University of Alaska Fairbanks Geochronology Laboratory $^{40}Ar/^{39}Ar$ analytical methods

For each sample, 250-350 mg of 'whole rock' (phenocryst-free groundmass) or concentrated separates of hornblende or biotite were packaged in an aluminum foil tube and irradiated in position 5C at the McMaster University nuclear reactor, in Hamilton, Ontario. Approximately 20 samples were irradiated at a time. Six packages containing ~20 mg of the standard mineral mmhb-1 (Samson and Alexander, 1987) with an age of 523.5 Ma (Renne et al., 1994) were also irradiated with the samples to determine the irradiation parameter (J) and the flux gradient in the reactor. Samples and standards were analyzed 45 to 90 days after irradiation. Upon their return from the reactor, the sample and monitors were loaded into 2 mm diameter holes in a copper tray that was then loaded in a ultra-high vacuum extraction line. The monitors were fused, and samples heated, using a 6-watt argon-ion laser following the technique described in York et al. (1981), Layer et al. (1987) and Layer (2000). The irradiated samples were step-heated on-line in a Modifications Ltd. low-blank furnace. Temperature control was better than 5 degrees and a maximum temperature in excess of 1600o C was achievable to ensure complete sample fusion. The extracted argon was purified in a two-stage process using a liquid nitrogen cold finger and two SAES Zr-Al getters. Prior to measurement of the Ar, the gas was collected on an activated charcoal finger. The purified Ar gas was measured using a Nuclide 6-60-SGA 15 cm mass spectrometer. The sensitivity of the spectrometer is 6.5 x 10-15 mol/mV and system noise is generally around 0.02 mV. System blanks are generally better than 1 X 10-14 mol for 40Ar. Argon isotopic measurements for both samples and standards were corrected for the system blanks, for decay of 37Ar and 39Ar, and for reactor-induced isotopic interferences. Ages were calculated using the equations and corrections from McDougall and Harrison (1999) and the constants from Renne et al. (2010). All errors on analyses are reported at the 1-sigma level.

For each mass spectrometer analysis, five Ar isotope abundances are measured. 36Ar is used to determine the amount of atmospheric or initial Ar in the sample, 37Ar provides an estimation of the Ca content in the mineral, 38Ar provides an estimation of the Cl content, 39Ar reflects the K content and 40Ar is a mixture of initial and radiogenic Ar. The age of the sample is proportional to the ratio of the amount of radiogenic 40Ar to the amount of 39Ar produced by neutron bombardment from 40K.
All samples were dated by the 40Ar/39Ar step-heating method. In this method, a sample is heated to progressively higher temperatures in a double-vacuum resistance-heated furnace, and the argon isotopes are measured and age determined for each step (fraction). The integrated age is the age given by the total gas measured in all fractions and is equivalent to a potassium-argon (K-Ar) age. The fractions are commonly plotted on an age spectrum plot for analysis. A spectrum provides a plateau age if three or more consecutive gas fractions represent at least 50% of the total gas release and are within two standard deviations of each other (Mean Square Weighted Deviation less than ~2.5). If the fractions do not meet the criteria for a plateau due to intrafraction scatter in ages, then a weighted mean age of 'plateau-like' fractions, with each fraction weighted by the volume of 39Ar released, is calculated.

References

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