PRECISE AGE DATING OF LUNAR ZIRCONOLITE WITH ELECTRON MICROPROBE.

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Introduction: Zirconolite ($CaZrTi_2O_7$) is an accessory mineral found in lunar basaltic [1-3] and granitic rocks [4]. It contains U (<~1 wt%), Th (<~2 wt%), and Pb (<~2 wt%) but negligible amounts of common Pb [5], apparently suitable for age dating. Zirconolite, however, in lunar rocks typically exhibits a needle-like morphology with a narrow width of approximately 1-2 µm. The thinness of zirconolite makes it challenging to use normal ion microprobe analysis for age dating. To overcome this limitation, electron microprobe analysis with its high spatial resolution (~1 µm at 15 keV accelerating voltage) has been employed for zirconolite dating [3, 4]. However, the precision of chemical dating using electron microprobe is lower compared to SIMS data. Recent advancements in trace element analysis using electron microprobe have shown potential for achieving higher precision in zirconolite dating. In this study, we present zirconolite crystallization age in a lunar granitic clast determined by electron microprobe with the MAN (mean atomic number) background correction and blank correction methods [6].

Methods: We studied a granophyric clast in the polished thin section of the DEW 12007 lunar meteorite. The clast consists of K-feldspar, silica, fayalitic olivine, and accessory phases including zircon, baddeleyite, zirconolite, tranquillityite, and apatite. Zirconolites exhibit a thin needle-like morphology (<~2 µm) with lengths ranging up to 100 µm. Mineral chemistry of zirconolite was analyzed using a field-emission electron probe microanalyzer (FE-EPMA; JEOL JXA-8530F) equipped with five wavelength dispersive X-ray spectrometers (WDS) at the Korea Polar Research Institute. Analytical conditions included an accelerating voltage of 15 kV, a beam current of 100 nA, and a beam size of 3 µm for standards and 1 µm for zirconolite. The MAN background correction and blank correction methods, implemented in the Probe for EPMA (PfE) software package, were employed to enhance data precision and reduce acquisition time. To establish precise MAN curves for all elements measured in this study, we selected 17 standard samples with average atomic numbers ranging from 18.7 to 35.2. X-ray peaks and high/low off-peak backgrounds were measured for 20 s and 10 s, respectively, from these standards. Detailed wavescans near the counting peaks and background positions were conducted to avoid potential interferences. Thanks to the MAN method, only X-ray peaks were measured for specific durations in lunar zirconolites: 20 s for Ti and Zr, 40 s for Ca and Fe, 100 s for Si, Al, and Y, 200 s for Ce, Cr, Mn, Mg, Nd, Sm, Dy, Ho, Er, Yb, Hf, and Nb, 300 s for Gd and Tb, 400 s for Th and Sc, 600 s for Tm and Pr, 700 s for La, 800 s for Eu, and 1000 s for U, Pb, Lu, and Ta. The detection limits for U, Th, and Pb were determined to be 30 ppm, 40 ppm, and 30 ppm, respectively. 91500 zircon standard [7] was used for blank correction for trace elements.

Results and Discussion: The zirconolites in the granophyric clast exhibit concentrations of ~4300 ppm, ~12500 ppm, and ~8000 ppm for U, Th, and Pb, respectively. Utilizing the method proposed by [8], assuming no common Pb, the crystallization age of the zirconolites was determined to be 4349 ± 23 (2SE, n=24) Ma. This chemical age is consistent with the Pb-Pb age of zircon (4340.9 ± 7.5 (2σ) Ma) determined by SHRIMP [9]. The precision and accuracy achieved in this study is significantly improved compared to previously reported chemical ages of lunar zirconolites measured with the electron microprobe [3, 4], thanks to the MAN background correction and blank correction methods. Because zirconolite is highly resistant to the electron beam, the precision can be further improve by employing a longer measurement time and/or higher beam current. Therefore, chemical age dating using the electron microprobe can be applicable to tiny U-Th-Pb bearing minerals in extraterrestrial materials, especially for return samples from Moon and Mars.

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