

# *DOE Bioenergy Technologies Office (BETO) 2021 Project Peer Review*

## *FCIC – Task 1: Materials of Construction*

Date: March 16, 2021

Technology Area Session: FCIC

ORNL: Jun Qu (Task Lead), Kyungjun Lee, Jim Keiser,  
Peter Blau

ANL: George Fenske (Task Co-Lead)

INL: Jeff Lacey and Vicki Thompson



# FCIC Task Organization

## Feedstock

**Feedstock Variability:**  
Develop tools that quantify & understand the sources of biomass resource and feedstock variability

## Preprocessing

**Preprocessing:**  
Develop tools to enable technologies that provide well-defined and homogeneous feedstock from variable biomass resources

## Conversion

**Conversion (High & Low-Temperature Pathways):**  
Develop tools to enable technologies that produce homogeneous intermediates that can be converted into market-ready products

**Materials Handling:**  
Develop tools that enable continuous, steady, trouble free feed into reactors

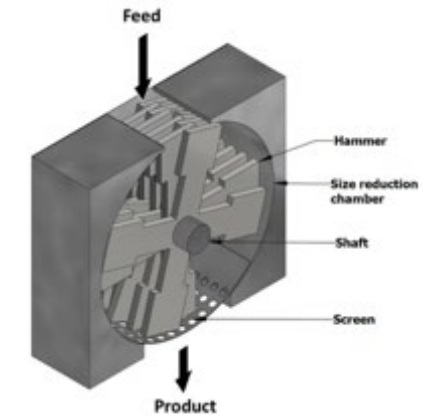
**Materials of Construction:**  
Develop tools that specify materials that do not corrode, wear, or break at unacceptable rates.

## Enabling Tasks

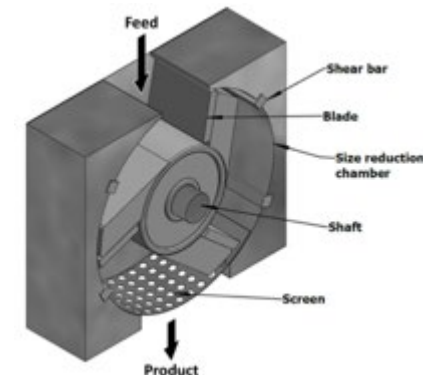
**Data Integration:** Ensure the data generated in the FCIC are curated and stored – FAIR guidelines

**Crosscutting Analyses TEA/LCA:**  
Works with other Tasks enable valuation and intermediate streams and quantify impact of variability.

- **Objective:** Use integrated efforts of **characterization, modeling, and testing** to:
  - gain fundamental understanding of the **failure modes and wear mechanisms** of biomass preprocessing equipment,
  - develop **analytical models** to predict wear and establish material property specifications,
  - select and evaluate **candidate mitigations**, via both feedstock modifications to reduce abrasiveness and developing more wear-resistant tools, and
  - **share** the fundamentals and mitigations with the biomass industry.
- **Current limitations:** Most current comminution equipment and tool materials are not optimized for processing biomass, particularly dirty feedstocks, and often fail or wore out prematurely.
- **Relevance:** This task uses science-based approach, in stead of trial-and-error, to develop effective mitigation strategies. The knowledge and mitigations from this Task **will enable rapid of selection of feedstock modification methods and wear-resistant tool materials for sustainable performance and product quality during biomass preprocessing.**
- **Risks:** Inconsistent wear behavior due to feedstock variations and mitigations difficult to implement due to high cost.



↓ *Focus transitioned from hammer mill to knife mill in FY20 Q4*



O. Oyediji, P. Gitman, J. Qu, E. Webb, ACS  
Sustainable Chemistry & Engineering 8 (2020) 2327.

# 1 – Management

Subtask	Lead(s)	Major Responsibilities
1.1: Understand and mitigate equipment wear in preprocessing	ORNL (Lead), ANL, and INL	Characterization of worn components, bench-scale testing of baseline materials of construction, identification and evaluation of advanced materials/coatings that resist wear and degradation
1.3: Mechanics of wear	ANL	Physical and mathematical modeling of wear phenomena active in milling operations. Prediction of wear metrics (wear volume and shape) based on material properties and operational parameters.



- **Risks/Mitigations:**

- Breakdowns or unavailability of instruments for characterization and testing

**Mitigation:** *Among the three national labs, we are confident to find alternatives*

- Wear models based solely on one wear mode would not capture other wear mechanisms

**Mitigation:** *To incorporate multiple wear modes into the overall wear model by conducting the root cause analysis*

- Candidate mitigations difficult to scale-up due to cost or technical challenges

**Mitigation:** *To select and identify appropriate mitigations using techno-economic analysis (TEA) in collaboration with FCIC Task 8*

- **Communication/Collaboration:**

- Communicating and coordinating with FCIC Tasks 2, 5, and 8 as well as the DFO project with Forest Concepts
- Communicating and collaborating with industry including milling OEMs (including EberBach and West Salem Machinery), material service providers for advanced coatings and surface treatments, and IAB members



# 2 – Approach

**Technical Approach:** Use a systematic quality-by-design approach with **integrated efforts of characterization, modeling, and testing** to gain fundamental understanding of the **failure modes and wear mechanisms** of biomass preprocessing tools, develop analytical **models to predict wear** and establish material property specifications, select and evaluate **candidate mitigations** based on modeling and lab-scale testing and identify top-performing mitigation for PDU validation, and **share** the fundamentals and mitigations with the biomass industry.

## Challenges:

- **Complex wear mechanisms** requiring correlation among biomass intrinsic and extrinsic inorganics, tool alloy hardness and fracture toughness, and wear performance in both erosion and abrasion
- Development of **effective AND low-cost** mitigations
- Quantification of **combined benefits** from increased tool life and throughput as well as reduced downtime and power consumption

## Metrics:

- Technical metrics: **wear modes, volumetric wear rate** in bench-scale erosion and abrasion testing, **blade edge sharpness** change in small knife mill testing
- Economic metrics: **costs** of tool materials and manufacturing, **cost savings** of reduced machine shutdown for tool replacement, **energy savings** by higher throughput, and improved **conversion efficiency** by more uniform particle sizes

# 3 – Impact

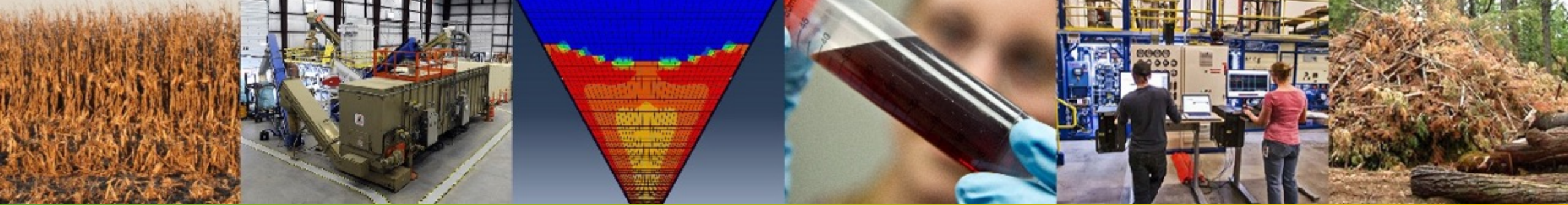
**Impact:** Meets the objectives of FCIC by providing fundamental understanding, predictive tools, and mitigations for equipment wear in preprocessing. QbD approach to identify CMAs and CPPs to provide CQAs on wear, reliability, durability, and performance for individual unit operations.

- Provides fundamental understanding for **tool wear mechanisms** in biomass preprocessing.
- Develops **cost-effective mitigations** for improving tool life and throughput and reduce downtime and power consumption.
- The biomass industry is eager to learn both the fundamentals and mitigation strategies produced in this study, which will potentially improve the operation reliability and efficiency for better economics.

## Dissemination:

- 3 peer-reviewed journal papers (including one selected for journal cover) and 1 conference proceeding in FY20
- 3 conference presentations including 2019 TMS Best Poster Award (3<sup>rd</sup> place)
- 3 technical reports (accessible through OSTI)
- 1 media report in *Biofuels Digest*
- Communications with hammer mill and knife mill manufacturers
- Communications with coating/surface treatment providers





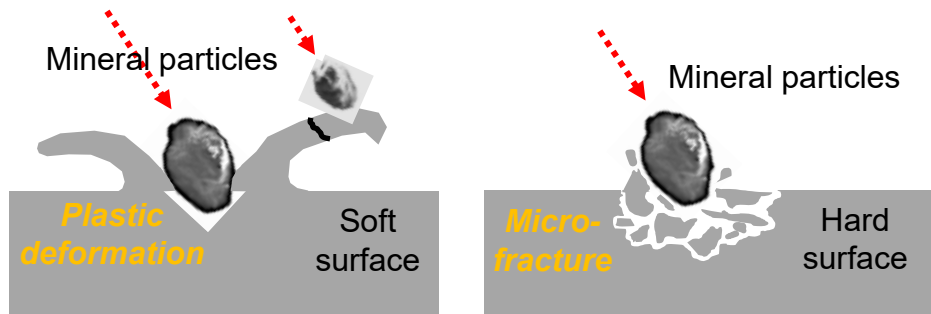
## *4 – Progress and Outcomes*





## General Types of Wear

### Erosive Wear

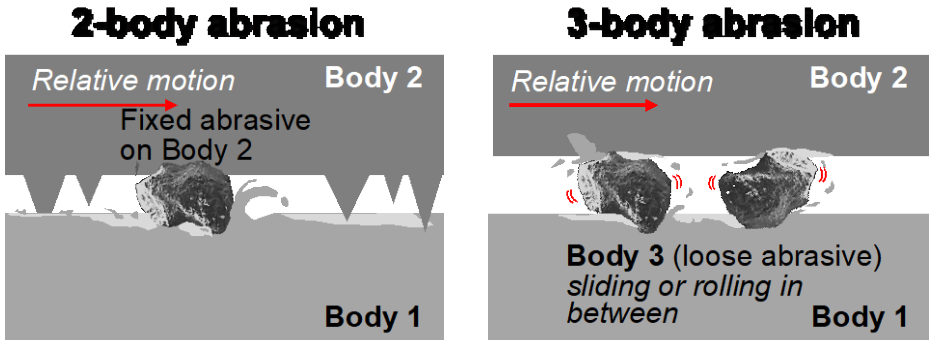


Manner of energy dissipation:  
**Plastic deformation, micro-fracture, heat**

Critical tool material mechanical properties:  
**Fracture toughness, hardness, fatigue ductility, yield strength**

Key processing parameters:  
 Particle hardness, velocity, and size, impingement angle

### 2-body/3-body Abrasive Wear



Manner of energy dissipation:  
**Groove plowing, cutting chips, grit fracture, heat**

Critical tool material mechanical properties:  
**Hardness, yield strength, fracture toughness**

Key processing parameters:  
 Abrasive grit shape/size, load, sliding speed/distance

**Other types:**  
 adhesive wear,  
 impact wear,  
 contact fatigue,  
 fretting wear,  
 oxidative wear,  
 corrosive wear,  
 etc.





# Tribosystem analysis of hammer mill to correlate wear with inorganic particles



Knowledge

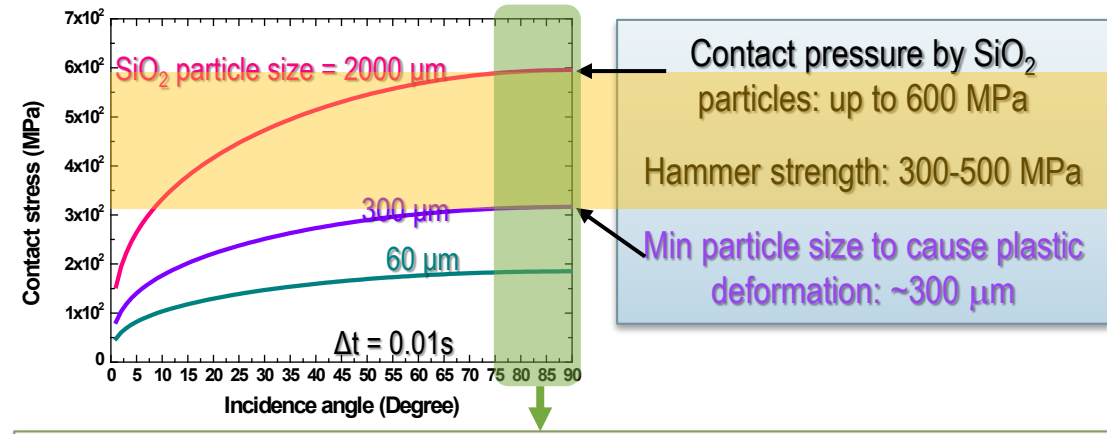
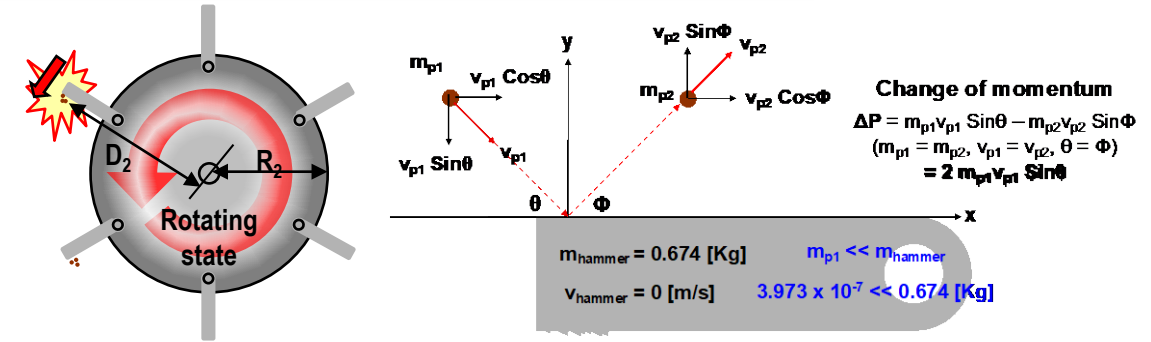
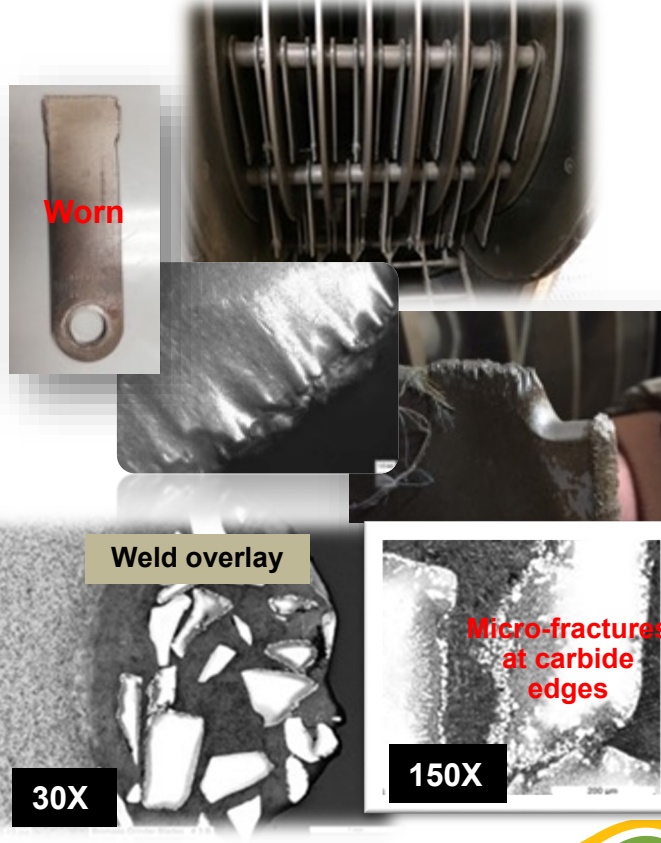


**Hammer Mill: Blunt blades; Comminution mode: Crushing**  
**Wear modes: Erosive wear (dominant) + 2-body/3-body Abrasive wear (secondary)**

Stage 1 Hammer mill

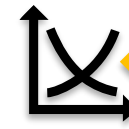


Stage 2 Hammer mill



- A smooth hammer would hit most particles at nearly 90 degree
- A very rough hammer would hit particles at various angles but biased towards large angles

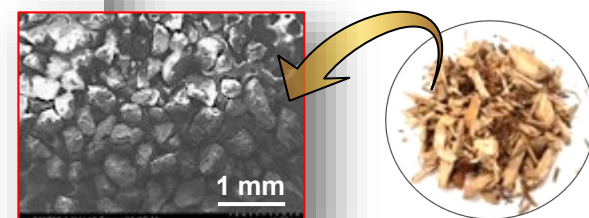
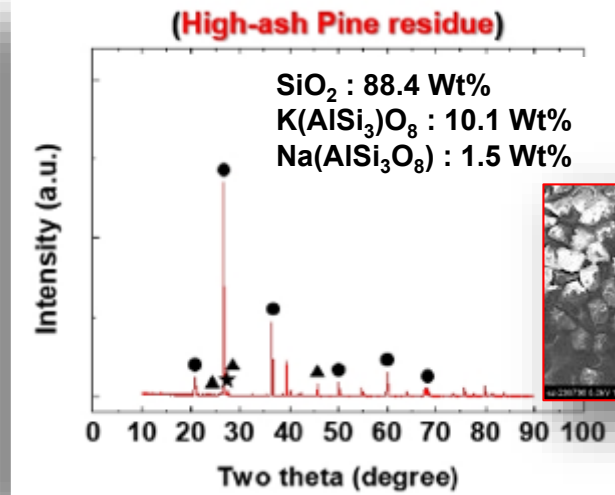
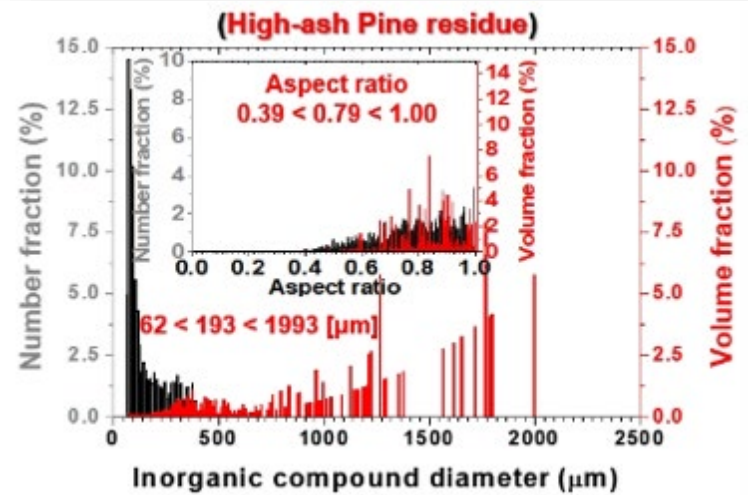
# Characterization of extrinsic inorganic particles



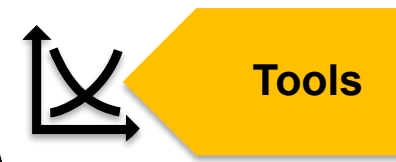
Tools



- Conventional measurement of inorganic content of biomass : ISO 176 and TAPPI T211 → combust wood at 525 °C  
ASTM D1102 → uses dry oxidation of wood at 580~600 °C  
ASTM D3174 → produces ash of coal in furnace at 750 °C
- ORNL newly developed composition-preserving extraction and characterization method allows to discover the original morphology, size, and composition of extrinsic inorganic particles.**

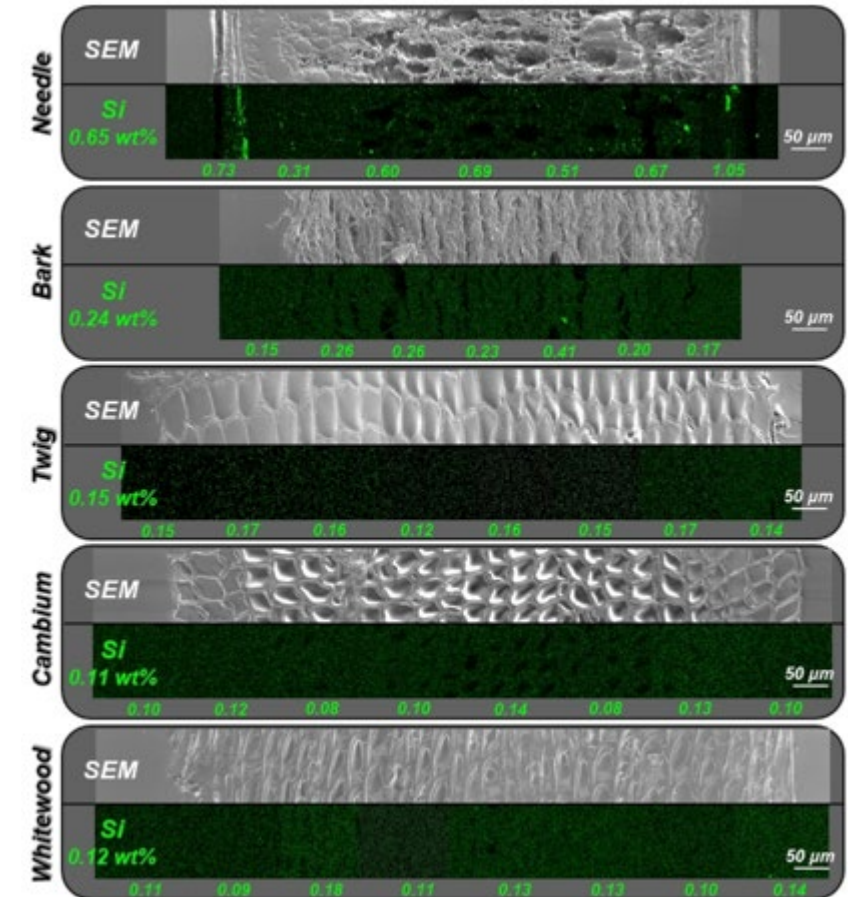
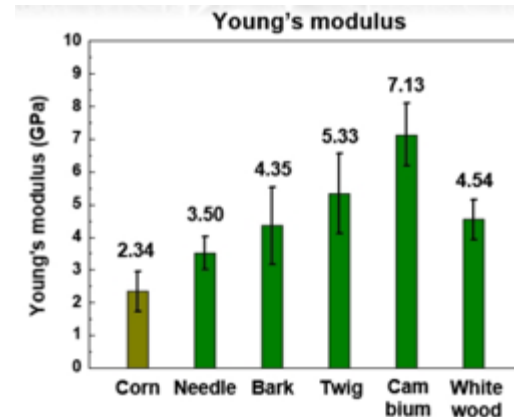
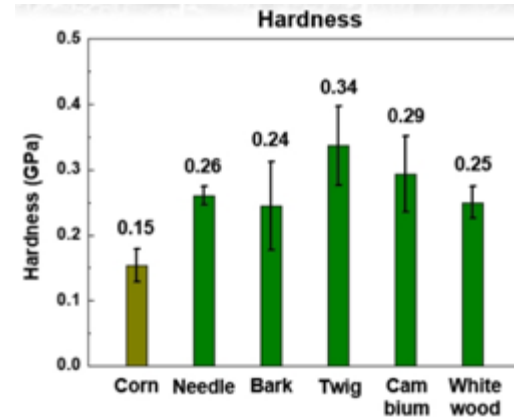
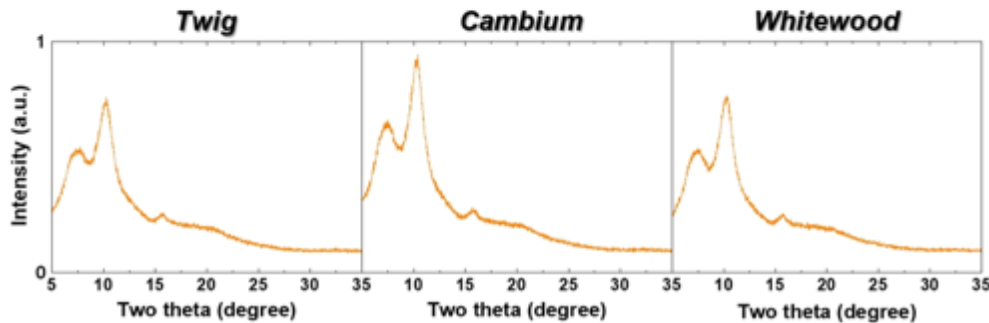
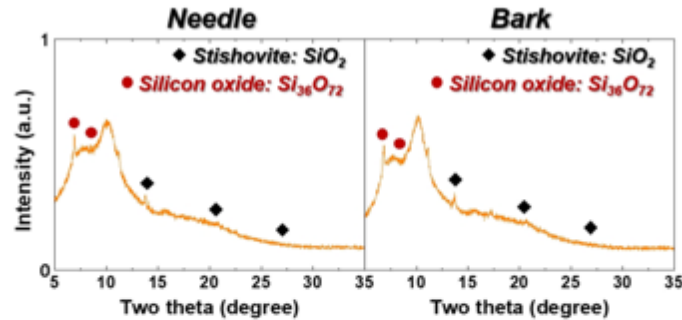


# Characterization of intrinsic inorganics



ORNL comprehensive characterization of intrinsic inorganics for pine anatomical fractions:

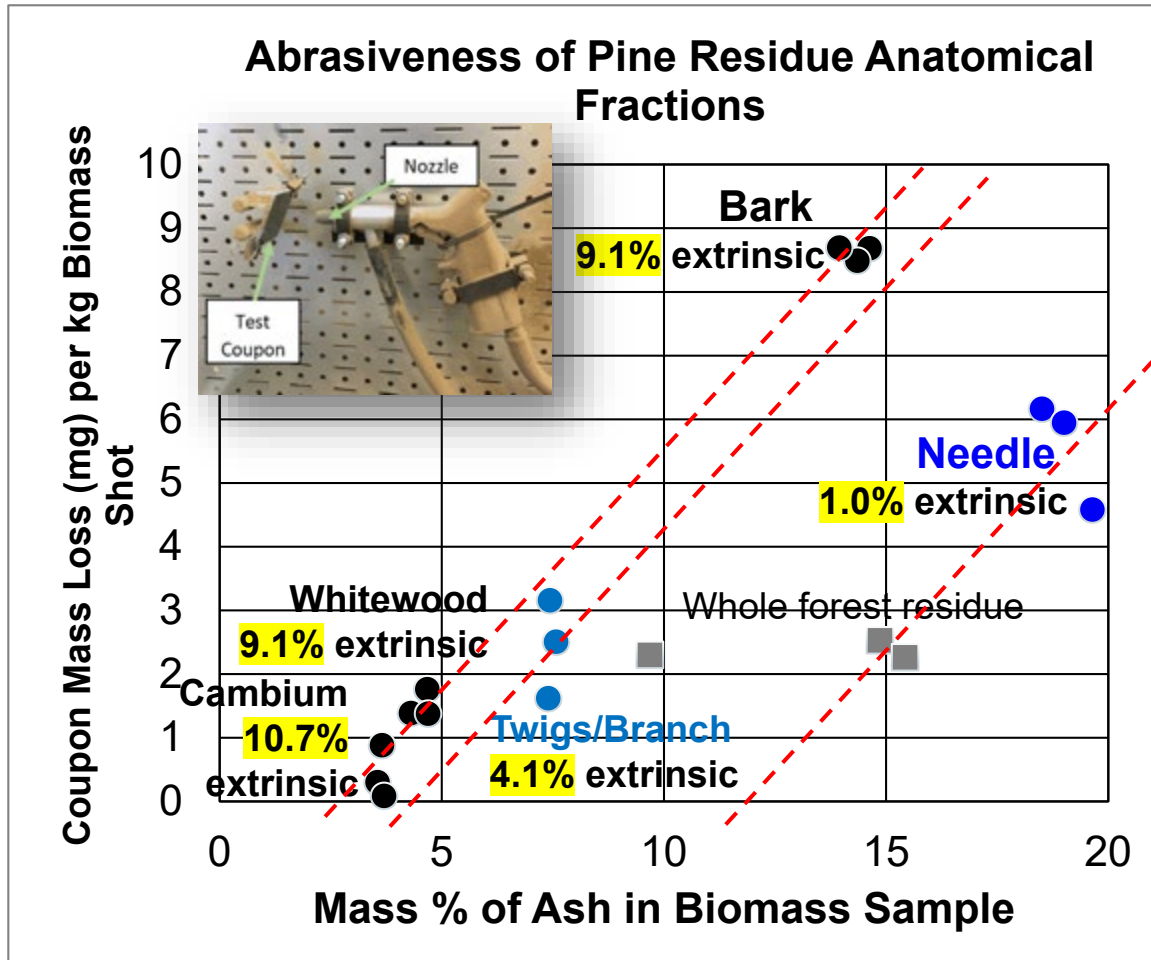
- Species identification by XRD
- Distribution inside biomass by EDS elemental mapping
- Mechanical properties by nanoindentation



# Contributions of macro-level anatomical fractions and inorganics to erosive wear



Knowledge

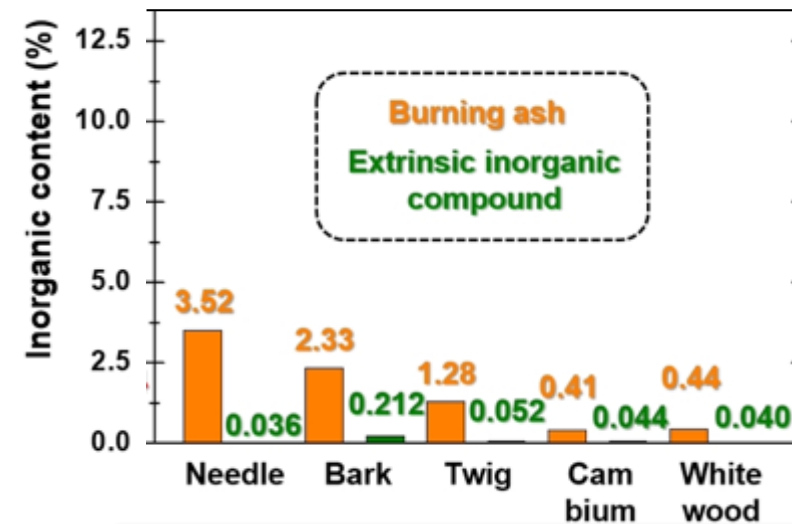


Blasting wear tests using coupons machined off actual hammers (low carbon steel)

Observations:

- Wear often increases along with the total ash content, *but not always*
- Wear is proportional to the percentile of extrinsic minerals in the total ash

**Conclusions:** Both intrinsic and extrinsic inorganic compounds are abrasive, but the **extrinsic minerals are much more abrasive** than the intrinsic inorganics.



1.0% 9.1% 4.1% 10.7% 9.1%

% of extrinsic inorganic particles (>60 μm) in the 'total ash'



# Feedstock modifications for ash reduction for hammer mill



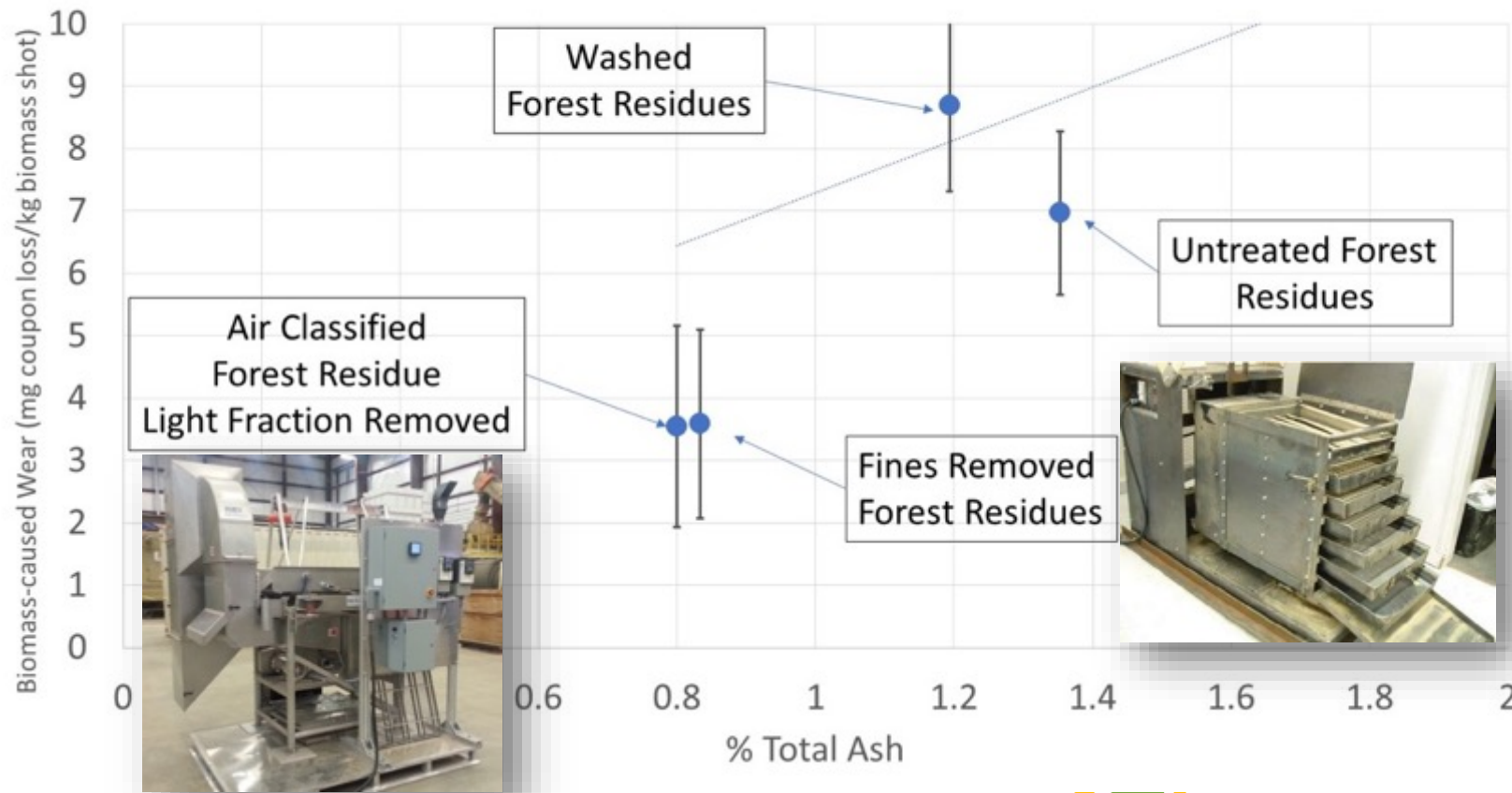
Knowledge



## Effectiveness of Air classification, Size separation, and Water washing

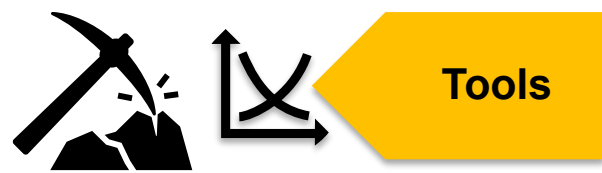
*Blasting wear tests using coupons machined off actual hammers (low carbon steel)*

Wear Properties of Forest Residues



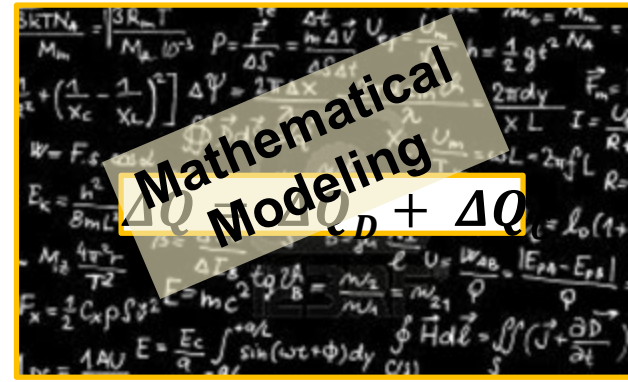
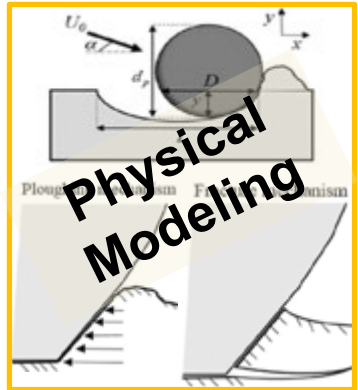
- **Air classification and size separation were effective in reducing erosive wear**
  - Removal of light fraction & fines
  - Modifications of forest residues led to 2X lower wear
  - Modifications of corn stover led to 4X lower wear
  - Cost: ~\$0.84 per ton
- Water washing had little effect in wear behavior
  - Major extrinsic inorganic compounds are water insoluble
  - Washing moved the minerals around, not removing them

# Analytical model of erosive wear



## • Technical progress:

- Guided by INL erosion wear studies and ORNL/ANL material characterization of worn components, **physical models of erosive wear were developed.**
- **Mathematical models of erosive wear were coded** to evaluate ability to predict impact of feedstock and material attributes and process parameters on erosive wear (QbD).
- Validated QbD approach against experimental studies – excellent agreement.
- **Validated wear model against prototypic hammer-milling operation** (Vermeer Stage 1 Hammer Mill).
- Model is guiding selection of candidate materials to mitigate wear of materials (hammers & knife mills).

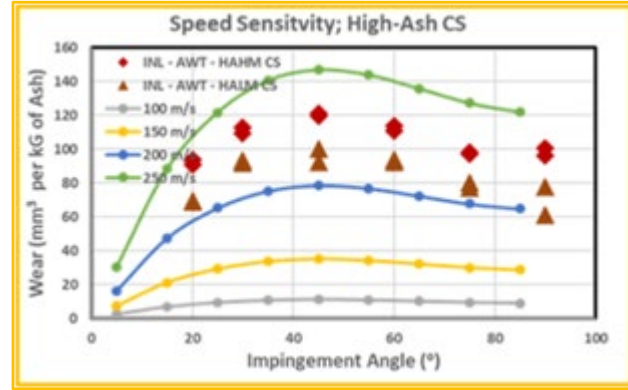


$$\frac{\Delta Q}{m_p} = C_D * \frac{\rho_p^{(1/4b)} * (U_0 \sin \alpha)^{(2+1/2b)}}{\eta^{(3/4b)} \epsilon_f^{(1/b)} H^{(1+1/4b)}} + C_C(1+f) * (1 - \exp(-200\alpha^2)) \frac{\rho_p^{1-f} d_p^{(1-f)}}{\eta^{1-f} H^{1-f} R^{(1-f)}} [U_0^{(3-f)} \cos^2(\alpha) \sin^{(1-f)}(\alpha)]$$

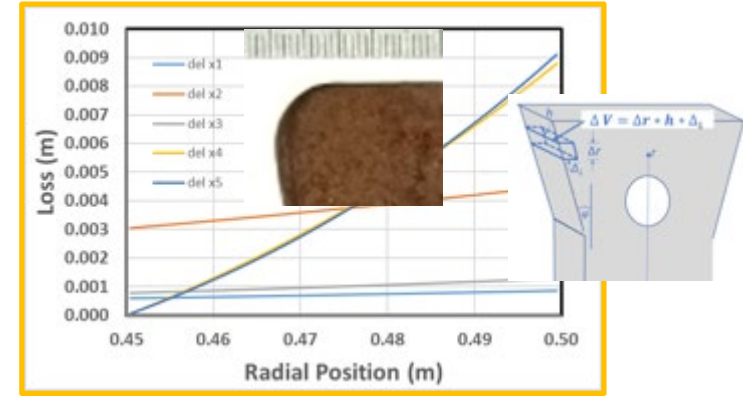
**Ben-Ami erosion wear model**

## • Impact:

- Erosive wear model that relates feedstock attributes, component attributes, and process parameters to critical quality performance attributes.
- Ability to screen effects of feedstock attributes on wear and performance, and more efficiently select wear resistant material solutions to mitigate wear.



Predicted & Measured Simulation Wear



Predicted Hammer Wear



# Transitioned from hammer mill to knife mill in FY20 Q4



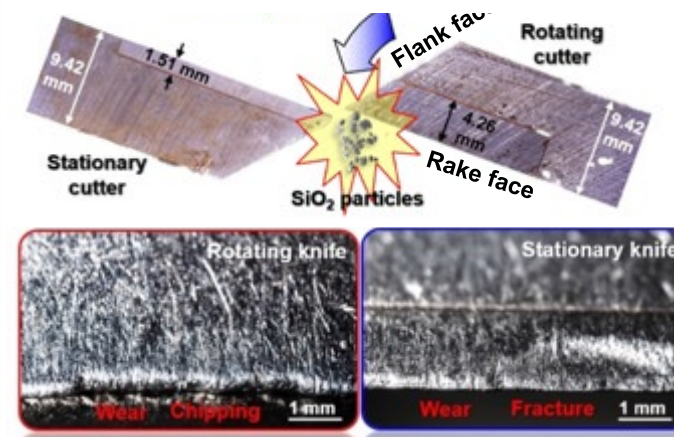
Knowledge



- A good portion of results from the hammer mill work have been transferred to the knife mill study:
  - Extrinsic and intrinsic inorganic species, mineral particle size & shape distributions,
  - Erosive wear model,
  - Feedstock modifications for ash reduction, and
  - Candidate alloys' wear resistance to 2-body abrasion.
- Tribosystem analysis determined different wear mechanisms for knife mill

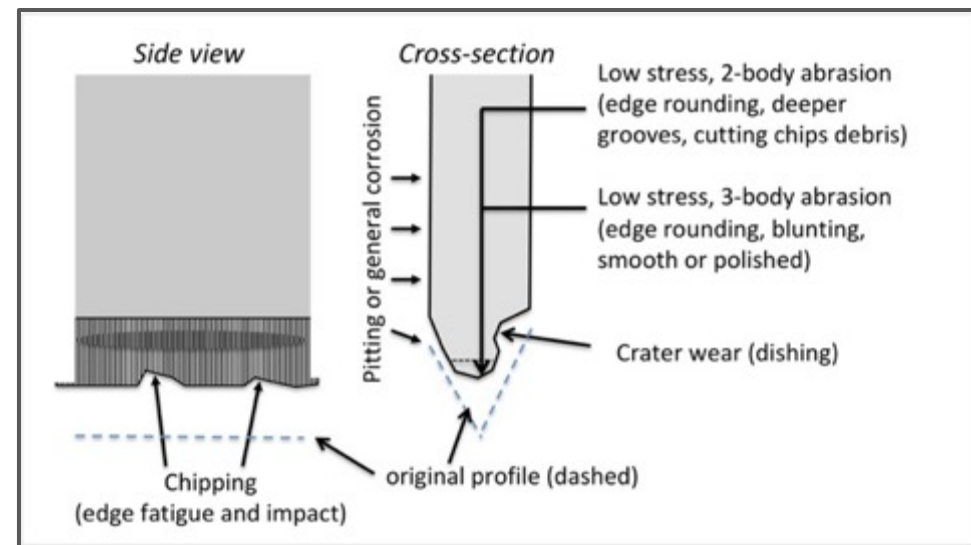


**Knife Mill** (Thomas Wiley Mill Model 4)



*Hammer Mill:* Blunt blades  
 Comminution mode: Crushing  
 Wear modes: Erosive wear (dominant) + Abrasive wear (secondary)

*Knife Mill:* sharp blades  
 Comminution modes: Cutting + Crushing  
 Wear modes: 2-body/3-body Abrasive wear + Erosive wear (both important)



## Knife mill: abrasive wear + erosive wear (both important)

### Technical progress:

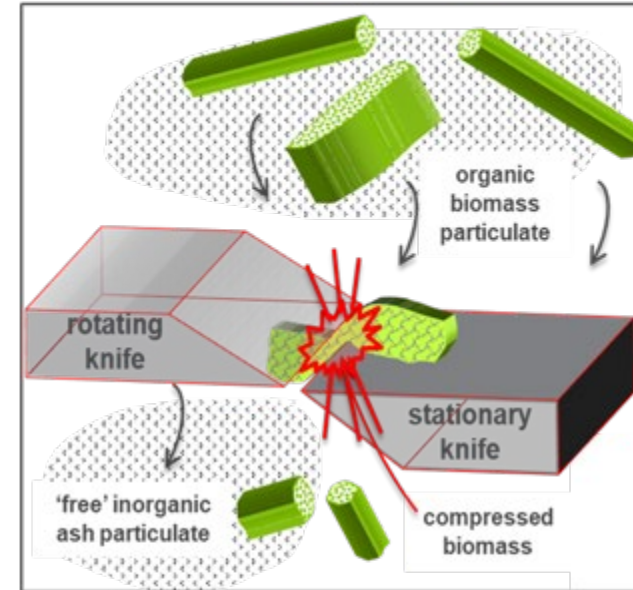
- Guided by literature and experimental characterization of knife blades, **physical models** and mathematical models are being developed to analytically predict wear (edge recession) of blades as functions of feedstock properties, knife blade properties, and milling parameters
- Erosive** and **abrasive** wear mechanisms both are present depending on blade speed and face.

#### Feedstock Attributes

- PSD & distribution of organic chips & fragments
- Ash content
- Inorganic ash PSD, shape, & distribution
- Intrinsic ash PSD, shape & density
- Density of mineral (e.g. SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>,...)
- Moisture

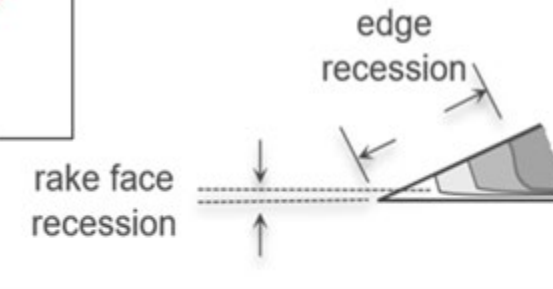
#### Knife Attributes

- Hardness
- Elastic modulus
- Fracture toughness
- Fatigue ductility, elongation
- Tensile Strength



#### Quality Attributes

- Material wear rate
- Knife shape rake and edge recession
- Product quality (size & shape)



#### Process Parameters

- Geometry/configuration/volume
- Knife geometry/design
- Rotational speed, velocity, impact speed
- Feed rate
- Residence time
- Ash content

*QbD approach under development to analytically model mechanical wear of knives used to mill biomass.*

$$\Delta V_{Knife} = \Delta V_{erosion} + \Delta V_{abrasion} + Syn$$



# Selection of candidate coatings & surface treatments to mitigate wear

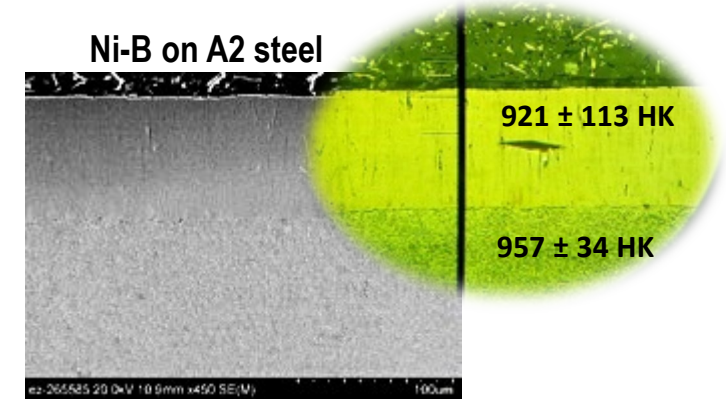
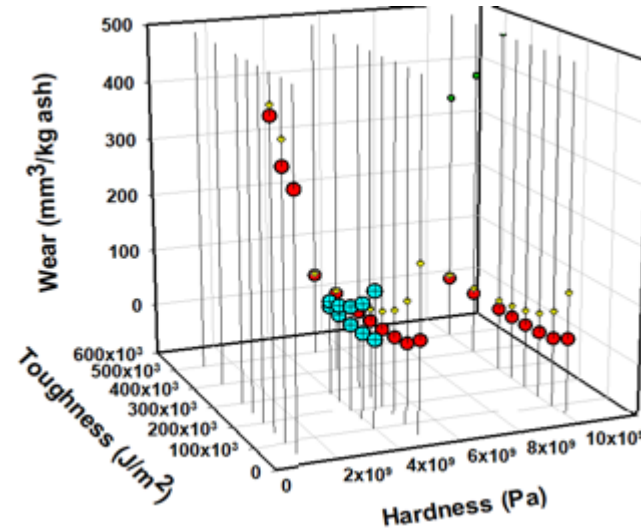


Knowledge

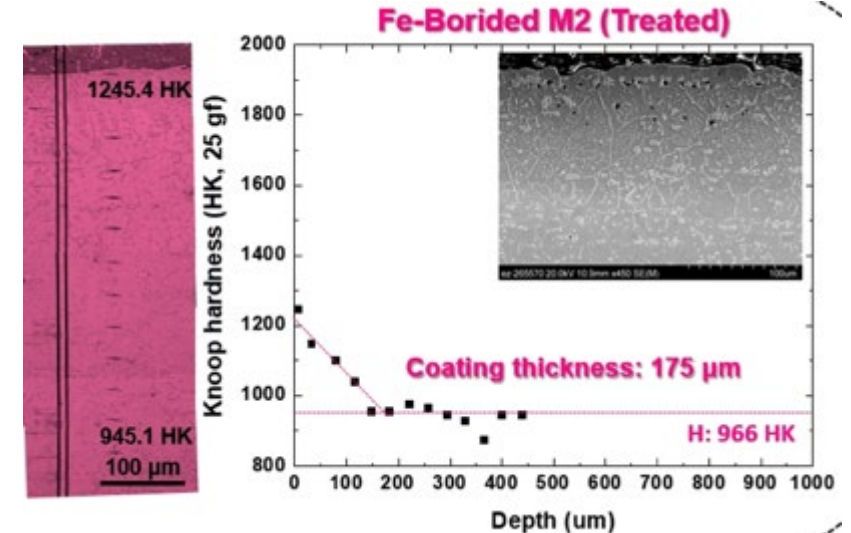


- Abrasive and Erosive wear can be mitigated by selecting tool materials with optimum mechanical properties
  - Increasing hardness – lowers abrasive wear
  - Increasing fracture toughness and fatigue ductility – reduces erosive wear

– Achieving all three attributes is a challenge and requires innovative material solutions.



	Nickel Boriding (coating)	Iron Boriding (case hardening)	Thick diamond-like carbon (DLC) coating
Hardness	Up to 1200 HV	1200-1900 HV	1000-2800 HV
Thickness	Up to 100 μm	Up to 300 μm	Up to 100 μm
Microstructure	columnar	columnar	amorphous
Process [Manufacturer]	Autocatalytic deposition (EXO) [UCT]	Deep case boriding (DCB) [IBC]	Plasma-enhanced chemical vapor deposition (PECVD) [C4E]
Deposition Temperature	RT followed by crystallization at 385 °C	1000+ °C followed by heat treat/tempering	< 300 °C



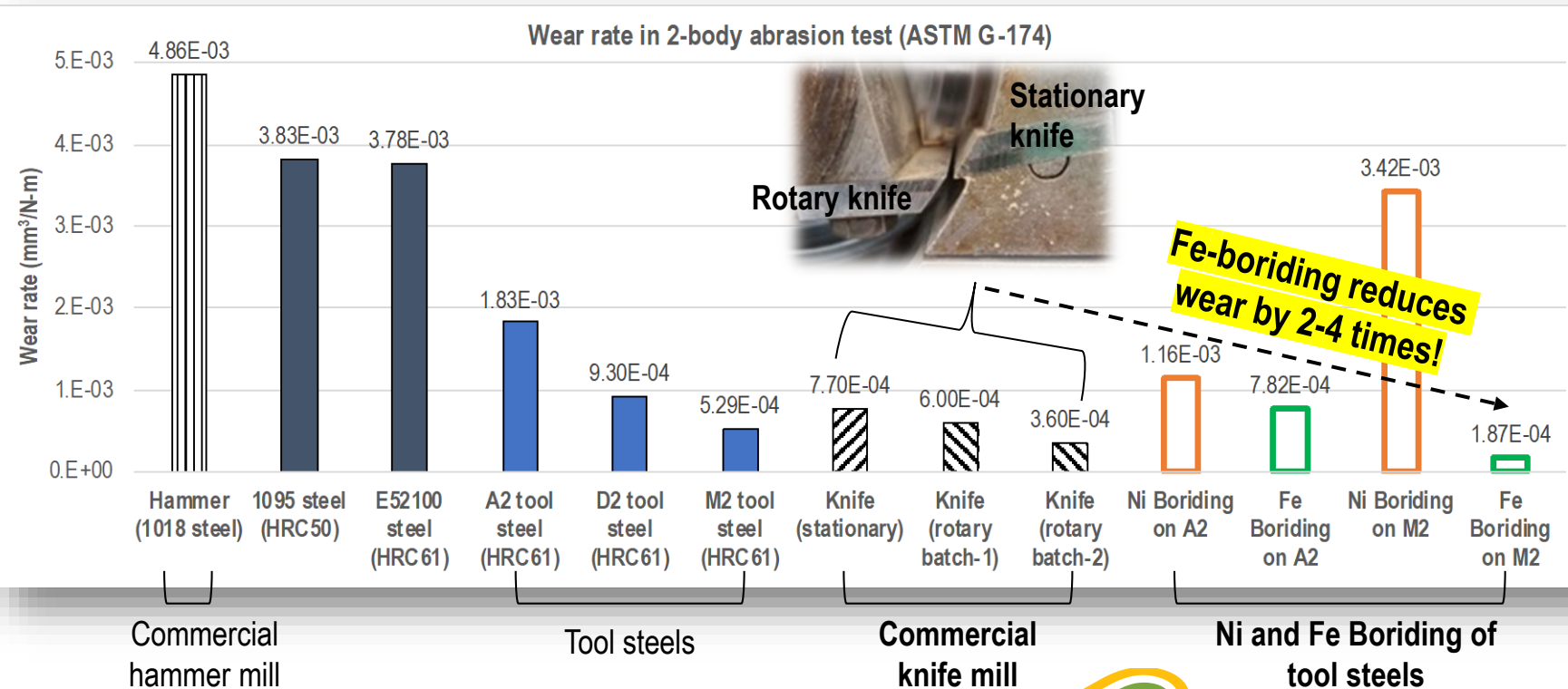
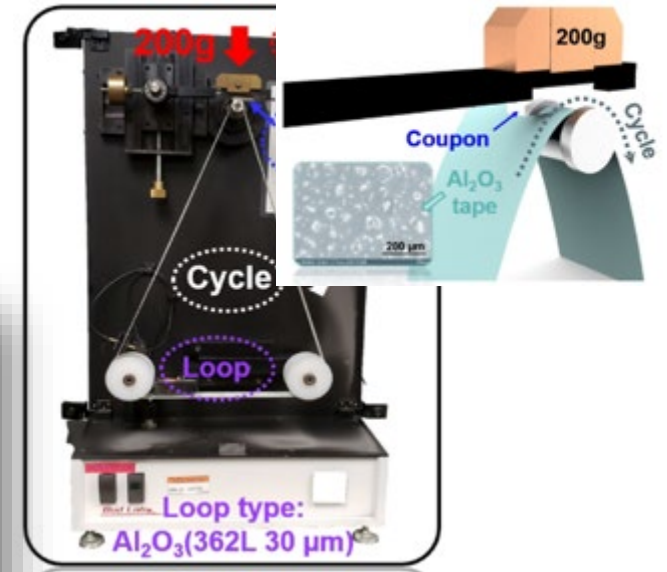
# Improve tool life for knife mill using more wear-resistant surface treatments



Knowledge



- Knife mill: 2-body/3-body abrasive wear + erosive wear (both important)
  - A candidate surface treatment showed significantly 2-4X improved wear resistance to abrasion in an ASTM standard 2-body abrasion test at ORNL
  - Their wear resistance to erosion is being evaluated using a blasting wear test at INL
  - More candidate coatings and surface treatments are being acquired and evaluated



Thomas Wiley Mill Model 4	Cost	Cost increase
Current tool steel knife set (10)	\$2,150	Baseline
<b>Fe-boriding a knife set (10)</b>	<b>\$120*</b>	<b>~7%</b>

\*\$12 per knife for a batch of 500 knives



**Management:** Collaboration of ORNL, ANL, and INL for joint experimental (Subtask 1.1) and modeling (Subtask 1.3) efforts.

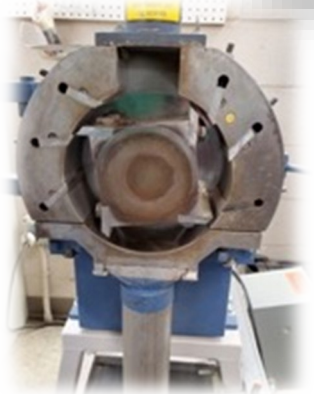
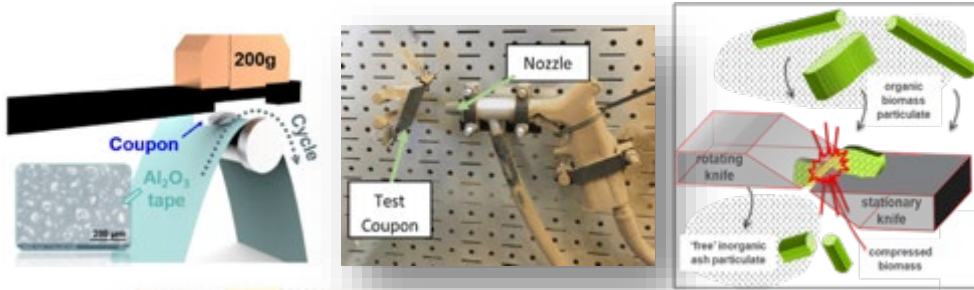
**Technical Approach:** Use integrated efforts of characterization, modeling, and testing to gain fundamental understanding of the failure modes and wear mechanisms of biomass preprocessing tools, develop candidate mitigations, and share findings with the biomass community.

**Impact:** Provides fundamental understanding for tool wear in biomass preprocessing, and develops cost-effective mitigations for improving the tool life, throughput, and particle size uniformity.

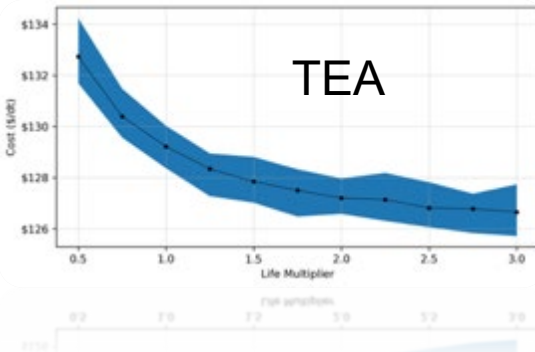
## Progress:

- Fundamental understanding of the **wear mechanisms of both hammer mill and knife mill** [complete]
- Protocols developed for **extraction and characterization of both extrinsic and intrinsic inorganics** [complete]
- **Analytical erosive wear model** for predicting wear of hammer mill [complete]
- **Low-cost feedstock modifications** developed for improving tool life [complete]
- Evaluation of **candidate tool coatings and surface treatments** using bench-scale abrasion and erosion tests [on-going]
- **Analytical abrasive wear model** to predict the impact of feedstock and tool material properties on knife mill durability and performance [on-going]

# Work plan for FY21 Q2-Q4



Small knife mill tests with feedstocks containing controlled, high content of extrinsic minerals to allow accelerated wear



Q2
Materials selection

- Continue **bench-scale tribo-testing** of candidate materials [Milestone 1]
- **Combined erosive and abrasive wear model** [Milestone 2]

Q3
Prototyping & testing

- **Prototype knives** to be using top candidate material(s) identified by bench-scale tribo-testing and wear modeling in Q2
- **Small knife mill testing** (Thomas Wiley Mill Model 4) to demonstrate >3X improved tool life [Milestone 3]

Q4
TEA

- **TEA** (in collaboration with Task 8) for potentially 2X or higher economics for knife mill OPEX [Milestone 4]
- **Share** fundamentals and mitigations with the biomass industry



# Quad Chart Overview

## FCIC, Task 1 Materials of Construction

### Timeline

- 10/1/2018 - 9/30/2021

	FY20	Active Project
<b>DOE Funding</b>	\$472K	FY19- \$1,064K FY20- \$472K FY21- \$472K Total- \$2,008K

Project Partners (N/A)

### Barriers addressed

- 19Ft-J, FSL, Operational Reliability
- 19Ct-B, CONV, Efficient Preprocessing and Pretreatment
- 19ADO-H, ADO, Materials Compatibility, and Equipment Design and Optimization

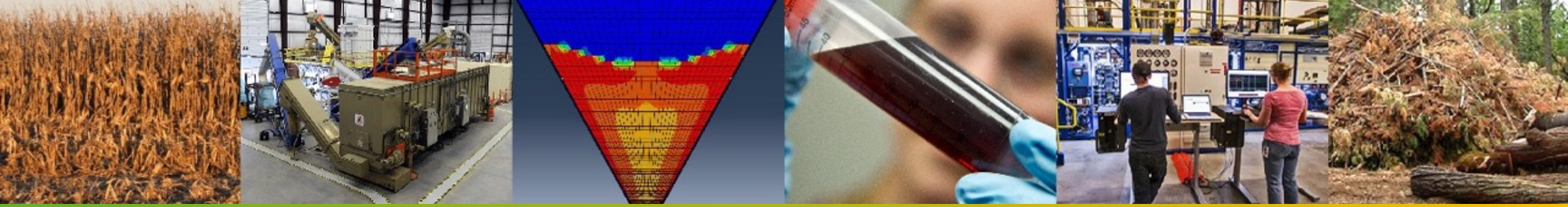
### Project Goal:

Using a systematic quality-by-design approach with integrated efforts of characterization, modeling, and testing to gain fundamental understanding of the failure modes and wear mechanisms of biomass preprocessing/preconversion equipment, develop analytical tools/models to predict wear and establish material property specifications, select and evaluate candidate mitigations based on modeling and lab-scale testing and identify top-performing mitigation for PDU validation, and share the fundamentals and mitigations with the biomass industry.

### End of Project Milestone:

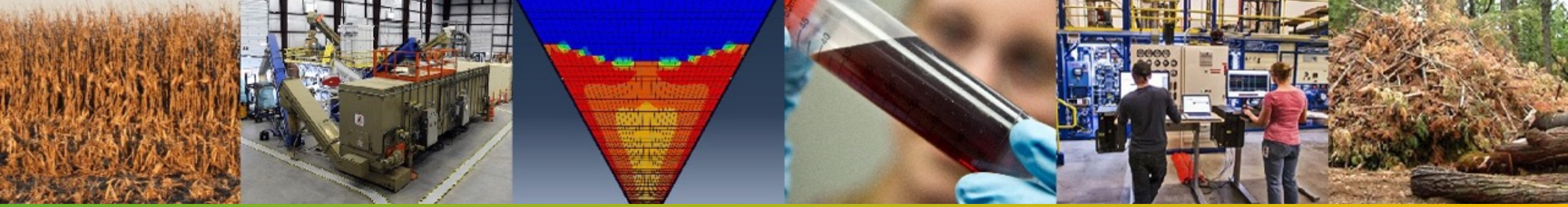
Deliver a TEA (in collaboration with Task 8) based on bench-scale and small knife mill wear testing as well as modeling for potentially 2X better economics for knife mill OPEX

Funding Mechanism (N/A)



*Thank you*  
**energy.gov/fcic**





## *Additional Slides*

# FY20-21 Task 1 Milestones

Milestone	Lead Lab	Milestone Description (Original)	Milestone Description (Revised)	Status
FY20 Q1	INL	Determine wear contributions from anatomical fractions of biomass and extrinsic ash.		<b>Complete – Milestone report submitted</b>
FY20 Q2	ANL	Experimental validation of particle erosion wear model against INL corn blaster wear data and ORNL characterization data.		<b>Complete – Milestone report submitted</b>
FY20 Q3 (GNG)	ORNL, ANL, INL	Down-select candidate wear mitigation strategies using bench tribo-tests, materials characterization, and wear modeling. Go: Developed effective mitigation strategies by using feedstock modifications and advanced tool materials. No-go: No improvement by any proposed mitigation strategy		[Delayed to FY20 Q4 due to pandemic] <b>Complete – Milestone report submitted</b>
FY20 Q3	ANL	<del>Workable predictive model of particle impact incorporating material and particle (ash) properties, speed, and impingement angle – validation against stage 1 hammer wear.</del>	Evaluate CPPs for knife mills and alternative bale deconstruction strategies (e.g. shredders, hybrid designs).	[Delayed to FY21 Q1 due to pandemic] <b>Complete – Milestone report submitted</b>
FY20 Q4	ORNL	<del>Identify the best combination of ash-reducing method and material/surface treatment and demonstrate at least 50% reduced wear rate in bench-scale wear tests.</del>	Determine the wear mechanisms, understand the baseline materials, and propose mitigation approaches for knife mills that include biomass ash reduction methods, milling control options, and blade material selection.	<b>Complete – Milestone report submitted</b>
FY21 Q1	ORNL		Down select candidate alloys and surface treatments by bench-scale erosion (blasting) and abrasion (2-body) accelerated wear testing in comparison with the baseline knife alloys.	[Delayed to FY21 Q2 due to pandemic]
FY21 Q2	ANL		Use erosion wear model and bench-scale testing results to define mechanical property operating envelope and tradeoffs for alloys and surface treatments used in construction of knives for industry.	
FY21 Q3	INL		Produce prototype knives using the top-performing candidate alloy and/or surface treatment and demonstrate at least 3X better tool life on a small knife mill at INL (Thomas Wiley Mill Model 4).	
FY21 Q4	ORNL		Deliver a TEA (in collaboration with Task 8) based on bench-scale and as well as modeling for potentially 2X or higher economics for knife mill OPEX.	



# Publications, Patents, Presentations, Awards, and Commercialization

## Publications:

- S. Roy, K. Lee, J.A. Lacey, V.S. Thompson, J.R. Keiser, J. Qu, “Material characterization-based wear mechanism investigation for biomass hammer mills,” *ACS Sustainable Chemistry & Engineering* 8 (2020) 3541–3546.
- K. Lee, S. Roy, E. Cakmak, J.A. Lacey, T.R. Watkins, H.M. Meyer, V.S. Thompson, J.R. Keiser, J. Qu, “Composition-preserving extraction and characterization of biomass extrinsic and intrinsic inorganic compounds,” *ACS Sustainable Chemistry & Engineering* 8 (2020) 1599–1610.
- O. Oyediji, P. Gitman, J. Qu, E. Webb, “Understanding the impact of lignocellulosic biomass variability on size reduction process: a review,” *ACS Sustainable Chemistry & Engineering* 8 (2020) 2327–2343.
- J. A. Lacey, J. E. Aston, S. Hernandez, V. S. Thompson, M. S. Intwan, K. Lee, J. Qu, “Wear and Why? How Ash Elements Can Help Define Wear Profiles of Biomass Feedstocks,” DOI: 10.13031/aim.201901446, *Proceedings of 2019 ASABE Annual International Meeting*, Boston, MA, July 7–10, 2019.

## Reports:

- George Fenske and Oyelayo Ajayi, An Analytical Model of Erosive Wear of BioMass Comminution Components, Argonne National Laboratory report # ANL/AMD-20/1, October 2020
- George Fenske and Oyelayo Ajayi, Application of an Erosion Wear Model to Predict Wear of Hammer Milling Components, Argonne National Laboratory, Argonne report # ANL/AMD-20/2, September 2020.
- George Fenske and Oyelayo Ajayi, Identification of Critical Process Parameters for Knife Milling and Alternative Comminution Strategies, Argonne National Laboratory, Argonne report # ANL/AMD-20/3, December 2020 (to be released)

## Presentations:

- K Lee, J Keiser, V Thompson, E Kuhn, E Wolfrum, J Qu, “Investigation of Equipment Wear Issues in Biomass Pre-Processing and Pre-Treatment,” *TMS 2019 Annual Meeting*, San Antonio, Mar. 10, 2019.
- K Lee, J Qu, E Kuhn, J Keiser, E Wolfrum, “Ash-induced wear on biomass pre-conversion equipment,” *74<sup>th</sup> STLE Annual Meeting*, Nashville, May 19, 2019.
- K. Lee, S. Roy, E. Cakmak, J.A. Lacey, H.M. Meyer, V.S. Thompson, J.R. Keiser, J. Qu, “Composition and abrasiveness of biomass extrinsic and intrinsic inorganic compounds,” *TMS 2020 Annual Meeting*, Feb. 23-27, 2020, San Diego.

## Awards:

- K Lee, J Qu, E Kuhn, J Keiser, E Wolfrum, “Ash-induced wear on biomass pre-conversion equipment,” 74<sup>th</sup> STLE Annual Meeting & Exhibition, Nashville, May 19, 2019. **Best Poster Award 3<sup>rd</sup> place**
- Jun Qu – **2020 Distinguished Researcher Award**, Recognition of sustained and distinguished accomplishments with high impact in science and engineering, ORNL/UT-Battelle Awards Night

## Media Report

- “ORNL’s latest on bio-oils corrosion and degradation: The Digest’s 2020 multi-slide guide to Oak Ridge National Laboratory,” *Biofuels Digest*, Jan. 10, 2021.