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Re–Os isotope systematics for the Lince–Estefanía deposit: constraints on the timing and source of copper mineralization in a stratabound copper deposit, Coastal Cordillera of Northern Chile

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Abstract The Lince–Estefanía stratabound copper deposit in the Michilla district is one of the most important deposits in the Coastal Cordillera of northern Chile and is one of the most representative of this type of deposit. Chalcocite and bornite characterize the main stage of hypogene copper sulfide mineralization. Rhenium and osmium isotopes are used here to constrain the age of hypogene mineralization and the source of osmium contained in these ore minerals. A Re–Os isochron yielded an age of 160 ± 16 Ma (2σ), with

an associated initial $^{187}\text{Os}/^{188}\text{Os}$ ratio of 1.06 ± 0.09 (mean square of weighted deviates=1.8). This age is consistent with available geochronological data from volcanic rocks that host the mineralization and associated alteration phases. The high initial $^{187}\text{Os}/^{188}\text{Os}$ ratio indicates a lower crustal component for the source of Os and, by inference, the Cu sulfides that contain this Os. Late hematite occurs as an isolated phase or, more commonly, is associated with the chalcocite–bornite and supergene chalcocite–covellite associations. Analyses performed on pure hematite indicate a disturbance of the Re–Os system, and hence, this mineral phase is not useful as a Re–Os geochronometer.

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Introduction

The copper ore deposits hosted in the Jurassic volcanic sequence of the La Negra Formation (García 1967), referred to either as “manto type” (Ruíz et al. 1971) or “stratabound copper deposits” (e.g., Boric et al. 1990; Vivallo and Henríquez 1998), represent, after porphyry copper and Fe-oxide–Cu–Au deposits (e.g., Candelaria), the third most important source of copper in Chile (Minera Michilla S.A. Annual Report, 2004). These deposits form a metallogenetic province that extends throughout the present Coastal Cordillera of northern Chile, between 22 and 26°S. In spite of their economic importance and wide distribution, the stratabound copper deposits are poorly understood and their epigenetic vs syngenetic formation has been a matter of debate (e.g., Ruíz et al. 1971; Losert 1973; Espinoza 1982; Sato 1984; Espinoza and Orquera 1988; Klohn et al. 1990; Vivallo and Henríquez 1998; Kojima et al. 2003).

To properly evaluate any genetic model, it is critical to determine the age of mineralization. Direct ages of Cu mineralization are not available and most geochronological constraints are inferred from the hosting volcanic rocks and

gabbroic–dioritic intrusions spatially related to the ore bodies (e.g., Espinoza and Orquera 1988; Niemeyer et al. 1990). Rogers and Hawkesworth (1989) and Venegas et al. (1991) reported Rb–Sr isotopic ages of 186 ± 14 Ma (whole rock) and 173 ± 19 Ma, respectively, for the hosting La Negra Formation volcanic rocks. Recently, Oliveros et al. (2004a) obtained ^{40}Ar – ^{39}Ar plagioclase ages of 172.3 ± 0.9 Ma and 173.6 ± 1.4 Ma for the La Negra Formation. K–Ar (whole-rock and plagioclase) and Rb–Sr geochronology on plutonic rocks that intruded the volcanic sequence yielded ages that range from 112 to 168 Ma (e.g., Palacios and Definis 1981; Astudillo 1983; Espinoza and Orquera 1988; Boric et al. 1990; Venegas et al. 1991; Oliveros et al. 2004b). Consequently, the timing of mineralization can be constrained to between 186 and 112 Ma.

The Michilla district in northern Chile is the largest copper district in the stratabound metallogenic province with a copper production of $\sim 50,200$ t in 2004 (Minera Michilla S.A. Annual Report 2004). The district contains some of the most representative ore deposits of this type. The Lince–Estefanía (former Susana–Lince) deposit is the most important ore body containing the highest Cu resource in this district and was selected for a Re–Os isotopic study to constrain the age of mineralization and the source of Os, and, by inference, the source of the ore metals.

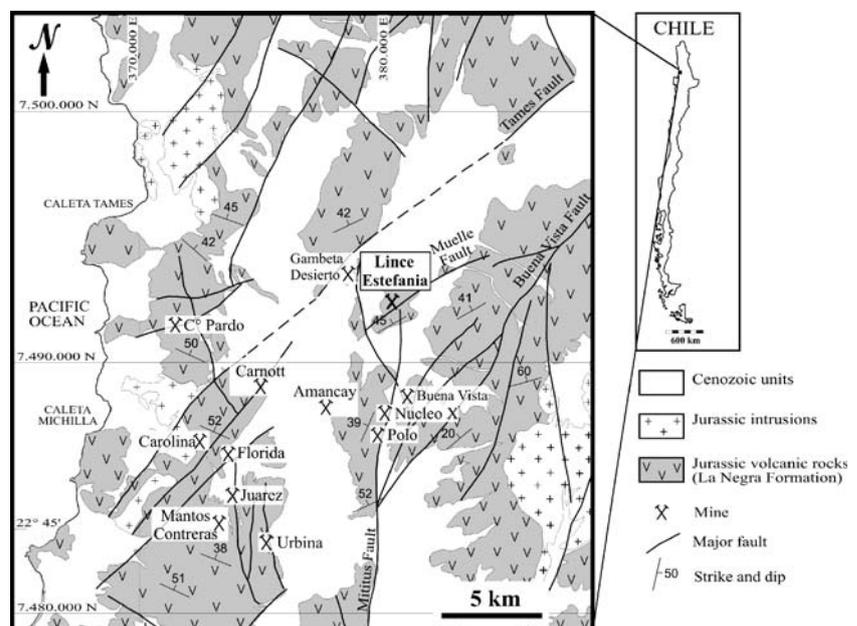
Geological background

The stratabound copper deposits of northern Chile are hosted mainly in the high-K calc-alkaline to tholeiitic andesitic–basaltic volcanic sequence of the La Negra Formation (i.e., García 1967; Chávez 1985). This formation was emplaced predominantly on a thinned pre-Jurassic continental crust, as a consequence of an extreme oblique

movement of the Pacific Phoenix Plate in the SSE direction relative to the continental margin (e.g., Scheuber and Reutter 1992; Scheuber and González 1999). Low initial $^{87}\text{Sr}/^{86}\text{Sr}$ ratios of these Jurassic volcanic rocks (0.7029 to 0.7035; Rogers and Hawkesworth 1989) and their major and trace element geochemistry support an extensional tectonic setting and are consistent with a mantle origin without appreciable crustal contamination (e.g., Lucassen and Franz 1994).

Plutonic bodies intruding the La Negra Formation have been grouped in the Coastal Batholith plutonic unit. The composition of these intrusions ranges from diorite to granite and they have initial $^{87}\text{Sr}/^{86}\text{Sr}$ ratios from 0.7032 to 0.7049 (Rogers and Hawkesworth 1989; Marinovic et al. 1995). Available geochronological data indicate that the emplacement of the plutonic bodies occurred mainly between 147 and 168 Ma (Espinoza and Orquera 1988; Boric et al. 1990; Maksiyev 1990). In addition, dioritic to gabbroic and andesitic subvolcanic bodies intruded the volcanic sequence as dikes and small stocks. The spatial relationship of some of these bodies with the Cu-stratabound deposits has been used to suggest a genetic link between these minor intrusions and the copper mineralization (Palacios and Definis 1981; Espinoza 1981, 1982; Espinoza and Palacios 1982; Wolf et al. 1990). The ages obtained from these subvolcanic bodies range between 112 and 168 Ma (e.g., Astudillo 1983; Espinoza and Orquera 1988; Boric et al. 1990; Venegas et al. 1991; Oliveros et al. 2004b). ^{40}Ar – ^{39}Ar plateau ages for plagioclase, biotite, and amphibole from intrusive rocks were recently reported by Oliveros et al. (2004a,b), with ages ranging from 136.1 ± 1.8 to 155.3 ± 1.6 Ma (2σ). These authors also provide indirect age determinations of the copper mineralization by dating associated intrusions and alteration minerals using the ^{40}Ar – ^{39}Ar method. Their results constrain the mineralization to between 138.1 ± 2.0 Ma and 154.4 ± 3.9 Ma (2σ) (Oliveros et al. 2004b).

Fig. 1 Geologic map and distribution of the main stratabound Cu deposits in the Michilla district, northern Chile



Lince–Estefanía geology

The Lince–Estefanía deposit, also known as Susana-Lince deposit (Kojima et al. 2003), is the most important source of copper in the Michilla district (Fig. 1). During 2004 it contributed ~80% of the Cu produced within the district, with an average grade of 0.8% Cu and 2.3% Cu at the Lince and Estefanía areas, respectively (Minera Michilla S.A. Annual Report, 2004). The deposit is located 130 km NNE from Antofagasta, northern Chile. At the deposit, the La Negra Formation consists exclusively of a thick sequence of andesitic lava flows forming a monoclin structure with a general orientation of N60°E/45°NW. Individual lava flows range typically from 2 to 10 m in thickness and show textural variations from breccia in the bottom of the flows, through aphanitic and porphyritic in the middle of the flows, and to amygdaloidal at the top of the flows.

Numerous dikes ranging between 10 cm and 30 m in thickness and small stocks of gabbroic–dioritic and dacitic composition intrude the volcanic sequence, nearly perpendicular to the bedding, with a NE orientation and SE dip. These dikes can be classified in two categories based on composition and temporal relation with the mineralization: (1) calc-alkaline diorite dikes interpreted as prehypogene Cu mineralization and (2) porphyritic hornblende-bearing calc-alkaline dacitic dikes that postdate Cu mineralization.

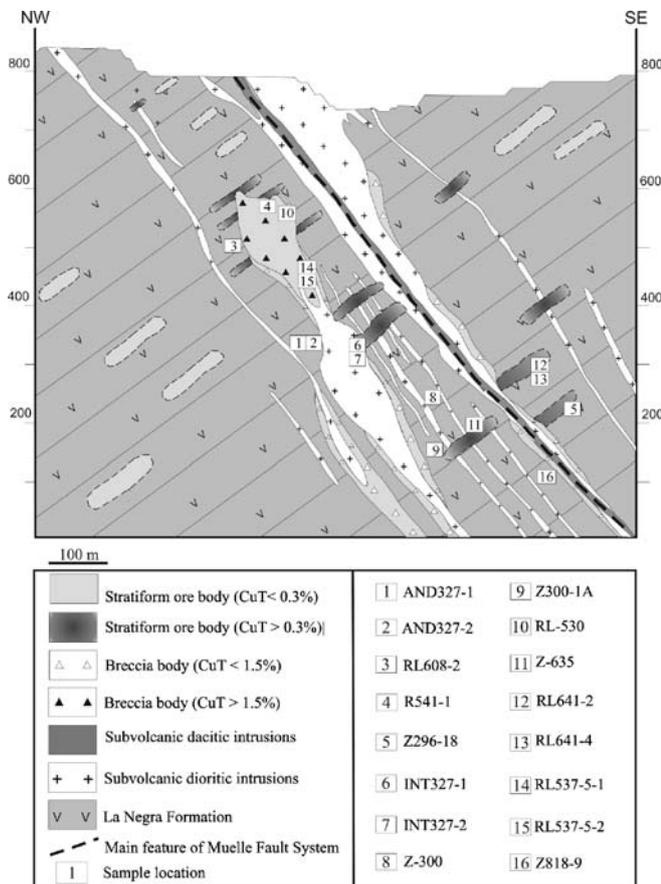


Fig. 2 Schematic cross-section showing the general geology of the Lince–Estefanía deposit and the location of samples used in the present study

The copper sulfides form parallel to subparallel (stratiform) and discordant breccia bodies, with veinlet formation locally. The stratiform bodies are the most common and abundant form of mineralization, and have the highest Cu content close to intrusives. The breccia bodies are generally located around the largest dioritic intrusions and extend laterally from the intrusions grading into stratiform bodies (“mantos”). Field and petrographic observations coupled with chemical analyses of copper ore suggest that copper sulfides in both types of ore bodies (stratiform or mantos and breccias) were formed by the same processes. Ore bodies are variable in size and range from 3 to 20 m in thickness with lateral extensions of up to 120 m along the strike and up to 200 m along the dip of the volcanic layers.

Ore minerals consist mainly of Cu sulfides and Cu oxides. Copper sulfides are represented by chalcocite (hypogene and supergene), bornite, covellite, and chalcopyrite, whereas Cu oxides predominantly include atacamite and chrysocolla. The ore minerals display a vertical zoning with sulfide phases occurring in the deepest levels [below 250 m above sea level (m.a.s.l.)] and oxides dominating near the surface (above 500 m.a.s.l.). A mixed-ore zone (oxide–sulfide) occurs between these two levels. Cu oxides also occur in trace amounts in the whole mineralized column (~1,000 m currently recognized). The alteration minerals related to the main Cu-sulfide mineralization are chlorite, epidote, and albite, with scarce quartz and actinolite (Wolf et al. 1990; Kojima et al. 2003), whereas the alteration mineral assemblage related to the Cu-oxide mineralization is calcite and gypsum with minor quartz.

Analytical methods

Representative samples were collected from drill core and underground works. Sample locations relative to the stratigraphy are shown in Fig. 2. Ore minerals and their associated alteration assemblages were studied under the microscope to determine the paragenetic sequence. Several well-constrained samples were analyzed using Re–Os systematics to determine the age of the hypogene mineralization. The isochron approach not only allows us to determine the age of mineralization but also the source of Os contained in the sulfides.

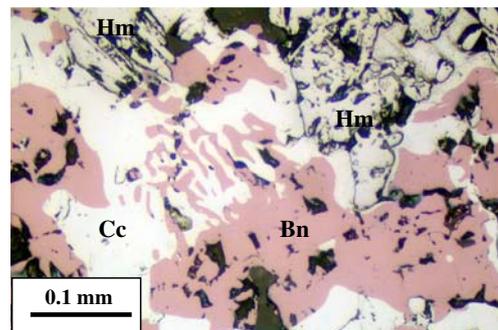


Fig. 3 Reflected light photomicrograph showing the textural relationship between bornite (Bn) and chalcocite (Cc), and late intergrown hematite (Hm)

Table 1 Re–Os data for pure mineral phases and mineral associations from the Lince–Estefanía deposit

Sample	Mineral association	Os (ppt)	Re (ppb)	$^{187}\text{Re}/^{188}\text{Os}$	$^{187}\text{Os}/^{188}\text{Os}$	$^{187}\text{Os}/^{188}\text{Os}$ initial
AND327-1	Cc	31	1.79	350.6±6.7	1.97±0.04	1.06 ^a
AND327-2	Cc	15	0.89	366.8±10.3	2.08±0.05	1.06 ^a
RL-541-1	Cc±Bn	15	0.19	75.8±3.8	1.27±0.07	1.06 ^a
RL-608-2	Cc±Bn	12	0.50	260.7±11.0	1.74±0.09	1.06 ^a
Z-296-18	Cc±Bn±Hm	22	0.62	179.6±5.7	1.75±0.06	1.23
INT327-1	Cc±Bn±Hm	24	2.30	747.1±20.4	3.89±0.12	1.82
INT327-2	Cc±Bn±Hm	26	2.49	773.5±30.0	4.93±0.19	2.75
Z-300	Cc±Bn±Hm	18	0.72	233.6±7.3	1.26±0.04	0.62
Z-300-1A	Cc±Bn±Hm	15	0.40	149.1±7.0	0.72±0.04	0.30
RL-530	Cc±Bn±Hm	11	0.50	246.4±9.4	1.06±0.03	0.40
RL-635	Cc±Bn±Hm	10	0.76	464.4±17.2	1.49±0.05	0.25
RL-641-2	Cc±Bn±Hm	7	0.23	193.0±13.1	0.91±0.06	0.38
RL-641-4	Cc±Bn±Hm	16	0.25	87.6±3.2	0.93±0.04	0.66
RL-537-5-1	Hm	18	9.42	2,934.5±117.4	0.91±0.03	−6.64
RL-537-5-2	Hm	18	3.68	1,128.2±41.6	0.90±0.03	−2.03
Z-818-9	Cc±Cv±Hm	16	5.06	1,883.3±55.4	1.74±0.05	−3.19

Calculated initial $^{187}\text{Os}/^{188}\text{Os}$ ratios are using an age of 160 Ma
Cc chalcocite, *Bn* bornite, *Hm* hematite, *Cv* covellite
^aInitial derived from isochron (Fig. 4a)

Two pure chalcocite samples (AND327-1 and AND327-2) and two samples of a mixture of chalcocite (90–95%) and bornite (10–5%) (RL608-2 and RL541-4) were analyzed for Re–Os isotopes to constrain the age of the hypogene mineralization. The close association of chalcocite and bornite in exsolution textures (Fig. 3) makes it virtually impossible to separate these different but cogenetic sulfide phases. In addition, nine samples containing the association chalcocite–bornite–hematite and one sample containing chalcocite–covellite–hematite were also analyzed (Table 1). In these cases, it was also impossible to separate the copper sulfides from hematite (Fig. 3) and the samples analyzed were mixtures of copper sulfides and late hematite. Replicate analyses of a single sample of pure hematite were also performed. The analyses of these associations were done to evaluate: (1) the influence of hematite in the Re–Os analyses and (2) to evaluate the mineral hematite for Re–Os geochronology.

Samples with fine-grained disseminations were ground, sieved, and separated by heavy liquids using methyl-iodide and a Frantz magnetic separator. The samples were then analyzed following the procedure described by Mathur et al. (2000) and Barra et al. (2003). Briefly, 0.7–1.7 g of sample was loaded in a Carius tube with Re (^{185}Re) and Os (^{190}Os) spikes and 16 ml of a 3:1 mixture of HNO_3 (16 N) and HCl (10 N) following the method indicated by Shirey and Walker (1995). To overcome the Claus effect and to achieve complete oxidation, 3 ml of hydrogen peroxide (30%) was also added in the Carius tube, as suggested by Frei et al. (1998). Samples and solutions were heated in an oven at 240°C for ~12 h. After complete dissolution and homogenization of samples, Os was separated from the solution in a two-step distillation process following the procedure described by Nägler and Frei (1997). Os was further purified using a microdistillation technique (Birck et al. 1997).

The Re remaining in solution from the distillation process was dried-down and redissolved in 0.1 N HNO_3 solution. Rhenium was purified through a two-stage

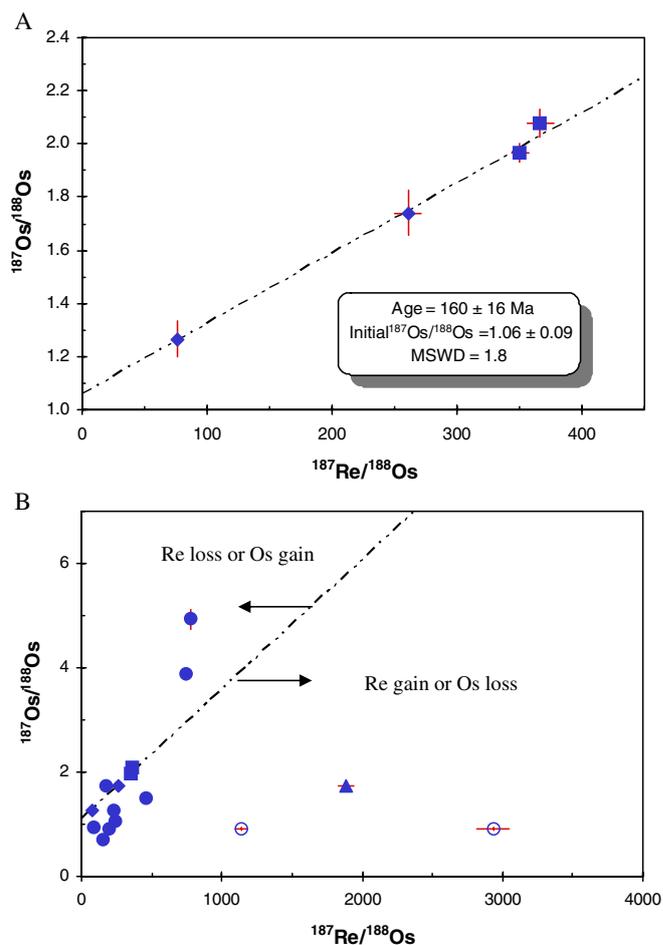


Fig. 4 Re–Os plots for mineral associations from Lince–Estefanía deposit. **a** Four-point isochron of hypogene Cu sulfide mineralization obtained from analyses of two pure chalcocite samples (*black squares*) and two chalcocite–bornite samples (*black diamonds*). **b** Re–Os analyses from chalcocite–bornite–hematite (*black circles*), chalcocite–covellite–hematite (*black triangle*), and pure hematite (*open circles*) mineral associations. The isochron from **a** is also plotted as reference (see text for discussion). Data plotted using Isoplot (Ludwig 2001)

column using AGI-X8 (100–200 mesh) resin. The column was washed with water and then treated with 8 N HNO₃ followed by 0.1 N HNO₃. The sample is loaded in the column and then rinsed three times with 0.1 N HNO₃. Rhenium is finally recovered with 8 N HNO₃. This solution is dried down and the whole process is repeated for the second stage using smaller-size columns. Finally, purified osmium and rhenium were loaded on platinum filaments with Ba(OH)₂ to enhance ionization and nickel filaments with Ba(NO₃)₂, respectively.

The samples were analyzed by negative thermal ion mass spectrometry (Creaser et al. 1991; Völkening et al., 1991) on a VG 54 mass spectrometer. Total blanks for low-concentration samples were 12±3 pg for Re and 1±0.5 pg of Os. The ¹⁸⁷Os/¹⁸⁸Os composition of the blank was 0.181±0.006. Isochron regression was calculated using Isoplot (ver. 2.49) of Ludwig (2001). All results are shown in Table 1 and Fig. 4.

Discussion

Age of hypogene copper mineralization

The Re–Os age of 160±16 Ma (2σ) obtained from samples of chalcocite and the chalcocite–bornite association, without hematite or covellite, is interpreted as the age of hypogene sulfide mineralization at the Lince–Estefanía deposit. This Re–Os age of 160±16 Ma (Fig. 4a) overlaps within error with the ⁴⁰Ar–³⁹Ar ages of 172.3±0.9 and 173.6±1.4 Ma reported for the La Negra Formation in the Antofagasta area (Oliveros et al. 2004a) and it is also in good agreement with ⁴⁰Ar–³⁹Ar plateau ages (138.1±2.0 to 154.4±3.9 Ma) obtained from alteration minerals in the Juárez ore deposit in the Michilla district (Oliveros et al. 2004b). These ages indicate that copper mineralization in the district occurred shortly after the emplacement of the volcanic rocks of the La Negra Formation, with several discrete and possibly contemporaneous pulses of mineralization between ~160 and ~140 Ma.

Hematite as a Re–Os geochronometer?

The presence of hematite associated with copper minerals and as a single phase presents an opportunity to evaluate the behavior of Re and Os in this oxide mineral. Replicate analyses of pure hematite gave variable ¹⁸⁷Re/¹⁸⁸Os at constant ¹⁸⁷Os/¹⁸⁸Os (Fig. 4b). If the Re–Os system has remained closed in this mineral, then these results would indicate that hematite precipitated recently and with a low ¹⁸⁷Os/¹⁸⁸Os initial ratio (~0.9; Fig. 4b). Moreover, the results show that pure hematite has similar Os concentrations than pure chalcocite and chalcocite–bornite samples, but significantly higher Re concentrations compared with the chalcocite, chalcocite–bornite, chalcocite–bornite–hematite and chalcocite–covellite–hematite mineral association than those of pure chalcocite and chalcocite–bornite samples, but pure hematite has significantly higher Re

concentrations. Experimental studies indicate that Re is 10²–10⁴ times more soluble than Os in high *f*O₂ fluids (Xiong and Wood 1999). The presence of abundant hematite at the Lince–Estefanía deposit indicates high *f*O₂ conditions and, consequently, Re mobility can be expected to be much higher than Os mobility. This oxidized fluid, probably of nonmagmatic origin (Kojima et al. 2003), interacted with some hypogene copper sulfides remobilizing preferentially Re over Os, and precipitating hematite with a high Re content.

Re–Os analyses of mineral associations that contain hematite do not form an isochron and none of these analyses fall within the isochron formed by chalcocite and samples from the chalcocite–bornite association (Fig. 4b). This is expected because petrographic evidence shows that hematite is mostly late in the system (Fig. 3). The analyses of samples of the chalcocite–bornite–hematite association are scattered within the Re–Os plot. These aberrant results are caused either by the presence of hematite and/or by the interaction of oxidized fluids, which were responsible for the formation of hematite, with the hypogene copper sulfides. Hence, the calculated ¹⁸⁷Os/¹⁸⁸Os initial ratios (Table 1) of this mineral association (chalcocite–bornite–hematite) have no geological meaning.

Numerous studies have shown open system behavior of the Re–Os isotopic system in various sulfide phases (Marcantonio et al. 1993; McCandless et al. 1993; Marcantonio et al. 1994; Foster et al. 1996; Lambert et al. 1998; Xiong and Wood 1999, 2000). In addition, some studies indicate that some phases are more susceptible to remobilization of Re and/or Os than others (Ruiz and Mathur 1999; Morelli et al. 2004). In the Urad–Henderson porphyry Mo deposit, USA, Ruiz and Mathur (1999) showed that magnetite was less susceptible to disturbance than associated sulfides. Morelli et al. (2004) concluded that sphalerite is subject to disturbance in the Red Dog Zn–Pb–Ag deposit, AK, whereas pyrite samples remained unaffected. In the present study we provide evidence that hematite is affected by open system behavior and constitutes a poor geochronometer, whereas the analyzed samples of pure chalcocite and samples of chalcocite intergrown or associated with bornite through exsolution textures (chalcocite–bornite association) have remained closed.

Source of osmium

The marked different chemical behavior of Re compared to Os during mantle melting processes makes this system a useful tool in evaluating crust–mantle interactions, and hence allows us to estimate the source of Os contained in ore minerals. In this study, the initial ¹⁸⁷Os/¹⁸⁸Os from the chalcocite and chalcocite–bornite isochron is 1.06±0.09 (Fig. 4a). This value is much more radiogenic than the chondritic reference ¹⁸⁷Os/¹⁸⁸Os ratio of ~0.13 at 160 Ma. In consequence, the Os contained in these sulfides has a significant crustal component, and hence we infer that the

Cu sulfides that contain this radiogenic Os also have a crustal component.

Two different possibilities can be evaluated as the source of Os (and copper) for these deposits: (1) volcanic rocks of the La Negra Formation and (2) lower crustal lithologies. The volcanic rocks of the La Negra Formation were mainly emplaced on a thinned pre-Jurassic continental crust, and geochemical studies support a mantle origin without appreciable crustal contamination (Rogers and Hawkesworth 1989; Lucassen and Franz 1994). Although there are no Re–Os data available for these rocks, it can be inferred that their $^{187}\text{Os}/^{188}\text{Os}$ ratio would be relatively low due to their proposed mantle origin. On the other hand, the consideration of the Cu concentration in these volcanic rocks (~100–300 ppm; Ruíz et al., 1971; Campano and Guerra 1979) and simple mass balance calculations indicate that a considerable volume of rock is required for the formation of these mantle deposits. This appears inconsistent with the relatively restricted volume of altered rock associated with the copper mineralization. Lower crustal rocks have highly variable Os compositions but are, in general, more radiogenic than upper crustal lithologies (Lambert et al. 1999). It follows then, and considering the geological setting of the Coastal Cordillera of northern Chile during Jurassic–Early Cretaceous times, that the Os, and possibly the copper, in the stratabound copper deposits have a lower crustal component.

Conclusions

The Re–Os isochron age of 160 ± 16 Ma obtained from chalcocite and chalcocite–bornite samples is the first direct age determination of the hypogene mineralization of the stratabound copper ore deposits in northern Chile. This age is in good agreement with geochronological information on associated volcanic host rocks and alteration minerals and supports the notion that Cu mineralization occurred during the evolution of the Jurassic to Early Cretaceous magmatic arc in the present Coastal Cordillera.

Re–Os analyses of samples of hematite and of the Cu sulfide–hematite association indicate that hematite is subject to open system behavior and is not a reliable mineral for Re–Os geochronology.

The initial $^{187}\text{Os}/^{188}\text{Os}$ ratio of 1.06 ± 0.09 obtained from the regression line of hypogene Cu and Cu–Fe sulfides indicates a lower crustal component for the source of Os and, by inference, for the hypogene copper sulfide mineralization.

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