

Molybdenite Re-Os dating of Mo-Th-Nb-REE rich marbles: pre-Variscan processes in Moldanubian Variegated Group (Czech Republic)

MILAN DRÁBEK¹ and HOLLY STEIN^{2,3}

¹Czech Geological Survey, Geologická 6, 152 00, Prague 5, Czech Republic; milan.drabek@alumni.uni-heidelberg.de

²AIRIE Program, Colorado State University, Fort Collins, CO 80523–1482, USA

³Centre for Earth Evolution and Dynamics, P.O. Box 1028, University of Oslo, 0316 Oslo, Norway; holly.stein@colostate.edu

(Manuscript received July 27, 2014; accepted in revised form March 12, 2015)

Abstract: In an effort to contribute to the discussion concerning the age of rocks of the Moldanubian Variegated Group, we have undertaken Re-Os dating of molybdenite of banded carbonatite-like marbles (CLM) from the graphite mine Václav at Bližná (Southern Bohemia), which belong to the metamorphic sequence of this group. The Re-Os model ages for the molybdenites range between 493 and 497 Ma and apparently correspond to the early stages of metamorphism connected with pre-Variscan rift-related tectono-metamorphic events, which affected and recrystallized sedimentary CLM material rich in Mo-Th-Nb-REE. The molybdenite bearing carbonatite like marbles situated in the footwall of Bližná graphite mine have been interpreted as carbonates with a large share of volcano-detritic material derived from contemporaneous primitive alkaline (carbonatite-like) volcanism deposited in a shallow marine lagoonal environment. There is no geological evidence for the participation of fluids mobilized from host rocks in the formation of the CLM. Because the Re-Os chronometer in molybdenite is demonstrably stable through later Variscan facies metamorphism, the molybdenite chronometer has not been affected by subsequent thermal overprints associated with the Variscan orogeny.

Key words: Re-Os dating, molybdenite, Moldanubicum, marble, Variegated group, carbonatite-like marbles, Bohemian Massif, Czech Republic.

Introduction

The Bohemian Massif comprises several metamorphic units among which the Moldanubian Zone is the southernmost. The Moldanubian Zone is a very complicated tectonic mélange of high-grade to medium-grade metamorphic rocks with a complex polyphase deformation history beginning with the Cadomian orogeny (Late Proterozoic–Early Paleozoic) and terminating with widespread Variscan tectonothermal activity (Chaloupský 1989; Cháb et al. 2010). The Moldanubian Zone consists of the Moldanubian Variegated Group, Moldanubian Monotonous Group and the Gföhl Unit (Cháb et al. 2010). The presently studied marbles belong to the Moldanubian Variegated Group (Fig. 1).

The age of individual tectonic slices varies from 2050 Ma (Wendt et al. 1992) to Paleozoic. The timing and stratigraphic division of the Moldanubian Zone is complicated by a strong Variscan overprint. Nevertheless, orthogneisses from Hluboká nad Vltavou dated by Vrána & Kröner (1995) using single grain evaporation yielded an age of 508 ± 7 Ma, which was interpreted as the time of granite emplacement. Whole rock Rb-Sr dating of the Choustník orthogneisses gave an isochron age of 459 ± 10 Ma (Rajlich et al. 1992). Pre-Variscan ages of 475–514 Ma for monazite were reported by Procházka et al. (2010) from the Sodoměřice leucogranite. *In situ* U-Pb dates of columbite and tantalite by La-SF-ICP-MS from Li-bearing Moldanubian pegmatites yielded emplacement ages of $\sim 333 \pm 3$ to 325 ± 4 Ma (Melleton et al. 2012). In an effort to contribute to the discussion concerning the age of the Mol-

danubian rocks, we have undertaken Re-Os dating of molybdenite from carbonatite-like marbles (CLM) from the Bližná graphite Václav Mine (Southern Bohemia, Czech Republic) which record the metamorphic sequence of this group.

Geological setting

The Bližná graphite mine Václav is situated in the southwestern part of the Moldanubian Variegated Group, correlative with the Český Krumlov unit (ČKU) (Zoubek 1979), 35 km SW of České Budějovice, Southern Bohemia, Czech Republic. The ČKU consists of biotite and amphibole-biotite paragneisses with frequent intercalations of quartzites, graphitic gneisses, calc-silicate rocks, calcite and dolomite marbles, and amphibolites. The abundance of scapolite in some marbles of the ČKU (Kříbek et al. 1997) has been used as an argument for their evaporitic origin. The rocks were metamorphosed under amphibolite facies conditions during regional Variscan metamorphism. The Bližná graphite deposit is confined to an intercalation of carbonate rocks in biotite paragneiss (Fig. 2) (Drábek et al. 1986, 1999; Veselovský et al. 1987). The carbonate rocks represent dolomite-calcitic and calcite-dolomitic marbles (metacarbonates). Graphitic marbles and calc-silicate rocks are represented to a lesser extent. Foliation is NE-SW with a steep NW dip, and in some parts of the deposit a SE dip. In addition to ordinary marble (OM), typical of the whole ČKU on the basis of mineralogy, chemistry and composition of radiogenic (Sr-Nd) and stable

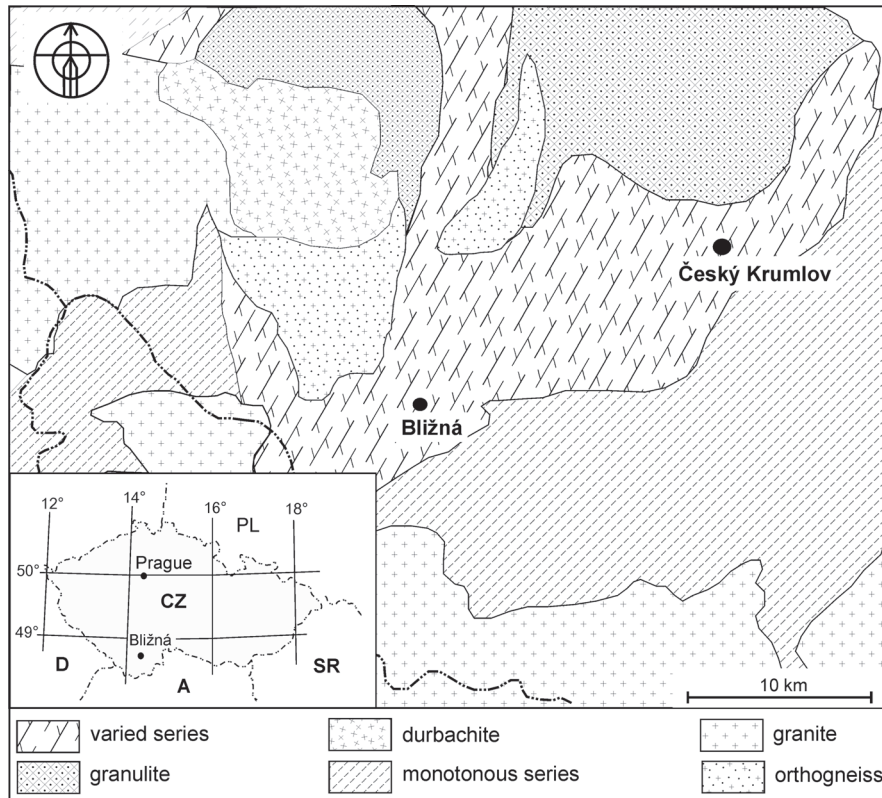


Fig. 1. Geological map of the southeastern part of the Bohemian Massif showing the location of Bližná (simplified from Kodym 1960 and Čech 1961). GPS coordinates of the studied area: N 48°43' 20", E 14°5' 48".

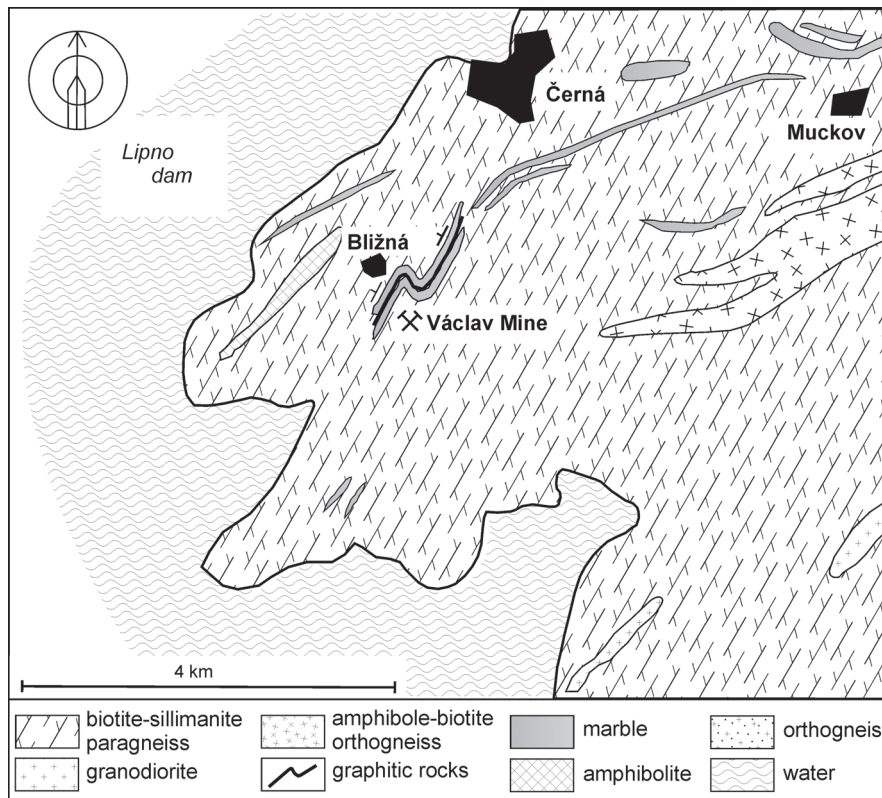


Fig. 2. Schematic geology of the Václav mine, Bližná (simplified from Kodym 1990).

(C-O) isotopes, Drábek et al. (1999) distinguished in the Bližná graphite mine two other types of marbles: carbonatite-like marble (CLM) and transitional marble (TM). The TM is less enriched in elements typical of CLM, however relations between the elements are preserved (Veselovský et al. 1987). The CLM differs markedly from other marbles of the ČKU, and chemically and isotopically resemble carbonatite compositions. CLM and TM occur only in the footwall of the graphite bed, where they form a single continuous stratabound layer. Only CLM are molybdenite bearing.

Banded carbonatite-like marble

The banded carbonatite-like marble (CLM) forms medium grained and silicate-rich intervals that are distinctly layered (Fig. 3). The darker layers are particularly enriched in silicates. The lighter layers consist primarily of calcite. Insoluble residues of CLM vary between 1.5 and 65.4 wt. %. The mineralogy of the insoluble residue of CLM is quite complex. The mineralogy of the CLM has been described by Drábek et al. (1986), Veselovský et al. (1987) and Drábek et al. (1999). The mineral assemblage of the CLM includes calcite, diopside, edenite, phlogopite, forsterite, antigorite, talc, chlorite and serpentinized forsterite. Edenite (subcalcic sodium edenite according to the IMA nomenclature Leake, B.E. 1978) is the most abundant silicate in the CLM. Edenite forms idiomorphic columnar crystals up to several mm in size (Fig. 4).

The Mo-Nb-Th-REE mineralization in CLM is represented by the following sulphides and oxides: molybdenite, pyrite, pyrrhotite, galena, chalcopyrite, sphalerite, pyrochlore, ilmenite, magnetite, rutile, euxenite and uranothorite. Barite is the only sulphate mineral, and apatite is the only phosphate mineral.

Molybdenite (MoS_2) is quite common in the CLM. Whole rock molybdenum contents vary from 7 to 1930 ppm (average, 754 ppm). Molybdenite flakes occur in both the darker and lighter layers, but in the darker layers the percentage of molybdenite is higher. Molybdenite forms perfect euhedral, isolated hexagonal thin platelet crystals, most commonly 1–3 mm but up to 7 mm in size (Fig. 5). Stacked molybdenite platelets comprise molybdenite crystals (Fig. 6). X-ray diffraction pattern for molybdenite correspond to the $2H$ polytype. Very sharp diffraction profiles attest to excellent crystallinity of molybdenite. Drábek et al. (1993) reported the following trace element compositions in molybdenite: 10 ppm Re, 20 ppm W, 51 ppm Se, and 4 ppm Te. These contents are low compared to molybdenites from other occurrences in the Bohemian Massif (Drábek et al. 1993). The isotopic composition of $\delta^{34}\text{S}$ in molybdenite is +4.2‰ (Drábek & Hladíková 1990). Pyrite, pyrrhotite and chalcopyrite occur together (Fig. 7). Pyrite forms small euhedral cubic crystals containing 0.2 ppm Te (AAS). Pyrrhotite forms small irregular grains and chalcopyrite forms small inclusions in other sulphides. Pyrochlore occurs together with ilmenite and magnetite. Pyrochlore (according to Atencio et al. (2010) classification, calciopyrochlore) forms grains up to 3 cm in size or small euhedral crystals with rounded edges typically 2 mm across. Pyrochlore is strongly enriched in Th



Fig. 3. Markedly layered CLM from the 3rd level of the Václav graphite mine. The dark layers are silica-rich.

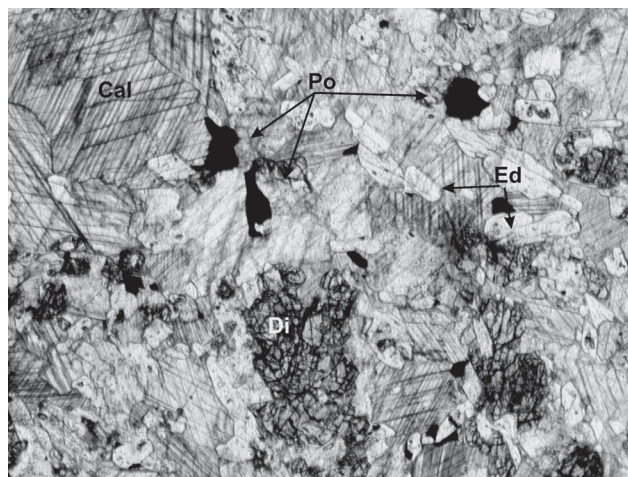


Fig. 4. Transmitted light microphotograph of CLM. Edenite replacing diopside in CLM. Plane polarized light. **Ed** — edenite, **Di** — diopside, **Cal** — calcite, **Po** — pyrrhotite.

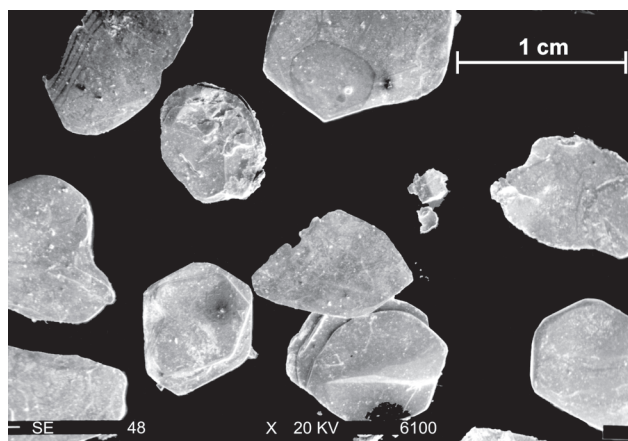


Fig. 5. Photomicrograph of euhedral molybdenite platelets. SEM.

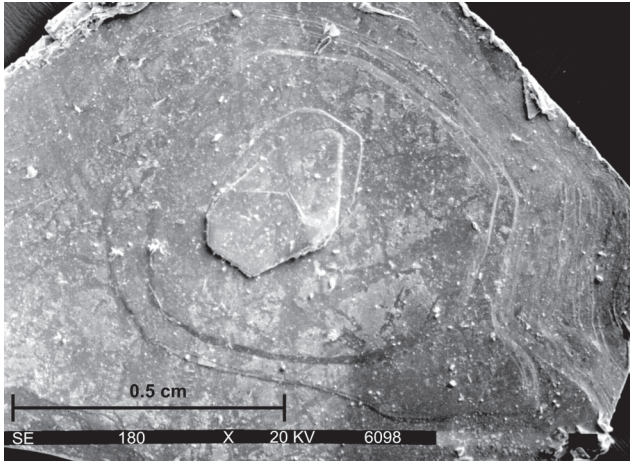


Fig. 6. Staked molybdenite plates, show sequential growth of the crystal. SEM.

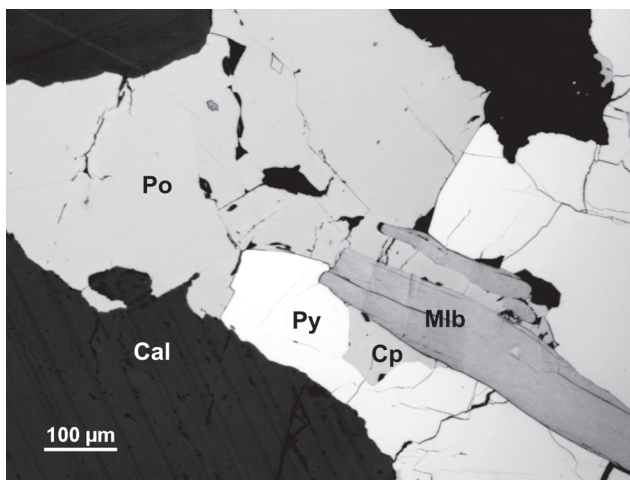


Fig. 7. Photomicrograph of molybdenite (Mlb), pyrite (Py), pyrrothite (Po), chalcopyrite (Cp), and calcite (Cal) intergrowths in CLM. Reflected light.

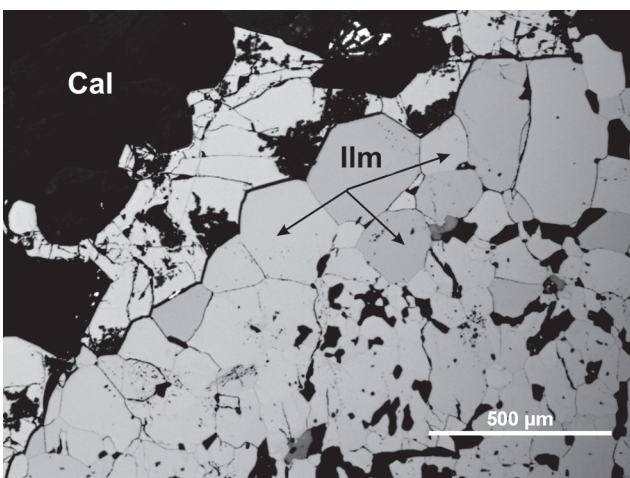


Fig. 8. Ilmenite grain. Photomicrograph of bubble foam structure of ilmenite (Ilm). Reflected light.

and REE. Ilmenite is most commonly found as individual grains commonly showing bubble foam structure (Craig & Vaughan 1981) indicating a recrystallization during thermal metamorphism (Fig. 8) or prismatic exsolution lamellae in magnetite. Magnetite forms rounded aggregates up to 10 mm across. Galena, rutile, euxenite and uranothorite were identified only as small (5–10 μm) inclusions in pyrochlore. In contrast to the OM, the CLM is graphite free. Representative chemical compositions of CLM and OM are given in Table 1.

The MgO content in CLM is low and ranges from 5 to 21 wt. %. The CLM are enriched in elements typical for carbonatites: Y, Th, Nb, Zr, Sr, Mo and the CLM is markedly enriched in REEs (e.g. Hoernle et al. 2002). Compared with OM,

Table 1: Representative chemical compositions of carbonatite-like marble (CLM) and ordinary marble (OM) from the 3rd level of the Václav mine, Bližná.

Sample No.	CLM BL 4	CLM BL 11	OM BL61
%			
SiO ₂	25.26	15.92	8.96
TiO ₂	0.15	0.07	0.02
Al ₂ O ₃	2.63	1.56	0.50
Fe ₂ O ₃	0.49	1.93	<0.10
FeO	0.42	2.62	0.27
MnO	0.13	0.41	0.21
MgO	7.24	7.21	20.00
CaO	38.12	39.42	29.61
SrO	0.02	0.15	0.01
BaO	0.02	0.16	<0.005
Li ₂ O	<0.005	<0.005	<0.005
Na ₂ O	0.09	0.47	0.01
K ₂ O	1.02	0.12	0.08
P ₂ O ₅	0.02	0.03	0.02
CO ₂	22.62	27.24	37.66
H ₂ O ⁺	0.40	<0.01	1.38
F	0.05	0.05	0.03
S	0.08	0.61	0.13
Σ	99.33	99.21	99.21
Nb	16	<7	<7
Y	307	409	<7
Zn	146	105	159
Cu	11	57	<7
Ni	<7	<7	<7
Pb	176	1334	31
Mo	465	1930	<7
Zr	28	40	<7
Th	222	b.d.	b.d.
La	56.21	72.70	1.41
Ce	258.56	332.97	3.72
Pr	46.82	60.53	b.d.
Nd	239.45	322.49	1.73
Sm	80.53	111.31	0.30
Eu	28.01	41.08	0.08
Gd	72.24	117.20	b.d.
Tb	10.72	18.89	b.d.
Dy	66.22	113.43	0.23
Ho	11.81	20.70	b.d.
Er	30.24	51.13	0.13
Tm	3.71	6.77	b.d.
Yb	22.34	38.92	0.15
Lu	2.44	4.67	b.d.
Σ	325.70	572.97	1.37
Au	5.20	17.2	2.00

b.d. — below detection, n.d. — not determined.

CLM are further enriched in Sr, Ba, Cu, Pb, and Zn. Concentrations of Cr, Ni and V are below their detection limits (<7 ppm). The chondrite-normalized pattern of REE is strongly enriched in Ce-Gd (Drábek et al. 1999). Compared to CLM, the OM has higher MgO contents and elements typical for CLM (Mo, Nb, Th) are below the detection limit (<7 ppm). The Sr and REE contents are also significantly lower in OM. Furthermore, the CLM have significantly lighter isotopic compositions of oxygen compared to OM from the ČKU. The isotopic composition of $\delta^{18}\text{O}$ and $\delta^{13}\text{C}$ in CLM vary from -15 to -19 ‰ and from -3 to -6 ‰, respectively. This isotopic composition falls into the range reported for carbonatite rocks (e.g. Heinrich 1966). The isotopic composition of Sr in CLM also differs from OM. According to Drábek et al. (1999) the Sr isotopic composition of the CLM is relatively primitive with the $^{87}\text{Sr}/^{86}\text{Sr}_{450} \sim 0.708$. On the other hand, the OM has a more evolved strontium ratio with the $^{87}\text{Sr}/^{86}\text{Sr}_{450} \sim 0.715$.

The CLM have been interpreted by Drábek et al. (1999) and Drábek & Stein (2003) as regionally metamorphosed carbonates (metacarbonates) with a large share of volcanodetrictic material derived from contemporaneous primitive alkaline (carbonatite-like) volcanism deposited in a shallow marine lagoonal environment that also produced evaporitic sequences. There is no geological evidence for the participation of fluids mobilized from host rocks in the formation of CLM. Mineralization in CLM is strictly stratiform (Drábek et al. 1990), and the surrounding rocks (graphite and paragneisses) contain only pyrite, pyrrhotite and rare sphalerite. Molybdenum was not detected in these rocks (Jifele 1984).

Sample description

The molybdenite sample used for dating comes from banded carbonatite like marble within the 3rd level of the Václav graphite mine. The molybdenite used in this study comes from two distinct mineral separates prepared at AIRIE (Applied Isotope Research Program, Colorado State, University) from a large hand-size sample of the dark grey variety of CLM. In this sample, molybdenite occurs as disseminated flakes 1–2 mm in diameter (Fig. 6).

Analytical technique

Molybdenite presents a unique set-up for the Re-Os method of dating in that it usually contains ppm level Re and essentially no initial or common Os, making it a single mineral chronometer. General principles and methodology for molybdenite dating are outlined in Stein et al. (1997, 2001). Sample-to-sample reproducibility for molybdenites illustrating the robustness of the chronometer has been documented (e.g. Watanabe & Stein 2000 — see summary in Stein 2014), and the chronometer is demonstrably robust through granulite facies metamorphism and ductile deformation (e.g. Stein & Bingen 2002; Bingen & Stein 2003). Sample size and preparation followed the procedures to obtain meaningful age results (e.g. Stein et al. 2006). The two molybdenite sep-

Table 2: Re-Os data for molybdenite from Carbonatite-like marbles (CLM), Václav graphite mine, Bližná.

AIRIE Run #	Sample	Re, ppm	¹⁸⁷ Os, ppb	Age, Ma
CT-509	sep. #1	5.462 (6)	28.54 (2)	497 ± 2
CT-558	sep. #2	10.24 (1)	53.05 (5)	493 ± 2
Absolute uncertainties shown, all at 2-sigma level.				
Decay constant used for ¹⁸⁷ Re is 1.666 × 10 ⁻¹¹ yr ⁻¹ (Smoliar et al. 1996).				
Ages calculated using ¹⁸⁷ Os = ¹⁸⁷ Re (e ^{λt} - 1) include all analytical and ¹⁸⁷ Re decay constant uncertainties.				

arates were 16 and 41 mg each. A Carius-tube digestion was used, whereby molybdenite is dissolved and equilibrated with ¹⁸⁵Re and ¹⁹⁰Os spikes in HNO₃-HCl (inverse *aqua regia*) by sealing in a thick-walled glass ampoule and heating for 12 hours at 230 °C. The Os is recovered by distilling directly from the Carius tube *aqua regia* into HBr, and is subsequently purified by micro-distillation. The Re is recovered by anion exchange. The Re and Os are loaded onto Pt filaments, and isotopic compositions were determined in 2002 using NTIMS on NBS 12-inch radius, 68° and 90° sector mass spectrometers at Colorado State University (now replaced by two Triton TIMS machines). Two in-house molybdenite standards, established and calibrated at AIRIE, are routinely run as an internal check (Markey et al. 1998). Blanks are insignificant (Re < 10 pg, ¹⁸⁷Os < 3 pg). The Re-Os data and ages are shown in Table 2 along with a footnote explanation of uncertainties in measurements and calculations.

Chemical analyses were performed in the Central Laboratory of the Czech Geological Survey, Prague. Major-elements were determined using wet chemical analysis; trace-elements were determined by ICP.

Discussion

The Re-Os ages 497 ± 2 and 493 ± 2 Ma obtained from the investigated samples are contradictory to a Precambrian age for the Moldanubian Variegated Group in Southern Bohemia suggested, for example, by Kodým (1966) Chaloupský (1978), Zoubek (1979), Konzalová M. (1981), Frank et al. (1990) and Procházka (2007). On the contrary, this age is consistent with that of Kröner et al. (2000). They reported two concordant zircon ages of 469.3 ± 3.8 Ma (cores with Variscan overgrowths) from regionally overlying granulite of the Gföhl Unit. A maximum age limit T_{DM}^{Nd} of 530 Ma of the betafite (Drábek et al. 1999) from CLM is also in agreement with our dating. The reported age is also in accordance with the suggestion made by Janoušek et al. (1997, 2008). These authors interpreted metabasic rocks from the ČKU as EMORB tholeiite basalts derived by Early Paleozoic melting of a strongly depleted mantle source ($\epsilon_{Nd}^{500} = +8.6$ to 9.4; $T_{DM}^{Nd} = 0.43$ –0.50 Ga). Friedl et al. (2004) also reported pre-Variscan geological events from the Austrian part of the Bohemian massif. Houzar & Novák (2002) distinguished events related to a polyphase metamorphism in the variegated units of the Bohemian Massif. According to these authors, diopside-bearing assemblages in marbles correspond to temperatures as

high as $T > 630\text{--}660\text{ }^{\circ}\text{C}$. Detailed field observations show that CLM forms a single continuous stratabound layer. There is no geological evidence for the participation of fluids mobilized from the host rocks or for metasomatic processes. A possible role for the evaporites is unclear. The high crystallinity and perfect shape of molybdenite crystals supports the concept of crystallization of molybdenite in a carbonate-rich shale matrix of a Ca-rich layer with a large component of volcano-detrital material (insoluble component up to 65.4 wt. %) enriched in trace elements typical of carbonatites, such as REE, Y, Th, Nb, Zr Sr and Mo. On the basis of trace element chemistry and the isotopic composition of C, O and Sr we suggest that the volcano-detrital material was derived from contemporaneous relatively primitive (alkaline carbonatite-like) volcanism which was deposited in a shallow marine lagoonal environment which also produced evaporitic deposits, and later, with metamorphism, graphite sequences. These constraints support the suggestion that the Re-Os molybdenite ages date geological processes that appear to have followed volcanic activity with unusual alkaline compositions captured in volcanic-detrital basinal sedimentation as intercalations within the carbonate layers. The Re-Os molybdenite ages presented in this paper correspond to post-Cadomian rifting in the Moldanubian Zone accompanied by intrusions of basic and acid magmas between 600 and 490 Ma (e.g. Gebauer & Grünenfelder 1982; Teufel 1988; Finger & Steyrer 1995; Fritz 1996; Friedl et al. 2004). Fiala (1976) also reported Proterozoic volcanism with a calc-alkaline trend in the Barrandian basin. Houzar & Novák (2002) described metacarbonates with a carbonatite-like signature from lithologically similar metamorphic sequences assigned to different Variegated geological units of the Bohemian Massif (Moravicum and Silesicum). Nevertheless, additional geochronological and petrological data are needed particularly for the ČKU.

Conclusions

Our Re-Os dating of molybdenite from the CLM provides ages for the formation of molybdenite at about 495 Ma (497 ± 2 and 493 ± 2). As expected, the Re-Os ages for molybdenite are not affected by high-grade Variscan metamorphism and the molybdenites preserve their pre-Variscan age of formation. We interpret the ages presented in this paper as a record of pre-Variscan metamorphism of limestones with an admixture of volcanic-detrital basinal sediments of alkaline composition related to rifting and crustal thinning on the Gondwana margin, during extensional tectonics.

Acknowledgments: This paper is dedicated to the late Professor Zdeněk Pouba. The Re-Os dating was supported by a U.S. National Science Foundation Grant EAR-0087483 to HS. Previous and on-going studies at the Václav graphite mine are supported by Czech Grant Agency Grant 205/96/563 to MD. We wish to thank V. Sixta and D. Weiss, (Czech Geological Survey, Prague) for the chemical analyses. Comments made by S. Vrána, M. Rieder and anonymous reviewers are gratefully acknowledged. We appreciate the valuable

comments of handling editor Igor Broska, which significantly improved our paper.

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