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Nondestructive high resolution composition mapping of additive manufactured materials for nuclear forensics analysis, and processing history analysis

TEXAS A&M UNIVERSITY

Motivation

The project will develop a non-destructive characterization method to map composition and fingerprint element distributions of additive manufactured nuclear materials, which are characteristics of specific processing and sources.

Advanced manufacturing methods, like additive manufacturing, are becoming common tools in rapid prototyping and deployment at industrial scales. The full spectrum of materials and forms is achievable ranging from ceramics and plastics to metals. The robust nature of additive manufacturing technologies pose a real challenge to non-proliferation efforts. Taking into account materials manufactured using advanced manufacturing technologies, key characteristic properties will be assessed taking advantage of ion beam analysis as a characterization method. Energy and type of ions will be optimized focusing on efficiency of signatures defining properties as well as products. Criteria for property-based alarm triggers will be developed. With ion and neutron options being available and evaluated, signatures will be developed and optimized with respect to efficiency of characterization – uniqueness of effects contributing to signatures characterizing both solid and liquid materials.

Methods

Nuclear materials for weapon applications, regardless of their processing details and resources, have certain fingerprints in both structures and compositions. The project will develop a unique method for nuclear forensics analysis of additive manufactured materials, towards determination of the origin and history of the materials, including processing history of additive manufacturing.

For this project, we will combine focused ion beam techniques with RBS (Rutherford backscattering spectrometry), PIXE (particle induced X-ray emission), NRA (nuclear reaction analysis), and ERD (elastic forward recoil detection) for a nondestructive high resolution composition analysis. We can obtain two dimensional mapping of composition of specimens at a spatial resolution of a few microns, but without any limitations on sample sizes. The analysis is nondestructive, which means other characterization such as localized transmission electron microscopy (TEM), scanning electron microscopy (SEM), hardness (through nanoindentation) and ductility measurement (through pillar compression), can be performed. This helps to link property variation to specific structural and composition details and to establish their correlations. By performing such 2-D composition analysis, in conjunction with other microscale characterization (indentation, pillar compression, transmission electron microscopy, atom probe tomography), we can obtain a comprehensive information on additive manufactured materials. Such information is valuable to identify the source of the materials, and shed lights onto processing details and even the purpose of materials applications.

Ion Beam Accelerator at Texas A&M University is the largest ion irradiation facility in the nation, equipped with five ion accelerators of terminal voltages of 10 keV to 3 MV. The lab has several unique capabilities including (1) an ion beam focusing system which is able to focus a beam (i.e. 2 MeV helium ions) from a typical beam spot size of a few millimeters down to a few microns, by using series of magnetic quadrupole lens system; (2) a high resolution RBS detector system which is able to reach an energy resolution of 1 keV, instead of >15 keV in traditional semiconductor solid state detector system. The high resolution is achieved through combination of a strong magnetic field and 2-D detector matrix; and (3) combination of RBS, PIXE, and NRA in one general chamber for a comprehensive analysis.

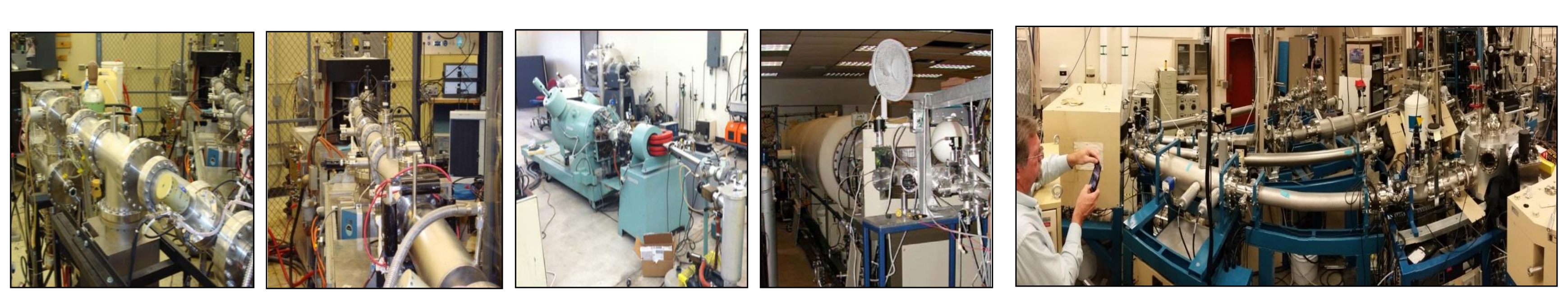

Figure 1. Five ion accelerators of various terminal voltages at Texas A&M University.

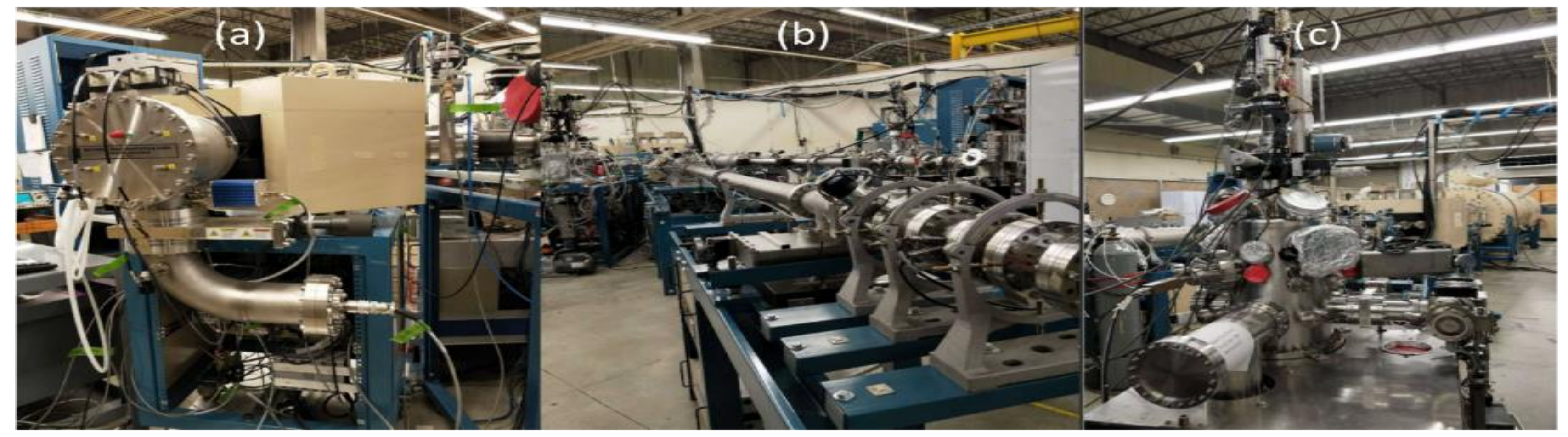

Figure 2. (a) High resolution RBS system, (b) ion beam focusing system, and (c) general purpose chamber of RBS+PIXE+NRA analysis.

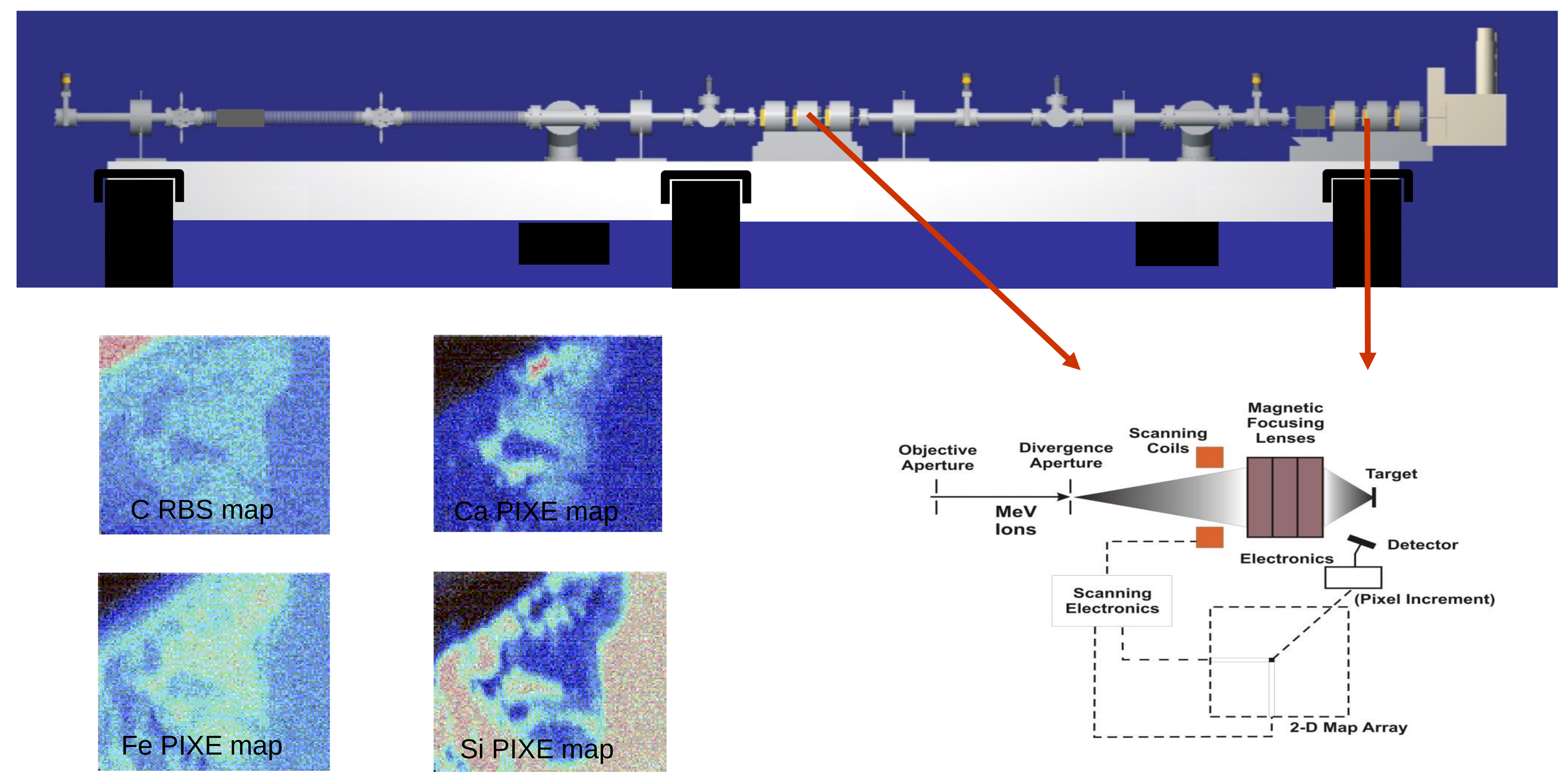

Figure 3. Micron beam characterization and elemental mapping of a rock over an area of 250 μm × 250 μm.

The project will develop the technique of micron proton beam induced X-ray fluorescence (μPIXRF). XRF uses characteristic X-ray emission for elemental analysis, upon excitation by a high energy X-ray beam. XRF has a sensitivity which is better than traditional Energy Dispersive X-Ray Spectroscopy (EDS, excited by electron bombardment). XRF offers a wide range of methodological advantages including (1) analysis from boron to uranium with high accuracy, (2) non-destructive, (3) high throughput, (4) rapid data collection, (5) no environmental contamination, and (6) highest quality and reproducibility standards. Figure 1 shows XRF yields as a function of X-ray energy for different elements, a schematic of the design and an image of a current commercial XRF device for soil contamination analysis.

The project will develop the technique of high resolution micron beam Rutherford backscattering spectrometry (μRBS). RBS measure the number and the energy of backscattered projectiles, to determine the concentrations and the atomic numbers of elements.

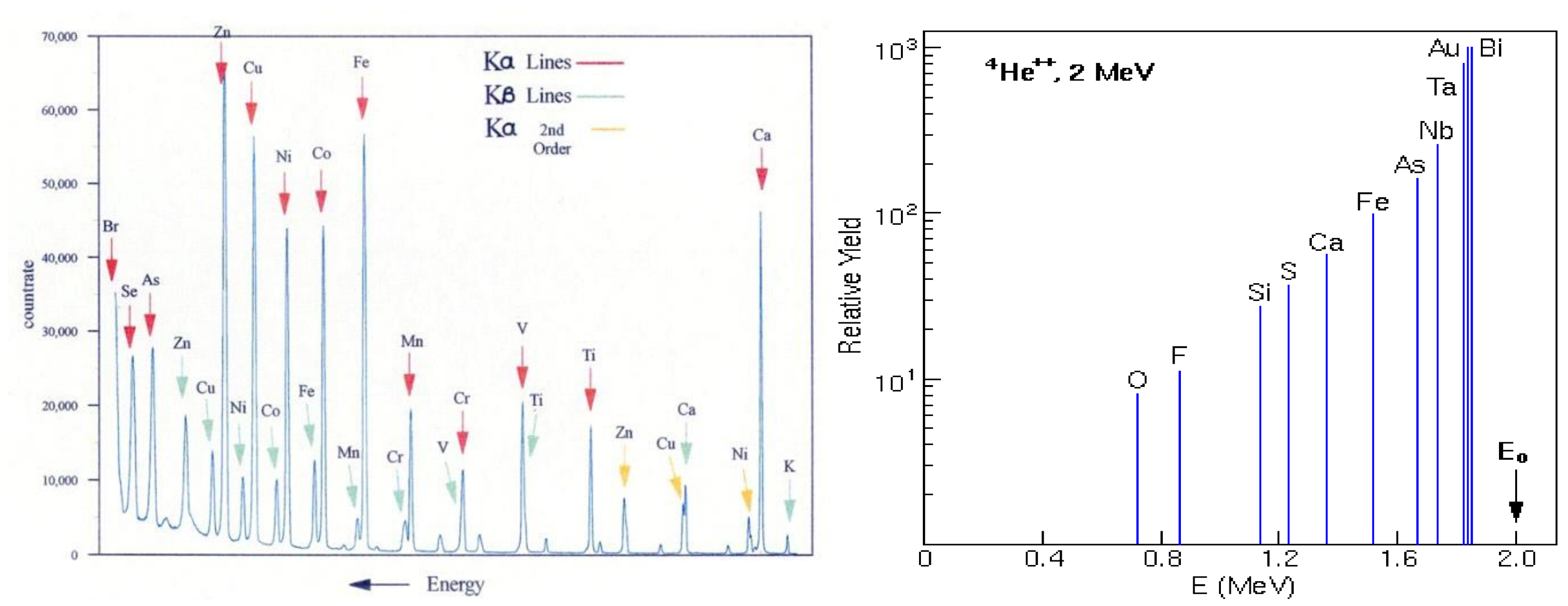

Figure 4. Typical (left) XRF spectra and (right) RBS spectra of various elements presented on a solid substrate.

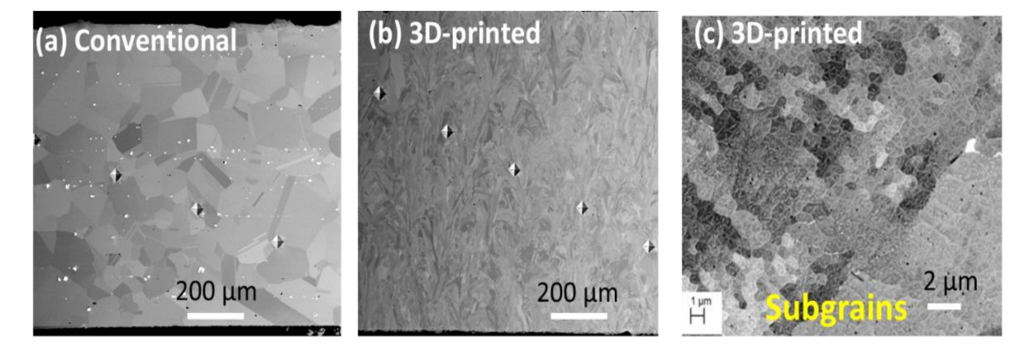

Figure 5. Additive manufactured materials

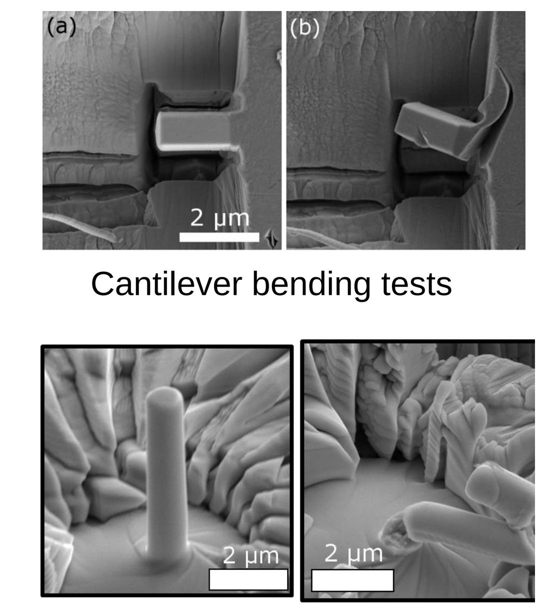

Figure 6. Micron scale mechanical testing

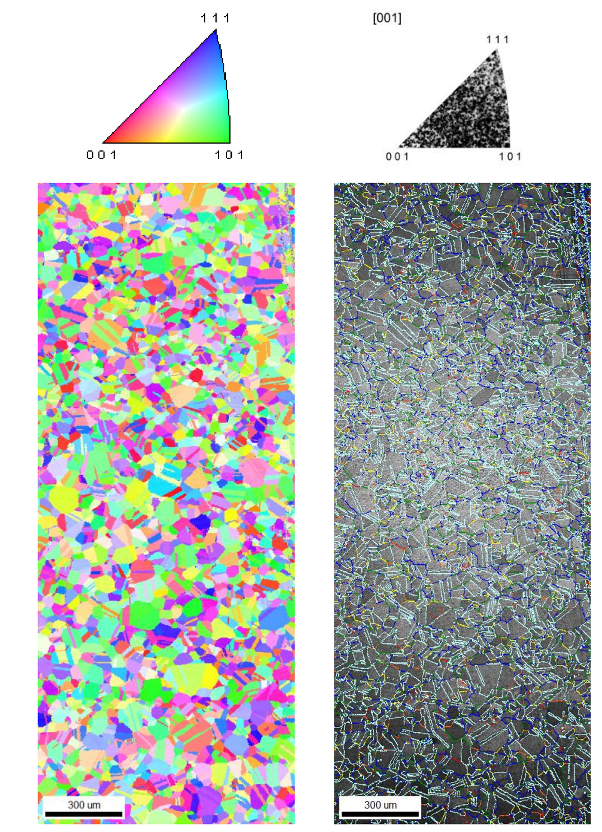
Figure 7. EBSD grain orientation mapping

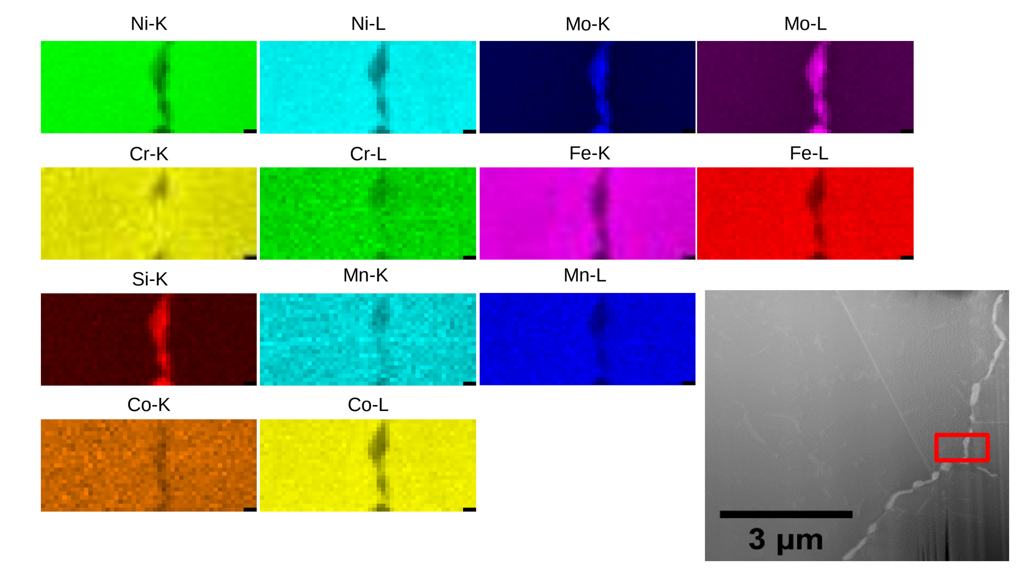

Figure 8. EDS elemental segregation mapping

Tasks

In collaboration with the project partners, this effort will consist of the following Tasks over 5 year:

Task 1: Advanced manufacturing methods screening and technology survey
- Database of advanced manufacturing methods and commercially available technologies
- Database of input/output materials involved in advanced manufacturing

Task 2: Nondestructive high-resolution composition analysis of advanced manufacturing technologies
- Advanced manufacturing samples library
- Sample interrogation using advanced nuclear methods (focused ion beam, RBS, PIXE, NRA, ERD)
- Sample mapping and advanced manufacturing signature development

Task 3: Nondestructive high resolution multi-dimensional nuclear signature method for advanced manufacturing technologies
- Nuclear signature-based data analytics and sample attribution in advanced manufacturing characterization
- Nuclear signature optimization
- Nuclear signature-based alarm triggers in advanced manufacturing technology domains
- Formulation of the nuclear signature method for advanced manufacturing technologies

Task 4: Sample-based nuclear-signature methodology demonstration
- Nuclear fuel cycle facilities including power plants and storage
- Supply chain streams associated with advanced manufacturing front-end
- Product streams associated with advanced manufacturing final products

NNSA National Nuclear Security Administration
Consortium for Enabling Technologies & Innovation