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Apparent partial loss age spectra of Neoarchean hornblende (Murmansk Terrane, Kola Peninsula, Russia): the role of biotite inclusions revealed by $^{40}\text{Ar}/^{39}\text{Ar}$ laserprobe analysis

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ABSTRACT

Metamorphic hornblende frequently yields spectra with progressively increasing $^{40}\text{Ar}/^{39}\text{Ar}$ age steps, often interpreted as caused by partial resetting due to thermally activated radioactive argon loss by solid-state diffusion. Yet, in many cases rising Ca/K ratio spectra for such samples imply the presence of minor inclusions of K-contaminant minerals. To avoid parts of grains with mineral inclusions or compositional zoning we drilled tiny discs from thin sections under a petrographic microscope. Laser step-heating of drilled biotite-free hornblende discs yielded flat age and ratio spectra. In contrast, furnace step-heated hornblende separates from the same samples produced apparent loss age spectra. Moreover, biotite-free samples yielded flat spectra by laser and furnace dating. Consequently, apparent loss spectra result from degassing of included substantially younger biotite before its hornblende host during laboratory step-heating; c. 2.640 Ma hornblende ages constrain the Murmansk Terrane’s cooling.


Introduction

$^{40}\text{Ar}/^{39}\text{Ar}$ age spectra with progressively rising apparent ages have been widely interpreted as caused by partial argon loss by diffusion during younger tectono-tectonic reworking or slow cooling (Turner, 1969; Dallmeyer, 1975; Harrison and McDougall, 1980; Berry and McDougall, 1986; Wijbrans and McDougall, 1987; Lister and Baldwin, 1996). Younger apparent ages for the early gas release during laboratory step-heating experiments were assumed to reflect the intragrain spatial distribution of argon in samples. However, abundant evidence exists that a large portion of Ar release during step-heating of amphiboles under vacuum occurs due to chemical and structural changes within the crystals, rather than by volume diffusion (Gaber et al., 1988; Lee et al., 1991; Wartho et al., 1991; Wartho, 1995a). Consequently, Ar may be released simultaneously from cores and rims of crystals, leading to homogenisation of age gradients (Lee et al., 1990; Kelley and Turner, 1991; Lee, 1993). This clearly implies that age plateaux can not $a$ priori be interpreted simply as reflecting crystal lattices with homogeneously distributed Ar and that minerals have been unaffected by Ar loss or gain. It similarly brings into question the interpretation that age spectra with progressively increasing apparent ages may point to Ar loss by volume diffusion - the classic interpretation. Trends in $^{40}\text{Ar}/^{39}\text{Ar}$ age and Ca/K and Cl/K ratio - proxies for $^{37}\text{Ar}/^{39}\text{Ar}$ and $^{38}\text{Ar}/^{39}\text{Ar}$, respectively - spectra for hornblende are often related, pointing to degassing of a heterogeneous phase. This may be due to chemical zonation of hornblende, the presence of exsolution features and/or included contaminant minerals (Berger, 1975; Berry and McDougall, 1986; Harrison and Pringle Gerald, 1986; Onstott and Peacock, 1987; Onstott and Pringle-Goodell, 1988; Ross and Sharp, 1988; von Blanckenburg and Villa, 1988; Baldwin et al., 1990; Kelley and Turner, 1991; Lee, 1993; Rex et al., 1993; Lo and Onstott, 1995; Wartho, 1995b; Villa et al., 1996, 2000; Ahn and Cho, 1998; Belluso et al., 2000).

To shed further light on the phenomenon of apparent partial loss age spectra we concentrated on hornblendes from the Murmansk Terrane that experienced a tectono-metamorphic evolution of about 1 billion years. We combined classic furnace step-heating of hornblende and biotite separates with laserprobe step-heating of tiny discs that were drilled from carefully selected inclusion-free hornblende grains in thin sections under a petrographic microscope, using the technique of Verschure (1978).

Murmansk Terrane and the Lapland-Kola Orogen

The Murmansk Terrane (MT) is one of the Neoarchean terranes in the Palaeoproterozoic Lapland-Kola Orogen in the Kola Peninsula of Arctic European Russia (Fig. 1) and separated from the other terranes by the northwest-trending subvertical Murmansk Shear Zone (Fig. 2). The MT predominantly comprises amphibolite-facies, leucocratic, tonalitic, trondhjemitic to granodioritic gneisses and intrusives with...
subordinate metasedimentary material (Batiyeva and Bel’kov, 1968; Mitrofanov, 2001). The few Rb-Sr whole-rock isochrons and U-Pb zircon ages for tonalitic gneisses and a variety of enderbites to granites span 2.6-2.8 Ga (Vetrin, 1988; Pushkarev, 1990; Balashov et al., 1992). Sm-Nd (Dm) model ages are between 2.68 and 3.06 Ga (Timmerman and Daly, 1995; Timmerman, 1996).

Following major crustal stretching at ca. 2.45 Ga (Timmerman, 1996; Balagansky et al., 2001) the orogen was at least partly peneplaned in the earliest Palaeoproterozoic (Zagorodny, 1982; Sturt et al., 1994; Bridgwater et al., 2001). Subduction of oceanic crust led to
accretion of 1.96-1.91 Ga juvenile island arcs and terranes. Granulite- and amphibolite-facies metamorphism occurred in the orogen’s suture zone at 1.92-1.90 Ga (Timmerman, 1996; Daly et al., 2001). At about 1.76 Ga (Vetrin et al., 2002) stitching plutons intruded terrane boundaries (Figs. 1, 2). Mica, when not affected by excess or inherited argon, yielded 1.75-1.70 Ga 40Ar/39Ar plateau ages in the Central Kola and Belomorian Terranes and the northernmost part of the Archaean Karelian Craton (Fig. 1; de Jong et al., 1999).

**Petrography and mineral chemistry**

Coarse-grained tonalitic gneiss MT-10 truncates an ill-defined sub-vertical principal gneissic layering. The main constituents quartz, oligoclase (An 25-28) and microcline define a high-grade equilibrium microstructure with straight or slightly curved high-angle mutual boundaries, without preferred orientation. Quartz occurs as equi-dimensional inclusions in feldspar, or may be concentrated in small crystals between feldspar grains at triple or quadruple points. Blue-green hornblende (<500 µm perpendicular to c-axes) is a minor constituent. Crystals are optically unzoned and electron probe microanalysis (EPMA) indicates a homogeneous composition (Tab. 1; Fig. 3).

Small crystals (<100 µm) of greenish biotite are partially replacing hornblende along cleavage planes (biotite’s (001) parallel to amphibole’s (110)), lattice imperfections, or form thin aggregates along grain boundaries. In the matrix, 200-500 µm biotite crystals occur as aggregates with low-angle rational impingement boundaries forming a decussate equilibrium microstructure. Biotite occurs intergrown with retrograde titanite (with ilmenite cores), epidote/clinozoisite and minor calcite (locally as veinlets). Matrix titanites are 0.43-0.46 phlogopites with 0.29-0.42 tschermak exchange component p.f.u. (Tab. 1).

MT-11 forms a lineated and foliated amphibolite band in gneiss MT-10. Variations in modal amounts of preferentially orientated plagioclase and green hornblende define a gneissic layering at the scale of several millimeters, along which brown biotite crystals have grown. Quartz is rare. Hornblende crystals (>2500 µm wide) may show some zoning towards lighter shades of green in rims and fractures and inside crystals too, giving rise to a mottled effect. Hornblende is locally replaced by biotite along lattice imperfections, cleavages and grain boundaries.
MT-27 constitutes an amphibolite band in a tonalite. This massive coarse-grained rock virtually lacks deformation fabrics or preferred orientation, but contains cm-thick boudins and isolated fold hinges of intensely deformed plagioclase veins. It comprises moss-green hornblende, plagioclase (with garnet inclusions) and quartz. It contains substantial amounts of salitic clinopyroxene that is replaced by hornblende along grain boundaries and parting and cleavage planes. Epidote is a rare constituent; titanite and biotite are absent. Hornblende (>3500 µm in cross section) may show some zoning towards lighter green colours in rims and fractures, as well as around included clinopyroxene relics. EPMA revealed an increasing actinolite component in hornblende toward such lighter coloured zones, yielding lower K, Na and Ti contents and much higher Ca/K ratios in these areas (Tab. 1; Fig. 3). Actinolite-rich zones are about 5-25 µm wide (Fig. 4), whereas individual actinolite inclusions and newly formed euhedral crystals measure up to 20 µm.
Many actinolite-rich zones in hornblende MT-10 and MT-11 contain biotite, but not in MT-27. This shows that biotite was not produced during retrogressive growth of actinolite in hornblende. The confinement of biotite to grain boundaries and lattice imperfections in hornblende imply localised ingress of an aqueous fluid with an increased activity of K ions. As biotite-bearing quartz-feldspar gneisses surrounding the amphibolites are K richer, the localised hydration implies an open system on at least the scale of several dm to metres.
Experimental Procedures

Argon isotopes were extracted from hornblende (MT-10, MT-11 and MT-27) and biotite separates by step-heating in a double-vacuum, resistance-heated furnace and measured on an AEI MS10 mass spectrometer at Leeds University. Mineral separates were prepared by handpicking the 75-100 µm size fraction obtained by standard procedures.

Ca. 1.5 mm diameter discs were drilled from much larger hornblende crystals (MT-11 and MT-27) in polished thin sections and step-heated with a continuous laser. The discs were thoroughly rinsed in acetone and subsequently in distilled water after being detached from the thin section glass support. The laserprobe in Amsterdam comprises a Spectra Physics 24 W argon ion laser and an MAP 215-50 noble gas mass spectrometer. To avoid uneven heating of the discs the laser beam was defocused to a focal distance of 580-590 mm (f:500 achromatic focusing lens) yielding a ca. 2 mm diameter laser spot that was truncated at the edges with an iris diaphragm to remove the low-energy halo around the beam.

Samples and neutron flux monitors were irradiated for 30 hours at the ECN/EU research reactor (Petten, Netherlands). Mineral separates in high purity aluminium foil envelopes were loaded into a Spectrosil phial for irradiation. Drilled hornblende discs were directly loaded in holes in a 22 mm diameter aluminium tray in a 25 mm OD standard irradiation cans. The ($^{36}\text{Ar}/^{37}\text{Ar})_C$, ($^{39}\text{Ar}/^{37}\text{Ar})_C$ and ($^{40}\text{Ar}/^{39}\text{Ar})_K$ ratios used in the corrections for Ca- and K-derived Ar isotopes produced in the unshielded High Flux Pool Side Isotope Facility (HFPIF) are: 0.000273, 0.000699 and 0.06051, respectively. Flux gradients were about 5% over the length of the cans and below 0.4% horizontally within the tray.

Experimental details are given in the footnotes to Tables 2 and 3 with $^{40}\text{Ar}/^{39}\text{Ar}$ analytical data.

Results

Furnace step-heating of hornblende separates yielded single, sharp peaks in the 950-1075°C range (Tab. 2; Fig. 5). Cl/K and Ca/K ratios referring to hornblende’s main degassing cluster tightly in chemical correlation diagrams (Fig. 6).

Hornblende separates MT-10 and MT-11 yielded spectra with progressively increasing Ca/K ratios and apparent ages to about 2.6 Ga, following an excessively old first increment for the latter sample, also observed for drilled grain MT-11 (Tabs. 2, 3; Figs. 7a, b). The first increment of MT-10 is as young as the youngest biotites of the MT, like MT-11 (Tab. 2; Figs. 7a, b). Ca/K ratios of 6-6.5 for separate MT-10 (970-1085°C range) are comparable to values of 6.1-7.7 obtained by EPMA (Tabs. 1, 2; Figs. 3, 7a); they steadily increase during the final 5% of 39Ar release to values of about 20. Interestingly, drilled grain MT-11 lacks the staircase-shaped section and shows slightly decreasing apparent ages to ca. 2.64 Ga instead (Tab. 3; Fig. 7b). Its fairly constant Ca/K ratio of about 12 is comparable to ratios of 10-11.5 of separate MT-11 above 980°C (Tab. 3; Fig. 7b). Trends in Cl/K and Ca/K ratios of separate MT-10 inversely correlate during the first 95% of gas release (Fig. 7a). In contrast, separate MT-11 lacks such decreasing Cl/K ratios (Fig. 7b). Cl/K ratios of drilled grain MT-11 are much lower than those of the separate (Tabs. 2, 3) and unrelated to trends in Ca/K ratios (Fig. 7b), which we explain by the small gas volume released by laser step-heating, in which ClAr was, apparently, close to the detection limit.
Fig. 6. Chemical correlation diagrams displaying Ca/K vs. CI/K ratios obtained by resistance furnace step-heating of hornblende separates MT-10 (a), MT-11 (b) and MT-27 (c) (open symbols with degassing temperatures indicated in °C) and by laser step-heating of drilled hornblende grains MT-11 (b) and MT-27 (c) (filled symbols). Hornblende's main degassing occurred around 1000 °C with tightly clustered Ca/K and CI/K ratios. Increments with deviating apparent ages (in Ma rounded to the closest age) with respect to the age of the main hornblende are indicated in bold between brackets. Mixing lines toward biotite (degassing at lower temperatures) and Ca-rich inclusions (degassing at higher temperatures) indicated by grey arrows. Dotted arrows (●) point toward a Ca-rich composition carrying excess Ar (fluid inclusions?) for hornblende MT-27 both separate (735 °C, 4865 Ma and 3242 °C) and single grain (first step age: 3152 Ma). The first furnace step-heating increment MT-11 (b) at 775 °C (3360 Ma) is probably a mixed degassing of excess Ar and biotite components, whereas the first laser step-heating increment of the drilled grain carried essentially only an excess Ar component (4403 Ma). Significant difference of integrated Ar/Ar ages for drilled grain and the hornblende separate of MT-11 (Tables 2 and 3) probably point to heterogeneous excess Ar incorporation at the grain scale in this sample. The first biotite-dominated heating increment of MT-10 (a) at 775 °C (1827 Ma) probably also contains gas released by fluid inclusions, given the elevated Ca/K ratio.
Hornblende separate MT-27 has a fairly flat age spectrum, whereas the drilled grain yielded slightly decreasing step ages from 2.67 to 2.62 Ga, following excess argon spikes for the first released gas fractions with high Cl/K ratios (Tabs. 2, 3; Fig. 7c). The average mean ages of the final 5 steps of the drilled grain (2641 ± 7 Ma) and of the main increments of the separate (2639 ± 6 Ma) are concordant. The similar Ca/K ratios for separate (12-12.5) and drilled grain (13-14), which are fairly constant over the entire 39Ar release (Tabs. 2, 3; Fig. 7c), agree with values of 13-14 obtained by EPMA (Tab. 1; Fig. 3b). Ca/K ratios steadily increase for the separate during final 10% of degassing, but remain constant for the drilled grain (Fig. 7c). The Cl/K ratios of separate MT-27 and the much lower values for the drilled grain and are essentially constant and unrelated to trends in Ca/K ratios (Tabs. 2 and 3, Fig. 7c).
Table 2 40Ar/39Ar analytical data of resistance-heated furnace step-heating of hornblende and biotite from the Murmansk Terrane

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Total age 2470.6 15.0

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Interpretation of the spectra

We have obtained flat as well as staircase-shaped $^{40}$Ar/$^{39}$Ar age spectra for closely spaced samples, which would sharply contrast the histories when classically interpreted by thermally activated $^{40}$Ar loss by solid-state volume diffusion. The finding of a flat age spectrum for drilled hornblende MT-11 and an apparent partial loss spectrum for the hornblende separate of this sample shows that such an interpretation is unrealistic. The similarity of Ca/K ratios obtained from EPMA and $^{40}$Ar/$^{39}$Ar analysis shows that the age information contained in the spectra of drilled grains and separates for the main degassing above 970°C refers essentially to hornblende. However, the low Ca/K ratios of hornblende
separates MT-10 and MT-11 for gas release below 970°C point to degassing of included K-rich phases, like the observed tiny biotite crystals that occur intergrown in the amphibole. The main degassing of biotite took place below 1000°C (Tab. 2; Figs. 7a, b). This interpretation corroborates work by authors who argued that typical partial loss age spectra of hornblende stem from small amounts of incorporated biotite (Berger, 1975; Rex et al., 1993; Lo and Onstott, 1995). The finding of spectra with fairly constant apparent ages and Ca/K ratios for hornblende grain MT-11, from which biotite inclusions could be avoided by well targeted drilling, agrees with this interpretation. The fairly constant age and Ca/K ratio spectra of biotite-free hornblende MT-27 further strengthen this explanation.

The Ca-rich component that degasses above 1119°C during the final 5-10% of 39Ar release of hornblende separates MT-10 and MT-27 (Tab. 2; Fig. 7a, c) is unlikely to be apatite as its characteristic elevated Cl/K ratio (Belluso et al. 2000) is lacking. Despite its Ca/K ratio above 40 (EPMA Tab. 1; Fig. 3b) actinolite is not a good candidate either as it likely degasses well below 1100°C (Villa et al. 2000). Hence, clinopyroxene (K-content below detection limit EPMA) and omnipresent in MT-27 may explain the elevated Ca/K ratio during the final degassing. Given the constant Ca/K ratios (Tab. 3; Fig. 7c), drilled grain MT-27 does not contain this component, in line with the drilling of pristine hornblende. Even for the hornblende separates the influence of these K-poor inclusions is minor as degassing occurred in a narrow peak (Fig. 5) instead of a broad temperature interval that would have characterised a multi-component amphibole separate as outlined by (Villa et al. 2000). This interpretation agrees with tight clustering of Cl/K and Ca/K ratios of hornblende (Fig. 6).

Conclusions

Our well-documented natural example has shown that apparent partial loss 40Ar/39Ar age spectra of hornblende stem from the presence of only minor quantities of intimately intergrown younger biotite that degas before the amphibole host during furnace step-heating experiments. Laser step-heating of hornblende discs microsampled with a microscope-mounted drill from parts of grains without biotite and other inclusions, yielded flat age and Ca/K ratio spectra.

The significant ca. 2640 Ma age of hornblende MT-27 has no direct bearing on the age of the peak metamorphism in the MT because the mineral was formed by retrogressive hydration of older metamorphic clinopyroxene. Instead, the data constrain the cooling of the rocks following hornblende formation.

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