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Isotopic Re-Os age of molybdenite from the Szklarska Poręba Huta Quarry (Karkonosze, SW Poland)

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New Re/Os isotopic data for molybdenite from the Szklarska Poręba Huta Quarry provide ages of 307 ± 2 Ma and 309 ± 2 Ma, respectively. The quarry is dominated by the porphyritic ("central") and equigranular ("ridge") varieties of the Karkonosze granite. Ore mineralisation hosted in aplogranite includes an assemblage of sulphides, sulphosalts, oxides and various rare phases. The molybdenite ages obtained are consistent with a previously published isotopic age of leucogranite (aplogranite?) from the same quarry and are only slightly older than a recently published, refined $^{206}Pb/^{238}U$ age of untreated zircons from the Szklarska Poręba Huta porphyritic granite. The age of the molybdenite corresponds moderately well to the younger stage of post-magmatic, pneumatolitic/hydrothermal activity of the Karkonosze granite (about 312 Ma).

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INTRODUCTION

The Variscan Karkonosze granite intrusion is located in southwestern Poland and in northeastern Bohemia. It belongs to a system of European Variscan granitoid plutons known from Poland, the Czech Republic, Germany, France, England, Spain and Portugal and elsewhere (see e.g., Seltmann and Faragher, 1994). The Polish members of this granitoid province are generally related to various stages of the Variscan orogeny although their geotectonic affinity is still disputed (e.g., Wilamowski, 1998; Kennan *et al*., 1999; Oberc-Dziedzic *et al*., 1999, 2010; Mazur *et al*., 2007; Mierzejewski, 2007).

As in other Variscan granitoids (see e.g., Seltmann and Faragher, 1994), the Karkonosze intrusion and its metamorphic envelope are well-known hosts of diverse, primary ore mineralisation resulted from the combined effect of the thermal energy transfer from the pluton and a related, hydrothermal solution circulation system. General data on ore mineralisation in the Karkonosze granite and its metamorphic envelope can be found in Berg (1913), Petrascheck (1933, 1937), Gajda (1960*a*), Kozłowski (1978), Mochnacka *et al*. (1995), Mochnacka (2000), Mochnacka and Banaś (2000), Michniewicz (2003) and Mikulski (2007) (and references therein).

A well-known site of hydrothermal ore mineralisation including molybdenite is the granite quarry in Szklarska Poręba Huta (Fig. 1). As molybdenite is a perfect material for Re-Os isotopic age determination due to relatively high contents of Re and to the origin of practically all Os from the decay of Re, the authors used MoS_2 specimens for age determination of part of the ore mineralisation from that site. It supplements existing age determinations of Karkonosze granite-related mineralisation.

GEOLOGICAL SETTING

The Karkonosze-Izera Massif (KIM) is the largest structure in the Western Sudetes. It comprises two main elements: the

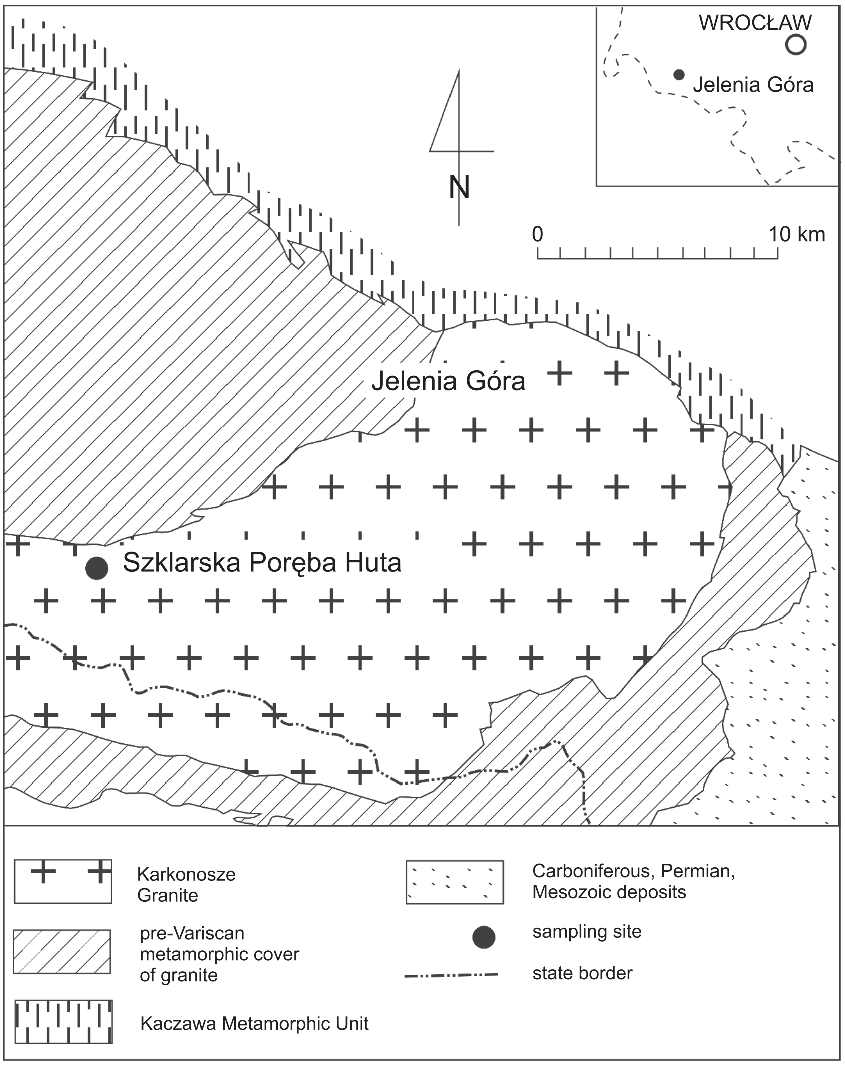

Fig. 1. Simplified geological map of the eastern part of the Karkonosze-Izera Massif (modified after Mochnacka *et al*., 2008)

Variscan Karkonosze granite intrusion and its Neoproterozic-Paleozoic metamorphic cover. The latter shows remarkable diversity in stratigraphy and metamorphic history, which enable distinction of four consecutive structural units: Izera-Kowary, Ješted, South Karkonosze and Leszczyniec, arranged as a stack of nappes formed in the Late Devonian and re-arranged in the early Carboniferous (for details see Mazur and Aleksandrowski, 2001). During the Variscan orogeny the nappe structure was intruded by the Karkonosze granite which resulted in the formation of an extensive contact aureole (Mierzejewski and Oberc-Dziedzic, 1990).

The Karkonosze granite is a complex intrusion comprising three petrographic varieties: central (porphyritic), ridge (equigranular) and granophyric (fine-grained), of roughly similar chemical compositions (corresponding to monzonites and granodiorites) but different structures, accessory mineral assemblages, and the frequency of schlieren and enclaves (for details see Borkowska, 1966). Various aspects of the Karkonosze granite were discussed by e.g., Cloos (1925), Borkowska (1966), Klomínský (1969), Mierzejewski and Oberc-Dziedzic (1990), Wilamowski (1998), Mierzejewski (2003, 2007), Žák and Klomínský (2007), Słaby and Martin (2008) and others.

Isotopic data (see Duthou *et al*., 1991; Mierzejewski *et al*., 1994) as well as geochemical modelling (Słaby and Martin, 2008) preclude a pure, crustal source of magma and suggest rather its origin from the mixing of crustal and mantle magmas (Mierzejewski *et al*., 1994; Mierzejewski, 2007), which resulted in the formation of porphyritic granite and subsequent fractional crystallisation leading to the equigranular variety (Słaby and Martin, 2008).

ORE MINERALISATION IN THE SZKLARSKA PORĘBA HUTA QUARRY

In the Szklarska Poręba Huta Quarry both the porphyritic ("central") and the equigranular ("ridge") granite varieties can be found. Both enclose common, bright (aplitic) and dark (biotitic) schlieren and dark, ellipsoidal, fine-crystalline enclaves, and both are cut by thin aplite veins. The porphyritic granite contains coarsely crystalline, pink K-feldspar accompanied by white plagioclase, grey quartz, and biotite with accessory zircon and apatite. A characteristic feature is the presence of large phenocrysts of pink K-feldspar rimmed by plagioclase (see Kryza *et al.*, 2012). Aplogranite, which accompanies the porphyritic granite, appears as a whitish, generally finely crystalline mass of grey quartz, whitish feldspars including plagioclases, and biotite. It encloses small (about 5 cm across), irregular, pegmatitic aggregates composed of euhedral/subhedral, white feldspars and grey quartz accompanied by biotite and pyrite. Field data and isotopic age determinations (Mikulski *et al.*, 2004) indicate that the aplogranite is younger than the porphyritic granite (see Table 2).

The Szklarska Poręba Huta Quarry is a well-known site of ore mineralisation described by many authors (see Berg, 1923; Gajda, 1960*a*, *b*; Karwowski *et al*., 1973; Kozłowski *et al*., 1975; Olszyński *et al*., 1976; Kozłowski *et al*., 2002; Pieczka and Gołębiowska, 2002; Kozłowski and Sachanbiński, 2007; Mikulski and Stein, 2007). A diverse ore mineral assemblage identified by these authors includes: Fe-wolframite, scheelite, cassiterite, native Bi, bismuthinite, molybdenite accompanied by arsenopyrite, pyrrhotite, chalcopyrite, pyrite, tetrahedrite, sphalerite, galena, marcasite, melnikovite, chalcocite, native Ag, Ti-magnetite, magnetite, wulfenite, stolzite, emplectite, nuffieldite, rutile, monazite, zircon, fergusonite, niobite, gadolinite, hingganite, thorite, titanite and a variety of secondary minerals.

We found ore mineralisation in bright, fine-crystalline aplogranite. Ore minerals form disseminated structures or, less commonly, larger aggregates, up to 2 mm across, accompanying coarsely crystalline, drusy quartz-feldspar intergrowths. Under the microscope we identified: pyrite, pyrrhotite, sphalerite and chalcopyrite accompanied by molybdenite, rutile, wolframite, scheelite, magnetite, cassiterite, marcasite, uranothorite? and trace amounts of native Bi, bismuthinite, chalcocite and ilmenite. At the present stage of observations, a detailed ore mineral succession cannot be established due to the small number of samples although at least two stages of this mineralisation are suggested.

Molybdenite was found in several samples as individual, disseminated crystals accompanying coarsely crystalline, drusy quartz-feldspar aggregates (Fig. 2). Molybdenite crystals show typical optical properties. Its chemical composition indicates trace admixtures of tungsten (0.01 and 0.07 wt.%).

Pyrrhotite usually forms anhedral crystals and aggregates. Some pyrrhotite grains resemble pseudomorphs after an unidentified flaky mineral. Pyrrhotite does not contain significant admixtures of trace elements.

Chalcopyrite forms intergrowths with pyrrhotite or individual crystals. The chemical composition of the chalcopyrite does not include remarkable trace elements.

Marcasite forms aggregates of elongated crystals. Their chemical composition reveals Mn (0.03 wt.%) and Ti (0.04 wt.%).

Pyrite was observed as disseminated crystals, locally accompanying magnetite aggregates.

Wolframite and scheelite form spotty aggregates composed of anhedral crystals. Their intercrystal spaces are filled with chalcopyrite, bismuthinite and native Bi. The chemical composition of the wolframite shows increased amounts of Mn (6.8 wt.%) whereas in scheelite Mn contents are low (0.02 wt.%).

Magnetite forms aggregates of anhedral crystals, which fill spaces between large crystals of rock-forming minerals. Lamellar intergrowths of ilmenite are common. Locally, magnetite aggregates are rimmed by ilmenite overgrowths. Some ilmenite crystals host minute cassiterite inclusions as well as uranothorite crystals. Magnetite-ilmenite intergrowths are usually surrounded by disseminated pyrite accumulations.

Ilmenite contains high amounts of MnO (up to 10 wt.%) and also TiO_2 (51.2–51.4 wt.%), FeO (2.0–3.0 wt.%) and V_2O_3 (0.2–0.3 wt.%).

The X-ray powder pattern enabled us to identify xenotime and monazite accompanied by ilmenite, pyrite and unidentified uranium minerals.

AGE DETERMINATIONS

AVAILABLE DATA

The age determinations of the Karkonosze granite are listed in Table 2. A wide range of results obtained with various methods of isotopic age studies give age limits of Karkonosze intrusion formation between 328 ± 12 Ma (Pin *et al*., 1987) and 292 Ma (Przewłocki *et al*., 1962) (see Table 2). The age of the porphyritic granite variety, which hosts our molybdenite crystals, was determined by several authors, giving ages between 328 ± 12 Ma (Pin *et al*., 1987) and 296 Ma (younger age provided by Depciuch and Lis, 1971). However, Kryza *et al*. (2012) showed that the young ages reported for the porphyritic granite (297 and 301 Ma) may be an effect of Pb loss.

There are few isotopic ages of ore minerals from the Karkonosze granite. Kucha *et al*. (1986) provided a U/Th/Pb age of disseminated, Th-bearing uraninite (bröggerite) collected from a core from the Jakuszyce IG 1 well at 299.8 Ma. Mikulski *et al*. (2004) determined, using the CHIME method (U, Th, Pb), the ages of monazite and xenotime in aplogranite from the Szklarska Poręba Huta Quarry at 271 ± 20 Ma and interpreted this age as the formation time of the aplogranite and of pneumatolitic/hydrothermal mineralisation. Mikulski and Stein (2007) provided Re/Os isotopic ages of molybdenite from the Karkonosze granite samples collected in the Łomnica Górna (326 ± 1 Ma) and the Michałowice (315 ± 1 Ma) quarries. Recently, Mikulski and Stein (2011) provided two Re/Os ages of

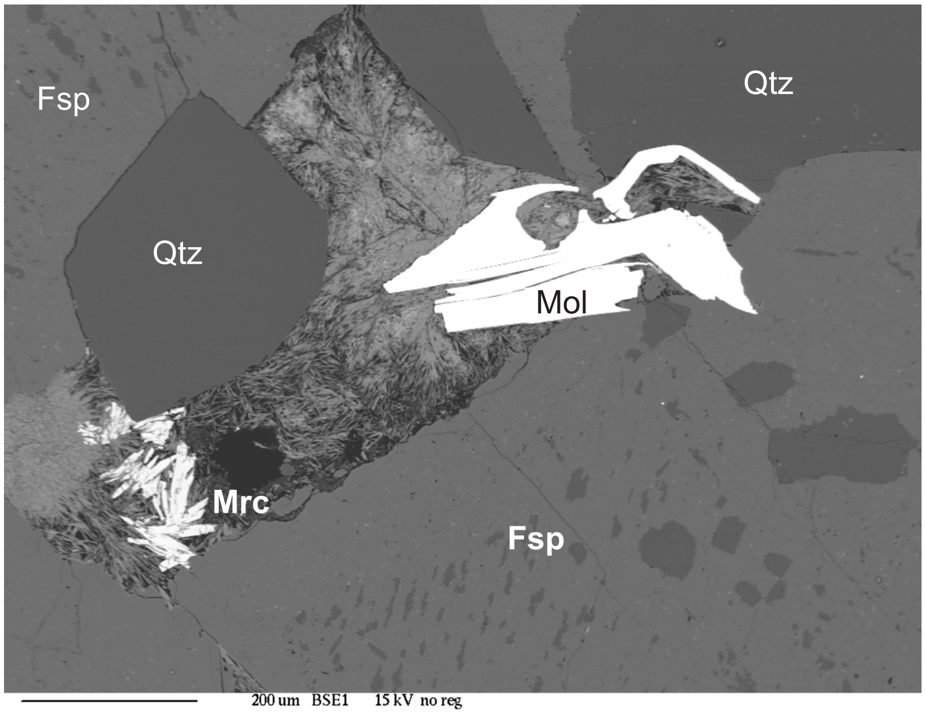

Fig. 2. Molybdenite crystals (Mol) intergrown with micas(?) at the contact of quartz (Qtz) and feldspar (Fsp)

At the bottom left radial marcasite (Mrc) crystals are visible, Szklarska Poręba Huta Quarry, sample SZP-4b, BSE image

vein-type and disseminated molybdenite from the Szklarska Poręba Huta Quarry at 310 ± 1 Ma and 323 ± 1 Ma, respectively.

GENERAL REMARKS

Molybdenite is naturally enriched in rhenium (Re) and contains insignificant amounts of Os, meaning that effectively all Os is derived from the decay of Re. To measure the abundance of Re and Os in molybdenite samples by isotope dilution, a mixed double spike solution was used containing isotopically enriched ^{185}Re together with isotopically enriched ^{188}Os and ^{190}Os (Selby and Creaser, 2004).

The ^{185}Re abundance in the tracer solution is calibrated directly against a gravimetric Re standard solution made from 99.999% Re metal and shows a reproducibility of better than ±0.20% 1σ (n = 6). The Os abundance in the tracer solution is calibrated directly against a gravimetric Os standard solution of known isotopic (Selby and Creaser, 2004) composition. The abundance of ^{187}Os in molybdenite is calculated by measuring the Os abundance after equilibrating ^{187}Os in molybdenite and the double ^{188}Os and ^{190}Os in the tracer solution. In this way, the isotopic analysis of Os can be corrected for instrumental fractionation during analysis, and potential "common" Os in the molybdenite quantified.

MATERIAL

For age determinations three samples were selected, labeled Mo-1, Mo-2 and Mo-3. All contained exposed grains of molybdenite in altered granite from the Szklarska Poręba Huta Quarry.

METHODS

Microprobe analyses of the molybdenite and of other ore minerals were carried on at the Joint-Institute Analytical Complex for Minerals and Synthetic Substances, Faculty of Geology, University of Warsaw under standard analytical conditions (see e.g., Mochnacka *et al*., 2008).

Re/Os isotopic age determinations were carried out at the Geochronology and Radiogenic Isotopic Analyses Laboratory, Geospec Consultants LTD., Edmonton, Canada.

The molybdenite was separated by full mineral separation procedures of crushing and milling, followed by magnetic and gravity concentration techniques (see Selby and Creaser, 2004).

The *Carius-tube* method was used for the dissolution of molybdenite and equilibration of sample, tracer Re and Os. Molybdenite samples were dissolved and equilibrated with a known amount of tracer in reverse aqua regia (2:1 16N HNO_3

Table 1

Re-Os ages of molybdenites from the Szklarska Poręba Huta Quarry

Sample ID	Re [ppm]	^{187}Re [ppm]	^{187}Os [ppb]	Age [Ma]	2σ uncertainty [Ma]
Molybdenite, Szklarska Poręba Huta Quarry (30 mg)	0.122 ± 0.001	0.0764 ± 0.0004	0.392 ± 0.002	307.4	±2.4
Molybdenite, Szklarska Poręba Huta Quarry (repeat) (24 mg)	0.125 ± 0.001	0.0788 ± 0.0008	0.407 ± 0.002	308.9	±2.1

Decay constant used: ^{187}Re = 1.666 × 10^{-11} yr^{-1} without uncertainty (Smoliar *et al*., 1996); blanks: Re – <2 pg, Os – 0.5 pg; initial ^{187}Os/^{188}Os ratio = 0.12

and 12N HCl, 3 ml) at 240°C for 24 h then cooled and refrigerated prior to Os and Re separation. Extraction of OsO_4 from the acid-sample mix was achieved using modified solvent extraction and microdistillation techniques. Mo was removed by solvent extraction from the acid-sample mixture after Os separation. Rhenium was then purified by HNO_3 + HCl-based anion exchange chromatography using standard techniques. Total procedural blanks for Re and Os were less than 2 picograms and 0.5 picograms, respectively. These procedural blanks are insignificant in comparison to the Re and Os concentrations in the molybdenite analysed here. The purified Re and Os separates were analysed by *Negative Thermal Ion Mass Spectrometry* (N-TIMS), and the abundances of ^{187}Re and ^{187}Os were calculated. A trial analysis established the Re abundance of the molybdenite concentrate at ~0.13 ppm, followed by the full Re-Os analyses. A second full Re-Os analysis was completed several weeks later than the first. The results of these analyses are presented below in Table 1.

The Chinese molybdenite powder HLP-5, used as an in-house "control" in the molybdenite laboratory, AIRIE, Colorado State University, was used as an external control of sample absolute age and reproducibility for this work. For this "control sample" using the *Carius-tube* technique with mixed-double spike described here, we obtained an average Re-Os age of 220.0 ± 1.0 Ma (2σ). This age is nominally younger than the long-term average age of 220.52 ± 0.24 Ma (2σ) reported by Selby and Creaser (2004) using the "Normal Addition" method for Os.

RESULTS AND DISCUSSION

The ore mineral assemblage found in aplogranite samples from the Szklarska Poręba Huta Quarry does not differ from that previously described from that site (see references above) neither in the index of minerals nor in their structures and textures. However, our molybdenite specimens represent the disseminated variety, not molybdenite hosted in quartz veins (see e.g., Kozłowski *et al*., 1975; Mikulski and Stein, 2011).

Our Re/Os isotopic age determinations revealed two consistent values for molybdenite disseminated in the Szklarska Poręba Huta aplogranite: 307 ± 2 Ma and 309 ± 2 Ma (Table 1). These values correspond best to leucogranite (aplogranite?) from the same quarry (310 ± 5 Ma; Mierzejewski *et al*., 1994). Simultaneously, our results are only slightly older than a recently published, refined ^{206}Pb/^{238}U age of untreated zircons from the Szklarska Poręba Huta porphyritic granite (306 ± 4 Ma; Kryza *et al*., 2012), and are consistent also with previously determined ages of equigranular granite (309 ± 3 Ma, Duthou *et al*., 1991 and 310 ± 14 Ma, Pin *et al.,* 1987) and a porphyritic granite enclave from Mały Staw (309 ± 17 Ma, Mierzejewski *et al*., 1994). In general, our results are consistent with the younger magmatic event in the Sudetes distinguished by Mazur *et al*. (2007). It suggests pene-contemporaneous crystallisation of disseminated molybdenite and granite rock-forming minerals in, at least, a part of the intrusion.

Considering the age determinations of the in-granite ore mineralisation, our molybdenite ages are consistent with the younger age (310 ± 1 Ma) of two different generations of molybdenite published by Mikulski and Stein (2011) for samples from the Szklarska Poręba Huta Quarry. This mineralisation may be related, to the younger stage of post-magmatic, pneumatolitic/hydrothermal activity distinguished by these authors (*ca*. 312 Ma, Mikulski and Stein, 2007). However, our molybdenite represents the disseminated type for which Mikulski and Stein (2011) determined a much older age (323 ± 1 Ma). Such time relationships may suggest diverse, long-lasting activity of pneumatolitic and hydrothermal systems of the Karkonosze granite, extending at least from 326 ± 1 Ma to 307 ± 2 Ma. This interpretation is supported by the multi-pulse character of the intrusion (Mierzejewski, 2007), which renewed the thermal capacity of the pluton and, thus, extended the driving force of the surrounding hydrothermal system. Robb (2005) proposed the lifetime of hydrothermal fluid circulation to be about 10^5 to 10^6 years for small intrusions (1–2 km wide) and Misra (2000) estimated a much shorter cooling time of "reasonably sized" intrusions (<10 km) to be some 10^4 years Obviously, as the Karkonosze intrusion is much larger, the hypothetical lifetime of its hydrothermal system was much longer.

CONCLUSIONS

1. Molybdenite from the Szklarska Poręba Huta Quarry disseminated in aplogranite was dated at 307 ± 2 Ma and 309 ± 2 Ma with the Re-Os isotopic method.

Table 2

List of isotope ages revealed by various methods applied to the Karkonosze granitoids (Polish part of the Karkonosze intrusion) and to related ore mineralisation

Rock/locality	Method	Granite age [Ma]	References	Mineralisation age [Ma]	References
Aplogranite, Szklarska Poręba Huta	U, Th, Pb CHIME monazite	271 ± 20	Mikulski *et al.* (2004)		
Karkonosze granite Szklarska Poręba	Rb/Sr K/Ar	292 304	Przewłocki *et al.* (1962)		
Aplogranite Szklarska Poręba Huta	U, Th, Pb CHIME xenotime	*ca*. 294	Mikulski *et al.* (2004)		
Aplites	K/Ar biotite	294–304	Depciuch and Lis (1971)		
Equicrystalline granite	K/Ar biotite	294–306	Depciuch and Lis (1971)		
Porphyritic, medium-crystalline granite	K/Ar biotite	296–307	Depciuch and Lis (1971)		
Karkonosze granite, Borów	K/Ar whole rock	299 ± 27	Borucki (1966)		
Karkonosze granite, Jakuszyce	U/Pb microprobe			299.8 bröggerite	Kucha *et al.* (1986)
Equigranular granite	U/Pb SHRIMP zircon	302 ± 6	Kusiak *et al*. (2008)		
Granophyric granite	K/Ar biotites	302–305	Depciuch and Lis (1971)		
Porphyritic, coarse crystalline granite	K/Ar biotites	303	Depciuch and Lis (1971)		
Microgranular enclave in equigranular granite	Pb/Pb, U/Pb SHRIMP zircon	304 ± 3	Kusiak *et al*. (2009)		
Porphyritic monzogranite	Pb/Pb evaporation, U/Pb, zircon	304 ± 14	Kröner *et al.* (1994)		
Porphyritic granite, Szklarska Poręba Huta	U/Pb SIMS, untreated zircons	306 ± 4	Kryza *et al*. (2012)		
Aplogranite, Szklarska Poręba Huta	Re/Os			307 ± 2 and 309 ± 2 molybdenite	this paper
"Ridge", equigranular granite	Rb/Sr whole rock isochrone	309 ± 3	Duthou *et al*. (1991)		
Porphyritic granite enclave Mały Staw	Rb/Sr whole rock	309 ± 17	Mierzejewski *et al.* (1994)		
Aplogranite, Szklarska Poręba Huta	Re/Os			310 ± 1 molybdenite	Mikulski and Stein (2011)
Leucogranite Szklarska Poręba Huta	Rb/Sr whole rock	310 ± 5	Mierzejewski *et al.* (1994)		
Ridge, equigranular	Rb/Sr whole-rock isochrone	310 ± 14	Pin *et al.* (1987)		
Izera village granite	K/Ar whole rock	310 ± 28	Borucki (1966)		
Microgranodiorite Karpacz–Janowice Wielkie dyke swarm	U/Pb SHRIMP zircon	313 ± 3–318 ± 3	Awdankiewicz *et al.* (2010)		
Porphyritic granite from NE part of intrusion	U/Pb SHRIMP zircon	314 ± 3–318 ± 4	Machowiak and Armstrong (2007)		
Equigranular granite	U/Pb SHRIMP zircon	314 ± 5	Kusiak *et al.* (2008)		
Michałowice granite	Re/Os			315 ± 1 molybdenite	Mikulski and Stein (2007)
Equigranular granite	Ar/Ar muscovite	315 ± 2	Marheine *et al.* (2002)		
Porphyritic granite (Liberec type)	Ar/Ar biotite	320 ± 2	Marheine *et al.* (2002)		

Tab. 1 cont.

Rock/locality	Method	Granite age [Ma]	References	Mineralisation age [Ma]	References
Porphyritic granite, Szklarska Poręba Huta	U/Pb SIMS, chemically abraded zircons	322 ± 3	Kryza *et al.* (2012)		
Mały Staw granite	K/Ar whole rock	323 ± 29	Borucki (1966)		
Aplogranite, Szklarska Poręba Huta	Re/Os			323 ± 1 molybdenite	Mikulski and Stein (2011)
Porphyritic granite, Łomnica Górna	Re/Os		326 ± 1 molybdenite	Mikulski and Stein (2007)	
Karkonosze granite	fission tracks, zircons	326 ± 32	Jarmołowicz-Szulc (1986)		
"Central", porphyritic granite Michałowice	Rb/Sr whole-rock isochrone	328 ± 12	Pin *et al.* (1987)		

2. Both ages are consistent with the newest ages provided by Mikulski and Stein (2012) for molybdenite from veins from the same locality and correspond well to a recently published, refined $^{206}Pb/^{238}U$ age of untreated zircons from the Szklarska Poręba Huta porphyritic granite (see Kryza *et al*., 2012) and with some earlier dates. Moreover, our results are consistent with the younger magmatic event in the Sudetes distinguished by Mazur *et al*. (2007).

3. Our results suggest penecontemporaneous crystallisation of a part of the molybdenite and the host aplogranite.

4. Our results are much older than the suggested age of pneumatolitic/hydrothermal Sn-W-Mo-Bi mineralisation well-known from the Szklarska Poręba Huta Quarry.

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