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Nanoscopic study on the chemical species during uranium electrodeposition for alpha spectrometry

C. G. Méndez (1), H. Esparza-Ponce (1), D. C. Burciaga-Valencia (1), A.M. Beesley (2), L. Fuentes (1), L. Fuentes-Montero (1), M. E. Montero-Cabrera (1) *

 (1) Advanced Materials Research Center, Chihuahua, Mexico. e-mail:elena.montero.cimav.edu.mx
 (2) School of Chemical Engineering and Analytical Science, The University of Manchester, Sackville Street, Manchester, M60 1QD, United Kingdom
 *Corresponding author.

Abstract – Alpha spectrometry (AS) has applications in works requiring determination of actinide contents. AS sources produced by electrodeposition consist of a radioactive deposit onto a metallic substrate. Natural U sources prepared by the Hallstadius method have co-deposited Pt. The purpose of this work is to better understand how Pt and U are deposited. Grazing incidence XRD patterns and new XAFS spectra were obtained. The surface roughness of the sources for 20, 40 and 60 minutes of electrodeposition was evaluated by AFM. Comparison of AFM image analysis with data obtained by α spectrometry shows that the best time is 20 minutes.

Alpha spectrometry (AS) has applications in nuclear decay data measurements, environmental, geological and nuclear wastes analyses and in other studies requiring the determination of actinide contents. AS sources must be thin and uniform, producing small FWHM to obtain accurate measurements. AS sources produced by electrodeposition consist of a radioactive deposit onto a metallic substrate (cathode of the electrolytic cell). Natural U sources prepared by the Hallstadius method have co-deposited Pt, originated from the dissolution of the anode during the electrodeposition. Sources obtained from natural U hold a large element mass while short half-life-isotopes present scarce mass input that could be practically buried under Pt. Recently [1] have reported a study on the morphology and spatial distribution of the U/Pt deposits with the related chemical speciation of U, using SEM/EDX, XPS and XAFS.

The purpose of this work is to review the Pt and U deposition process. Sources are very thin, having a thickness about 500 U atoms by nm². We have obtained new spectra of the U LIII edge XAFS by total electron yield at Stanford Synchrotron Radiation Lightsource (SSRL). Grazing incidence (GI) XRD patterns were obtained at SSRL, BL 11-3. GI-XRD patterns show uniform crystal orientation of Pt, with grains ~ 4 nm; uranyl hydroxide diffraction signals suggest 001 texture of ~1 nm grains, i.e. with low crystallization. EXAFS spectra were fitted assuming the structure of uranyl hydroxide, according to the Hansen theory about actinide electrodeposition. U-U path shows low intensity that also may be a result of low crystallization.

The surface roughness (Rms) of the sources for 20, 40 and 60 minutes of electrodeposition was evaluated by AFM with a NanoScope v. IV. The roughness was evaluated in two zones, center and out of center. Table 1 presents the roughness average values for both zones. It may be observed that the roughness in the center increases for 40 but decreases for 60 minutes. However, the roughness out of center increases with the time of electrodeposition. Comparison of AFM image analysis with data obtained by α spectrometry shows that the best electrodeposition time is 20 minutes. In this case the roughness is homogenous on different areas of the source.

	Zone/time in minutes	20	40	60	
-	Center	29.599	55.050	37.992	
-	Out of center	29.685	58.186	94.231	
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 Table 1: Roughness average values in samples from the center and out of center.

Figure 1. Topographic images in the source center for 20, 40 and 60 minutes, respectively.

[1] A M Beesley et al doi:10.1016/j.apradiso.2009.03.031