Contribution ID: **20** Type: **Oral**

Development of a FIB-SEM-ToF-SIMS microanalysis method for potential applications in the investigation of nuclear forensics samples and safeguards

We describe the development of a comprehensive microanalysis method using focussed ion beam- scanning electron microscopy (FIB-SEM) in combination with time-of-flight-secondary ion mass spectroscopy (ToF-SIMS) for potential applications in the investigation of nuclear forensic samples and safeguards. Possible materials for examination include bulk uranium and powder specimens with varying isotopic enrichments. The integration of FIB-SEM and ToF-SIMS instrumentation into a single microanalytical platform is still an emerging capability. Its use for the precise and accurate isotopic characterisation of discrete particles or grains within nuclear materials is an area we are just starting to explore at ANSTO. Analysis of these isotopic signatures is useful in deconvoluting the history of how nuclear materials are formed and it can also provide further insight during the investigation of unknown samples.

SIMS has been extensively used as a technique for the surface elemental mapping and depth profiling of materials at the nano-scale, with detection sensitivity from the ppm to ppb range. Recent developments in SEM and related FIB
instrumentation have led to advanced capabilities for performing in-situ SIMS measurements using a dual-beam FIB-SEM system. Microanalysis by FIB-SEM with ToF-SIMS involves using the ion beam to remove material at the sample surface by mechanical sputtering and ionisation. Secondary ions (SI) from the sputtered material are extracted and then pulsed through a ToF-SIMS analyser where they are separated according to their mass/Q ratio and the measured time of flight to the detector (Figure 1a). In comparison to energy dispersive X-ray spectroscopy (EDS), which is the more commonly used microanalysis technique in the SEM, FIB- based ToF-SIMS has a lower interaction volume allowing for high surface sensitivity and greater spatial resolution for isotope mapping (Figure 1b).

At ANSTO, a Xe plasma TESCAN FERA FIB-SEM is equipped with an EDS and a ToF-SIMS detector. Experiments have been carried out on a range of inactive materials that include bulk, thin film and powder samples. A typical workflow begins with high-resolution SEM imaging, followed by EDS and finally ToF-SIMS analysis of specific regions of interest for each material (Figure 2). ToF-SIMS spectra obtained in the negative and positive ion modes may reveal distinct compositional and isotopic variations in very small volumes (within nm3 to µm3 range). Comparison to EDS spectra can be used to confirm the elemental composition and the possible detection of contaminants in the material. From our initial measurements using inactive materials, we believe that the method can be successfully applied for the routine investigation of individual particles within a broad range of nuclear forensic and safeguards applications.

Primary author: KRACICA, Masturina (Nuclear Forensics Facility, Nuclear Stewardship, Nuclear Science and Technology Department, ANSTO)

Co-authors: OLDFIELD, Daniel (Electron Microscopy Facility, Nuclear Materials Characterisation and Development, Nuclear Science and Technology Department, ANSTO); YOUNG, Emma (Nuclear Forensics Facility, Nuclear Stewardship, Nuclear Science and Technology Department, ANSTO); KEEGAN, Liz (Nuclear Forensics Facility, Nuclear Stewardship, Nuclear Science and Technology Department, ANSTO); SHORT, Ken (Electron Microscopy Facility, Nuclear Materials Characterisation and Development, Nuclear Science and Technology Department, ANSTO)

Presenter: KRACICA, Masturina (Nuclear Forensics Facility, Nuclear Stewardship, Nuclear Science and Technology Department, ANSTO)

Session Classification: Oral Session #6 – Novel Techniques Applied to Nuclear Forensic Examinations

Track Classification: 1. Nuclear Forensics Capability Building: Initiation and Sustainability: 1.2 New Technologies, R&D and Signature Research in Nuclear Forensics