

### **Tourmaline $^{40}\text{Ar}/^{39}\text{Ar}$ analysis**

Tourmalines were separated for  $^{40}\text{Ar}/^{39}\text{Ar}$  analysis by manually separating the cleanest sample from the pegmatites into a ca. 20 mg aliquot. After hand picking the cleanest tourmaline crystals without trace of alteration, inclusions or pre-existing cores, the selected tourmalines were leached in diluted HF for one minute and then thoroughly rinsed with distilled water in an ultrasonic cleaner.

Samples were loaded into a large well of one 1.9 cm diameter and 0.3 cm depth aluminium disc along with unrelated samples.

The disc containing the tourmaline sample was irradiated for 3 hours alongside FCs standards (Jourdan and Renne, 2007), for which an age of 28.294 Ma ( $\pm 0.13\%$ ) was used (Renne et al., 2011).

The disc was Cd-shielded (to minimize undesirable nuclear interference reactions) and irradiated in the Oregon State university nuclear reactor (USA) in central position. The mean J-value computed from standard grains within the small pits yielded values of 0.0009134 ( $\pm 0.67\%$ ). Mass discrimination was monitored using an automated air pipette and provided a mean value of 0.987368 ( $\pm 0.03\%$ ) per dalton (atomic mass unit). The correction factors for interfering isotopes were  $(^{39}\text{Ar}/^{37}\text{Ar})\text{Ca} = 6.95 \times 10^{-4}$  ( $\pm 1.3\%$ ),  $(^{36}\text{Ar}/^{37}\text{Ar})\text{Ca} = 2.65 \times 10^{-4}$  ( $\pm 0.84\%$ ) and  $(^{40}\text{Ar}/^{39}\text{Ar})\text{K} = 7.30 \times 10^{-4}$  ( $\pm 12.4\%$ ; Renne et al., 2013).

The  $^{40}\text{Ar}/^{39}\text{Ar}$  analyses were performed at the Western Australian Argon Isotope Facility at Curtin University. A multi-grain aliquot of tourmaline was step-heated using a continuous 100 W PhotonMachine© CO<sub>2</sub> (IR, 10.4  $\mu\text{m}$ ) laser fired on the

crystal population and jogged during 60 seconds. Each of the standard crystals was fused in a single step.

The gas was purified in an extra low-volume stainless steel extraction line of 240cc and using one SAES AP10 and one GP50 getter. Ar isotopes were measured in static mode using a low volume (600 cc) ARGUS VI mass spectrometer from Thermofisher© (Ware and Jourdan, 2018) set with a permanent resolution of ~200. Measurements were carried out in multi-collection mode using four faradays to measure mass 40 to 37 and a low background compact discrete dynode ion counter to measure mass 36. We measured the relative abundance of each mass simultaneously using 10 cycles of peak-hopping and 16 seconds of integration time for each mass. Detectors were calibrated to each other electronically and using Air shot beam signals. The raw data were processed using the ArArCALC software (Koppers, 2002) and the ages have been calculated using the decay constants recommended by Renne et al. (2011). Blanks were monitored every 3 to 4 steps. All parameters and relative abundance values are provided in in Annex 1 and have been corrected for blank, mass discrimination and radioactive decay. Individual errors in Annex 2 are given at the  $1\sigma$  level.

Our criteria for the determination of plateau are as follows: plateaus must include at least 70% of  $^{39}\text{Ar}$ . The plateau should be distributed over a minimum of 3 consecutive steps agreeing at 95% confidence level and satisfying a probability of fit (P) of at least 0.05. Plateau ages are given at the  $2\sigma$  level and are calculated using the mean of all the plateau steps, each weighted by the inverse variance of their individual

analytical error. A mini-plateau follows the same definition except it includes only between 50 and 70% of the total  $^{39}\text{Ar}$  released and is deemed less reliable than its >70% plateau counterpart. The final age uncertainties include all sources of uncertainties.

Our criteria for the determination of plateau are as follows: plateaus must include at least 70% of  $^{39}\text{Ar}$ . The plateau should be distributed over a minimum of 3 consecutive steps agreeing at 95% confidence level and satisfying a probability of fit(P) of at least 0.05. Plateau ages are given at the  $2\sigma$  level and are calculated using the mean of all the plateau steps, each weighted by the inverse variance of their individual analytical error. Mini-ages ( $2\sigma$ ) are calculated using the total gas released for each Ar isotope. Inverse isochron includes the maximum number of steps with a probability of fit  $\geq 0.05$ . The uncertainties on the  $^{40}\text{Ar}^*/^{39}\text{Ar}$  ratios of the monitors are included in the calculation of the integrated and plateau age uncertainties, but not the errors on the age of the monitor and on the decay constant (internal errors only, see discussion in (Min et al., 2000)).

### **Monazite U(-Th)-Pb analysis**

Monazite LA ICP-MS U-(Th)-Pb geochronology was carried out at State Key Laboratory for Mineral Deposits Research, School of Earth Sciences and Engineering, Nanjing University, utilizing a system consisting of ASI RESolution S-155 193nm ArF Excimer laser coupled to Thermo Scientific iCAP Qc quadrupole ICP-MS.

U/Pb age data were collected by ablating with laser beam diameters of 20  $\mu\text{m}$ , a beam energy density of  $\sim 3 \text{ J/cm}^2$  and a repetition rate of 3 Hz respectively. Each

analysis includes 20 s of background, 50 s ablation and 20 s of washout. With an ablation rate of ca 0.1  $\mu\text{m}/\text{pulse}$ , 50s ablation at the smallest beam diameter will produce an ablation crater with a diameter/depth ratio of  $<1$ . A fully analytical session typically consists of several blocks of 6 standard analyses (two monazite standard Treblicock, one monazite standard M4 ( $525.3 \pm 2.4$  Ma, Liu et al., 2012), two NIST610 standard glass, one NIST612 standard glass) followed by 8 unknown samples.  $^{207}\text{Pb}/^{206}\text{Pb}$ ,  $^{206}\text{Pb}/^{238}\text{U}$ ,  $^{207}\text{U}/^{235}\text{U}$  ( $^{235}\text{U} = ^{238}\text{U}/137.88$ ) and  $^{208}\text{Pb}/^{232}\text{Th}$  ratios were corrected using monazite standard Treblicock as an external standard (TIMS data at  $272 \pm 2$  Ma, Tomascak et al. 1996). Data reduction were undertaken using ICPMSDataCal (Liu et al., 2008) and Isoplot version 4.15 (Ludwig, 2003) was used to make concordia plots and calculate weighted averages.

### **Tourmaline major and trace element analysis**

Major elements of tourmaline were analyzed using a JEOL JXA 8230 electron microprobe at the Key Laboratory of Mineralogy and Metallogeny, Guangzhou Institute of Geochemistry, Chinese Academy of Sciences (GIG-CAS), Guangzhou, China. The operating conditions of an accelerating potential of 15 kV, a probe current of 20 nA and a beam diameter of 1  $\mu\text{m}$  were adopted for all elemental analysis. Peak and background counting times were 10 and 5s for Na, K, and F, 20 and 10s for Si, Al, Fe, Mg and Ca, and 40 and 20s for Ti and Mn. The standards used for analyses were kaersutite (for Si), rutile (for Ti), almandine garnet (for Al), magnetite (for Fe), olivine (for Mg), rhodonite (for Mn), diopside (for Ca), albite (for Na), orthoclase (for K), and  $\text{Ba}_2\text{F}$  (for F). Analytical results were reduced using the ZAF correction routines.

Relative precisions are better than  $\pm 5\%$ . Tourmaline structural formulae were calculated by normalizing to 15 cations in the tetrahedral and octahedral sites ( $T + Z + Y$ ) according to the suggestion of Henry and Dutrow (1996), based on the general formula  $XY_3Z_6T_6O_{18}(BO_3)_3V_3W$ , where  $X = Na^+, Ca^{2+}, K^+, \square[\text{vacancy}]$ ;  $Y = Mg^{2+}, Fe^{2+}, Mn^{2+}, Al^{3+}, Ti^{4+}$ ;  $Z = Al^{3+}, Mg^{2+}$ ;  $T = Si^{4+}, Al^{3+}, (B^{3+})$ ;  $V = OH^-, O^{2-}$ ; and  $W = OH^-, O^{2-}$  and  $F^-$  (Henry et al. 2011). The  $B_2O_3$  and  $H_2O$  contents were calculated based on the stoichiometry for  $B = 3$  apfu and  $OH + F = 4$  apfu.

Tourmaline trace elements were measured with an ELEMENT XR (Thermo Fisher Scientific) ICP-SF-MS coupled with a 193-nm (ArF) Resonetics RESolution M-50 laser ablation system in the State Key Laboratory of Isotope Geochemistry, GIG-CAS. Laser condition was set as following: beam size,  $45\mu m$ ; repetition rate, 5Hz; energy density,  $\sim 4 J cm^{-2}$ . A smoothing device (The Squid, Laurin Technic) was used to smooth the sample signal. Each spot analysis consisted of 20 s gas blank collection with the laser off, and 30 s sample signal detection with the laser on. Signals of the following elements were detected:  $^7Li$ ,  $^9Be$ ,  $^{31}P$ ,  $^{45}Sc$ ,  $^{51}V$ ,  $^{53}Cr$ ,  $^{55}Mn$ ,  $^{59}Co$ ,  $^{60}Ni$ ,  $^{63}Cu$ ,  $^{66}Zn$ ,  $^{71}Ga$ ,  $^{85}Rb$ ,  $^{88}Sr$ ,  $^{89}Y$ ,  $^{90}Zr$ ,  $^{93}Nb$ ,  $^{95}Mo$ ,  $^{107}Ag$ ,  $^{118}Sn$ ,  $^{133}Cs$ ,  $^{137}Ba$ ,  $^{139}La$ ,  $^{140}Ce$ ,  $^{141}Pr$ ,  $^{146}Nd$ ,  $^{149}Sm$ ,  $^{151}Eu$ ,  $^{157}Gd$ ,  $^{159}Tb$ ,  $^{161}Dy$ ,  $^{165}Ho$ ,  $^{166}Er$ ,  $^{169}Tm$ ,  $^{173}Yb$ ,  $^{175}Lu$ ,  $^{179}Hf$ ,  $^{181}Ta$ ,  $^{208}Pb$ ,  $^{232}Th$ . Si, pre-measured with EPMA, was selected as the internal standard element. NIST610 was used as the calibration standard. The oxide molecular yield, indicated by the  $^{238}U^{16}O/^{238}U$  ratio, was less than 0.3%. The detailed experiment procedure and data reduction strategy are described in Zhang et al. (2019). NIST612 was measured as unknown samples. 30 analyses of NIST612 indicate most

elements are within 8% of the reference values and the analytical precision (2RSD) was better than 10% for most elements.

### **Tourmaline Boron isotopes**

The in situ tourmaline boron isotopic compositions were measured on polished thin sections using a Neptune Plus MC-ICP-MS (Thermo Fisher Scientific) connected to a 193 nm excimer laser ablation system (Resolution M-50, Resonetics LLC, USA) at the State Key Laboratory of Isotope Geochemistry, GIG-CAS. The detailed description of the two instruments can be found in Zhang et al. (2014). An X skimmer cone in the interface was used to improve the instrumental sensitivity. Two Faraday cups L3 and H3 collected the signals of  $^{10}\text{B}^+$  and  $^{11}\text{B}^+$  statically and simultaneously. The laser parameters were set as follow: beam diameter, 33 $\mu\text{m}$ ; repetition rate, 6 Hz; energy density,  $\sim 4 \text{ J cm}^{-2}$ . Helium was chosen as the carrier gas (800 ml min $^{-1}$ ). Each analysis consisted of 400 cycles with an integration time of 0.131 s per cycle. The first 30 s was used to collect the gas background with the laser beam off, followed by 30 s laser ablation for sample signals collection with laser beam on. During the measurement of this study, the  $^{10}\text{B}$  and  $^{11}\text{B}$  signals of the gas background were less than 1 mv and 3 mv respectively, which were subtracted from the raw time-resolved signal intensities for each boron isotope. The mass bias of the instrument was calibrated using the standard-sample-bracketing method (SSB). The IAEA B4 standard ( $\delta^{11}\text{B} = (-8.71 \pm 0.18\text{‰})$ ) (Tonarini et al., 2003) was chosen as the external standard. The analytical quality was assessed by replicate analyses of tourmaline reference IMR RB1 (Hou et al., 2010). Twelve measurements of the IMR RB2

standard during the course of this study yielded a weighted mean of  $\delta^{11}\text{B} = -12.60 \pm 0.49\text{‰}$  (2SD), which is consistent within error with the reported value of  $-12.53 \pm 0.57\text{‰}$  (Hou et al., 2010).

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