Disturbance of the Re-Os chronometer of molybdenites from the late-Caledonian Galway Granite, Ireland, by hydrothermal fluid circulation

KATSUHIKO SUZUKI,^{1*} MARTIN FEELY² and CIAN O'REILLY³

¹Department of General Systems Sciences, Graduate School of Arts and Sciences, University of Tokyo, Komaba, Meguro, Tokyo 153-8902, Japan

²Department of Geology, National University of Ireland, Galway, Ireland

³Navan Resources, Kennedy Rd., Navan, Co. Meath, Ireland

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Re-Os ages were determined for molybdenites from the late-Caledonian Galway Granite in the west of Ireland. Molybdenites from three localities within the batholith, Mace Head, Murvey, and Travore, yield respective Re-Os ages of 425.2 ± 3.1 Ma, 383.2 ± 8.1 and 426.1 ± 5.5 Ma, and 338.9 ± 5.6 and 464 ± 28 Ma. The molybdenites from the three localities are believed to be contemporaneous. However, the Re-Os ages obtained for each locality are not only in disagreement with the ages of the samples from the other localities but also are inconsistent within a single locality. Replicate analyses of a molybdenite standard (HLP-5) characterized by AIRIE using our method (Suzuki *et al.*, 1992) yields reproducible Re-Os ages, which are in agreement with the results of Markey *et al.* (1998). Therefore, the age discrepancies in Galway molybdenites in this study should not be attributed to analytical errors. Suzuki *et al.* (2000) revealed experimentally that the Re-Os system in molybdenite can behave as an open-system when immersed in warm aqueous NaCl or NaHCO₃ bearing fluid. Detailed fluid inclusion studies of molybdenite vein quartz and granite quartz (Gallagher *et al.*, 1992; O'Reilly *et al.*, 1997) have shown that the Galway Granite was the locus for repeated fluid events, i.e., 1. Expulsion of aqueous carbonic magmatic fluids during the final stages of crystallization, 2. Meteoric fluid convection probably driven by waning magmatic heat and 3. Incursion of basinal brines associated with fluorite mineralization at least 100 Ma after granite consolidation. We suggest that younger hydrothermal fluids perhaps associated with fluorite mineralization may have perturbed the Re-Os systematics of molybdenites from the Galway Granite.

INTRODUCTION

The Re-Os isotopic system is a useful tool for the direct determination of formation ages of sulfide ore deposits, because the chalcophile and siderophile properties of Re and Os lead to relatively high abundances of these elements in sulfide minerals, unlike such lithophile elements as K-Ar, Rb-Sr, Sm-Nd and U-Pb systems. Among the varieties of sulfide minerals, molybdenite (MoS₂) has been a target for the application of the Re-Os system because of its high concentration of Re

(Hintenberger *et al.*, 1954; Fleischer, 1969).

To obtain an accurate age, it is necessary that the mineral has been kept closed for Re and Os since its formation. Luck and Allègre (1982) first presumed open-system behavior of Re in molybdenite. Suzuki *et al.* (1992, 1993, 1996), however, demonstrated that the age discrepancy may have resulted from analytical problems rather than geological open-system behavior. McCandless *et al.* (1993) examined molybdenite alteration in detail with regard to Re behavior. They reported that high transparency of

*Present address: Institute for Geothermal Sciences, Kyoto University, Noguchibaru, Beppu, Oita 874-0903, Japan

*Corresponding author (e-mail: suzuki@bep.vgs.kyoto-u.ac.jp)

molybdenite under near infrared (NIR) light and/or existence of certain secondary minerals indicated alteration and also Re loss in post-depositional alteration. Suzuki *et al.* (2000) experimentally demonstrated that disturbance of the Re-Os system depends on the alteration condition. On the contrary, Frei *et al.* (1998) claimed that the Re-Os system in molybdenite is resistant to alteration, and Stein *et al.* (1998a, b) demonstrated closed system behavior in molybdenite with a proper “whole molybdenite” mineral separation that is relatively fine-grained.

In this study, the Re-Os ages of molybdenite from the Galway granite, Ireland, were determined. The variation in the obtained Re-Os ages is discussed in the context of repeated hydrothermal fluid events that occurred both syn- and post-consolidation of the granite.

GEOLOGICAL SETTING

The Galway Granite batholith is exposed over an area of approximately 600 km² along the north shore of Galway Bay; a southerly offshore extension of several km is indicated on gravity and aeromagnetic maps (Murphy, 1952; Max *et al.*, 1983; Madden, 1987). It is a late-Caledonian “I-type” intrusion emplaced *ca.* 400 Ma ago (Leggo *et al.*, 1966) into the 474.5–462.5 Ma metagabbro-gneiss suite (MGGS) to the north (Friedrich *et al.*,

1999), and into Lower Ordovician greenschist facies rocks (the South Connemara Group) to the south (McKie and Burke, 1955; Williams *et al.*, 1988). The batholith is composed of lithologies that range from diorites through granodiorites and adamellites to alkali-feldspar granites (Leake, 1974, 1978; Max *et al.*, 1978; El Desouky *et al.*, 1996; Crowley and Feely, 1997).

Disseminated and quartz vein-hosted molybdenite mineralization occurs throughout the Galway Granite (Derham, 1986; Max and Talbot, 1986; Derham and Feely, 1988; Feely and Hoegelsberger, 1991; Gallagher *et al.*, 1992; Derham, 1993). The most notable occurrences are at the western end of the batholith, i.e., at Mace Head and Murvey (Fig. 1). At Mace Head, molybdenite bearing quartz veins (5–30 cm thick) trend NE-SW their orientation controlled by early jointing in the host granite (Derham, 1986; Max and Talbot, 1986). Additional vein minerals include chalcopyrite, pyrite, magnetite and muscovite. Mineralized and altered granite extends over an area of 2 km² and in addition to the vein mineralization, quartz-magnetite pods and an intrusive K-feldspar breccia also occur (Derham and Feely, 1988). At Murvey, a highly evolved leucogranite contains molybdenite intergrown with muscovite. Quartz veins in the area also contain Mo-mineralization. Gallagher *et al.* (1992) interpreted the Mo-mineralization in both areas,

Fig. 1. Geological settings of the Galway Granite. Molybdenite sample localities are 1. Murvey, 2. Mace Head and 3. Travore Bay.

using geochemical, fluid inclusion and stable isotope (O, H, S and C) data, as having been produced by highly fractionated granite magma. Three localities were sampled in the Galway Granite, i.e., quartz vein-hosted molybdenite from Mace Head and Travore Bay, and granite-hosted molybdenite from Murvey (Fig. 1).

ANALYTICAL METHOD

Molybdenite samples were separated by hand-picking from the quartz veins and granite. Os and Re abundances were determined by the isotope dilution method (Suzuki *et al.*, 1992). Briefly, after Re and Os spikes were added, molybdenite was decomposed through the microwave digestion method. The decomposed solution was added with $K_2Cr_2O_7$ to decompose molybdic acid and to completely oxidize Os to OsO_4 . Osmium was separated by distillation and Re was separated by anion exchange resin. An inductively coupled plasma mass spectrometer (ICP-MS), VG PQ2[®] at the Institute of Physical and Chemical Research and VG PQ3[®] at the Institute for Geothermal Sciences were used for measurements of Os and Re isotope abundances. Mass discrimination in Os isotope measurement is corrected by a method modified from that of Hirata *et al.* (1988).

Recently, Markey *et al.* (1998) and Stein *et al.* (1997, 1998a) obtained a much higher precision (10 $\times$) by employing alkaline fusion and negative thermal ionization mass spectrometry (NTIMS). Our study was undertaken through ICP-MS and, therefore, does not achieve precision levels currently possible with NTIMS. However, we believe the quality of the isotopic data presented is sufficient to justify the first-order conclusions presented in this paper.

RESULTS AND DISCUSSION

Because of the negligible quantity of common Os contained in the molybdenite, model age can be obtained using the following equation:

$$T = \{\ln[(^{187}Os/^{187}Re) + 1]\}/\lambda$$

where λ is the decay constant of ^{187}Re and equals to $(1.666 \pm 0.017) \times 10^{-11} \text{ yr}^{-1}$ (Smoliar *et al.*, 1996).

Re and Os abundances and the resultant ages for molybdenites are presented in Table 1 and Fig. 2. Molybdenites from the three localities in the Galway Granite yield the Re-Os ages of 425.2 ± 3.1 Ma for Mace Head, 383.2 ± 8.1 and 426.1 ± 5.5 Ma for Murvey, and 338.9 ± 5.6 and 464 ± 28 Ma for Travore Bay. Previously, Hirt *et al.* (1963) analyzed the molybdenite sample from Murvey Granite and obtained the Re-Os age of 365 ± 13 Ma (recalculated using $\lambda(^{187}Re) = 1.666 \times 10^{-11} \text{ yr}^{-1}$, Smoliar *et al.*, 1996). Since molybdenites from the three localities were genetically related and were formed contemporaneously from the granitic magma (Gallagher *et al.*, 1992), it is reasonable to expect that they would give harmonious Re-Os ages. However, the Re-Os ages obtained for each locality in this study and by Hirt *et al.* (1963) are not only inconsistent with the ages of the samples from the other localities but also are in disagreement even within each locality (Fig. 2). There are two reported ages of the host Galway Granite; the Rb-Sr age of 398 ± 10 Ma (Leggo *et al.*, 1966, recalculated using $\lambda(^{87}Rb) = 1.42 \times$

Fig. 2. Re-Os ages for molybdenites from Mace, Murvey, and Travore in the Galway Granite. The Re-Os age obtained by Hirt *et al.* (1963) are also shown.

Table 1. Results of Re and Os analysis of molybdenites from Galway, Ireland

Sample	Re (ppb)	Os (ppb)	Re-Os age (Ma)
This study			
Galway Mace	76140 ± 360	340.2 ± 1.9	425.2 ± 3.1
Galway Murvey	9492 ± 42	38.21 ± 0.79	383.2 ± 8.1
	10954 ± 84	49.05 ± 0.51	426.1 ± 5.5
Galway Travore	408.8 ± 5.1	1.455 ± 0.016	338.9 ± 5.6
	467 ± 26	2.281 ± 0.061	464 ± 28
Hirt <i>et al.</i> (1963)			
Galway Murvey	15200 ± 400	58.2 ± 1.4	365 ± 13

Errors are $2\sigma_m$

Re-Os age calculations were based on the ^{187}Re decay constant of $1.666 \times 10^{-11} \text{ yr}^{-1}$ (Smoliar *et al.*, 1996).

Errors in all Re-Os ages do not include the 1.2% uncertainty of the decay constant. The age obtained by Hirt *et al.* (1963) was also recalculated using Smoliar *et al.* (1996) ^{187}Re decay constant.

10^{-11} yr^{-1} by Steiger and Jäger, 1977) and the U-Pb age of $412 \pm 15 \text{ Ma}$ (on zircons, Pidgeon, 1969, recalculated using the decay constant of U by Jaffey *et al.*, 1971). However, since these data do not have enough quality available today and there is unusual statistical handling of data in Rb-Sr age, we do not use these ages in our discussion.

The age discrepancies found in the Re-Os ages of molybdenites in this study are not the result of the analytical procedures followed in the present study. We made replicate analyses of a molybdenite “standard” (HLP-5) provided by the AIRIE group, Colorado State University. As shown in Table 2, our obtained Re-Os ages for HLP-5 (average $222.0 \pm 2.1 \text{ Ma}$, $n = 4$) reproduce within analytical errors for the ICP-MS method, and, in addition, agree with the results ($221.3 \pm 0.24 \text{ Ma}$, $n = 19$) of Markey *et al.* (1998). These results illustrate the reliability of our technique for the Re-Os analysis of a powdered molybdenite standard, though precision of our ICP-MS age is 10 times greater than that of Markey *et al.* (1998) with NTIMS.

The range of Re-Os ages for molybdenites from the three localities in the Galway Granite may have been caused by post-ore deposition alteration accompanied by Re-Os fractionation. Frei *et al.* (1998) claimed that the Re-Os system in molybdenite was resistant to post-mineralization alteration and Stein *et al.* (1998a, b) demonstrate

that the Re-Os chronometer in molybdenite is robust in a variety of different environments. In contrast, McCandless *et al.* (1993) reported that molybdenite might be susceptible to Re loss through hydrothermal alteration processes at temperatures as low as 150°C , which affected Re-Os chronometry significantly. They determined the spatial Re distribution in altered rim (secondary minerals) and unaltered core of molybdenite and demonstrated evidence of Re remobilization at the boundary of the molybdenite mineral. In addition, Suzuki *et al.* (2000) demonstrated experimentally that the Re-Os system of molybdenite has an open-system behavior in certain alteration conditions. They found that the molybdenite, which was exposed to NaCl and NaHCO_3 solutions at a temperature of 180°C for 20 days, gave greatly deviated Re-Os ages relative to that of the unaltered sample, especially in the outer part of the altered molybdenite. When the molybdenite was immersed in aqueous AlCl_3 and CaCl_2 solutions, it yielded an age consistent with the unaltered original molybdenite. Suzuki *et al.* (2000) claimed that, under similar conditions to the above, Re-Os fractionation would be triggered in molybdenite. These two papers showed that the Re-Os system of molybdenite could be disturbed by post-depositional alteration at least at a micro scale. On the other hand, Stein *et al.* (1998b) indicate reproducible Re-Os ages for molybdenites by

Table 2. Results of Re and Os analysis of molybdenite standard HLP-5, provided by the AIRIE group, Colorado State University

Run	Re (ppm)	^{187}Os (ppb)	Re-Os age (Ma)
1	283.9 $\pm$ 5.0	661 $\pm$ 12	221.8 $\pm$ 5.5
2	282.5 $\pm$ 3.1	663.0 $\pm$ 3.3	223.7 $\pm$ 2.7
3	281.3 $\pm$ 2.6	652 $\pm$ 13	220.8 $\pm$ 4.8
4	290.1 $\pm$ 6.9	673.9 $\pm$ 4.7	221.5 $\pm$ 5.5
		average	222.0 $\pm$ 2.1
<i>n</i> = 7	Stein <i>et al.</i> (1997)	average	221.5 $\pm$ 0.3
<i>n</i> = 19	Markey <i>et al.</i> (1998)	average	221.3 $\pm$ 0.2

Errors are 2σ .

Re-Os age calculations were based on the ^{187}Re decay constant of $1.666 \times 10^{-11} \text{ yr}^{-1}$ (Smoliar *et al.*, 1996).

Errors in our four Re-Os ages do not include the 1.2% uncertainty in the ^{187}Re decay constant.

producing a sort of “whole-molybdenite” mineral separate, i.e., they collected all the molybdenites from a sample rock. They finely chop larger molybdenite crystals to produce a relatively fine-grained mineral separate. Their procedure could cancel the effect of subtle within-crystal alteration on the Re-Os system. However, if the sample experienced intense alteration, where Re and/or ^{187}Os have left the system, it might be dangerous to pulverize the whole sample at one time and produce sample powder, as Suzuki *et al.* (2000) suggested. The disturbed molybdenite would be mixed into unaltered, which leads to an erroneous age, though the Re-Os age would reproduce.

It is now well documented that the Galway Granite has experienced three major fluid events since its intrusion at *ca.* 400 Ma (Feely and Hoegelsberger, 1991; Gallagher *et al.*, 1992; O'Reilly *et al.*, 1997). The earliest fluid to be trapped was a carbonic fluid of magmatic origin and was responsible for the molybdenite mineralization. The second fluid was a H_2O -NaCl-KCl, low-moderate salinity (0–10 wt% NaCl eq.), moderate temperature (270–340°C), high δD (–18 $\pm$ 2‰), low $\delta^{18}\text{O}$ (0.5–2.0‰) fluid of meteoric origin. The circulation of this fluid probably occurred as the batholith was cooling. The final fluid in-

cursion involved low temperature (*ca.* 150°C) basinal brines that deposited calcite-quartz-fluorite and Cu-Pb sulfide veins in the granite. This event took place at least 100 Ma after granite consolidation and maybe as late as the Triassic, because field evidence reveals that these fluorite-bearing veins cut a 300 Ma dolerite dyke which intruded the granite. Both the first and second fluids are never found in these late fluorite veins, while fluid inclusions from all three events occur in quartz samples from the molybdenite veins and mineralized granite (Feely and Hoegelsberger, 1991; Gallagher *et al.*, 1992; O'Reilly *et al.*, 1997).

Based on the above consideration, we suggest, in line with McCandless *et al.* (1993) and Suzuki *et al.* (2000), that the Re-Os system in molybdenite from the Galway Granite has been significantly perturbed by hydrothermal alteration associated with at the second and third pervasive hydrothermal fluid events. The inconsistent Re-Os age reported by Hirt *et al.* (1963) may also reflect this alteration, although the analytical errors in their neutron activation analyses and mass spectrometry cannot be ruled out. As shown in Fig. 2, the Re-Os ages of molybdenite from Travore deviate the most significantly compared with the ages of the samples from Murvey and Mace, probably indicating the more intensive alteration. In addition, retrograde alteration of the granite (plagioclase to sericite, biotite and hornblende to chlorite $\pm$ epidote, calcite and reddening of K-feldspars) are associated with the second and third fluid events. The results obtained in this study combined with the previous experimental studies provide evidence that hydrothermal fluids may be responsible for disturbance of the molybdenite Re-Os system.

CONCLUSIONS

Analytical results of Re and Os reveals that contemporaneous molybdenites from Mace, Murvey, and Travore in the Galway Granite give inconsistent Re-Os ages even within each locality. Fluid inclusion studies indicate that the

molybdenites used in this study possibly experienced hydrothermal alteration after their deposition. These results allow us to suggest that the Re-Os system in molybdenite may show open-system behavior during post-depositional exposure to hydrothermal fluids, and that these fluids represent an event of as yet uncertain age and origin. This hydrothermal event must be significantly younger than the age of molybdenite deposition in order to achieve the results presented in this paper, and could be related to much younger fluorite mineralization.

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