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Analytical Procedure for the Accurate U-Pb zircon dating by High Resolution Secondary Ion Mass Spectrometer

We report here new analytical procedures for the U-Pb zircon dating by a High Resolution Secondary Ion Mass Spectrometer, Cameca ims-1270 with multi-collection system. The most outstanding advance in U-Pb zircon dating has been achieved by the development of a Sensitive High Resolution Ion Micro Probe (SHRIMP), which is capable of measuring areas small as ~40 μm in diameter in zircon (e.g., Compston et al., 1984). In order to determine the U-Pb age from micro zircons (< 20 μm in diameter), we have improved the techniques using a focused primary ion beam and multi-collection method of secondary ions for Pb isotope analysis. Pursuing these improvements, we were confronted by the following difficulties.

1) Pb contamination from the sample surface: Applying the focused primary beam, a conical sputtering crater was formed and enlarged with time during the analysis. Pb contamination from the sample surface, therefore, significantly affected Pb isotope composition through the analysis. In order to overcome this problem, chemical washing was performed on polished samples with 0.1M HF and subsequently 0.5M HNO₃ for 1 and 3 minutes, respectively, in an ultrasonic bath just prior to gold coating. This procedure effectively reduced the 206Pb signal due to the surface contamination to negligible level (< 0.1 cps), which was comparable with the background of the electron multiplier.

2) Preparation of precise zircon standards: The key of accurate zircon dating by SIMS was how to prepare the reliable standards. For this purpose, two gem-quality zircon, 91500 zircon standard (1066 ± 2 Ma, 1σ) and Sri Lanka zircon (561 ± 2 Ma, 1σ), were prepared. These zircon standards were confirmed with homogeneity of major element composition, Pb/U atomic ratio and Pb isotope composition by SEM and Cameca ims-1270, and then precise 206Pb/235U and 208Pb/235U ratios were determined by conventional ID-TIMS method (Usui et al., 2002, in detail).

3) Direct calibration of Pb/U atomic ratio: The empirical linear calibration method using UO²⁻/U⁺ to determine Pb/U atomic ratio has been applied in the zircon dating by SHRIMP (e.g., Compston et al., 1984). We, however, did not obtain the significant variation of UO²⁻/U⁺ ratio to correct Pb/U atomic ratio using our standards, on the typical vacuum condition in the sample chamber of Cameca ims-1270 (~5 x 10⁻⁶ torr). We, therefore, applied direct Pb/U calibration by yielding Pb/UO²⁻ ratio from the standards and obtained a highly reliable linear calibration curve (Fig. 1).

In order to confirm our new analytical technique, the running standard of young zircon (Mutum, Brazil, donated by Dr. Sato, Univ. of Sao Paulo) was repeatedly analyzed. The obtained concordia age was 91.2 ± 2.6 Ma (2σ, n=16 weighted mean value), which is consistent with the age determined by the conventional ID-TIMS method (93 Ma). It is, therefore, concluded that the reliable age determination applying our new technique by Cameca ims-1270 can be obtained for young (< 100 Ma) and micro (< 20 μm) zircons.

Keywords: zircon, HR-SIMS, direct Pb/U calibration