# DEVELOPMENT OF LOW-COST RARE EARTH ELEMENT ANALYSIS AND SORTING METHODS

Submitted to:

Ms. Karlene Fine North Dakota Industrial Commission Lignite Research Program State Capitol 600 East Boulevard Avenue, Department 405 Bismarck, ND 58505-0840

Funding Requested: \$224,767

Submitted by:

Ms. Shuchita Patwardhan Project Manager Microbeam Technologies Inc. 4200 James Ray Drive, Ste 193 Grand Forks, ND 58202

July 1, 2017

## TABLE OF CONTENTS

1	AB	STRACT1								
2	PRO	DJECT SUMMARY								
	2.1	The Solution and Technical Approach								
3	PRO	DJECT DESCRIPTION								
	3.1	Overall Project Goal7								
	3.2	Project Objectives								
	3.3	Scope of Work								
	3.4	Anticipated Results								
	3.5	Facilities and Resources								
	3.6	Environmental and Economic Impacts during Project1								
	3.7	Technical and Economic Impacts of Proposed Technology								
	3.8	Project Need14								
4	STA	ANDARDS OF SUCCESS								
5	BA	CKGROUND INFORMATION15								
	5.1	Introduction15								
	5.2	Coal and Coal Byproducts – Alternative REE Resources								
	5.2.	1 REE Associations in Coal								
	5.2.	2 Evaluation of Coal and Coal Byproducts as an REE Resource								
	5.2.	3 REE Content in North Dakota Lignite Coal20								

5	5.3 Me	easuring Inorganic Composition of Coal and Abundance and Association of REE	
	5.3.1	Inductively Coupled Plasma – Mass Spectroscopy (ICP-MS)	
	5.3.2	X-ray Fluorescence (XRF)	
	5.3.3	Scanning Electron Microscopy with Energy Dispersive X-ray Spectrometry (SEM-H	EDS)25
	5.3.4	Chemical Fractionation	
	5.3.5	Prompt Gamma Neutron Activation Analysis	
	5.3.6	Coal Properties and Correlations for Coal Sorting and Blending	
5	5.4 Sur	mmary of the Research Motivation	27
6	QUALI	FICATIONS	
7	VALUE	E TO NORTH DAKOTA	
8	MANAO	GEMENT	
9	TIMETA	ABLE	
10	BUDO	GET	
11	MAT	CHING FUNDS	
12	TAX	LIABILITY	
13	CONI	FIDENTIAL INFORMATION	
14	APPE	ENDICES	

#### 1 ABSTRACT

Rare earth elements (REE) consist of the lanthanide series of elements and are crucial materials in an incredible array of electronic devices, energy system components and military defense applications. However, the global production and entire value chain for REEs is dominated by China, with the U.S. being 100% import reliant for these critical materials. Traditional REE mineral ores, however, have several challenges. Chief among these is that the content of the most critical and valuable of the REEs are deficient, making mining uneconomical. Further, the supply of these most critical REEs is nearly 100% produced in China from a single resource that is only projected to last another 10 to 20 years. The U.S. currently considers the REEs market a national security issue, given the supply risks and the importance of their end uses to both energy applications and military defense applications. Therefore, given the complete import reliance and dwindling Chinese reserves, it is imperative that alternative domestic sources of REEs be identified and methods developed to produce them. Recently, coal and coal byproducts have been identified as one of these promising alternative resources. This project targets the development of advanced sensor technology combined with novel predictive algorithms to enable selective mining and sorting of high rare earth element (REE) content coal and coal-related feedstocks. This project will determine the feasibility of the integrated application of portable x-ray fluorescence (pXRF) analyzers, gamma ray core log analysis, REE 'fingerprinting' algorithms, and prompt gamma neutron activation analysis (PGNAA) sensors to identify REE-rich layers in coal seams using a simple, fast and field deployable method integrated with selective mining and real-time on-belt coal sorting using PGNAA. This approach overcomes the challenge of dilution of REE-rich coal with coals of lower REE content during the mining process. The duration of the project will be two years. The total project cost is expected to be \$449,534 that includes \$168,767 of in-kind cost share, \$56,000 in cash cost share from project sponsors currently being identified, and \$224,767 from NDIC. The project participants include: Energy Technologies Inc., University of North Dakota, and Microbeam Technologies Inc. Microbeam will work closely with the North Dakota Geological Survey in field sampling and selection of samples for analysis.

#### 2 PROJECT SUMMARY

In recent years, due to their recognition as crucial materials for a huge variety of important end use applications such as military defense, wind turbines, hybrid/electric vehicles, electronics and many others, the secure supply of rare earth elements (REE) and REE-based products is considered an issue of national security. China, in part due to its deposits of a unique REE resource (ion-adsorbed clays) that combines high quantities of the more valuable heavy REE, as well as simple and low cost extraction, dominates the global supply market. In 2010, China established new quotas on exports of REEs, which resulted in huge increases in REE prices peaking in 2011 due to an expected supply shortage for critical applications such as in permanent magnets used in wind turbines and motors in hybrid/electric vehicles. As a result, production at the California-based Mountain Pass Mine (Molycorp, Inc.) was re-started after several years of dormancy. However, after peaking in price in 2011, prices have dropped substantially to slightly above 2010 levels, challenging the profitability of non-China based production which consists mainly of hard rock carbonatite deposits that are deficient in the more valuable heavy REE. Some researchers have noted that *mining of the Mountain Pass and similar resources will neither mitigate the crisis in REE resources nor eliminate the shortage of the most critical REE, but will only result in overproduction of excessive cerium.* 

According to the USGS 2017 Mineral Commodity Summary report, China accounted for about 83% of the total global REE supply in 2016, down from about 95% prior to 2010. Meanwhile, the US production was zero, with the Mountain Pass mine having declared bankruptcy and closing operations in the last quarter of 2015. <u>The U.S. is currently 100% import reliant for REEs</u>. Although still dominating global supply of the heavy REE, researchers have estimated that the Chinese ion-adsorbed clays resource will only last another 10-20 years. The Chinese clays represents essentially the entire global supply of the most critical of the REEs. Due to its limited supply, and because the Chinese clay resource is rich in the most critical REE, while most other traditional resources are deficient in these less common and more valuable elements, it is imperative that new domestic sources of REEs, especially the most critical REE, be

identified and processes be developed to produce them. Coal and coal byproducts have recently been identified as one of these potential new resources for REEs. This proposed work is aimed at development of a novel technology to separate and concentrate REEs in North Dakota lignite coal and lignite-related materials.

However, there are some specific challenges regarding coal and coal byproducts as REE resources. Besides the technical challenges associated with REE recovery from coal, there are challenges with mining as well. An important finding from ongoing work by UND looking at evaluating the feasibility of recovering REE from North Dakota lignite and lignite-related feedstocks, is that the REE content is highest in certain locations within seams and in certain seams. For instance, in the Falkirk Mine, the feed coal to the Coal Creek Station contains a blend of Hagel A and B coals (majority lower REE Hagel A), which results in a dilution of the REE content in the combustion fly ash (~240 ppm vs. 400-600 ppm in Hagel B ash). *To maximize concentration of REE, selective mining will be needed, a prospect that may not be feasible at large power plants. To aid in selective mining and sorting procedures, it would be desirable to develop expedient, low-cost and reliable analysis techniques that are field/mine-deployable to identify, separate and stockpile the high REE content material for processing.* Standard methods of REE measurement are both time and cost intensive and are not compatible with field or mine settings.

#### 2.1 The Solution and Technical Approach

This project targets the development of advanced sensor technology combined with novel predictive algorithms to enable low-cost and accurate selective mining and sorting of high rare earth element (REE) content coal and coal-related feedstocks. This project will determine the feasibility of the integrated application of portable x-ray fluorescence (pXRF) analyzers, gamma ray core log analysis, predictive REE 'fingerprinting' algorithms, and prompt gamma neutron activation analysis (PGNAA) sensors to identify REE-rich layers in coal seams using a simple, fast and field deployable method integrated with selective mining and real-time on-belt coal sorting using PGNAA. This approach overcomes the challenge of dilution of REE-rich coal with coals of lower REE content during the mining process, as well as eliminates the need

for time and cost-intensive REE-specific analysis techniques that would not be feasible for field deployment.

Our novel approach is illustrated in Figure 1, and consists of the following integrated elements:

- Detailed understanding of the abundance and association of REE in the lignite or lignite-related materials that allows for genetic classifications such as those discussed later in Section 5.2.1.
- Novel REE-predictive algorithms derived from genetic classifications, enrichment mechanisms, and correlations developed via gamma ray log measurements as well as major/minor/trace element concentrations that are easily/accurately measured by pXRF. Direct measurement of REEs is not required.
- Enabling of selective mining practices through in-mine or field use of the pXRF and gamma ray core log analysis data combined with REE-predictive algorithms to accurately identify locations within the mine with the highest REE content.
- Integration of novel REE algorithms with on-belt PGNAA analysis to separate, sort and stockpile REErich coal and associated sediments.

The association of REE in the coals will be used to identify the elements that will provide a chemical signature or fingerprint indicating the presence of REE. The elements must be present in sufficiently high concentration to enable accurate and fast measurement by pXRF and PGNAA. Bryan and others (2015) [1] and work conducted by Microbeam/UND showed promise that pXRF can be used as a screening tool to identify REE-rich coals. In this project we plan to use the pXRF not only as a screening tool, but more importantly as a tool to reliably identify areas within the mine/seam with the highest REE content that can be selectively mined.

Another method that has shown promise for indirect measurement of REE in a field or mine setting is use of gamma ray well logging or core analysis. Uranium and thorium will provide positive gamma ray signals, and due to their chemical similarity to REEs (actinides are after lanthanides in the periodic table), uranium and thorium are often found in elevated concentrations in REE-enriched coals and coal sediments.

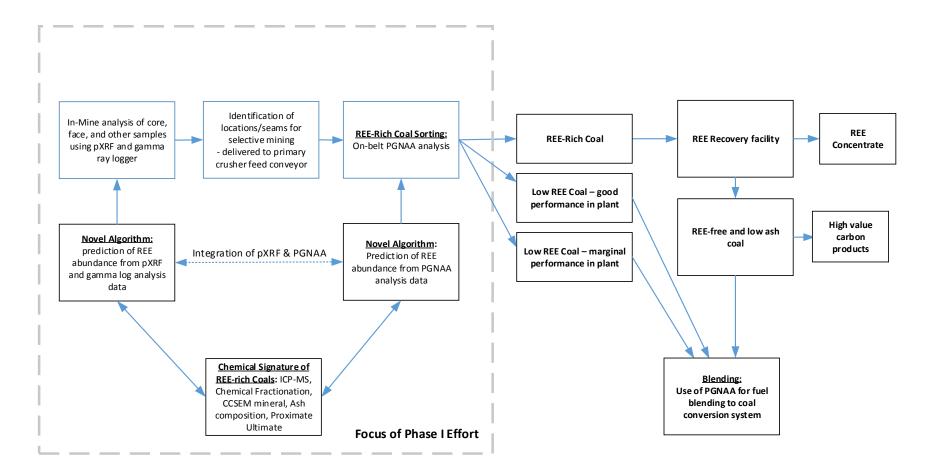


Figure 1. Overall conceptual diagram of the proposed approach to enable low-cost selective mining and sorting of REE-rich coal feedstocks.

Dai and Finkelman (2017) [2] state that positive natural gamma ray anomalies in well or core logging should be considered for Nb-Zr-REE-Ga deposits, as these are often associated with intra-seam alkali tonstein layers or alkali tuffs in coal-bearing sequences. Further, southwestern North Dakota is host to uraniferous lignites, which were actually mined specifically for uranium production in the 1960s and 1970s. The areas highlighted in Figure 2 denote areas with significant positive gamma ray signals, representing known areas of high uranium content lignites within 200 feet of the surface. In ongoing sampling efforts by the North Dakota Geological Survey (NDGS), these same areas have been shown to contain exceptionally high REE content as well [3, 4, 5]. The NDGS targeted the southwestern portion of the state specifically for the known uranium/REE relationship (Ed Murphy, NDGS, personal communication June 2017). In this project, we plan to evaluate the use of a portable gamma ray core logger to analyze drill core samples and quantify the relationship between gamma ray signal and REE content. Similarly to the pXRF, the gamma ray core log analysis can be used as a screening tool for elimination/selection of samples for additional characterization, but we also aim to develop robust correlations that can be used to accurately identify high REE stratigraphic layers in the cores without direct REE measurement.

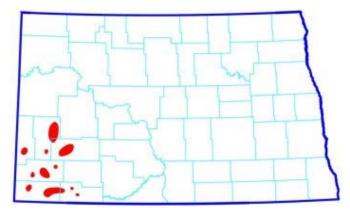


Figure 2. Known areas of uranium occurrence within 200 feet of the surface in western North Dakota. This information was plotted from positive gamma logs on file with the NDGS [6].

To further enable coal and associated sediments to be economic REE resources, we have incorporated a sorting analyzer based on PGNAA analysis that is calibrated/tuned to match the pXRFderived REE-predictive fingerprinting algorithms. Using REE thresholds established through synchronizing the pXRF and PGNAA analysis results, the PGNAA provides fast, real-time analysis that can be used to separate run-of-mine from REE-rich coals. Microbeam and ETI have extensive experience in implementing a similar algorithm-based coal sorting and blending method using a PGNAA [7]

The overall impact of our novel approach is reduced cost and increased efficiency/reliability of REE selective mining, enabling of simple, real-time process sorting of coals based on REE content thresholds, and thus improvement of the economics of recovery of REEs from coal and coal byproducts.

#### **3 PROJECT DESCRIPTION**

#### 3.1 Overall Project Goal

The overall goal of this project is to develop novel REE fingerprinting algorithms via pXRF and gamma ray analysis that will enable low-cost field/mine identification of high REE-content coals and associated sediments, combined with selective mining and on-belt sorting or REE-rich from run-of-mine coals.

#### 3.2 **Project Objectives**

To achieve the above project goal, specific project objectives have been identified as follows:

- Survey the ability of pXRF to perform analysis of a range of REE containing coals in order to identify key elements that can be quantified
- Validate pXRF measurement with standard laboratory XRF to calibrate the results
- Develop novel correlations between elements quantifiable by pXRF in coal and the abundance and association of REEs
- Incorporate REE fingerprinting correlations into pXRF analysis scheme and test on known samples
- Use gamma ray core log analysis to quantify the correlation with REE content
- Determine the ability of PGNAA to detect pXRF key elements and ability to utilize pXRF-derived REE correlations
- Perform proof-of-concept sorting tests with the calibrated PGNAA
- Develop the conceptual strategy for integrated commercial use of pXRF/gamma log/PGNAA/REEpredictive algorithms to enable selective mining and sorting of REE-rich feedstocks.

#### 3.3 Scope of Work

The technical approach in the Phase I project is to collect and analyze a range of ND lignite coals to establish a database with a wide range of chemical composition so that we may effectively develop novel algorithms that can identify REE 'markers' without direct measurement of the REEs. Initial analysis of about 100 samples will be conducted by pXRF in a controlled laboratory setting. Based on the pXRF results, about 20 samples covering the widest range of composition will be selected for additional analysis, including standard laboratory XRF ash composition (as control for pXRF), proximate/ultimate, CCSEM, chemical fractionation and REE determination by ICP-MS. The combined data will be utilized with the pXRF data to establish novel algorithms that can accurately predict the REE content of ND lignite coal without direct measurement of the REEs. We will also evaluate the relationship between gamma ray core log signal and REE content by evaluating about 10 drill core samples provided by industrial sponsors. Based on the results of the analysis, from each core sample, we will select up to about five specific stratigraphic layers (representing markedly different gamma log signals) and will be further analyzed by pXRF or inductively coupled plasma – mass spectroscopy (ICP-MS).

Based on the combined results, up to five larger samples (~200 lbs) will be collected from industrial sponsor properties and analyzed via pXRF and ICP-MS. Static measurements with PGNAA will be made with each of the known compositional splits, and results calibrated to match the pXRF-derived REE-predictive algorithms. Once calibrated, proof-of-concept sorting testing will be performed to determine performance of the PGNAA to separate coal into REE content splits. The combined results of the project will be utilized with the support of industrial sponsors to develop the preliminary conceptual strategy for commercial integration of our selective mining/sorting methodology within the existing mine infrastructure/procedures.

Specific tasks to be completed in the proposed project are detailed in the following sections.

#### Task 1 – Identification and Procurement of Samples

**Work to be Performed**: We will identify and procure about 100 split samples (~1-2 kg) for subsequent analysis in Task 2. A combination of samples from the existing UND sample library, as well as drill core samples and samples collected from exposed seams at the industrial sponsors' properties will be included. This effort will not include collection of run-of-mine coal samples, but rather will focus on specific stratigraphic layers that have the potential to contain elevated REE levels. This task will be performed jointly by Microbeam and UND. <u>Methodology</u>: We will collect samples that cover a wide range of chemical composition. To ensure this occurs, we will utilize the existing analysis by UND and industry sponsors as well as the CoalQual Database and ND Geological Survey data (as available) to identify samples for collection. We will work with industry sponsors to collect samples during drilling activities and leverage their analysis when possible.

#### Task 2 – Survey Analysis

Work to be Performed: The collected samples will be analyzed at Microbeam, UND and an external laboratory, with Microbeam leading the data analysis efforts. Methodology: All samples collected (~100), with the exception of drill core samples to be evaluated by gamma ray log analysis, will be ground to uniform size (-1/4 inch) and analyzed via UND's existing pXRF instrument in a controlled laboratory setting with constant optimized measurement parameters (measurement time, measurement angle, pXRF operating conditions). X-ray spectra will be used to identify and quantify inorganic elements. With the pXRF data, approximately 20 samples will be selected covering the widest range possible of chemical composition. These samples will be exposed to more detailed analysis including proximate/ultimate (external lab), laboratory XRF ash composition (external lab), computer controlled scanning electron microscopy (CCSEM) (Microbeam) and chemical fractionation (Microbeam), as well as ICP-MS (UND) to determine REE content and modes of occurrence. The pXRF data will be compared to the laboratory XRF data for calibration purposes. Also in this task, up to ten selected drill core samples will be analyzed via UND's

existing gamma ray core logger instrument. Based on the gamma log results, for each of the core samples up to five specific stratigraphic layers will be further evaluated by pXRF or ICP-MS.

#### Task 3 – REE-Predictive Algorithm Development

Work to be Performed: With the data generated in Task 2, we will develop novel REE fingerprinting algorithms to predict REE content in ND lignite coal based on the bulk chemistry of the coal and the gamma ray log data. Microbeam will perform the work in this task, with assistance as necessary from UND. Methodology: Our preliminary analysis of the UND sample database has shown favorable correlations for elements such as Zn, Zr and Ti. We expect that upon collection of a larger range of REE levels in coals that better correlations can be revealed. The modeling will employ linear and non-linear curve fits, multi-variate analysis, and cluster analysis. Specialized parameters such as ratios or combined parameters may be developed which better describe relationships. We will investigate total REE content and individual REE and groupings of REE (light, mid and heavy). We will utilize known associations between REE and select minerals to generate specialized parameters to assess correlations. Statistical analysis will determine robustness of relationships. Advanced algorithms using custom-built software may be developed as needed. Microbeam has access to Microsoft Excel, Tableau, and SPSS software and has customized software development capabilities to aid in the modeling process. We will also explore data normalization techniques such as that used by Seredin and Dai (2012) [8] in which elemental 'enrichment markers' will be correlated to enrichment of REE via normalization of the coal composition to the composition of the upper continental crust.

#### **Task 4 – Large Sample Collection**

**Work to be Performed**: Up to five larger samples will be collected (~200 lbs) to be used in PGNAA testing in Task 5. We will select large samples that each have markedly different pXRF spectra and REE content. Microbeam and UND will work with industry sponsors in this task to select locations for sampling, with Microbeam and UND jointly participating in the sampling activity. <u>Methodology</u>: Selection of these samples will be determined based on previously sampled areas within the mines and/or trace element

analysis available via industry sponsors or the CoalQual Database. The REE-predictive algorithms developed in Task 3 will also be used in the selection process to ensure samples represent a range of REE content. Once collected, samples will be ground to a uniform size and analyzed via pXRF to obtain the baseline spectra.

#### Task 5 – PGNAA Calibration and Verification Testing

Work to be Performed: This task will involve PGNAA testing of the five large samples collected in Task 4 in order to obtain analysis data to allow for identification of materials with high levels of REE. The primary aim of the PGNAA is to obtain for calibration to the pXRF data and the REE-predictive algorithms. The PGNAA analysis calculations will be adjusted as necessary to synchronize with the pXRF. Once calibrated, verification testing will be performed that will include semi-continuous measurement of the partially pre-mixed coal (simulating delivery of truck loads to a commercial conveyor belt system). Based on the combined results of the project, including the verification testing, we will develop the preliminary conceptual strategy for implementing our selective mining/sorting methodology at the commercial scale. ETI will lead the PGNAA testing, identification of key detectable elements, and calibration in this task using their existing analyzer/facility. Microbeam and UND will be responsible for sample and data analysis. Microbeam and ETI will work with project industry sponsors to formulate the commercialization strategy. Methodology: For verification testing, we will partially pre-mix the samples and feed them into the calibrated PGNAA system in a semi-continuous mode or reduced belt rate, with the goal of being able to effectively distinguish between the five coal compositions once feeding through the partially mixed fractions. In this testing, we will separate the coals based on the PGNAA analysis to simulate sorting. Subsequent ICP-MS analysis of each split will be used to determine sorting performance.

#### 3.4 Anticipated Results

The anticipated results of the proposed project are the establishment of novel REE fingerprinting algorithms based on low-cost and field/mine deployable pXRF analysis, which can be combined with selective mining procedures and real-time REE-rich coal sorting using PGNAA to separate and stockpile high REE content

coal for processing. Our methodology will eliminate the need for time and cost-intensive ICP-MS analysis as a screening method. This effort will develop preliminary data based on limited sampling, but will serve as the proof-of-concept and will be a foundation for subsequent, more detailed sampling and analysis that can improve the robustness and precision of the REE-predictive algorithms. The primary questions to be answered in this proposed work are:

- What elements or element combinations in data produced by pXRF serve as markers to predict the abundance of REE in coal and coal bypdroducts?
- Can gamma ray core logging be used to quantitatively predict the REE content of coal and coal byproducts?
- Can the pXRF/REE correlations be successfully applied to PGNAA, allowing real-time sorting of REE-rich coals?
- How can pXRF, gamma logging and PGNAA be combined to aid in selective mining and sorting of REE-rich coals on a commercial-scale?

#### 3.5 Facilities and Resources

A summary of the facilities and resources available to the project via the project team organizations are provided below. More details are contained in Appendix C attached with this application.

MTI has laboratory and office space located in the Center for Innovation at the University of North Dakota. Our automated scanning electron microscope is equipped with x-ray microanalysis capabilities. The laboratory has sample preparation equipment, a small-scale fluidized bed combustor, a small-scale gasifier simulator equipped with a syngas cooler, a high-temperature 1700 °C refractory testing furnace, an ash-fusion furnace, chemical-fractionation analysis equipment, and other laboratory equipment. The equipment is specifically designed and optimized to characterize coal and coal ash-related materials. In addition, MTI has developed numerous data analysis procedures designed to interpret the results of analysis of fuel and fuel-ash related materials for clients worldwide. These techniques are used to assist combustion and gasification facilities to improve reliability and decrease maintenance costs through fuel

selection/blending and optimized operating conditions. MTI has conducted over 1550 projects that involve the analysis of fuel, ash, slag, and metal materials.

The Materials Characterization Laboratory (MCL) at the UND Institute for Energy Studies has numerous analytical capabilities that will complement the MTI facilities and equipment. Specifically, this project will leverage the use of UND's existing pXRF instrument and gamma ray core logger instrument. The project will also use UND's ICP-MS instrument for measurement of the abundance of REEs.

ETI's office is equipped with a PGNAA unit designated for research and development of new techniques for analysis as well as software improvements incorporating these techniques. PGNAA technology is used in conjunction with ash meter technology to provide absolute ash, as a total weight percent, as well as mineral composition. PGNAA has the ability to analyze large numbers of samples, provide spectral/statistical data for every measurement period (3 second, 6 second, 10 second, 60 second, etc.) and can be modified to capture the existence of rare earth elements in coal.

#### **3.6 Environmental and Economic Impacts during Project**

The environmental impacts resulting from performance of the proposed work are negligible. Any waste materials produced as a part of testing will be disposed of via the existing mechanisms at Microbeam, UND and ETI. Economic impacts include employment opportunities for MTI, UND and ETI research staff, students and support staff. The project will train the next generation of engineers/scientists that will benefit the North Dakota labor force.

#### 3.7 Technical and Economic Impacts of Proposed Technology

Existing analysis methods to determine the abundance of REEs in coal and coal-related materials are both time and cost-intensive and are not compatible with field/mine deployment, and large-scale mining practices result in a run-of-mine coal that significantly lower in REE content than specific stratigraphic layers within the mine. The major impact of the proposed work is the development of advanced sensor technology and sorting methodology that will enable low-cost selective mining and sorting of high REE content coal with minimal impact to existing mining infrastructure/procedures. This work will enable use

of coal as an alternative domestic resource for REEs, limiting foreign dependence for critical end-use applications such as renewable energy components. New markets for coal will be established and the value of domestic coal resources will be increased.

#### 3.8 Project Need

With increasing environmental regulations on fossil-fuel based power and decreasing coal production nationwide, new opportunities for marketable use of lignite coal are required to maintain the existing mining/lignite use infrastructure in the State. The recovery of REEs from ND lignite and related materials has potential to be a significant new industry for the state that will both maintain existing jobs and create new jobs and revenue for the State. The results of ongoing work by UND evaluating REE recovery methods for lignite are extremely encouraging, and the unique properties of ND lignite coal make recovery of REEs a simpler and lower-cost proposition than either higher rank coals or traditional mineral ore-based resources. Additionally, the U.S. is currently 100% import reliant for REEs, and critical end-use applications in growing market sectors require a stable, domestic supply. Under the current conditions, extreme instability/uncertainty exists due to monopolistic control of the entire REE value chain by China. To ensure the national and economic security of the U.S., it is imperative that domestic sources of REEs be identified and processes developed to produce them. However, one of the key challenges associated with use of coal and coal byproducts as REE resources is that the REE content is not consistent along the stratigraphic column in a mine, and thus selective mining methods will be required to effectively target the REE-enriched zones, a prospect that may be economically challenging at large scales. At current, the standard method of measuring the abundance of REEs in coal is both time and cost-intensive and is not economically feasible for large-scale screening activities that would be needed to aid in selective mining. To solve this challenge, the proposed project will develop advanced sensor technology combined with novel algorithms to quickly, accurately and cheaply identify zones of REE-enriched coals or coal sediments. This can be combined with selective mining and novel sorting methods to separate and stockpile the high REEcontent lignites for use in REE processing with minimal impact to the mine operations. This project will

enable lower-cost mining and separation of high-REE content North Dakota lignite coal that will improve the economics of REE recovery.

#### 4 STANDARDS OF SUCCESS

The standards of success for the proposed project are summarized below:

- Provision of about 100 samples of REE-containing feedstocks with a sufficiently wide chemistry range to permit development of robust REE-predictive algorithms
- Development of statistically accurate REE-predictive algorithms based on pXRF data without direct measurement of the REEs.
- Demonstration of the ability of real-time REE-rich coal sorting via PGNAA calibrated using the pXRF/REE algorithms.

#### **5 BACKGROUND INFORMATION**

#### 5.1 Introduction

Rare earth elements (REEs) include a group of elements with atomic numbers from 57-71. This includes the elements lanthanum (La), cerium (Ce), praseodymium (Pr), neodymium (Nd), samarium (Sm), europium (Eu), gadolinium (Gd), terbium (Tb), dysprosium (Dy), holmium (Ho), erbium (Er), thulium (Tm), ytterbium (Yb), and lutetium (Lu). Yttrium (Y) and Scandium (Sc) are often included in the group because of their similar properties. The rare earth elements are classified as light (LREE) and heavy (HREE). The LREE include La to Eu. The HREE include Gd to Lu, and Y. Another grouping is critical REEs that are classified based on the combination of their supply scarcity and end use importance, and are typically considered to include Nd, Eu, Tb, Dy, Er and Y. According to the National Energy Technology Laboratory [8], REEs provide significant value to our national security, energy independence, environmental future, and economic growth. REEs are utilized in a suite of high importance end-uses, such as cell phones, hybrid vehicles, magnets, computer components, catalysts and many others.

China, in part due to its deposits of a unique REE resource (ion-adsorbed clays) that combines high quantities of HREE and critical REE, as well as simple and low cost extraction, dominates the global supply market. In 2010, China established new quotas on exports of REEs, which resulted in huge increases in REE prices peaking in 2011 due to an expected supply shortage for critical applications such as in permanent magnets used in wind turbines and motors in hybrid/electric vehicles. As a result, production at the California-based Mountain Pass Mine (Molycorp, Inc.) was re-started after several years of dormancy. However, after peaking in price in 2011, prices have dropped substantially to slightly above 2010 levels, challenging the profitability of non-China based production which consists mainly of hard rock carbonatite deposits, that are deficient in critical REE and HREE. Seredin and Dai (2012) [9] have noted that *mining of the Mountain Pass and similar resources will neither mitigate the crisis in REE resources nor eliminate the shortage of the most critical REE, but will only result in overproduction of excessive Ce.* 

According to the USGS 2017 Mineral Commodity Summary report, China accounted for about 83% of the total global REE supply in 2016, down from about 95% prior to 2010 [10]. Meanwhile, the US production was zero, after the temporarily opened Mountain Pass Mine stopped operations at the end of 2015 after Molycorp declared bankruptcy. Although still dominating global supply, Chegwidden and Kingsnorth (2011) [11] have estimated that the Chinese ion-adsorbed clays resource will only last another 15-20 years, based on estimated remaining reserves [12, 13]. *Due to its limited supply, and because the Chinese resource is rich in HREE and critical REE, while most other traditional resources are deficient in these less common and more valuable elements, it is imperative that new domestic sources of REEs, especially the HREE and critical REE, be identified and processes be developed to produce them. The aim of this proposal is to develop a technology to enable production of one of these promising alternative resources – coal and coal byproducts.* 

#### 5.2 Coal and Coal Byproducts – Alternative REE Resources

Ackmann (2012) [14] conducted a prospectivity analysis of REE + Y in coals and coal byproducts and found that the levels of REEs were enriched in some coal beds and formations above crustal average. The

study also estimated that the "unintended" production of REEs from coal mining was greater than 40,000 tons in 2010. Although recovery of REEs from coal presents several challenges, namely the relatively low REE content, it also offers several advantages compared to traditional recovery from mineral resources. Seredin and Dai (2012) [9] present data that indicates that coal ash is a better source of critical REE components than conventional mineral resources, mainly due to the enrichment of the HREE and critical REE in some coals.

#### 5.2.1 REE Associations in Coal

The abundance and association of the REE in coal is controlled by the type of REE-containing source materials. Source materials can be derived from minerals and volcanic ash (detrital origin) that are accumulated with the organic materials in a swamp [15, 16, 17]. During the coal formation process the REE-containing volcanic ash and minerals are exposed to the influence of ground water and other processes that can result in the transfer of the REE to the organic fraction or to new authigenic minerals. This process can result in the enrichment of REE in selected layers in the coal bearing stratigraphic sequences.

Very fine-grained REE-bearing minerals are commonly found in coals and authigenic minerals are typically more abundant than detrital minerals in high REE content coals. These can be discrete minerals contained in the organic matrix, or as minerals associated with clay minerals. Often, the REE in clay-bound minerals are enriched or contained only within the partings of the coal or margins of the seam. Seredin and Dai (2012) have categorized the main genetic types of high REE accumulation in coals as shown in Table 1.

Туре	REE Content in Ash, %	Associated Elements
Terrigenous	0.1–0.4	Al, Ga, Ba, Sr,
Tuffaceous	0.1–0.5	Zr, Hf, Nb, Ta, Ga
Infiltrational	0.1–1.2	U, Mo, Se, Re
Hydrothermal	0.1–1.5	As, Sb, Hg, Ag, Au, etc.

Table 1. Main genetic classifications of REE accumulation in coal [9]

Many low-rank subbituminous and lignite coals contain a high proportion of REE associated with the organic fraction as ions attached to carboxylic acid groups or as coordination complexes or adsorbed onto the organic matter. Hower and others (2016) [18] note that HREE generally have higher affinity to organic matter and form more stable organic complexes than LREE. Eskenazy evaluated a Bulgarian lignite coal and found that REE cations bound to –COOH and –OH groups replaced the alkali cations [19]. Other studies have indicated correlations of REE content with ash content and specific gravity fraction, with highest REE content in the low ash and low density fractions, which were considered to be inferred organic associations [18]. In the ongoing work by UND, the association of REEs in North Dakota lignites has been determined to be primarily (80-90%) with the organic matter as coordination complexes of oxygen functional groups. Further, a tuffaceous origin is suggested by the data collected, with an infiltrational mechanism of surface water leaching of REE-containing volcanic ash being adsorbed by the organic matter during coalification [20].

Understanding REE associations in coal and their depositional origins is critical, not only in the development of extraction processes, but also in development of fingerprinting algorithms that can identify REE-rich coals and sediments based on mineralogy and bulk chemistry.

#### 5.2.2 Evaluation of Coal and Coal Byproducts as an REE Resource

Seredin and Dai (2012) [9] have established criteria for the determination of promising coals for REE recovery. These include a cutoff of about 800-1000 ppm (oxide ash basis) total mixed REE+Y content combined with metrics that evaluate the content of the individual elements within the total. An ideal feedstock would have the highest concentration of critical REE and lowest concentration of excessive REE (i.e. Ce). Figure 3 displays their evaluation criteria and shows the percent critical elements on the Y-axis and an outlook coefficient (metric describing ratio of critical to excessive REE) on the X-axis. Based on their extensive sampling of coals globally, they have prepared three general groupings and have compared the coal ashes to various mineral and ion-adsorbed clay deposits. Their analysis indicates that many coal ashes are more promising than traditional mineral resources such as the Mountain Pass mine. Based on the

ongoing UND and NDGS work, the North Dakota lignites analyzed to date fit exclusively in the promising and highly promising zone [20, 3].

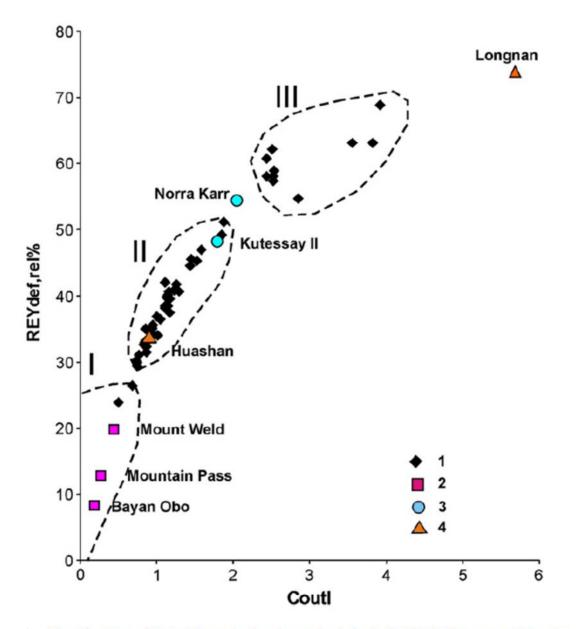


Figure 3. Classification of REE-rich coal ashes by outlook for individual REY composition in comparison with selected deposits of conventional types.1 – REE-rich coal ashes; 2 – carbonatite deposits; 3 – hydrothermal deposits; 4 – weathered crust elution-deposited (ion-adsorbed) deposits. Clusters of REE-rich coal ashes distinguished by outlook for REY composition (numerals in figure): I – unpromising, II – promising, and III – highly promising.

#### 5.2.3 REE Content in North Dakota Lignite Coal

As part of an ongoing DOE project at UND [21], efforts are focused on determining the feasibility of recovering REE from ND lignite coal and byproducts. UND has performed sampling and analysis campaign on a range of coals and associated sediments (byproducts) from multiple seams in the state. At the Falkirk Mine, analysis has shown that on an ash basis, the REE are more concentrated in certain locations and certain coal seams than in the associated roof/floor sediments, as shown in Figure 4. Detailed analysis using chemical fractionation [22] and scanning electron microscopy/x-ray microanalysis (methods described later in Section 5.3) has shown that the bulk of the REE are organically associated in the lignite coals. Even though REE-enriched layers have been identified at the Falkirk Mine, sampling work led by NDGS [3] in the west and southwest portions of the state have identified exceptionally enriched REE coals and associated sediments. For example, samples collected from an exposed outcropping of the Harmon-Hansen coals in Slope County have shown REE content exceeding 600 ppm on a dry whole coal basis, or more than 2000 ppm on an ash basis. Several additional samples have also been collected by NDGS that have much higher content than the Falkirk samples. These represent unusually elevated REE levels, and are as high as anything reported in the literature for U.S. coals [23, 24, 25]. The high content of REEs found in North Dakota lignites to date, combined with the massive lignite reserves in the State suggests that commercially feasible (mineable) quantities of REE-rich coals are present and could be economically attractive targets for REE recovery.

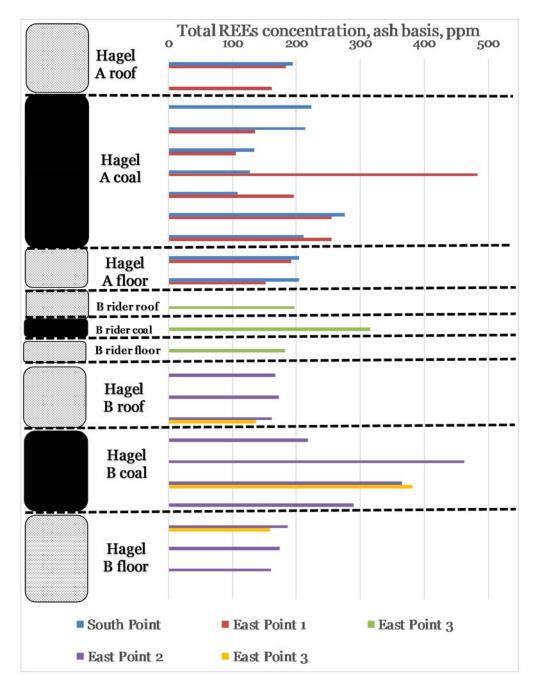


Figure 4. Rare earth element content (ash basis) in the Falkirk Mine stratigraphic column at multiple locations

Further, the weakly-bonded organic association of the REEs in North Dakota lignite, presents a unique opportunity for REE recovery. In the ongoing UND work [21], a low-cost and highly effective novel REE extraction process has been developed. The process involves a highly dilute acid leach at ambient temperature of the raw coal (not the coal ash) that produces very high recovery of the REE, Y and Sc. The

resulting residual coal has lower ash content and can be utilized in any number of coal conversion processes. Up to 90% of the REEs can be recovered from the lignites using this process, which also recovers several other high value metals, such as germanium, gallium and cobalt. The combination of extremely high REE recovery and exceptionally simple extraction method (no physical beneficiation/pre-processing required) are expected to result in significantly lower costs than recovery from coal fly ash, higher rank coals, or traditional mineral deposits. Also important is that the process results in a beneficiated coal that has reduced ash, but preserved organic content.

#### 5.3 Measuring Inorganic Composition of Coal and Abundance and Association of REE

There are numerous methods used to determine the abundance and association of inorganic elements such as REE in coals. The following is a brief overview of applicable methods that can be used to develop methods to identify, separate, and sort REE-rich coal.

#### 5.3.1 Inductively Coupled Plasma – Mass Spectroscopy (ICP-MS)

ICP-MS is an excellent method for determining the abundance of REE and non-REEs in bulk samples of coal, fly ash and associated sediments. However, ICP-MS is a time and cost-intensive method that is not compatible with field or in-process analysis. The analysis procedure includes multiple steps, including ashing to remove carbon, fusion/acid digestion to dissolve REEs into solution, and analysis of the REE-containing solution by ICP-MS [26].

#### 5.3.2 X-ray Fluorescence (XRF)

XRF methods can be used to determine the abundance of REE and non-REE elements. The method is not as sensitive as ICP-MS. Hand-held portable X-ray fluorescence (pXRF) spectrometers are being evaluated for their ability to be used as a tool to indicate the presence of REE in coal and coal waste materials [1]. Commercial pXRF systems have the ability to detect and measure Y and light REE up to the molecular weight of Nd (addition of a radioactive source is required for heavier elements). The time required for a measurement will depend on the nature of the sample and the levels of the elements of interest. High elemental levels will take a few seconds while part-per-million levels take a few minutes.

The concentration of REE in many of the coals are too low to detect and measure with pXRF. However, previous work (discussed below) by others and the Microbeam/UND team has indicated that it is possible to use surrogate elements as markers to indicate the presence of REE.

Bryan and others (2015) [1] were successful in making correlations with elements or combinations of elements measured by pXRF in coals and coal waste materials with REEs measured by laboratory techniques (i.e. ICP-MS). They concluded that the pXRF method can be used as a low-cost screening tool to determine if samples have sufficiently high REE levels to warrant additional analysis. They also indicated that certain elements such as Y could be used as an indication of the presence of light REE and were able to distinguish between low, medium and high levels of light REE. *Thus, an ideal application for pXRF would be to identify elements in the coal that are available in sufficient quantity to permit fast measurement and accurate quantification, and are also determined (via single elements or combinations of elements) to have positive correlations to REEs.* 

Microbeam and UND have conducted preliminary analysis of samples of ND lignite coal using UND's Bruker Tracer IV-SD pXRF. Coal samples available from UND's ongoing REE work were analyzed using pXRF using operating conditions for mid and heavy elements (Ca, Ba, Ti, Cr, Mn, Fe, Co, Ni, Cu, Zn, Ga, As, Pb, Th, Rb, U, Sr, Y, Zr). The samples were analyzed previously for REE content via ICP-MS. The quantified pXRF results were plotted against total REE content to identify positive correlations. Promising correlations were observed between REE and several elements with the best fits for Zn, Ti and Zr (Figure 5). Correlations with Zr suggest volcanic ash origins (see Table 1). This data is a promising start, but most samples analyzed were from the Falkirk Mine that have narrow range of total REE content. Additional samples covering a larger range of REE content will be required to generate a more complete (and useful) data set with this methodology. This is a major objective of the proposed project.

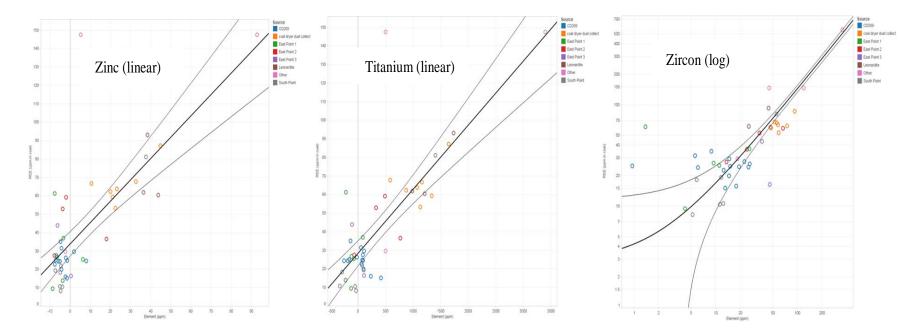


Figure 5. Preliminary REE-predictive correlations based on pXRF measurements of existing UND samples

# 5.3.3 Scanning Electron Microscopy with Energy Dispersive X-ray Spectrometry (SEM-EDS)

SEM-EDS is an excellent method to determine the size, composition, and abundance of mineral grains in coal. Microbeam has conducted SEM-EDS analysis of nearly 10,000 samples of coal, ash related materials, and materials of construction, and extensively uses computer controlled SEM (CCSEM) methods to conduct automated size and composition analysis of thousands of particles in a sample. Microbeam has routine mineral typing schemes for 33 mineral types based on composition and an excellent method for determining associations of elements in coal and coal byproducts [27].

#### 5.3.4 Chemical Fractionation

Chemical fractionation methods are used to selectively extract elements based on their solubility in water, ammonium acetate, and hydrochloric acid [22]. Water removes water soluble minerals. The ammonium acetate removes ion exchangeable elements. The hydrochloric acid removes elements associated as carbonates and coordination complexes, and the residual material after extraction includes elements associated as clays, other aluminosilicates, and sulfides. Karner and others [28, 29] used chemical fractionation, correlations with a mineral content and lithologic layering, and SEM-EDS mineral analysis to identify associations of REE in lignite.

#### 5.3.5 Prompt Gamma Neutron Activation Analysis

Prompt gamma neutron activation analysis (PGNAA) is an analytical technique that has been applied to measuring coal properties on moving belts at coal mines and power plants. Project partner ETI has developed full stream elemental analyzers (FSEA) that incorporates PGNAA to determine the inorganic components present along with sensors to measure moisture and ash content and has over 200 installations worldwide. The FSEA is a cross-belt mounted system that measures the chemical composition of material on a conveyor belt in real time, providing the opportunity for sorting of feed materials based on composition thresholds. A discussion of the commercial application of ETI's FSEA is provided in the following section.

#### 5.3.6 Coal Properties and Correlations for Coal Sorting and Blending

Microbeam and ETI have worked together to apply the FSEA to "fingerprint" coal types that allows for sorting and blending to optimize plant performance [30]. The system was tested and standardized with coals that have a range of properties and known response in the plant as shown in Figure 6.

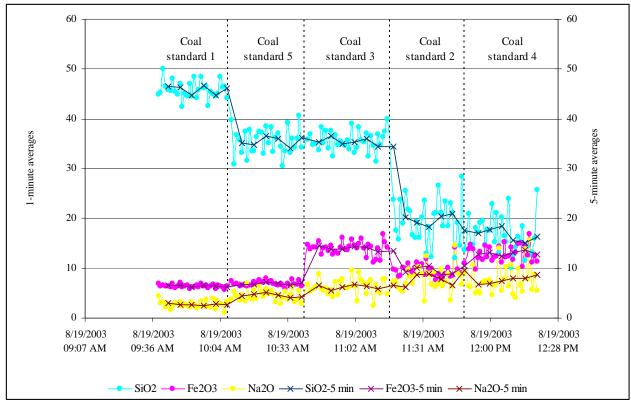


Figure 6. PGNAA analysis of multiple coal standards using ETI's Full Stream Elemental Analyzer

A simple example of a Microbeam-developed correlation derived from the FSEA-PGNAA system includes the base-to-acid ratio expressed as oxides ( $B/A=(Na_2O+MgO+CaO+K_2O+Fe_2O_3)/(SiO_2+Al_2O_3+TiO_2)$ ) of the fuel impurities, sodium level, and ash content. The correlations are used to optimize coal boiler performance through blending and sorting. The sorting and blending process schematic shown in Figure 7 utilizes two analyzers at Milton R. Young Unit 2 (~470MW) (Center, ND) [31, 32]. The ETI PGNAA system is able to successfully sort coals into groupings based on the B/A ratio and ash content, and a second ETI PGNAA analyzer is utilized in a feedback control loop to blend the coal to optimize plant performance.

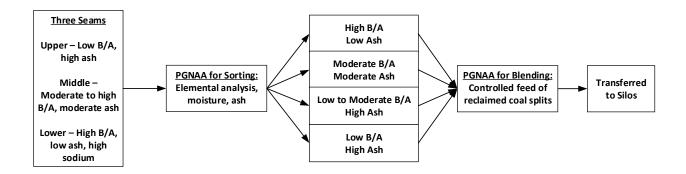


Figure 7. Sorting and blending of coal using Microbeam's fuel performance algorithms and ETI's PGNAA analyzer at Milton R. Young power station.

#### 5.4 Summary of the Research Motivation

Due to unsustainable foreign supplies of critical and heavy REE, upon which the US is currently reliant, it is imperative that alternative domestic sources of REEs be identified and processes be developed to produce them. Most traditional REE resources, such as the US Mountain Pass mine are deficient in the critical REEs. However, coal and coal byproducts have been identified as promising resources. Coals throughout the world have shown elevated levels of REE, specifically elevated levels of HREE and critical REE compared to traditional mineral resources, making them an attractive alternative resource. However, the content of REEs within the coal is not uniformly distributed throughout the stratigraphic sequence, and thus selective mining procedures will need to be employed to maximize the content of REE in the feed coal to a REE recovery process. *To aid in the development of selective mining, new analysis/sensor technology combined with predictive algorithms must be developed to reduce the cost and time of REE measurement, while also providing the possibility of field and in-process sorting capabilities to separate and stockpile coal with the highest REE content. The proposed work will develop such technology.* 

#### **6** QUALIFICATIONS

The project team compiled is uniquely qualified to perform the proposed work, having extensive expertise in all areas pertinent to successful completion of the project – advanced coal and coal-related materials characterization, rare earth elements chemistry and geochemistry in coals, and similar algorithm

development expertise related to coal sorting. The key personnel for each organization are identified below with relevant qualifications.

<u>Ms. Shuchita Patwardhan will be the Principal Investigator (PI) on this project and will lead the project</u> <u>effort.</u> Ms. Patwardhan, Project Manager at Microbeam, has a BS degree in Chemical Engineering and an MS in Environmental Engineering from the University of North Dakota. She currently manages and works on projects aimed at minimizing the impact of fuel impurities on efficient and environmentally acceptable use of coal and other fuels in combustion and gasification systems. She works with fuel preparation and blending facilities equipped with PGNAA technologies for on-line fuel analysis to optimize fuel blending to improve plant performance. She leads projects on generating mathematical correlations for fuel properties, transport mechanisms, deposit growth and slag flow in high temperature combustion and gasification systems. She has extensive experience in interpreting CCSEM, chemical fractionation and pXRF field data along with plant performance. She has also worked on several projects associated with the analysis on coal byproducts for rare earth elements.

Mr. David Stadem, Research Scientist at Microbeam, has a BS degree in Chemistry and is working on an MS in Energy Engineering at UND. Mr. Stadem currently conducts interprets coal and coal ash mineralogy and phase analysis determined using CCSEM and XRF methods. He has conducted analysis coal and related materials derived from mining and associated with combustion and gasification systems. He develops algorithms to predict the impact of fuel properties and plant performance. He has experience with pXRF in the field and laboratory applications.

Dr. Steven Benson is the President of Microbeam Technologies, Inc and has strong expertise in fuel analysis, fuel properties, combustion, gasification, ash transformations, and pollution control. Dr. Benson and MTI have performed numerous projects for many coal-fired power plants, and will bring expertise in the area of SEM and coal chemical composition analysis and recovery of REEs from coal. Dr. Benson will assist the PI for MTI in managing the scope of work as laid out in the task descriptions above.

Dr. Daniel Laudal, Manager: Major Projects at UND Institute for Energy Studies, brings expertise in the areas of rare earth element extraction processes for coal and coal byproducts, coal and REE geochemistry, advanced materials/fuels characterization and coal utilization processes. Dr. Laudal's Ph.D. research work was focused on development of novel REE recovery methods from North Dakota lignites. Dr. Laudal will be the PI for UND's scope of work.

Mr. David Swindell, President, Energy Technologies Inc. has over 22 years of experience in designing, manufacturing and installing bulk material analyzers and process control equipment in the coal industry. Mr. Swindell's expertise in on-line fuel analyzers will help this project integrate lab scale analysis with field application. Mr. Swindell will serve as the PI for ETI's scope of work.

#### 7 VALUE TO NORTH DAKOTA

North Dakota produces over 30 million tons of lignite annually. The state's economy is heavily invested in the production and use of lignite. Successful completion of the proposed project will enable a new high value commercial opportunity for lignite use. The project will support the development of REE recovery processes by lowering the cost of mining REE-rich coals and providing the best opportunity to deliver a high-grade feedstock to the REE recovery process. If REE recovery technologies for lignite and lignite-related feedstocks can be successfully realized commercially, a completely new industry will formed, providing new opportunities for high-paying jobs and new tax revenues for the State. The results of the work extends far beyond REEs. It also will provide information on the ability to measure other elements such as sodium, sulfur, and trace elements that are also important to the utilization of lignite coal from ND. The project will also provide additional information on the ability to selectively mine and blend lignite to improve plant performance.

#### 8 MANAGEMENT

The team assembled to perform the proposed work includes Microbeam Technologies Incorporated, the UND Institute for Energy Studies, and Energy Technologies Incorporated. The project will be led by Ms. Shuchita Patwardhan at MTI, who will be the overall Project Manager. Ms. Patwardhan will be the contact person for MTI and the overall project team, and will be responsible for managing resources and project schedule and will coordinate meetings and conference calls with the NDIC/LRC, project cosponsors and project participants. Dr. Steve Benson from MTI will assist Ms. Patwardhan in the project management activities and will provide technical expertise related to coal and REE geochemistry and advanced characterization techniques. The UND team will be led by Dr. Dan Laudal, who will provide technical expertise related to rare earth elements content, distribution and modes of occurrence in North Dakota lignite-related materials. Dr. Laudal will coordinate the analysis efforts with UND's pXRF and gamma ray core log instruments and will work closely with the MTI team to develop REE-predictive algorithms based on the results. Mr. David Swindell from ETI will lead the ETI team, and will coordinate the PGNAA calibration and verification efforts associated with Task 5.

The project activities have been divided by task, each with specific technical objectives, with the tasks to be implemented and completed under the direction of each task leader. Figure 8 displays the management structure for the project, with task leaders and key personnel identified. Cost management will be coordinated by the Administrative Resource Manager who will be responsible for tracking all costs for each of the project tasks.

Project meetings and conference calls will be held, at least, on a weekly basis to conduct project activities, review project timelines, upcoming milestones/deliverables, costs and challenges associated with the completion of the project tasks. Microsoft Project management tools will be utilized. Project review meetings with sponsors will also be held on a monthly or quarterly basis to ensure communication and discussion of accomplishments, plans and management of project risks.

### 9 TIMETABLE

The proposed project is planned for a 2-year duration, with an anticipated start date of October 1, 2017. The project Gannt chart is displayed in Figure 9. Major milestones and planned completion dates are provided below.

- 1. Collect approximately 100 samples with distributed composition for analysis (Month 12)
- 2. Down-selection of about 20 samples for detailed chemical analysis (Month 13)
- 3. Successfully identify correlations for REE content via pXRF and gamma ray core log (Month 17)
- 4. Successfully verify ability to sort REE-rich coals via calibrated PGNAA (Month 24)

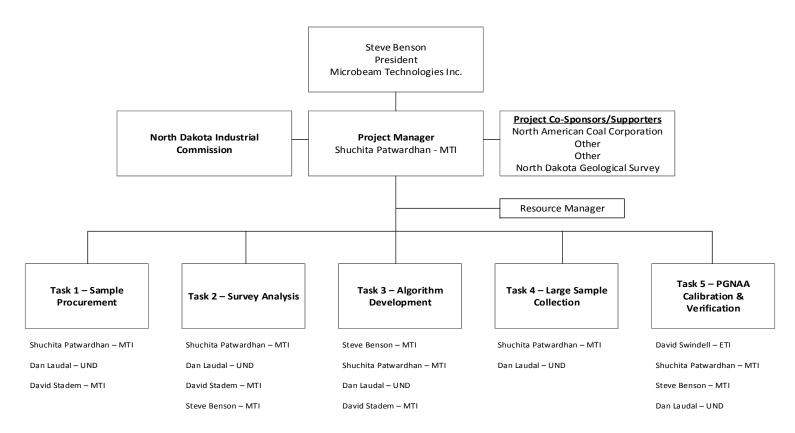


Figure 8. Project management structure

D	Tethler	Start	Finish	Q4 17			Q1 18			QZ 18			Q3 18			Q4 18			Q1 19			Q2 19			Q3 19		
	Task Name			Oct	Nov	Dec	Jan	Fab	Mar	Apr	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec	Jan	Fab	Mar	Apr	May	Jun	Jul	Aug	Sep
1	Task 1 - Identification and Procurement of Samples	10/2/2017	9/28/2018																								
2	Task 2 – Survey Analysis	11/1/2017	12/31/2018																								
з	Task 3 – REE-predictive Algorithm Development	10/1/2018	2/28/2019																								
4	Task 4 – Large Sample Collection	6/1/2018	2/28/2019																								
5	Task 5 – PGNAA Calibration and Verification Testing	3/1/2019	9/27/2019																								

Figure 9. Project schedule

# **10 BUDGET**

The overall budget is as follows. Budget notes and information from subcontractors is included in

Appendix B.

# **RESEARCH & RELATED BUDGET - Cumulative Budget**

Section A, Senior/Key Person         50,284.17           Section B, Other Personnel         22,540.60           Total Number Other Personnel         4           Total Salary, Wages and Fringe Benefits (A+B)         72,794.77           Section C, Equipment         6,125.00           Section D, Travel         6,125.00           1. Domestic         6,125.00           2. Foreign         6           Section F, Participant/Trainee Support Costs         1           1. Tuition/Fees/Health Insurance         1           2. Stipends         1           3. Travel         1           4. Subsistence         106,772.14           5. Other         106,772.14           1. Materials and Supplies         5,141.94           2. Publication Costs         100,053.00           3. Consultant Services         100,053.00           4. ADP/Computer Services         100,053.00           5. Subawards/Consortium/Contractual Costs         100,053.00           6. Equipment or Facility Rental/User Fees         1,4terations and Renovations           8. Other 1         1,677.20           9. Other 2         100           10. Other 3         105,691.91           Section G, Direct Costs (A thru F)         105,691.91		Totals (\$)		
Total Number Other Personnel         4           Total Salary, Wages and Fringe Benefits (A+B)         72,794.77           Section C, Equipment         6,125.00           Section D, Travel         6,125.00           1. Domestic         6,125.00           2. Foreign         6           Section E, Participant/Trainee Support Costs         1           1. Tuition/Fees/Health Insurance         1           2. Stipends         1           3. Travel         1           4. Subsistence         106,772.14           5. Other         106,772.14           1. Materials and Supplies         5,141.94           2. Publication Costs         106,772.14           1. Materials and Supplies         5,141.94           2. Publication Costs         100,053.00           3. Consultant Services         100,053.00           4. ADP/Computer Services         100,053.00           5. Subawards/Consortium/Contractual Costs         100,053.00           6. Equipment or Facility Rental/User Fees         1           7. Alterations and Renovations         1           8. Other 1         1,577.20           9. Other 2         1           10. Other 3         105,691.91           Section G, Direct Costs (A th	Section A, Senior/Key Person		50,254.17	
Total Salary, Wages and Fringe Benefits (A+B)       72,794.77         Section C, Equipment       6,125.00         Section D, Travel       6,125.00         1. Domestic       6,125.00         2. Foreign       9         Section E, Participant/Trainee Support Costs       1         1. Tuition/Fees/Health Insurance       9         2. Stipends       9         3. Travel       9         4. Subsistence       9         5. Other       9         6. Number of Participants/Trainees       9         Section F, Other Direct Costs       106,772.14         1. Materials and Supplies       5,141.94         2. Publication Costs       106,772.14         3. Consultant Services       100,053.00         6. Equipment or Facility Rental/User Fees       100,053.00         7. Alterations and Renovations       1,577.20         9. Other 2       9       105,691.91         10. Other 3       9,075.27         Section G, Direct Costs       29,075.27	Section B, Other Personnel		22,540.60	
Section C, Equipment         12779171           Section D, Travel         6,125.00           1. Domestic         6,125.00           2. Foreign         Section E, Participant/Trainee Support Costs           1. Tuition/Fees/Health Insurance	Total Number Other Personnel	4		
Section D, Travel         6,125.00           1. Domestic         6,125.00           2. Foreign         Section E, Participant/Trainee Support Costs           1. Tuition/Fees/Health Insurance	Total Salary, Wages and Fringe Benefits (A+B)		72,794.77	
1. Domestic       6,125.00         2. Foreign	Section C, Equipment			
6,123.00         2. Foreign         Section E, Participant/Trainee Support Costs         1. Tuition/Fees/Health Insurance         2. Stipends         3. Travel         4. Subsistence         5. Other         6. Number of Participants/Trainees         Section F, Other Direct Costs         1. Materials and Supplies         5. /141.94         2. Publication Costs         3. Consultant Services         4. ADP/Computer Services         5. Subawards/Consortium/Contractual Costs         1. Materials and Renovations         8. Other 1         9. Other 2         10. Other 3         Section G, Direct Costs (A thru F)         Section H, Indirect Costs	Section D, Travel		6,125.00	
Section E, Participant/Trainee Support Costs         1. Tuition/Fees/Health Insurance         2. Stipends         3. Travel         4. Subsistence         5. Other         6. Number of Participants/Trainees         Section F, Other Direct Costs         1. Materials and Supplies         5. /141.94         2. Publication Costs         3. Consultant Services         4. ADP/Computer Services         5. Subawards/Consortium/Contractual Costs         100,053.00         6. Equipment or Facility Rental/User Fees         7. Alterations and Renovations         8. Other 1         9. Other 3         Section 6, Direct Costs (A thru F)         Section 7, Direct Costs         10. Other 3	1. Domestic	6,125.00		
1. Tuition/Fees/Health Insurance         2. Stipends         3. Travel         4. Subsistence         5. Other         6. Number of Participants/Trainees         Section F, Other Direct Costs         1. Materials and Supplies         2. Publication Costs         3. Consultant Services         4. ADP/Computer Services         5. Subawards/Consortium/Contractual Costs         100, 053.00         6. Equipment or Facility Rental/User Fees         7. Alterations and Renovations         8. Other 1         9. Other 2         10. Other 3         Section 6, Direct Costs (A thru F)         105, 691.91         Section H, Indirect Costs	2. Foreign			
2. Stipends         3. Travel         4. Subsistence         5. Other         6. Number of Participants/Trainees         Section F, Other Direct Costs         1. Materials and Supplies         5. onliant Services         3. Consultant Services         3. Consultant Services         3. Consultant Services         3. Consultant Services         4. ADP/Computer Services         5. Subawards/Consortium/Contractual Costs         100,053.00         6. Equipment or Facility Rental/User Fees         7. Alterations and Renovations         8. Other 1         9. Other 2         10. Other 3         Section G, Direct Costs (A thru F)         Section G, Direct Costs         9. other 2         10. Other 3	Section E, Participant/Trainee Support Costs			
3. Travel         4. Subsistence         5. Other         6. Number of Participants/Trainees         Section F, Other Direct Costs         1. Materials and Supplies         5. publication Costs         3. Consultant Services         4. ADP/Computer Services         5. Subawards/Consortium/Contractual Costs         1. Alterations and Renovations         8. Other 1         9. Other 2         10. Other 3         Section G, Direct Costs         10. Other 3         Section H, Indirect Costs	1. Tuition/Fees/Health Insurance			
4. Subsistence         5. Other         6. Number of Participants/Trainees         Section F, Other Direct Costs         1. Materials and Supplies         5. 7,141.94         2. Publication Costs         3. Consultant Services         4. ADP/Computer Services         5. Subawards/Consortium/Contractual Costs         100,053.00         6. Equipment or Facility Rental/User Fees         7. Alterations and Renovations         8. Other 1         9. Other 2         10. Other 3         Section G, Direct Costs (A thru F)         Section H, Indirect Costs	2. Stipends			
5. Other	3. Travel			
6. Number of Participants/Trainees         Section F, Other Direct Costs         1. Materials and Supplies         2. Publication Costs         3. Consultant Services         4. ADP/Computer Services         5. Subawards/Consortium/Contractual Costs         100,053.00         6. Equipment or Facility Rental/User Fees         7. Alterations and Renovations         8. Other 1         9. Other 2         10. Other 3         Section G, Direct Costs (A thru F)         Section H, Indirect Costs	4. Subsistence			
Section F, Other Direct Costs       106,772.14         1. Materials and Supplies       5,141.94         2. Publication Costs       1         3. Consultant Services       1         4. ADP/Computer Services       1         5. Subawards/Consortium/Contractual Costs       100,053.00         6. Equipment or Facility Rental/User Fees       1         7. Alterations and Renovations       1,577.20         8. Other 1       1,577.20         9. Other 2       1         10. Other 3       185,691.91         Section G, Direct Costs (A thru F)       185,691.91         Section H, Indirect Costs       39,075.37	5. Other			
1. Materials and Supplies       5,141.94         2. Publication Costs	6. Number of Participants/Trainees			
1. Materials and Supplies       5,141.94         2. Publication Costs	Section F, Other Direct Costs		106,772.14	
3. Consultant Services         4. ADP/Computer Services         5. Subawards/Consortium/Contractual Costs         100,053.00         6. Equipment or Facility Rental/User Fees         7. Alterations and Renovations         8. Other 1         9. Other 2         10. Other 3         Section G, Direct Costs (A thru F)         Section H, Indirect Costs	1. Materials and Supplies	5,141.94		
4. ADP/Computer Services         5. Subawards/Consortium/Contractual Costs         100,053.00         6. Equipment or Facility Rental/User Fees         7. Alterations and Renovations         8. Other 1         1,577.20         9. Other 2         10. Other 3         Section G, Direct Costs (A thru F)         Section H, Indirect Costs	2. Publication Costs			
5. Subawards/Consortium/Contractual Costs       100,053.00         6. Equipment or Facility Rental/User Fees	3. Consultant Services			
6. Equipment or Facility Rental/User Fees       100,053.00         7. Alterations and Renovations       1         8. Other 1       1,577.20         9. Other 2       1         10. Other 3       1         Section G, Direct Costs (A thru F)       105,691.91         Section H, Indirect Costs       39,075.37	<ol> <li>ADP/Computer Services</li> </ol>			
7. Alterations and Renovations         8. Other 1       1,577.20         9. Other 2         10. Other 3         Section G, Direct Costs (A thru F)         185,691.91         Section H, Indirect Costs	5. Subawards/Consortium/Contractual Costs	100,053.00		
8. Other 1       1,577.20         9. Other 2	6. Equipment or Facility Rental/User Fees			
9. Other 2         10. Other 3         Section G, Direct Costs (A thru F)         Section H, Indirect Costs	7. Alterations and Renovations			
10. Other 3           Section G, Direct Costs (A thru F)           185, 691.91           Section H, Indirect Costs           29, 075.37	8. Other 1	1,577.20		
Section G, Direct Costs (A thru F) 185, 691.91 Section H, Indirect Costs 39, 075.37	9. Other 2			
Section H, Indirect Costs 29,075.27	10. Other 3			
	Section G, Direct Costs (A thru F)		185,691.91	
	Section H, Indirect Costs		39,075.37	
Section I, Total Direct and Indirect Costs (G + H) 224, 767.28	Section I, Total Direct and Indirect Costs (G + H)		224,767.28	
Section J, Fee 0.00	Section J, Fee		0.00	

### 11 MATCHING FUNDS

The total project cost is expected to be \$449,534 that includes \$168,767 of in-kind cost share, \$56,000 in cash cost share from project sponsors currently being identified, and \$224,767 from NDIC. Microbeam is in contact with coal companies, utilities, and others regarding participation in the project. Letter of support will be provided to NDIC prior to project initiation.

# 12 TAX LIABILITY

No outstanding liabilities to the state of North Dakota

# **13 CONFIDENTIAL INFORMATION**

No confidential information has been included with this application.

# **14 APPENDICES**

- A. References
- B. Budget summary and budget justification
- C. Letters of support and cost share contributions
- D. Additional facilities and equipment documentation
- E. Resumes of key personnel

#### **APPENDIX A – REFERENCES**

- R. C. Bryan, D. Richers, H. T. Andersen and T. Ackman, "Study on the utilization of portable handheld XRF spectroscopy as a screening tool for rare earth elements in coal and coal waste products," Document No: 114-910178X-100-REP-R001-01, 2015.
- [2] S. Dai and R. B. Finkelman, "Coal as a promising source of critical elements: Progress and future prospects," *International Journal of Coal Geology*, p. in press, 2017.
- [3] N. W. Kruger, L. D. Moxness and E. C. Murphy, "in prep., Rare Earth Element Concentration in Hell Creek and Fort Union Strata in Western North Dakota," North Dakota Geological Survey, Report of Investigation no. 117, in preparation.
- [4] N. W. Kruger and North Dakota Department of Mineral Resources, "Rare Earths in Coal," January 2017. [Online]. Available: https://www.dmr.nd.gov/ndgs/documents/newsletter/2017Winter/Rare%20Earths%20in%20Coal.pd f.
- [5] N. Kruger and North Dakota Department of Mineral Resources, "A "Rare Opportunity"," January 2016. [Online]. Available: https://www.dmr.nd.gov/ndgs/documents/newsletter/Winter%202015/A%20Rare%20Opportunity.p df.
- [6] N. D. D. o. M. Resources, "Mineral Resources of North Dakota: Uranium," North Dakota Geological Survey, [Online]. Available: https://www.dmr.nd.gov/ndgs/mineral/uranium.asp.
- [7] S. A. Benson, A. Ruud, S. Patwardhan, E. Sirianni, E. Flynn and D. Stadem, "Optimization of tools to manage coal properties and plant operations - Final Report," North Dakota Industrial Commission, FY13-LXXIV-182, 2015.
- [8] V. V. Seredin and S. Dai, "Coal deposits as potential alternative sources for lanthanides and yttrium," *International Journal of Coal Geology*, vol. 94, pp. 67-93. DOI: 10.1016/j.coal.2011.11.001, 2012.
- [9] U.S. Department of Energy National Energy Technology Laboratory, "Rare Earth Elements From Coal and Coal By-Products," 2017. [Online]. Available: https://www.netl.doe.gov/research/coal/rare-earth-elements.
- [10] U.S. Department of the Interior; U.S. Geological Survey, "Mineral Commodity Summaries 2017," USGS, 2017.
- [11] J. Chegwidden and D. J. Kingsnorth, "Rare earths an evaluation of current and furture supply," 2011. [Online]. Available: http://www.tremcenter.org/index/php?option=com\_attachements&task=download&id=412011.

- [12] R. Chi and J. Tian, Weathered crust elution-deposited rare earth ores, New York: Nova Science Publishers, 2008.
- [13] Z. Bao and Z. Zhao, "Geochemistry of Mineralization with Exchangeable REY in the Weathering Crusts of Granitic Rocks in South China," *Ore Geology Reviews*, vol. 33, pp. 519-535, 2008.
- [14] T. Ackman, J. Ekmann, C. Kirchner, E. Lopert and J. Pierre, "Rare earth elements in coal the case for research and development into co-production with coal," Leonardo Technologies, Inc., 2012.
- [15] V. Bouska and J. Pesek, "Quality parameters of lignite of the North Bohemian Basin in the Czech Republic in comparison with the world average lignite," *International Journal of Coal Geology*, vol. 40, pp. 211-235, 1999.
- [16] S. Dai, D. Ren, C. L. Chou, R. B. Finkelman, V. V. Seredin and Y. Zhou, "Geochemistry of trace elements in Chinese coals: a review of abundances, genetic types, impacts on human health, and industrial utilization.," *International Journal of Coal Geology*, no. doi:10.1016/j.coal.2011.02.003, 2011.
- [17] S. Dai, X. Wang, Y. Zhou, J. C. Hower, D. Li, W. Chen and X. Zhu, "Chemical and mineralogical compositions of silicic, mafic, and alkali tonsteins in the late Permian coals from the Songzao Coalfield, Chongqing, Soutwest China," *Chemical Geology*, vol. 282, pp. 29-44, 2011.
- [18] J. C. Hower, E. J. Granite, D. B. Mayfield, A. S. Lewis and R. B. Finkelman, "Notes on Contributions to the Science of Rare Earth Element Enrichment in Coal and Coal Combustion Byproducts," *Minerals*, vol. 6, no. 32, 2012.
- [19] G. Eskenazy, "Rare earth elements in a sampled coal from the Pirin Deposit, Bulgaria," *International Journal of Coal Geology*, vol. 7, pp. 301-314. https://doi.org/10.1016/0166-5162(87)90041-3, 1987.
- [20] D. A. Laudal and S. A. Benson, "Rare earth elements in North Dakota lignite coal and lignite-related materials," in *Clearwater Clean Energy Technology Conference*, Clearwater, Florida, 2017.
- [21] S. A. Benson and D. A. Laudal, "Investigation of rare earth element extraction from North Dakota coal-related feedstocks," in 2017 NETL Crosscutting Research Meeting, Pittsburgh, PA, 2017.
- [22] S. A. Benson and P. L. Holm, "Comparison of inorganics in three low-rank coals," *Ind. Eng. Chem. Prod. Res. Dev.*, vol. 24, pp. 145-149, 1985.
- [23] "Rare earth elements from coal and coal by-products Energy Data Exchange portfolio," National Energy Technology Laboratory, [Online]. Available: https://edx.netl.doe.gov/ree/.
- [24] R. Matthias and L. Ruppert, "COALQUAL Database," United States Geological Survey, 6 4 2017. [Online]. Available: https://ncrdspublic.er.usgs.gov/coalqual/.
- [25] "Project review meeting for crusscutting research rare earth elements research portfolios," National Energy Technology Laboratory, 2017. [Online]. Available:

https://www.netl.doe.gov/events/conference-proceedings/2017/2017-project-review-meeting-for-crosscutting-research-gasification-systems-and-rare-earth-elements-research-portfolios.

- [26] T. Bank, E. Roth, P. Tinker and E. Granite, "Analysis of rare earth elements in geologic samples using inductively couple plasma mass spectrometry," U.S. DOE Topical Report - DOE/NETL-2016/1794, 2016.
- [27] "Analysis of Coal Minerals," Microbeam Technologies Incorporated, 2017. [Online]. Available: www.microbeam.com.
- [28] F. R. Karner, S. A. Benson, H. H. Schobert and R. G. Roaldson, "Geochemical varioation of inorganic constituents in a North Dakota lignite," in *In The Chemistry of Low-Rank Coals; Schobert, H.H., (Ed); American Chemical Society Symposium Series 264*, 1984.
- [29] F. R. Karner, H. H. Schobert, S. K. Falcone and S. A. Benson, "Elemental distribution and association with inorganic and organic components in North Dakota lignites," in *In Mineral Matter* and Ash in Coal; Vorres, Karl S. (Ed); ACS Symposium Series 301, 1986.
- [30] A. A. Benson, M. L. Laumb and A. Ruud, "Analysis of factory acceptabnce test for online coal analyzer using prompt gamma neutron activation analysis," Microbeam Technologies Incorporated, for Minnkota Power Cooperative, 2014. MTI Project 692.
- [31] S. A. Benson, "Application of online coal analyzers to plant performance," in *Minnesota Energy Ingenuity Conference, Great River Energy*, 2008.
- [32] S. A. Benson, S. Patwardhan, A. Ruud, A. Freidt and J. Joun, "Ash formation and partitioning in a cyclone fired boiler," in *Impacts on Fuel Quality on Power Production Conference*, Snowbird, Utah, 2014.

# **APPENDIX B – SUBCONTRACTOR LETTERS OF SUPPORT AND BUDGETS**

INSTITUTE FOR ENERGY STUDIES COLLEGE OF ENGINEERING AND MINES UPSON II ROOM 366 243 CENTENNIAL DRIVE – STOP 8153 GRAND FORKS, NORTH DAKOTA 58202-8153 PHONE (701) 777-3852 FAX (701) 777-4838

July 1, 2017

Ms. Shuchita Patwardhan Project Manager Microbeam Technologies Inc. 4200 James Ray Drive, Ste 193 Grand Forks, ND 58202

Re: Support of the proposal entitled "Development of Low-Cost Rare Earth Element Analysis and Sorting Methods." Submitted to the North Dakota Industrial Commission Lignite Research Council.

Dear Ms. Patwardhan:

The University of North Dakota (UND) Institute for Energy Studies is pleased to provide this letter of support for Microbeam Technologies Incorporated in this collaborative project that will develop novel analysis techniques and predictive algorithms that will enable low-cost identification, mining and separation of high rare earth content coals and coal byproducts.

As part of an ongoing DOE project looking at the feasibility of extracting REE from North Dakota lignite coal-related feedstocks, we have identified multiple seams and locations in the state with high REE content. We have also shown that ND lignite resources present a unique opportunity due to the ability to utilize a simple and low cost extraction procedure, making them a highly attractive alternative domestic source of REE, especially the critical and heavy REE which are enriched in the lignite materials. However, our work has also shown that the REE content both within and between seams is not uniformly distributed. Therefore, we believe that to enable economic REE recovery from coal-related feedstocks it will be necessary to deploy selective mining methods in combination with real-time separation methods to maximize REE content of the feed material to a recovery process.

In addition to the UND scope of work for this project, as part of our ongoing work we have collected numerous samples and have an extensive analysis database, both of which we will make available to this proposed project. Work to be conducted by UND is summarized below.

**Task 1 – Identification and Procurement of Samples:** In this task, UND will work with Microbeam, as well as industry supporters to identify and procure lignite coal samples for analysis in this project. UND will assist in analyzing existing databases from drill core records, the USGS Coal Quality Database, and the existing UND database to identify locations and seams within the state to target.

Task 2 – Survey Analysis: In this task UND will perform analysis of approximately 100 samples using its existing handheld XRF instrument and five drill core samples using its existing gamma ray core logger instrument. UND will assist Microbeam, as necessary, in evaluating the analysis data to select approximately 20 of these samples for REE content determination using UND's existing inductively coupled plasma mass spectrometry system. Additionally, UND will work with Microbeam in calibration of the handheld XRF results based on standard laboratory XRF to ensure accurate and repeatable data.

**Task 3 – REE-Predictive Algorithm Development:** In this task, UND will support Microbeam in the development of novel algorithms that can predict the REE content within coal samples based on the bulk chemistry as measured by handheld XRF, without the need to directly measure the REE content.

Task 4 – Large Sample Collection: In this task, based on the results of previous tasks, UND will participate in collection of larger samples of coal that will be used in testing of project partner ETI's coal sorting analyzer. UND will also prepare the samples for testing in this task via grinding and homogenization using its existing sample preparation equipment.

Task 5 – PGNAA Calibration and Verification Testing: In this task, UND will provide input regarding calibration and testing of ETI's PGNAA analyzer, and will also assist Microbeam and ETI in data analysis efforts. UND will also perform ICP-MS analysis to determine REE content in the separated coal fractions from PGNAA testing.

UND is excited to team with Microbeam and ETI on this project, and we look forward to a successful proposal outcome. The overall budget for the UND scope of work is \$80,178 as detailed in the attached budget justification, with an anticipated start date of September 1, 2017. If you have any questions, please do not hesitate to contact us at the letterhead information or the contact information below.

Sincerely,

Davy Yul

Daniel A. Laudal Manager: Major Projects **UND Institute for Energy Studies** daniel.laudal@engr.und.edu 701-777-3456

#### **UNIVERSITY OF NORTH DAKOTA**

Institute for Energy Studies

#### **Budget Justification**

#### <u>Salaries</u>

Salary estimates are based on the scope of work. The labor rate used for specific personnel is based on their current salary rate, average labor rates are used for general labor categories. The personnel cost breakdown is listed in the table below.

Personnel	Role	Labor Rate Basis	Proposed Effort (%)	Base Salary	Labor Cost
	Senior				0.050
Michael Mann	Management	Annual salary	1	191840	1918
		• • • •			
Daniel Laudal	PI/PD	Annual salary	4	85000	3400
Xiaodong Hou	Laboratory Mgr	Annual salary	10	59322	5932
Research					
Engineer	Research Support	Average salary	12	75000	9000
Graduate	Student	Average salary	25	28000	7000
Undergraduate	Student	Average salary	37	12480	4654
Resource					
Manager	Administrative	Average salary	2	33705	674
Total					\$32,579

\*Any reference to hours worked on this grant is for budgeting purposes only. The University tracks employee's time on the basis of effort percentage and will not track or report employees time worked on this project in hours. Final numbers may not agree due to rounding.

#### **Fringe**

Fringe benefits are estimated for proposal purposes only, on award implementation, only the true cost of each individual's fringe benefit plan will be charged to the project. Fringe benefits are figured at a rate of 35% of total salary for all personnel, with the exception of graduate/undergraduate students which are figured at a rate of 5% of total salary. Estimates are based on historical averages.

#### Equipment

None

#### **Travel**

Two trips are planned to collect samples for the project. Two people for two days are budgeted for each trip. Departure from Grand Forks, ND with a destination at Underwood, ND is used for budgeting purposes. This represents a trip to the Falkirk Mine. We have estimated each trip to cost \$300/person, for a total of \$1200. Estimates are based on standard vehicle rental fees, mileage, standard per diems and lodging rates.

#### **Materials and Supplies**

\$1000 for materials and supplies is included in the budget - \$500 each specified for office supplies and sample storage supplies. Office supplies include materials such as paper, pens/pencils, ink, notebook, folders...etc. Sample storage supplies are sample containers for storage of samples collected during the project. Each of these categories are estimated based on experience.

#### **Other Direct Costs**

Other direct costs include sample analysis costs, such as hand held XRF, gamma ray core log and ICP-MS that will be performed at UND. Additional costs are included for sample preparation, primarily use of grinding equipment to grind samples prior to analysis. Costs are broken down in the following table.

Category	# Samples	\$/hr	\$/smpl	Total \$	Notes
XRF	120	24		1440	30 minutes/sample
Gamma Ray Core Logger	10	104		1040	
ICP-MS	35		313	10955	
Sample Prep	125	75		1562	10 minutes/sample

#### **Indirect Cost**

The indirect cost rate included in this proposal is the federally approved rate for the University of North Dakota 39%. Indirect costs are calculated based on the Modified Total Direct (MTDC), defined as the Total Direct costs of the project less individual items of equipment \$5000 or greater, subcontracts in excess of the first \$25,000 for each award, and graduate tuition waivers.

#### **Budget Summary**

An overall budget summary is contained on the following page.

BUDGET OUTLINE	F & A	39.00%
	Start date:	
	End date:	
DESCRIPTION		
SALARIES - REGULAR	19,006	
SALARIES - OTHER (Graduate)	11,654	
SALARIES - FACULTY	1,918	
FRINGE BENEFITS	7,906	
TOTAL PERSONNEL	40,485	
TRAVEL	1,200	
COMMUNICATIONS-PHONE		
COMMUNICATIONS-POSTAGE		
INSURANCE		
RENTS/LEASES-EQUIPMENT & OTHER		
RENTS/LEASES-BUILDING/LAND		
OFFICE SUPPLIES		
PRINTING-COPIES, DUPLICATING		
REPAIRS		
UTILITIES		
SUPPLIES-IT SOFTWARE		
SUPPLY/MATERIALS	1,000	
SUPPLIES-MISCELLANEOUS		
IT EQUIPMENT <\$5,000		
OTHER EQUIPMENT <\$5,000		
FEES-OPERATING FEES & SERVICES		
FEES-PROFESSIONAL FEES & SERVICES	14,997	
PROFESSIONAL DEVELOPMENT		
FOOD AND CLOTHING		
WAIVERS/SCHOLARSHPS/FELLOWSHPS		
TOTAL OPERATING	17,197	
EQUIPMENT >\$5,000		
TOTAL DIRECT COST	57,682	
TOTAL INDIRECT COST	22,496	
TOTAL COST	80,178	



June 29, 2017

Microbeam Technologies Inc. 4200 James Ray Drive, Suite 193 Grand Forks, ND 58202

Attention: Ms. Shuchita Patwardhan, Project Manager

Re: Support of the proposal entitled "Development of Low-Cost Rare Earth Element Analysis and Sorting Methods"

Dear Ms. Patwardhan:

This letter is a commitment of support to Microbeam Technologies Inc. (Microbeam) in its proposed project to develop and test methods to measure key elements that indicate the presence of Rare Earth Element Analysis (REE) in samples of coal and associated sediments.

Energy Technologies Inc. (ETI) is the largest supplier of on-line analyzers in North America with over 200 installations. Our state of the art, prompt-gamma neutron activation analyzers (PGNAA), are installed within full stream elemental analyzers (FSEA) which measure the chemical composition of material on a conveyor belt in real-time, providing the opportunity for sorting of feed materials based on composition. In the past, we have worked with Microbeam to apply the FSEA to "finger print" coal types that allow for sorting and blending of coal in order to optimize plant performance. Our system is able to successfully sort coals into groupings based on the coal compositional parameters developed by Microbeam. This system currently operating in this configuration at the 734 MW<sub>e</sub> Milton R. Young power station (Center, ND).

ETI's scope of work under this project is summarized below.

# Task 5 – PGNAA Calibration and Verification Testing

ETI will lead the PGNAA testing and calibration effort in this task using our existing analyzer/facility. Under this task, we will conduct the PGNAA testing of the five large samples in static belt mode to collect analysis data for calibration to the pXRF data and the REE-predictive algorithms. The PGNAA analysis calculations will be adjusted based on Microbeam's input as necessary to synchronize with the pXRF data. Once calibrated, verification testing will be performed that will include semi-continuous measurement of the partially pre-mixed coal (simulating delivery of truck loads to a commercial conveyor belt system). As a proof-of-concept of our integrated selective mining and feed coal sorting

methodology, in Phase I, we will demonstrate on-belt sorting tests with our REE algorithm-calibrated PGNAA using a semi-mixed feed coal with splits of known pXRF analysis and REE content.

In addition to scope of work for this project, we have an extensive database that covers a wide range of fuel properties which we will make available to this proposed project. We are committed to provide all the necessary relevant information regarding PGNAA analysis to Microbeam for this project. We will also work with Microbeam on developing the commercialization strategy in order for it to be efficiently implemented within existing mining infrastructure and procedures.

ETI is excited to team with Microbeam and UND on this project, and we look forward to a successful proposal outcome. The overall budget for the ETI scope of work is \$19,872.00, with an anticipated start date of September 1, 2017. If you have any questions, please do not hesitate to contact me by phone at (865)-927-9330 or by email at <u>david.swindell@energytechninc.com</u>.

Sincerely,

Dailk. AM

David Swindell President

# **APPENDIX C – LETTERS OF SUPPORT**



North Dakota Geological Survey

Edward C. Murphy - State Geologist Department of Mineral Resources Lynn D. Helms - Director North Dakota Industrial Commission

https://www.dmr.nd.gov/ndgs/

June 30, 2017

Ms. Shuchita Patwardhan Project Manager Microbeam Technologies Inc. 4200 James Ray Drive, Ste 193 Grand Forks, ND 58202

Re: Support of proposal "Development of Low-Cost Rare Earth Element Analysis and Sorting Methods"

#### Dear Ms. Patwardhan:

The North Dakota Geological Survey is pleased to support the proposal from the Microbeam, University of North Dakota and Energy Technologies Incorporated team to develop methods to identify, selectively mine, and sort coal rich in rare earth elements (REE). The proposed method identifies REE-rich coal layers in coal seams based on a unique REE enrichment marker algorithm integrated with selective mining and coal sorting that overcomes the challenge of dilution of REE-rich coal with coals that contain low levels of REE during the mining processes.

Recently, the NDGS has been working with the UND project team related to its efforts in development of methods to extract and concentrate the REEs from lignite-related feedstocks. We have supported those efforts by providing samples for analysis and testing and by providing insights into the geology of North Dakota lignites. As part of a separate NDIC-funded effort, we have been sampling areas in the western portion of the State, and have gathered a large number of samples with elevated REE-levels. We support the proposed project to the extent that it does not duplicate our ongoing efforts, but rather complements them.

We are interested in the proposed project because we feel it can improve the efficiency of field sampling efforts to identify REE-rich materials at lower cost. We will support the proposed project by assisting in sample collection, determining locations for sampling and in assisting with the understanding of the geochemistry of the samples and the depositional environments that led to REE-accumulations in the lignites.

We believe that, if successful, this project can significantly contribute to the development of an alternative and low cost domestic production of REEs and will reduce foreign dependence. It will also help the coal mines to find new markets for coal related products and we wish Microbeam and its team success in their effort.

If you have questions and require additional information please contact.



600 E Boulevard Ave - Dept 405, Bismarek, North Dakota 58505-0840 Phone (701)328-8000 Fax (701)328-8010



July 19, 2017

Ms. Shuchita Patwardhan Project Manager Microbeam Technologies Inc. 4200 James Ray Drive, Ste 193 Grand Forks, ND 58202

Re: Support of the proposal entitled "Development of Analyzers for Rare Earth Element Separations"

Dear Ms. Patwardhan:

Westmoreland Coal Company is pleased to support the proposal from the Microbeam, University of North Dakota and Energy Technologies Incorporated team to develop methods to identify, selectively mine, and sort coal rich in rare earth elements (REE).

This project targets the development of advanced sensor technology for field and in-process measurement of the rare earth element (REE) content within coal and coal-related materials. The proposed approach involves the integrated application of portable x-ray fluorescence (XRF) analyzer, REE enrichment marker algorithms, and in-process based prompt gamma neutron activation analysis (PGNAA) sensors that identifies and sorts REE-rich coals. The proposed method identifies REE-rich coal layers in coal seams based on a unique REE enrichment marker algorithm integrated with selective mining and coal sorting that overcomes the challenge of dilution of REE-rich coal with coals that contain low levels of REE during the mining processes.

Westmoreland Coal Company, through its subsidiaries operate lignite mining complexes that are of specific interest to this project. The lignite mines include Savage in Montana and the Beulah mine in North Dakota. As such, we are happy to help by providing the Microbeam team with access to available cores and/or core logs to assist in sample selection associated with mines we operate in Montana and North Dakota.

Westmoreland will work closely with the Microbeam team in the testing and development of the technology by providing access to coal samples and mines to test the technology. Westmoreland will also advise the team on the feasibility of the application of the methods to large scale mining operations.

We believe that, if successful, this project can significantly contribute to the development of an alternative and low cost domestic production of REEs and will reduce foreign dependence. It will also help the coal mines to find new markets for coal related products and we wish Microbeam and its team success in their effort.

If you have questions, or require additional information, please feel free to contact me at (720) 354-4541.

Sincerely,

Westmoreland Coal Company

Paul Fritzler Sales Director

# **APPENDIX D – ADDITIONAL FACILITIES AND EQUIPMENT**

# UND FACILITIES, EQUIPMENT AND OTHER RESOURCES

This project will take advantage of the exceptional laboratory-, bench- and pilot-scale equipment and facilities, as well as analytical laboratories available at the University of North Dakota Institute for Energy Studies (IES) and Energy & Environmental Research Center (EERC). A description of the equipment and facilities available to the proposed project are provided in the following sections.

# A. MATERIALS CHARACTERIZATION LABORATORY AND UND IES EQUIPMENT

### **Thermal Analysis Equipment**

**Simultaneous Thermogravimetric Analyzer/Differential Scanning Calorimeter (TGA/DSC):** UND has 2 TGA/DSC units, one of which operates under atmospheric conditions (TA Instruments) and the other can operate under pressure and with steam injection (Linseis Inc.). The information derived from the TGA will include any weight loss or gain with increasing temperatures, or isothermal operation when exposed to oxidizing and reducing environments. The DSC measures exothermic and endothermic transitions as a result of reactions, phase transformations, decompositions, and melting.

### **Scanning Electron Microscopes**

**FEI Quanta 650 FEG SEM**: Field emission SEM capable of obtaining high-resolution data from almost any sample material. This system was purchased in 2014. The instrument is operable in both high and low vacuum modes. The x-ray microanalysis system consists of an energy dispersive Bruker QUANTAX 200 x-ray detector. The system is equipped with backscattered and secondary electron imaging. The backscattered imaging allows for discerning materials based on atomic number. The presence of higher atomic number materials increases the brightness and allows for easy identification and subsequent analysis. The instrument is able to achieve 1-3 nm resolution. The imaging software package allows for performing analysis of mineral association with coal and other minerals.

*Hitachi Scanning Electron Microscope with an Energy Dispersive System (SEM/EDS).* The SEM is equipped with backscattered and secondary electron detectors for imaging and is automated with energy dispersive x-ray detectors for chemical composition analysis. The system can perform computer controlled scanning electron microscopy (CCSEM) of particles to determine the size, composition (major, minor, trace elements), and mineral typing. The system is also equipped to perform included/excluded analysis that provides information on association of minerals with coal particles or gangue materials. The system is also a good tool for examining the microstructure of the laser clad specimen, for examining the integrity at the clad/substrate interface, for determining microstructure of the laser melted surfaces, and for studying corrosion properties. This instrument allows samples to be viewed at a high magnification and to acquire information about the coating thickness, porosity, adhesion, microstructure analysis, and elemental composition.

### X-ray Fluorescence Spectrometers

**Rigaku Supermini 200** XRF: This XRF is a wavelength dispersive bench-top XRF able to provide low ppm detection limits for major, minor, and trace elements. The instrument is equipped with a 12 sample autosampler and can analyze either solids or liquids. The software allows rapid analysis of known and unknown samples. The system provides the ability to perform quantitative analysis and qualitative survey scans to identify the presence of elements. The system has been demonstrated to detect the REEs in a semi-quantitative analysis.

**Bruker Tracer IV Geo handheld XRF (pXRF):** The Tracer IV Geo is equipped with a large area silicon drift detector as well as a vacuum system for the analysis of lighter elements. This portable instrument can be taken to field sites. The flexibility of the system also allows for analysis of bulk samples (e.g., coal core samples, clays and other sediments for major elements) in the field without any sample preparation. This system has been used to identify REE 'marker' elements in REE-rich materials, such that it can be used to estimate the REE content of a sample without direct measurement of the REEs.

# X-ray Diffraction

The Rigaku SmartLab is a fully automated XRD that utilizes cross-beam optics (CBO) enabling fast and easy changing of the incident X-rays by substituting selection slits. The instrument can operate in either Bragg-Brentano or parallel beam focusing methods. The flexible design allows for analysis of samples ranging from loose powder to large pieces of sample. The instrument is equipped with both a scintillation acquisition. A Ka1 system with a monochromator is also available for high intensity measurements. The system is equipped with a CCD camera for imaging of specific areas on a sample and has a variety of stages allowing analysis of a wide array of sample types and applications. Once the x-ray diffraction pattern is obtained it is analyzed to determine the crystalline phases present. The system can also be used to perform quantitative XRD analysis.

# Gamma Ray Core Logger

UND IES has a OFITE SGR 740 Spectral Gamma Ray Core Logger, which measures the radiation that comes from the radioactive decay of uranium, thorium and potassium in core samples. The core gamma ray logger is built around a 6" wide by 7.5' long conveyor belt. The speed is variable from about 0.25 to 6 feet/minute. Slower speeds provide greater definition in the log of a small-diameter core. Belt spped and radiation data are collected by a PC. The belt is synchronized to the gamma ray readings with a shaft encoder interfaced to the PC. The gamma ray detector is a 3" by 3" NaI cystal mounted under the conveyor belt so that the distance between the core and detector will not vary with core size. During a test, the PC records the data from the core and displays in on-screen in real time. After the test, the data, including information about the core sample, is saved to disk for later recall. The unit can easily be trailer mounted for mobile lab testing, such as at a field sampling location or mine site.

### **Gravity Concentration Equipment**

UND IES has a bench-scale pulsed air jig particle separation system that can be used in the proposed project to prepare feedstocks for testing by recovering the lighter specific gravity fractions of the materials. The system is fully instrumented, uses continuous feed and discharge and has a throughput of about 50-100 lb/hr. The system will be available to the proposed project if required.

### **Continuous Flow Drying Equipment**

UND IES has a pilot-scale coal/sorbent drying system available to the project. It consists of an 11 inch rotary tube shell with external heaters, as depicted in Figure 1. It is capable of performing partial drying of coal (i.e. from as-received moisture to about 25% moisture) at a throughput of about 10 to 20 kg/hr.

### Sample Preparation

To take advantage of the above equipment, UND has a fully-equipped sample preparation lab, with all of the necessary capabilities for the sample preparation requirements contained in the proposed work.



Figure 1. UND IES coal/sorbent dryer

# **B. ANALYTICAL RESEARCH LABORATORY (UND EERC)**

The Analytical Research Laboratory (ARL) provides quality data, flexibility, and rapid turnaround time in support of research activities at the EERC. The lab is equipped for routine and specialized analyses of inorganic and organic constituents, which are performed using classical wet-chemistry and state-of-the-art instrumental procedures. Established analytical techniques allow for the chemical characterization of a variety of environmental and biological sample types, including fossil fuels, biomass, combustion by-products, geologic materials, fine particulate matter, groundwater, wastewater, fish tissue, and plant materials. Particular attention is directed toward trace element analysis, including arsenic, mercury, and selenium.

### Inductively Coupled Plasma – Mass Spectrometry (ICP-MS)

UND has two ICP-MS systems available. Both are the Thermo Electron iCAP SQ Quadrupole models from Fisher Scientific. The ICP-MS measures trace and major element analysis at the sub- part per trillion levels. Samples are prepared by a digestion method. To ensure total digestion and recovery of the trace elements, the sample is first ashed and then mixed with a borate fluxing agent (lithium metaborate and lithium tetraborate) and heated to  $1000^{\circ} - 1100 \text{ }^{\circ}\text{C}$  to form a glass bead. The bead is then dissolved in dilute acid and brought to a known volume with reagent water. The solution is analyzed by ICP-MS and results are reported on a  $\mu g/g$  (ppmw) or a dry whole sample basis and ash basis.

Additional tools of this laboratory include the following:

• 4200-ft<sup>2</sup>, fully equipped, exceedingly clean laboratory with seven fume hoods

- VG PQ ExCell inductively coupled plasma-mass spectrometer (ICP-MS) with collision cell technology
- Perkin Elmer Optima 2100 ICP-atomic emission spectroscope (AES)
- CETAC M6000A cold-vapor atomic absorption spectrometer (CVAAS) mercury analyzer
- PS Analytical Millennium Merlin cold-vapor atomic fluorescence spectrometer (CVAFS)
- PS Analytical Millennium Excalibur hydride generation atomic fluorescence spectrometer (HGAFS)
- Varian Spectra AA-880Z graphite furnace atomic absorption spectrometer (GFAAS)
- Mitsubishi TOX-100 chlorine analyzer with oxidative hydrolysis microcoulometry
- Dionex ISC3000 ion chromatograph (IC) with conductivity detection
- Dionex 2020i IC with UV-VIS, conductivity and electrochemical detection
- CEM MDS 2100 microwave with temperature and pressure control
- Shimadzu total organic carbon analyzer TOC-L

# C. FUEL ANALYSIS LABORATORY (UND EERC)

The Fuel Analysis Laboratory (FAL) is an integrated and fully equipped laboratory providing research support for many EERC research programs. In addition to performing proximate and ultimate analyses, the FAL provides a wide variety of testing: helium air pycnometry, surface area determination, laser particle sizing, dry and wet sieve analysis, and ash fusion. The lab analyzes coal samples from various state universities and institutions to determine the quality of the coal used in the boilers. The lab performs analyses according to ASTM International (ASTM) standards.

Laboratory capabilities include the following:

- Leco TGA-701 analyzer Thermogravimetric analysis includes moisture, volatile matter, and ash analysis. The volatile matter value is used to determine the coal rank.
- Leco TruSpec CHN analyzer Carbon and hydrogen values are used to determine the amount of oxygen required in combustion processes and calculations of efficiency of combustion processes. Nitrogen values can be used to evaluate the potential formation of nitrogen oxides as a source of atmospheric pollution.
- Leco TruSpec sulfur analyzer The sulfur analysis determines the total sulfur percentage of major elements in coal ash.
- Leco AC-350 isoperibol calorimeter The heating value is used to classify coal samples. It is also used to evaluate the effectiveness of beneficiation processes for research.
- Micromeritics Flowsorb surface area analyzer The surface area of granulated and powdered solids or porous materials is measured by determining the quantity of gas that adsorbs as a single layer of molecules on the sample.
- Micromeritics helium air pycnometer The multivolume pycnometer determines the skeletal density by measuring the reduction of gas volume in the sample chamber caused by the presence of the research sample.
- Malvern 2600 particle-size analyzer The sample is illuminated by the light from a low-power visible wavelength laser. The particles scatter some of the light at angles characteristic of their size, forming a series of diffraction patterns that are focused onto the detector and interfaced with the computer (size range is  $0.5-564 \mu m$ ).
- Fusibility of coal and coke ash furnace Ash fusibility temperatures predict whether the ash will perform properly in the process for which the coal was chosen.
- Facilities for sieving, grinding, and sample preparation The lab has a variety of coal and research sample preparation equipment. Physical tests are also performed, including wet and dry sieve analysis and bulk density.

# D. NATURAL MATERIALS ANALYTICAL RESEARCH LABORATORY (UND EERC)

The Natural Materials Analytical Research Laboratory (NMARL) offers analytical services designed specifically to address engineering problems in a wide range of fields. Analytical facilities combined with an experienced team of researchers provide a full range of advanced materials characterization and data interpretation. The laboratory is equipped with the following instrumentation:

- Scanning electron microscopes (SEMs). The NMARL has the following SEMs available: JEOL 5800 with a NORAN Instruments energy-dispersive x-ray detector system, GW Electronics enhanced backscatter detector, and NORAN Instruments Voyager IV microanalysis system and JEOL 5800 LV (LV is for manual control of the vacuum system to obtain a low vacuum) with a PRINCETON GAMMA TECH (PGT) SPIRIT Instruments energy-dispersive x-ray detector system and microanalysis system. Elements higher than atomic number 6 can be analyzed with an accuracy of 0.1 wt%. Standard and standardless quantification is available.
- X-ray fluorescence (XRF). Rigaku ZSX PRIMUS II is a wavelength-dispersive x-ray system that is good for elements above atomic number 6, with accuracies that can be attained to the ppm level (traditional reporting to 0.1 wt%). Standards must be available for elements to be quantified.
- X-ray diffraction (XRD). BRUKER AXS D8 ADVANCE is a state-of-the-art research-grade XRD instrument for conducting phase identification, ab initio structure determination, and quantitative phase analysis (QPA).

# E. PROCESS CHEMISTRY AND DEVELOPMENT LABORATORY (UND EERC)

The Process Chemistry and Development Laboratory has facilities for the analysis of different types of product and by-product streams. These analyses provide the data necessary for the calculation of material balances, conversions, and product qualities for several ongoing engineering projects at the EERC. Equipment is in place for ashing, solubility testing, numerous ASTM standard tests, coal cleaning, and a variety of general and specialized analytical testing, including wet chemical testing. To perform analyses, the laboratory utilizes the following equipment:

- Thermal analysis
  - Pressurized thermogravimetric analysis: TG-Minireactor capable of operation at up to 900°C and 600 psig
  - Thermogravimetric analysis: DuPont 951 thermogravimetric analyzer (TGA) interfaced with DuPont 2100 thermal analyzer
  - Differential scanning calorimetry: DuPont 910 differential scanning calorimeter (DSC) interfaced with DuPont 2100 thermal analyzer
  - Thermal reactivity: Cahn TGA System 113-DC and 1000 thermal reactivity system
  - Surface area: Micromeritics Flowsorb II 2300 (by arrangement with the UND Department of Chemical Engineering)
- Gas analysis
  - Hewlett-Packard 5880a refinery gas analyzer
  - HP-7890 GC with autosampler

- HP-7890 GC/MS with autosampler
- Liquids and solids analysis
  - Leeman Labs Model CE440 elemental analyzer
  - LECO sulfur analyzer
  - Lab Industries Karl Fischer titrator for trace water analysis
  - Bausch and Lomb Spectronic 20 spectrometer
  - Servall centrifuge
  - Perkin-Elmer/Coleman flame photometer
  - Abbe refractometer
  - ASTM D1160 distillation apparatus
  - ASTM D86 distillation apparatus
  - Coal washability equipment
  - ASTM and low-temperature ash furnaces
  - Haake RV100 Rotovisco viscometer
  - Hydrometers
  - Brookfield rheology viscometer
  - High-pressure liquid chromatograph
  - Waters Associates ALC/GPC 201 gradient liquid chromatograph
  - Hewlett-Packard 1040A diode array detector
- Ultraviolet spectrometry
  - Hitachi Model 340 UV Vis spectrometer
- Infrared spectroscopy
  - Perkin-Elmer 283 infrared spectrometer
  - Bomem 110c infrared spectrometer
- A full complement of micro and analytical balances and volume-measuring glassware

All laboratory as well as engineering and pilot-scale operational staff maintain offices and workstations complete with computerized systems for document creation, utilization, and control. These work areas are accessible by EERC personnel 24 hours per day, 7 days per week, as needed to complete project objectives.

The EERC designs and creates all machinery in-house for demonstrating and testing technologies. The EERC routinely has requirements for the design and construction of the highly specialized equipment. These requirements often involve using materials and equipment at or beyond normal design limits. In order to ensure the safety of researchers and the integrity of the research equipment, the EERC has a mechanical design group to address these needs. The design group uses Intel-based PCs as well as Sun workstations.

Engineering design packages currently being used by the mechanical design group include the following:

- AutoCAD drafting and solid modeling
- CAEPipe piping flexibility analysis using finite element analysis (FEA) method
- AutoSTAAD/MAX model preprocessor for structural analysis using FEA method
- STAADIII/ISDS structural analysis using FEA method
- ABAQUS general stress and heat-transfer analysis (linear and nonlinear) using FEA method
- Cosmos/M general stress and heat-transfer analysis using FEA method
- Cosmos/M Designer general stress and heat-transfer analysis using FEA method
- Fluent CFD applications using FEA method

- HyperMesh model preprocessor and postprocessor for FEA method
- MathCad general purpose math package
- Code26 software package developed in-house to design equipment in accordance with the American Society of Mechanical Engineers (ASME) Boiler and Pressure Vessel Codes Section VIII Div. 1 and Div. 2 and ASME Pressure Piping Code B31.3
- ZPrint rapid prototyping 3-D color printing of prototype models using ZCorp 3-D printer hardware

In the event that systems need repair or the need arises for the fabrication of new parts the EERC is fully qualified to design and build high-temperature/pressure reactors, sampling and measurement systems, and other specialized test facilities for performing research and development on a scale from micrograms to tons of processed material. Beyond fabrication the facility also maintains electronic control systems through its own electronics shop. The EERC has the capability to engineer and construct the following:

- Atmospheric and pressurized laminar-flow furnaces
- Pulverized-fuel combustors
- Emission controls: scrubbers, fabric filters, and electrostatic precipitators
- Atmospheric and pressurized fluidized-bed combustors
- Fixed-bed, fluidized-bed, and entrained-flow gasifiers
- Hot-gas filter test modules
- Processes for coal liquefaction, carbonization, and hydrothermal treatment
- Low-temperature oxygen plasma reactors
- Wastewater treatment reactors
- Cooling tower simulators
- Gas flow measurement and high-velocity thermocouple equipment
- Process instrument systems and gas sample conditioning equipment
- Specialized test well equipment

Access to these capabilities can be given within minutes of need. Scheduling of work needs/requirements for fabrication and controls are address on a weekly basis, more often if needed, and a full staff can be available around the clock.

# **APPENDIX E – RESUMES**

# Shuchita Patwardhan

# Education

Master of Science, Environmental Engineering University of North Dakota (UND), ND, USA

Bachelor of Engineering, Chemical Engineering University of Pune, India

# **Research and Professional Experience**

# Project Manager

May'17 - Present

# Microbeam Technologies Inc.

- Managed and developed projects to understand and minimize the impacts of fuel impurities on the performance of combustion and gasification systems. The combustion systems include cyclone fired combustion systems, pulverized coal fired systems, bubbling and circulating fluidized bed combustion systems, and biomass fired boilers. The gasification systems include high temperature entrained flow gasifiers, fixed bed and fluidized bed.
- Worked with fuel preparation and blending facilities equipped with prompt gamma neutron activation analysis (PGNAA) technologies for on-line fuel analysis to optimize fuel blending to improve plant performance.
- Reviewed plant design, operational parameters and Computer-Controlled Scanning Electron Microscopy (CCSEM) analysis results to identify solutions to operational challenges in coal fired power plants and developed an algorithm to enhance energy system performance.
- Identified business opportunities through discussion with potential clients and prepared proposals to meet client needs.

# **Research Engineer**

# Microbeam Technologies Inc.

- Performed analysis on coal, fly ash and fouling deposit samples to detect mineral impurities using computer controlled scanning electron microscopy and X-ray diffraction analysis.
- Designed laboratory as well as field scale experiments to determine the impacts of firing conditions on partitioning of fuel impurities between slag and entrained ash during combustion and gasification.
- Worked on projects associated with the analysis on coal byproducts for rare earth elements.
- Generated mathematical correlations for fuel properties, transport mechanisms, deposit growth and slag flow in high temperature combustion and gasification systems which are currently utilized in algorithms to predict the impact of fuel properties on energy system performance.

Feb'13 – April'17

Jun'06 - Jun'10

Aug'10 - Dec'12

- Administered the field work by coordinating with plant operators and managers to optimize field test conditions. Supervised team efforts for onsite material collection and storage.
- Supervised efforts on air flow rates and heat transfer simulations in high temperature combustion and gasification systems using computational fluid dynamics. Also determined phase separations using thermochemical equilibrium calculation to define the interactions between solid and liquid slag in order to better understand and predict slag viscosity-temperature profiles.

# Research Assistant

Jan'11 - Dec'12

# Department of Chemical Engineering, UND

Research Project Title - Evaluation of a Low Corrosion Method to Increase Mercury Oxidation and Scrubber Capture.

- Designed a lab scale reactor to conduct experiments in order to understand thermal desorption of multiple mercury forms from taconite pellets.
- Modified the temperature and form of mercury release by using various formulations of raw materials and additives which also facilitated the capture of mercury in the scrubber.
- Proved the ability of the carbon based additive to improve mercury oxidation by testing raw taconite material from five taconite production facilities.

# **Publications**

- Patwardhan, S., Evaluation of a Low Corrosion Method to Increase Mercury Oxidation and Scrubber Capture, Master's Thesis, University of North Dakota, 2012.
- Benson, S.A., Nasah, J., Thumbi, C., Patwardhan, S., Yarbrough, L., Feilen, H., Korom, S., and Srinivasachar, S., Evaluation of Scrubber Additives and Carbon Injection to Increase Mercury Control, Minnesota Dept. of Nat. Resources. St. Paul, MN, 67 p., 2012a.
- Benson, S.A., Lentz, N., Patwardhan, S., Nasah, J., Thumbi, C., Feilen, H. and Srinivasachar, S., Evaluation of a Low Corrosion Method to Increase Mercury Oxidation and Scrubber Capture. Minnesota Dept. of Nat. Resources. St. Paul, Minnesota: 66 p., 2012b.
- Benson, S.A., Patwardhan, S, Ruud, A., Freidt, A., Joun, J., Ash Formation and Partitioning in a Cyclone Fired Boiler, Presented at Impacts of Fuel Quality on Power Production Conference, Snowbird Utah, October 26-31, 2014.

# **Grants and Awards**

Small Business Technology Transfer (STTR) Project; Entitled "Spouted Fluid Beds for Mercury Removal from Coal", DOE AWARD NO. DE-SC0017060; 2017.

# David John Stadem

# Education

Augustana University, Sioux Falls, SD Bachelor of Arts in Chemistry

# Experience

#### Microbeam Technologies, Inc., Grand Forks, ND **Research Scientist**

- Assisted in management of projects to understand impacts of fuel impurities on the performance of cyclone fired combustion systems, bubbling and circulating fluidized bed gasification systems, and biomass fired boilers.
- Utilized MS Excel, Tableau, and custom-built data manipulation programs for data analysis and • interpretation
- Built, developed, and maintained algorithms and software programs; integrated software into DCS software • for customers in the ND lignite industry
- Performed advanced SEM-EPMA analysis on field samples as well as laboratory-produced samples; identified/quantified REE in SEM-EPMA analysis of samples.
- Used Bruker pXRF instrument for analysis of ND lignite coal samples; worked on method development for robust, accurate sample analysis.
- Worked in the field collecting samples; worked on accurate, representative sample ٠ collection/handling/storage procedures.
- Worked with colleagues on data analysis models using SPSS, FactSage, and Fluent. •
- Managing main office and laboratory.

# Novartis Animal Health, Inc., Larchwood, IA

**R&D** Scientist, Analytical Development

- Developed/improved USDA-compliant ELISA and other assays for use in the production of novel/existing • animal vaccines.
- Modified Specific Outlines (SO's) to maintain USDA compliance. .
- Drafted new SOPs and Quality Records (QRs) according to SO's in compliance with GMP-like standards. •
- Collaborated with Quality Control representatives to ensure compliance with internal and external • standards as well as remove barriers to successful tech transfer.
- Collaborated with Quality Assurance representatives to ensure compliance with internal and external • standards.

# Novartis Animal Health, Inc., Larchwood, IA

Lab Technician, Quality Control ELISA

Responsible for ELISA analysis of samples submitted to the OC ELISA department for antigen quantitation • and relative potency; communicate results to departmental associates and to internal customers; Reviewed records, SOPs, and Special Outlines to ensure consistency; drafted new SOPs and records according to Special Outlines in compliance with GxP-like standards; performed various tasks relating to upkeep of QC ELISA laboratory.

# Gevo, Inc., Luverne, MN

Analytical Chemist, Analytical

Prepared and analyzed samples in complex matrices for HPLC and GC analysis, recording and communicating data to analytical team and to internal customers; reviewed chromatograms diligently, recognizing and identifying chromatographic anomalies; learned trouble-shooting and periodic maintenance of GC and HPLC instruments; prepared reagents and standards, performed calibrations and restocked supplies to maintain an orderly lab setting.

June-Oct 2012

July 2013 – July 2014

Nov 2012 – June 2013

Oct 2014 – present

May 2012

### STEVEN A. BENSON President, Microbeam Technologies Inc. Associate VP for Research and Professor, University of North Dakota

#### Areas of Expertise

Dr. Benson's principal areas of interest and expertise include development and management of complex multidisciplinary education and research programs that are focused on educating the next generation energy experts and solving environmental and energy problems. These programs include: 1) technologies to improve the performance of fuel resource recovery, refining, conversion and environmental control systems; 2) transformations and control of fuel impurities in combustion and gasification systems; 3) carbon dioxide separation and capture technologies, 4) advanced analytical techniques to measure the chemical and physical transformations of inorganic species in gases; 5) computer-based models to predict the emissions and fate of pollutants from combustion and gasification systems; 6) advanced materials for power systems; 7) impacts of power system emissions on the environment; 8) national and international conferences and training programs; and 8) state and national environmental policy.

#### Education and Training

Minnesota State University	Chemistry	B.S. 1977
Pennsylvania State University	Fuel Science	Ph.D. 1987

#### **Research and Professional Experience**

- 2015 Present Associate Vice President for Research, Energy & Environmental Research Center, University of North Dakota -- Dr. Benson is responsible for developing and managing projects on the clean and efficient use of fossil and renewable fuels.
- 2010 2014 Director/Chair, Petroleum Engineering Program and Institute for Energy Studies coordinate energy related education and research activities that involve faculty, research staff, and student.
- 2008 Present Professor, University of North Dakota (Part time) -- Dr. Benson is responsible for teaching courses on energy production and associated environmental issues. Dr. Benson conducts research, development, and demonstration projects aimed at solving environmental, efficiency, and reliability problems associated with the utilization of fuel resources in refining/combustion/gasification systems that include: petroleum coke utilization, transformations of fuel impurities; carbon dioxide separation and capture technologies, advanced analytical techniques, and computer based models.
- 1999 2008 Senior Research Manager/Advisor, Energy & Environmental Research Center, University of North Dakota (EERC, UND) -- Dr. Benson is responsible for leading a group of about 30 highly specialized group of chemical, mechanical and civil engineers along with scientists whose aim is to develop and conduct projects and programs on combustion and gasification system performance, environmental control systems, the fate of pollutants, computer modeling, and health issues for clients worldwide.
- 1994 1999 Associate Director for Research, EERC, UND -- Dr. Benson was responsible for the direction and management of programs related to integrated energy and environmental systems development. Dr. Benson led a team of over 45 scientists, engineers, and technicians.
- 1991 Present President, Microbeam Technologies Incorporated (MTI) -- Dr. Benson is the founder of MTI whose mission is to conduct service analysis and consulting associated with efficient and clean combustion and gasification of fossil and renewable fuels. MTI began operations in 1992 and has conducted over <u>1500</u> projects for industry, government, and research organizations. MTI has extensive databases of fuel, fireside deposits, fly ash, and corrosion project that totals approximately 10,000 samples.

- 1989 1991 Assistant Professor of Geological Engineering, Department of Geology and Geological Engineering, UND -- Dr. Benson was responsible for teaching courses on fuel geochemistry, fuel/crude behavior in refining, combustion and gasification systems, and analytical methods of materials analysis.
- 1986 1994 Senior Research Manager, Fuels and Materials Science, EERC, UND -- Dr. Benson was responsible for management and supervision of research on the behavior of inorganic constituents in fuels in combustion and gasification.
- 1984 1986 Graduate Research Assistant, Fuel Science Program, Department of Materials Science and Engineering, The Pennsylvania State University, Mr. Benson took course work in fuel science, chemical engineering (at UND), and ceramic science and performed independent research leading to a Ph.D. in Fuel Science.
- 1983 1984 Research Supervisor, Distribution of Inorganics and Geochemistry, Coal Science Division, UND Energy Research Center -- He was responsible for management and supervision of research on coal geochemistry.
- 1977 1983 Research Chemist, Energy Resources Development Administration (ERDA) and U.S. Department of Energy Grand Forks Energy Technology Center, Grand Forks, North Dakota

# Selected Relevant Publications and Presentations

- 1. Laudal, D.,Benson, S.A., Recovery of Rare Earth Elements from North Dakota Lignite Coal and Related Feedstocks, Clearwater Clean Energy Conference, Cleawater Florida, 2017
- Benson, S.A., Patwardhan, S, Ruud, A., Freidt, A., Joun, J., Ash Formation and Partitioning in a Cyclone Fired Boiler, Presented at Impacts of Fuel Quality on Power Production Conference, Snowbird Utah, October 26-31, 2014. (Submitted to Fuel Processing Technology (in review)).
- 3. James, D.W., Krishnamoorthy, G., Benson, S.A., and Seames, W.S., "Modeling trace element partitioning during coal combustion," Fuel Processing Technology, 126 (2014) 284-297\
- Ma, Z.; Iman, F.; Lu, P.; Sears, R.; Vasquez, E.; Yan, L.; Kong, L.; Rokanuzzaman, A.S.; McCollor, D.P.; Benson, S.A. A comprehensive slagging and fouling prediction tool for coalfired boilers and its validation/application, Fuel Processing Technology 88 (2007) 1035–1043.
- 5. Matsuoka, K.; Suzuki, Y.; Eylands, K.E.; Benson, S.A.; Tomita, A. CCSEM Study of Ash-Forming Reactions During Lignite Gasification. *Fuel* 2006, *85*, 2371–2376.
- 6. Benson, S.A. and Laumb, M.L. Advances in Predicting Ash Behavior in Western Coal Fired Power Plants. In *Proceedings of the Symposium on Western Fuels: 20th International Conference on Lignite, Brown, and Subbituminous Coals Workshops*; Denver, CO, Oct 23, 2006.
- Benson, S.A.; Zygarlicke, C.J.; Steadman, E.N.; Karner, F.R. Geochemistry and Mineralogy of Fort Union Lignites. In Geology and Utilization of Fort Union Lignites; Finkelman, R.B.; Tewalt, S.J.; Daly, D.J., Eds.; Environmental and Coal Associates: Reston, VA, 1992; pp 111–120.
- Benson, S.A.; Falcone, S.K.; Karner, F.R. Elemental Distribution and Association with Inorganic and Organic Components in Two North Dakota Lignites. Prepr. Pap.—Am. Chem. Soc., Div. Fuel Chem. 1984, 29 (4), 36–47.
- 9. Benson, S.A.; Zygarlicke, C.J.; Karner, F.R. Distribution of Detrital, Authigenic, and Absorbed Inorganic Constituents in Lignite from the Beulah Mine, North Dakota. In Proceedings of the Rocky Mountain Coal Symposium; Haughton, R.L.; Clausen, E.N., Eds.; N.D. Geological Society, Report No. NB84-1; 1984; pp 22–27.

Patents – 4 patents issued and several applications pending

7,574,968 - Method and apparatus for capturing gas phase pollutants such as sulfur trioxide.

7,628,969 - Multifunctional abatement of air pollutants in flue gas.

7,981,835 -System and method for coproduction of activated carbon and steam/electricity.

8,277,542- Method for capturing mercury from flue gas

#### Daniel A. Laudal

#### Principal Areas of Expertise

Dr. Laudal's principal areas of expertise include rare earth element recovery processes, rare earth elements and coal geochemistry,  $CO_2$  capture, fuels characterization, advanced power generation systems and emissions control. He has specifically focused on process and system design and has worked with numerous types of lab, bench and pilot-scale systems. Dr. Laudal has more than nine years of experience working in large multidisciplinary and multi-organizational research projects.

Education and Training		
University of North Dakota	Chemical Engineering	B.S. 2006
University of North Dakota	Chemical Engineering	Ph.D. 2017

#### **Research and Professional Experience**

#### 2016-Present Manager: Major Projects, UND Institute for Energy Studies.

Primary roles include developing and writing major funding proposals, managing major research projects, coordinating IES research staff and students, and process design/development of innovative solutions to challenges in the energy industry. Primary research areas include recovery of rare earth elements from coal and coal byproducts, chemical looping combustion, post-combustion  $CO_2$  capture, novel gas/solid contacting reactor designs, and development of novel designs for the aging fleet of North Dakota University System steam generation plants.

#### 2012-2015 Research Engineer, UND Institute for Energy Studies.

Research areas included CO<sub>2</sub> capture, advanced fuel conversion systems and natural gas processing. Work included concept development, process design and testing of innovative solid-sorbent based technologies. Principal Investigator on multiple projects and key contributor on several successful research proposals. Lead research engineer on multiple projects relating to Chemical Looping Combustion (CLC) Technology. Developed concepts for innovative methods to characterize both the physical attrition and reactivity of oxygen carriers for CLC. Co-developer of a unique technology for segregation of oxygen carriers and fuel combustion products (ash, unburned char) in CLC, a significant challenge in advancing the technology. Developing new oxygen carrier compositions and optimizing process conditions to maximize fuel conversion and increase carrier durability. Lead research engineer developing UND's CACHYS<sup>TM</sup> technology for post-combustion CO<sub>2</sub> capture. Led the design, construction and testing of the small pilot-scale slipstream test system installed at the UND steam plant. Co-inventor and lead developer of a novel sorbent-based technology for capture and processing of associated natural gas for reduction of gas flaring from oil fields.

#### 2008-2012 Research Engineer, UND Energy & Environmental Research Center.

Research involved design and operation of various lab and pilot-scale gasification, combustion and advanced power systems. Lead researcher on a project aimed at developing a process for the production of hydrogen by catalytic hydrolysis of biomass. Gained invaluable experience with high pressure and high temperature systems and fluidized beds.

#### 2006-2008 Field Engineer, Schlumberger Oilfield Services.

Design, execution and evaluation of well cementing operations in the Williston Basin. Lead a team of 3-5 operators in performing various types of cement and work-over operations. Lead cement lab operator – designed, tested and validated cement compositions for each job.

#### **Publications/Reports/Presentations**

**Laudal, D**.; Benson, S. "Recovery of Rare Earth Elements from North Dakota Lignite Coal and Related Feedstocks." Conference Proceedings: 2017 Clearwater Clean Energy Conference. June 2017, Clearwater, Florida.

Van der Watt, J., **Laudal, D.** "Development of a spouted bed reactor for chemical looping combustion." Conference Proceedings: 2017 Clearwater Clean Energy Conference. June 2017, Clearwater, Florida.

Feilen, H., **Laudal, D.** "Development of an advanced oxygen carrier attrition characterization methodology for chemical looping combustion." Conference Proceedings: 2017 Clearwater Clean Energy Conference. June 2017, Clearwater, Florida.

Benson, S., **Laudal, D.** "Investigation of rare earth element extraction from North Dakota coal-related feedstocks." 2017 NETL Crosscutting Research & Analysis Portfolio Review. March 2017, Pittsburgh, PA.

Pei, P., Nasah, J., Solc, J., Korom, S. **Laudal, D**., Barse, K. "Investigation of the feasibility of underground coal gasification in North Dakota, United States." Energy Conversion and Management. Volume 113, 1 April 2016, pages 95-103.

Pei, P., **Laudal, D**., Nasah, J., Johnson, S., Ling, K. "Utilization of Aquifer Storage in Flare Gas Reduction." Journal of Natural Gas Science and Engineering. Volume 27, Part 2, November 2015, 1100-1108.

Benson, S., Srinivasachar, S, **Laudal, D**., Browers, B. "Evaluation of Carbon Dioxide Capture from Existing Coal Fired Plants by Hybrid Sorption using Solid Sorbents." Final Technical Report. US Department of Energy Award Number: DE-FE0007603. May 2015

Benson, S., Srinivasachar, S., **Laudal, D**. "CO<sub>2</sub> Capture Using Hybrid Sorption with Solid Sorbents (CACHYS<sup>TM</sup>)". Thirteenth Annual Conference on Carbon Capture, Utilization & Storage. April 2014.

Emerson, S., Zhu, T., Davis, T. Peles, A., She, Y., Willigan, R., Vanderspurt, T., Swanson, M., **Laudal**, **D**. "Liquid Phase Reforming of Woody Biomass to Hydrogen". International Journal of Hydrogen Energy, August 2013.

Swanson, M., Sondreal, E., **Laudal, D**., Hajicek, D., Henderson, A., Pavlish, B. "JV Task-129 Advanced Conversion Test – Bulgarian Lignite" US Department of Energy Cooperative Agreement No. DE-FC26-98FT40321. June 2009.

Swanson, M., **Laudal, D**. "Subtask 7.4 – Powder River Basin Subbituminous Coal-Biomass Cogasification Testing in a Transport Reactor." US Department of Energy Cooperative Agreement No. DE-FC26-98FT40320. May 2009

Swanson, M., **Laudal, D**. "Advanced High-Temperature, High-Pressure Transport Reactor Gasification" Period of 2005-2008. US Department of Energy Cooperative Agreement No. DE-FC26-05NT42605. December 2008.

### Synergistic Activities

Introduction to Mineral Processing Short Course - Colorado School of Mines

• Completed 2.0 Continuing Education Units – July, 2016

**Proposal Reviewer** 

• University Coalition for Fossil Energy Research

# David K. Swindell

Security Clearance: DoD Secret (Inactive), DOE Q (Inactive)

**Education**: University of Tennessee: B.S. Mechanical Engineering, 1981 University of Tennessee: M.S. Business Administration

# Experience:

Mr. Swindell has more than 30 years of experience in the technical and financial management of engineering projects and in maintenance and performance engineering, mechanical, and electrical design and analysis. He has performed project engineering functions in support of safety analysis assessments of Department of Energy (DOE) facilities and radioactive materials packages. He is experienced in the identification and development of new products and services for the defense, utility, mining, and other industries and with the design and analysis of American Society of Mechanical Engineers Section VIII pressure vessels.

# Employment:

<u>Energy Technologies Inc.</u> – 1994- Present As Chairman and CEO, Mr. Swindell is responsible for business management and development. He also serves as Radiation Safety Officer.

<u>Energy Technologies Inc.</u> - As President, Mr. Swindell managed the design, manufacture, and installation of instrumentation and associated services into the utility and mining industries. He led an investor backed group buyout of the division he built and managed at SAIC.

<u>SAIC</u> – 1987-1994 As Division Manager, Mr. Swindell built an instrument manufacturing and services business. He provided maintenance technical support and initiated and expanded preventive maintenance programs into compliance with the Institute of Nuclear Power Operations (INPO) and DOE guidelines. He also provided detailed structural and thermal analyses and performed project engineering functions in support of safety analysis assessments of mining, and other industries.

John B. Long Company (formerly Power Diagnostics, Inc) - As Vice President, Mr. Swindell's responsibilities included overseeing the engineering, manufacturing, and purchasing departments and supervising 31 employees. Products included underground mining equipment, coal handling and sampling systems, related instrumentation, and maintenance engineering services. He was also responsible for integrating PDI capabilities into the John B. Long Company. His projects included predictive maintenance sales and service, metal fabrication and electrical assembly, and PC-based monitoring systems.

<u>Technology for Energy Corporation</u> (TEC) - A Project Engineer, Mr. Swindell supervised design, manufacture, and installation of Reactor Vessel Level Monitoring and Reactor Vessel (In-Core) Power Monitoring Systems. His responsibilities included marketing and sales support, assembly of project teams, budgeting, cost control, and scheduling.

<u>Nuclear Assurance Corporation</u> (Norcross, GA) - Mr. Swindell prepared structural and mechanical analyses, specifications, procedures, manuals, and proposals for products and services offered. He prepared structural and mechanical analysis for inclusion in safety analysis reports for packaging for spent fuel transport packages, licensed under 10 CFR 71, and topical reports for spent fuel storage packages licensed under 10 CFR 72. These reports were submitted to nuclear utility customers and to the Nuclear Regulatory Commission.

<u>Combustion Engineering</u> (Chattanooga, TN) - Mr. Swindell managed research and development projects in the test laboratory in areas of secondary side flow, and thermal, structural, and vibration experimentation. He served as a key member of a design team working with civilian and naval engineers to incorporate advances in the commercial nuclear industry into the U.S. Naval Reactor Program. This included design and analyses of naval reactors as well as subsystems and preparation of specifications for implementation. Earlier, as an engineering student for Combustion Engineering, Mr. Swindell participated in a rotating training program with maintenance engineering, quality assurance, the nuclear test laboratory, and the machine design laboratory. These assignments included both production and research positions.