Effect of impact energy on U–Pb zircon dating by a sensitive high-resolution ion microprobe (SHRIMP-II)

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A sensitive high resolution ion-microprobe (SHRIMP-II) is the first instrument offering the possibility to derive spatially resolved age information from micro areas of polished samples, and has played a pivotal role in geochronology using accessory minerals such as zircon, monazite, titanite, and rutile and isotope geochemistry over the last 30 years. The calibration of ionization efficiency between Pb and U is based on the empirical observation that in homogeneous single-age zircon grains, the Pb^+/U^+ ratio is covariant with the UO^+/U^+ or UO_2^+/U^+ ratio. Although the empirical calibration has been used for several decades and in thousands of peer-reviewed publications, the mechanism is not well known. Similar calibrations are also used for the CAMECA IMS-1270/1280/1280 HR (hereafter, 12X0) series instruments, although UO_2/U is usually preferred over UO/U in the 12X0. The Pb ionization efficiency in the IMS series instruments is lower than in the SHRIMP under high vacuum conditions. However, this deficiency has been rectified by use of oxygen flooding in the IMS instruments, where ionization is improved by a factor of two. Oxygen flooding experiments in SHRIMP have been perfunctory but appear to indicate an improvement closer to 20% than 200%. The difference in Pb ionization efficiency during flooding remains unresolved. There are differences for the ion source geometries and potentials between SHRIMP and 12X0 in two main respects. First, the SHRIMP uses a two stage secondary ion extraction system, where the secondary ions are extracted from the sample at ~700 V, before being accelerated to 10 kV acceleration farther down the secondary ion column. In contrast, the IMS series instruments accelerate to the full secondary acceleration directly from the sample surface. The SHRIMP has a working distance of 2 mm, giving an extraction field of 350 V/mm, while the 12X0 instruments have a typical working distance of 5 mm, giving an extraction field of 2000 V/mm. The second difference is that the primary column of SHRIMP floats at the initial extraction potential (~700 V). The primary energy is set by biasing the duoplasmatron relative to the primary column. Thus, for 10 kV sample potential and 10 kV primary acceleration of negative ions, the primary beam feels only the 700 V difference between the sample and the extraction plate, namely impacting at 10,700 V. In the 12X0 series instruments, the primary column referes Earth ground, and the source is biased relative to ground. The impact energy for negative primary ions bombarding a positively biased sample is the sum of the primary and secondary acceleration. Therefore, a sample at +10,000 V and a duoplasmatron source at -13,000 V yield a primary ion impact energy of 23,000 V.

In this study, relationship between calibration from Pb^+/U^+ ratio to elemental Pb/U ration and the primary energy was assessed by using SHRIMP-IIe at the National Institute of Polar Research, Japan. The primary energy was adjusted at 10,000 V, 9,000 V, and 8,200 V with the secondary extraction voltage of ca. 800 V, namely impact energy of 10.800 V, 9,800 V, and 9,000 V, respectively. The transition of the primary energy dramatically changes the focus of ion optics, and voltage of condensor lons (objective lens) correlates with the primary energy. Intensities of the primary beam show relationship with the primary energy and then depth of analytical pit, namely sampling volume. The slope of relation curve beween Pb^+/U^+ and UO^+/U^+ is 2 among the various primary energy, and the Pb^+/U^+ ratio increases with decrease of the primary energy, which indicates that the ionization efficiency of Pb improve at the lower primary energy.