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A Comparison of Oxide Thickness Measurements of Uranium Dioxide and Tantalum Pentoxide Using Both User-Acquired and Built-In EDS Standards

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In this work we describe thickness measurements from oxide layers on uranium and tantalum using Energy Dispersive Spectroscopy (EDS) combined with Oxford Instruments software calculating the layer thickness from measured X-ray intensities. A comparison of the oxide distribution is shown between (1) a user standardised system with an older SDD detector and INCA ThinFilmID and (2) the latest large area SDD detector and the AZtec LayerProbe software using built in standards (i.e. standardless analysis). As system (2) contains significantly improved routines for standardless analysis and better integration with the X-ray acquisition software it reduces the analysis time significantly over system (1).

There is general agreement that the presence of the oxide film on uranium metal mediates its interaction with hydrogen[1]. Consequently, models of the initial stages of the uranium-hydrogen reaction have been developed in terms of the hydrogen transport properties and thickness distribution of this surface oxide[2]. For the purposes of validation or quantification of such models, therefore, it is essential to have a good understanding of the way in which oxide films grow on this metal (as a function of oxidising conditions) and how the mean oxide thickness (and later thickness distribution) depends on both oxidising conditions and exposure time.

A Tantalum pentoxide standard BCR – 261T certified by the Institute for Reference Materials and Measurements (IRMM) was used to provide a oxide thickness reference to test both the user standardised system running ThinFilmID and the AZtec LayerProbe system using built-in standards. Ta2O5 was chosen over other metal oxides as it is available commercially as a well characterised heavy element oxide standard. Composition and estimated thickness values for the tantalum pentoxide and uranium dioxide samples were entered into the ThinFilmID and LayerProbe set-up files and using this data the optimum analysis conditions were deduced from calculations of the relative statistical variation at different beam voltages (kV)[3]. The intensity of the Oxygen line O-Kα at 0.52eV was used to calculate the oxide thickness.

On both systems measurement of the Si Kα signal at 1.740 keV from a bulk pure element standard of Si was used as a reference to normalise the X-ray count from the thin film samples and obtain measurements independent of beam current. For INCA ThinFilmID accuracy was improved by taking additional measurements on standards, again referenced to a measurement on the pure Si standard at the same beam energy. For AZtec LayerProbe, the factory default calibration was used. The results of the thickness measurements on the Ta_2O_5 standard (Table 1) show that system (1) and system (2) give essentially the same results which validates the improvements made to standardless analysis accuracy in AZtec LayerProbe Our comparative study shows AZtec LayerProbe as a viable technique to measure the

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thickness of native oxides on heavy elements. The data obtained will be used to further refine theoretical models of the oxidation and reaction kinetics of heavy elements such as Uranium.

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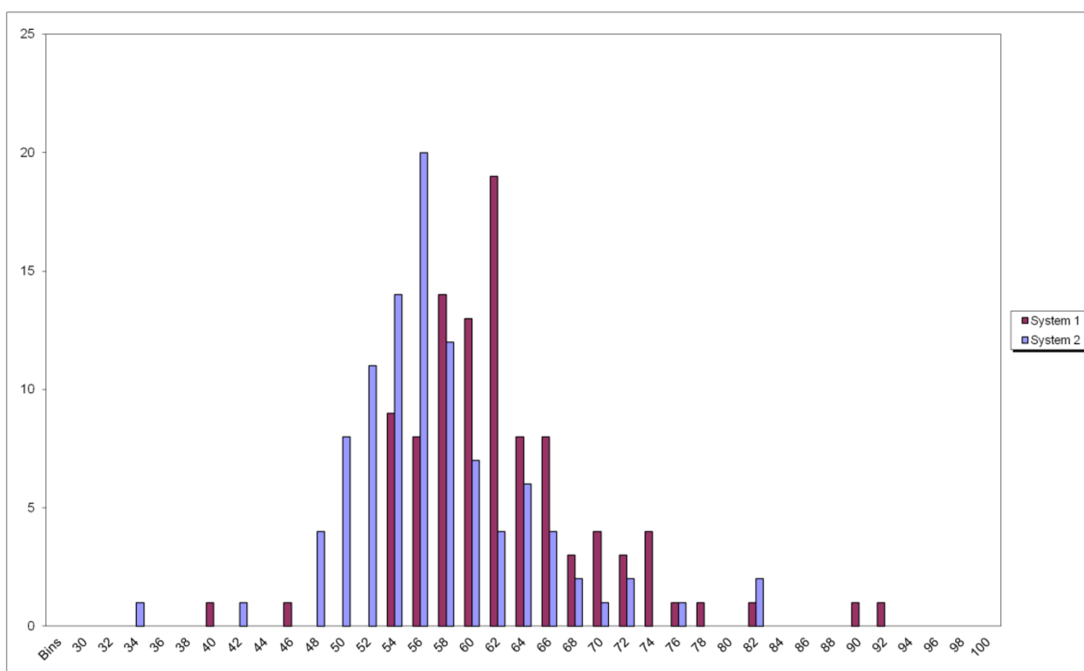

Figure 1 A comparison of the Uranium Dioxide thickness distribution between System 1 (AWE -INCA ThinFilmID) and System 2 (OINA-AZtec LayerProbe)

System	Mean (nm)	Std Dev	Min(nm)	Max(nm)
1 (AWE)	32.3	0.5	32.0	33.0
2 (OINA)	34.4	0.8	33.4	35.2

Table 1 Comparison of thickness measurements from a Tantalum Pentoxide (Ta_2O_5) reference material

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