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Increasing the Accuracy of EPMA of Microparticles Using Lower Energy Electron Beam and FIB Slicing

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Technique for increasing the accuracy of energy probe microanalysis (EPMA) of particles is proposed. EPMA with scanning electron microscope (SEM) and energy dispersive x-ray spectrometer is reasonable express method to search and analyze microparticles of nuclear materials. Accuracy of elemental analysis of uranium microparticles could be low if results are calculated with pre-installed commercial software. Such software provides good accuracy only for polished beam-perpendicular sample. The size this sample should be larger than free pass of incident electrons. Microparticles are not such samples.

Traditional technique of EPMA is to place microparticles on a planchet randomly and to analyze them by using electron beam with energy about 20÷30 keV. In this case results of analysis have not good accuracy. Measured concentrations of uranium for the set of microparticles manufactured of the same UO2 sample differ by more than 10%. Moreover systematic error of measure of elemental composition depends on a size of a particle. At the same time the difference of measured concentrations for individual particle could be up to 10% due to relative orientation of the particle, electron beam and X-ray detector.

Using lower electron beam energy (5÷8 keV) higher accuracy for the same particles obtained. Difference of measured concentrations is less 10% regardless of the particle size if this size is larger 0.5 μm.

Most modern SEM equipped with focused ion beam (FIB). Preparing of particles surface with FIB allows to get even higher accuracy of microanalysis. Particles of UO2 with the smallest size of 2 μm were sliced by using 30 kV Ga+ beam. Difference of measured concentrations for individual sliced particle decreases down to 1%. For the set of sliced particles this difference decreases down to 2%.

Country or International Organization

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