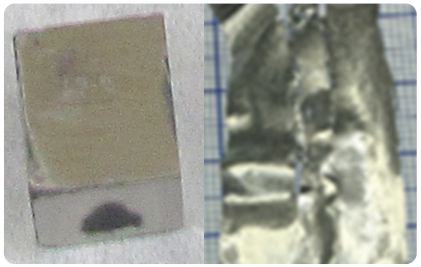
Development of Sustainable High Performance Magnetic Materials for Exceptional Power Density Electric Drive Motors

Iver E. Anderson (PI), Jun Cui, Matthew J. Kramer



Ames Laboratory
June 22, 2021

Project ID: elt215

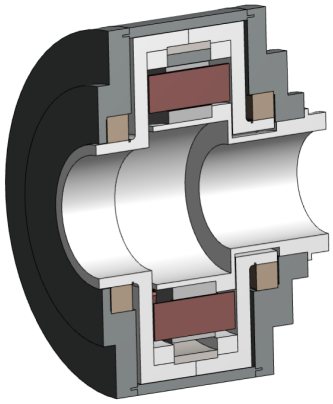


Figure adapted from ORNL

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Overview

Timeline

- Start: October 1, 2018
- End: September 30, 2023
- Percent complete: 30%

Budget

- Project funding estimate
  - $2,250 K (Federal)
  - $0 K (Cost share)
- Funding for FY 2021: $450K (Permanent Magnet portion)

Barriers and targets

- Barriers addressed
  - Permanent magnet (PM) cost and heavy rare earth (HRE) element scarcity and price volatility
  - Non-rare earth PM electric motor has low power density
- Targets
  - Exceptional drive motor power density and reduced cost (50 kW/l at $3.3/kW).

Partners

- Oak Ridge National Laboratory
- National Renewable Energy Laboratory
- Sandia National Laboratory
- University Consortium Partners

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Sandia National Laboratories

Consortium Relevance

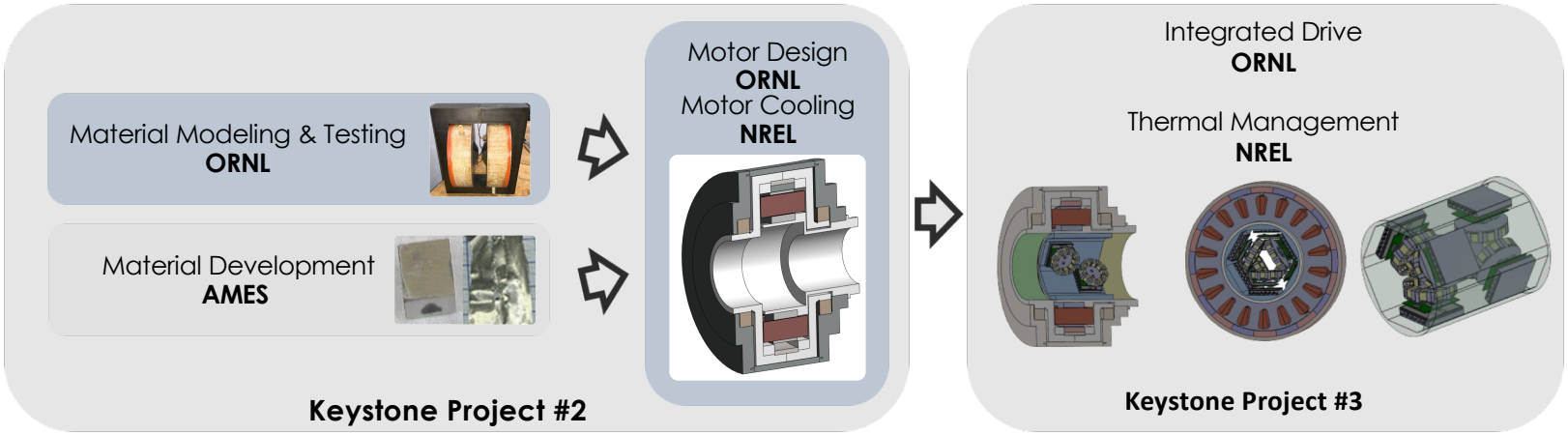
Magnetic Material Development

Electrical steel core losses

- Lower machine inductances lead to worse pulse-width modulation induced core losses
- Lower surface area to volume ratio makes accurate prediction of hot spot temperatures more important for cooling system analysis
- Vector hysteresis and eddy current losses exhibit different flux density magnitude dependency than uniaxial properties

Heavy rare earth free permanent magnets

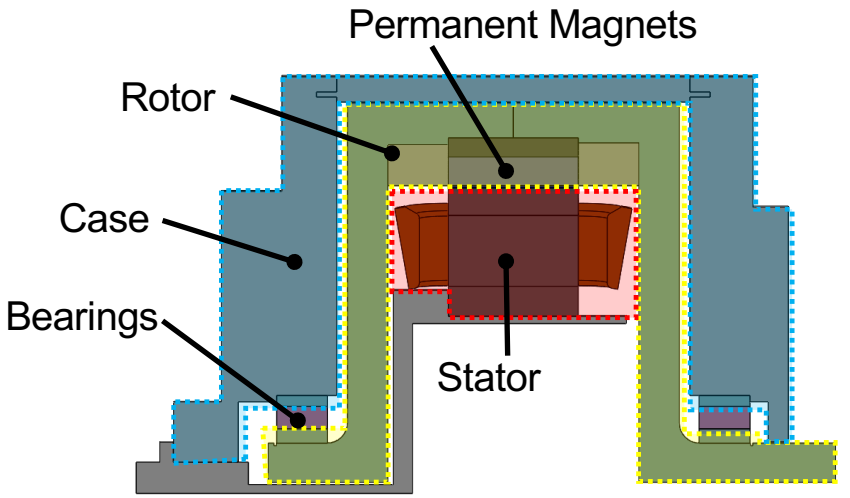
- Low coercivity leads to reliability issues from demagnetization
- Complex magnet arrangements lead to vector demagnetizing fields that are not well modeled with commercial software



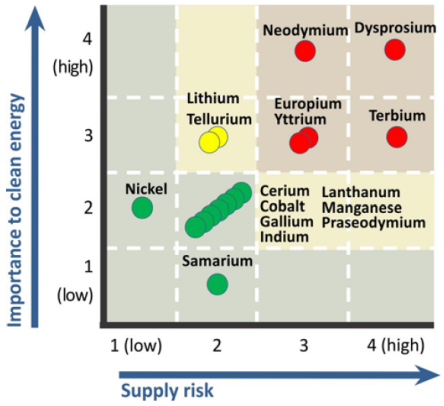
Any proposed future work is subject to change based on funding levels

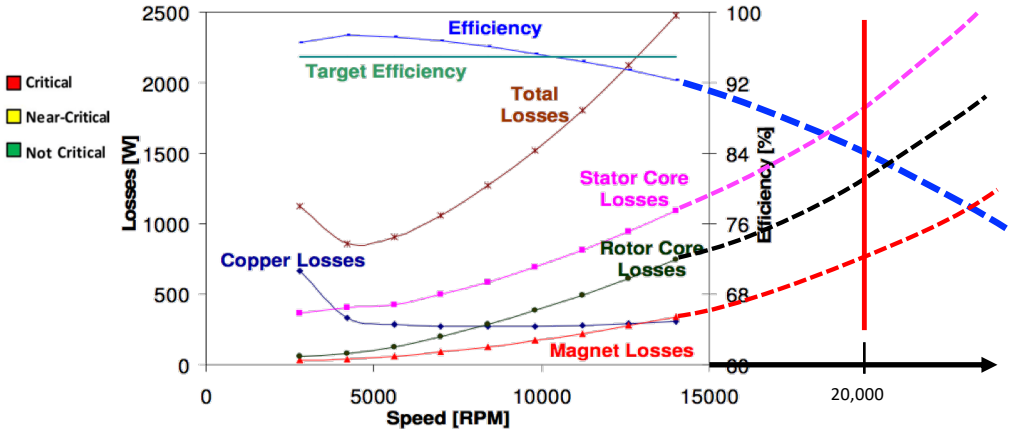
Relevance

- Objective
  - Develop permanent magnetic (PM) materials and processes to avoid scarce/costly heavy rare earth (HRE) metals, but have suitable performance for electric motors with exceptionally high power density.
- Impact
  - Reduces permanent magnet (PM) motor magnet eddy current losses at increased frequency (up to 20,000 RPM)
  - Improve PM motor power density for new outer rotor PM motor designs (ORNL)
  - Maintain drive system cost-effectiveness and high efficiency at elevated temperature to minimize PM cooling needs.



Motor Design (ORNL)
Figure adapted: Kevin Bennion (NREL)





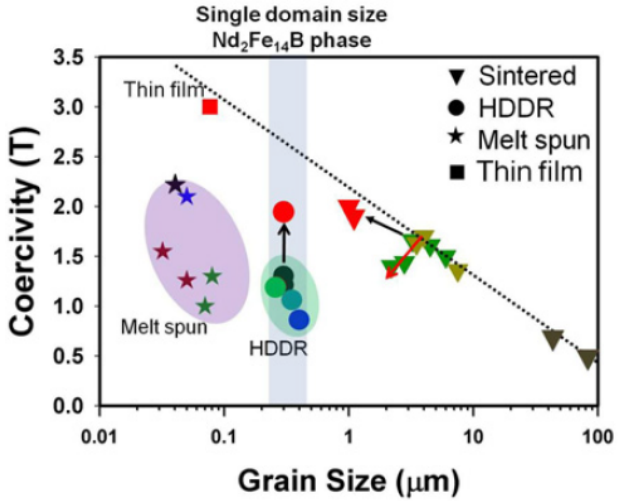
Milestones: Permanent Magnets

Tasks #	Description	Status
FY21-Q1	1) Down-select TLP sintering-aids and utilize conventional bonded magnet alloy (without Nd-enrichment) powder to develop TLP sintering at reduced temperatures, compared to previous results with intrinsic (Nd-rich) TLP alloy powders 2) Assemble and test a tabletop jet-mill; testing the capability to produce (<5µm) powder, including particle size, particle size distribution, and oxygen passivation, compared to experimental ball-milled powder.	100%
FY21-Q2	Q2 Demonstrate approach to superfine powder production with single jet mill to particle size 3-5 microns and proof of concept for alternative (non-oxidation) passivation	70%
FY21-Q3	1) Develop the capability of the single-jet mill to produce superfine (1-3 µm), passivated powder and produce fine-grained sintered RE magnets with TLP sinter aids recognized target erosion impurities for preliminary measurement of magnetic properties. 2) Arrange with vendor (Netzsch) to use their demonstration lab multi-jet mill to demonstrate superfine powder production with narrow size cut and conventional passivation procedure (3 microns or less, producing 1 kg)	50%
FY21-Q4	1) Demonstrate alternative passivation approach at vendor during production of superfine magnet alloy powder using the multi-jet-milled and passivated powder. 2) Fabricate superfine-grain sintered magnet using the passivated single jet-milled powder, combining extrinsic TLP sintering aids and low temperature sintering conditions and characterize magnetic properties up to 400K.	10%

Task 2.7 Develop ultrafine-grain HRE-free RE-PM with high coercivity at high temp.

Challenge: ***Highly refined grain size in HRE-free RE-PM with improved magnetic & mechanical strength***

Advantages



- Dy (a HRE) in RE-PM for drive motors maintains high coercivity at high operating temperatures.
- Ultrafine grain HRE-free RE-PM also can raise coercivity and stabilize high temperature properties.
- New ORNL outer rotor design should lower mechanical property needs for permanent magnets.

Hono et al. Scripta Materialia 67, 2012 (530)

Challenges

- Difficult to produce and handle fine powder in manufacturing process
  - Extreme milling time/intensity is required for finer powder, need refinement without contamination.
  - High flammability of fine RE-PM powder needs highly controlled processing, leads to extra cost
- Difficult to fabricate into bulk magnet
  - Fragmented shape/high surface area impedes grain alignment & promotes rapid grain growth.
  - Multi-jet milling needs to be developed promoting ultrafine (<2um) rounded powders & passivation.
- Deterioration of bulk mechanical properties
  - Residual surface oxides embrittle microstructure and lower saturation magnetization.
  - Should develop strategies to promote passivation without oxides to reduce embrittlement.

Approach: Processing Ultrafine Grain Size HRE-free RE-PM

- **Investigate production of ultrafine powder/uniform composition HRE-free (e.g.,Dy): Down-selected**
  - Feedstock: (commercial) strip cast-HD, (lab capability) melt spun/thin profile chill cast
  - Size Refinement: (commercial) multi-jet milling, (lab capability) ball milling (BM)
- **Surface oxidation protection:**
  - Minimal oxide growth (lab glove box processing)
  - Alternative surface reaction during milling (prove concept, scalable?)
- **Investigate processing of aligned, ultrafine grain bulk magnets:**
  - Loose powder aligned by external pulsed field: die compact or CIP
  - Full density sintering: liquid phase sintering aid addition to reduce temperature
- **Alternative novel processing approaches:**
  - Alternative surface reaction during milling (experimental concept test delayed)
  - Additions to minimize grain growth by reducing sintering temperature (in-progress)

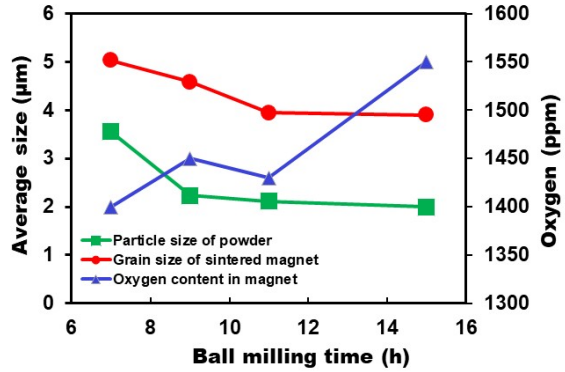
	Planned milestones and annual go/no-goes
2020	• Enable fine grain approach as a replacement for heavy RE stabilization of coercivity at high temperature.
2021	• Demonstrate superfine powder production at full size refinement and with alternative passivation (1-3 micron).

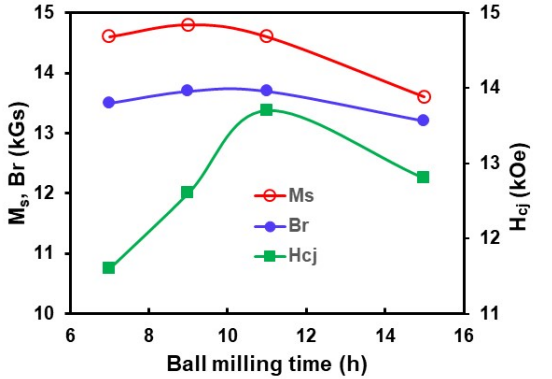
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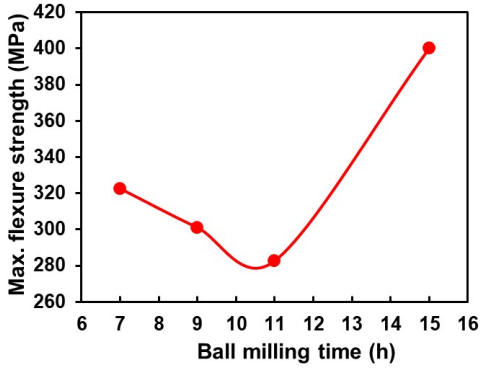
Accomplishments: ***limit of ball milling on grain size refinement and higher magnet performance***

Approach: will extended ball milling time for 15 h lead to further improvement?

- Average powder and magnet grain sizes don't change for 15 h, compared to 11 h milling time.
- Oxygen in 15 h-milled magnet is considerably increased.
- M_s and B_r trend downward after 9 h to 15 h.
- H_{cj} first increased to best at 11h milling and then decreased after 15h milling.
- Maximum flexural strength decreased to 11 h, then increased for 15 h: seems to be abnormal behavior-----might be attributed to the oxygen increase in the magnet.
- Besides the effect of finer grains, the oxygen content may play an important role in the magnetic and mechanical properties.





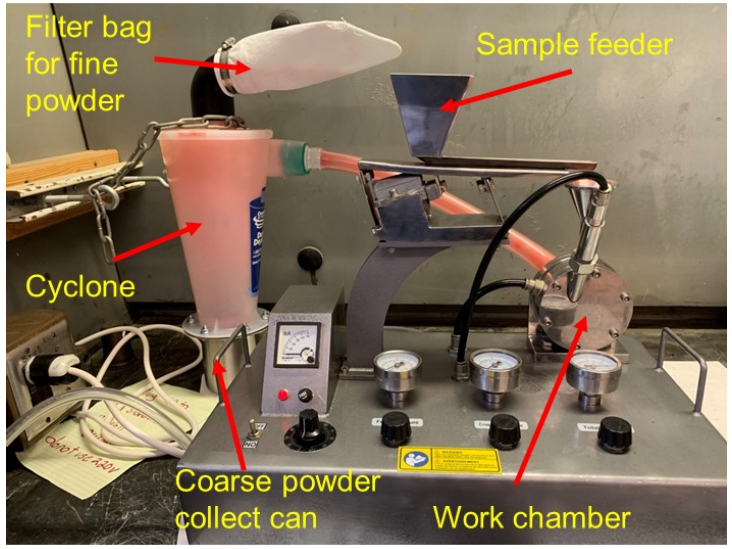


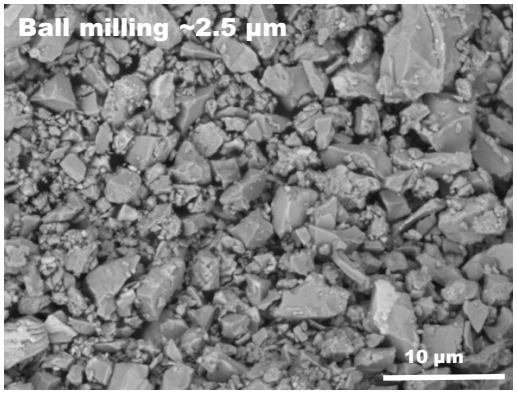
Accomplishments: Jet milled fine powders

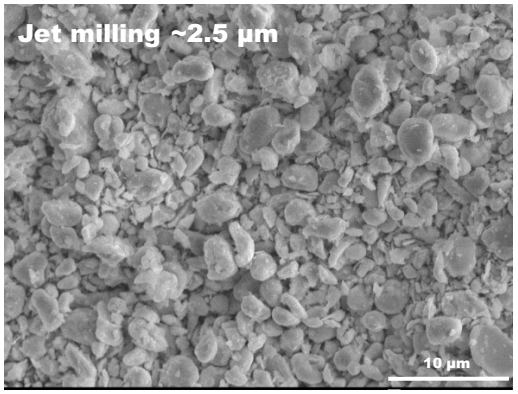
Approach:

- Use bakelite powder (red in color) for trial runs. The particle size of the feedstock bakelite is 1~3 mm.
  - 90% of the powder in collection can (50 μm)
  - 10% of the powder in filter bag (10 μm).
- Jet milled powder is more rounded, improved shape can promote better alignment during pulse magnetizing.
- The lab-type jet milled powder size target is around 3 μm (commercial size). Extended runs combined with custom-designed cyclone classification may push the ultimate powder size to 1 μm.
- Developing experimental plans and setting up a small lab-type multi-jet mill and applied for EHS approval for operation in controlled atmosphere.

Jet mill setup in a hood





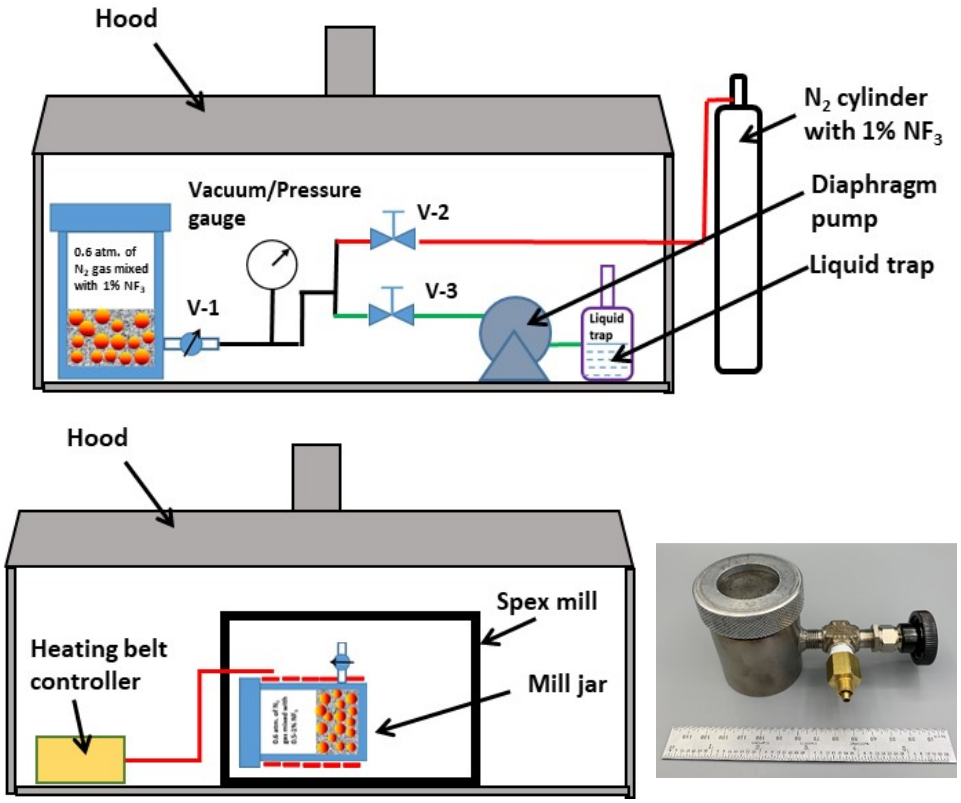


Accomplishments: *Powder passivation concept test*

Approach: Develop fine-powder passivation technology. The concept involves milling powder under a reactive gas at elevated temperatures.

- ❑ Modify a milling device for evacuation & back-filling with reactive gas and for heating to permit reactive milling above ambient.
- ❑ Demonstrate passivation reaction during Spex ball milling with sealed reactive atmosphere.
- ❑ Fabricate bulk magnets using the passivated fine powders and compare properties with previous experimental magnets.
- ❑ Completed the system design and operation procedures for passivation process. Passed safety readiness review. First experimental trials pending receipt of special gas.

Process chart of NF_3 filling and exhaust



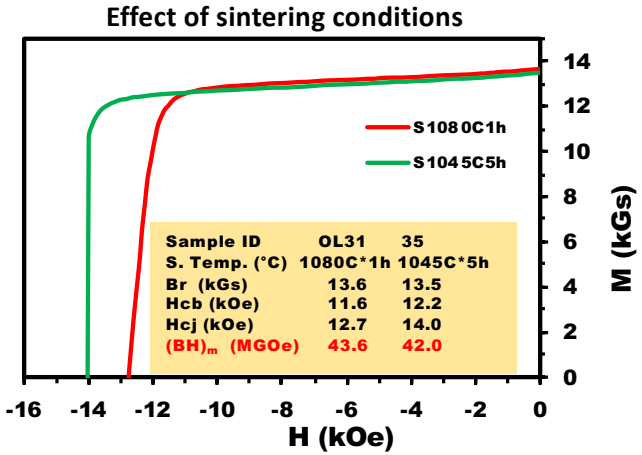
Spex mill NF_3 passivation

Spex mill jar with an inlet port and valve

Accomplishments: Grain growth suppression

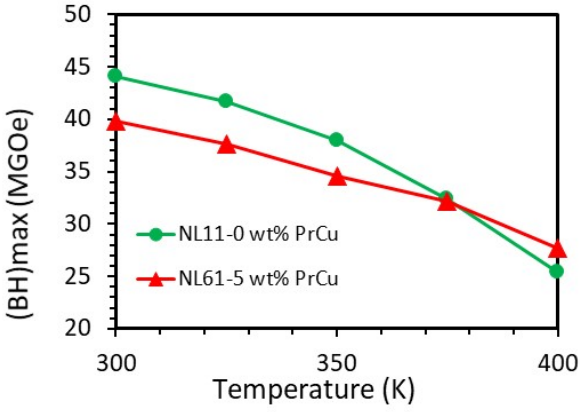
Approach I: Control of sintering condition

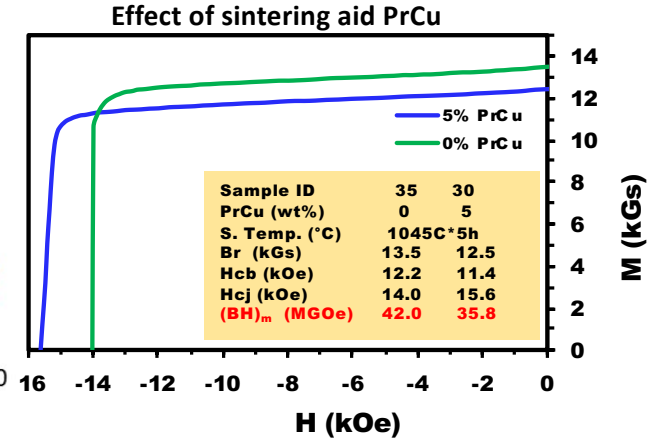
- Lowering sintering temperature and extending sintering time produce sufficient densification with min. grain growth.
- Hcj is increased from 12.7 to 14.0 kOe and the squareness of demag. curve is further improved. The resulting magnet achieved a smaller grain size (still 2X feedstock powder size) with uniform sized grains.



Approach II: Addition of external sintering aid; low melting alloy

- At the same sintering conditions, the addition of 5 wt% Pr-Cu boosts up Hcj from 14.0 to 15.6 kOe. Need further improvements to decrease final grain size.
- With Pr-Cu addition, the magnet starts to outperform when T>375 K

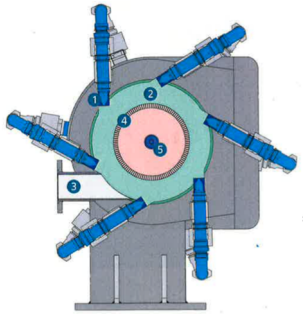




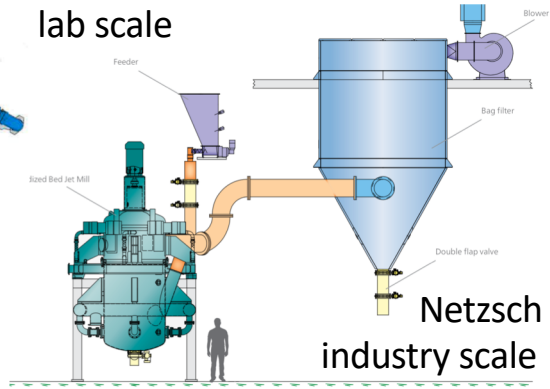
Responses to Previous Year Reviewers' Comments

Question 4: Given the lack of domestic availability of jet-milling equipment, alternate approaches for size refinement should be identified, if they exist. More focus on scalability is needed.

Ames response: The selected jet mill manufacturer is Netzsch, a German company specialized in grinding technology. Netzsch uses the same method for autogenous milling in lab and industrial scales. An alternative Japanese manufacturer with equivalent technology is Aishin Nano-Technologies. Major scale-up effort is planned for FY22 activities.



Netzsch lab scale



Netzsch industry scale

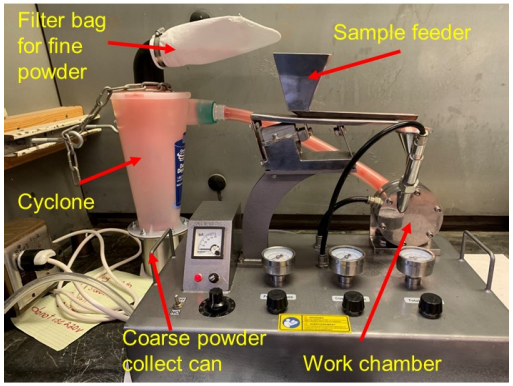
It is a challenge (oxide contamination, flammability) to handle superfine metal powders in large scale. This drives us to develop powder passivation with minimum effect on magnetic properties, using a simple stagnant atmosphere analogue, a "Spex" milling capsule.



Spex milling capsule.

Question 6: Most reviewers considered the resources to be adequate, although one reviewer thinks resources may not be enough to complete the jet milling work. It was suggested to work more closely with OEMs.

Ames response: Resources are tight to implement the jet-milling work, but a small tabletop low-power jet mill (MTI Corp.) was acquired. It will be modified for demonstrating the passivation treatment concept (for IP purposes) with flow-through reactive atmosphere. Perhaps alternative funding sources (including OEMs) will grasp the impact of this technology development on permanent magnet manufacturing and EV drive systems before our off-shore competitors.



MTI low-power jet mill.

Collaboration and Coordination



- Collaboration on motor design advances and sharing expected material magnetic & mechanical properties.
- Guidance on modeling of geometry-corrected demagnetization lines of high M_{sat}/high H_{ci} graded magnet types.
- System level performance modeling.



- Investigation of thermal and mechanical properties of newly developed magnetic materials.



- Coordination of efforts of university partners who are actively engaged in permanent magnet development for associated motor designs.
- Cooperative development of composite permanent magnet processing and designs.

Remaining Challenges and Barriers

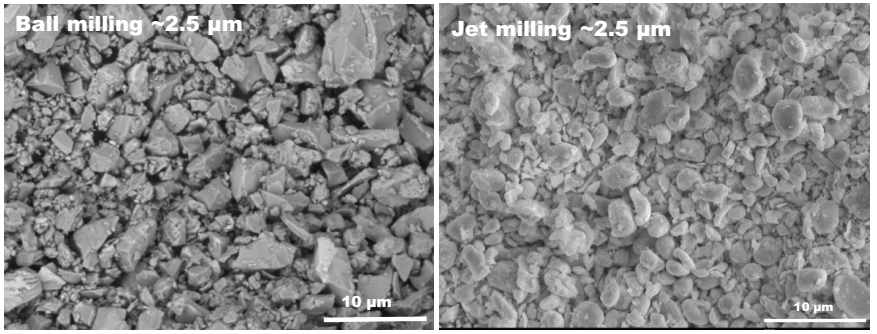
- **Multi-jet milling method selected for controlled atmosphere production of ultrafine $Nd_2Fe_{14}B$ powder with uniform size, but not available in US.**
  - Experiments run with common commercial feedstock particulate from strip casting/HD in Dy-free composition.
  - Size refinement by multi-jet milling uses intense energy and controlled atmosphere to produce high purity, rounded ultrafine (< 2 μm) particles preferred for alignment.
  - Capability compatible with plans for minimized oxide growth by development of surface reaction, dry filter box collection, and glove box processing.
  - Co-operation on system purchase is being sought between consortium members.

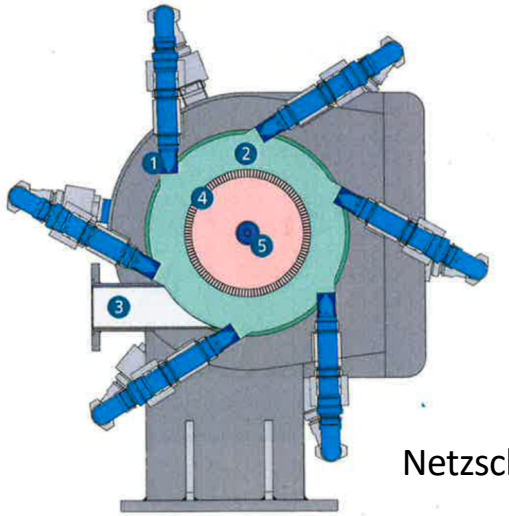
- **Inert handling, alignment, and consolidation methods need to be extended to retain advantages of new ultrafine jet milled powders.**
  - SEM or automated size analysis needs to verify uniform refinement and rounded shape of as-milled powder.
  - Need to investigate if pulse magnet for loose powder alignment may be incorporated with high pressure CIP densification to suppress any misalignment of powder before sintering.

- **Powder sintering parameters need to be modified to fully exploit reduced temperature of TLP additives activity.**
  - Alloy composition and wt.% of TLP sintering additive needs to be finally selected, along with particle size.
  - Temperature stability of magnetic properties of resulting ultrafine grain magnets must be tested.

Proposed Future Research

- Must gain access and sufficient expertise with multi-jet milling system to perform critical experiments on alternative methods for passivation of resulting ultrafine (dia.< 2 μm) powder of HRE-free RE-PM alloys.
- Need to finalize sintering additive composition, quantity, & particle size for blending with HRE-free RE-PM alloy powder to maximize retention of ultrafine grain size during full density consolidation without compromising magnetic property gains.
- Validate the hypothesis that mechanical properties improve for ultrafine-grain magnet if powder surface coating is thinner and Nd-rich phase on grain boundary suppresses microcrack propagation.
- For ultrafine grain RE-PM magnets, should develop final shape processing to minimize material waste, only needing final grinding to dimensions.





Netzsch Jet mill

Multi-jet milling system schematic.

Any proposed future work is subject to change based on funding levels.

Summary

- We can make bulk magnets with magnetic properties superior to commercial grade magnets from similar RE-rich (HRE-free) Nd-Fe-B alloy
  - Feedstock powder size refined via extensive ball milling
  - Powder loaded in glove box into alignment capsule for pulsed magnet (9T) alignment.
  - High density green compact made with a new high pressure CIP setup (500 MPa)
  - All powder & green compacts transferred into operations/equipment without air exposure
  - Sintered magnet grain size reduced by 20% from typical 5 ± 0.2 mm to 4 ± 0.2 mm via two-step sintering treatment (using feedstock powder with finer particle size) without sintering aids.

- Developed critical experiment for ultrafine feedstock powder production where demonstration of the novel passivation technology of heavily milled powder is in-progress.

- Selected suitable external TLP additive, but need further improvement of processing and sintering conditions to limit magnet grain growth, while maintaining full density and sufficient mechanical strength.