Analytical techniques

Zircon evaporation: Single zircons were handpicked after careful optical inspection and analyzed by the evaporation method (Kober 1987). ²⁰⁷Pb/²⁰⁶Pb isotopic ratios were measured on a Finnigan-MAT 261 mass spectrometer at the Max-Planck-Institut für Chemie in Mainz, and the procedures, as well as comparisons with conventional and ion-microprobe zircon dating, are detailed in Kröner *et al.* (1991) and Kröner & Hegner (1998).

The calculated ages and their uncertainties are based on the means of all ratios and their 2-s (mean) errors. Mean ages and errors for several zircons from the same sample are presented as weighted means of the entire data set. Repeated analysis of an internal zircon standard suggests an error of about 0.2 % as a best estimate for the reproducibility of the 207 Pb/ 206 Pb ages. In the case of combined data-sets the 2-s_m error may become very low, and whenever this error was less than the reproducibility of the internal standard, we have used the latter value (i.e. 0.2 %).

The ²⁰⁷Pb/²⁰⁶Pb spectra are shown in histograms that permit visual assessment of the data distribution. Due to the lack of U concentrations there is no *a priori* way to determine whether the zircons behaved as closed systems and whether the measured ²⁰⁷Pb/²⁰⁶Pb ratio reflects a concordant age. However, comparative studies using evaporation, conventional U/Pb dating, and ion-microprobe analysis have shown excellent agreement, even for zircons from complex metamorphic terrains (for references see Kröner *et al.* 2003; Schulmann *et al.* 2005).

SHRIMP II analyses: Single zircon grains from samples M02/107 and M05/287 were handpicked and mounted in epoxy resin, together with chips of the Perth Consortium zircon standard CZ3 (206 U/ 238 U age 564 Ma, U-content 551 ppm); the handling procedure is described in Kröner *et al.* (1999). Isotopic analyses of zircons from M02/107 were performed on the Perth Consortium SHRIMP II ion microprobe, whereas zircons from M05/287 were analyzed on the Beijing SHRIMP in the Chinese Academy of Geological Sciences. The analytical procedures are described in Compston *et al.* (1992), Claoué-Long *et al.* (1995), and raw data reduction followed the method described by Nelson (1997). CZ3 was used for Pb/U vs. UO/U calibration (Claoué-Long *et al.* 1995) on both the Perth and Beijing SHRIMP, and the calibration errors were 1% for the Perth data and 1.3 % for the Beijing data. Common Pb is considered to be surface-related (Kinny 1986), and corrections have been applied using the ²⁰⁴Pb-correction method and assuming the isotopic composition of Broken Hill lead (Cumming & Richards 1975). Errors for single analyses are based on counting statistics (6 mass-scans) and are at 1-sigma (63 % confidence), whereas pooled ages are reported at the 2-sigma level.

Sm-Nd isotopic analyses: Whole-rock powders were spiked with a 150 Nd- 149 Sm tracer solution and dissolved in a mixture of HF-HClO₄ (Hegner *et al.* 1995). The isotopic ratios

were measured on a Finnigan MAT 251 mass spectrometer at Munich University using a dynamic quadruple mass collection mode for Nd and a dynamic single cup collection mode for Sm. Total procedural blanks for both elements are <60 pg and negligible for the samples of this study. The ¹⁴³Nd/¹⁴⁴Nd ratios were normalized to ¹⁴⁶Nd/¹⁴⁴Nd = 0.7219 and Sm isotopic ratios to ¹⁴⁷Sm/¹⁵²Sm = 0.51086. The long-term average for ¹⁴³Nd/¹⁴⁴Nd in our Ames metal Nd solution is 0.512141 ± 11 (2- σ N=35) that corresponds to 0.511853 in the La Jolla Nd-standard. The ¹⁴⁷Sm/¹⁴⁴Nd ratios are precise to 0.2% as verified with the CalTech-Sm/Nd standard solution.

Multigrain zircon study (sample B611): Zircons were extracted from the crushed rock sample using standard heavy liquid and magnetic separation techniques. Hand-picked zircon crystals were air abraded with pyrite (Krogh 1982) at air pressures around 5.0 PSI, until about 20% of the grain size was removed. Samples (three multi-grain aliquots) were spiked with a 205 Pb- 233 U- 235 U tracer (Parrish & Krogh 1987) before digestion. Chemical separation followed that of Krogh (1973) with modification after Corfu & Noble (1992). Isotope ratios of Pb and U were measured respectively as Pb⁺ and UO₂⁺, using a VG-354 thermal ionization mass spectrometer (TIMS) at the Natural Environment Research Council Isotope Geosciences Laboratory (NIGL), UK. Procedural blanks for U and Pb from zircons are 0.4 and 10 pg, respectively. Errors on presented ratios are at the 1-sigma, and those on ages are at the 2-sigma limit. Errors were calculated after the method of Roddick (1987). Error ellipses plotted in the figures are at the 2-sigma limit. The decay constants of Steiger & Jager (1977) were used for age calculation, and corrections for common Pb were made using the values of Stacy & Kramers (1975), with an assumed uncertainty of 2.0%.

⁴⁰*Ar*-³⁹*Ar dating:* Biotite and amphibole grains were concentrated from crushed samples by standard density and magnetic techniques, followed by hand-picking. The mineral concentrates were wrapped in Al-foil packets and loaded in quartz ampoules together with monitors. After vacuum removal, the ampoules were soldered and placed in Al-containers. Irradiation was performed in the VEK-11 carrier of the VVR-K research reactor at the Technical University (Tomsk, Russia) during 40 hours (total fluxes of 5*10¹⁷ n×cm⁻²). Cadmium shielding, 0.5 mm thick, was used to reduce the thermal neutron influence received by the samples. Corrections for interfering nuclear reactions on K and Ca were monitored by including salts CaF₂ and K₂SO₄ in ampoules with mineral concentrates and monitors. Correction factors determined were (39 Ar/ 37 Ar)_{Ca} = 0.000808 ± 0.000032; (36 Ar/ 37 Ar)_{Ca} = 0.000346 ± 0.000038; (40 Ar/ 39 Ar)_K = 0.01500 ± 0.00023. The Russian standard sample for K-Ar dating - biotite MCA-11 - was used as a monitor. Conventional step heating experiments with biotite MCA-11 and hornblende MMhb-1 (split 6-52-1/4, reference age of 523.2 ± 0.9 Ma) (Spell & McDougall 2003) showed a former age of 313.7 ± 1.3 Ma.

Isotopic analyses of mineral concentrates and step heating experiments were performed using a MI-1200V mass-spectrometer operated statistically with an ion beam collected on a Faraday cup. Isotopic analyses were carried out at the United Institute of Geology, Geophysics and Mineralogy, Novosibirsk, Russia. Experiments were started at a temperature range of 450-550 °C and ended at 1100-1200 °C. Each analysis was corrected for mass discrimination, system blanks, decay of ³⁷Ar and ³⁹Ar, and interfering reactions on calcium and potassium during irradiation. Total blanks were ³⁷Ar - 1÷4×10⁻¹⁷ mol; ³⁹Ar - 0.5÷2.0×10⁻¹⁷ mol; ³⁶Ar - 5×10⁻¹⁶ mol (< 2%); ³⁸Ar - 9×10⁻¹⁷ mol (< 10%); ⁴⁰Ar - 15×10⁻¹⁴ mol (< 0,2%).

All ⁴⁰Ar-³⁹Ar ages have been calculated using the decay constants of Steiger & Jäger (1977). Three or more analytically indistinguishable contiguous steps comprising more then 50% of the total ³⁹Ar were used for calculation of plateau ages (Fleck *et al.* 1977). Total uncertainties have been calculated according to Dalrymple & Lanphere (1971).

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