

## TITANITE-ILMENITE ASSEMBLAGE IN REDWITZITES OF THE SLAVKOVSKÝ LES MTS. (BOHEMIAN MASSIF, CZECH REPUBLIC)

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### ABSTRACT

Titanium-rich biotite from granodiorite belonging to redwitzite suite of the Slavkovský les Mts. alters to mixture of low-Al titanite, Mn-enriched ilmenite, REE-fluorocarbonate (parisite) and very fine-grained mixture of chlorite and clay minerals. Titanite consists from 1.1 to 2.3 wt. %  $\text{Al}_2\text{O}_3$  and from 0.4 to 1.0 wt. %  $\text{Fe}_2\text{O}_3$ . Titanite shows some  $\text{Al}+\text{Fe}^{3+}$  excess over F with significant amount of  $(\text{Al}+\text{Fe}^{3+})\text{-OH}$  titanite component (4-9 mol. %). Ilmenite contains considerable amount of pyrophanite component (5-8 mol. %). The retrograde assemblage of titanite with considerable amount of  $(\text{Al}+\text{Fe}^{3+})\text{-OH}$  titanite component and presence of ilmenite, together with chlorite, argued for low-temperature breakdown of biotite in reducing conditions.

**KEYWORDS:** Bohemian Massif, Saxothuringian Zone, granodiorite, titanite, ilmenite

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### INTRODUCTION

Titanite and ilmenite are common accessory minerals in igneous rocks, especially in calc-alkalic granites (s.l.) with I-type characteristic (e.g., Lee and Silver, 1964; Piccoli et al., 2000; Helmy et al., 2004; Broska et al., 2007). In hydrothermally altered plutonic rocks secondary titanite can occur together with ilmenite (Piccoli et al., 2000; Ciesielczuk and Janeczek, 2004; Broska et al., 2007).

In the present study distinct intergrowths of titanite and ilmenite are described from a granodiorite dyke of the Krudum granite body in the Horní Slavkov-Krásno ore district. These titanite-ilmenite intergrowths originated from the breakdown of biotite during its hydrothermal alteration. The aim of this paper is to describe and discuss association of titanite and ilmenite, their chemical composition and the conditions controlling hydrothermal alteration of granodiorite.

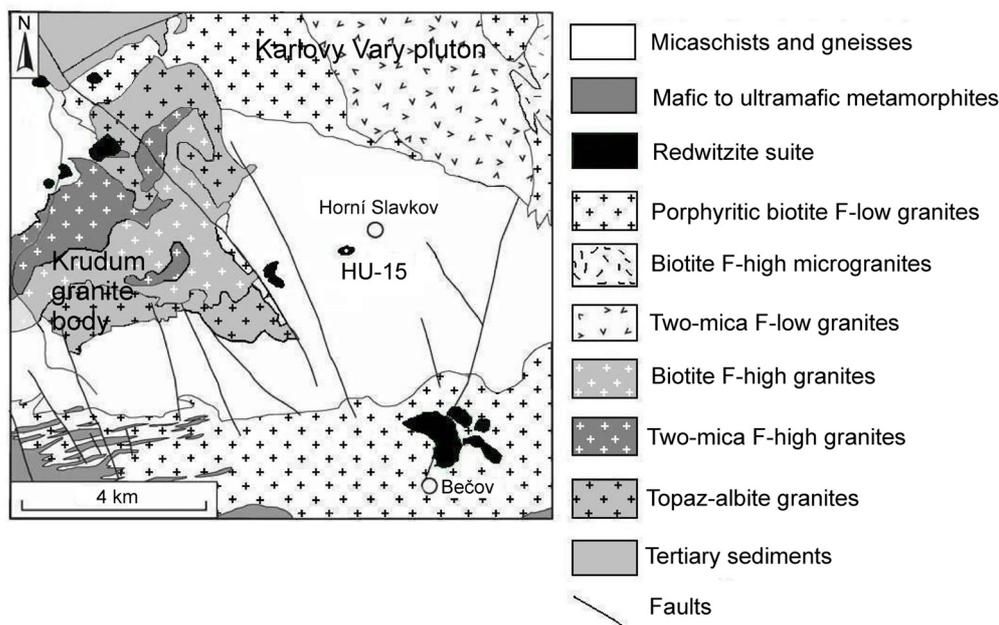
The examined granodiorite is part of granodiorite-tonalite-diorite magmatic suite which is recently considered as a part of the redwitzite suite (Kovářiková et al., 2010). Redwitzites were originally defined in NE Bavaria at the north-western edge of the Bohemian Massif by Willmann (1920). This local name was given to unusual textured magmatic rocks of largely dioritic and granodioritic composition occurred in area of the Marktredwitz. The redwitzites from original occurrence are fine- to medium-grained, generally non-porphyritic tonalites to quartz monzodiorites. They consist amphibole, biotite, plagioclase, minor amounts of quartz, K-feldspar,

pyroxene, titanite and apatite (Troll, 1968; Siebel et al., 1995, 2003; Freiburger et al., 2001). Mafic magmatic rocks similar to redwitzites were described from the Bor pluton by René (2000), from western part of the Krušné hory/Erzgebirge granite batholith by Kovářiková et al. (2007), and more recently also from the Slavkovský les Mts. by Kovářiková et al. (2010).

### ANALYTICAL METHODS

Major element analyses on two whole-rock samples were performed in the laboratory of Institute of Rock Structure and Mechanics of the AS CR, Prague by conventional wet chemical methods. Trace elements were determined by ICP MS at Activation Laboratories Ltd., Lancaster, Canada on a Perkin Elmer Sciex ELAN 6100 ICP mass spectrometer. The decomposition of rock samples for ICP MS analyses involved lithium metaborate/tetraborate fusion.

Analyses of titanite, ilmenite, biotite, plagioclase and chlorite were performed using a CAMECA SX 100 electron microprobe working in WDX mode at the Institute of Geological Sciences, Masaryk University Brno and at Mineralogical Institute, Leibniz University Hannover, Germany. The accelerating voltage and beam currents were 15 kV and 20 nA, respectively, with beam diameters 2  $\mu\text{m}$ . The raw data were corrected using PAP matrix corrections (Pouchou and Pichoir, 1985). The following standards and were used: Al on sanidine, Ca on titanite, Cl on NaCl, Cr on chromite, F on topaz, Fe on andradite, K on orthoclase, Mg on olivine, Mn on

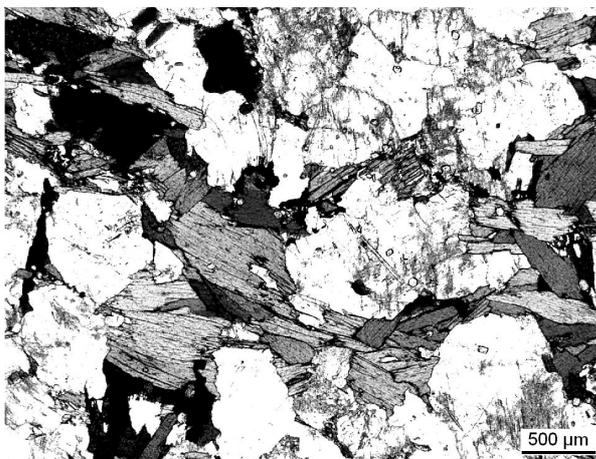


**Fig. 1** Geological sketch map of the Horní Slavkov-Krásno ore district (after Kovářková et al., 2010, slightly modified by author).

spessartine, Na on albite, Nb on columbite, Si on titanite, Ti on titanite and Zn on gahnite. Elements were measured for 20 s at the peak and for 10 s for each background.

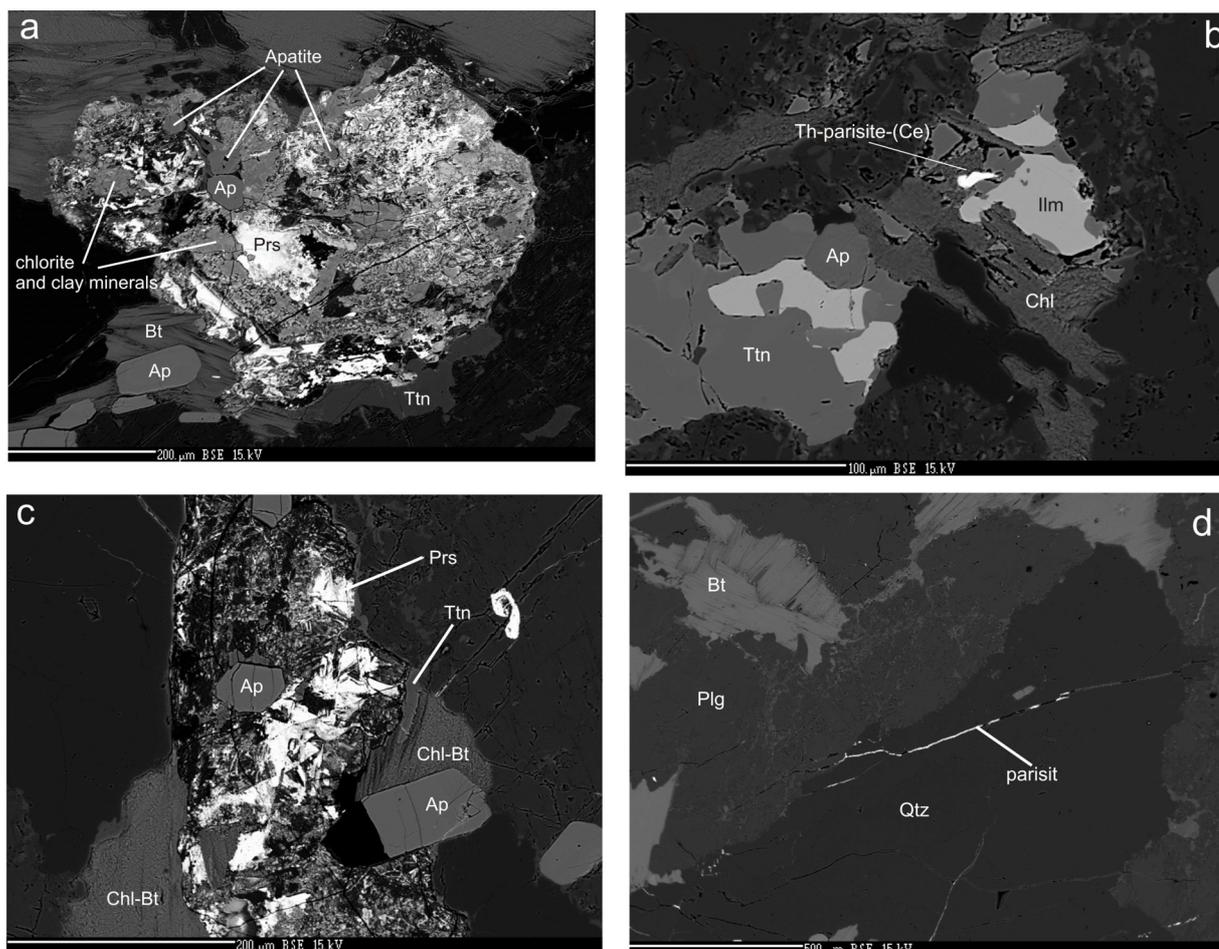
#### INVESTIGATED ROCK MATERIAL

Examined biotite granodiorite of the redwitzite suite occurs as relatively small irregular magmatic bodies and/or as sills in metamorphic rocks of the Slavkov crystalline unit, that forms a mantle of underlying granites of the Krudum granite body (Fig. 1). The medium-grained biotite granodiorite formed relatively thin sills (2-20 m) have a massive structure. Plagioclase (An<sub>27-37</sub>) (30-40 vol. %) prevails over K-feldspar (up to 20 vol. %). Tabular plagioclase shows weak zoning with more sodic rim (oligoclase to andesine) and more calcic cores (basic andesine). Plagioclase is sometimes partly argitilised. Quartz (18-20 vol. %) with undulatory extinction usually fills intergranular spaces. Distinctly pleochroic biotite occurred usually as irregular flakes contains inclusions of accessories (apatite, zircon). Some biotite tables are partly deformed (Fig. 2). Biotite is often chloritised and in quite chloritised biotite was found newly formed titanite, together with ilmenite and rare REE-fluorocarbonate (parisite). Partly chloritised biotite contains intratified laths of chlorite in biotite tables, sometimes together with newly formed titanite. Whole altered biotite, together with argitilised plagioclase forms highly irregular aggregates which contain euhedral to anhedral grains of original magmatic apatite, flakes of chlorite, fine tabular grains of parisite and interstitial, very fine-



**Fig. 2** Microphotography of biotite granodiorite with irregular flakes and partly deformed biotite tables. Sample R-1359, borehole HU-15, Horní Slavkov-Krásno ore district.

grained, mixture of chlorite and clay minerals (kaolinite, smectite?) (Fig. 3a). The more precise identification of individual minerals in interstitial mixture of clay minerals by microprobe analyses is excluded. However, occurrence of newly formed epidote group minerals it can be eliminated. Titanite, together with ilmenite forms in these aggregates large anhedral grains in intergranular spaces (Fig. 3b) and or subhedral laths in partly chloritised biotite. The distinctly more mobile REE fluorocarbonates (parisite) also fills thin irregular fissures in unaltered granodiorite matrix (Fig. 3d).



**Fig. 3** BSE image of titanite-ilmenite-chlorite-parisite assemblage, a - aggregates contained euhedral to anhedral (partly corroded?) grains of apatite (ap), fine parisite laths (prs), anhedral grains of titanite (ttn) and interstitial mixture of very fine-grained chlorite and clay minerals (kaolinite, smectite?), b - titanite (ttn)-ilmenite (ilm)-apatite (ap) assemblage in partly altered biotite granodiorite, c - assemblage of partly chloritized biotite (bt) with small lath of newly formed titanite (ttn), anhedral grain of parisite (prs) and subhedral grains of original magmatic apatite (ap), d - small vein of hydrothermally formed parisite (prs).

The  $\text{SiO}_2$  and  $\text{MgO}$  contents in examined granodiorite are relatively low (57.1-57.4 wt.%  $\text{SiO}_2$ , 2.6 wt.%  $\text{MgO}$ ), however  $\text{TiO}_2$  content is, in comparison with data of Kovářiková et al. (2010), distinctly higher (1.6-1.7 wt. %), (Table 1). Potassium content is also relatively high (3.0-3.3 wt. %  $\text{K}_2\text{O}$ ) and  $\text{K}_2\text{O}/\text{Na}_2\text{O}$  ratio ranges from 0.9 to 1.1, showing that examined granodiorite belongs to high-K magmas (Gill, 1981). Granodiorite shows I-type signature ( $A/\text{CNK} = 1.0-1.1$ ). Sr content, in comparison with values for granodiorite from Bečov area (Kovářiková et al., 2009), is distinctly high (734-764 ppm). However, examined granodiorite shows low content of Cr (21-29 ppm), Ni (16-19 ppm) and V (41-44 ppm). The REE pattern of granodiorite normalized to chondrite shows the predominance of LREE over HREE with absence of obvious Eu anomaly.

#### MINERAL CHEMISTRY

The composition of titanite ranges from 89 to 94 mol. % titanite end-member (Table 2). The Al and Fe contents range from 0.04 to 0.09 atoms per formula unit (a.p.f.u.) and 0.01 to 0.03 a.p.f.u., respectively, together with small amount of F (0.0-0.04 a.p.f.u.). Titanite shows some  $\text{Al}+\text{Fe}^{3+}$  excess over F, which indicates the presence coupled substitution of  $(\text{Al}, \text{Fe}^{3+}) + (\text{F}, \text{OH}) \Leftrightarrow \text{Ti} + \text{O}$  (Fig. 4). Calculation of OH content allows the estimation of the  $(\text{Al}+\text{Fe}^{3+})\text{-OH}$  titanite component, which ranges from 4 to 9 mol. %. The content of  $(\text{Al}+\text{Fe}^{3+})\text{-F}$  component is distinctly lower than the amount of OH-titanite, and ranges from 0 to 4 mol. %.

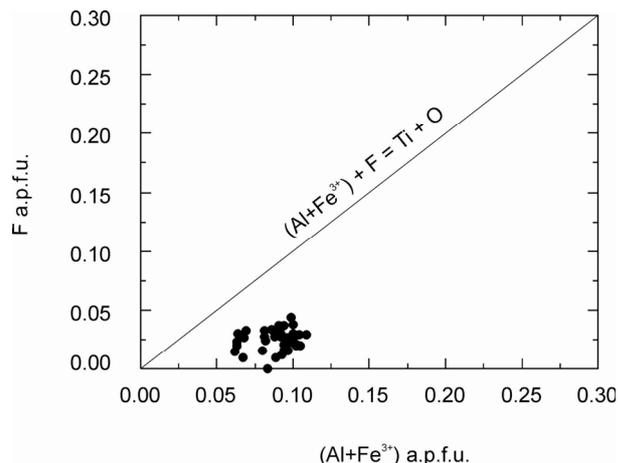
Ilmenite from analysed biotite granodiorite contains  $\text{MnO}$  as the dominant admixture with lesser amount of  $\text{Nb}_2\text{O}_5$ ,  $\text{MgO}$ ,  $\text{ZnO}$ ,  $\text{CaO}$  and  $\text{Al}_2\text{O}_3$

**Table 1** Major element oxides (in wt. %) and trace element composition (in ppm) of biotite granodiorite from the Horní Slavkov-Krásno ore district (borehole HU-15).

Sample	R-1359	R-1360
SiO <sub>2</sub>	57.10	57.41
TiO <sub>2</sub>	1.58	1.65
Al <sub>2</sub> O <sub>3</sub>	17.16	17.11
Fe <sub>2</sub> O <sub>3</sub>	1.88	1.73
FeO	5.02	5.04
MnO	0.09	0.09
MgO	2.61	2.57
CaO	4.13	4.59
Na <sub>2</sub> O	3.10	3.33
K <sub>2</sub> O	3.31	2.99
P <sub>2</sub> O <sub>5</sub>	0.55	0.48
H <sub>2</sub> O <sup>+</sup>	1.86	1.57
H <sub>2</sub> O <sup>-</sup>	0.43	0.31
Total	98.82	98.87
A/CNK	1.06	1.00
Ba	2100	2110
Rb	144	134
Sr	734	764
Y	22	20
Zr	322	430
Nb	17	20
Th	24	27
Ni	19	16
V	44	41
Cr	21	29
Hf	7	10
Cs	14	13
Ta	1.2	1.7
Co	15	13
U	3.1	4.4
La	86	96
Ce	172	187
Pr	17.6	19.2
Nd	71	78.4
Sm	11.1	12.3
Eu	2.4	2.7
Gd	6.5	7.2
Tb	0.83	0.91
Dy	4.1	4.1
Ho	0.67	0.59
Er	1.8	1.6
Tm	0.24	0.2
Yb	1.5	1.2
Lu	0.19	0.16

(Table 3). The examined ilmenite typically has FeTiO<sub>3</sub> amount between 91-94 mol. % and is enriched in the MnTiO<sub>3</sub> (pyrophanite) component (5-8 mol. %). The content of geikielite component (MgTiO<sub>3</sub>) is negligible and hematite component is missing.

For original, unaltered biotite is characteristic distinctly high content of TiO<sub>2</sub> (3.8-4.8 wt. %), partly



**Fig. 4** (Al + Fe<sup>3+</sup>) vs. F plot indicating that the titanite grains from altered biotite granodiorite contain additional OH.

higher Fe/(Fe+Mg) ratio (0.57-0.59) and rather low amount of F (0.09-0.18 wt. % F). Biotite could be consequently classified as Mg-siderophyllite to Fe-phlogopite (Table 4).

#### DISCUSSION

The process of chloritisation of biotite and consecutive origin of secondary titanite is common in granitic rocks and has been studied in detail by numerous authors (e.g., Ferry, 1979; Eggleton and Banfield, 1985; Janeczek, 1994; Piccoli et al., 2000; Ciesielczuk and Janeczek, 2004; Broska et al., 2007). In examined magmatic rocks of the redwitzite suite from the Horní Slavkov-Krásno ore district this process affects biotite even in nearly unaltered biotite granodiorite.

Origin of titanite, partly associated with ilmenite by breakdown of biotite could be described by following reaction:



Often is this alteration accompanied by sericitization and argilization of plagioclase. Following argilization of plagioclase occurs also in examined granodiorite. Association secondary titanite with ilmenite argued for alteration in reducing conditions, since origin of ilmenite would involve Fe<sup>2+</sup> as opposed to Fe<sup>3+</sup>. Model reactions which could be induced from schematic Schreinemakers diagrams (e.g., Harlov et al., 2006; Broska et al., 2007) show that ilmenite formation is in these cases always associated with hydration of the primary mineral assemblage.

During hydrothermal alteration originated titanite has a wide compositional range of (Al+Fe<sup>3+</sup>) = 0.05-0.55 a.p.f.u., together with highly variable content of F (Enami et al., 1993). Some of these

**Table 2** Representative analyses of titanite (wt. %).

Sample	R1359-8	R1359-21	R1359-121	R1359-122	R1359-124	R1359-128
SiO <sub>2</sub>	30.42	30.07	30.63	30.78	30.56	30.63
TiO <sub>2</sub>	38.93	37.67	37.61	38.06	38.00	37.78
Nb <sub>2</sub> O <sub>5</sub>	0.30	0.08	0.00	0.00	0.00	0.00
Al <sub>2</sub> O <sub>3</sub>	1.14	1.78	2.23	1.66	1.88	2.13
Fe <sub>2</sub> O <sub>3</sub>	0.98	0.64	0.70	0.79	0.70	0.93
MnO	0.14	0.08	0.08	0.08	0.02	0.08
CaO	28.86	29.18	28.96	28.69	29.05	29.30
Na <sub>2</sub> O	0.01	0.00	0.02	0.01	0.04	0.00
F	0.26	0.32	0.19	0.00	0.10	0.19
O=F	0.08	0.10	0.06	0.00	0.03	0.06
Total	100.96	99.72	100.36	100.07	100.32	100.98
Si	1.000	1.000	1.000	1.000	1.000	1.000
Ti	0.963	0.942	0.924	0.930	0.935	0.928
Nb	0.004	0.001	0.000	0.000	0.000	0.000
Al	0.044	0.070	0.085	0.064	0.072	0.082
Fe <sup>3+</sup>	0.024	0.016	0.017	0.019	0.017	0.023
Mn	0.004	0.002	0.002	0.002	0.001	0.002
Ca	1.016	1.040	1.013	0.999	1.018	1.025
Na	0.001	0.000	0.001	0.001	0.003	0.000
F	0.027	0.034	0.020	0.000	0.010	0.020
OH	0.041	0.052	0.082	0.083	0.079	0.085
Σ cations	3.056	3.071	3.042	3.015	3.046	3.060
X(Ttn)	0.934	0.916	0.901	0.918	0.913	0.898
X(Al,Fe <sup>3+</sup> -F)	0.026	0.033	0.019	0.000	0.010	0.019
X(Al,Fe <sup>3+</sup> -OH)	0.040	0.051	0.080	0.082	0.077	0.083

Formulae calculated on the basis of 1 Si; OH – Al+Fe<sup>3+</sup>-F; Ttn: titanite, Al,Fe<sup>3+</sup>-F: Al,Fe<sup>3+</sup>-F titanite; Al,Fe<sup>3+</sup>-OH: Al,Fe<sup>3+</sup>-OH titanite.

secondary titanites are enriched in Al (Janeczek, 1994; Ciesielczuk and Janeczek, 2004; Broska et al., 2007). In secondary titanite from altered granites of the Tribeč Mts. (Slovakia) (Broska et al., 2007) reaches  $X_{Al}$  ratio values of 0.07-0.09, but in secondary titanite from granites of the Strzelin granite (Poland) (Janeczek, 1994; Ciesielczuk and Janeczek, 2004) ranges  $X_{Al}$  from 0.18 to 0.82. Similar Al-enriched secondary titanite was found by Enami et al. (1993) in altered hornblende-biotite granite from Japan ( $X_{Al}$  = 0.48-0.53). However, in examined titanite from the Horní Slavkov-Krásno ore district ranges this ratio only from 0.04 to 0.09. Analysed titanite from the Horní Slavkov – Krásno ore district shows also relatively low F content, whose source was highly probable fluorine from altered biotite. For original magmatic biotite from examined biotite granodiorite is characteristic high content of Ti (3.8-4.8 wt.% TiO<sub>2</sub>) and rather low amount of F (0.09-0.18 wt.% F). The distinctly low  $X_{Al}$  ratio in examined titanite, together with its low content of fluorine indicates that sometimes observed dissolution of original magmatic apatite (Fig. 3a) played rather subordinate part during origin newly formed titanite.

Secondary titanite originated during low-temperature breakdown of biotite often shows a minor

Al+Fe<sup>3+</sup> excess over F, which indicates the presence of an Al-involving substitution reaction such as (Al, Fe<sup>3+</sup>) + OH ⇌ Ti + O. Amount of the (Al + Fe<sup>3+</sup>)-OH component in low-temperature titanite usually ranges from 5 to 10 mol. % (Enami et al., 1993) which is in good concordance with amount of this component in titanite from the Horní Slavkov-Krásno ore district (4-9 mol. %). Occurrence of REE enriched fluorocarbonate (parisite) argues for presence of some CO<sub>2</sub> in hydrothermal fluids, responsible for breakdown of original biotite and enclosed REE-bearing accessories (apatite, zircon). Similar occurrence of fluorocarbonates in presence of chlorite from hydrothermally altered rocks was described by Pan et al. (1994) and Lottermoser (1995).

## CONCLUSION

Titanium enriched biotite was significantly affected by H<sub>2</sub>O and CO<sub>2</sub> enriched fluids. Mineral assemblage of hydrothermally altered biotite consists hydrothermal titanite and ilmenite accompanied by highly rare REE fluorocarbonate (parisite) and very fine-grained mixture of chlorite and unknown clay minerals (kaolinite, smectite ?). The composition of titanite ranges from 89 to 94 mol. % titanite end-member. Titanite shows some Al+Fe<sup>3+</sup> excess over F

**Table 3** Representative analyses of ilmenite (wt. %).

Sample	R1359-5	R1359-6	R1359-17	R1359-20	R1359-24	R1359-25
SiO <sub>2</sub>	0.01	0.00	0.01	0.02	0.06	0.04
TiO <sub>2</sub>	53.28	53.12	53.17	53.26	53.54	53.12
Al <sub>2</sub> O <sub>3</sub>	0.00	0.00	0.01	0.00	0.02	0.01
Cr <sub>2</sub> O <sub>3</sub>	0.00	0.00	0.00	0.00	0.00	0.00
Fe <sub>2</sub> O <sub>3</sub>	0.00	0.00	0.00	0.00	0.00	0.00
FeO	42.82	43.88	44.87	43.49	43.30	43.66
MnO	3.83	3.37	2.14	3.70	3.69	3.90
MgO	0.04	0.07	0.30	0.06	0.06	0.10
CaO	0.05	0.07	0.04	0.06	0.06	0.03
ZnO	0.03	0.02	0.07	0.00	0.03	0.01
Nb <sub>2</sub> O <sub>5</sub>	0.07	0.03	0.02	0.07	0.17	0.00
Total	100.13	100.56	100.63	100.66	100.93	100.87
Si	0.000	0.000	0.000	0.001	0.001	0.001
Al	0.000	0.000	0.000	0.000	0.001	0.000
Ti	1.007	1.001	1.000	1.002	1.003	0.999
Cr	0.000	0.000	0.000	0.000	0.000	0.000
Fe <sup>3+</sup>	0.000	0.000	0.000	0.000	0.000	0.000
Fe <sup>2+</sup>	0.899	0.920	0.939	0.910	0.902	0.999
Mn	0.081	0.072	0.045	0.078	0.078	0.083
Mg	0.001	0.003	0.011	0.002	0.002	0.004
Ca	0.001	0.002	0.001	0.002	0.002	0.001
Zn	0.001	0.000	0.001	0.000	0.001	0.000
Nb	0.001	0.000	0.000	0.001	0.002	0.000
Σcations	1.991	1.998	1.997	1.996	1.992	2.001
X(Hm)	0.000	0.000	0.000	0.000	0.000	0.000
X(Ilm)	0.916	0.925	0.944	0.920	0.920	0.913
X(Gk)	0.002	0.003	0.011	0.002	0.002	0.004
X(XPph)	0.082	0.072	0.045	0.078	0.078	0.083

Hm – hematite, Ilm – ilmenite, Gk – geikielite, Pph – pyrophanite. Recalculations of FeO<sub>i</sub> using the method of Carmichael (1967).

with significant amount of (Al+Fe<sup>3+</sup>)-OH titanite component (4-9 mol. %). Ilmenite contains considerable amount of pyrophanite component (5-8 mol.%). Assemblage of titanite with considerable amount of (Al+Fe<sup>3+</sup>)-OH titanite component and presence of ilmenite, together with chlorite, argued for low-temperature breakdown of biotite in reducing conditions.

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**Table 4** Representative analyses of biotite (wt. %).

Sample	3	7	9	14	16	17	20	22
SiO <sub>2</sub>	34.72	34.65	35.62	35.19	35.35	35.03	35.31	35.60
TiO <sub>2</sub>	4.21	3.95	4.39	4.29	4.35	4.79	4.31	4.68
Al <sub>2</sub> O <sub>3</sub>	14.15	14.29	14.25	14.10	14.04	14.15	14.24	14.08
FeO	22.74	22.79	22.95	23.36	22.73	23.08	22.67	22.85
MnO	0.19	0.25	0.19	0.22	0.30	0.23	0.26	0.25
MgO	9.07	9.48	9.00	9.27	9.10	8.85	9.17	9.07
CaO	0.01	0.02	0.05	0.01	0.00	0.00	0.00	0.00
Na <sub>2</sub> O	0.11	0.11	0.13	0.08	0.17	0.11	0.12	0.12
K <sub>2</sub> O	8.42	7.68	8.17	8.43	8.07	8.07	8.44	8.27
F	0.17	0.18	0.16	0.16	0.19	0.17	0.19	0.13
Cl	0.06	0.07	0.07	0.07	0.07	0.06	0.07	0.06
O=F,Cl	0.09	0.09	0.08	0.08	0.10	0.09	0.10	0.07
Total	93.76	93.38	94.90	95.10	94.27	94.45	94.68	95.04
Si <sup>+4</sup>	2.748	2.743	2.775	2.750	2.773	2.748	2.762	2.770
Al <sup>IV</sup>	1.252	1.257	1.225	1.250	1.227	1.252	1.238	1.230
Al <sup>VI</sup>	0.068	0.077	0.083	0.049	0.072	0.056	0.075	0.061
Ti <sup>+4</sup>	0.251	0.235	0.257	0.252	0.257	0.283	0.254	0.274
Fe <sup>+2</sup>	1.033	0.933	1.043	1.005	1.009	0.957	1.029	0.993
Fe <sup>+3</sup>	0.472	0.576	0.452	0.521	0.482	0.557	0.454	0.494
Mn <sup>+2</sup>	0.013	0.017	0.013	0.015	0.020	0.015	0.017	0.016
Mg <sup>+2</sup>	1.070	1.119	1.045	1.080	1.064	1.035	1.069	1.052
Ca <sup>+2</sup>	0.001	0.002	0.004	0.001	0.000	0.000	0.000	0.000
Na <sup>+1</sup>	0.017	0.017	0.020	0.012	0.026	0.017	0.018	0.018
K <sup>+1</sup>	0.850	0.776	0.812	0.840	0.808	0.808	0.842	0.821
OH	1.949	1.946	1.951	1.951	1.944	1.95	1.944	1.960
F	0.043	0.045	0.039	0.040	0.047	0.042	0.047	0.032
Cl	0.008	0.009	0.009	0.009	0.009	0.008	0.009	0.008
Fe/(Fe+Mg)	0.584	0.574	0.589	0.586	0.584	0.594	0.581	0.586

Calculations of biotite formulae was done on the basis of a 22+z positive charges according to Yavuz (2003).

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