Argon redistribution during a metamorphic cycle: Consequences for determining cooling rates

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Argon redistribution during a metamorphic cycle: Consequences for determining cooling rates

Christopher S. McDonald, Clare J. Warren, Darren F. Mark, Alison M. Halton, Simon P. Kelley, Sarah C. Sherlock

Abstract

$^{40}$Ar/$^{39}$Ar thermochronology is commonly used to constrain the rates and times of cooling in exhumed metamorphic terranes, with ages usually linked to temperature via Dodson’s closure temperature ($T_C$) formulation. Whilst many metamorphic $^{40}$Ar/$^{39}$Ar data are consistent with the timing of crystallisation or cooling within a chronological framework defined by other, higher temperature, chronometers, other $^{40}$Ar/$^{39}$Ar data are more difficult to interpret. We report white mica and biotite single grain fusion and laser ablation $^{40}$Ar/$^{39}$Ar ages from felsic gneisses of the Western Gneiss Region, Norway. The rocks record isothermal decompression from peak eclogite-facies conditions (white mica stable) to amphibolite-facies conditions (biotite stable) at c. 700 °C. White mica and biotite yield dispersed single grain fusion dates from 416 to 373 Ma and 437 to 360 Ma respectively. In-situ laser ablation analyses provide a similar range, with white mica spot ages ranging from 424 to 370 Ma and biotite spot ages ranging from 437 to 370 Ma. The dates span the duration of the metamorphic cycle suggested by previous studies, and cannot be reconciled with the results of simple models of Ar loss by diffusion during cooling.

1. Introduction

Understanding the timing of when, and the rate at which, metamorphic terranes are exhumed through the Earth’s mantle and crust is important for constraining geodynamic models of tectonic processes. $^{40}$Ar/$^{39}$Ar mica thermochronology is typically employed to constrain exhumation and cooling rates, with age most commonly being linked to temperature via the Dodson closure temperature ($T_C$) formulation (Dodson, 1973). This solution to the diffusion equation is only applicable for geological applications under the following boundary conditions: (1) negligible initial lattice-hosted $^{40}$Ar during crystallisation (i.e. a very low mineral:fluid partition coefficient), (2) $\text{Ar}$ distribution within the mineral controlled only by thermally-activated volume diffusion that adheres to Fick’s 2nd law of diffusion (modified for a source term), (3) an ‘open’ grain boundary network (i.e. the grain boundary effectively has a negligible concentration of Ar) during the temperature interval over which within-grain Ar diffusion is efficient and (4) initial crystallisation at a temperature at which within-grain Ar diffusion is efficient.

As geochronological data continues to be collected at ever-higher spatial resolution and analytical precision, it is becoming possible to test some, but not all, of the listed boundary conditions. This is important for determining the geological scenarios in which the Dodson $T_C$ formulation is justifiably applicable vs. those situations where it is not. For example mineral thermobarometers provide estimates of...
metamorphic pressure-temperature conditions at different stages of the rock evolution, which, when coupled to experimental diffusion parameters, provide insight into whether a specific rock reached temperatures high enough for efficient diffusion in different minerals (c.f. Warren et al., 2012c). In-situ laser ablation profiles across grains of sufficient size can provide evidence for diffusion (e.g. Wartho and Kelley, 2003). Petrological modelling and a temporal framework based on independent higher-temperature chronometers coupled with diffusion modelling provide a solid platform for assessing open system behaviour and/or incorporation of initial $^{40}$Ar during crystallisation.

Over the last few decades, a significant number of studies have shown that micas in high pressure (> 15 kbar) metamorphic rocks appear to be particularly prone to yielding $^{40}$Ar/$^{39}$Ar ages that are “too old” relative to other independently constrained ‘events’ along their P-T paths. For example, despite temperatures high enough for theoretically effective diffusion, $^{40}$Ar/$^{39}$Ar data from high pressure micas commonly produce ages that are older than the timing of peak metamorphism from zircon U-Pb data, or relative to the petrographically-constrained timing of mica crystallisation. (Foland, 1979; Li et al., 1994; Arnaud and Kelley, 1995; Scaillet, 1996; Ruffet et al., 1997; Sherlock and Arnaud, 1999; Baxter et al., 2002; Warren et al., 2012a). Studies suggest that the incorporation of “excess” $^{40}$Ar ($^{40}$Ar$_{ex}$) decoupled from its parent $^{40}$K) by diffusion from the grain boundary into the mineral lattice and/or lattice defects, or during deformation or (re)cristallisation may modify $^{40}$Ar concentrations (Li et al., 1994; Ruffet et al., 1997; Pickles et al., 1997; Villa, 1998; Sherlock and Kelley, 2002; Wartho and Kelley, 2003; Di Vincenzo, 2004; Warren et al., 2011). Other studies suggest that non-zero grain boundary conditions were experienced during or after crystallisation: high Ar concentrations in the grain boundary would hinder efficient diffusive loss of Ar, and lead to inherited ages that are older than predicted by the Dodson $T_C$ formulation, but younger than the timing of mineral crystallisation (c.f. Baxter et al., 2002). Recent experimental data additionally suggest that Ar diffusion in white mica is pressure- as well as temperature-dependent (Harrison et al., 2009), suggesting higher Ar retention than previously suspected at metamorphic pressures > 10 kbar.

$^{40}$Ar/$^{39}$Ar data may therefore relate to the timing of mineral crystallisation, the timing of cooling through a specific temperature or temperature interval (specifically whether they adhere to the Dodson $T_C$ formulation), or may be geologically meaningless. Determining between these options is important for quantifying rates and timescales of tectonic processes. Here we track the incorporation, release, and transport of Ar within and between different minerals during a metamorphic cycle, and especially during the exhumation-related, retrograde metamorphic reactions. The study of such processes informs the assessment of the main mechanism(s) for redistributing Ar within minerals, and provides example cases for which $^{40}$Ar/$^{39}$Ar may or may not be reliably linked to temperature via the Dodson $T_C$ formulation.

Mid- to lower-crustal felsic gneisses exposed in the Western Gneiss Region (WGR), western Norway provide an excellent natural laboratory for determining Ar behaviour during a burial-exhumation cycle because the gneisses are broadly similar in composition but preserve different stages of the metamorphic evolution. The WGR experienced a high temperature evolution (~700 °C) for 10–15 Ma (Wain et al., 2000, Hacker, 2007, Spencer et al., 2013, Kylander-Clark & Kylander-Clark and Hacker, 2014) – long enough for diffusive processes to have been, in theory, efficient enough for all micas of the same grain size and composition to retain the same $^{40}$Ar/$^{39}$Ar age (Warren et al., 2012b). Published white mica $^{40}$Ar/$^{39}$Ar multi- and single-grain step heating ages from the Outer Nordfjord area range from 409 to 380 Ma and are interpreted as cooling ages (Root et al., 2005; Young et al., 2011). Published white mica and biotite $^{40}$Ar/$^{39}$Ar multi- and single-grain step heating ages from the Outer Nordfjord area range from 389 to 374 Ma and 402 to 375 Ma, respectively, and interpreted as cooling ages (Lux, 1985; Berry et al., 1995; Hacker and Gans, 2005; Root et al., 2005; Walsh et al., 2007; Young et al., 2011; Walsh et al., 2013). More recent single grain fusion and in-situ laser ablation techniques have yielded highly variable white mica $^{40}$Ar/$^{39}$Ar ages, both within and between grains in the same sample, as well as between different samples (Warren et al., 2012a). The range generated by the single grain fusion data was greater than the range expected from diffusive loss in different grain sizes. Furthermore, the spatial patterns that the in-situ data showed were inconsistent with diffusive loss on cooling.

Here we systematically document age populations from samples that show evidence for different physical processes including mineral breakdown/replacement, deformation, partial melting, and hydration, and compare them with age populations from samples that have not experienced or recorded evidence for these processes. Our data show that all samples generate a range of $^{40}$Ar/$^{39}$Ar ages spanning at least 15 Ma. Samples that preserve evidence for significant recrystallization and deformation displayed younger white mica, but older biotite, single grain fusion age populations than more pristine samples. Amphibolite-facies gneisses preserving evidence for significant partial melting yield younger biotite
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<td>Rim</td>
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| Total ions per 22 O | 184 | 184 | 184 | 184 | 184 | 184 | 184 | 184 | 184 | 184 | 184 | 184 | 184 | 184 | 184 | 184 | 184 | 184 | 184 | 184 | 184 | 184
ages than samples that record evidence for fluid infiltration during the amphibolite-facies overprint. Overall our results show that most of the 40Ar/39Ar mica dates from the WGR are inconsistent with the interpretation that they record the time at which the rock cooled through a fixed temperature interval. Instead the 40Ar concentrations appear to trace other physical processes that affected the rocks during exhumation.

1.1. Regional geology

The Western Gneiss Region (WGR) exposed in western Norway is a 50,000 km² basement window within the Scandinavian Caledonides, formed during the subduction of the leading edge of Baltica beneath Laurentia during the Scandinavian Phase of the Caledonian Orogeny (Gee, 1975; Roberts, 2003; Hacker, 2007). The dominant lithology in the WGR is amphibolite-facies felsic gneiss, with local eclogite enclaves (Eskola, 1921; Bryhni, 1966). Evidence for ultrahigh-pressure metamorphism (UHP, >2.6 GPa) is preserved across c. 5000 km² of the WGR (Smith, 1984; Root et al., 2005). Coesite (the high pressure polymorph of quartz whose presence confirms that UHP conditions were reached) has been documented mostly within eclogite, but rare occurrences have also been documented in the felsic host gneisses (Wain, 1997). There are three confirmed UHP domains: the Nordfjord-Stadlandet (southernmost), the Sorøyane (central) and the Nordøyane (northern). This study focuses on the Nordfjord-Stadlandet domain, where the record/preserved P-T gradient ranges from quartz eclogite-facies in the southeast (−2.5 GPa and ~600 °C) to coesite-stable eclogite-facies in the northwest (−3.0 GPa and ~700 °C) across 2500 km² (Labrousse et al., 2004; Root et al., 2005; Young et al., 2007). Following near-isothermal decompression from (U)HP conditions, the entire WGR experienced retrograde amphibolite-facies metamorphism at ~700 °C and 1 GPa (Walsh and Hacker, 2004).

Multiple radioisotopic dating techniques provide an independent temporal framework constraining the timing of peak metamorphism and exhumation across the Outer Nordfjord Region. Sm-Nd and Lu-Hf garnet ages suggest prograde metamorphism and burial from c. 440 Ma to c. 404 Ma (Griffith and Brueckner, 1980; Mearns, 1986; Carswell et al., 2003; Kylander-Clark et al., 2007; Peterman et al., 2009). U-Pb zircon ages in mafic eclogite bodies suggest that peak eclogite-facies metamorphic conditions were achieved by c. 410–400 Ma (Root et al., 2004, Young et al., 2007, Kylander-Clark and Hacker, 2014, Young and Kylander-Clark, 2015). U-Pb titanite ages of c. 399 to 379 Ma from the Nordfjord area suggest host gneiss recrystallization under amphibolite-facies conditions during exhumation (Spencer et al., 2013), and U-Pb zircon ages from an undeformed granitic pegmatite dyke suggest post-eclogite-facies partial melting and crystallisation at c. 389 Ma (Kylander-Clark and Hacker, 2014).

2. Petrology and mineral chemistry

Major-element compositions of white mica and biotite were analysed using the Open University Cameca SX-100 electron microprobe using a spot size of 10 μm, conditions of 15 kV, 20 nA and 30 s collection time. Calibrations were performed on natural standards, and analyses were corrected using a ZAF matrix correction routine. Analyses were bracketed by analyses of secondary standards to check for major element reproducibility of 1%. Average white mica and biotite compositions are reported in Tables 1 and 2 respectively; full data are reported in Supplementary Table S.1.

The felsic gneisses form two petrographically-distinct groups: garnet-bearing gneisses that record exhumation from eclogite-facies conditions (Group 1, Fig. 2A) and biotite-epidote gneisses that record the amphibolite-facies overprint (Group 2, Fig. 2B and C; a full petrographic description of each sample is provided in Supplementary material S.2). Major element XRF data suggest similar bulk compositions for these different groups (Young and Kylander-Clark, 2015).

The garnet-bearing (Group 1) gneisses comprise three sub-groups: those that still preserve HP relics (Group 1a), those that lack HP minerals but still contain white mica, albeit heavily replaced by biotite-plagioclase symplectites (Group 1b) and those that lack HP relics and no longer contain white mica (although do preserve symplectites indicative of the former presence of white mica; Group 1c).

Group 1a gneisses are found in a low-strain region adjacent to a large (>300 m long) eclogite boudin at Krokkenakken (samples NF40, NF42, and NF43; Fig. 2D). They are medium-grained, unfoliated gneisses with a peak assemblage of quartz, white mica and garnet with accessory apatite, rutile, zircon, clinozoisite, and kyanite. Symplectites of clinoxyroxyene and plagioclase provide evidence for precursor omphacite (Wain et al., 2000). White micas are 0.5–2.5 mm in length, with Si ranging from 6.8 per 22 oxygens per formula unit (pfu) in the cores to 6.3 pfu in the rims. White mica is commonly almost completely replaced by biotite-plagioclase symplectites at the rims. Biotite grains within the symplectite are generally <0.5 mm in size and are chemically variable with Mg/Mg + Fe (Mg#) of 0.52–0.73 and Ti concentrations of 0.2–0.3 pfu.

Group 1b gneisses occur at all three localities (Krokkenakken: NF106; Flatraket Harbour: NF48; Fig. 2E) and Drage: NF88, NF89, NF96 and NF99). These gneisses are foliated and composed of quartz, plagioclase feldspar, garnet, white mica, biotite, and rare alkali feldspar with accessory rutile, apatite, and zircon. White micas, 0.5–2.5 mm in length, are zoned in Si from 6.8 pfu in the core to 6.1 pfu in the rim. Narrow 0.5–1 μm wide rims are altered to biotite + plagioclase symplectite. Biotite grains <0.5 mm in length also forms fabric-defining euhedral laths. These grains show a wide range in composition with Mg# ranging from 0.46–0.51 and Ti concentrations between 0.23 and 0.34 pfu from core to rim.

Subordinate in volume to Group 1b gneisses, Group 1c gneisses preserve neither HP mineral relics nor white mica (Flatraket Harbour: NF47, NF50 and NF112; Drage: NF87). These gneisses are located close to the contact with the mafic eclogites and are also found close to the contact with the amphibolite-facies biotite-epidote gneisses described below. They contain quartz, plagioclase feldspar, garnet, epidote (commonly with allanite cores), and biotite with accessory rutile, apatite, and zircon. Rare biotite-plagioclase symplectites preserved in quartz ribbons attest to the former presence of white mica (Fig. 2F). Further biotite laths are generally <1 mm in length and yield a narrow range in composition with Mg# values from 0.53–0.58 and Ti concentrations from 0.20–0.34 pfu.

Group 2 biotite-epidote gneisses form approximately 70% by volume of the felsic gneisses in the study area (e.g. Peterman et al., 2009). They are typically foliated and preserve the lowest metamorphic grade assemblages. At Drage (Fig. 1), within the core of the antiformal Nordfjord-Stadlandet UHP domain, Group 2 gneisses are intensely migmatised.
Group 2a gneisses preserve relict white mica, whereas Group 2b do not. Group 2a are located close to the contact with Group 1c and contain an amphibolite-facies assemblage of biotite, epidote, quartz and plagioclase. They also commonly preserve skeletal relics of white mica and garnet (Fig. 2D). The white mica shows evidence of melting, being displaced by alkali feldspar + quartz at their rims. This type of gneiss occurs only at Krokkenakken (NF105) and at Drage (NF98; Fig. 2C). Relict white mica grains are <0.5 mm in length and form subhedral to euhedral laths that define the fabric. Biotite preserves a broad range of compositions with Mg# ranging from 0.40–0.51 and Ti concentrations from 0.12–0.42 pfu.

Group 2b samples include NF35 from Krokkenakken, NF51 from Flatraket Harbour (Fig. 2H) and samples NF90 and NF91 from Drage. They contain quartz, plagioclase, biotite, epidote (with common allanite cores and forming prominent porphyroblasts), minor amphibole, and K-feldspar. Accessory phases include titanite, ilmenite, apatite, and zircon. Biotite defines the fabric and is often inter-grown with titanite. The fabric both wraps around and is overprinted by the epidote porphyroblasts, suggesting epidote growth both during and after amphibolite-facies conditions (Fig. 2H). Biotite grains are ~1 mm in length and preserve a broad range in chemistry, with Mg# varying from 0.37 to 0.55 and with Ti concentrations from 0.20 to 0.37 pfu.

### Table 2

**Biotite data.**

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<tr>
<th>Sample</th>
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### 3. 40Ar/39Ar analyses

19 samples were selected for single grain fusion analysis to determine the presence of any age variations within and between different samples. For single grain fusion analysis, ~50 grains of white mica and biotite were picked from each crushed sample. Grains showing the least deformation and the fewest inclusions were selected. Care was taken to avoid picking white mica grains rimmed by biotite-plagioclase symplectites. Grains were washed in acetone or methanol and distilled water before being packed into aluminum foil packets. Polished thick sections (~250 μm) for in-situ laser ablation analysis were prepared by adhesion to glass slides with cyanoacrylate (“super glue”). Sections were removed from the glass by soaking in acetone for up to 24 h to dissolve the glue, and then cut into 5 × 5 mm squares before washing and packing as above.

Samples were analysed at two facilities: the 40Ar/39Ar and Noble Gas Laboratory at the Open University (OU), and the NERC-funded Argon Isotope Facility (AIF) housed at the Scottish Universities Environmental Research Centre (SUERC). Analytical details are summarised below; full details are provided in Supplementary document S.3. Two mass spectrometers were used at the OU. Total fusion of single grains of the unknowns and flux monitors was achieved using a 1062 nm CSI fibre laser coupled to an automated gas handling vacuum system and admitted to a MAP 215-50 noble gas mass spectrometer (Warren et al., 2012a). Samples were fused over 60–90 s and gasses cleaned for 210 s through two SAES AP10 Zr-Al getters (one at room temperature and the other at 450 °C). A liquid nitrogen cold trap provided additional gas cleaning prior to inlet to the mass spectrometer.

High spatial resolution single spot and traverse analyses on polished thick sections were achieved using a New Wave Systems Nd-YAG 213 nm ultraviolet (UV) laser coupled to a Nu Instruments Noblesse gas mass spectrometer (Sherlock et al., 2005). UV analyses consisted of ablating 30 μm diameter spots for 90 s, at 20 Hz and an on-sample fluence of 3.8 mJ cm$^{-2}$. Gasses were subsequently cleaned for 90 s through two SAES AP10 Zr-Al getters (one at room temperature and the other at 450 °C).

At SUERC, single grain fusions were achieved using a Merchantek 25 W CO2 laser coupled to a GVI ARGUS multi-collector mass spectrometer (Mark et al., 2009; Mark et al., 2011). Samples were fused over 20 s prior to 300 s gas clean-up using two GP50 SAES getters (one at room temperature and the other at 450 °C).

Isotope data were reduced using in-house software (ArMaDiLo), at the Open University and Berkeley Geochronology Centre ‘Mass Spec’ software at SUERC using a decay constant value of 5.530 × 10$^{-8}$ a$^{-1}$ (Renne et al., 2011). The isotope data were corrected for blank, radioactive decay, mass discrimination and interfering reactions. The 40Ar/39Ar measurements from the SUERC ARGUS and OU Nu instruments were corrected for atmospheric argon via the measured 39Ar. The measured 39Ar on the OU MAP instrument approached detection limits, and unlike on the Nu and ARGUS, the mass discrimination is not sensitive...
enough to discriminate between $^{36}$Ar and $^{36}$hydrocarbon. $^{40}$Ar measurements from this machine were therefore only corrected for atmospheric argon when the measured $^{36}$Ar was $>2 \times$ that of the background measurement (c.f. Sherlock et al., 2005; Supplementary Table S.4). The dispersion in ages within and between samples vastly out-magnifies the differences in errors on the ages that these changes in correction scheme induce. Both corrected and uncorrected ages are reported in Supplementary Table S.4. Age uncertainties are reported to 2σ.

4. $^{40}$Ar/$^{39}$Ar results

The summary $^{40}$Ar/$^{39}$Ar age data are provided in Table 3. Full isotope data for the single grain fusion analyses are reported in Supplementary Table S.4 and all ages are plotted in Fig. 3. Full UV laser ablation isotope data are in Supplementary Table S.5; ages are plotted on Fig. 4. Plots of $^{39}$Ar/$^{39}$Ar, $^{37}$Ar/$^{39}$Ar or $^{38}$Ar/$^{39}$Ar vs. $^{40}$Ar/$^{39}$Ar age show no correlation.

4.1. Single grain fusion data

4.1.1. Group 1: Garnet-bearing Gneisses

Three Group 1a samples from Krokenakken (NF40, NF42 and NF43), totalling 34 white mica and 34 biotite analyses, yielded white mica single grain fusion (SGF) dates between 405 ± 6 Ma and 377 ± 4 Ma and biotite dates between 389 ± 2 Ma and 377 ± 2 Ma (ignoring outliers; Fig. 3A).

Six Group 1b samples, from Krokenakken (NF106); Flatraket Harbour (NF48) and Drage (NF88, NF89, NF96 and NF99), totalling 107 white mica and 107 biotite analyses, generated white mica dates between 416 ± 8 and 373 ± 8 Ma and biotite dates between 412 ± 6 and 360 ± 6 Ma (Fig. 3B).

Four samples of white-mica-free Group 1c gneisses from Flatraket Harbour (NF47, NF112 and NF50) and Drage (NF87), yielded 63 biotite dates between 423 ± 8 and 381 ± 5 Ma (Fig. 3C).

4.1.2. Group 2: Biotite-epidote gneisses

Two samples of Group 2a gneisses from Krokenakken (NF105) and Drage (NF98) yielded 30 white mica and 30 biotite dates between 415 ± 6 and 377 ± 9 Ma (white mica) and 393 ± 6 to 365 ± 6 Ma (biotite; Fig. 3D).

Four Group 2b samples from Krokenakken (NF35), Flatraket Harbour (NF51) and Drage (NF90 and NF91) generated 94 biotite dates between 429 ± 4 and 377 ± 6 Ma (Fig. 3E).

4.2. UV laser ablation data

45 spots on four white mica grains from Group 1a sample NF40 and 87 spots on three white mica grains from NF43 (Fig. 4A) yielded spot dates ranging from 409 ± 3 to 380 ± 5 Ma and c. 424 ± 3 to 370 ± 6 Ma respectively (ignoring outliers, Supplementary Table S.5). Each grain provided a similar spread in dates but none showed consistent core-rim variations. 20 spots on five biotite grains from NF40 produced younger dates, from 393 ± 4 to 378 ± 3 Ma. 21 spots on five grains from sample NF43 generated dates between 400 ± 5 and 370 ± 7 Ma. 19 spots on one large grain (~1 mm) of white mica and 18 spots on nine biotite grains from a Group 1b sample (NF48) generated dates from 390 ± 3 to 372 ± 5 Ma and 393 ± 4 to 378 ± 3 Ma respectively (ignoring outliers; Fig. 4B).

33 spots on eight grains of biotite, three included within a garnet and five within the matrix, were analysed from a sample of Group 1c gneiss (NF50) from Flatraket Harbour (Fig. 4C), producing dates between 420 ± 4 to 371 ± 4 Ma. No notable difference was determined between biotite included in garnet and those in the matrix. 24 spots on eight grains of biotite from the matrix of Group 2b sample NF51 (Fig. 4D) from Flatraket Harbour yielded dates between 433 ± 4 and 376 ± 4 Ma.

5. Diffusion modelling

The $^{40}$Ar/$^{39}$Ar ages expected under the assumptions inherent in the Dodson T formulation (Dodson, 1973) for the published WGR P-T constraints were modelled using the finite-difference code DiffargP.
Fig. 2. Field photographs and photomicrographs of samples from the five petrological groups. (A) Field photograph of the garnet-bearing gneisses. (B) Field photograph of non-migmatised Group 2b gneisses at Flatraket Harbour. (C) Field photograph of migmatised Group 2b gneisses at Drage. (D) Group 1a gneisses (e.g. NF43) preserve high-pressure relicts. (E) Group 1b gneisses (e.g. NF48) contain relict garnet but no other HP relics. Note the formation of white mica fish and foliation defined by biotite. (F) Group 1c gneisses (e.g. NF112) show relict symplectites of biotite + plagioclase after white mica preserved in quartz ribbons. (G) Group 2a gneisses (e.g. NF86) preserve highly degraded garnet and minor flakes of white mica and (H) Group 2b gneisses (e.g. NF51) preserve large epidote porphyroblasts but no white mica or garnet. Mineral abbreviations after Whitney & Evans (2010), Wm = white mica and Symp = symplectite.
1.0 GPa was reached 7 Ma later in the models. These constraints were

starts. Decompression to amphibolite-facies conditions of 700 °C at

"instantaneous in an open system, so regardless of when the model

et al., 2012a) have shown how at the temperatures, Ar removal is geolog-

ically instantaneous in an open system, so regardless of when the model

clock starts, the grain has an effective age of zero at the time cooling

in-situ ages.

IR single grain fusion ages

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nd = not determined; n^a = number of grains or spots

Krokkenakken 61°54′ N 005°12″ 54.0 38.0
Drage 62°06′30″ N 005°14″ 48.0 7.0
Flatraket Harbour 61°58′ N 005°20″ 42.0 80.0

(Wheeler, 1996; Warren et al., 2012c). The P and T-dependent diffusion parameters of Harrison et al., 2009 were used to model the behaviour of white mica and the T-dependent diffusion parameters of Harrison et al., 1985 used to model the behaviour of biotite. Following petrographic observations, both micas were modelled at 0.5 mm grain radii.

Peak eclogite-facies conditions in the Nordfjord region appear to have reached 2.5–3.0 GPa and 600–700 °C at c. 410–400 Ma (U-Pb zircon), followed by isothermal decompression to 1 GPa and 700 °C by c. 399–379 Ma (U-Pb titanite: Cuthbert et al., 2000, Root et al., 2005, Young et al., 2007, Walsh and Hacker, 2004, Spencer et al., 2013). White mica and biotite were modelled separately to reflect the fact that they experienced different post-crystallisation histories, as shown by the petrographic observations (Section 2) and as described below.

5.1. White mica models

In the Nordfjord felsic gneisses, white mica appears to be a stable member of the peak eclogite-facies assemblage. White mica models therefore assumed crystallisation under eclogite-facies conditions of 650–750 °C at 2.5–3.5 GPa. Although the white mica may have crystallised along the prograde path, previous modelling (e.g. Warren et al., 2012a) has shown that at these temperatures, Ar removal is geologically instantaneous in an open system, so regardless of when the model “clock” starts, the grain has an effective age of zero at the time cooling starts. Decompression to amphibolite-facies conditions of 700 °C at 1.0 GPa was reached 7 Ma later in the models. These constraints were based on the time difference between the youngest (400 Ma) U-Pb zircon ages assumed to constrain the timing of peak pressure metamorphism in the Nordfjord region (Root et al., 2004; Young et al., 2007), the weighted mean of the U-Pb titanite ages in the region (393 Ma; Spencer et al., 2013) and the crystallisation age of an assumed decomposition-related granitic dyke (391 Ma, Kylander-Clark & Kylander-Clark and Hacker, 2014). In the models, linear cooling subsequently proceeded at a rate of 25 °C Ma⁻¹ from amphibolite-facies conditions. This reference cooling rate is somewhat arbitrary, but uncertainties in modelled age related to the choice of cooling rate are discussed below.

5.2. Biotite models

In all the mica-bearing gneisses, biotite appears to have replaced white mica during decompression from eclogite- to amphibolite-facies conditions, and was not stable at the metamorphic peak. Biotite models therefore assumed crystallisation of ~0.5 mm radius grains at amphibolite-facies conditions of 700 °C, 1.0 GPa, followed immediately by cooling at a linear rate of 25 °C Ma⁻¹. Ar diffusion in an open system is geologically instantaneous in biotite at 700 °C, so regardless of when the biotite crystallised along the decompression path, it should only record a zero age at the time cooling initiated.

Full model results, including sensitivity testing, are presented in Table 4. The models indicate that 0.5 mm radius white mica grains that decompressed from peak eclogite-facies conditions and then cooled after reaching amphibolite-facies conditions should yield ages that are
Fig. 3. (A) Compilation of current geochronological data for the Outer Nordfjord area, showing the timing of prograde metamorphism, peak metamorphism, and exhumation. Sm-Nd Grt-Cpx-Whole Rock (WR) and Lu-Hf data from Griffin and Brueckner (1980); Mearns (1986); Carswell et al., 2003; and Peterman et al. (2009). U-Pb zircon data from Root et al. (2004); Young et al. (2007); Kylander-Clark and Hacker (2014); and Young and Kylander-Clark (2015). U-Pb titanite data from Spencer et al. (2013). (B–F) Cumulative graphs of white mica and biotite single-grain fusion 40Ar/39Ar data from the five petrological types.
10 Ma younger than the timing of attainment of eclogite-facies metamorphism. Similarly, 0.5 mm radius biotite grains should provide ages that are 6 Ma younger than the time at which the rocks started to cool.

The diffusion model results are sensitive to a number of input uncertainties, which can be divided into those that cause spread within the modelled ages and those that shift the whole population. The only variable that causes a spread in the resulting modelled bulk ages is grain size. Variations in grain radius between 0.25 and 1 mm produce a 1 Ma uncertainty in the modelled bulk ages, a ± 2 Ma uncertainty on the core-rim ages of white mica and ± 1 Ma uncertainty on the core-rim ages of biotite. These uncertainties are well within the 1σ error of the single grain fusion and in-situ 40Ar/39Ar ages observed in this study.

Input variables that shift the whole model population include the diffusion parameters, the modelled cooling rates, and the peak temperature. The uncertainties in the experimentally-determined diffusion parameters (Ea and D0) shift the results by ± 4 Ma (Warren et al., 2012a). Varying the cooling rate from 10 to 50 °C Ma⁻¹ causes a +4/−2 Ma variance in the modelled white mica age and a +9/−3 Ma variance in the modelled biotite age. Finally, a ± 50 °C variation in the temperature at which cooling initiates shifts the resulting model age by ± 1 Ma.

6. Discussion

6.1. New single grain fusion and in-situ data

Overall, the white mica and biotite separates from all localities and petrological groups yield a broad spread in 40Ar/39Ar ages from 416 to 373 Ma in white mica and 437–360 Ma in biotite. In-situ UV laser data

Fig. 4. A. Photomicrographs showing the in-situ UV laser ablation 40Ar/39Ar ages and analysis pits. (A) White mica in NF43 (Group 1a), (B) White mica in NF48 (Group 1b).
shows that the distribution of Ar both within and between grains of white mica and biotite is non-systematic and highly variable, generating ages between 424 and 370 Ma (white mica) and 440–370 Ma (biotite). Overall the in-situ analyses provided a similar, yet wider, range in the age populations compared to the single grain fusion analyses. There is no discernible difference between the ranges of ages generated from the SGF data at SUERC or the OU, despite differences in hardware.

6.2. Comparison with previously published age data

The whole 40Ar/39Ar dataset spans the known timing of the entire metamorphic cycle from prograde metamorphism at 420–404 Ma (Sm-Nd and Lu-Hf garnet; Kylander-Clark et al., 2007, Peterman et al., 2009), through peak metamorphism at c. 410–400 Ma (U-Pb zircon; Root et al., 2004; Young et al., 2007; Kylander-Clark and Hacker, 2014; Young and Kylander-Clark, 2015) to the timing of amphibolite-facies
recrystallization documented by U-Pb titanite at 399–379 Ma (Spencer et al., 2013).

Published white mica $^{40}$Ar/$^{39}$Ar step heating plateau ages for the Outer Nordfjord area, range from c. 409–380 Ma (Root et al., 2005; Young et al., 2011). White mica SGF and in-situ $^{40}$Ar/$^{39}$Ar ages from this study span a greater range from 416 to 373 Ma and 424–370 Ma, respectively. The ages generated in this study are also broadly similar to previously reported SGF and in-situ ages for felsic gneisses and garnet mica schists from the Outer Nordfjord area (413 ± 4 Ma to 379 ± 2 Ma and 507 ± 6 Ma to 388 ± 6 Ma, respectively; Warren et al., 2012a).

Published biotite $^{40}$Ar/$^{39}$Ar step heating plateau ages from the WGR, range from 467 to 379 Ma (Berry et al., 1995; Hacker and Gans, 2005; Root et al., 2005). Biotite SGF and in-situ ages from this study range from 437 to 360 Ma and 447–370 Ma, respectively, within the previously reported range.

Previous studies have interpreted white mica and biotite step heating ages from Western Norway as representing the timing of cooling through 400 °C and 350 °C, respectively (Root et al., 2005; Hacker, 2007; Walsh et al., 2007; Young et al., 2011; Walsh et al., 2013). However, the age ranges documented in this study both within and between grains, and within and between samples, suggest there may be a more complex link between $^{40}$Ar/$^{39}$Ar age and metamorphic evolution. The different lithologies studied here allow the ages to be considered in relation to different potential influences.

### 6.3. $^{40}$Ar/$^{39}$Ar distribution during a metamorphic cycle

There are several possible explanations for the broad range of $^{40}$Ar/$^{39}$Ar dates - spanning 45/47 Ma (SGF/in-situ) in white mica and 77/70 Ma in biotite. These include the loss or gain of Ar due to diffusion, recrystallization, deformation, partial melting and/or the availability and/or influence of fluids. The effect of each of these processes can be critically assessed by comparing and contrasting age populations of different samples.

#### 6.3.1. Diffusion

The diffusion models show that in open conditions at temperatures of ~700 °C, volume diffusion in white mica and biotite should have been sufficient to reset mica ages in the Nordfjord area prior to the initiation of cooling. White mica grains of 0.5 mm diameter that crystallised at eclogite facies conditions should provide ages that are ~10 Ma younger than their crystallisation age. Similarly, 0.5 mm biotite grains that crystallised under amphibolite facies conditions should yield ages that are ~6 Ma younger than their crystallisation age, assuming cooling started soon after they crystallised. White mica grains from the study area should therefore produce total fusion ages of ~390 Ma with core-rim ages that range from 391 to 389 Ma. Biotite should provide bulk (total fusion) ages of ~387 Ma and core-rim ages that range from 388 to 378 Ma. However only ~20% of the white mica and biotite single grain fusion ages lie within the suggested model ages ±2 Ma (Fig. 3). 50% of the white mica SGF ages and 65% of the white mica in-situ ages, and 48% of the biotite SGF ages and 59% of the biotite in-situ ages are older, suggesting that diffusion may not be the principle mechanism for Ar redistribution within and between the micas of these felsic gneisses.

A key prediction of the pure diffusion model is the development of systematic core-rim age variations: older cores and younger rims. The in-situ white mica $^{40}$Ar/$^{39}$Ar data show patchy age distributions, with no clear core-rim age pro

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### Table 4

Diffusion modelling results.

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<td>391.8</td>
<td>388.6</td>
<td>390.4</td>
<td>0.0</td>
</tr>
</tbody>
</table>
analytical uncertainties for the biotite in-situ data (around ±6 Ma), the
±1 Ma core–rim age variation suggested by the diffusion modelling is
not resolvable.

Together, the spread in SGF and in-situ ages within and between
both grains in the same sample and across different samples suggests
that diffusion was not the sole mechanism by which Ar was
redistributed within both white mica and biotite, despite cooling from
ambient temperatures of ~700 °C.

6.3.2. White Mica breakdown

Mica recrystallization during metamorphism may exert a far greater
influence on isotopic resetting than diffusion, and should outpace it (e.g.
Villa, 1998; Allaz et al., 2011). In our samples, the white mica appears to
have been the only stable K-bearing phase at peak eclogite-facies condi-
tions. As exhumation and decompression progressed, white mica re-
crystallized to biotite + plagioclase. Various samples of Group 1 gneiss
document differential progress of this reaction, culminating in some
samples in the complete loss of white mica (NF43, NF48, and NF50; Fig.
5A and B). White mica and biotite age populations in these samples
were compared using Student T Tests to evaluate whether any statisti-
cally different significance were exhibited. The results are presented in
Table 5.

Sample NF43 (Group 1a) preserves the initial stages of white mica
breakdown to biotite. In this sample, white mica SGF ages range from
404 to 387 Ma and biotite SGF ages from 390 to 383 Ma (Table 3,
Fig. 5A and B). Sample NF48 preserves more advanced breakdown of
white mica and yields a statistically younger white mica age population
of 391–380 Ma. The biotite age population in sample NF48, however, is
statistically identical to sample NF43 (391–382 Ma; Fig. 5A and B). This
suggests that even though 40Ar was being released during white mica
breakdown, it was not incorporated into the concurrently crystallising
biotite in either sample. Conversely, biotite in sample NF50, which pre-
serves no white mica, generated statistically significantly older ages of
400–383 Ma (Fig. 5B). This shift towards older dates implies that the
co-crystallising biotite incorporated some of the 40Ar released from
the white mica, assuming all the biotite crystallised during exhumation.
A recent study suggests that in some samples, lower pressure minerals
persisted metastably during the Caledonian orogeny (Young and
Kylander-Clark, 2015). If this was the case for biotite, some of the
older ages may imply argon inheritance. Either way, the data suggest
that an open system was not consistently or concurrently present in
all rock types during exhumation.

6.3.3. Deformation

The effect of exhumation-related deformation on the 40Ar/39Ar ages
yielded by the felsic lithologies was investigated by comparing the
white mica and biotite SGF ages from Group 1a and Group 1b gneisses,
which are assumed to be of the same bulk composition (Fig. 5C and D).
Group 1a gneisses preserve biotite-plagioclase symplectites that form
from the breakdown of white mica. These delicate structures suggest
that little or no deformation affected these gneisses during and/or
after decomposition (e.g. Hacker et al., 2010). These samples also vari-
able preserve HP relics such as omphacite or kyanite that are not pres-
ent in the other gneiss groups. In contrast, Group 1b gneisses do not
preserve the delicate symplectite structure, but are instead deformed,
with a gneissic foliation defined by biotite + plagioclase. White micas
are kinked, folded and form “fish” and HP minerals or their relics are
not present. The Si content of the remnant white mica in Group 1b is
more variable than that in Group 1a.

White mica and biotite in Group 1a samples yielded SGF ages be-
tween 405 and 377 Ma and 437–360 Ma, respectively (Table 3).
Group 1b samples generated broader, but statistically identical age
ranges (416–373 Ma and 412–360 Ma; Tables 3 and 5). The age popula-
tion similarity between the two groups suggests that in this area, forma-
tion of the gneissic fabric does not result in a statistically significant
change in the white mica and biotite 40Ar/39Ar age populations.

Assuming the fabric is related to Caledonian deformation, rather than
inherited from the Proterozoic protolith (e.g. Young and Kylander-
Clark, 2015); the implication is that deformation alone did not control
40Ar redistribution in the felsic gneisses.

6.3.4. Partial melting

Migmatites have been documented in many exhumed HP metamor-
phic complexes (e.g. Faure et al., 1999; Hermann et al., 2001; Ganzhorn
et al., 2014), with melting generally linked to decomposition from eclogite- to the amphibolite-facies conditions at high temperatures (e.g. Labrousse et al., 2002). At Drage, the amphibolite-facies gneisses show abundant field and petrological evidence for partial melting (Fig. 2C, Supplementary information S2). The $^{40}\text{Ar}/^{39}\text{Ar}$ ages of key samples NF90 and NF91. Further studies involving a greater number of samples NF90 and NF91. The in fluence of partial melting on the $^{40}\text{Ar}/^{39}\text{Ar}$ signature. NF90 and NF91 were collected from Flatraket Harbour) were compared to assess the influence of partial melting in these samples (Group 2b gneisses) consumed fluids. Together these reactions show that water was both locally produced and consumed in the felsic rocks during exhumation, and that fluid was likely mobile at least on the cm scale.

The influence of fluid availability on the biotite $^{40}\text{Ar}/^{39}\text{Ar}$ ages between the garnet- and epidote-bearing samples is clear (Fig. 5F). The garnet-bearing rocks of Group 1c yielded biotite ages of 423–381 Ma, whereas the epidote-bearing rocks of Group 2b generated a statistically older and broader population between 429 and 377 Ma. Fluid in the Group 2b gneisses, likely internally-derived from the white mica breakdown reaction, therefore appears to not have been very mobile. The older biotite population and the increased age spread suggest that the grain boundary network was (partially) closed during exhumation, and that water was both locally produced and consumed in the felsic rocks during exhumation, and that fluid was likely mobile at least on the cm scale.

The felsic rocks exposed in the Outer Nordfjord region preserve evidence for fluid-producing reactions during their decompression history, including the white mica breakdown reaction. In contrast, the replacement of garnet (Group 1c gneisses) by epidote–group minerals (Group 2b gneisses) consumed fluids. Together these reactions show that water was both locally produced and consumed in the felsic rocks during exhumation, and that fluid was likely mobile at least on the cm scale.

6.3.6. Summary

The lack of clear core-rim age variations expected from diffusive loss and the wide spread in ages shown by the SGF data suggest that diffusion was not the primary mechanism by which $^{40}\text{Ar}$ was redistributed in micas from the WGR felsic gneisses. Furthermore, the lack of statistical difference between age populations in samples that record strain during the Caledonian orogeny vs. those that do not suggests that in this region at least, deformation was unimportant for redistributing Ar. Conversely, white mica breakdown, partial melting and the release/absorption of fluids appear to have played a far greater role in controlling grained $^{40}\text{Ar}$ concentrations.

Table 5

<table>
<thead>
<tr>
<th>White Mica</th>
<th>All Data</th>
<th>Group 1a</th>
<th>Group 1b</th>
</tr>
</thead>
<tbody>
<tr>
<td>Group 1a</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Group 1b</td>
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<table>
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<th>Partial Melting</th>
<th>NF90 Bt</th>
<th>NF91 Bt</th>
<th>NF51 Bt</th>
</tr>
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<tbody>
<tr>
<td>NF90 Bt</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>NF91 Bt</td>
<td>0.00</td>
<td></td>
<td></td>
</tr>
<tr>
<td>NF51 Bt</td>
<td>0.04</td>
<td>0.00</td>
<td></td>
</tr>
</tbody>
</table>

Values < 0.05 indicate significant difference at 95% confidence
Values 0.05–0.1 indicate a possible difference at 95% confidence
Values > 0.1 indicate no significant difference at 95% confidence

(WM = white mica, Bt = biotite)
Samples in which white mica is no longer present (but for which textural evidence of precursor white mica is still visible) yield older biotite age populations than samples in which white mica is still present. Rocks in which there is evidence for partial melting produce younger biotite age populations than rocks which do not appear to have melted, suggesting that melting facilitated $^{40}$Ar removal. The older biotite age population in more hydrated samples compared to drier samples suggests that fluid released during white mica breakdown acted as a source for excess $^{40}$Ar in the biotite and that the fluid had limited mobility.

### 7. Conclusions

Felsic gneisses with similar bulk compositions from the Outer Nordfjord area of the UHP Western Gneiss Region, Norway, preserve textural and petrological evidence of different stages of their burial and exhumation history. $^{40}$Ar/$^{39}$Ar data show that Ar is incorporated into, hosted by, and ultimately lost by white mica and biotite differently in these different rock types despite their shared metamorphic history.

White mica and biotite grains yield dispersed single grain fusion age populations from 416 to 373 Ma and 437 to 360 Ma, respectively. In situ UV laser ablation white mica analyses reveal a similar, but older, 424 to 370 Ma spread within individual grains, and no clear core-rim patterns. In-situ biotite analyses yield a similar, and slightly older, spread, from 447 to 370 Ma.

Numerical modelling of open system diffusion predicts that due to the high temperatures experienced by the WGR during the metamorphic cycle, white mica and biotite ages should reflect the timing of cooling after reaching amphibolite-facies conditions. However both micas yield age populations that are mostly older than the model ages and span the timing of the entire metamorphic cycle. Such an age spread cannot be reconciled with simple open system diffusion and therefore the Outer Nordfjord $^{40}$Ar/$^{39}$Ar mica ages likely do not strictly represent the timing of cooling through a discrete temperature or temperature interval. Whilst Ar must have diffused readily within minerals during the exhumation of the WGR at temperatures near 700 °C, age populations in samples that record different parts of the metamorphic cycle suggest that processes such mineral breakdown, partial melting, and fluid availability had a greater effect on Ar redistribution and eventual $^{40}$Ar/$^{39}$Ar age at the grain- to outcrop-scale. In detail, samples recording significant white mica breakdown produced younger white mica dates than white mica in less retrogressed samples. These ages were overall too old to be interpreted as crystallisation or cooling ages. In the same sample suite, biotite grains yielded the same age population in samples that still contained white mica or white mica remnants, but an older population in the samples where white mica had completely broken down. Samples that showed significant evidence for partial melting generated a younger biotite population than samples showing no evidence for partial melting. More hydrated (epidote-present) samples provided significantly older biotite age populations than less hydrated (garnet-present) samples. The older biotite population in the more hydrated samples suggests high $^{40}$Ar concentrations were present in the fluids and that local fluid mobility was limited. Finally, contrary to previous studies, deformation did not appear to affect the $^{40}$Ar/$^{39}$Ar mica age populations in these samples.

The spread of ages generated both within and between different samples has repercussions for detrital mica provenance studies. These studies assume that the source material can be defined by specific $^{40}$Ar/$^{39}$Ar age populations that reflect the cooling and exhumation history of the source. In the WGR, this source material is predominately felsic gneiss, which, in this study, yield spreads of up to 46 Ma (white mica) and 74 Ma (biotite) from the same exposure horizon. Matching detrital mica populations to a change in source in this region is therefore impossible.

This study shows that the interpretation of $^{40}$Ar/$^{39}$Ar age data collected from high temperature metamorphic terranes is assisted by analysing different Ar chronometers and multiple samples that record different parts of the metamorphic cycle. Data from different rock types needs to be considered on a case-by-case basis, in conjunction with detailed petrographic analysis, before $^{40}$Ar/$^{39}$Ar mica ages can be interpreted.

Supplementary data to this article can be found online at http://dx.doi.org/10.1016/j.chemgeo.2016.09.028.

### Acknowledgements

CJM was funded on an Open University Charter Studentship. CJW acknowledges financial support from a NERC Advanced Fellowship (NE/H016279/1). Laboratory and technical assistance from Michelle Higgins, Sam Hammond and James Malley was gratefully received. David Young, Jan. Wijbrans and Klaus Mezger are all thanked for insightful comments that improved the manuscript.

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