NEW K-Ar COOLING AGES OF GRANITOIDS FROM THE STRZEGOM-SOBÓTKA MASSIF, SW POLAND

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Abstract: The Strzegom-Sobótka Variscan Massif (Fore-Sudetic Block, NE Bohemian Massif) consists of various post-kinematic Variscan granitoids emplaced into Palaeozoic low grade metamorphic rocks. Biotite from five samples representing the hornblende-biotite monzogranite, biotite monzogranite and biotite granodiorite has been dated using the K-Ar method for two size-fractions (0.25-0.35 and 0.35-0.5 mm). Finer fractions show more intense chloritization and therefore the results for them were rejected in further discussion. Coarser fractions with higher potassium content were accepted as yielding a maximum estimate approaching the true K-Ar biotite cooling ages. The results are clustered in two groups. The older age group (308.8±4.6 Ma and 305.5±4.3 Ma) comprises results obtained from the biotite granodiorites. They are generally consistent with the zircon crystallization age of 308.4±1.7 Ma reported by Turniak *et al.* (2005) and imply rapid cooling of the biotite granodiorite from magmatic temperatures down to the closure temperature of K-Ar isotopic system in biotite. The younger group is defined by 291.0±4.4 Ma and 298.7±5.2 Ma ages for the hornblende-biotite monzogranite and 294.2±4.3 Ma age for the biotite monzogranite. In the absence of precise U-Pb or Pb-Pb zircon data further geochronological studies are needed to decipher precisely the cooling history of the monzogranites.

Keywords: Variscan granitoids, biotite, cooling age, K-Ar dating, Strzegom-Sobótka Massif

1. INTRODUCTION

The Strzegom-Sobótka Massif (SSM), composed of various Variscan post-kinematic granitoids, is situated in the central part of the Fore-Sudetic block (NE Bohemian Massif; **Fig. 1**). Four main rock varieties exposed in SSM – hornblende-biotite monzogranite and biotite monzogranite (in the west of SSM), two-mica monzogranite (in the centre) and biotite granodiorite (in the east) are interpreted by Puziewicz (1985) as separate intrusions. Details on their petrography and geochemistry are given by Kural and Morawski (1968), Majerowicz (1972) and Puziewicz (1990).

Based on Rb-Sr investigations, the massif was shown to be a complex body with at least two stages of development: intrusion of the two-mica granite (324±7 Ma) followed by emplacement of the biotite granodiorite and monzogranites dated at about 280 Ma (Pin *et al.*, 1989). The latter age was also confirmed by Domańska-Siuda *et al.* (2004) for the hornblende-biotite monzogranite. Ages of 309.1±0.8 Ma and 306.4±0.8 Ma were obtained from two-mica granite using ID-TIMS method on monazite and xenotime, respectively (Turniak and Bröcker, 2002). The emplacement age of the biotite granodiorite was established at 308.4±1.7 Ma by Turniak *et al.* (2005) on the basis of Pb-evaporation zircon datings. The hornblende-biotite and biotite monzogranites yielded a spectrum of zircon $^{207}Pb/^{206}Pb$ ages: 299±8 Ma (Paszowice), 300±11 Ma (Morów), 302.7±3.4 Ma (Strzegom), 306.6±4.2 Ma (Kostrza) and 307.2±4.4 Ma (Graniczna). Comparable results of 304-309 Ma are reported from Re-Os studies on molybdenite from quartz veins cross-cutting the hornblende-biotite granite exposed in the Paszowice quarry (Mikulski and Stein, 2005).
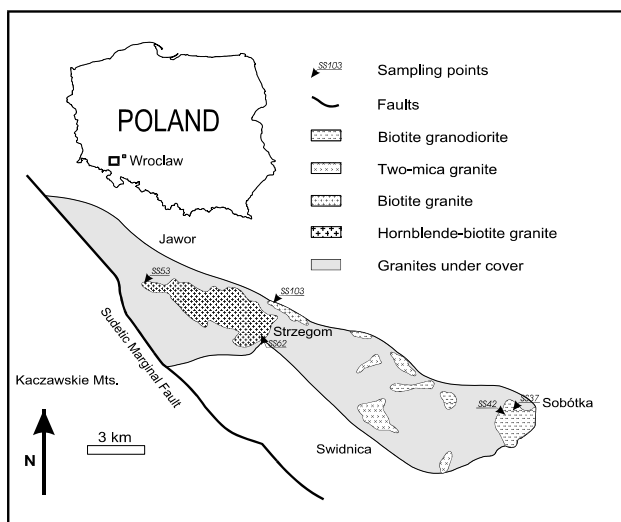
Fig. 1. *Geological sketch map of the Strzegom-Sobótka Massif with sampling points (based on Majerowicz, 1972).*

Although the ages of individual intrusions comprised in SSM seem to be well estimated, the cooling history of the massif remains poorly constrained. Early K-Ar investigations on biotites extracted from the granitoids revealed a wide age range from 224 to 318 Ma (Borucki, 1966). Depciuch (1971), using the same isotopic system and mineral, obtained about 266 Ma.

In this study, we report new results of biotite K-Ar dating for 5 samples representing the hornblende-biotite monzogranite, biotite monzogranite and biotite granodiorite of SSM. Our study provides first isotopic evidence for different cooling histories of the granitoids exposed in the western and eastern parts of the Strzegom-Sobótka Massif.

2. ANALYTICAL PROCEDURES

The rock samples were crushed using a jaw crusher and sieved into 0.25-0.35 and 0.35-0.5 mm size-fractions. The material was then washed in water and dried in an oven in temperature at about 50°C. Magnetic separation, paper-shaking and handpicking under a binocular microscope produced about 300 mg biotite separates which were subsequently cleaned in distilled water in an ultrasonic bath.

The potassium concentrations in the biotite samples were determined by AAS using a Pye Unicam SP9 spectrophotometer at the Institute of Geological Sciences, University of Wrocław. Argon was analysed using a modified mass spectrometer MS-10 at the Institute of Physics of the Maria Curie-Skłodowska University in Lublin. A low blank ultra-high-vacuum line described by S. Halas (2007) was used for argon extraction and purification. For K-Ar age calculation the constants λ_e=0.581*10^{-10} 1/a, λ_β=4.962*10^{-10} 1/a, $^{40}K/K$=0.01167% (Steiger and Jaeger, 1977) were adopted. Uncertainty in the K-Ar age was calculated from the error propagation formula.

Chemical composition of the biotite was investigated by a CAMECA SX-100 electron-microprobe (WDS mode) in the Electron Microprobe Laboratory at the In-

ter-Institute Microanalytical Complex for Minerals and Synthetic Substances, Warsaw University. Measurement conditions were as follows: 10 nA beam current, accelerating voltage 15 kV and counting time 20 s. The PAP procedure was employed for matrix correction.

XRD analyses were carried out for SS62 sample to compare semi-quantitively chlorite admixtures in the biotite fractions analysed. For each fraction an oriented mount with uniform thickness was prepared. X-ray diffraction patterns were obtained at the Institute of Geological Sciences, University of Wrocław, using a Siemens 5005 instrument operating at 30 kV, 25 mA and CoK$_{\alpha 1}$ Fe-filtered radiation. Scans were made from 4° to 75° 2Θ with a step of 0,02° 2Θ and a step-time of 1 s.

3. BIOTITE DESCRIPTION

Ten biotite concentrates extracted from 5 granitoid samples were analysed using the K-Ar isotope method. They represent three of four main lithological varieties of the Strzegom-Sobótka Massif: biotite granodiorite (SS37 – Strzeblów, SS42 - Chwałków), hornblende-biotite monzogranite (SS53 – Paszowice, SS62 – Strzegom, "Żbik" quarry) and biotite monzogranite (SS103 – "Graniczna I" quarry). The sampling sites are indicated in **Fig. 1**.

The biotite forms mostly subhedral to anhedral plates up to 2 mm long, sometimes grouped into aggregates. Grain boundaries are often ragged or lobate (**Fig. 2**). Pleochroic colour for γ direction change gradually from cinnamon-brown (the biotite granodiorite) through red-brown (the biotite monzogranite) to dark-brown, almost black (the hornblende-biotite monzogranite). Heavy minerals – apatite, ilmenite, zircon and allanite are common inclusions in the mica from hornblende-biotite monzogranite. Additionally monazite an xenotime are found in the biotite from the granodiorite and biotite monzogranite. The biotite is partly replaced by chlorite – mainly along cleavage planes, fractures and marginal parts of grains (**Fig. 2**).

The biotite composition changes from biotite granodiorite through biotite monzogranite to hornblende-biotite monzogranite manifested mainly by the decrease of Al and Si contents coupled with the increase of $Fe^{2+}/(Fe^{2+}+Mg)$ ratio (**Table 1**). It corroborates findings reported previously by Wichrowska (1974 and 1977) and Puziewicz (1994). Biotite from the biotite granodiorite shows the highest concentrations of Al, occupying both tetrahedral and octahedral sites, whereas the mica from the hornblende-biotite monzogranite contains less Al than necessary to substitute for Si^{4+} implying that possibly also Fe^{3+} is confined in the tetrahedral sites. The $Fe^{2+}/(Fe^{2+}+Mg)$ ratio is 0.59-0.63 for the biotite granodiorite, 0.68-0.71 for the biotite monzogranite and 0.78-0.83 for the hornblende-biotite monzogranite. Potassium content in the pristine biotite is highest in the biotite granodiorite (SS37: 7.92(12)wt.%, SS42: 7.90(15)wt.%). Smaller concentrations were shown by samples representing the hornblende-biotite monzogranite (SS62: 7.65(14)wt. %, SS53: 7.73(8)wt.%) and biotite monzogranite (SS103: 7.60(15)wt.%). It is worth to note that

Table 1. *Chemical composition of biotites from the Strzegom-Sobótka Massif.*

Representative chemical analyses of biotite from dated samples

	Biotite granodiorite						Biotite monzogranite			Hornblende-biotite monzogranite					
	SS37			SS42			SS103			SS62			SS53		
	#2	#4	#10	#18	#23	#30	#2	#5	#9	#1	#9	#11	#3	#9	#14
SiO$_2$	35.25	35.56	35.31	35.23	35.31	35.24	34.31	34.71	35.32	34.94	34.13	34.01	34.42	34.40	34.18
TiO$_2$	3.70	3.16	3.08	3.99	3.68	3.33	4.06	4.40	4.14	2.99	4.37	3.90	3.50	3.79	3.72
Al$_2$O$_3$	16.55	16.38	16.64	16.79	15.65	15.85	14.56	14.50	14.00	12.70	12.65	12.69	13.54	13.08	13.09
Cr$_2$O$_3$	0.06	0.00	0.00	0.00	0.00	0.00	0.05	0.00	0.04	0.00	0.00	0.00	0.00	0.00	0.00
FeO	22.58	23.13	23.28	21.72	22.81	23.04	26.35	25.97	26.18	31.81	31.82	32.68	31.12	31.66	31.87
MnO	0.64	0.71	0.69	0.49	0.35	0.49	0.82	0.75	0.64	0.50	0.57	0.47	1.00	0.67	1.01
MgO	7.22	7.39	7.41	7.79	7.82	7.90	6.12	6.18	6.28	3.85	3.31	3.39	3.62	3.69	3.42
CaO	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Na$_2$O	0.15	0.06	0.13	0.14	0.12	0.12	0.12	0.10	0.12	0.04	0.04	0.05	0.02	0.01	0.06
K$_2$O	9.62	9.53	9.34	9.42	9.44	9.82	8.97	9.19	8.90	9.30	9.09	9.13	9.39	9.28	9.16
BaO	0.19	0.26	0.24	0.71	0.40	0.00	0.71	0.31	0.28	0.00	0.00	0.00	0.00	0.00	0.00
Total	95.94	96.16	96.12	96.26	95.58	95.78	96.07	96.12	95.90	96.12	95.97	96.32	96.60	96.59	96.49

Cations calculated basing on 22 oxygens; Fe3+/Fe2+ after Puziewicz (1994)

	SS37 (18 analyses)			SS42 (7 analyses)			SS103 (15 analyses)			SS62 (10 analyses)			SS53 (10 analyses)		
	min	max	mean	min	max	mean	min	max	mean	min	Max	mean	min	max	mean
Si	5.40	5.54	**5.47(4)**	5.46	5.48	**5.47(1)**	5.32	5.48	**5.41(5)**	5.25	5.42	**5.31(5)**	5.29	5.43	**5.35(5)**
Al^{IV}	2.46	2.60	**2.53(4)**	2.52	2.54	**2.53(1)**	2.52	2.66	**2.57(4)**	2.28	2.41	**2.33(3)**	2.38	2.57	**2.46(6)**
Fe^{IV}	0.00	0.00	**0.00(0)**	0.00	0.00	**0.00(0)**	0.00	0.09	**0.02(3)**	0.26	0.47	**0.36(7)**	0.03	0.33	**0.20(10)**
Al^{VI}	0.40	0.65	**0.52(7)**	0.36	0.61	**0.48(10)**	0.00	0.08	**0.03(3)**	0.00	0.00	**0.00(0)**	0.00	0.00	**0.00(0)**
Ti	0.34	0.44	**0.39(3)**	0.39	0.46	**0.43(3)**	0.42	0.51	**0.47(3)**	0.35	0.55	**0.46(6)**	0.26	0.44	**0.41(5)**
Cr	0.00	0.01	**0.00(0)**	0.00	0.01	**0.00(0)**	0.00	0.01	**0.00(0)**	0.00	0.01	**0.00(0)**	0.00	0.01	**0.00(0)**
Fe^{3+}	0.20	0.21	**0.21(0)**	0.20	0.22	**0.20(1)**	0.02	0.10	**0.08(3)**	0.04	0.23	**0.14(7)**	0.17	0.44	**0.29(9)**
Fe^{2+}	2.62	2.81	**2.73(5)**	2.62	2.87	**2.71(9)**	3.24	3.38	**3.31(4)**	3.63	3.73	**3.67(3)**	3.45	3.70	**3.56(8)**
Mn	0.07	0.10	**0.09(1)**	0.04	0.06	**0.06(1)**	0.07	0.11	**0.09(1)**	0.06	0.08	**0.07(1)**	0.08	0.13	**0.11(2)**
Mg	1.66	1.81	**1.72(4)**	1.63	1.83	**1.76(6)**	1.38	1.51	**1.44(3)**	0.74	0.89	**0.79(5)**	0.79	0.99	**0.85(6)**
Σ	5.53	5.82	**5.65(7)**	5.57	5.71	**5.64(4)**	5.34	5.52	**5.42(5)**	5.07	5.21	**5.13(4)**	5.17	5.26	**5.22(3)**
Na	0.02	0.04	**0.03(1)**	0.02	0.04	**0.03(0)**	0.01	0.04	**0.03(1)**	0.01	0.02	**0.01(0)**	0.00	0.02	**0.01(1)**
K	1.85	1.94	**1.89(3)**	1.83	1.94	**1.88(4)**	1.76	1.87	**1.81(3)**	1.77	1.88	**1.82(3)**	1.81	1.87	**1.84(2)**
Ba	0.00	0.03	**0.01(1)**	0.00	0.04	**0.02(2)**	0.00	0.04	**0.02(1)**	0.00	0.02	**0.00(1)**	0.00	0.00	**0.00(0)**
Σ	1.90	1.98	**1.94(1)**	1.90	1.98	**1.94(2)**	1.81	1.91	**1.86(3)**	1.78	1.90	**1.84(3)**	1.83	1.88	**1.85(2)**
Xfe	0.60	0.63	**0.61(1)**	0.59	0.62	**0.61(1)**	0.68	0.71	**0.70(1)**	0.80	0.83	**0.82(1)**	0.78	0.82	**0.81(1)**
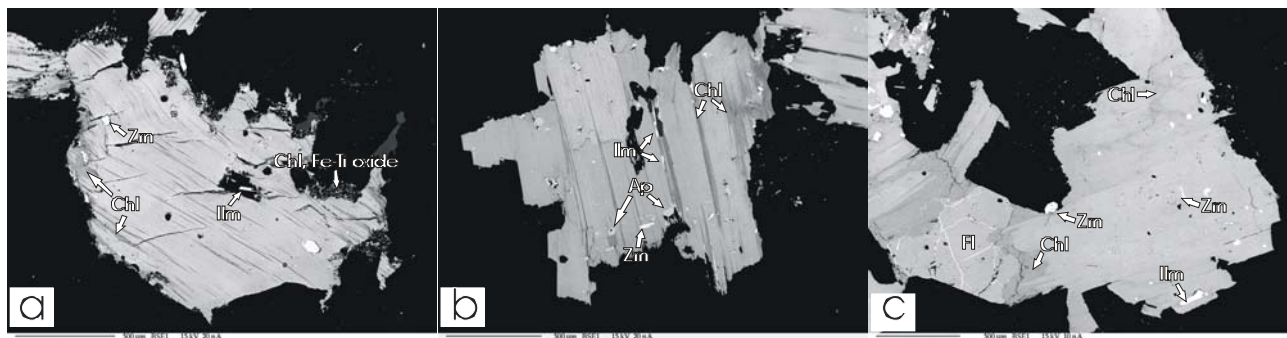
Fig. 2. *Back-scattered electron images of biotites from the Strzegom-Sobótka Massif. a) Deformed biotite crystal from the biotite granodiorite (SS42) partly replaced by chlorite. b) Biotite plate from the biotite monzogranite with chlorite interlayers developed along biotite cleavage. c) Aggregate of biotite individuals from the hornblende-biotite granite (SS62). Chlorite distributed along fractures, cleavage and in the vicinity of fluorite grain. Mineral abbreviations after Kretz (1983).*

chemical analyses of the biotite from the granodiorite show slightly elevated Ba concentrations suggesting that Ba^{2+} partly replaces K^+ in the interlayer site.

4. RESULTS AND DISCUSSION

The results of K-Ar isotopic analyses are summarized in **Table 2**. The K-Ar ages decrease while K contents increase consequently with a particle size of the analysed material. On the other hand, $^{40}Ar^*$ concentrations are similar for different fractions of the same sample. The exception is SS42 where the coarser fraction yielded $^{40}Ar^*$ of 4087 pmol/g and the age of 305.5±4.3 Ma, whereas the finer one – 4428 pmol/g and 334.5±5.8 Ma. The latter result is significantly higher than the zircon Pb-Pb age obtained for the biotite granodiorite (308.4±1.7 Ma; Turniak *et al.*, 2005) and therefore probably meaningless geologically. Abundances of potassium measured by AAS for the biotite separates are below EPMA values recorded for pristine biotite. It can be explained assuming that chlorite and/or inclusions of heavy minerals were present in the AAS samples analysed. To verify this, XRD study was carried out for the biotite separates from sample SS62. Both XRD patterns are characterised by intense reflections corresponding to biotite (00l) spacings with weak peaks of chlorite. No other mineral was detected in the sample. The intensity ratios $(003)_{biotite}$ / $(004)_{chlorite}$ of 100/4 and 100/8 obtained for fractions 0.35-0.5 and 0.25-0.35 mm respectively testify that the latter fraction is more contaminated by

chlorite. Many studies have shown that chlorite contamination of biotite separates disturbs K-Ar and ^{40}Ar-^{39}Ar age spectra (e.g. Mitchell and Taka, 1984; Roberts *et al.*, 2001, Di Vincenzo *et al.* 2003). Consequently, since coarser fractions contain least chlorite and highest potassium content, it is most correct to reject K-Ar results based on 0.25-0.35 mm fractions and accept remaining values as yielding a maximum estimate that approach the true K-Ar biotite cooling ages.

The K-Ar cooling ages presented in this paper are clustered in two groups. The older age group (308.8±4.6 Ma and 305.5±4.3 Ma) comprises results obtained from the biotite granodiorites. They are generally consistent within error limits with the zircon crystallisation age reported by Turniak *et al.* (2005) that implies a rapid cooling of the biotite granodiorite exposed in the eastern part of SSM from magmatic temperatures down to the closure temperature of K-Ar isotopic system in biotite (300±50°C; Dodson and McClelland-Brawn, 1985). This is consistent with earlier conclusions based on the outcomes of petrological studies (Kural and Morawski, 1968) according to which the brittle deformed granodiorites from the eastern part of SSM were first to experience consolidation.

The younger group is defined by 291.0±4.4 Ma and 298.7±5.2 Ma ages for the hornblende-biotite monzogranite and 294.2±4.3 Ma age for the biotite monzogranite. A similar age of 290.5±6.6Ma (Rb-Sr, whole-rock) was obtained by Domańska-Siuda (2006) for hornblende-biotite monzogranite and enclosed mafic enclaves. The biotite monzogranite was probably emplaced 307.2±4.4 Ma as indicated by Pb-Pb zircon evaporation data (Turniak *et al.*, 2005). This age is in agreement within error limits with the results obtained for the granodiorites. Maciejewski and Morawski (1975) reported a several cm thick vein of the hornblende-biotite granite cutting across the biotite monzogranite suggesting that the first rock was emplaced later. Following their observation, it seems plausible that the late K-Ar cooling age of the biotite monzogranite can be explained by thermal influence of the adjacent hornblende-biotite monzogranite which records similar cooling ages.

In the absence of exact U-Pb or Pb-Pb zircon data, a minimum emplacement age for the hornblende-biotite monzogranites is probably best constrained by the

Table 2. *K-Ar ages for the granitoids of the Strzegom-Sobótka Massif.*

Sample	Fraction (mm)	K (%)	Error (%)	$^{40}Ar^*$ (pmol/g)	$^{40}Ar^*$ (%)	Age (Ma)	Error (2σ)
SS37	0.25-0.35	7.32	0.56	4337	98.3	312.9	4.7
SS37	0.35-0.5	7.42	0.56	4334	98.4	308.8	4.6
SS42	0.25-0.35	6.95	0.52	4428	97.5	334.5	5.8
SS42	0.35-0.5	7.08	0.50	4087	98.4	305.5	4.3
SS62	0.25-0.35	6.97	0.59	3875	93.1	295.1	5.0
SS62	0.35-0.5	7.20	0.57	3942	97.8	291.0	4.4
SS53	0.25-0.35	6.59	0.62	3800	98.1	305.2	4.9
SS53	0.35-0.5	6.82	0.52	3841	98.1	298.7	5.2
SS103	0.25-0.35	7.42	0.56	4164	85.4	297.7	5.9
SS103	0.35-0.5	7.54	0.54	4178	98.4	294.2	4.3

304-309 Ma molybdenite cooling ages (Mikulski and Stein, 2005). Comparing K-Ar, Re-Os and Pb-Pb ages for the biotite granodiorite and hornblende-biotite monzogranite it might be suggested that the latter was subjected to slower cooling. Further geochronological studies are needed to precisely decipher the cooling history of the monzogranite.

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