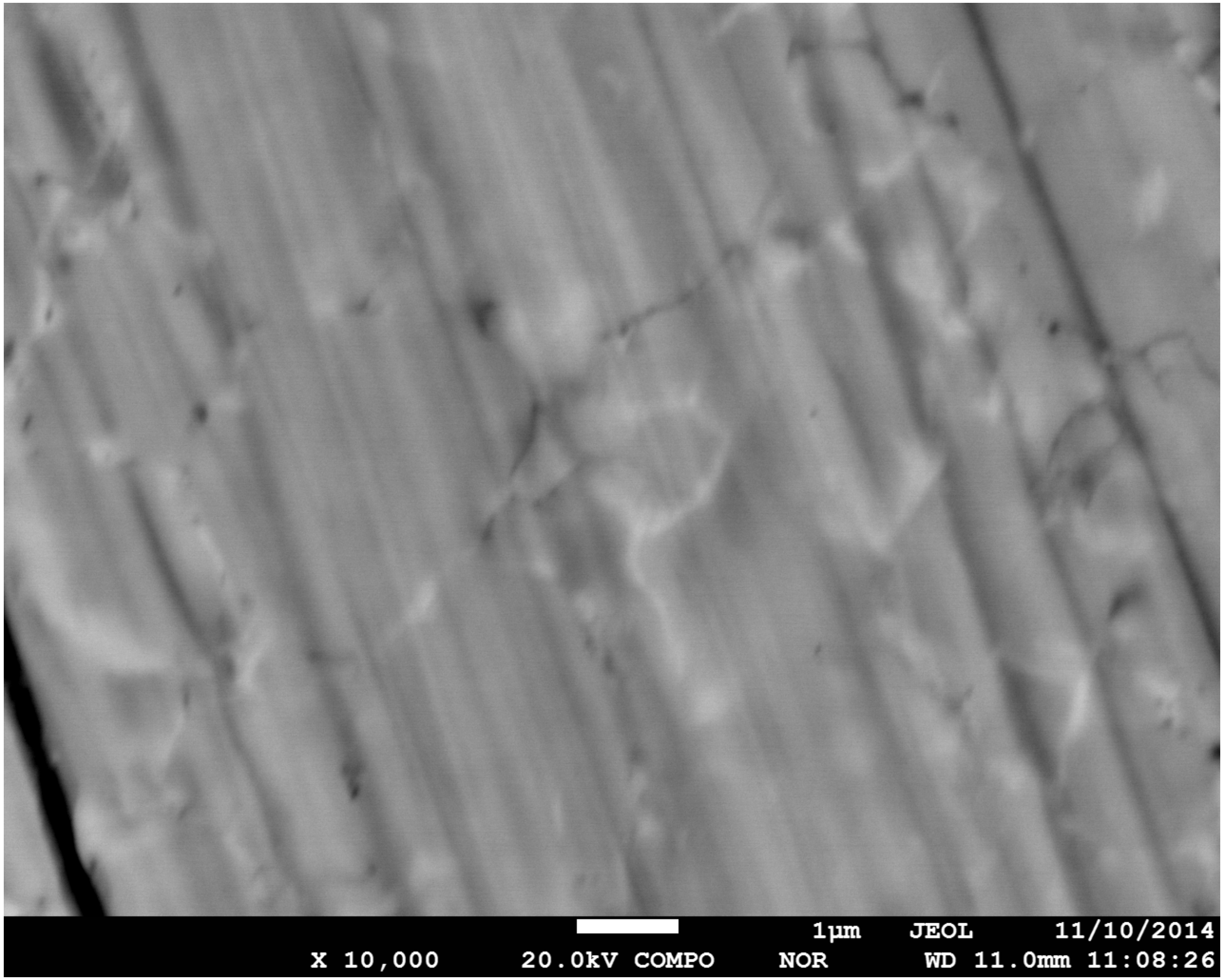
Density of SPSed Fe_4N samples

$\rho_{theory} = 7.212$ g/cm^3



- Density increases with increasing SPS temperature and pressure
- Higher degree of variation in SPSed samples using as-received powder
- Milling improves density and uniformity

Toroid Surface SEM and EDS

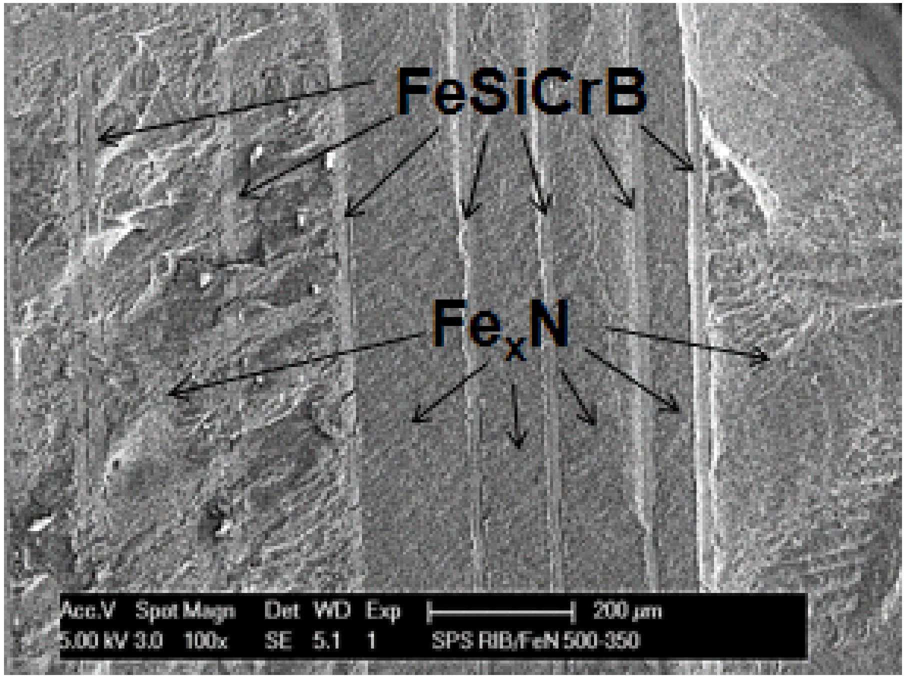


- Small variation in composition between grain boundary and center
- Grain center stoichiometry ≈ Fe_4N
- Grain boundary is ≈ 3 Atomic% richer in iron

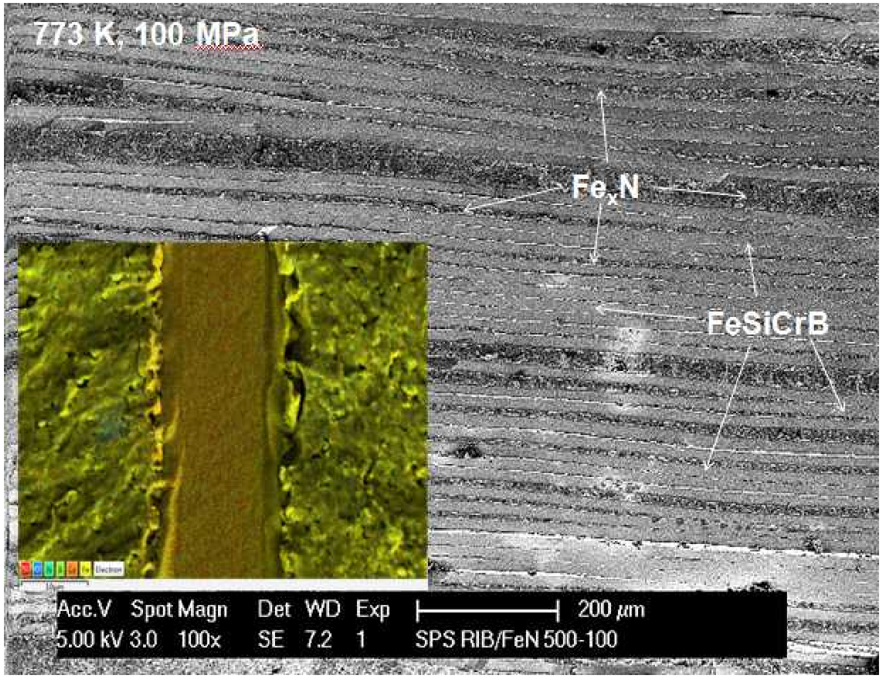
*SEM/EDS analysis completed by Dick Grant (SNL)

Location	Fe (Atomic %)	N (Atomic %)
Grain center	81.3	18.7
Grain boundary	84.2	15.8

SEMs of FeSiCrB and Fe_xN composites

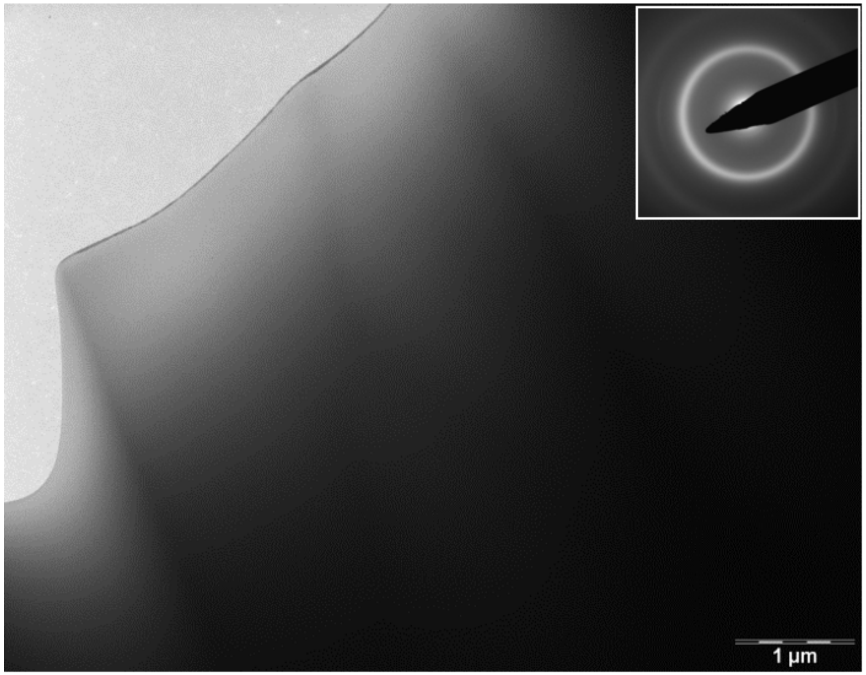


10 vol% FeSiCrB ribbon

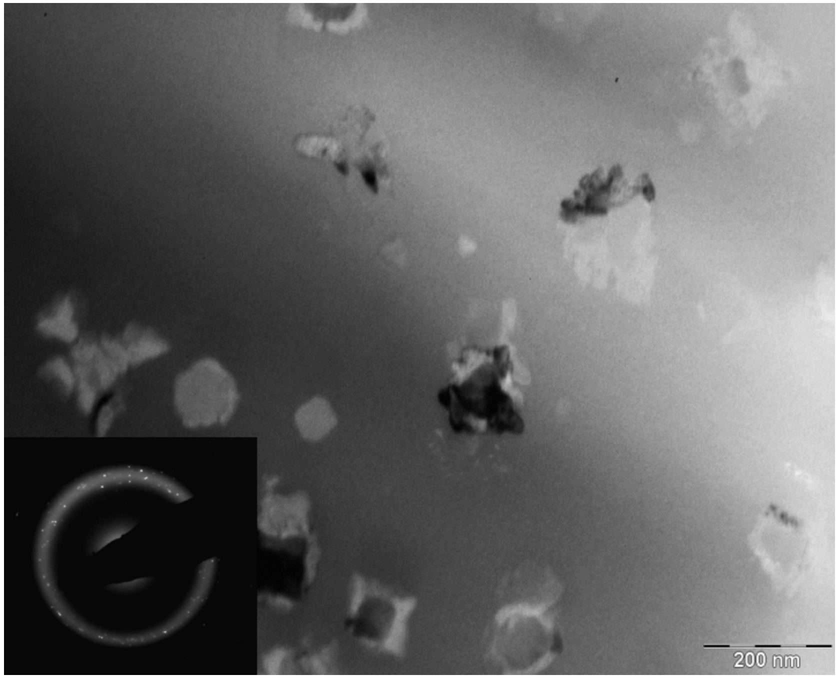


90 vol% FeSiCrB ribbon plus EDS mapping (inset)

TEMs of FeSiCrB before and after sintering

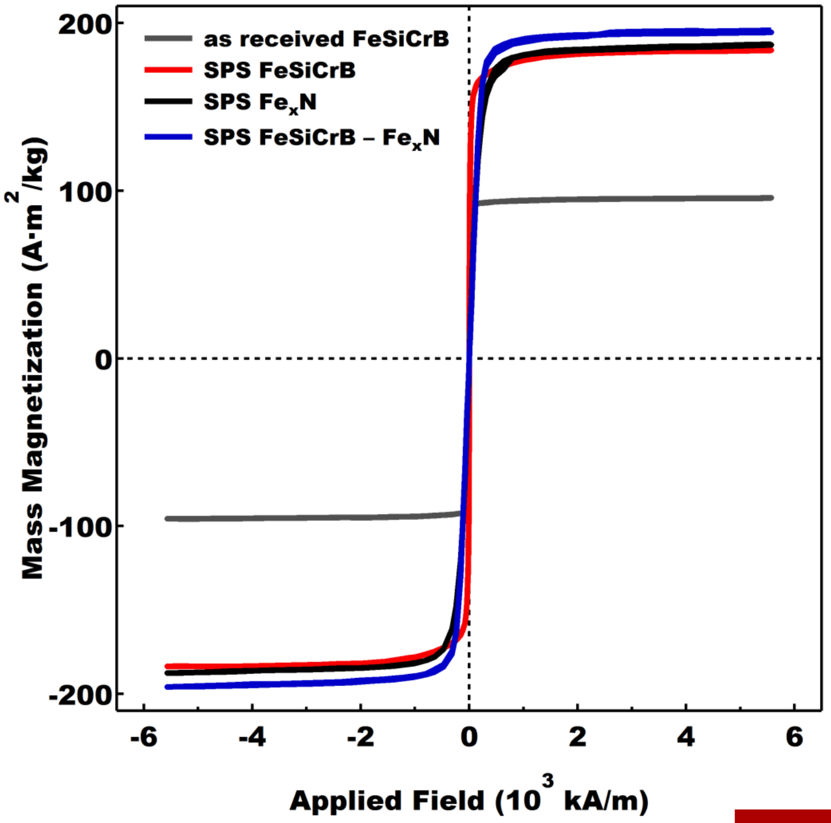


Before SPS



After SPS

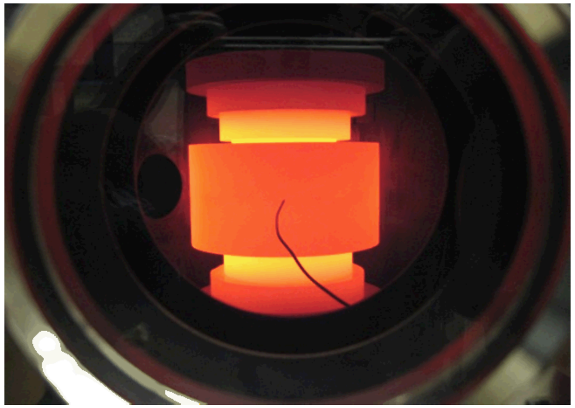
Composite DC Magnetic Response



Sample	M_{sat} ($A{\cdot}m^2/kg$)
As received FeSiCrB ribbon	96.0
SPSed FeSiCrB ribbon	185.3
SPSed Fe_xN	188.0
SPSed FeSiCrB–Fe_xN	196.5

γ'-Fe_4N Conclusions

- γ'-Fe_4N has the potential to serve as a new low cost, high performance transformer core material
  - M_{sat} > Si steel
  - Increased current and field (and therefore power) carrying capability
  - Resistivity 200X greater than nanocrystalline and amorphous alloys
  - Only requires low cost and abundant elements (Fe & N)
  - High temperature (T) operation complementing Sandia development of high T capacitors and WBG semiconductors
- The fabrication of bulk γ'-Fe_4N using SPS has been demonstrated
  - SPS can consolidate iron nitrides without material decomposition
  - Parts can be fabricated directly using net-shaping



2017 Spring MRS Symposium

Outline

- Synthesis of bulk γ'-Fe_4N soft magnetic materials
  - Why soft magnetic materials are important
  - History of soft magnetic materials & current state of the art
  - γ'-Fe_4N R&D
- **Highly magnetostrictive electrodeposited CoFe materials and devices**
  - **New electrodeposition chemistry**
  - "Dumb" vs. smart tags and sensors
  - First ever electroplated CoFe micro-resonators

Magnetostrictive materials: Current state of the art & limitations

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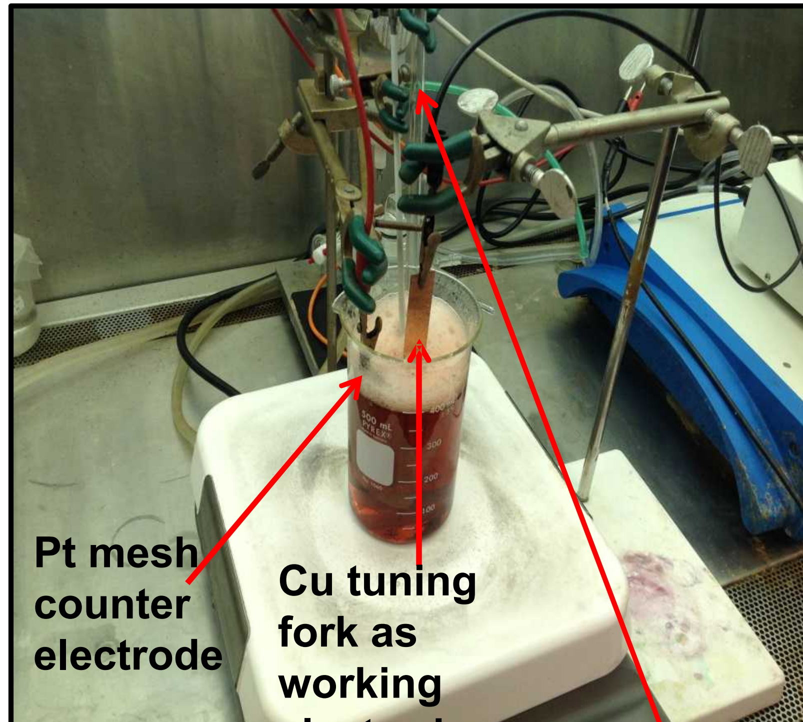
- Terfenol-D, terbium, iron, and dysprosium
  - Highest known magnetostriction at about 2000 microstrains (λ_s = 2000ppm)
  - Difficult manufacturing process and expensive due to rare earth materials
  - Poor piezomagnetic coefficient – high bias and AC field needed
- Galfenol, gallium alloyed with iron (λ_s~ 400ppm)
  - Difficult to electrodeposit and large intrinsic stress
  - Gallium salts for electrodeposition expensive
  - $Ga_2(SO_4)_3$ at $138 for 5g
- Cobalt and iron (CoFe) can be formed into a magnetostrictive alloy with common stoichiometries of 50% cobalt with 50% iron and 70 to 75% cobalt and 25 to 30% iron
  - Theoretical saturation magnetostriction of $\lambda_s \approx 1000$ ppm
  - $CoSO_4$ at $2.10 for 5g
- Metglas
  - Only commercially available material and therefore used in a lot of current device work
  - Three alloys: 2826MB, 2605SA1
  - $\lambda_s = 12 – 27$ ppm

New method to electrodeposit CoFe

Improvements over existing electrodeposition methods

- **New Fe cation source**
- **Oxygen scavengers**
- **Grain refiners, levelers**
- **Surfactant**
- **Pulsed plating**



Example of previous work:

Osaka, T.; Yokoshima, T.; Shiga, D.; Imai, K.; Takashima, K. *Electrochem Solid St* 2003, 6, (4), C53-C55.

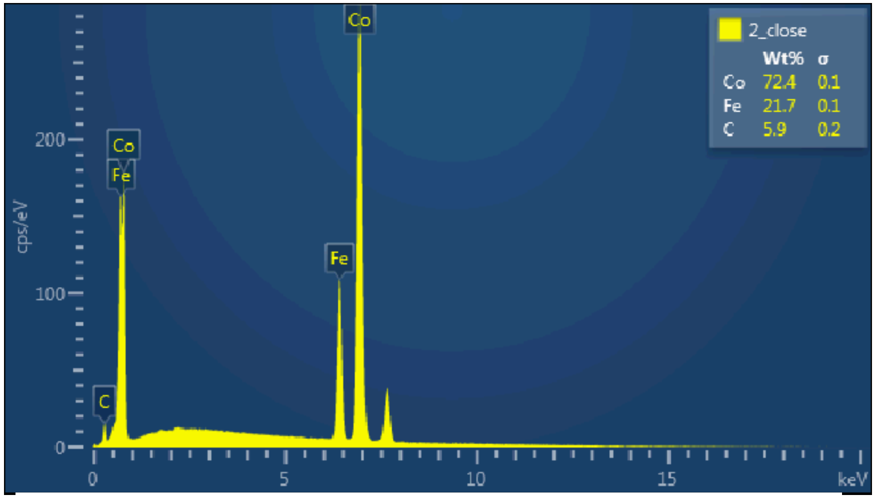
Our work:

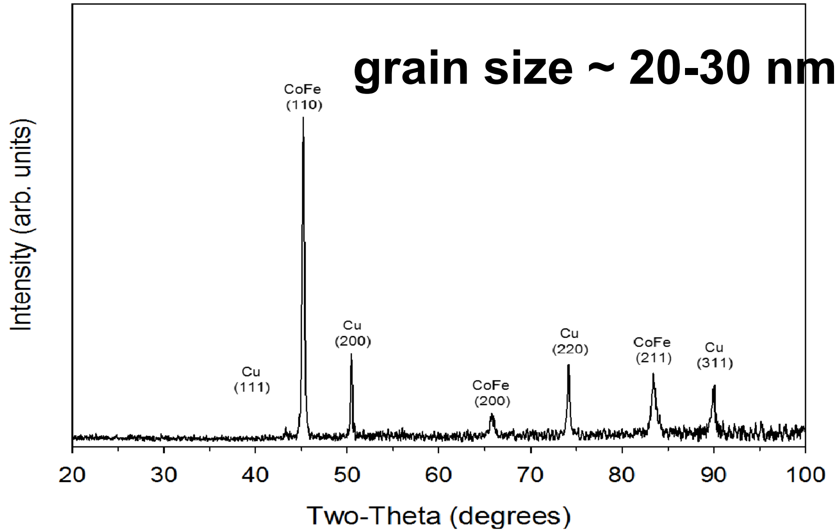
Jamin Pillars, Eric Langlois, Christian Arrington, Todd Monson, Andrew Hollowell, Mark Rodriguez, "Electrodeposition of a High Magnetostriction CoFe Film," to be submitted to Electrochimica Acta

Jamin Pillars, Eric Langlois, Christian Arrington, Todd Monson, "Electrodeposition Processes for Magnetostrictive Resonators," U.S. Patent Application #14876652, October, 2015.

High quality, low oxide (< 8 at%) CoFe films

- **Achieving $Co_{0.7}Fe_{0.3}$ ratio reported to have "giant magnetostriction" by Hunter et al.***
  - As-sputtered films λ_S = 84 ± 5 ppm
  - 800°C anneal and slow cool, λ_S = 156±7 ppm
  - 800°C anneal and quench, λ_S = 260±10 ppm
  - Anneal produced fcc (111) β-Co peak
  - (fcc+ bcc)/bcc phase boundary
- **Note on limitations of sputtering**
  - Line-of-sight deposition
  - Limited thickness **(<2 micron)** and high intrinsic stress
  - High temperature anneal would degrade CMOS devices
  - High cost and wasteful
  - Lack of anisotropic etch



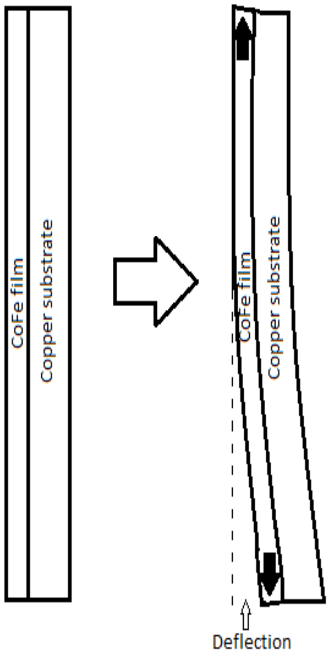


* Hunter, D., et al., *Giant magnetostriction in annealed Co(1-x)Fe(x) thin-films.* Nat Commun, 2011. **2**: p. 518.

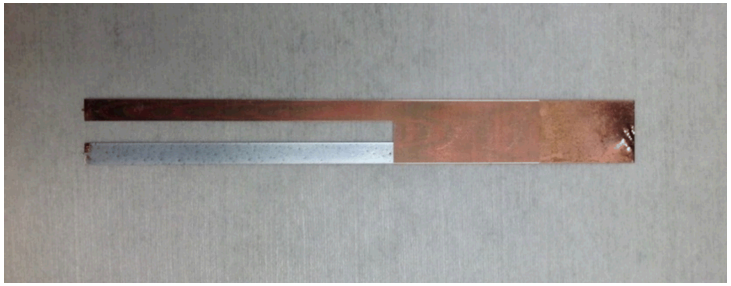
Novel rapid magnetostriction testing

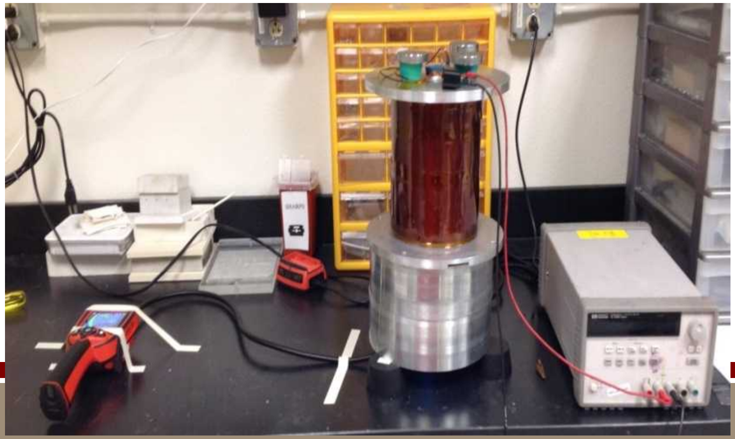
- **Klokholm (1976) observed deflection of bimorph when magnetized**
- **The expression du Tremolet de Lacheisserie and Peuzin is:**

$$\lambda_{eff}(D_{sat}) = \frac{2(D_{\parallel} - D_{\perp})E_s {t_s}^2(1+\upsilon_f)}{9E_f L^2 t_f(1+\upsilon_s)}$$



- **Solenoid for *in situ* displacement measurements**
  - Cu tuning fork substrate
- **Calculated magnetostriction for CoFe compared with a Metglas standard**



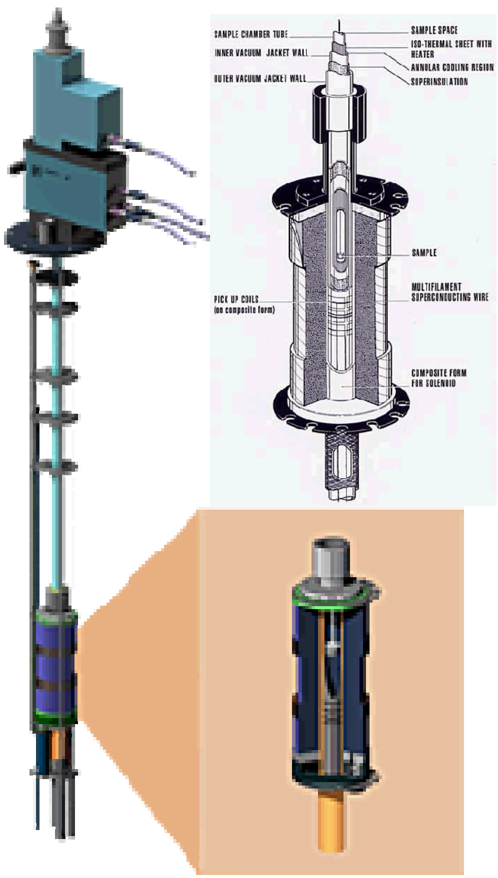


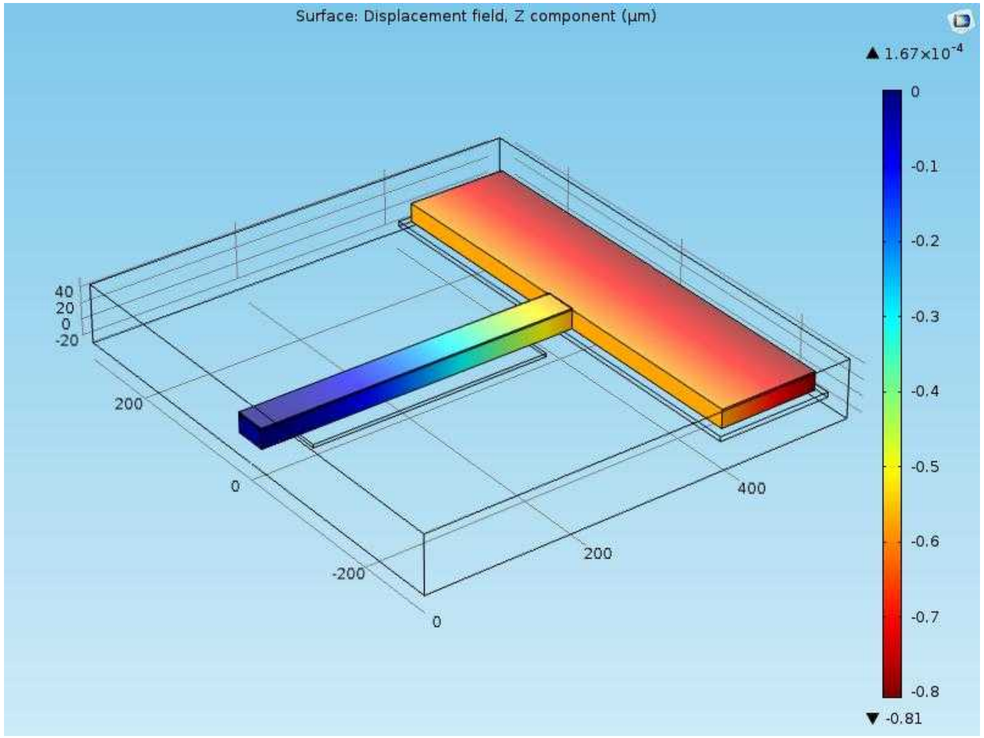
Calibrated solenoid used for testing

MEMS cantilever capacitors for λ_s measurement, coming soon to our lab

Sandia National Laboratories

Quantum Design Magnetic Property Measurement System (MPMS) probe assembly

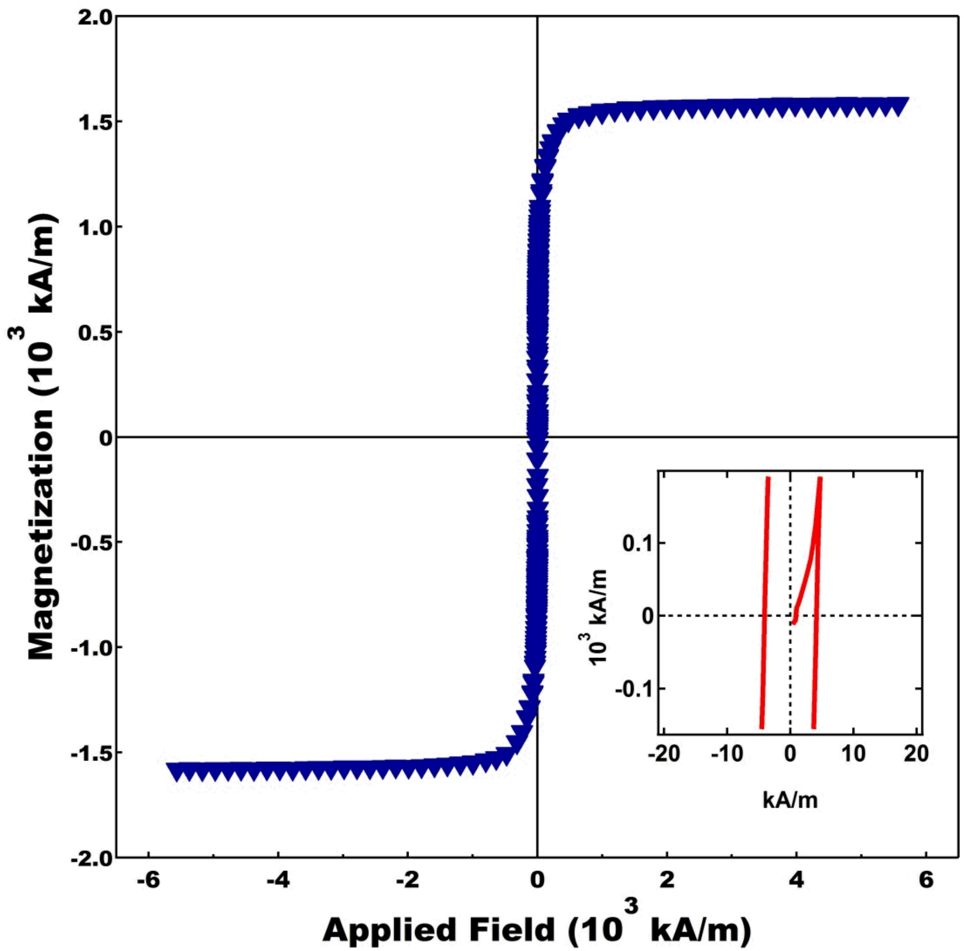




- **Will enable more precise λ_s measurements**
- **Two orthogonal capacitors will allow for measurement of both $D_{\parallel}$ and $D_{\perp}$**

Electrodeposited CoFe magnetic properties

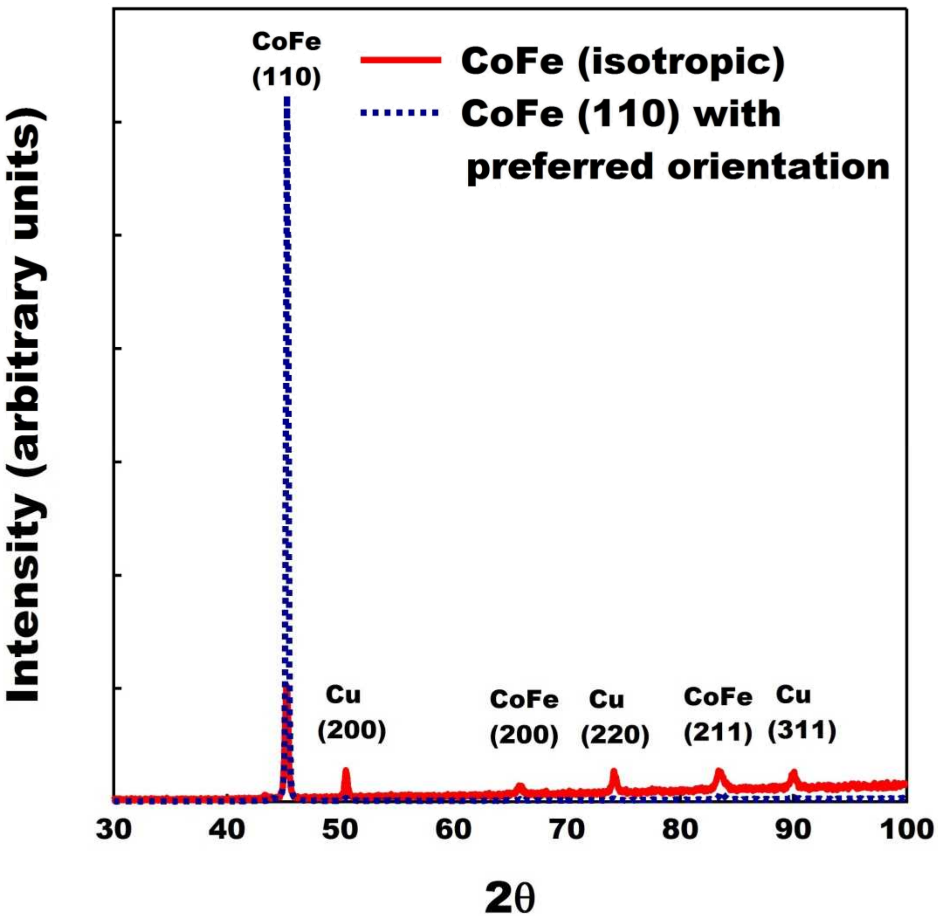
Sandia National Laboratories



Sample	M_s (A/m)	H_c (A/m)	χ
CoFe	1.58×10^6	4.13×10^3	165.6
Metglas 2605SA1	1.07×10^6	1.16×10^3	140.8

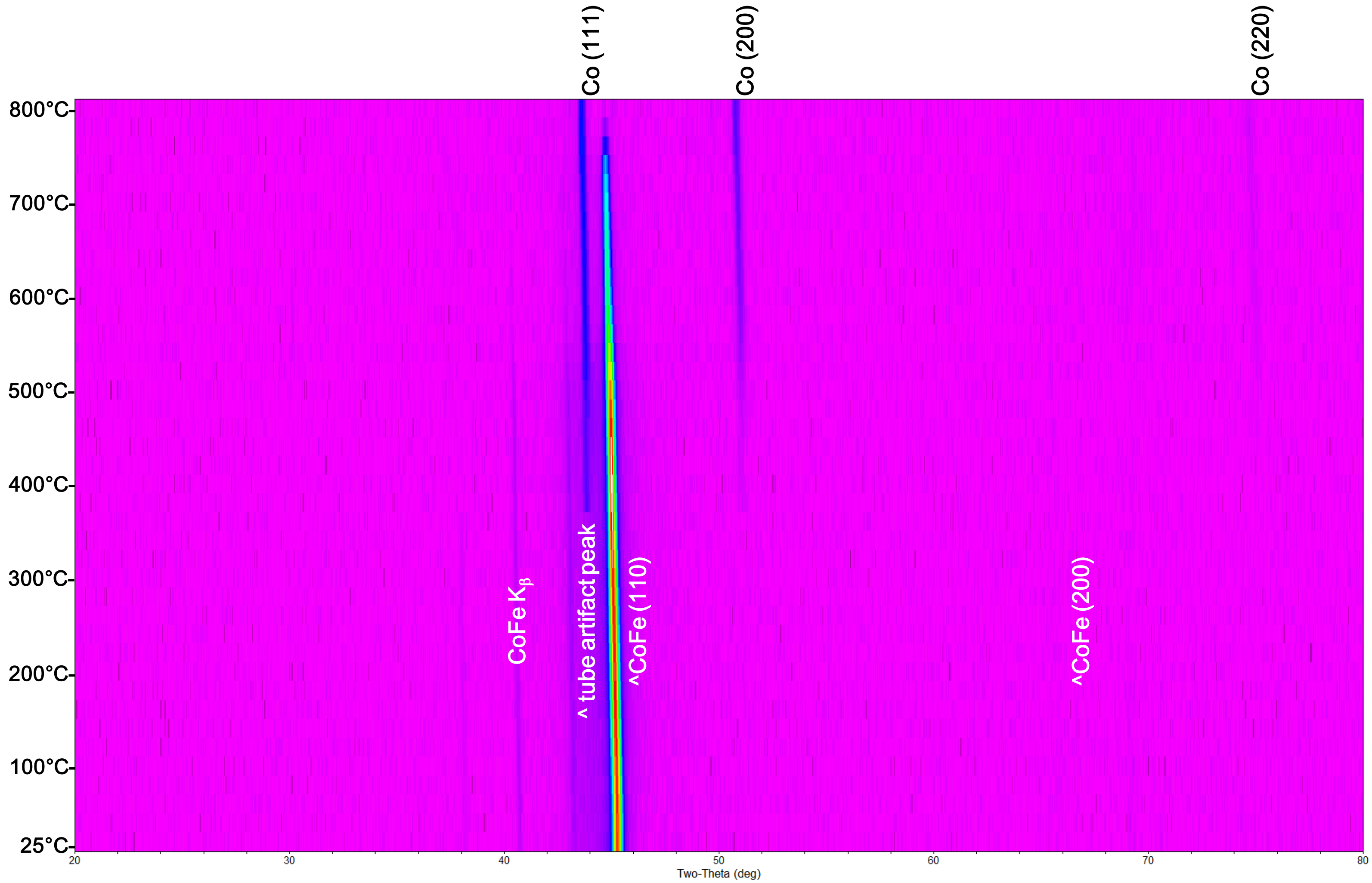
Sample	λ_s (ppm)
CoFe (isotropic)	172 ± 25
CoFe (110) preferred orientation	229 ± 32
Hunter et al. CoFe as-sputtered	84 ± 5
Hunter et al. CoFe 800°C anneal and quench	260 ± 10
Metglas 2605SA1	26 ± 4

(110) preferred orientation



- Sample 1: isotropic CoFe
- Sample 2: CoFe with strong (110) out-of-plane preferred orientation
- Results in enhanced magnetostriction
  - As reported on previous slide

HT XRD in gHe (ramped at 20° C/min)



- Separation of FCC Co > 350 °C
- We thank James Griego for collecting this data!

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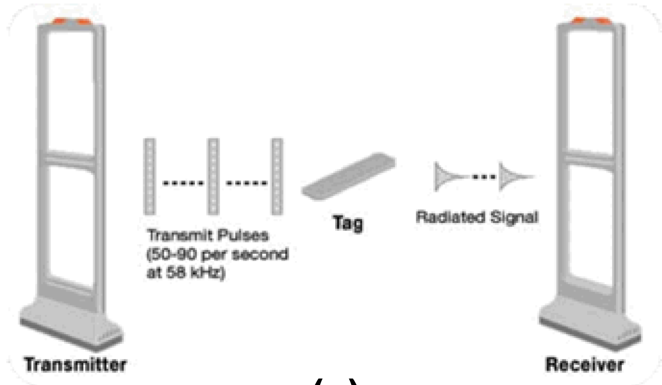
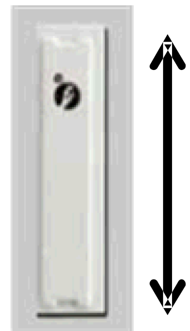
Magnetoelastic sensor overview

“Dumb



(a)

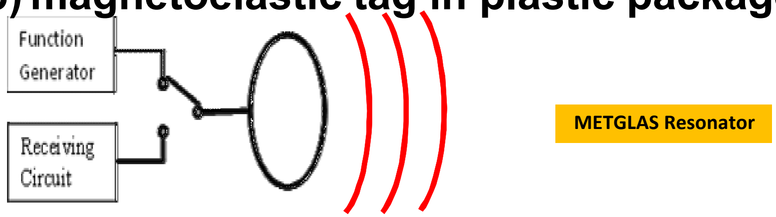


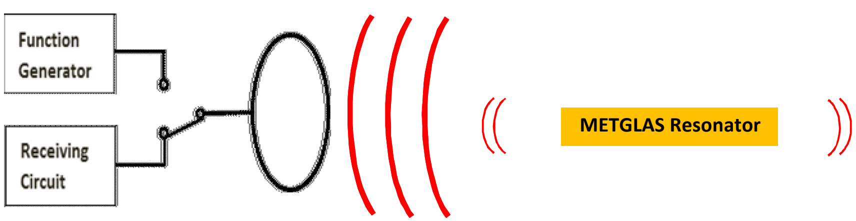
45 mm

(b)

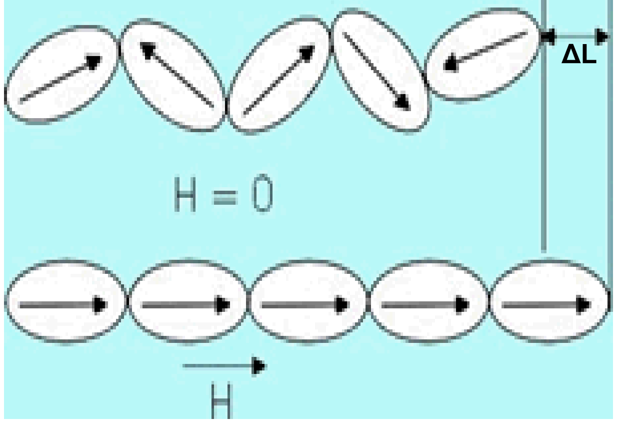
amorphous magnetic material such as METGLAS™ ($Ni_{40\text{-}50}Fe_{40\text{-}50}Mo_{5\text{-}10}B_{1\text{-}5}$)

a) magnetic dipole antenna interrogation zone
b) magnetoelastic tag in plastic package



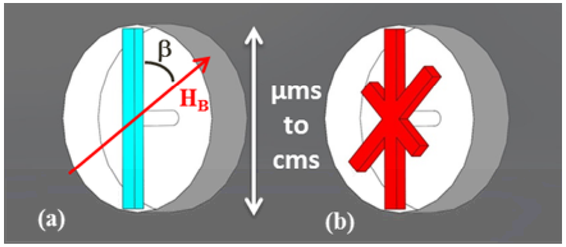


Magnetostriction, $\lambda=\Delta L/L$



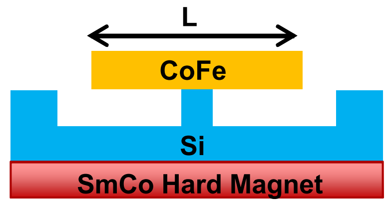
Magnetic Smart Tag (MaST)

"Smart



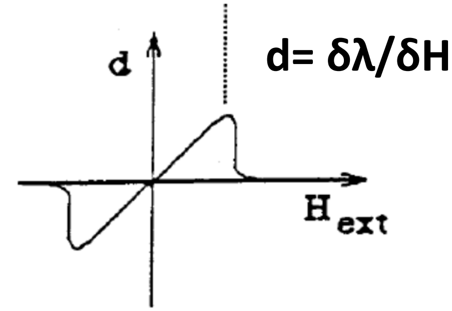
a) Single frequency resonator
b) Multi-Frequency resonator (3)

$$f_r = \frac{1}{2L}\left[\sqrt{\frac{\rho}{E_0} + \frac{9\lambda^2{}_s\rho\left((|H_B|\cos(\beta))\right)^2}{J_s H_A{}^3}}\right]^{-1}$$

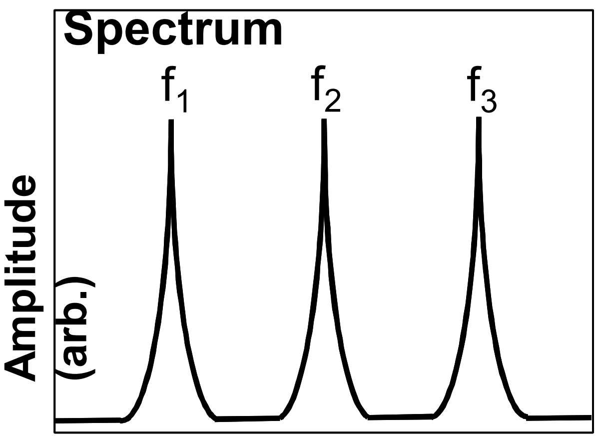


MagSens resonator cross section

Optimal ME coupling point

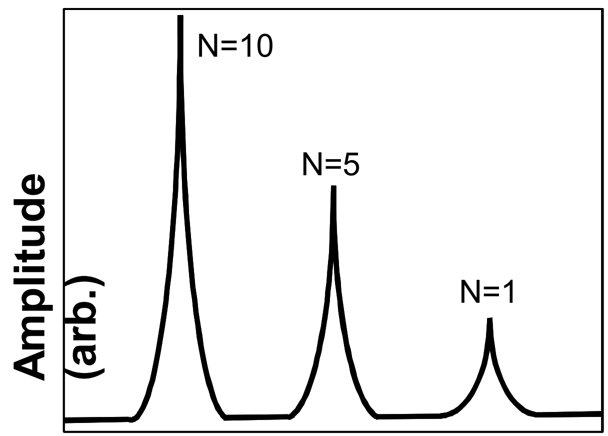


Multi-Frequency Spectrum



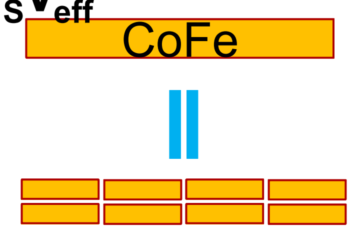
Peak frequency constitutes primary tag identity!

Multi-Frequency/Multi-Amplitude Spectrum



Relative amplitude constitutes secondary tag identity!

Signal strength is related to effective volume:
$\Delta m = \chi H_{ex} V_{eff} < M_S V_{eff}$



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First ever electroplated CoFe micro-resonators

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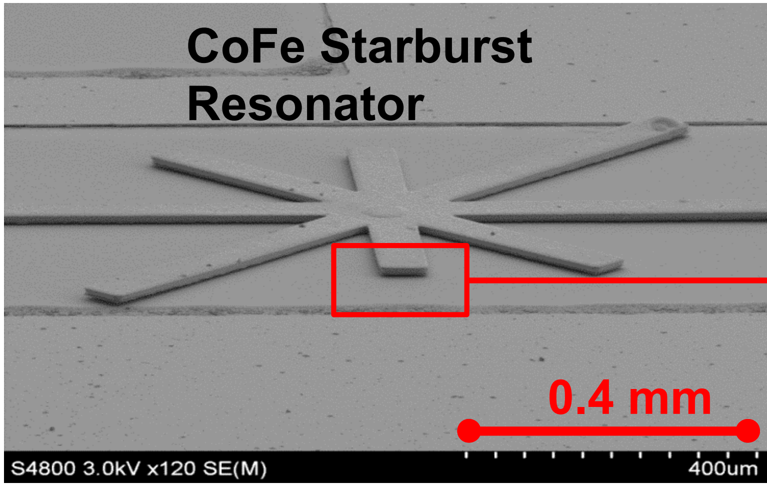
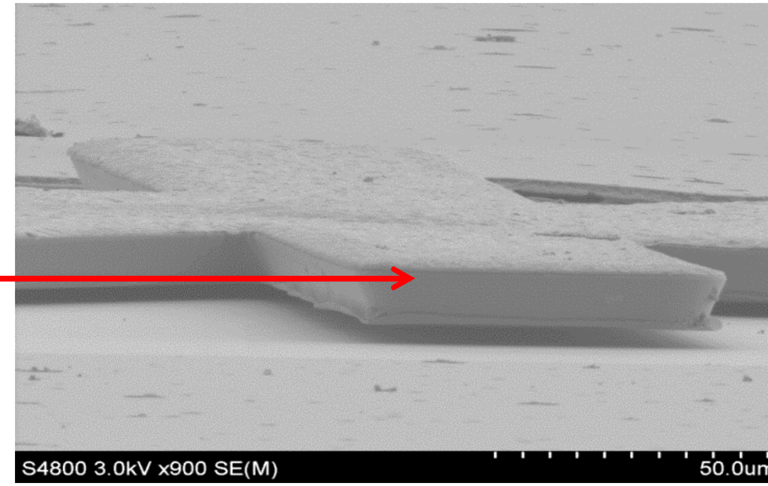
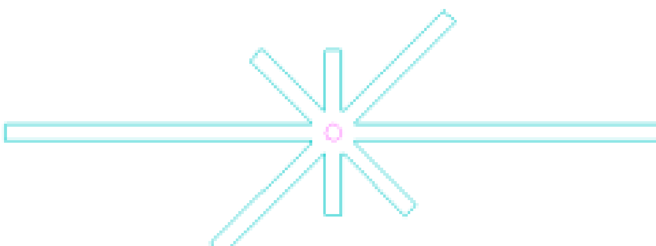
2.4 mm long freestanding resonator

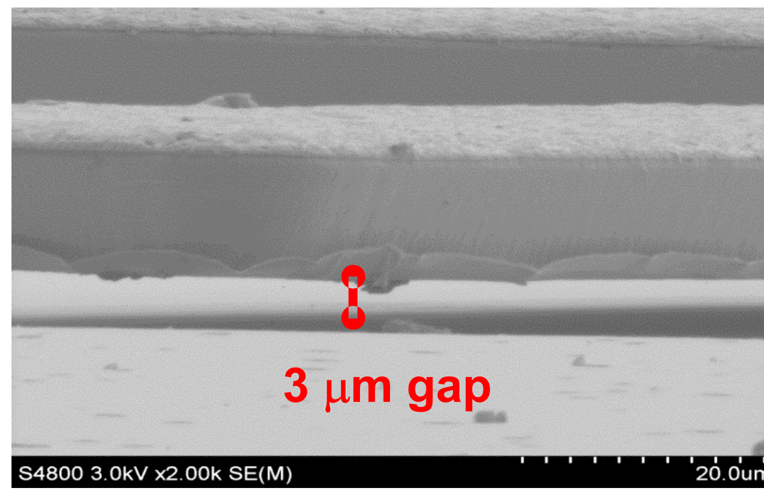




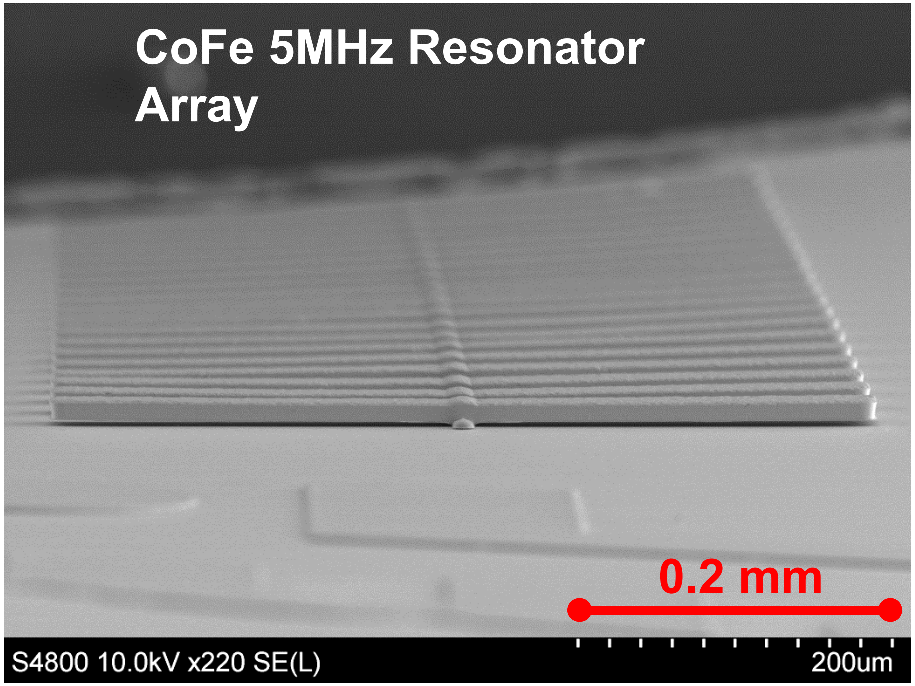
Multi-Frequency Micro Resonator, "Starburst Pattern"

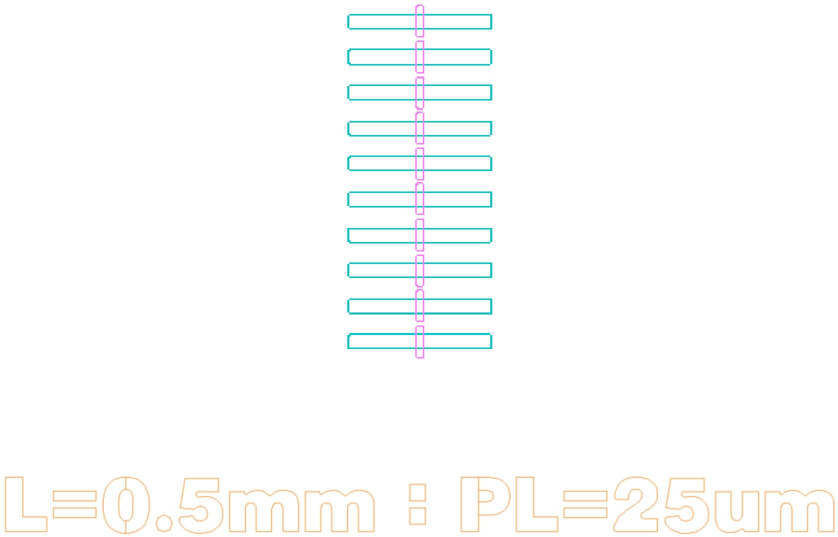


L=0.6mm-2.4mm



First ever electroplated CoFe micro-resonators





- Arrays of resonators increase signal amplitude
- Via proper design resonators can measure a host of environmental parameters
- Temperature, pressure, current, gases, stress/strain, angular position, and many more!

Electrodeposited CoFe Conclusions

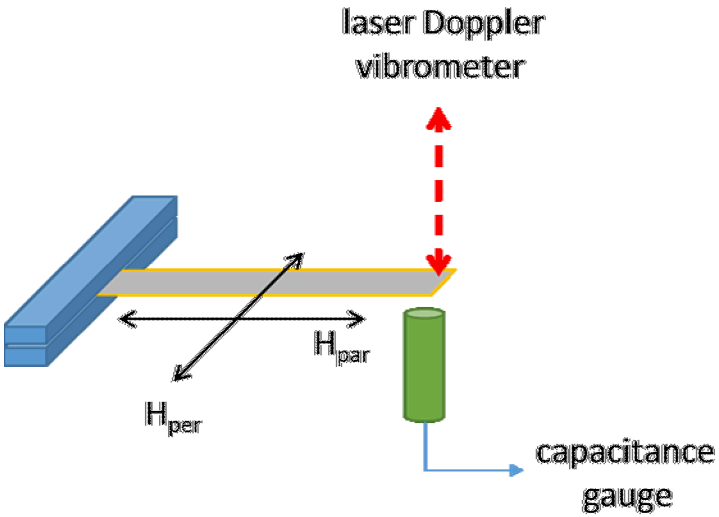
- New method to electrodeposit thick (10+ μm), low oxide (< 8 at%) low stress, highly magnetostrictive CoFe
  - $\lambda_S = 229 \pm 32$ ppm for CoFe with (110) out-of-plane preferred orientation
  - Significantly exceeds λ_S of Metglas 2605SA1 and is comparable to CoFe sputter deposited by Hunter et al.
  - U.S. patent application (#14876652) filed
  - Electrodeposited CoFe bypasses issues associated with sputtering and avoids high cost and manufacturing challenges of Terfenol-D and Galfenol
- First ever free standing CoFe micro-resonators demonstrated
- MEMS CoFe resonators can be used as smart tags and to measure a host of environmental parameters
  - Temperature, pressure, current, gases, stress/strain, angular position, and many more!
- Demonstration of these devices coming soon!

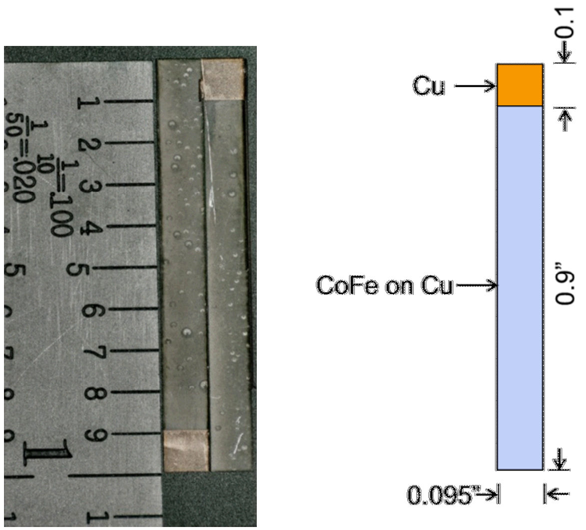
Acknowledgements

- 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
- CoFe material and device R&D supported by Sandia's Laboratory Directed Research and Development (LDRD) program
- We thank Robert Delaney (El Dorado High School) for his assistance with magnetic data fitting and analysis

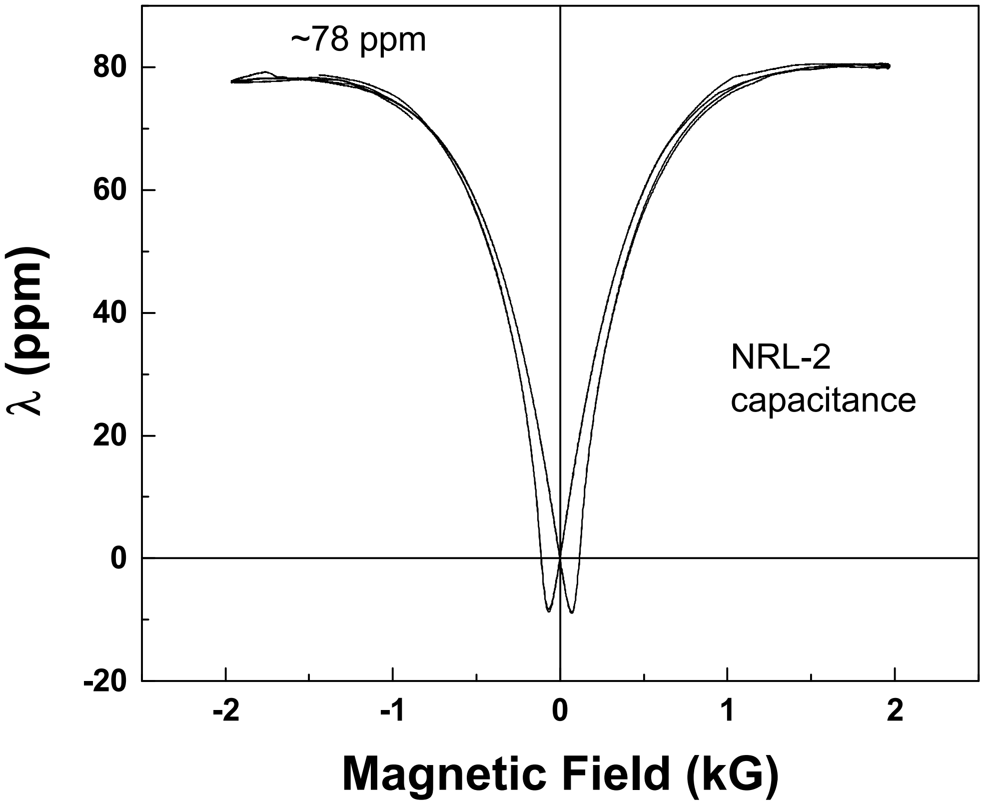
Extra Slides

NRL measurements of CoFe Cantilevers

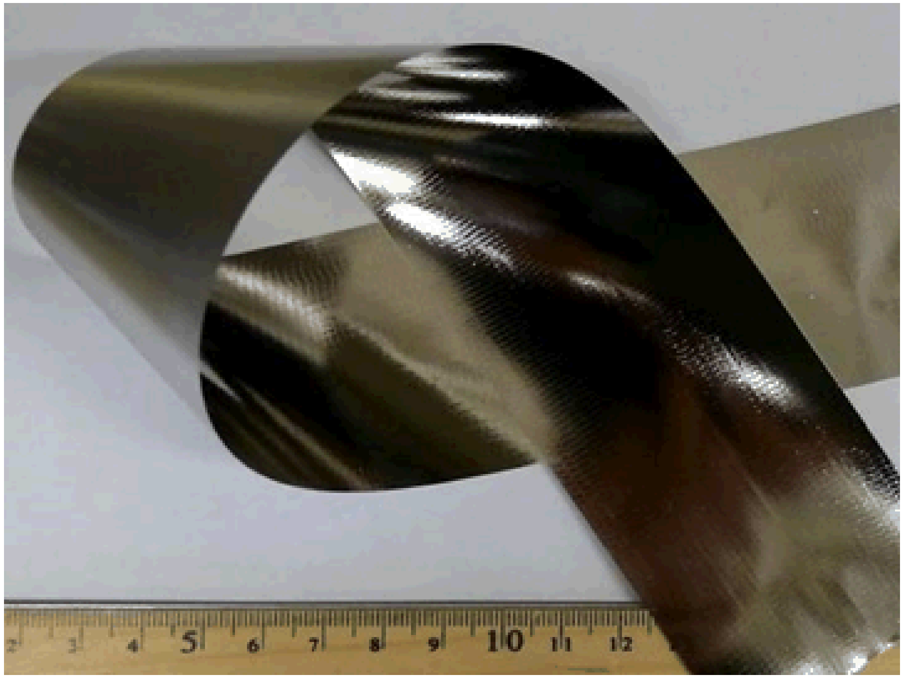




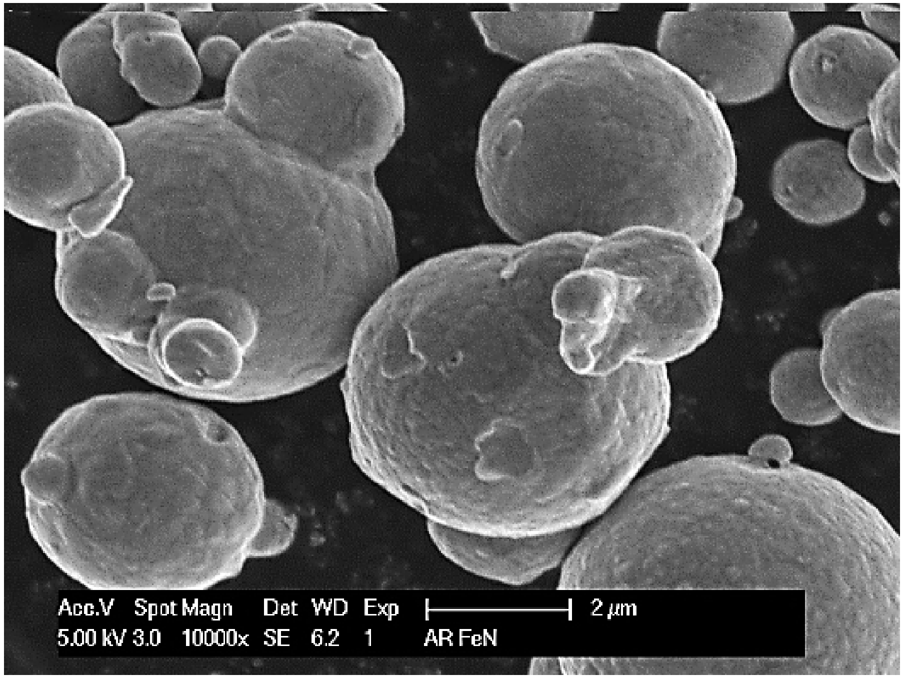
Smaller CoFe coated Cu tine machined using ns-laser.



FeSiCrB ribbons and Fe_xN powder



FeSiCrB ribbon



Fe_xN powder

Iron nitrides crystalline structure

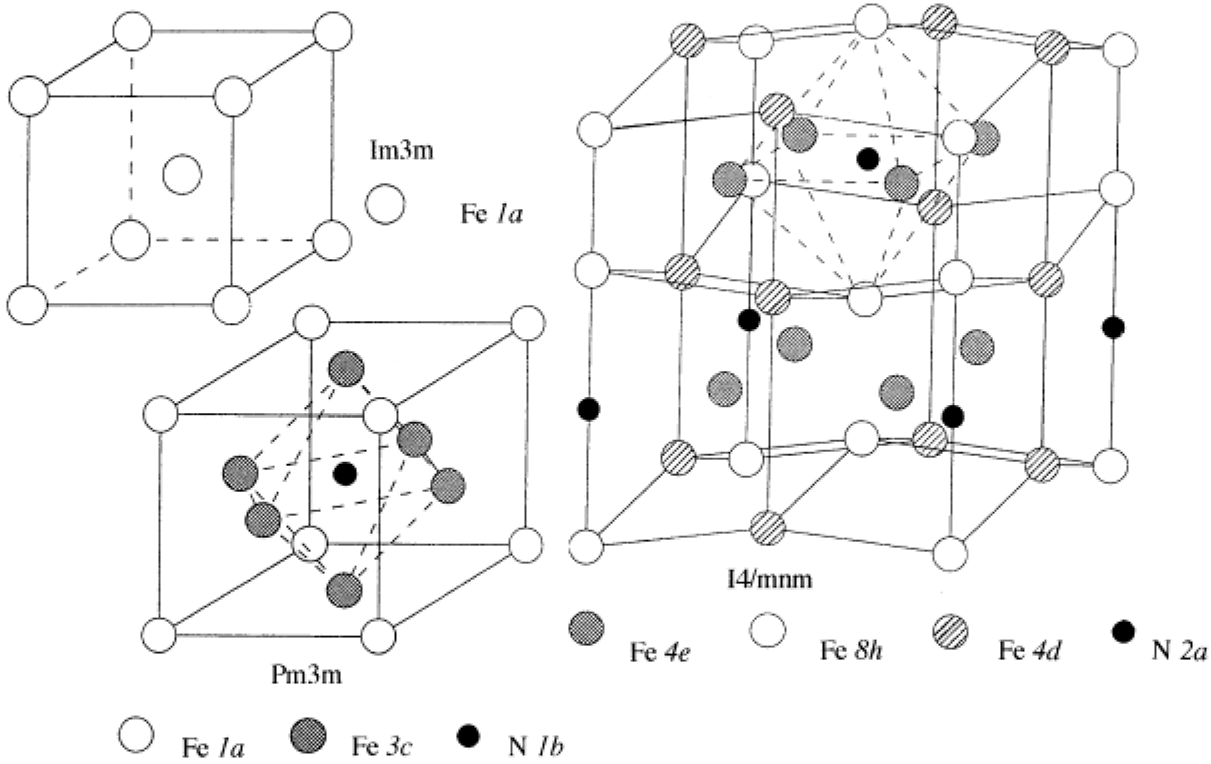


Fig. 1. Crystal structures of αFe, γ′Fe_4N and α″$Fe_{16}N_2$, drawn to scale.

J.M.D. Coey, P.A.I. Smith, Magnetic Nitrides, J. Magn. Mag. Mat., 200, 405 (1999).

Soft Magnetic Material Permeability

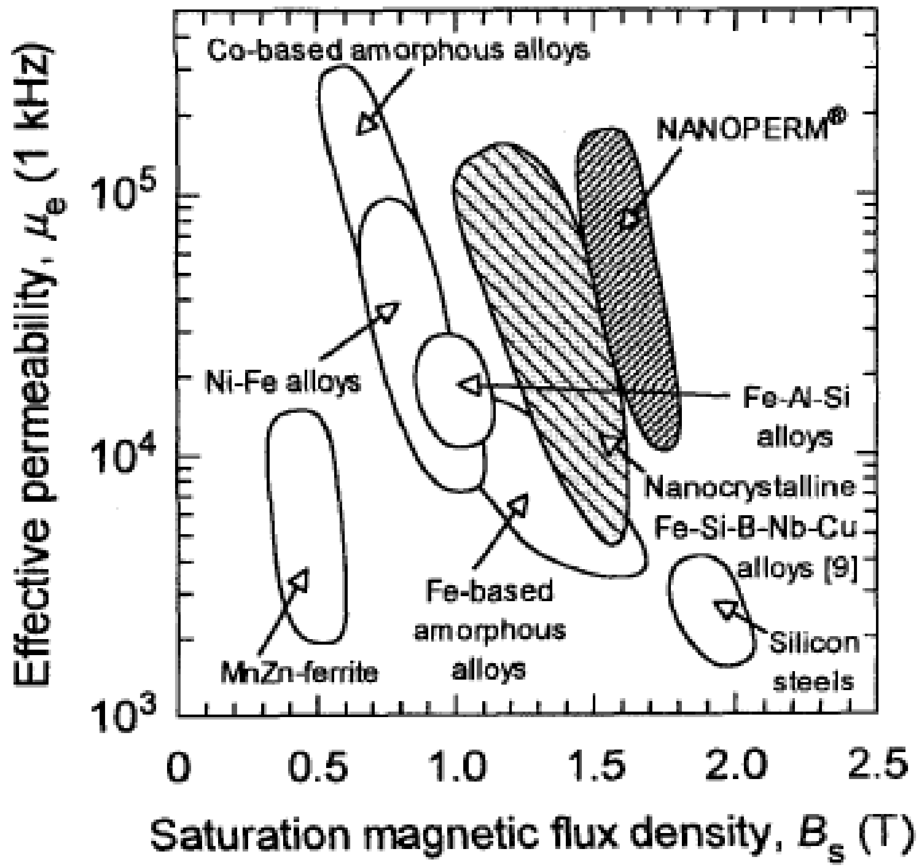


Fig. 2. Relation between B_s and μ_e at 1 kHz for NANOPERM®, the nanocrystalline Fe-Si-B-Nb-Cu alloys [9] and conventional soft magnetic materials

A. Makino, et. al., Applications of Nanocrystalline Soft Magnetic Fe-M-B (M = Zr, Nb) Alloys “NANOPERM”, IEEE Trans. Magn., 33, 3793 (1997).

Magnetic Moment Variation with Nitrogen Concentration

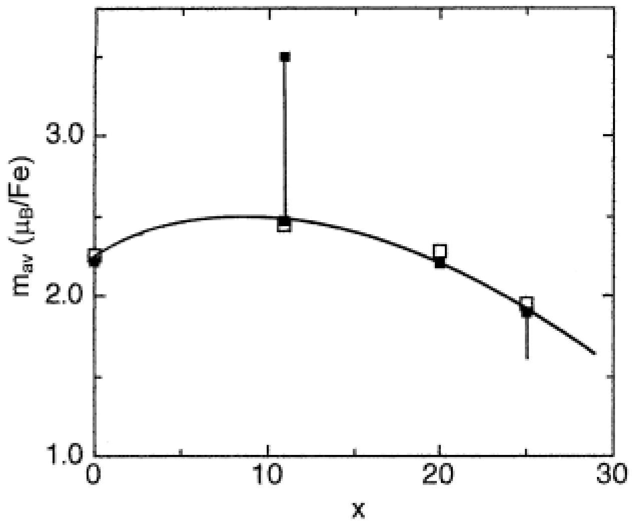


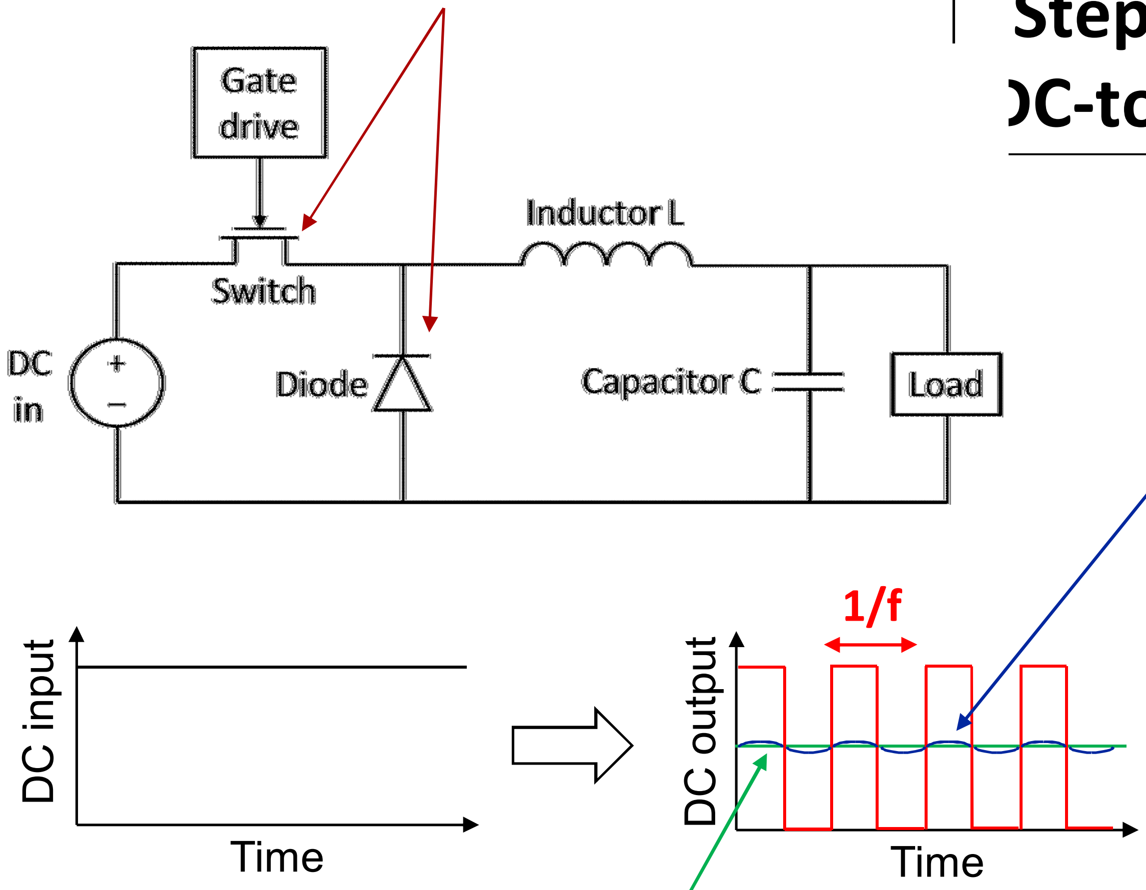
Fig. 3. Calculated (open symbol) and measured (solid symbol) average iron moments in Fe–N compounds.

J.M.D. Coey, P.A.I. Smith, Magnetic Nitrides, J. Magn. Mag. Mat., 200, 405 (1999).

Higher Switching Frequency Enables Reduction in Passive Element Volume and Weight

Step-Down (Buck) DC-to-DC Converter

Power semiconductor devices

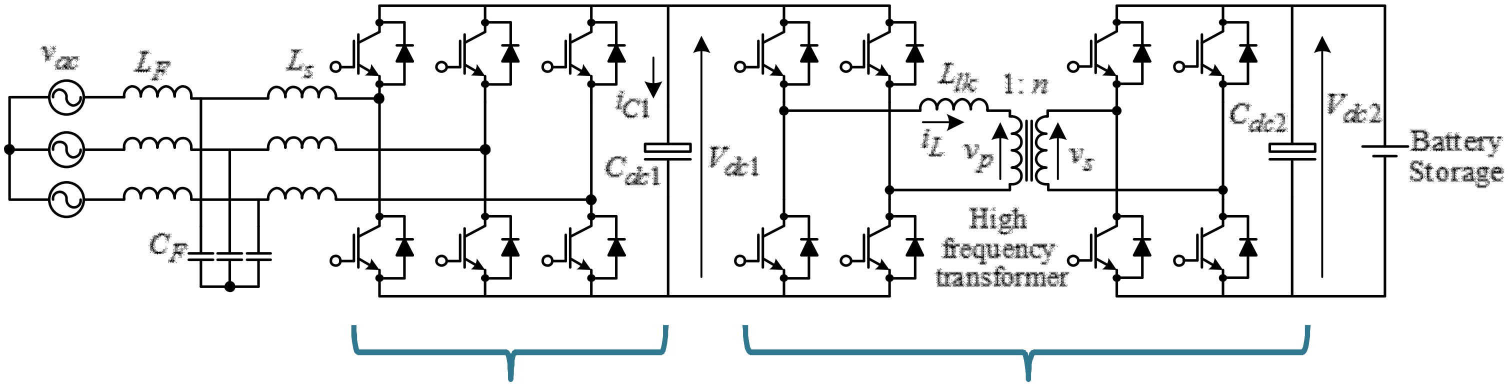


$V_{out} = DV_{in}$

$$\frac{V_{ripple}}{V_{out}} = \frac{1 - D}{8LCf^2}$$

Increasing f allows one to reduce L and C while keeping the ripple constant

High Frequency Link Power Conversion System Configuration



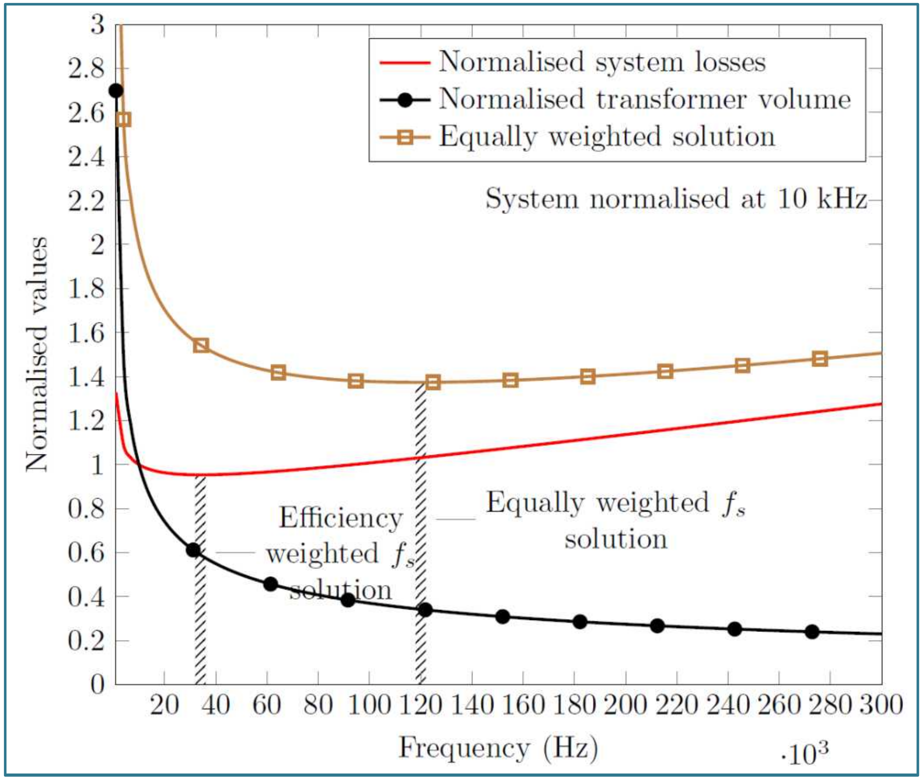
- Optimal switching frequency based on the losses at this stage

Volume occupied by heat sinks and non-magnetic components were not considered !!

- Optimal switching frequency based on:
- Switching and conduction losses in the switch
- Volume of the transformer
- Losses in the transformer
  - Copper losses in transformer windings
  - Core losses of the transformer

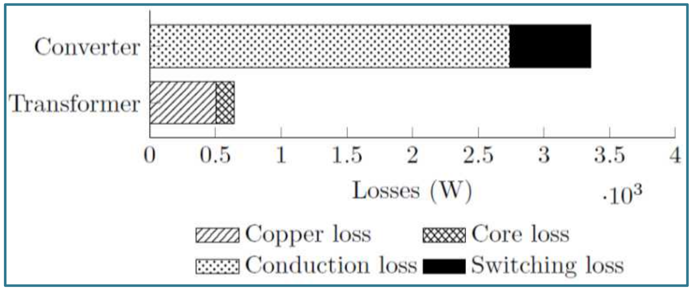
S. Kulasekaran, R. Ayyanar, Analysis, Design, and Experimental Results of the Semidual-Active-Bridge Converter, IEEE Transactions on Power Electronics, 29 (2014).

Optimum Frequency with SiC MOSFETs



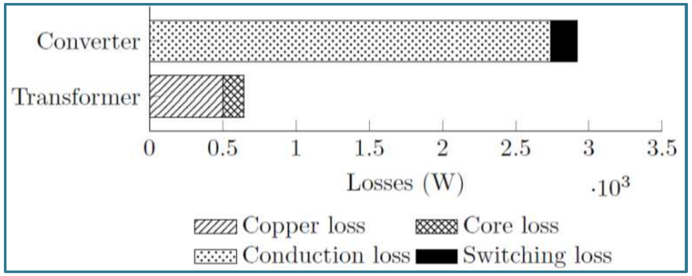
Equally weighted solution:

The converter has optimal solution at a frequency of **120 kHz** with equal weights on volume and losses



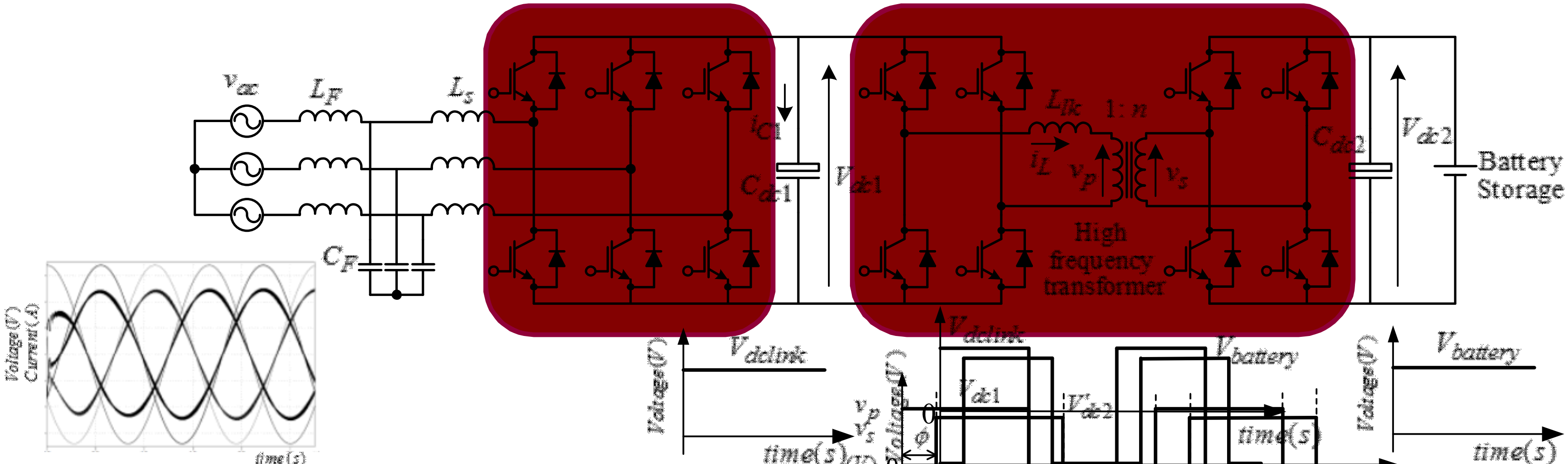
Efficiency driven solution:

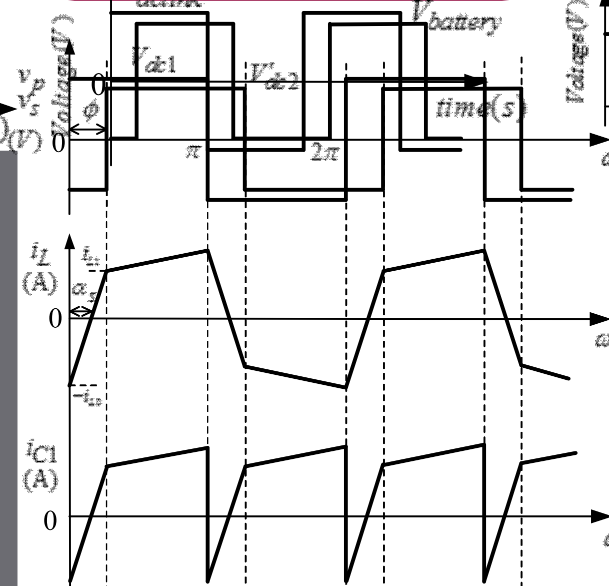
The optimal solution for minimum loss condition is **35 kHz**



S. Kulasekaran, R. Ayyanar, Analysis, Design, and Experimental Results of the Semidual-Active-Bridge Converter, IEEE Transactions on Power Electronics, 29 (2014).

System Configuration





$$P = \frac{V_{dc1} V_{dc2}}{2\pi fL} \phi \left(1 - \frac{\phi}{\pi} \right)$$

Parameters	Specifications
Power Level	250 kW
Maximum RMS current	370 A
Maximum voltage	1100 V ± 10 %
Transformer core material	Ferrite
Transformer turns ratio	1:1
Transformer leakage inductance	18 µF
Snubber capacitance	20 nF
DC Capacitor (Input/ Output)	4.4 mF

Loss and Volume Estimation

Transformer Volume Estimation:

Power handling capacity of the cores are identified by Area product, A_P (cm^4)

$$A_p = \frac{S}{k_w J_{max} \Delta B_{max} f}$$

A_c = Area of the core

A_w = Window area

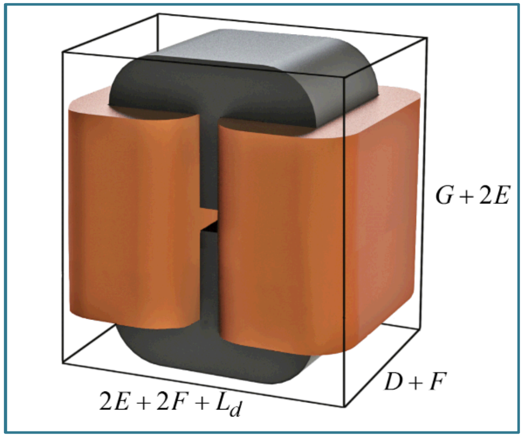
S = Total volt-amp rating of the transformer (VA)

k_w = window utilization factor

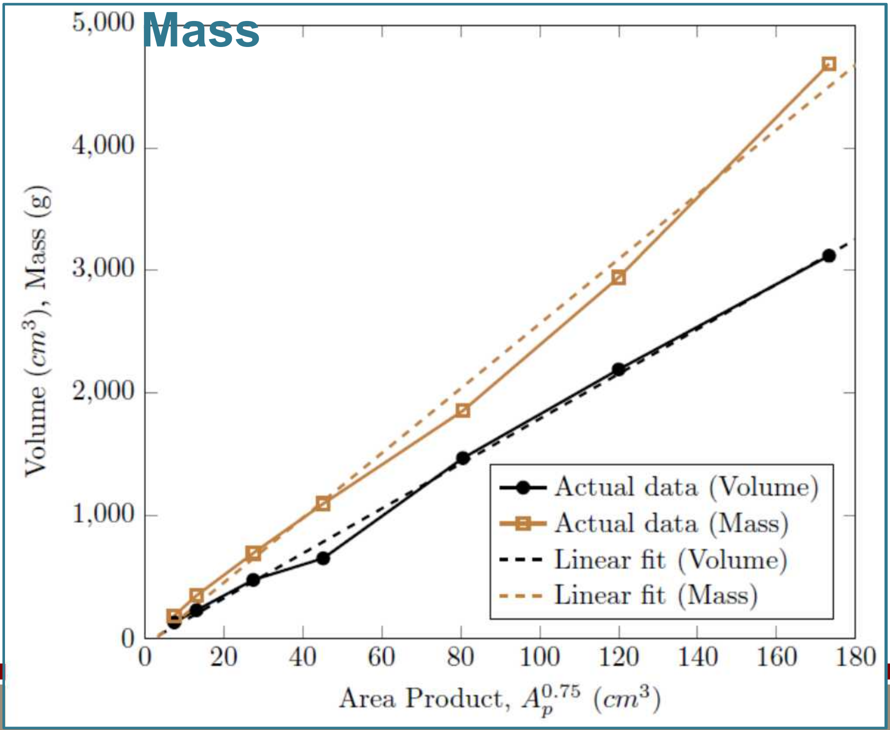
J_{max} = Current density (A/cm²)

ΔB_{max} = flux density (T)

f = switching frequency (Hz)



Area Product vs Volume, Mass



$$\text{Volume} = K_v A_p^{0.75}; K_v = 18.302$$

$$\text{Mass} = K_p A_p^{0.75}; K_p = 26.41$$

$$\text{Volume} = \frac{8.328 \times 10^6}{\Delta B^{0.75} f^{0.75}} cm^3$$

Transformer Losses

Transformer copper loss:

$$P_{cu} = 2K_r \frac{N_{pri}\rho l}{A_{cond}} I_{rms}^2$$

$$N_{pri} = \frac{V_{pri}}{\Delta B_{max} A_c f}$$

$$P_{cu} = \frac{288475.68}{\Delta B^{0.75} f^{0.75}} W$$

Mean turns length :

$$l = 2\,(E + D) + \pi\, F/2$$

where,

$$N_{pri} = \frac{1703.1}{\Delta B^{0.5} f^{0.5}}; R_{dc} = \frac{1.0536}{\Delta B^{0.75} f^{0.75}}$$

Transformer Iron loss:

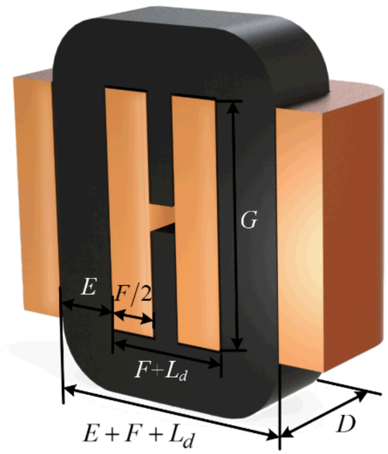
$$P_{fe} = \left(k f_{eq}^{\alpha-1} \hat{B}^{\beta}\right) f$$

$$f_{eq} = \frac{2}{\Delta B^2 \pi^2} \int_0^T \left(\frac{dB}{dt}\right)^2 dt$$

← **Modified Steinmetz equation**

The total iron losses in a 3C93 ferrite core is

$$P_{fe} = 4.77 f^{0.6} \Delta B^{2.45}$$



Optimal flux density

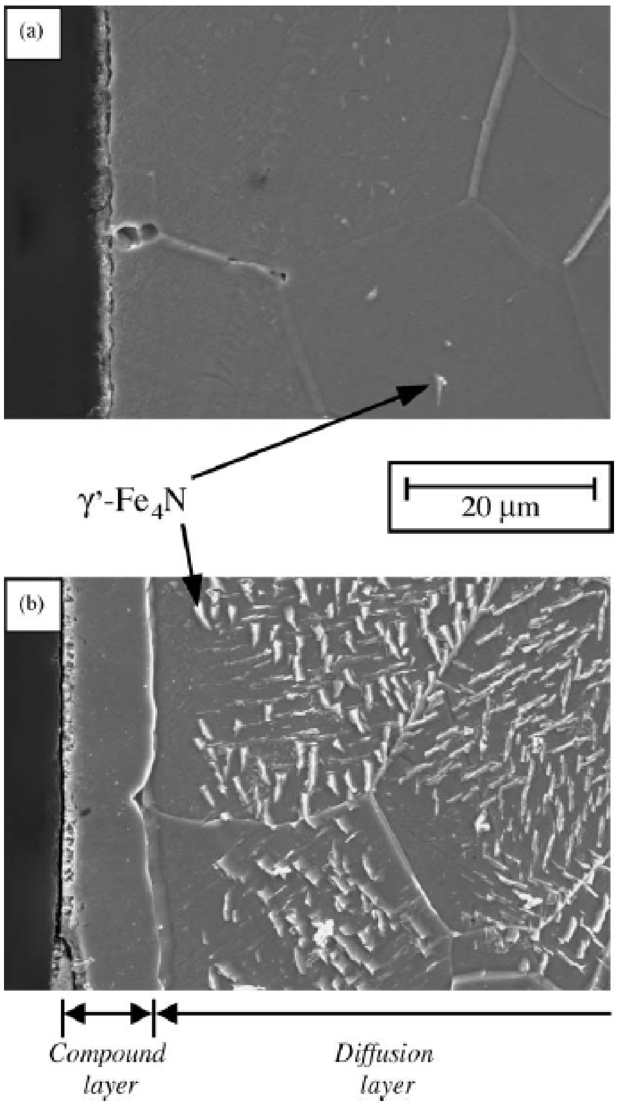
$$P_T = P_{cu} + P_{fe}$$

$$= \frac{288475.68}{\Delta B^{0.75} f^{0.75}} + 4.77 f^{0.6} \Delta B^{2.45}$$

$$\frac{dP_T}{dB}(f = const) = 0$$

$$B_{opt} = \frac{21.557}{f^{0.425}}$$

In House Synthesis of Raw Materials: Electrochemical Nitriding of Iron



- Growth of γ'-Fe_4N demonstrated by Japanese electrochemists
- Formed γ'-Fe_4N at the surface of Fe(0) electrode using Li_3N as nitride source
- Demonstrates electrochemical synthesis of iron nitride possible
- Our goal is to demonstrate autonucleation of iron nitride with flowing N_2

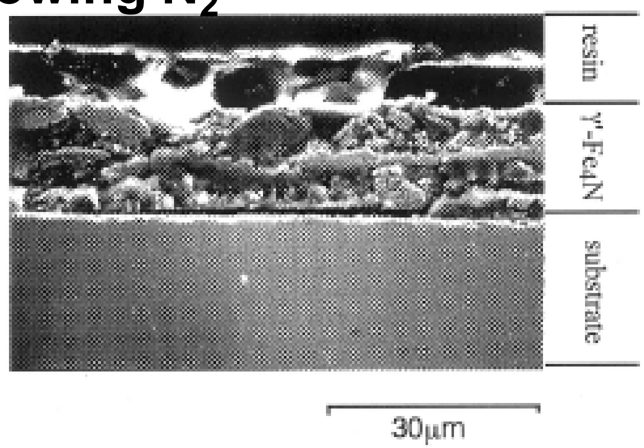
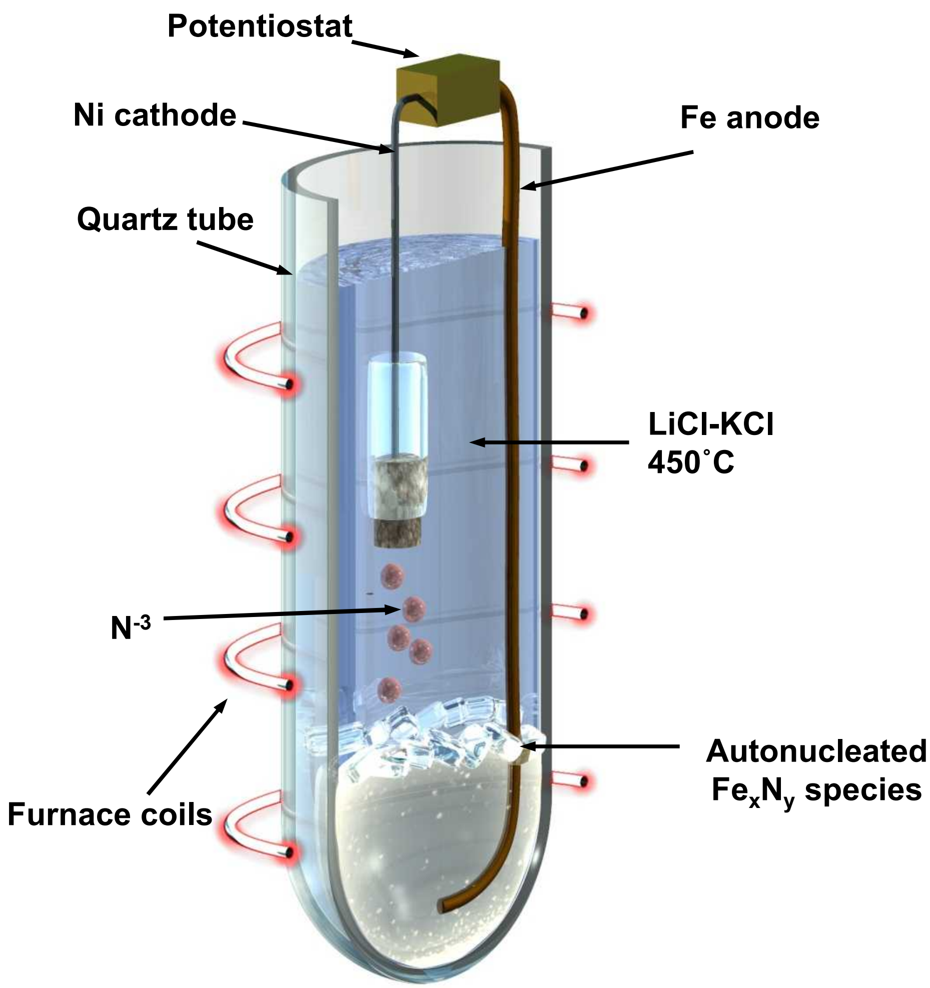


Fig. 10. Cross-sectional SEM image of iron electrode after potential pulse electrolysis for 1 h.

H. Tsujimura, T. Goto, Y. Ito, Journal of Alloys and Compounds, 376, 246 (2004)

T. Goto, R. Obata, Y. Ito, Electrochimica Acta, 45, 3367 (2000)

Electrochemical Solution Growth of Magnetic Nitrides



- **Not electroplating!!!!**
- **Molten salt solution growth of GaN developed and patented at Sandia**
- **Create ionic precursors electrochemically**
- **Use salt transport to deliver precursors**
- **Increase growth rate through flux of reactants (increase currents, N_2 flow also has an effect)**
- **Can control oxidation state of transition metal**
- **Produces high quality material**

$$\frac{1}{2}N_2 + 3e^- \rightarrow N^{-3}$$

$$Fe \rightarrow Fe^{+3} + 3e^-$$

Precursors can be replenished as they are consumed

Advantage: Continuous, isothermal or steady-state growth

U.S. Patent Filed November 2014

Other Magnetic Nitrides of Interest

Material	Phase	σ_s (Am²/kg)	J_s (T), if available	T_c (K)	H_c (A/m)
FeN	rocksalt (fcc or fct)	209			
γ'-Fe_4N	antiperovskite-like	209	1.89	769	460
α''-$Fe_{16}N_2$	tetragonal	230 - 286	2.3	810	
α''-$Fe_{90}N_{10}$		230			
g-C_4N_3	graphitic	62			
MnN	rocksalt	194-308			4000
α-Fe	bcc	217	2.15	1044	70

- **Nitrides will have higher resistivities than current transformer core materials and will not require laminations of inactive material to mitigate eddy current losses**

Grid-connected inverter with high-frequency DC link

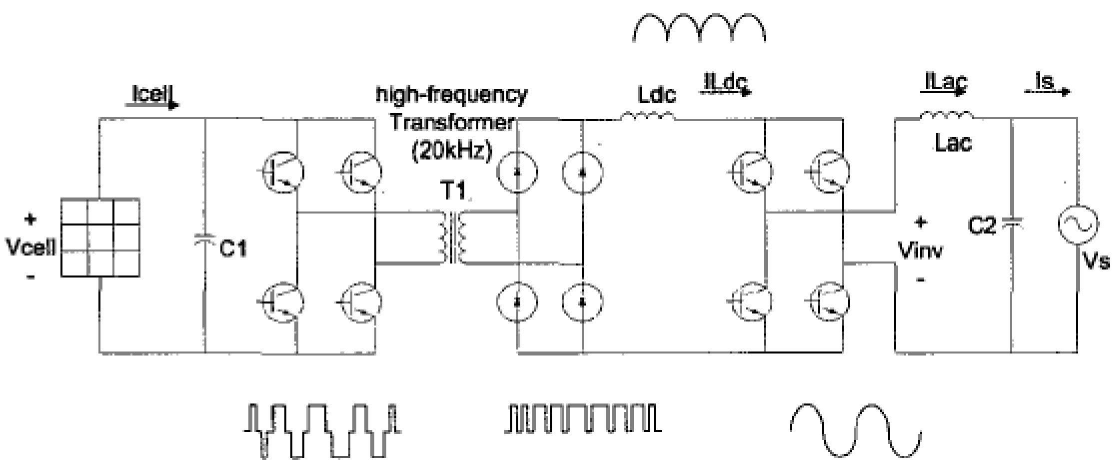
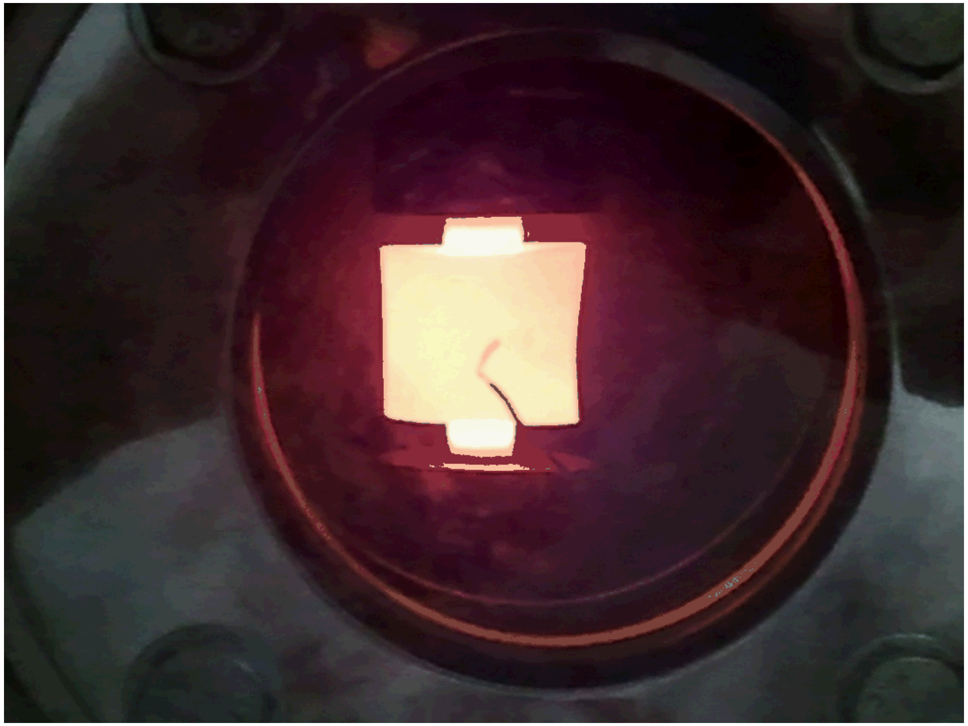


Fig. 1. Grid-connected inverter with high-frequency DC link.

Y. Jung, et. al., High-frequency DC link inverter for grid-connected photovoltaic system, IEEE, 1410 (2002).

Spark Plasma Sintering (SPS)

- DC current
  - ON(1-99 ms)/OFF(1-9 ms) pulse
- Surface activation
  - Electromigration
- Short time
  - 5~30 min
  - Max. heating rate ~ 400 °C/min
- SPS-825S
  - Max. force: 250 kN
  - Max. current: 8000 A
  - Sample dimension: Φ80 mm



- Offers the ability to fine tune grain size in sintered devices

SPS for Manufacturing Ceramics

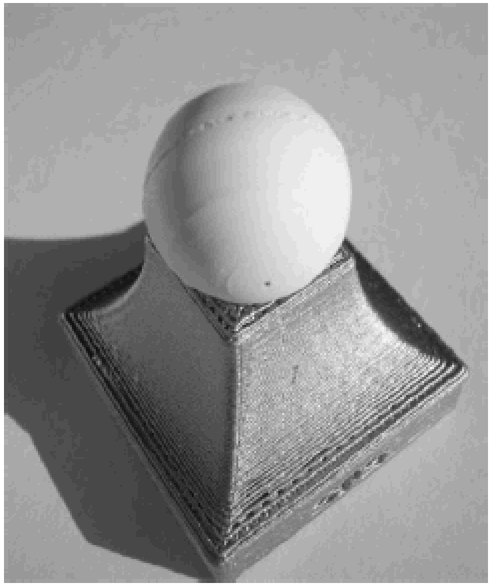


Fig. 9. Al_2O_3 sphere obtained in one single step by SPS.[63]

J. Galy, Private Communication, 2007.

Hungría et. al., Adv. Eng. Mater. Vol. 11 (2009) 616
DOI: 10.1002/adem.200900052

SPS System Manufacturers:

- **Fuji Electronic Industrial Co. (Japan)**
- **FCT Systeme GmbH (Germany)**
  - **Can make components up to 500 mm (~20") in diameter**
- **Thermal Technology LLC (Santa Rosa, CA)**

- **Size of equipment increasing to accommodate commercial needs**
- **Technology for continuous SPS under development**
- **A large number of companies have acquired SPS but often request this info to not be made public to maintain a competitive advantage**