EDS and SEM Analysis to Expound Nuclear Target at IUAC,

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Introduction

Experimental insights into nuclear and astrophysical studies require advanced experimental facilities like high energy accelerators, target chambers and detectors with associated electronics. These experiments also demand enriched, pure and sustainable thin films to be used as targets. Required targets and their preparation methods chosen depends on the nature of material and purpose of experiment (reaction studies, spectroscopy or astrophysics). The fabricated targets can be examined before, as well as after experiments with different characterization techniques like Rutherford Backscattering Spectroscopy (RBS), Scanning Electron Microscopy (SEM), Energy Dispersive Spectroscopy (EDS), X-Ray Fluorescence(XRF), and X-Ray Diffraction (XRD) etc. [1,2]. EDS-SEM is a fast, non-destructive technique used to observe surface morphology and analyze elemental composition. This paper describes how this technique helps in understanding experimental isotopic targets, with examples.

EDS-SEM

Focused Electron Beam (FEB) in a voltage gradient system accelerate towards the target material. The electron interaction with material yields the various signals such as backscattered electron, secondary electron and X-rays. These signals contain wrathful information regarding surface morphology, topology and elemental fractional composition of the target sample. In SEM, surface morphology has been extensively studied for nuclear targets [1]. To associate an EDS technique with SEM system, an X-ray detector setup is installed. The FEB interaction with target atoms results the electron excitation of few nm depth from the surface of target. These excited electrons de-excite to ground state by emitting the characteristic X-rays. The characteristic X-rays of particular energy (shell) can reveal the many more properties of the target material. The quantification is by *K* ratio, where *K = X-ray Intensity of standard / X-ray intensity of measured*. Since, some correction factors in connection to electron-atom interaction-emission may have X-ray absorption (attenuation) during passage through, photoelectric absorption of primary X-rays and Bremsstrahlung (continuum) are introduced as *ZFC* correction and given C_{Speed} /C_{Std} = *KZAFc,* Where *Z* is the atomic number of target material; *A* is the absorption correction; *F* and *C* are the correction for characteristics and continuum.

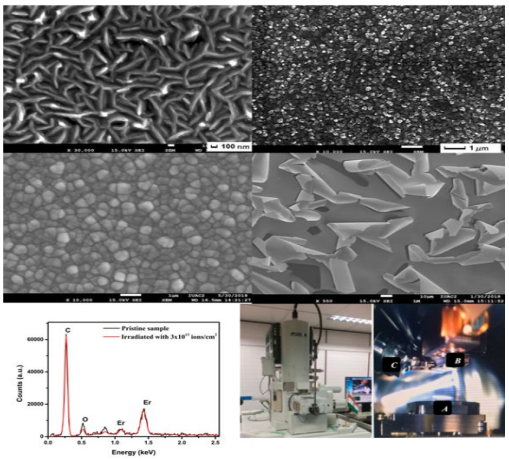

Fig 1.SEM images of (A) ^{130}Te after experiment and (B) before experiment. (C) ^{62}Ni. D) Au on Carbon. (E) EDS Spectra of C/Er/C. (F) FESEM facility. (G) SSD for EDS setup.

Experiment details

The SEM-EDS measurements were performed at Geochronology-FESEM facility at IUAC using JEOL's JSM-7610F FESEM with Octane Plus Silicon Drift Detector (SSD) for EDS attachment. Imaging was done at 15KV, 6nA at normal scan rate. EDS was performed at the current of 9nA with varying voltage (5-30KV), depending on target thickness. Samples were placed horizontal to FEB in vacuum (5.1e-5 Pa). Working distance was adjusted to 12 nm to ensure SSD gets full exposure for X-rays.

Table 1: Details of Sample * Rolling method

Samples	Deposition Method	Details	Ref
^{130}Te	Thermal	Carbon back	3
^{164,166}Er	Rolling* & Thermal	Free standing C cap/back	4 6
^{144,154}Sm	Electron gun	C cap &Al back	5
^{Nat}Ni ^{61,62}Ni	Thermal	Free standing C back	7
Pb, Ca	Thermal	C cap & back Au cap&back	8
Au	Thermal	$SiO_2/BaCl_2/C$	-

Results and Discussion

The ^{130}Te samples of 170-200 μm/cm^2 with carbon backing of 22 μm/cm^2 are found to be stable with ^{32}S/^{35}Cl beam (DC-Pulsed respectively) in the range of 70-155 MeV. The SEM-EDS investigation shows micron sized wrinkled surface with elemental composition remaining same, as shown in Fig 1A & 1B. The quantity of Er in carbon sandwiched sample is too low as compared to Er-cold rolled sample. Both samples are studied for radiation protection shielding with 180 MeV Si beam (thick Er sample) and 80 MeV ^{19}F beam (thin C-Er-C sample). Radiation stability and surface morphology of post irradiation modifications were studied using EDS and SEM. The ^{144,154}Sm films were prepared using electron gun evaporation and were suspected of having Ta or Cu as impurities, which might have come from the crucible during deposition.The EDS results shows no impurities and these samples were successfully used in main experiments. The ultra thin ^{nat}Ni free standing film was successfully prepared but in case of Sn, it was not possible to confirm by SEM observation. In Ni macro sized clusters were found held together whereas Sn sits on carbon as well separated nano particles. The parting agent contamination in Pb films is seen and $BaCl_2$ replaced by KCl yielded zero contamination as confirmed by EDS. The Ca thin film oxidizes immediately when exposed to atmosphere. The film without capping gets oxidized. Preventing it from oxidation to maximum extent can be achieved by Au capping rather than carbon capping. Thermally deposited Au on glass/$BaCl_2$/C was observed to be in broken clusters which could not be successfully floated. SEM images clearly show that deposited Au found in peel off from the carbon surface.

Conclusion

SEM-EDS studies are an effective, simple and non-destructive tool for target characterization. The post irradiation studies are more interesting to investigate the status of target after the experiment. Changes in the surface morphology could easily be seen: like material degradation and nano shapes appearance, and corresponding elemental composition at particular positions could be investigated. The EDS serves as complementary technique for RBS and XRF. In addition SEM allows to visually investigate the surface of the material.

References

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