Clay mineral dating of displacement on the Sronlairig Fault: implications for Mesozoic and Cenozoic tectonic evolution in northern Scotland

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Abstract
Temporary excavations during the construction of the Glendoe Hydro Scheme above Loch Ness in the Highlands of Scotland exposed a clay-rich fault gouge in Dalradian Supergroup psammite. The gouge coincides with the mapped trace of the subvertical Sronlairig Fault, a feature related in part to the Great Glen and Erich–Laidon faults, which had been interpreted to result from brittle deformation during the Caledonian orogeny (c. 420–390 Ma). Exposure of this mica-rich gouge represented an exceptional opportunity to constrain the timing of the gouge-producing movement on the Sronlairig Fault using isotopic analysis to date the growth of authigenic (essentially synkinematic) clay mineralization. A series of fine-size separates was isolated prior to K–Ar analysis. Novel, capillary-encapsulated X-ray diffraction analysis was employed to ensure nearly perfect, random orientation and to facilitate the identification and quantification of mica polytypes. Coarser size fractions are composed of greater proportions of the 2M1 illite polytype, with no evidence of 2M1 illite in the finest fractions. A series of Illite Age Analysis plots produced excellent R2 values with calculated mean ages of 296 ± 7 Ma (Late Carboniferous–Early Permian) for the oldest (2M1) illite and 145 ± 7 Ma (Late Jurassic–Early Cretaceous) for the youngest (1M) illite. The Late Carboniferous–Early Permian (Faulting event 1) age may represent resetting of earlier-formed micas or authigenesis during dextral displacement of the Great Glen Fault Zone (GGFZ). Contemporaneous WNW(ESE) extension was important for basin development and hydrocarbon migration in the Pentland Firth and Moray Firth regions. The Late Jurassic–Early Cretaceous (Faulting event 2) age corresponds with Moray Firth Basin development and indicates that the GGFZ and related structures may have acted to partition the active extension in the Moray Firth region from relative inactivity in the Pentland Firth area at this time. These new age dates demonstrate the long-lived geological activity on the GGFZ, particularly so in post-Caledonian times where other isotopic evidence for younger tectonic overprints is lacking.

Keywords: illite, X-ray diffraction, K–Ar analysis, polytype, Dalradian, Great Glen Fault Zone

Faults and fractures originate from strains that arise from stress concentrations around flaws, heterogeneities and physical discontinuities, on scales from the microscopic to continental, in response to lithostatic, tectonic and thermal stresses and high fluid pressures (National Research Council, 1996). Faults and fractures that affect rock strength and the mechanical behaviour of rock units, provide both flow-paths and barriers for fluids and often form the host for economic mineralization, are amongst the most important of geological structures (Gillespie et al., 2011).

For many years, the dating of brittle near-surface faults was commonly achieved by indirect geological deduction (Lyons & Snellenberg, 1971). However, reactive surfaces created by movement within these localized zones led to the retrogressive development of fault gouges composed of rock fragments and authigenic phyllosilicates (Bense et al., 2014). The most recent fault activity is revealed by mineralogical and isotopic investigations of the finest particle size fractions of these fault gouges in the fault core (Kralik et al., 1987). The commonly illitic composition of the gouge phyllosilicates lends itself to age determination using radiogenic 40K–39Ar or, following microencapsulation, 39Ar–39Ar geochronometers (Poland et al., 1992).

The importance of understanding basin thermal history in the hydrocarbon industry led to the development of the seminal Illite Age Analysis (IAA) technique (Peev, 1992; Vrolijk et al., 2018) and its further application to fault dating to estimate hydrocarbon trap timing and with the potential for evaluating earthquake hazards (Peev, 1992).

A plethora of fault-dating studies have since been published (e.g. Vrolijk & van der Pluijm, 1999; Ylagan et al., 2002; Solum & van der Pluijm, 2005, 2007; van der Pluijm et al., 2006; Mántárrí et al., 2007; Haines & van der Pluijm, 2008; Schleicher et al., 2010; Zwingmann et al., 2010, 2011; Duvall et al., 2011; Rahl et al., 2011; Verdel et al., 2011; Pleuger et al., 2012; Donohue, 2013; Hetzel et al., 2013; Viola et al., 2013).

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displacement in the latest Carboniferous to Early Permian (Speight & Mitchell, 1979); transtensional opening and expansion across the Moray Firth–Pentland Firth region from the Permian and through the Mesozoic (Underhill, 1991; Underhill & Brodie, 1993); and dextral oblique wrench in the Paleocene–Eocene as Atlantic Ocean opening progressed (Holgate, 1969; Watts et al., 2007).

In the Glen Roy District of the Central Highlands, the Sronlairig Fault splays off the GGFZ and is indicated as a simple fault plane trending NE–SW (British Geological Survey, 1995). Northwards, throughout the Strathdearn area, the Sronlairig Fault then appears as a more complex zone of faulting with associated subparallel faults and minor splays cutting granitic intrusions to form wide zones of brecciation and shearing (British Geological Survey, 2004). Further north in the Nairn district, it is shown on published geological maps as twin fault structures trending NNE–SSW, locally constraining small grabens filled with Middle Devonian conglomerate (British Geological Survey, 2013).

The history of displacement on the Sronlairig Fault is poorly constrained. In the southwestern part of the Grampian terrain around Glendoe, folds and shear zones that affect the Caledonian metamorphic rocks are cut by mylonite and cataclasite fabrics as exposed in Glen Buck and the River Tarff (British Geological Survey, 1995; Key et al., 1997). The Sronlairig Fault was probably initiated at least in the early part of the Caledonian orogeny, and, as orogenic movements declined, strain generated lesser offsets and displacements along the boundaries of the late Caledonian (Silurian–Devonian) granite plutons that intrude the metasedimentary complex (Haselock & Leslie, 1992; British Geological Survey, 1996, 2004). The youngest onshore sedimentary strata affected by the Sronlairig Fault are sandstones of Upper Devonian (Famennian) age (British Geological Survey, 2012). It is not clear if the Sronlairig Fault extends offshore, but faults mapped offshore in the Moray Firth and Pentland Firth later accommodated the geological evolution of the late Paleozoic and Mesozoic basins in that region, in part by reactivating the Caledonian structures (Dewey & Strachan, 2003).

Although large faults like the Sronlairig Fault have clearly played an important role in the tectono-sedimentary evolution of parts of onshore and offshore Scotland, they are poorly exposed and the evidence for their younger tectonic history has not been reported onshore. Temporary excavations during dam construction for the Glendoe Hydro Scheme provided a rare opportunity to study the exposed trace of the Sronlairig Fault, as well as an exceptional chance to sample and analyze a non-weathered homogeneous brown/grey clay-rich fault gouge and to use a combination of K–Ar geochronology and X-ray diffraction (XRD) techniques to constrain the timing of post-orogenic movements.

illite Age Analysis and fault dating

Two different mineral transformations in clay gouges have proved suitable for IAA (Pevear, 1999): the illitization of illite-smectite (I-S) and the neocrystallization of authigenic 1Md/1Md illite (Haines & van der Pluijm, 2008). Mixtures of illite and I-S can be identified and the individual components can be quantified relatively simply using XRD profile modelling programs such as NEWMOD® (Reynolds & Reynolds, 1996; e.g. Solum & van der Pluijm, 2005; van der Pluijm et al., 2006). Identifying and quantifying mixtures of illite polytypes have proved to be more challenging.
Experimental studies indicate that the 2M\textsubscript{i} polytype is the most stable and is thought to form above 280°C (e.g. Šrodon & Eberl, 1984), while the 1M/1M\textsubscript{g} polytypes are thought to form at significantly lower temperatures, below ~200°C (Velde, 1965). The sequence 1M\textsubscript{lg}−1M−2M\textsubscript{i} has been proposed to be indicative of increasing temperature and pressure, with authigenic 2M\textsubscript{i} indicating the onset of the lowest-grade metamorphic zone (the anchizone 280–360°C) (Merriman & Kemp, 1996).

Early fault-dating studies of clay gouges considered that the fine-grained fraction of illite (<2 µm) was entirely composed of low-temperature 1M or 1M\textsubscript{g} authigenic illite, with no detrital (2M\textsubscript{i}) illite present (e.g. Lyons & Snellenberg, 1971; Kralik et al., 1987). However, more recent studies have revealed that early illite dating produced erroneously old ages due to the presence of detrital (2M\textsubscript{i}) material, even in the very fine particle-size fractions (Pevear, 1992; Grathoff et al., 2001). The most widely accepted interpretation is that 2M\textsubscript{i} illite in fault rocks is derived from the wall rocks and the 1M (and/or 1M\textsubscript{g}) component is authigenic material formed in the fault during movement (i.e. synkinematic; e.g. Hower et al., 1963; Reynolds, 1963; Grathoff et al., 2001). However, it should be noted that none of the many published fault-dating studies has provided direct evidence that 1M authigenesis is synkinematic. Nevertheless, recent research on Alpine, Scandinavian and Pyrenean faults in mostly high-grade metamorphic terranes consistently describes authigenic 2M\textsubscript{i} illite developing within fault gouges (e.g. Zwingmann et al., 2010; Viola et al., 2013, 2016; Torgersen et al., 2015; Abd Elmola et al., 2018). Because these faults may contain a mixture of wall rock-derived and authigenic 2M\textsubscript{i} illite, age dates obtained for these populations should be regarded as minima.

Research has therefore focused on methods to discriminate and quantify the various illite polytypes. Initial methods involved using peak-area or peak-height measurements for polytype-specific XRD peaks, usually dividing these by the area of the 2.58 Å band that is common to all illite polytypes (e.g. Reynolds, 1963; Maxwell & Hower, 1967; Callière et al., 1982). Later, computer modelling programs such as WILDFIRE\textregistered (Reynolds Jr, 1994) were employed to determine quantitatively the amounts (as percentages) of authigenic and wall rock-derived clay in various size fractions (e.g. Grathoff & Moore, 1996; Ylagon et al., 2002; Solum & van der Pluijm, 2005, 2007; van der Pluijm et al., 2006; Haines & van der Pluijm, 2008).

Once determined, the percentage of wall rock-derived illite in each of the particle-size fractions is plotted against their apparent K–Ar (or 40Ar–39Ar) total gas ages and extrapolated linearly to 0% and 100% in order to find the ages of the ‘end-member’ wall-rock and authigenic illite populations (Pevear, 1999).

The K–Ar dating technique was one of the earliest isotopic methods, and it remains a popular geological technique due to its ease of measurement and ideal half-life (1250 Ma; Kelley, 2002). The later 40Ar–39Ar dating technique offers the advantage that K and Ar are measured simultaneously in the same sample and therefore can be applied to very small and heterogeneous samples (Kelley, 2002). Both the K–Ar and 40Ar–39Ar techniques are based on the decay of naturally occurring K and Ar, but they differ in the way K is measured. In K–Ar dating, K is usually measured using flame photometry, atomic absorption spectroscopy or isotope dilution, while Ar isotope measurements are made by mass spectrometry on a separate subsample. In 40Ar–39Ar dating, K is measured by the transmutation of 39Ar to 39Ar by neutron bombardment; ages are determined from the ratio of Ar isotopes. While K–Ar dates are absolute, 40Ar–39Ar dates are relative to a mineral standard analysed simultaneously.

Both K–Ar and 40Ar–39Ar dating techniques have been applied to illite fault-dating studies (e.g. van der Pluijm & Hall, 2015; Zwingmann, 2015). However, recoil effects during irradiation result in the loss of up to 30% 39Ar and severely compromise 40Ar–39Ar dating of fine-grained species such as illite. More recent encapsulation methodologies have captured the recoil 39Ar and allow non-standardized corrections for age calculations (Dong et al., 1995; Clauer et al., 2012).

Sample site on the Sronlairig Fault

During construction of a dam, forming part of the supporting infrastructure of the Glendoe hydro Scheme, a 50 m-long section through the Sronlairig Fault was revealed briefly in 2007. At the site (Figs 1b, 2a), the fault juxtaposes granitic rocks of the Allt Crom Pluton (Caledonian Supersuite) against psammitic rocks of the Glen Doe Psammitic Formation (Corrieyairack Subgroup/Grampian Group/Dalradian Supergroup). The latter were deposited originally as muds and sands ~650–700 Ma (Key et al., 1997). The sinistral offset on the Sronlairig Fault, as
measured by surface displacement of the pluton contact, is ~2–5 km.

The southern (rear) wall of the temporary exposure revealed fractured psammitic with well-defined discrete planar fractures aligned parallel (060°N) to the mapped trace of the Sronlairig Fault. Immediately adjacent (on the NNW side) is a band of greenish–grey to blue cataclasite developed in psammitic, which is relatively persistent and ~20–30 cm thick where best developed. This band is separated by a sharp boundary from a band up to 2.5 m wide of clay-rich, brown and greenish–grey fault gouge enclosing fragments of psammitic rock, which is taken to be the fault core (or at least the core of one substantial fault strand within the Sronlairig Fault zone). The fault gouge is malleable and can easily be moulded and shaped by hand. The band of fault gouge pinches and swells along the fault trace, becoming irregular. In other respects, the character of the fault gouge does not vary (when viewed at the outcrop and hand-sample scale). The colour bands are inferred to be due to a redox overprint; the brown banding appears as a local overprint on the grey fault gouge, possibly caused by localized penetration of oxidizing groundwater.

The fault gouge locally preserves clear evidence of a shape fabric in the brown and greenish–grey clay-rich domains, indicating sinistral shear in the lifetime of the fault structure (see below). S–C fabrics are developed in the clay-rich gouge. Representative samples of fault gouge were collected for characterization and dating.

Materials and methods

Samples

Four samples (VY351, 358, 359 and 360) of undisturbed clay-rich gouge for petrographic characterization were carefully collected using Kubiena tins (Kubiena, 1953). Three of these were collected from the brown fault gouge in mutually orthogonal orientations (Fig. 2b). The fourth was collected to include significant proportions of both grey and brown gouge. The samples, initially wrapped in cling film to minimize drying and shrinking, were slowly impregnated with epoxy resin under vacuum to consolidate the soft fault gouge and preserve delicate structures. A single, large-format (200 mm × 200 mm × 100 mm) Kubiena tin contained each, which was then ground in an agate pestle and mortar to pass a 2 mm sieve. A subsample of the <2 mm crushate was ball-milled, then a portion of each ball-milled sample was micronized following the methodology of Kemp et al. (2016a). The samples were then spray-dried following the method and apparatus described by Hillier (1999) and front-loaded into a standard, stainless steel sample holder for analysis.

Further size fractionation

In order to prepare fine-size fractions for K–Ar analysis and age dating, <2 µm fractions were isolated. However, in order to retrieve all of the fine-grained material, the gravity separation process was repeated until no further particles were visible in suspension.

For capillary mount preparation, small quantities (<30 mg) of each size fraction were ground in an agate pestle and mortar to pass a 32 µm sieve cloth. The powdered samples were then loaded into borosilicate glass capillary tubes (80 mm long, external diameter 0.7 mm, wall thickness 0.01 mm) and sealed with a flame. A Capillary Boy (Huber Diffractionstechnik GmbH & Co.) was employed to accelerate the capillary-filling process. Each capillary was then mounted in a brass collet for XRD analysis.

Petrographic analysis

The thin sections were first examined under plane-polarized light and cross-polarized light conditions using a Zeiss Axioplan 2 polarizing microscope. Following the application of a carbon coating, further examination of the thin sections was undertaken using a LEO 535VP variable-pressure scanning electron microscope (SEM) equipped with an Oxford Instruments INCA Energy 450 energy-dispersive X-ray microanalysis (EDX) system for qualitative phase identification. The sections were analysed under high vacuum with a 15 kV accelerating voltage and a working distance of 19 mm. Photomicrographs were collected under backscatter electron imaging conditions.

• Sample VY352 [245422 802845] consists of chocolate brown (hematite-bearing) fault gouge and is visually clay-rich with variably comminuted/digested rock fragments and wisps and patches of greenish–grey clay, the latter commonly associated with remnant rock fragments. Two subsamples were prepared and analysed: VY352/G1 consists almost entirely (>95%) of chocolate brown fault gouge, while VY352/G2 contains a larger proportion (~10%) of visible rock fragments and grey fault gouge.

• Sample VY361/G1 [245404 802872] consists almost entirely of grey fault gouge, with occasional small, dark green rock fragments and rare wisps of brown (hematite-bearing) gouge.

Illite Age Analysis

Initial sample preparation

Representative subsamples were dried overnight in a moisture extraction oven at 55°C and then hand-crushed in a pestle and mortar to pass a 2 mm sieve. A subsample of the <2 mm crushate was ball-milled, then a portion of each ball-milled sample was micronized following the methodology of Kemp et al. (2016a). The samples were then spray-dried following the method and apparatus described by Hillier (1999) and front-loaded into a standard, stainless steel sample holder for analysis.

Nominal <2 µm fractions were isolated from further portions of the <2 mm crushate and oriented mounts prepared using the methodology outlined by Kemp et al. (2016b).

Further size fractionation

In order to prepare fine-size fractions for K–Ar analysis and age dating, further <2 µm fractions were isolated. However, in order to retrieve all of the fine-grained material, the gravity separation process was repeated until no further particles were visible in suspension.

The dispersed <2 µm fractions were then centrifuged (MSE Blue Force) for a calculated time period and speed (e.g. Hathaway, 1956) and the <0.2 µm supernatant decanted into a stock beaker. The dispersion and centrifugation process was then repeated three times, adding the decanted <0.2 µm suspension to the stock beaker each time. Finally, the 2.0–0.2 µm sediment was removed from the base of the centrifuge bottles, dried at 55°C and stored. Similar procedures, using a higher-speed Sorvall RC 5B Plus centrifuge, were performed sequentially to produce finer particle-size fractions (0.2–0.1, 0.10–0.05 and <0.05 µm). All fractions were dried at 55°C and stored.

For capillary mount preparation, small quantities (<30 mg) of each size fraction were ground in an agate pestle and mortar to pass a 32 µm sieve cloth. The powdered samples were then loaded into borosilicate glass capillary tubes (80 mm long, external diameter 0.7 mm, wall thickness 0.01 mm) and sealed with a flame. A Capillary Boy (Huber Diffractionstechnik GmbH & Co.) was employed to accelerate the capillary-filling process. Each capillary was then mounted in a brass collet for XRD analysis.
X-ray diffraction analysis

X-ray diffraction analysis was carried out using a PANalytical X’Pert Pro series diffractometer equipped with a cobalt-target tube and X’Celerator detector and operated at 45 kV and 40 mA. The spray-dried samples were scanned from 4.5 to 85°2θ at 2.06°2θ/min. Diffraction data were initially analysed using PANalytical X’Pert HighScore Plus version 4.1 software coupled to the latest version of the International Centre for Diffraction Data (ICDD) database.

Following identification of mineral species, quantification was achieved using the Rietveld refinement technique (e.g. Snyder & Bish, 1989) using PANalytical X’Pert HighScore Plus software. Errors for the quoted mineral concentrations are typically better than ±1% for concentrations >50 wt.%, ±5% for concentrations between 20 and 50 wt.%, and ±10% for concentrations <10 wt.% (Kemp et al., 2016a). Where a phase was detected but its concentration was indicated to be below 0.5%, it is assigned a value of <0.5%, as the error associated with quantification at such low levels becomes too large.

The <2.0, 2.0–0.2, 0.2–0.1, 0.10–0.05 and <0.05 µm oriented mounts were scanned from 2 to 40°2θ at 1.02°2θ/min after air-drying, ethylene glycol solvation and heating at 550°C for 2 h. Clay mineral species were then identified from their characteristic peak positions and their reaction to the diagnostic testing program (Moore & Reynolds, 1997). Modelling of the <2 µm XRD profiles was carried out using Newmod-for-Window3™ (Reynolds & Reynolds, 1996) software following the procedure outlined by Kemp et al. (2016b).

For capillary analysis, the configuration of the diffractometer was altered to convert the diverging X-ray beam into a high-intensity, narrow parallel beam and Kβ radiation was removed using a PANalytical parabolic mirror. Each capillary and brass collet was individually mounted in the goniometer head of the diffractometer and aligned to the X-ray beam using a bespoke microscope attachment. The capillary was rotated at a speed of 5 revolutions/s. To increase the counting statistics obtained from such small amounts of material, extremely slow scans from 5 to 75°2θ were performed at a speed of 0.16°2θ/min.

In order to remove the component of the XRD trace produced by the borosilicate glass capillary, an empty capillary was scanned using the same analytical program and the ‘blank’ subtracted from each sample trace.

Illite polytypes were identified from their characteristic peak positions, intensities and widths measured using PANalytical X’Pert HighScore Plus version 4.1 software. Peak data were then compared with standard data from the latest version of the ICDD database, publications (e.g. Dalla Torre et al., 1994; Grathoff & Moore, 1996; Haines & van der Pluijm, 2008) and modelled profiles obtained from the WILDFIRE® software package (Reynolds Jr, 1994). No attempt was made to differentiate between mica/illite 1M and 1M₄ polytypes in this study.

K–Ar analysis

The K–Ar dating technique followed standard methods described in detail by Dalrymple & Lanphere (1969). K content was determined by atomic absorption with a pooled error of duplicate K determinations (on several samples and standards) better than 2.0%. Ar isotopic measurements were based on a procedure described by Bonhomme et al. (1975). Clay fraction sample portions were preheated under vacuum at 80°C for several hours to reduce the amount of atmospheric Ar adsorbed onto the mineral surfaces during sample preparation. Blanks for the mass spectrometer and extraction line were systematically determined and the mass discrimination factor was determined by airshots (small amounts of air for 40Ar/36Ar ratio measurement). Approximately 10 mg of sample material was required for Ar analyses. Special care was taken in the preparation of both K and Ar sample portions due to the hygroscopic nature of clays. For Ar analysis by noble gas spectrometry, sample portions were loaded onto clean Mo foil (Goodfellow molybdenum foil, thickness 0.0125 mm, purity 99.9%), weighed and subsequently preheated to 80°C overnight to remove moisture, and reweighed using a Mettler AT20 balance. The measured dry weight was used in the K–Ar age calculation. Samples were stored prior to loading into the Ar purification line in a desicator. During the analysis, seven international standards (3 HD-B1, 3 LP6 and 1 GLO) were measured. The error for Ar analyses was below 1.00% and the 40Ar/36Ar value for airshots averaged 295.46 ± 0.37 (n = 4) (Tables 1, 2). The K–Ar ages were calculated using the 40K abundance and decay constants recommended by Steiger & Jäger (1977). The age uncertainties take into account the errors during sample weighing, 40Ar/36Ar and 40Ar/38Ar measurements and K determination. The reported errors for K–Ar age values represent 2σ uncertainty. Ages are stated according to the stratigraphical timescale of Gradstein et al. (2004).

Results

Petrographic analysis

The three mutually orthogonal thin sections show a dense, brown (hematite-bearing), cryptocrystalline ‘clay’ matrix enclosing scattered 100 µm- to centimetre-scale porphyroclasts of quartz and lithic fragments (Fig. 3a). The lithic fragments, which are probably derived from both granitic and psammitic protoliths, are hematized to varying degrees (Fig. 3a). Some of the fragments consist of cataclastic fault rock (clasts in a matrix), with a fabric of brittle–ductile character (at the 100 µm–cm scale) that can be markedly discordant with that in the enclosing fault gouge. The deformation within these fragments clearly predates the deformation that produced the enclosing clay-rich matrix, but it is not

| Table 1. K–Ar standards data. Standard error to references GLO (Odin et al., 1982). HD-B1 (Hess & Lippolt, 1994) and LP6 (Odin et al., 1982). |
|---|---|---|---|---|---|
| Standard | K (%) | 40Ar mol/g | Age (Ma) | Error (Ma) | Error to reference (%) |
| GLO-157 | 6.55 | 1.1093E−9 | 94.5 | 95.1 | 1.5 | +0.06 |
| HD-B1-122 | 7.96 | 3.3607E−10 | 92.2 | 24.2 | 0.4 | −0.08 |
| HD-B1-123 | 7.96 | 3.3590E−10 | 92.1 | 24.2 | 0.4 | −0.12 |
| HD-B1-124 | 7.96 | 3.3776E−10 | 92.6 | 24.3 | 0.4 | +0.41 |
| LP6-136 | 8.37 | 1.9254E−9 | 97.2 | 128.0 | 2.0 | +0.06 |
| LP6-137 | 8.37 | 1.9398E−9 | 97.6 | 128.7 | 1.9 | +0.59 |
| LP6-138 | 8.37 | 1.9237E−9 | 96.7 | 127.9 | 1.9 | −0.02 |

| Table 2. Airshot data. |
|---|---|---|
| Airshot ID | 40Ar/36Ar ± | ± |
| AS118-AirS-1 | 295.17 | 0.56 |
| AS119-AirS-1 | 296.36 | 0.34 |
| AS119-AirS-2 | 294.76 | 0.30 |
| AS120-AirS-1 | 295.55 | 0.29 |
clear if it occurred during an earlier episode of fault displacement or earlier in the same episode that produced the enveloping clay-rich gouge. One thin section, cut vertically and normal to the fault plane, shows a strong foliation dipping ∼70° towards SSE, with subtle kinematic indicators (e.g. mantled porphyroclasts) suggesting a possible reverse sense of movement (SSE side up) in the lifetime of the fault structure (Fig. 3a). A second thin section, cut vertically and parallel to the fault plane, is essentially isotropic (lacking a foliation). The third, cut horizontally, shows a relatively weak ENE–WSW-trending foliation with a sinistral sense of displacement (developed at some point in the lifetime of the fault structure) and both sliding and rotation of objects/domains in the shear fabric.

Taken together, the preserved evidence indicates that a sinistral–oblique deformation was a significant part of the history of movement on this fault structure. Any foliation developed in association with the earlier phase(s) of displacement (indicated by the porphyroclasts of deformed rock) has been obliterated by the fabric developed in association with later displacement. There is no microscopic evidence that the fabric now dominating the fault gouge has been modified since it formed by subsequent displacement on the fault.

The fourth thin section, cut to include a boundary between dominantly brown and dominantly grey fault gouge, reveals a highly complex distribution of the two colour variants at the thin-section scale (and evidence for sinistral shear), but confirms that the gouge otherwise displays no discernible difference in character (Fig. 3a,b).

Higher-magnification SEM examination provided further evidence of the very fine-grained nature and oriented fabric of the
clay-rich gouge (Fig. 3c,d). As well as the larger quartz and lithic grains, occasional silt- and sand-grade, generally sub-rounded quartz, K-feldspar, Ti oxide and apatite grains were observed together with chlorite flakes and submicron monazite grains (Fig. 3c,d). The EDX analyses suggest that the gouge matrix is composed of a mixture of illite and chlorite (Fig. 3e).

**X-ray diffraction analysis**

In broad agreement with the petrographic analysis, powder XRD analysis indicates that the whole-rock mineralogy of the fault-gouge samples is dominated by undifferentiated mica (‘mica’ possibly including muscovite, biotite, illite and I-S) and quartz with subordinate amounts of K-feldspar and chlorite and traces of calcite. Minor amounts of plagioclase feldspar were detected in sample VY361/G1, along with traces of hematite in the brown samples (Table 4). Reconnaissance <2 µm oriented-mount XRD indicated that the whole-rock mineralogy of the fault-gouge samples and their full width at half maximum (FWHM) measurements.

**Table 4. Summary of the relative proportions of clay minerals in the <2 µm fractions extracted from the fault-gouge samples and their full width at half maximum (FWHM) measurements.**

| Sample          | Illite (%) | Chlorite (%) | Non-clay minerals | FWHM (3°2θ) |
|-----------------|------------|--------------|-------------------|-------------|
| VY352/G1, brown | 96         | 4            | Quartz, hematite  | 0.88        |
| VY352/G2, brown | 93         | 7            | Quartz, hematite  | 0.71        |
| VY361/G1, grey  | 93         | 7            | Quartz            | 0.77        |

**Table 5. Summary of size-fraction clay mineral assemblages and K-Ar analyses.**

| Sample          | Size fraction (µm) | Proportion of clay minerals in size fraction (%) | Newmod II crystallite size dist. (low-high n, mdff) | Proportion of illite, Chlorite (%) | K (%)   | Rad. 40Ar (mol/g) | Rad. 40Ar (%) | Age (Ma) | Error (Ma) |
|-----------------|--------------------|-----------------------------------------------|-----------------------------------------------|-----------------------------------|---------|------------------|---------------|----------|------------|
| VY352/G1, brown | 2.0–0.2            | 84, 16                                        | 0.590, 0.348                                 | 1–21, 11, 1–30, 10                | 4.81    | 2.427E–2         | 97.02         | 269.7    | 5.4        |
|                 | 0.2–0.1            | 95, 5                                         | 0.718, 0.493                                 | 1–19, 7, 1–21, 8                  | 5.49    | 2.333E–3         | 93.23         | 229.7    | 4.6        |
|                 | 0.10–0.05          | 99, 1                                         | 0.898, 0.747                                 | 1–12, 6, 1–13, 7                  | 5.51    | 1.923E–3         | 89.20         | 190.8    | 3.8        |
|                 | <0.05              | 99.5, 0.5                                     | 1.242, 1.220                                 | 1–10, 4, 1–9, 4                   | nd      | 4.94, 1.327E–3   | 81.64         | 146.6    | 3.0        |
| VY352/G2, brown | 2.0–0.2            | 83, 17                                        | 0.573, 0.334                                 | 1–22, 11, 1–32, 10                | 4.94    | 2.546E–3         | 97.26         | 275.0    | 5.6        |
|                 | 0.2–0.1            | 95, 5                                         | 0.686, 0.483                                 | 1–19, 8, 1–22, 8                  | 5.67    | 2.551E–3         | 94.66         | 242.3    | 4.9        |
|                 | 0.10–0.05          | 98, 2                                         | 0.924, 0.761                                 | 1–12, 7, 1–13, 7                  | 5.63    | 2.082E–2         | 90.79         | 201.6    | 4.1        |
|                 | <0.05              | 99.4, 0.6                                     | 1.324, 1.403                                 | 1–9, 3, 1–8, 4                    | nd      | 4.80, 1.259E–3   | 79.95         | 145.2    | 2.9        |
| VY361/G1, grey  | 2.0–0.2            | 83, 17                                        | 0.507, 0.295                                 | 1–26, 12, 1–40, 10                | 5.48    | 2.848E–3         | 97.33         | 277.2    | 5.6        |
|                 | 0.2–0.1            | 91, 9                                         | 0.690, 0.480                                 | 1–19, 8, 1–22, 8                  | 5.67    | 2.564E–3         | 94.51         | 243.5    | 4.9        |
|                 | 0.10–0.05          | 98, 2                                         | 0.948, 0.727                                 | 1–11, 6, 1–14, 7                  | 5.45    | 2.083E–3         | 91.34         | 207.9    | 4.2        |
|                 | <0.05              | 99.4, 0.6                                     | 1.449, 1.216                                 | 1–9, 3, 1–7, 4                    | nd      | 4.81, 1.348E–3   | 79.66         | 154.7    | 3.2        |

nd = not detected; dist. = distribution; mdff = mean defect free distance.
Despite the excellent random orientations achieved, illite polytype identification was still a difficult task due to peak overlap and the paucity of diagnostic reflections (Fig. 7). Initial examination suggested that the coarser size separates were composed of greater proportions of 2M1 illite, while the finer grain sizes showed increasing proportions of 1M illite. No evidence was obtained for 2M1 polytype in the finest-size separates (<0.05 µm).

A range of approaches was trialled for quantifying the proportions of each illite polytype. The WILDFIRE© (Reynolds Jr, 1994) modelling approach, favoured in several recent studies (e.g. Solum & van der Pluijm, 2007; Haines & van der Pluijm, 2008; Song et al., 2014), proved successful at generating generally realistic 2M1 polytype profiles. However, despite repeated attempts using the full range of available options, the generated profiles failed to provide satisfactory simulations for the observed 1M illite. This inability to match the 1M illite profile in the sample’s finest fractions, where it is found in its greatest abundance, means that any quantification in coarser fractions, where its degree of crystal order might be different, is highly uncertain. Similar difficulties with WILDFIRE© modelling have been experienced by other current researchers (Vrolijk et al., 2018; A. Schleicher, pers. comm., 2017; M. Raven, pers. comm., 2019). Quantification using the Rietveld refinement technique (Rietveld, 1969; Bish & Post, 1993) was also precluded by a lack of suitable structural data for illite polytypes in the Inorganic Crystal Structure Database (2018).

Therefore, in this study, we reverted to a more simplistic, corrected peak-area-measurement methodology. Dalla Torre et al. (1994) conducted a review of XRD mica polytype quantification methods and recommended the approach of Callière et al. (1982), and these guidelines were followed here. Specifically, this involved measuring the peak areas (I) of the 2M1-specific 3.00 Å and 1M-specific 3.06 Å peaks (Fig. 7) and ratioing these using the simple formula:

\[
\%2M_1 = \frac{I(3.00 \text{ Å})}{I(3.00 \text{ Å}) + I(3.06 \text{ Å})}
\]  

(1)

As the chosen peaks form slightly different relative intensities (3.00 Å, 76%; 3.06 Å, 83%), these were further refined by normalizing on the basis of peak intensity data from ICDD (2014) standard patterns (Fig. 7, Table 5).

To establish absolute errors for the quantification method, Dalla Torre et al. (1994) produced a series of synthetic mixtures of pure 2M1 and 1M polytypes and determined their proportions using the polytypic reflections suggested by Callière et al. (1982). The resultant calibration curve showed a high degree of reproducibility and a good correlation \( R^2 = 0.99 \). Ten samples containing 50% 2M1 produced a mean value of 0.47 (standard deviation, \( \sigma \): 0.02) and an absolute error of ± 2%. A more conservative error of ± 5% was applied in this study.

**K-Ar analysis**

Twelve K–Ar illite ages, covering four grain-size ranges comprising: (1) a very fine <0.05 µm size; (2) intermediate 0.10–0.05, 0.20–0.1 µm sizes; and (3) a coarse 2.0–0.2 µm size were obtained (Fig. 8, Table 5). The K–Ar ages range from 145.2 ± 2.9 Ma (Lower Cretaceous–Berrisian, VY352/G2, <0.05 µm) to 277.2 ± 5.6 Ma (Permian–Cisuralian–Artinskian, VY361/G1, 2.0–0.2 µm). Radiogenic 40Ar contents range from 79% to 97% indicating reliable analytical conditions for all analyses with no significant radiogenic 40Ar contamination. K contents range from 4.80% to 5.67%. The lower K concentration in some size fractions is caused by contamination with other mineral phases, such as quartz and chlorite, which is supported by XRD data (Table 4).

The K–Ar ages increase with increasing grain sizes (Fig. 8). A detailed comparison of the analyzed grain-size fractions with a discussion of mineralogical constraints is presented below. In general, illite content increases with decreasing grain size (Table 5, Fig. 8). The 2.0–0.2 µm fractions yield ages within error and an average age of 274.0 ± 5.5 Ma, suggesting an homogeneous illite source. The K content for the coarse fractions ranges from 4.81% to 5.48%, with the range reflecting contamination with non-K-bearing mineral phases (Table 4).
The 0.2–0.1 µm size fractions yield ages ranging from 229.7 ± 4.6 Ma to 243.5 ± 4.9 Ma. K content ranges from 5.49% to 5.67%, caused by minor changes in mineralogy (Table 5). The 0.10–0.05 µm fractions yield ages ranging from 190.8 ± 3.8 Ma to 207.9 ± 4.2 Ma with similar K contents to the 0.2–0.1 µm grain size, ranging from 5.45% to 5.63% and again caused by minor variations in mineralogy (Table 5).

The <0.05 µm fractions yield ages ranging from 145.2 ± 2.9 Ma to 154.7 ± 3.2 Ma and contain less, homogeneous K, ranging from 4.80% to 4.94%, again caused by minor mineralogical variations (Table 5).

**Illite Age Analysis modelling**

A series of IAA plots was produced using the proportion of the 2M1 polytype plotted against the function $\exp(-\lambda t)$ (where $\lambda$ is the decay constant of potassium and $t$ is time) with error bars of ±5% applied. The ‘inclined spectra’ (Pevear, 1999)
shown in Fig. 9 are conceptually similar to those obtained by other fault studies (Torgersen et al., 2015). As noted by Haines & van der Pluijm (2008), the percentage of $2M_1$ illite is linearly related to the exponential decay of potassium and not to chronological time. Excellent $R^2$ values of $>0.97$ were obtained with predicted mean ages of $296 \pm 7$ Ma (‘Faulting event 1’) and $145 \pm 7$ Ma (‘Faulting event 2’) (Fig. 9a,b,c). The similarity of the IAA dates suggests that trace amounts of K-feldspar in the coarse (2.0–0.2 $\mu$m) fraction of sample VY361/G1 has had a negligible effect, as increased temperatures in the fault zone might have partially or fully reset its isotopic signatures as described by Zwingmann et al. (2010).

**Geological interpretation**

Field evidence and the dating of synkinematic plutons suggest a mid- to late-Silurian age for the initiation of sinistral displacements on the major Scottish strike-slip faults such as the GGFZ (e.g. Stewart et al., 2001). Based on the presence of overlying Devonian sedimentary rocks, sinistral movements along the GGFZ ceased by c. 390 Ma (Stewart et al., 1999). Primary, sinistral strike-slip displacements along the Walls Boundary Fault Zone, a northerly extension of the GGFZ, were probably initiated later than movements in the GGFZ as a whole (before c. 400 Ma) and ceased before c. 370 Ma (Watts et al., 2007).

Based on field observation and these previous indirect dating studies, we expected our illite dating study to prove similar ages for the Sronlairig Fault gouge. Our initial hypothesis envisaged that the oldest event age from the Sronlairig Fault gouge would reflect mica derived from Dalradian metasedimentary rocks (south wall, >470 Ma) and/or the Allt Crom Pluton (north wall, c. 434 Ma). These would almost certainly have been reset during the metamorphism associated with the Caledonian orogeny (490–390 Ma; McKerrow et al., 2000). However, the IAA age of $296 \pm 7$ Ma for Faulting event 1 is much younger than the wall rock or orogenic features and therefore requires a different interpretation (Fig. 10).

**Faulting event 1 age**

The Faulting event 1 age might reflect mica derived from Late Carboniferous protolith, but we consider this extremely improbable due to a lack of suitable sources. Instead, the age may reflect the full or partial resetting of earlier-formed, inherited or authigenic micas (resulting in lost Ar or gained K) or the authigenic, synkinematic crystallization of $2M_1$ illite-muscovite during an
earlier, hotter fault-displacement event. Diffusion of $^{40}$Ar may have been caused by regional heating, frictional heating or high-temperature fluid advection during fault reactivation (e.g. Tagami, 2012; Tonai et al., 2016).

Generally, the Earth has a one-dimensional (vertical) temperature profile, averaging $\sim$30°C/km in the upper continental crust (Ehlers, 2005). At a regional scale, the background thermal history depends on a range of factors, including surface topography, spatial variation of geothermal structure, tectonic tilting and ductile deformation at depth (Tagami, 2012). Normal or slightly elevated geothermal gradients as a result of end-Caledonian granite plutonism are suggested as appropriate during the earlier stages of shearing along the GGFZ (Stewart et al., 1999). The development of 2$M_1$ illite in a brittle fault gouge due to cooling of the host rock and its passage through epizonal conditions contemporaneous to faulting and retrograde metamorphism (Bense et al., 2014) is improbable in the Sronlairig Fault.

Brittle deformation may cause fault frictional heating, which is characterized by episodic, several-second temperature increases of up to 1000°C that reach several millimetres into the wall rock (Brown, 1998; Tagami, 2012). Such changes can result in melting and, where pressures exceed 0.7 GPa, the development of pseudotachylytes (Lavallée et al., 2015). Leloup et al. (1999) suggested shear heating increases of $\sim$590°C at the Moho, $\sim$475°C at 20 km depth and lower temperatures (still sufficient to cause high-grade metamorphic conditions) in strike-slip shear zones.

Mineralogical response such as crystal–plastic straining of quartz (250–300°C) and fracturing and brittle deformation of feldspar (<500°C) provide useful indicators of syntectonic temperatures in the GGFZ (Stewart et al., 1999). Micas deform mainly by mechanical sliding along basal 00l crystal planes, but may also become kinked or fragmented and locally experience intense subgrain development and dynamic recrystallization close to the centre of the GGFZ (Stewart et al., 1999).

Fig. 8. Comparison of the clay mineralogy and K–Ar age data for the isolated size fractions. Dashed lines indicate the mean K–Ar age for the differently sized fractions.

Fig. 9. IAA plots for the three Sronlairig Fault samples (a) VY352/G1, (b) VY352/G2 and (c) VY361/G1. Fraction error bars reflect ±5% error in quantification of the %2$M_1$ illite polytype. Propagated errors for end-member ages are shown in labels only.
Fluid advection, often related to volcanism, with temperatures above the Ar-closure temperature of micas (>280°C), may also be responsible for Ar-resetting in the micas, as noted in faults and thrust zones elsewhere (e.g. de Jong et al., 1992; Kondo et al., 2005; Rowe et al., 2009; Tagami, 2012). Petrographic examination of red- to brown-coloured gouges from the Walls Boundary Fault (Fig. 1a) suggests widespread sericitization and chloritization reactions attributed to chemically active fluid alteration (Watts et al., 2007). These hematitic, phyllosilicate-rich gouges appear to be very similar to those observed in the present Sronlairig Fault study (Fig. 3). However, the Sronlairig Fault gouge appears to lack veins of calcite, quartz and ore deposits typically developed by fluid advection and in situ chemical precipitation that might offer other dating opportunities (e.g. U–Th disequilibrium; Watanabe et al., 2008).

Stewart et al. (1999) noted that the original feldspar-dominated protoliths along the GGFZ produced load-bearing frameworks that were converted to weaker, phyllosilicate-dominated, foliated cataclasites and phyllonites. Any post-Devonian reactivation appears to have been concentrated within or along the margins of these hydrated assemblages.

The ~20–30 km of dextral displacement on the GGFZ (Coward et al., 1989; Rogers et al., 1989), considered to be of late Carboniferous age on the basis of regional geological considerations (Séranne, 1992), would appear to offer the most probable event for resetting of early-formed micas in the Sronlairig Fault or authigenesis of 2M1 illite-muscovite (Fig. 10). The dextral movement has also been recognized on the Walls Boundary Fault by an inversion event within the Devonian rocks of Shetland, as well as the Orcadian Basin on Orkney and in northern Scotland (Watts et al., 2007). Further north, significant Carboniferous to Permian movements are also seen along the Norwegian Møre–Trøndelag Fault Complex, although these are sinistral rather than dextral (e.g. Osmundsen et al., 2006).

Post-Caledonian tectonic patterns were established in response to the movement of Baltica relative to Laurussia (Coward, 1993). In the Late Carboniferous, regional stresses generated by the relative south-westwards movement of Baltica drove dextral shear in the GGFZ, coincident with strongly partitioned strain in the North Sea basin interior. The Sronlairig Fault is therefore responding along with other structures in this region (e.g.
the Banff and Wick faults) to the WNW(NW)–ESE(SE) extension, which becomes important for basin development and hydrocarbon migration in the Pentland and Moray Firth regions (Dichiarante et al., 2016; Leslie et al., 2016).

Faulting event 2 age

The faulting event 2 IAA age may relate to an instantaneous event such as contact metamorphism (e.g. Aronson & Lee, 1986) or hydrothermal activity (e.g. Lonker & FitzGerald, 1990) or alternatively to gradual burial activity (e.g. Grathoff & Moore, 1996). Previous studies have demonstrated that the age of the finest fraction constrains the timing of faulting (or last movement), as this fraction is usually dominated by the most recently grown authigenic illite (Torgersen et al., 2013).

The lack of any foliation and the preservation of the syntectonic fabric within the clay gouge suggests only very minor (if any) movement post-illite growth. Whether the illite formed during individual slip events, gradually during aseismic creep or between individual seismic events due to fluid flow along the fault is unclear (cf. Haines & van der Pluijm, 2008). As the gouge provides no evidence of further slip, direct precipitation of the 1M illite polytype would indicate formation temperatures of <200°C (Velde, 1965).

Alternatively, the illite-rich assemblage in the Sronlairig Fault gouge may, in part, result from burial metamorphism of a precursor, authigenic smectite or I-S. The completion of the thermally driven dehydration of smectite to illite would have required temperatures at the higher end of the 120–150°C range (Merriman & Kemp, 1996). There is no evidence for contact metamorphism or further extensive hydrothermal activity at this time in the region.

In a rare case, Duvall et al. (2011) noted that the authigenic illite age does not always represent the latest phase of fault motion and may document the initiation of faulting. Those authors also suggested that, using XRD analysis, it would be impossible to distinguish between different preserved populations of authigenic 1M illite produced by multiple fault-slip episodes. Here, the pseudauthigenic illite age would record a mixture of ages rather than the time of a particular event. However, in this case, the excellent linear relationship between the Sronlairig Fault illite age and the %2M1 illite-muscovite component (Fig. 9), requiring illite polytypes to have grown in the same relative proportions during each event for all size fractions, is strongly indicative of a single faulting episode (or successive events within the uncertainty of the dating methodology) (Duvall et al., 2011).

The Late Jurassic–Early Cretaceous (145 ± 7 Ma) faulting event 2 IAA age does not correspond with any known local geological event, but coincides with possible dextral movements along the GGFZ and Mesozoic rift, graben and basin development in the Inner Moray Firth (McQuillan et al., 1982) (Figs 1, 10). Later seismic interpretations have disputed the contribution of the GGFZ to this episode of basin development, arguing that the GGFZ lay dormant during the Mesozoic (Underhill, 1991). However, the GGFZ and related structures may have helped to partition active extension in the Moray Firth region from relative inactivity in the Pentland Firth area (Thomson & Underhill, 1993; Dichiarante et al., 2016).

The GGFZ resumed activity as a divergent wrench structure as part of limited oblique slip in the Tertiary Alpine Orogeny, probably contemporaneous with uplift of the Scottish Highlands during Paleocene–Eocene northeast Atlantic rifting (Underhill, 1991) or Oligo-Miocene tectonics (Thomson & Underhill, 1993; Underhill & Brodie, 1993). More recently, field observations of Jurassic outcrops in northeast Scotland and their ‘beef’ structures suggest right-lateral reactivation of the order of 10–18 km during the Late Eocene to Late Oligocene, c. 37–26 Ma (Le Breton et al., 2013).

The Late Jurassic–Early Cretaceous Faulting event 2 IAA age therefore suggests that the Sronlairig Fault was only subject to low-temperature frictional heating and low-temperature fluid flow during the Tertiary period. The drive of the right-lateral reactivation of the GGFZ and the heat provided by the Iceland mantle plume were clearly insufficient to reset the 1M illite component of the Sronlairig Fault gouge.

Conclusions

Fault cores and their component fault rocks are very rarely preserved in natural exposures, and especially so in the Scottish Highlands, where differential erosion along faults and the degree of vegetation act together to often completely obscure such features. Temporary exposures therefore provide important opportunities to interrogate regionally important tectonic frameworks.

By employing novel capillary XRD analysis techniques to facilitate polytype identification and quantification, IAA of gouge from the Sronlairig Fault has revealed two event ages of 296 ± 7 and 145 ± 7 Ma, respectively.

We suggest that the earlier age may either represent resetting of the wall rock-derived Sronlairig Fault micas or growth of authigenic 2M1 illite-muscovite during Late Carboniferous–Early Permian dextral displacement of structures related to the GGFZ. The Late Jurassic–Early Cretaceous age coincides with possible dextral movements along the GGFZ and related structures, helping to partition active extension in the Moray Firth region from relative inactivity in the Pentland Firth area.

These new age dates therefore record younger parts of the record of the long-lived activity on the GGFZ. In contrast to the offshore region where the Mesozoic to Cenozoic lithostratigraphy provides good constraints for deformation superimposed on those successions, the absence of these younger successions onshore means that such constraints are lacking in those regions. However, movements associated with the GGFZ are unlikely to have been restricted to the Moray Firth region at this time. These new data and this methodology reveal important new insights into an integrated regional-scale record of tectonic displacement in the GGFZ that has the potential to link the onshore and offshore domains.

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