THE TIMING OF GOLD MINERALIZATION AT THE RATTLING BROOK DEPOSIT, WHITE BAY: CONSTRAINTS FROM Re–Os AND ⁴⁰Ar–³⁹Ar GEOCHRONOLOGY

A. Kerr, O. van Breemen¹, and R.A. Creaser² Mineral Deposits Section

ABSTRACT

The Rattling Brook deposit is a low-grade, disseminated to stockwork-style gold deposit hosted by Precambrian granites and Cambrian sedimentary rocks in western Newfoundland. Gold mineralization and alteration is also present in foliated and metamorphosed mafic rocks, which are interpreted as pre-mineralization metadykes, possibly late Precambrian in age. Auriferous granites are, in turn, cut by relatively fresh, unaltered, locally chilled diabase dykes, interpreted as post-mineralization Paleozoic intrusions.

Attempts to date pyrite and arsenopyrite directly using Re-Os geochronology produced imprecise data due to very low Os content. The isochron ages of 327 to 315 Ma have large errors and seem unreasonably young. Better age constraints are provided by identical ${}^{40}Ar - {}^{39}Ar$ ages from a gold-bearing metadyke and a post-mineralization diabase. The post-mineralization diabase gave an amphibole age of 413 ± 4.3 Ma, which is interpreted as the time of its crystallization, and provides a younger limit for the timing of gold mineralization. The pre-mineralization metadyke has a mean biotite age of 412.3 ± 2.3 Ma, which can be interpreted in two ways. It could represent post-metamorphic cooling, or it could represent resetting of metamorphic biotite during alteration related to gold mineralization. In either case, the time of mineralization is not likely to greatly exceed the ${}^{40}Ar - {}^{39}Ar$ biotite age, provided that the ambient temperature during the mineralization event was not significantly above the closure temperature for argon isotopic systems in biotite (~300°C). On this basis, gold mineralization at Rattling Brook occurred between 419.6 and 408.7 Ma, during the earliest Devonian.

The result resembles a latest Silurian U-Pb age $(420 \pm 5 \text{ Ma})$ for hydrothermal zircon in the Stog'er Tight gold deposit, but is older than Middle to Late Devonian U–Pb ages for the Nugget Pond gold deposit $(384 \pm 8 \text{ Ma})$ and the Titan gold prospect $(<381 \pm 5 \text{ Ma})$. Curiously, the two age-groupings defined by these Newfoundland data resemble those indicated by recent Re–Os dating of vein-hosted gold mineralization in the Meguma Zone of Nova Scotia. There is an obvious need for better timing constraints on gold mineralization, from the perspective of metallogenic studies and also regional tectonic syntheses.

INTRODUCTION

Establishing the exact timing of epigenetic hydrothermal mineralization is a persistent problem in metallogenic studies. In some cases, host rocks can be dated precisely using U–Pb geochronology, or other whole-rock methods, but this provides only a maximum age constraint. If mineralization occurs in pegmatites that can be convincingly linked to local plutonic rocks, the latter may yield age constraints. However, many epigenetic deposits, such as mesothermal gold lodes or Carlin-type gold deposits, may not be of magmatic origin, and quartz or quartz-carbonate veins generally do not contain minerals amenable to radiometric dating. It may also be possible to constrain epigenetic mineralization by direct dating of hydrothermal alteration minerals, e.g., sericite, using K–Ar or Ar–Ar techniques.

¹ Geological Survey of Canada, 601 Booth Street, Ottawa, ON, K1A 0E8

² Department of Earth Sciences, University of Alberta, Edmonton, AB, T6G 2E3

However, interpretation depends on the subjective link between mineralization and alteration, and some host rocks may have unrelated pre- or post-mineralization alteration. In Carlin-type deposits, the rare ore-stage mineral galkhaite is amenable to precise Rb–Sr dating; this technique indicates that several large deposits in Nevada formed during Eocene times, whereas previous dating of alteration assemblages indicated a wide range of post-Carboniferous ages (Arehart *et al.*, 2000; Hofstra and Cline, 2000). Mesothermal-type gold veins rarely contain ore-stage minerals amenable to dating, and they are difficult to constrain.

The problem of dating epigenetic gold deposits is illustrated very well by Paleozoic gold mineralization in the Newfoundland Appalachians. On a regional scale, most of the gold deposits on the Island are hosted by Cambrian to Ordovician volcanic and/or sedimentary rocks of the Dunnage Zone. Only a few examples of gold mineralization are known in Silurian rocks, most notably in the Sops Arm Group, White Bay (Saunders, 1991; Kerr, this volume) and the Botwood and/or Indian Islands groups north and west of Gander (Squires, 2005; McNicol et al., this volume). A general perception that epigenetic gold mineralization in Newfoundland is broadly Silurian, is based on an inferred linkage to "Salinic" deformation, metamorphism and plutonism, prevalent throughout the Central Mobile Belt (e.g., Tuach et al., 1988; Evans, 2001; Wardle, 2004). The direct constraints on the precise timing of gold mineralization are few in number, and not all of them support this idea.

EXISTING DATES FOR NEWFOUNDLAND GOLD DEPOSITS

At the Hammerdown deposit near Springdale, goldbearing veins cut Ordovician mafic volcanic rocks, and also cut felsitic dykes that share the structural trend of the veins. Ritcey et al. (1995) obtained a U–Pb zircon age of 437 ± 4 Ma from the felsitic dykes, and interpreted this as a maximum age for the mineralized quartz veins. At the Stog'er Tight deposit near Baie Verte, Ramezani (1992) obtained a U–Pb zircon age of 420 ± 5 Ma from "hydrothermal" zircon present in skarn-like alteration of the gabbroic host rock. Of these two ages, the first provides a maximum age constraint only, but the second is more diagnostic, assuming that the hydrothermal zircon does not itself represent a discrete event. At the Nugget Pond deposit, also on the Baie Verte Peninsula, gold mineralization occurs in sedimentary rocks within Ordovician mafic volcanic sequences above the Betts Cove ophiolite (Sangster and Pollard, 2001; Sangster, in press). Xenotime occurs in an unusual quartz-feldspar-carbonate alteration assemblage that is spatially associated with auriferous zones, and this mineral yielded a U-Pb age of 374 ± 8 Ma (R. Parrish, quoted in Sangster and Pollard, 2001). More recently, a U-Pb SHRIMP study on zircons

indicated a crystallization age of 381 ± 5 Ma for a mafic dyke that hosts gold-bearing veins at the Titan gold prospect north of Gander (McNicol *et al., this volume*), providing an upper age limit. At the Road Breccia showing south of Glenwood, a U–Pb SHRIMP zircon date of 411 ± 5 Ma was obtained from an unaltered mafic dyke that shares the trend of auriferous quartz veins cutting siltstones of the Indian Islands Group (McNicol *et al., this volume*). However, the lack of alteration in the dyke does not necessarily indicate that it is younger than the gold mineralization, as the alteration haloes around nearby quartz veins are very narrow. In summary, the five age determinations indicate that gold mineralization in Newfoundland occurred during the latest Silurian and middle Devonian.

POSSIBLE APPROACHES FOR DIRECT DATING

The ideal approach to dating mineralization is to use minerals that form part of the ore assemblage; in the case of epigenetic gold deposits, this means dating sulphides. Sphalerite is one possible candidate, as it can be dated using Rb-Sr techniques; however, it is not widely present in veinhosted gold mineralization. Regrettably, most of the radiogenic isotopes that have half-lives suitable for geochronology are simply not present in common sulphide minerals such as pyrite, pyrrhotite, chalcopyrite and arsenopyrite. The only exception is the Re-Os geochronometer, based on the decay of ¹⁸⁷Re to ¹⁸⁷Os. This method has been used to accurately date molybdenites in recent years, because this mineral contains abundant Re, but negligable Os; model ages can therefore be obtained from a single isotopic analysis (e.g., Stein et al., 2001). In the last decade, increasing analytical precision has allowed application of the Re-Os system to more common sulphide minerals, mostly through isochron methods. It has also been shown that some sulphides (notably pyrite and arsenopyrite) contain low levels of highly radiogenic Os, and can yield valuable geochronological data. These techniques were successfully used to date gold mineralization in Australia (Arne et al., 2001), Scandinavia (Stein et al., 2000) and also in Nova Scotia (Morelli et al., 2005), despite very low concentrations of radiogenic Os. The method is not necessarily applicable to all types of gold mineralization, as some (e.g., Carlin-types) have very low levels of Re and consequently extremely low Os contents; in such samples, blank corrections become significant (H. Stein, personal communication, 2004).

In cases where direct dating is impossible, it may be possible to constrain the timing of mineralization using mineralized host rocks and rocks that visibly postdate mineralization. This approach depends on reliable geological relationships, but it may work very well in specific cases. For example, Carlin-type gold deposits in Nevada are associated with unaltered to altered mafic and felsic dykes that may be derived from plutons at depth. The altered dykes locally contain gold mineralization (Henry and Boden, 1998; Ressel *et al.*, 2000a,b). The mineralized dykes and unaltered post-mineralization dykes give closely similar Eocene ⁴⁰Ar-³⁹Ar ages on primary biotite and secondary illite, and were thus probably emplaced synchronously with mineralization of the older carbonate rocks that they intrude (Ressel *et al.*, 2000a, b).

PROJECT OVERVIEW

The Rattling Brook gold deposit (McKenzie, 1987; Saunders and Tuach, 1991; Kerr, 2004, 2005) was chosen as a test case for Re-Os dating in 2003 because its age has always been contentious, and the mineralization is rich in arsenopyrite. Rhenium-osmium analyses were conducted at the University of Alberta using mineralized samples from drill core. The results were inconclusive because Re and Os concentrations were very low, requiring proportionately large blank corrections. Further work in 2004 allowed the definition and sampling of both mineralized, metamorphosed mafic dykes and unmineralized fresh mafic dykes. Dating of hornblende and biotite separates from these preand post-mineralization dykes using ⁴⁰Ar-³⁹Ar methods provides better constraints on the age of the gold mineralization at Rattling Brook. This short article summarizes both datasets, and discusses future initiatives for constraining the age(s) of epigenetic gold mineralization in Newfoundland.

GEOLOGY AND MINERALIZATION

The Rattling Brook gold deposit is located in White Bay, western Newfoundland (Figure 1), and is the most significant gold deposit in the area. It was discovered in the early 1980s and was explored further by BP-Selco (e.g., McKenzie, 1986, 1987) and more recently by Kermode Resources. The geology and mineralization are described by McKenzie (1986, 1987), Saunders and Tuach (1988, 1991) and Kerr (2004, 2005). The regional geology of western White Bay is summarized by Smyth and Schillereff (1982) and Kerr (2005, *this volume*).

The Rattling Brook gold deposit is located along the unconformable contact between Precambrian granites and Cambrian sedimentary rocks of the Bradore and Forteau formations (Figure 1). The unconformity is well-preserved in outcrop and in drill core, but strong deformation is present in the overlying sedimentary rocks, including a major detachment within phyllites of the Forteau Formation (Kerr and Knight, 2004; Figure 1). This structure cuts out part of the stratigraphy, and places Cambrian dolostones (Port au Port Group) or lower Ordovician limestones (St. George Group) against the older Cambrian rocks (Figure 1). Gold mineralization at Rattling Brook is present in four main zones, the Incinerator Trail, Beaver Dam, Road, and Apsy zones (Figure 1). The gold is mostly hosted by Precambrian granites, but also occurs in Bradore Formation quartzites and the basal limestone member of the Forteau Formation at the Beaver Dam Zone and Apsy Zone. It is not present in phyllites, limestones and dolostones exposed to the west. The granites are cut by two generations of mafic dykes. The older dykes are metamorphosed, altered and gold-bearing, whereas the younger dykes are fresh and unaltered, and cut granite-hosted gold mineralization. Dykes are rarely observed in drill core from the sedimentary rocks, although they are present in some outcrops, where they are fresh and unaltered. Metamorphosed dykes are not seen in the Cambro-Ordovician sedimentary rocks, implying that the emplacement age of the older dykes is Precambrian.

Mineralization at Rattling Brook consists of disseminated and veinlet-style pyrite and arsenopyrite in granite, quartzite and limestone (Saunders and Tuach, 1988, 1991; Kerr, 2005). Alteration relationships suggest that there was an early episode of potassic alteration, manifested by secondary microcline in plutonic rocks and sericitic alteration in metadykes and quartzites. This was followed by silicification and local albitization associated with the introduction of gold. Gold grades vary widely, but most samples contain <4 ppm Au; the highest grades seem to be associated with mineralized dykes, mafic variants of the granite, or impure carbonate rocks at the Bradore-Forteau formational boundary (Kerr, 2004, 2005). The geochemical features of the mineralization suggest some similarities to sedimentaryrock hosted "micron" gold deposits (also known as "Carlintype") or non-carbonate-hosted disseminated gold mineralization (e.g., Poulsen et al., 2000), rather than to gold-bearing quartz veins of mesothermal or "orogenic" type (e.g., Groves et al., 1999; Poulsen et al., 2000). Further discussion of the local geology and the gold mineralization are given by Saunders and Tuach (1991), Kerr (2004, 2005) and Kerr and Knight (2004).

From the perspective of this study, the relationships between gold mineralization and mafic dykes are critically important. Exploration work recognized the presence of "early-series" and "late-series" dykes (McKenzie, 1987) but did not clearly distinguish between them in all areas.

The mafic rocks that host alteration and mineralization are foliated and recystallized, suggesting that they also predate metamorphism and deformation. Many of these metadykes are biotite-rich. In mineralized metadykes, sericitic alteration visibly penetrates along foliation planes, but also overprints the fabric, and is visibly overprinted by siliceous alteration zones associated with the sulphides. Tuach and French (1986) and Kerr (2005) suggested that the mineralized metadykes belong to the Precambrian Long



Figure 1. Geological map of the Rattling Brook gold deposit and surrounding area, showing the locations of drillholes containing samples analyzed in Re–Os and ${}^{40}Ar$ – ${}^{39}Ar$ studies. For location of the map area and details of regional geology, see *Kerr* (2005).

Range dyke swarm, dated elsewhere at ~615 Ma (Stukas and Reynolds, 1974; Kamo *et al.*, 1989). These dykes commonly display a metamorphic and structural overprint of Paleozoic age, which is most intense in the southeastern part of the Long Range Inlier, where the study area is located. In this area, metamorphic mineral assemblages in the dykes indicate upper greenschist- to epidote–amphibolite-facies conditions (Owen, 1991), suggesting peak metamorphic temperatures up to 450°C. The timing of Paleozoic metamorphism is not well constrained.

Unaltered and unmineralized diabase dykes are very fresh in appearance, retain igneous textures including plagioclase phenocrysts, and have well-preserved chilled margins. These dykes cut auriferous granites in outcrop and drill core, but contain no alteration of any kind and lack sulphides. Geochemical data (A. Kerr, unpublished data) indicate only background levels of gold; these dykes are thus viewed as post-mineralization. Crosscutting relationships between the two generations of dykes have not been observed in outcrop or in drill core. Similar dykes cut Cambro-Ordovician carbonate rocks on the shores of Coney Arm (northeast of the area shown in Figure 1), implying that the post-mineralization dykes are Middle Ordovician or younger (Kerr and Knight, 2004).

The K-feldspar porphyritic granodiorite that forms the predominant host to mineralization (the Apsy Granite, Figure 1) was dated at ~1006 Ma using U-Pb zircon methods (Heaman et al., 2002), but the dykes have never been dated in this part of the Long Range Inlier. The gold mineralization was initially suggested to be Precambrian, because it could not be traced across the unconformity into adjacent Paleozoic rocks (Tuach and French, 1986). However, subsequent exploration documented gold in Cambrian quartzites and limestones, indicating that mineralization must be Paleozoic (Tuach, 1987). Saunders and Tuach (1991) suggested a Silurian or post-Silurian timing because there is gold mineralization in Silurian sedimentary rocks elsewhere in White Bay, and implied a genetic link to the Devils Room Granite (located some 15 km south of Figure 1), which at that time gave an imprecise U-Pb age of ~400 Ma (Erdmer, 1986). Subsequent geochronological work conducted by Heaman et al. (2002) now shows that the Devils Room Granite is of Silurian age (~425 Ma).

Re–Os GEOCHRONOLOGY

METHODS AND TECHNIQUES

The Re–Os geochronometer is based on the decay of ¹⁸⁷Re to ¹⁸⁷Os, and its application is identical to the more familiar Rb–Sr method used in silicate rocks. Unless the samples contain purely radiogenic Os, an isochron must be

constructed from measurements of ¹⁸⁷Re and ¹⁸⁷Os ratios, with reference to ¹⁸⁸Os. The samples used for Re–Os analysis were collected from mineralized granite in drillhole RB-08, completed by BP Resources in 1986 at the Road Zone (Figure 1), and now stored at the Department of Natural Resources core library in Pasadena. An initial test of Re contents from pyrite separates suggested that samples contained 3 to 5 ppb Re, which should provide enough radiogenic Os for dating samples of Paleozoic age.

Rhenium-osmium analysis was conducted at the University of Alberta. Four pyrite separates were prepared, but it proved very difficult to separate fine-grained arsenopyrite from the generally coarser pyrite. About 0.2 grams of each separate was weighed and transferred to a thick-walled borosilicate glass Carius tube for digestion. The samples were spiked using a mixed ¹⁸⁵Re and ¹⁹⁰Os spike, and Re and Os separated by chemical procedures (Shirey and Walker, 1995; Cohen and Waters, 1996; Birck et al., 1997). The procedural blanks were measured to be less than 5 picograms Re and 2 picograms Os. The Re and Os isotopic compositions were measured by negative thermal ionization mass spectrometry. The decay constant for ¹⁸⁷Re in isochron calculations was 1.666 x 10⁻¹¹ a⁻¹ (Smoliar et al., 1996). Results are reported at the 2σ level. More details on procedures are provided by Morelli et al. (2005).

RESULTS AND INTERPRETATION

Rhenium-osmium abundance and isotopic data are shown in Table 1. The pyrite separates have moderate Re/Os ratios, but have very small amounts of radiogenic Os. Rhenium abundances are significantly lower than those initially estimated, and three of four separates contained <1 ppb Re. The reasons for the discrepancy are unknown, but calibration problems are suspected. The amounts of Os are thus very low, ranging from 40 to 127 ppt (parts per trillion), representing absolute amounts of only 4 to 26 picograms Os. The precision of Os isotopic analysis is adversely affected by these low levels, particularly for fraction B, for which Os contents are only twice the blank value. The results are shown as an isochron plot in Figure 2. Regression of all four fractions yields an age of 315 ± 47 Ma and an initial 187 Os/ 188 Os ratio of 0.09 \pm 0.3. This is Late Carboniferous, but the error permits ages between the end of the Devonian and the Early Permian. Exclusion of Fraction B is justifiable on the basis of its very large uncertainty, and the resultant isochron has a slightly older age of 327 ± 58 Ma. The larger error reflects the fact that this isochron is effectively a 2point line as Fractions A and D yielded almost identical results, and it is thus difficult to estimate the true error through regression. Attempts were made to analyze additional fractions, and also to analyze a rare example of coarse-grained (>3 mm) arsenopyrite, but these proved unsuccessful due to negligable Re and Os abundances.

 Table 1. Rhenium–osmium concentration and isotopic data for pyrite separates from mineralized Precambrian granite in hole RB-08 at the Rattling Brook gold deposit; see text and Creaser (2005) for details of analytical procedures and data treatment

Sample	Re	Os	¹⁸⁷ Re -	¹⁸⁷ Os–	Error	
Fraction	(ppb)	(ppt)	¹⁸⁸ Os	¹⁸⁸ Os	(2 σ)	
RB-8A	0.91	111.50	40.45	$0.305 \pm$	0.02	
RB-8B	0.29	19.49	75.84	0.437 \pm	0.14	
RB-8C	0.66	41.69	80.31	$0.524 \pm$	0.01	
RB-8D	1.04	127.00	40.42	0.307 \pm	0.01	

Notes

1. ppb = parts per billion; ppt = parts per trillion

2. Uncertainty in 187 Re $-{}^{188}$ Os estimated at $\pm 1\%$

3. For details of procedures, see contract report by Creaser (2005)



Figure 2. *Rhenium–osmium isochron plot for pyrites from mineralized granite in hole RB-08.*

The Re–Os age obtained from the pilot study is not particularly useful as it is imprecise; however, it does confirm that mineralization hosted by the Precambrian granitic host rock is Paleozoic, as suggested by the occurrence of gold in Cambrian sedimentary rocks. The possibility that the occurrences in younger rocks represent remobilization of Precambrian gold is therefore discounted by the data.

⁴⁰Ar-³⁹Ar GEOCHRONOLOGY

The ⁴⁰Ar–³⁹Ar geochronological technique has its roots in K–Ar geochronology, based on the decay of ⁴⁰K to ⁴⁰Ar, but is now more commonly used than K–Ar methods. It has the advantage of avoiding problems associated with excess ⁴⁰Ar in some minerals, which can yield unreasonably old ages. More importantly, it allows for progressive release of Ar by stepwise heating of samples, so that post-crystallization ⁴⁰Ar loss related to later low-grade metamorphism and/or alteration can be detected, and discounted in calculation of primary igneous or metamorphic ages. The closure temperatures for common minerals such as feldspar, biotite and hornblende cover a wide range (200 to 550°C), so the technique often gives inferences into the thermal history of samples. The theory underlying ⁴⁰Ar–³⁹Ar geochronology is complex, as it involves the irradiation of samples to produce ³⁹Ar from ³⁹K, prior to direct measurement of argon isotopic ratios on a single sample. Background information on the method, techniques and assumptions is given in standard texts (e.g., Faure, 1986; MacDougall and Harrison, 1999). Isotopic analyses were conducted at the laboratories of the Geological Survey of Canada in Ottawa.

SAMPLE DETAILS

Two samples were analyzed, representing pre- and postmineralization mafic dykes, respectively. Both generations of mafic dykes can be observed in outcrops, but the sampling was conducted from drill core to avoid weathering and because Au assays were available from samples and wall rocks (data from BP-Selco; McKenzie, 1986, and Kermode Resources; N. Briggs, personal communication, 2004). The two drillholes chosen for sampling are at the Road Zone, and are located about 250 m northeast of drillhole RB-08, which was sampled for Re–Os analysis (Figure 1).

Rock Type and Relationships

Sample KC-04-034 was collected from drillhole JA-04-12, completed by Kermode Resources Ltd. The drillhole consists of Precambrian granite, and fine-grained mafic intervals, both of which are altered and mineralized (Kerr, 2005). The core was sampled at a depth of 34 m, where it is a variably altered and mineralized, metamorphosed mafic rock, extending from 26.4 to 42.2 m. Note that chilled margins are not visible, so the intrusive relationship is not unequivocal, but the same rock type has a clear dyke-like form in surface outcrops by the drillhole collar, and there is no indication that it is intruded by the surrounding granite. In the field, these metamorphosed mafic rocks were interpreted as Precambrian dykes of the Long Range dyke swarm (see Kerr, 2005, and above). The term "metadyke" is used in subsequent discussions for clarity. In drill core, the metadyke varies widely in appearance due to the presence of superimposed sericitic alteration and siliceous alteration, but the least altered sections are a fine-grained, featureless black, biotite-rich rock that has a visible fabric, along which sericitic alteration has locally penetrated. However, pervasive sericitic alteration clearly overprints the fabric in the metadyke, as do later zones of silicification associated with sulphides and gold. The alteration and mineralization of the metadyke are described by Kerr (2005). The metadyke is invaded by white quartz veins throughout the intersection, but these were avoided during sampling. The sampled interval contains only 0.09 ppm Au (90 ppb), as it is essentially unaltered, but adjacent sections of the metadyke contain up to 6.5 ppm Au (N. Briggs, personal communication, 2004; see also Figure 4 of Kerr, 2005). In general, the mafic metadykes and melanocratic granites in this hole contain the highest gold assays, suggesting that these rocks more effectively precipitated gold than the surrounding leucocratic granites.

Sample KC-04-036 was collected from nearby drillhole RB-09 (Figure 1), completed by BP-Selco in 1985. The drillhole consists of variably altered and mineralized Precambrian granite, similar to the dominant rock type in JA-04-12, and contains three intervals of fresh diabase, which exhibit clear chilled contacts against the granite; a drillhole log is provided by McKenzie (1986). The core was sampled at approximately 62.3 m depth, where it consists of fresh, plagioclase-porphyritic diabase. The mineralized granite above the diabase contains up to 2.3 ppm Au. Below the lower contact of the diabase dyke, which is less distinct, there is a greyish, siliceous rock type (sample KC-04-037) described by McKenzie (1986) as "green pyritic alteration" of the diabase; this contains disseminated pyrite, and grades downward into altered and mineralized granite within 10 to 20 cm. The mineralized granite is visibly cut by fresh, locally chilled diabase at greater depths. The "green pyritic alteration", sampled together with some of the underlying granite, contains 1.4 ppm Au (BP-Selco assays). Detailed examination of drill core with a hand lens and in thin section demonstrates that the grey altered rock was originally a granite and not a mineralized section of the dyke. The drillhole thus represents variably mineralized granites that are cut by post-mineralization diabase dykes; the granites are perhaps also included as screens and xenoliths within the diabase. There is no sign of any hornfels development adjacent to the contacts of the late dykes, which commonly have clear chilled margins.

Petrography

The pre-mineralization dyke (KC-04-034) is a completely recrystallized rock composed mostly of biotite, Kfeldspar and quartz, with lesser amounts of epidote and sericite. It contains no indication of relict igneous textures and, based on petrography, no clear evidence of having originated as a dyke. The biotite, quartz and K-feldspar are all fine grained and intergrown, and a strong fabric is defined by the biotite and quartz. Larger K-feldspar grains are visible, up to 1 mm in diameter, but the relationship between these and the fabric is not entirely clear; however, they contain quartz inclusions, suggesting that they may have overgrown the fabric as porphyroblasts. Minor epidote is associated with the biotite, and also defines the strong fabric. Sericite is present as an alteration product of biotite, and also forms discrete veinlets; some of the veinlets are parallel to the fabric, whereas others crosscut the fabric. No amphibole was observed in the sample. The field relationships imply that the sample represents a mafic metadyke, but its biotite-rich composition suggests either a lamprophyric parent, or metasomatic effects that introduced potassium prior to or during regional metamorphism.

The post-mineralization dyke (KC-04-036) is completely different in appearance. It is a fine-grained, plagioclase-porphyritic diabase that retains very good primary igneous textures, despite some incipient hydration and alteration. It is dominated by turbid to saussuritic plagioclase, which forms numerous tiny laths and scattered larger phenocrysts, up to 3 mm in diameter. These are surrounded by variably altered oikocrystic mafic minerals, giving a typical ophitic or "diabasic" texture. The most abundant primary mafic mineral is orthopyroxene, although a few heavily oxidized clots may be derived from minor primary olivine. Magnetite is also interstitial in form and is part of the primary igneous assemblage. Amphibole is widespread, and may include more than one generation. The largest and bestformed amphibole crystals are associated with relict pyroxene, and are probably late-magmatic in timing, formed soon after (and from) the pyroxenes. Finer-grained amphibole elsewhere in the sample may be secondary or deuteric in origin, but the main mafic alteration mineral is chlorite. Both epidote and sericite occur locally in veinlets, but there is no sign of any widespread sericitic alteration.

The "altered diabase dyke" (KC-04-037) described by McKenzie (1986) is dominated by coarse-grained quartz, alkali feldspar, calcite and disseminated sulphides, with areas of fine-grained quartz that likely represent silicified zones. There is no sign of any relict ophitic texture or of ferromagnesian alteration products. The sample is an altered, mineralized, leucocratic granite akin to those forming the wall rocks to the diabase dykes.

ANALYTICAL METHODS AND PROCEDURES

The samples were crushed to granules in the size range 0.25 to 0.50 mm. From sample KC-04-034 aggregates rich in biotite, but also containing quartz and other minerals (likely epidote and sericite), were selected by hand. For sample KC-04-036, it was possible to hand-pick grains consisting entirely of a poor quality amphibole with opaque inclusions. Two aliquots of KC-04-034 were analyzed in order to better constrain precision and reproduceability.

Several grains and/or aggregates were loaded into aluminum foil packets along with a single grain of Fish Canyon Tuff Sanidine (FCT-SAN) to act as flux monitor (apparent age = 28.03 Ma; Renne et al., 1998), and irradiated for 12 hours at the research reactor of McMaster University. Laser ⁴⁰Ar-³⁹Ar step-heating analysis was carried out at the Geological Survey of Canada laboratories in Ottawa, Ontario. Heating of individual sample aliquots in steps of increasing temperature was achieved using a CO₂ laser. The released Ar gas was analyzed isotopically using the secondary electron multiplier system of a VG3600 gas source mass spectrometer; details of data collection protocols can be found in Villeneuve and MacIntyre (1997) and Villeneuve et al. (2000). Error analysis on individual steps follows numerical error analysis routines outlined in Scaillet (2000); error analysis on grouped data follows algebraic methods of Roddick (1988). Gas-release spectra show step-heating data from two aliquots of KC-04-034 (Figure 3) and one aliquot of sample KC-04-036 (Figure 4), normalized to the total volume of ³⁹Ar released for each aliquot. Such plots provide a visual image of the evidence for ⁴⁰Ar-loss in the low temperature steps, and the error and apparent age of each step. Correction for neutron flux gradients during irradiation was accomplished using the sanidine flux monitors, from which J-factors were estimated for each aliquot (Table 2). The error on individual J-factor values is conservatively estimated at $\pm 0.6\%$ (2 σ). Correction for J-factor uncertainty was applied after calculation of dates from isotopic correlation diagrams (Roddick, 1988). No evidence for excess ⁴⁰Ar was observed in any of the samples and, therefore, all regressions are assumed to pass through the ⁴⁰Ar/³⁶Ar value for atmospheric air (295.5) All errors are quoted at the 2σ level of uncertainty.

RESULTS

Corrected argon isotopic data are listed in Table 2 and presented as gas-release spectra and inverse-isochron diagrams in Figures 3, 4 and 5 (after Roddick *et al.*, 1980). The gas-release spectrum for sample KC-04-034 (Figure 3) contains step-heating data from two aliquots. Both aliquots show well-defined multi-step plateaus representing 72% and 74%, respectively, of the total released ³⁹Ar for each aliquot. The combined weighted average ⁴⁰Ar/³⁹Ar age for 7 Ar



Figure 3. Plateau diagram showing ⁴⁰Ar–³⁹Ar ages plotted against percentage of total ³⁹Ar gas released during stepheating of biotite separates from the pre-mineralization metadyke (sample KC-04-034).



Figure 4. Plateau diagram showing ⁴⁰Ar-³⁹Ar ages plotted against percentage of total ³⁹Ar gas released during stepheating of amphibole separates from the post-mineralization diabase dyke (sample KC-04-036).

Table 2. Argon isotopic data and related parameters for step-heating analysis of biotite and amphibole separates from a pre-
mineralization metadyke (hole JA-04-12) and a post-mineralization diabase dyke (hole RB-09) at the Rattling
Brook gold deposit; see text and van Breemen (2005) for details of analytical procedures and data treatment

Power	Volume ³⁹ A x10 ⁻¹¹ cc	r ³⁶ Ar/ ³⁹ Ar	³⁷ Ar/ ³⁹ Ar	³⁸ Ar/ ³⁹ Ar	⁴⁰ Ar/ ³⁹ Ar	% ⁴⁰ Ar ATM	* ⁴⁰ Ar/ ³⁹ Ar	f ₃₉ ^b (%)	Apparent Age Ma ^c			
	34 Diatitas 1	L_0 00207700d						()	8			
Alignot. A												
2.8	0.3381	0.0189+0.0102	0.018+0.282	0.088+0.012	49.17+1.25	11.3	43.60+3.25	0.7	220.2+15.5			
3	0.3933	0.0106 ± 0.0087	0.082 ± 0.243	0.085 ± 0.012	74.14 ± 1.14	4.2	71.00 ± 2.82	0.9	345.9 ± 12.5			
3.5	0.8929	0.0039+0.0038	0.261 ± 0.108	0.100+0.011	84.08+0.64	1.4	82.92+1.30	2	398.0+5.6			
3.9	2.2086	0.0019±0.0015	0.135±0.044	0.111±0.011	86.70±0.77	0.7	86.13±0.89	4.9	411.8±3.8			
4.2	2.7999	0.0017+0.0012	0.001 ± 0.034	0.106+0.011	86.52+0.32	0.6	86.01+0.48	6.2	411.3+2.1			
4.6	2.3027	0.0024 ± 0.0015	0.004 ± 0.041	0.096 ± 0.011	86.79±0.39	0.8	86.07±0.59	5.1	411.5±2.5			
5.5	2.3053	0.0024 ± 0.0014	0.028 ± 0.033	0.124 ± 0.011	87.07±0.31	0.8	86.37±0.52	5.1	412.8±2.2			
5	1.6226	0.0013+0.0019	0.009+0.054	0.101+0.011	86.13+0.38	0.4	85.74+0.69	3.6	410.1+3.0			
6	3.3796	0.0011±0.0009	0.004 ± 0.026	0.130±0.011	86.78±0.30	0.4	86.46±0.41	7.4	413.2±1.7			
6.5	3.4826	0.0018+0.0009	0.012 ± 0.025	0.130 ± 0.011	87.04+0.31	0.6	86.50+0.41	7.7	413.4+1.8			
7	3.6204	0.0008+0.0010	0.010+0.025	0.126+0.011	87.74+0.33	0.3	87.49+0.44	8	417.6+1.9			
13	3.0168	0.0014 ± 0.0012	0.076±0.030	0.124 ± 0.011	87.85±0.31	0.5	87.44±0.46	6.6	417.4±2.0			
				Aliquot: B								
2.8	0.3389	0.0308 ± 0.0087	0.151±0.280	0.113±0.012	69.83±1.15	13	60.730±2.80	0.8	299.8±12.7			
3	0.7314	0.0048+0.0039	0.068+0.130	0.098+0.011	75.44+0.61	1.9	74.02 ± 1.30	1.6	359.3+5.7			
3.5	1.4586	0.0029 ± 0.0020	0.042 ± 0.065	0.101 ± 0.011	83.70±0.38	1	82.84±0.71	3.2	397.7±3.0			
3.9	1.0473	0.0074 ± 0.0028	0.132 ± 0.091	0.097±0.011	86.78±0.50	2.5	84.61±0.95	2.3	405.3±4.1			
4.2	2.0093	0.0018 ± 0.0014	0.087 ± 0.047	0.098 ± 0.011	86.72±0.81	0.6	86.19±0.91	4.4	412.0±3.9			
4.6	1.6503	0.0018±0.0017	0.082 ± 0.058	0.094 ± 0.011	86.59±0.61	0.6	86.05±0.79	3.6	411.5±3.4			
5	1.628	0.0018 ± 0.0018	0.102 ± 0.058	0.115 ± 0.011	86.66±0.47	0.6	86.12±0.70	3.6	411.8±3.0			
5.5	2.9118	0.0014 ± 0.0010	0.043±0.033	0.122±0.011	86.64±0.28	0.5	86.24±0.41	6.4	412.2±1.8			
6	1.9395	0.0006 ± 0.0015	0.050 ± 0.051	0.121±0.011	86.65±0.39	0.2	86.46±0.59	4.3	413.2±2.5			
6.5	1.708	0.0014 ± 0.0017	0.084 ± 0.056	0.120 ± 0.011	86.74±0.55	0.5	86.34±0.74	3.8	412.7±3.2			
7	2.5118	0.0008 ± 0.0011	0.040 ± 0.038	0.112 ± 0.011	87.74±0.32	0.3	87.49±0.47	5.5	417.6±2.0			
13	1.1531	0.0026 ± 0.0025	0.461 ± 0.084	0.100 ± 0.011	86.51±0.77	0.9	85.73±1.06	2.5	410.1±4.5			
KC-04-()36 Amphibo	ole; J=0.00297340ª										
				Aliquot: A								
3	0.0405	0.7047±0.0495	67.225±3.298	0.417 ± 0.044	286.01±9.95	72.8	77.78±16.56	2.3	375.3±72.1			
3.9	0.12	0.2397±0.0152	53.223±1.584	0.185 ± 0.018	156.95 ± 4.66	45.1	86.11±6.03	6.8	411.3±25.8			
4.6	0.7023	0.0154 ± 0.0025	7.034±0.194	0.033 ± 0.011	91.04 ± 0.87	5	86.49±1.12	39.5	412.9±4.8			
5	0.4703	0.0175 ± 0.0037	11.750±0.310	0.123 ± 0.012	91.67±1.22	5.6	86.50±1.62	26.5	412.9±6.9			
6	0.0908	0.0313±0.0158	21.804±1.278	0.226±0.025	95.31±4.43	9.7	86.05±6.35	5.1	411.0±27.1			
6.5	0.0281	0.0557 ± 0.0946	37.844±3.977	0.451 ± 0.050	109.34±10.13	15.1	92.88±29.64	1.6	439.9±124.7			
7.5	0.0537	0.0348 ± 0.0500	37.821±2.372	0.474 ± 0.037	102.79±5.62	10	92.50±15.77	3	438.4±66.4			
13	0.2714	0.0137 ± 0.0107	15.424±0.509	0.175 ± 0.013	90.76±1.41	4.5	86.71±3.44	15.3	413.8±14.7			
a. As m	easured by	laser in % of full n	ominal nower ((10W)								

b: Fraction ³⁹Ar as percent of total run

c: Errors are analytical only and do not reflect error in irradiation parameter J

d: Nominal J, referenced to FCT-San =28.03 Ma (Renne et al., 1994)

All uncertainties quoted at 2s level

release fractions from aliquot A and 7 from aliquot B is 412.3 ± 2.3 Ma. The highest power steps at 7.0 and 13 for fraction A and at 7 for fraction B (Table 2) yield slightly older ages (~417 Ma). Unfortunately, the very fine grain size of the sample precluded complete separation of mineral phases. For amphibole from sample KC-04-036, an age plateau for 7 power steps on a single aliquot represents 98%

of the total argon released (Figure 4). The weighted average age for these Ar release fractions is 413 ± 4.3 Ma. An identical age was obtained using a regression analysis for an isotopic correlation diagram (Figure 5). Note that two stepheating increments, at power steps 6.5 and 7.5, gave imprecise results, and these plot off the reverse-isochron line in Figure 5. Excluding these results, the Ca/K ratio of the



Figure 5. Reverse isochron diagram(s) showing ${}^{39}Ar/{}^{40}Ar$ and ${}^{36}Ar/{}^{40}Ar$ data from sample KC-04-036.

increments is consistent, indicating homogeneity. The results listed in Table 2 and illustrated in Figures 3 to 5 demonstrate that the apparent ages of the foliated, altered, gold-bearing, pre-mineralization metadyke (KC-04-034) and the fresh, unaltered post-mineralization diabase dyke (KC-04-036) are identical within respective errors, and correspond to the earliest Devonian (417 to 409 Ma).

INTERPRETATION OF DATA

Although their apparent ages are identical, the relationships documented in the field and in drill core clearly indicate that the metadyke was emplaced, metamorphosed, and then altered and mineralized before the fresh post-mineralization diabase was emplaced. Furthermore, because the ages come from biotite and hornblende, respectively, and the pre-mineralization metadyke is metamorphosed, these identical ⁴⁰Ar-³⁹Ar ages do not necessarily demonstrate simultaneity of crystallization.

The closure temperature of Ar in hornblende is around 550°C (McDougall and Harrison, 1999). Much of the hornblende in the post-mineralization dyke appears to be latemagmatic and there is no indication that this rock was subjected to such high temperatures during a later event. Thus, the cooling age for KC-04-036 likely also records its time of crystallization, and it provides a minimum age for alteration and gold mineralization in the granitic rocks that the dyke intrudes. Given that mafic rocks in the Rattling Brook deposit generally contain the most obvious alteration and typically have the best gold grades, it is hard to argue that lack of mineralization in a dyke that has mineralized wall rocks is coincidental.

The closure temperature of Ar in biotite is typically around 300°C (Hodges, 1991), although it can be significantly higher (McDougall and Harrison, 1999). This low closure temperature complicates interpretation of the data from the pre-mineralization metadyke, and it is unlikely that the age records its initial emplacement and crystallization. It must instead represent a cooling age that is younger than both crystallization and subsequent metamorphism.

There are three possible interpretations of the combined age data, which are illustrated schematically in Figure 6, and discussed below.

- 1. The age from the metadyke records cooling through \sim 300°C following regional metamorphism. Metamorphism was followed (or accompanied) by alteration and mineralization, and then by emplacement of the post-mineralization dyke. The timing of peak regional metamorphism is thus prior to 417.6 ± 2 Ma, which is the oldest step-heating age from KC-04-034 (Figure 6a), assuming that there is no excess Ar. The relative timing of biotite cooling and gold mineralization is an interesting problem, discussed fully below.
- 2. The age from the metadyke records complete resetting of pre-existing metamorphic biotite during alteration related to gold mineralization, which was then followed by emplacement of the post-mineralization dyke. The timing of peak regional metamorphism is again prior to 417.6 ± 2 Ma, but could be much earlier if the dyke cooled through 300°C and was then reheated during the mineralization event (Figure 6b).
- 3. The age records resetting of pre-existing metamorphic biotite *and* alteration minerals related to gold mineralization during a thermal event associated with the emplacement of the post-mineralization dykes. In this case, the timing of peak regional metamorphism and the timing of the mineralization event are unconstrained, and both could be much earlier than 417.6 ± 2 Ma (Figure 6c).

Option (3) is very unlikely, because post-mineralization dykes are not common, and there is no evidence of hornfelsing effects or mineral growth in country rocks adjacent to their contacts. There is thus no indication that they had widespread thermal effects, and purely local effects can be excluded because there is no indication of any younger diabase in drillhole JA-04-12. Options (1) and (2) above cannot be discriminated by the geochronological data alone, and both remain viable interpretations.



Figure 6. (opposite) Schematic illustrations of possible cooling paths for the pre-mineralization metadyke and the post-mineralization diabase, with respect to the timing and temperature regime of gold mineralization. (a) Option 1, where the metadyke cools following regional metamorphism. If the temperature regime during mineralization is below the blocking temperature for biotite, the metadyke age provides an upper limit for mineralization. If the temperature regime during mineralization exceeds the blocking temperature for biotite, the metadyke age could actually be younger than the mineralization. (b) Option 2, where the metadyke cools following metamorphism and is then reheated during gold mineralization, and its biotite age is reset in a thermal regime that exceeds the blocking temperature for biotite. (c) Option 3, where the timing of metamorphism and mineralization are unconstrained because the biotite age of the metadyke was reset by heating related to the emplacement of the post-mineralization dyke. See text for further discussion.

The simplest interpretation of the data is that gold mineralization and related alteration in the Rattling Brook deposit occurred during the earliest Devonian, between 417.6 ± 2 Ma (the oldest incremental ages from KC-04-034) and 412.9 \pm 4.25 Ma (the age of the post-mineralization dyke KC-04-036). However, if option (1) applies, the true upper limit for the timing of mineralization may be older than 417.6 \pm 2 Ma, if the ambient temperatures during the mineralization event were significantly above the closure temperature of biotite. Under these circumstances, the biotite age from the pre-mineralization metadyke could be younger than the timing of mineralization, as shown in Figure 6a. If option (2) applies, the age of 412.2 ± 2.3 Ma directly records the timing of mineralization, and ambient temperatures during the mineralization event must have exceeded the closure temperature of biotite, as shown in Figure 6b.

The temperature regime during gold mineralization at the Rattling Brook deposit is not constrained by fluid-inclusion studies, but Saunders and Tuach (1991) suggest a temperature range of 300 to 350°C, based on alteration signatures, and indicated 400°C as an upper limit, based on the presence of secondary microcline in altered granites. They considered the mineralization to be of broadly "mesothermal" affinity, for which temperatures are generally considered to be in the range of 250 to 400°C (e.g., Groves *et al.*, 1998). More recently, Kerr (2005) suggested that mineralization at Rattling Brook has affinities to sedimentary-rockhosted micron gold deposits ("Carlin-type") or non-carbonate-hosted stockwork-disseminated gold deposits, both of which are believed to form at temperatures below 300°C (Poulsen *et al.*, 2000; Cline *et al.*, 2000). It thus appears unlikely that the ambient temperature during the mineralization event was significantly higher than the closure temperature for argon isotopic systems in biotite. On this basis, it is suggested that the geochronological data do essentially bracket the mineralization event, regardless of which of the two options applies. However, the suggested upper limit may need revision if future work suggests higher ambient temperatures during mineralization.

DISCUSSION AND CONCLUSIONS

There are few constraints on the timing of gold mineralization in Newfoundland, and some of the existing data require subjective interpretation. The ⁴⁰Ar-³⁹Ar data presented here strongly suggest that hydrothermal gold mineralization at the Rattling Brook deposit is earliest Devonian. An earliest Devonian gold mineralization event could also be responsible for vein-hosted Au-Ag (± Cu, Pb, Zn) mineralization in the Silurian Sops Arm Group, located south of the area shown in Figure 1 (Saunders, 1991; Kerr, this volume). These auriferous veins were emplaced following deformation of the host sedimentary and volcanic rocks, which (based on faunal evidence) are as young as the Pridoli stage (uppermost Silurian). However, the style, mineralogy, and geochemical associations of mineralization in the Sops Arm Group are different from Rattling Brook, where there is no association with base metals, and a generally higher Au/Ag ratio (Kerr, 2005, this volume).

The indicated age of mineralization at Rattling Brook is slightly younger than the U-Pb age of hydrothermal zircon obtained by Ramezani (1992) from the Stog'er Tight Deposit, which essentially corresponds to the Silurian–Devonian boundary (420 \pm 5 Ma). The younger dates reported from the Nugget Pond Deposit (374 ± 8 Ma; R. Parrish, in Sangster and Pollard, 2001) and the Titan Prospect ($<381 \pm 5$ Ma; McNicol *et al.*, *this volume*) indicate that there was also a Middle to Late Devonian mineralizing event in Newfoundland. It is interesting to note that these two events resemble those defined by Re-Os dating of mesothermal lode gold deposits in the Meguma Zone of Nova Scotia. In this district, Morelli et al. (2005) report that gold mineralization at the Ovens prospects yielded an age of 408 ± 4 Ma, whereas the Dufferin and Touquoy deposits gave coincident ages of 381 ± 3 Ma. However, given the limited database in both Newfoundland and Nova Scotia, and the different methods involved, it is premature to ascribe too much significance to this observation. The Re-Os isotopic data from Rattling Brook were not very useful, as low Os abundances led to large relative errors and blank corrections. The imprecise Re-Os isochron age of ca. 327 ± 58 Ma is contradicted by the 40 Ar $-{}^{39}$ Ar data from the post-mineralization dyke, and must be erroneously young.

The upper limit indicated by the Re–Os data is ca. 385 Ma, some 30 Ma younger than the lower limit given by the ${}^{40}\text{Ar}-{}^{39}\text{Ar}$ data.

In a broader sense, there is a need for better constraints on the timing of gold mineralization in the Newfoundland Appalachians, and such information may also prove useful in understanding the timing of orogenic and "accretionary" events. The example discussed here is unusual as pre- and post-mineralization dykes permit bracketing of the mineralizing event. Unfortunately, field relationships of this type are the exception rather than the rule. Although our attempts to date the Rattling Brook gold deposit using the Re–Os technique were not successful, the technique holds great promise, as evidenced by the studies of Stein *et al.* (2000), Arme *et al.* (2001) and Morelli *et al.* (2005), amongst others. A further test of the method at other sites needs to be preceded by a reconnaissance study of Re contents in pyrite and (preferably coarse-grained) arsenopyrite.

ACKNOWLEDGMENTS

The cooperation of Mineral Lands Division geologists Alvin Harris and Stuart Cochrane in sampling archived drill core for geochronological studies is very much appreciated by the authors. This article was reviewed by Don Kontak of the Nova Scotia Department of Natural Resources, who suggested improvements and additions.

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