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Development of a SIMS Method for Measurement of Oxygen Isotope in Nuclear Forensic Applications

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Introduce:
As a new branch of forensic science, nuclear forensics was developed with combination of nuclear radiochemistry and forensics. It aims to identify the origin, transfer path, process and purpose of the seized nuclear materials by analyzing their characteristic attributes. The main characteristic properties are dimensions, isotopic composition, impurities and microstructure, and so on. But sometimes these properties do not enable accurate identification of the origin of the sample, signatures need to be found and analyzed.
The oxygen isotopic ratio 18O /16O varies ranging from 1% to 5% in nature[, , ,], and the oxygen element are concomitant with uranium ores and uranium products in the nuclear fuel cycle. So, the oxygen composition is usually regarded as one of these signatures. The classical method for analyzing the oxygen isotopic ratio need more sample consuming, complex chemical process and long measuring time. It is necessary to develop a reliable, fast technique to determine accurately the oxygen ratio in the uranium oxides.
SIMS is a convenient method for analyzing the oxygen isotope ratios in natural materials. The spatial resolution can reach 0.5-1 μm by the ion microprobe mode and the compositions, depth of tiny samples could be analyzed[ ]. So contaminated particles adhere to samples can be analyzed.

Experiments:
In the work, the sample was sputtered with magnetically filtered 133Cs+primary ions of 10 kV impact energy. The primary current was about 10 pA. The secondary accelerated volt was negative 5 kV.
To reduce the background contribution from residual atmospheric oxygen, the source housing was flushed with N2 gas until the background counts could not be detected.
Secondary ions 16O- and 18O- were detected by an electron multiplier (EM) with correction for dead time (25ns), the ion collection mode was peak jumping.
Oxidated metal-uranium sample was stuck to the graphite disk by conductor glue.

Result and discuss:
The intensity of secondary ion was almost linear with the primary ion current until it reached the maximum counts of EM. The measuring RSD of n(18O-)/n(16O-) was about 9.5% and 1% when the intensity of 18O- was 50˜100 and 100˜3000 counts per second respectively.
During sputtering, the intensity of secondary ion increased rapidly at the beginning of 300 seconds and the intensity became stable subsequently.
According to the measurement result of n(18O-)/n(16O-) , there were no different between using focused primary ion and unfocused one.
The results were same at the mass resolution of 300 and 2542, but the intensity of secondary ion at 2542 was only 20% of that at 300.

Summary:
These operational parameters of SIMS were studied for measuring the oxygen isotope ratio in metal-uranium and the method of SIMS was developed. The same sample was measured at different time, the value of n(18O-)/n(16O-) ratios were same. This proved that the method was reliable and suited to measure the oxygen isotope ratio for nuclear forensic science.

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