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Development of an in-situ K-Ar dating instrument

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We have been developing an in-situ dating method based on the K-Ar system for future planetary landing missions. The K-Ar dating method employs radiometric decay of 40K into 40Ar with half-life of 1.25 Gyr [Steiger & Jager, 1977]. Our system measures K and Ar with two techniques at the same laser irradiation spot on a sample: laser-induced breakdown spectroscopy (LIBS) and quadrupole mass spectrometry (QMS), respectively (LIBS-QMS system). Potassium and argon are extracted from a sample simultaneously by the laser ablation, in which the sample is vaporized by a series of intense ($>$ 1GW/cm2) laser pulses. We used a Nd:YAG laser with 6 ns of pulse width and 1064 nm of wavelength (Surelite I, Continuum). The laser energy was set at 100 mJ and the spot diameter was ˜500 micron. The pulse repetition rate was 2 Hz. We used a small spectrometer with a charge couple device (CCD) (HR 2000+, Ocean Photonics Inc.), to simulate a small and simple spectrometer for the spacecraft missions. The light emission from plasma was collected by a lens and transmitted through an optical fiber to the entrance slit of the spectrometer. The spectral acquisition time was 1 ms and the shutter was opened before the laser pulses reached the sample; time-integrated plasma emission was observed to simulate a non-gated operations on the planetary missions. The intensity of the K line at 769 nm was normalized by that of the O emission line at 777 nm in order to reduce signal fluctuations.

The gas extracted from the sample was purified with a Ti-Zr getter. The purified Ar gas was trapped on the charcoal trap cooled by liquid nitrogen. The Ar isotopes, 36Ar, 38Ar and 40Ar, are measured with the quadrupole mass spectrometer. Blank mass spectra were also acquired and subtracted from the main data. Finally, the volume of laser ablation pit was measured with a laser microscope to obtain the concentration of 40Ar within the pit.

In order to construct a calibration curve for K2O, 24 geologic samples with known K2O concentration were measured with our LIBS system. The calibration line can be fitted by a power law: I=0.11C$^{0.55}$-0.00686, where I and C are the signal intensity and K2O concentration (wt%). The detection limit and the quantification limit of our LIBS system were 300 ppm and 1 wt%, respectively. Also the detection limits of 36Ar and 40Ar were measured to be 2x10^{-12} and 2x10^{-11} [cm3 STP], respectively, in this study. As a result, if a rover encounters a rock with K2O=1 wt%, as Mars Exploration Rover found at Gusev crater, our instrument is expected to measure K and Ar from a rock sample; i.e., the error in LIBS measurement would be $<$20% and the S/N for QMS signals would be sufficient (=200).

Using our instrument, we measured three samples whose K concentrations and ages have been measured previously with flame photometry and a sector mass spectrometer: a hornblende (K2O=1.12 wt%, 1.75 Ga), a biotite (K2O=8.44 wt%, 1.79 Ga), and a plagioclase (K2O=1.42 wt%, 1.77 Ga) [Nagao, unpublished data]. We obtained the model ages of 2.1+/-0.3, 1.8+/-0.2, and 2.0+/-0.3 Ga, respectively.

Since the three samples have similar ages and different K concentrations, we should be able to construct a ”virtual” isochron by plotting the concentrations of K and 40Ar$_{rad}$. The slope of the isochron simulated with our experimental data yields 1.34 Ga of age. The data with known values yields 1.79 Ga. Such underestimation probably results from both overestimation for K and underestimation for 40Ar in the biotite data, which have large weight for the regression. Nevertheless, a positive correlation between [K] and [40Ar$_{rad}$] is obvious. Although further improvement in the accuracy of our measurements is necessary, the data obtained in this study demonstrate that our LIBS-QMS method can reproduce the trend essential for quantitative isochron-based age measurements.

Keywords: Decadal Survey, In-situ age measurements, Planetary landing missions, K-Ar dating