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A CRITICAL APPROACH TO TI IN THE BIOTITE GEOTHERMOMETER. CASE STUDY ON BIOTITES FROM PEGMATITES OF ROMANIA

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Abstract

The present study reveals that, while the use of the Ti in the biotite geothermometer on biotites from metamorphic rocks seems to provide acceptable data on temperature, in the case of biotite from pegmatites, the temperature data have only limited credibility. As the geothermometer lies on the chemical composition of biotites, the analytical technique that provides the chemistry of the mineral is of major importance. Despite the criticism that might be brought to the method, the wide range of temperatures determined for the biotite from pegmatites of the Carpathian Pegmatite Province is obvious; it seems to prove the multistage character of the mineralogical associations of pegmatites, which can be produced either by successive stages of one single phase (i.e. pegmatitic, pneumatolytic, and hydrothermal) or the successive development of the three genetic phases. The obviously higher temperature of biotites from the metamorphic rocks, compared to that of biotites from pegmatites, suggests that the metamorphic processes develop enough temperature to generate fluids of pegmatite composition; this fact could be in agreement with the theory of genesis of some pegmatite bodies through metamorphic differentiation.

Key words: pegmatite, medium-grade metamorphic rocks, biotite, geothermometry, Ti in biotite.

Introduction

In terms of micas as part of the mineralogical associations in the pegmatite bodies from Romania, biotite has a much lower quantitative participation in comparison with muscovite. Biotite frequently appears into the border zone of large pegmatite bodies (Mârza, 1985;

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Hann, 1987; Murariu, 2001; Stumbea, 2001) as a result of both mechanical (redistribution of mafic material through the pegmatite body) and chemical segregation processes (Stumbea, 1999; 2001). The most common paragenesis of biotite includes tourmaline, muscovite, quartz and, accidentally, garnet.

Generally, biotite appears as small crystals even in the basification zone developed at the contact of some pegmatite bodies with the host metamorphic rocks (Stumbea, 2001). In addition, biotite could also be identified within the inner zones of pegmatite bodies (intermediate and blocky zones and/or quartz core); when it appears as a relic mineral from the metamorphic rocks it underwent intense muscovitization processes. Other transformations that modify the biotite from pegmatites are the following: the removal of pre-existing Fe, Mg, Mn, during the pegmatite genesis, followed by their redistribution in the zone of basification; the formation of muscovite through the biotite depletion in Fe, under hydrolytic conditions, the conversion taking place gradually, until the complete transition of biotite into muscovite; the corrosion of biotite by quartz.

During the last three decades, the biotite of pegmatite bodies from Romania has been the subject of many scientific approaches in terms of its mineralogy and geochemistry (Murariu, 1979, 2001; Mârza, 1985; Hann, 1987; Stumbea, 1999, 2001; Rădășanu, 2002; Androne, 2005). In the framework of their studies, some of these authors also produced data of geothermometry, based mainly on the relation between the chemical features of biotites and temperature (Murariu, 2001; Stumbea, 2001).

The purpose of the present study is to make a critical comparison between the data of previous attempts to determine the temperature of biotites and the new data determined by using the geothermometer proposed by Henry et al. (2005), which lies on the relation between the amount of Ti in biotite and its temperature.

Geological setting

All previous studies carried out on the pegmatites from Romania show that pegmatite bodies are associated, with hardly any exception, to the medium-grade metamorphic rocks of Romanian Carpathians; as Mârza (1980) first emphasized, pegmatite occurrences belong to the so-called Carpathian Pegmatite Province, divided into four subunits which overlay specific mountainous units, as follows (fig. 1):

- the Gilău-Muntele Mare Pegmatite subprovince: pegmatite bodies associated with the metamorphic rocks of the Someș Group (granitoids, migmatite, gneiss, almandine-kyanite-sillimanite–bearing micaschist) and the Baia de Arieș Series (almandine-kyanite-sillimanite–bearing micaschist, gneiss, amphibolites);

- the Preluca Pegmatite subprovince: pegmatite bodies within the metamorphic rock formations of the Răzoare Series (almandine-kyanite–bearing paragneiss, tremolite bearing–limestone and dolomite) and the Preluca Series (staurolite-kyanite–bearing paragneiss and micaschist);

- the Rodna Pegmatite subprovince: pegmatite occurrences within the Rebra Group (paragneiss and micaschist);

- the Getic Pegmatite subprovince: pegmatite bodies associated with the Sebeș-Lotru Group (granitoids, migmatite, almandine-kyanite-staurolite-sillimanite–bearing gneiss and micaschist).

In an overview on pegmatite occurrences from Romania, Mârza (1985) points out some of their main features: the areal association of pegmatites with rocks of the same metamorphic grade and type, suggesting similar genetic conditions; the pegmatite occurrence as small bodies (tens to hundreds of meters long, rarely more); a generally simple and invariable paragenetic mineralogy; the almost exclusive presence of black tourmaline (schörl); genetic processes dominated by alternating abundances of Na or K; the main minerals with an economic potential are the feldspars, quartz and micas.

The genesis of the pegmatite bodies from Romania is a subject of controversy, various hypotheses being recorded, from that of magmatic differentiation processes, sometimes supposed to act even in areas where no magmatic (granitoid) bodies were reported, to that of metamorphic differentiation, responsible especially for occurrences of small pegmatite bodies.

Fig. 1 Sketch of Carpathian Pegmatite Province (modified from Mârza, 1985). 1-limit of pegmatite subprovinces; 2-overthrust fault; 3-granitoid bodies; 4-pegmatite occurrence; 5-low-grade metamorphic rocks; 6-medium-grade metamorphic rocks. I-Preluca pegmatite subprovince; II-Rodna pegmatite subprovince; III-Gilău-Muntele Mare pegmatite subprovince; IV-Getic pegmatite subprovince

Ti in the biotite geothermometer. Description of method

To obtain the optimal fit of the biotite data, Henry et al. (2005) normalized the biotite formulae in a 22 oxygen atom basis, assuming all Fe as Fe^{2+} in order to calculate Ti atoms per formula unit (apfu). They then fit the entire data set, containing 529 analyses, to equations that relate Ti in peraluminous biotites at 4.6 Kbar to T (°C) and Mg/(Mg+Fe) values. The authors tried more than 500 equations, sorted the F-statistic criterion (ratio of the mean squared regression to the mean squared error) in order to optimize the accuracy of the equation and to maintain the simplicity of its form. Using this criterion, Henry et al. (2005) found that the optimal equation is:

$$\ln z = a + bx^3 + cy^3 \qquad (1)$$

where c = T (°C), y = Mg/(Mg+Fe), and z = Ti (apfu).

The correlation coefficient (r^2) of 0.924 determined by Henry et al. (2005) is very good, displaying a significant improvement relative to the previous results of Henry and Guidotti (2002), who obtained an r^2 of 0.866. An encouraging aspect of this fit is that the Ti-saturation surface, although derived from natural data, is consistent with the general Ti trends observed in the biotite experiments carried out earlier by Patiño Douce and Johnston (1991).

Henry et al. (2005) also provide a graphic image of isotherms projected on a Ti vs. Mg/(Mg + Fe) diagram for biotite, which provides an instructive way of viewing the influence of temperature on biotite chemistry; isotherms are relatively closely spaced at temperatures below about 600 °C and at more magnesian compositions. As Henry et al. (2005) stated, their data only covers Ti values ranging from 0.04 to 0.60 apfu, Mg/(Mg + Fe) values from 0.275 to 1.000, temperatures between 480 and 800°C, and pressures of 4 to 6 Kbar, so the extrapolation of isotherms beyond these ranges may introduce significant errors.

Moreover, Henry et al. (2005) made an estimation of errors induced by equation 1, as in table 1.

Tab. 1 Estimation of errors induced by the equation 1 (after Henry et al., 2005)

Range of temperature (°C)	Error (°C)
480 – 600	± 24
600 – 700	± 23
700 – 800	± 12

Analytical techniques

The present study is based on chemical data regarding the biotites from pegmatites and from metamorphic rocks that host pegmatite bodies, obtained through the wet chemical method, X-ray fluorescence technique and electron microprobe method; the chemical data used in the present study are displayed in tables 2, 3 and 4.

In order to establish the influence of an overall interpretation of the analytical data obtained through the three different methods on the accuracy of the geochemical conclusions, the Ti/X(Mg) ratios have been calculated (tab. 2).

Tab. 2 Ti/X(Mg) ratios of the chemical composition of biotites obtained through different analytical techniques

Analytical technique	Mean of Ti/X(Mg) ratio
wet chemical analyses	0.357
Microprobe	0.327-0.622
XRF	0.599

The estimation of the impact of different Ti/X(Mg) ratios on the final temperature data reveals differences between values larger than the estimation of error induced by equation 1 of Henry et al. (2005) (tab. 1). Thus, in order to avoid the misinterpretation induced by the use of chemical data obtained through different analytical techniques, separate discussions were carried out on temperatures, according to each analytical technique that provided the chemical data.

Results

A subordinate to accessory status of biotite from pegmatite bodies of Romania is typical to geochemically primitive pegmatites; this feature is found mainly in pegmatites of ceramic and mica-bearing types, as is the case of almost all pegmatite bodies from Romania. Biotite of the ceramic and mica-bearing pegmatite types from Romania occurs in three generations: as thin elongate crystals in outer zones of the pegmatite bodies that display granite-like textures; biotite flakes in the blocky zones of the pegmatite bodies; large, but thin, platy biotite in quartz cores. Often, the elongate crystals type turns gradually into biotite flakes.

The present study relies on temperature data determined using the geothermometer of Henry et al. (2005) on biotite from both pegmatites (tabs. 2, 3,and 4) and metamorphic rocks that host pegmatite bodies (tab. 6).

The data of table 2 show quite similar mean temperatures of biotite from all pegmatite subprovinces, ranging between 416 and 450°C; in terms of the whole population of temperature data, the range is obviously wider (208-516°C), even in the case of pegmatite bodies that occur in the same area (i.e. the range of biotite temperature in Rodna Pegmatite subprovince is 314-516°C), which is in agreement with the multistage occurrence of biotite during the pegmatite genetic process.

Temperature data determined using the chemical composition of biotites acquired through XRF and electron microprobe techniques (tabs 3 and 4) reveal higher values, such as: 620-659°C (XRF – biotites from pegmatite bodies that occurr in the Getic Pegmatite province); 498-568°C (electron microprobe – biotites from pegmatite bodies occurring in the Getic Pegmatite province); 453-641°C biotites from pegmatite bodies occurring in the Preluca Pegmatite province).

Tab. 2 Temperature data of biotite from pegmatites of Romania (based on the geothermometer of Henry et al., 2005)

Sample	Oxides (wt %)			Cations (apfu)*			X (Mg)	T (°C)
	TiO_2	$(FeO+Fe_2O_3)$	MgO	Ti	Fe_{tot}	Mg		
Gilău-Muntele Mare Pegmatite subprovince								
B-510	1.30	23.48	7.44	0.143	2.816	1.621	0.365	475
B-540	0.46	14.30	13.98	0.051	1.761	3.084	0.637	und**
B-1483	1.10	17.76	8.12	0.120	2.100	1.756	0.456	443
Preluca Pegmatite subprovince								
B-418	1.30	18.26	6.17	0.142	2.170	1.333	0.381	476
B-434	1.32	17.98	6.81	0.143	2.163	1.466	0.404	485
B-467	0.81	23.58	6.61	0.092	2.961	1.493	0.335	208
B-1454	1.30	21.59	7.12	0.152	2.747	1.647	0.375	496
Temperature mean								416
Rodna Pegmatite subprovince								
B-85	1.36	22.50	6.61	0.153	2.774	1.476	0.347	493
B-91	1.36	22.00	6.75	0.153	2.703	1.507	0.358	495
B-141	1.50	24.20	6.00	0.170	2.982	1.347	0.311	516
B-351a	0.92	23.26	5.83	0.104	2.868	1.300	0.312	314
B-351b	0.78	22.42	6.00	0.088	2.782	1.348	0.326	und**
B-381	1.33	24.75	6.30	0.150	3.037	1.412	0.317	482
B-396	0.92	22.00	6.73	0.104	2.738	1.512	0.356	336
Temperature mean								439
Getic Pegmatite subprovince								
BC-Cataracte	0.08	25.55	7.89	0.009	3.123	1.764	0.361	und**
7-Boutari	1.20	22.63	6.60	0.136	2.825	1.486	0.345	455
8-Vosilova	1.24	24.07	5.90	0.139	2.899	1.314	0.312	456
9-Armenis	1.35	23.87	8.90	0.153	2.913	2.002	0.407	506
10-Armenis	1.20	24.14	6.80	0.139	3.065	1.561	0.337	460
b-20	0.91	23.68	9.09	0.105	1.894	2.086	0.524	424
b-34	0.90	24.85	7.77	0.104	1.912	1.785	0.483	397
Temperature mean								450

X(Mg) means Mg/(Mg + Fe) ratio; * normalized in a 22 oxygen atoms basis; und**- undetermined temperature for the reason that either Ti amount or X(Mg) is out of ranges stated by Henry et al. (2005) (see text above)

Higher temperature values were also reported by Murariu (2001), who used different geothermometers based on the relation between temperature and the chemical composition of biotite in terms of either major or minor elements: geothermometer based on the (Mg/Mg+Fe) ratio in biotite, geothermometer based on the Sc amount in biotite;

geothermometer based on the amount of Li in biotite (tab. 5). Moreover, Murariu (2001) provides some data on pressure, regarding the biotite-garnet mineralogical association (6-7Kbar).

Tab. 3 Temperature data of biotite from pegmatites of the Getic Pegmatite subprovince (Conțu-Negovanu pegmatite district) (based on the geothermometer of Henry et al., 2005)

Sample	Oxides (wt %)*			Cations (apfu)**			X (Mg)	T (°C)
	TiO_2	$(FeO+Fe_2O_3)$	MgO	Ti	Fe_{tot}	Mg		
Bi-1[1]	2.57	21.67	9.18	0.295	2.486	2.087	0.456	654
Bi-7[1]	2.13	21.32	8.37	0.248	2.485	1.934	0.438	620
Bi-34[1]	2.27	19.53	10.78	0.266	2.290	2.498	0.522	651
Bi-54[1]	2.72	22.46	9.37	0.305	2.524	2.085	0.452	659
Bi-131A[2]	1.23	19.70	10.13	0.139	2.468	2.262	0.478	498
Bi-132A[2]	1.39	20.41	10.72	0.158	2.568	2.404	0.484	534
Bi-133A[2]	1.44	18.99	10.67	0.165	2.421	2.425	0.500	550
Bi-131B[2]	1.52	18.68	11.00	0.171	2.336	2.451	0.512	562
Bi-132B[3]	1.60	19.52	11.06	0.178	2.412	2.435	0.502	568
Temperature mean (all data)								588
Temperature mean (XRF data)								646
Temperature mean (electron microprobe data)								542

* chemical data from Androne (2005); ** normalized in a 22 oxygen atoms basis; [1] XRF technique; [2] electron microprobe technique

Tab. 4 Temperature data of biotite from pegmatites of the Preluca Pegmatite subprovince (based on the geothermometer of Henry et al., 2005)

Sample	Oxides (wt %)*			Cations (apfu)**			X (Mg)	T (°C)
	TiO_2	$(FeO+Fe_2O_3)$	MgO	Ti	Fe_{tot}	Mg		
Bt-IX	2.49	19.75	7.84	0.284	2.502	1.771	0.414	641
Bt-X	2.30	22.68	7.26	0.268	2.936	1.675	0.363	623
Bt 2	1.14	22.17	5.64	0.138	2.977	1.350	0.312	453

* chemical data from Rădășanu (2002), through electron microprobe technique; ** normalized in a 22 oxygen atoms basis

Tab. 5 Comparative temperature data determined using different geothermometers on pegmatite biotites from the Romanian Pegmatite Province

	Mg/(Mg+Fe) in biotite*	Sc (ppm) in biotite	Li (ppm) in biotite
Range of data**	525 – 595°C	550 – 675°C	520 – 640°C
Mean temperature**	556°C	609°C	624°C

* Mg/(Mg+Fe) in biotite from garnet-biotite association; ** determined using temperature data published by Murariu (2001)

Tab. 6 Temperature data of biotite from some metamorphic rocks of Romania (based on the geothermometer of Henry et al., 2005)

Sample	Oxides (wt %)			Cations (apfu)			X (Mg)	T (°C)
	TiO_2	($FeO+Fe_2O_3$)	MgO	Ti	Fe_{tot}	Mg		
Metamorphic rocks from Preluca Pegmatite subprovince								
bM-2	1.27	12.10	6.69	0.140	1.480	1.464	0.497	508
bG-433	1.59	19.64	10.13	0.179	2.416	2.267	0.484	565
Metamorphic rocks from Rodna Pegmatite subprovince								
bM-313	2.20	20.23	9.64	0.244	2.450	2.121	0.464	623
bM-378	2.20	17.50	8.33	0.241	2.102	1.811	0.463	620
bM-384	2.05	16.60	8.65	0.224	1.970	1.871	0.487	611
bM-1199	1.06	16.75	11.70	0.113	1.944	2.461	0.559	468
Temperature mean								581
Metamorphic rocks from Getic Pegmatite subprovince								
1180	1.58	21.63	9.46	0.175	2.633	2.078	0.441	548
bG-16	2.21	21.16	8.39	0.240	2.499	1.802	0.419	610
bM-4	1.77	16.48	11.21	0.192	1.923	2.408	0.556	600
2	1.59	19.64	10.16	0.180	2.416	2.274	0.485	565
3	1.96	20.40	9.10	0.225	2.511	2.069	0.452	604
4	1.75	20.54	9.84	0.201	2.580	2.237	0.464	584
Temperature mean								585

As the data of table 6 show, the temperatures of the biotite samples from the medium-grade metamorphic rocks that host pegmatite bodies of the Carpathian Pegmatite Province are higher than the temperature data of biotite from pegmatites (tab. 2); temperatures range between 508 and 623°C, the mean value being over 580°C. The comparison involves temperature data based on chemical analyses obtained through the same technique, i.e. wet chemical analysis method.

Figures 2 and 3 show the distribution of temperature data regarding the frequency ranges; it is apparent that the range of highest frequency is 400-500°C in the case of the biotite samples from pegmatites, while most temperature data of the biotites from metamorphic rocks are higher than 600°C. The mean value of temperature of the biotites from pegmatites (444°C) falls in the range of highest frequency, whereas the mean value of temperature of the biotites from metamorphic rocks (559°C) belongs to the 500-600°C frequency range. The ranges of temperatures shown in figures 2 and 3 were not established based on a statistical approach because of the limited amount of data. The temperature values that are the subject of the present paragraph were determined based on chemical analyses obtained exclusively through the wet chemical technique.

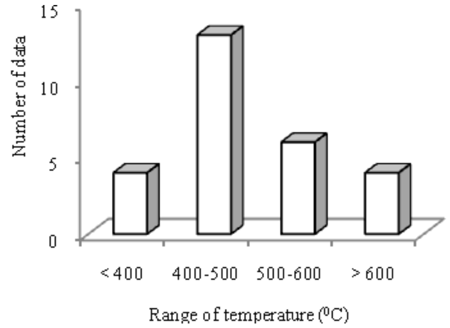

Fig. 3 Frequency of temperature data of pegmatite-hosted biotite samples

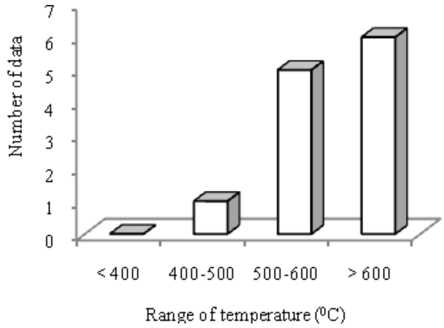

Fig. 4 Frequency of temperature data of metamorphic rocks-hosted biotite samples

Discussion

The present attempt to use the geothermometer proposed by Henry et al. (2005) in order to determine the temperature of biotites from pegmatites and medium-grade metamorphic rocks of the Carpathian Pegmatite Province (Romania) generates discussions on two issues.

The first issue is whether the use of the geothermometer of Henry et al. (2005) in the case of the biotites from pegmatites is advisable, as long as the temperature values of biotite from metamorphic rocks determined through the same method seems to be acceptable.

Thus, the temperature data regarding the biotites from the medium-grade metamorphic rocks, determined using the wet chemical analysis method, range between 508 and 623°C; if we consider the occurrence, the mean values of temperature are higher than 550°C. Otherwise, considering the whole population of temperature data, the mean value is 580°C and it does not fall into the class interval of highest frequency (> 600°C); however, about 90% of temperature data belong to this latter class interval. Quite similar values of temperature were found by Eric et al. (2009) who used the same geothermometer on mica-schist samples from Central Serbia (530-630°C).

Despite the plausible data regarding the temperature of biotite from metamorphic rocks, the temperatures of biotites from pegmatites raise some questions. Thus, the temperature data, based on analyses performed through wet chemical method, are obviously lower (means ranging between 400 and 450°C) than the temperatures based on the chemical composition determined through XRF and electron microprobe techniques (means falling in the 550-650°C interval). The latter temperatures are quite analogous to the data of Murariu (2001), who used geothermometers based on the amount of Sc and Li in biotites. However, these higher temperatures could be contested, since even though the XRF and electron microprobe techniques provide more precise results, the amounts of Ti higher than 0.22 apfu led to less accurate temperature results, as Eric et al. (2009) stated.

A second issue is related to the opportunity of comparing the temperature data obtained by using the geothermometer of Henry et al. (2005) to chemical data determined through the same analytical method (i.e. wet chemical analysis). Thus, the mean temperature of biotite from all four pegmatite subprovinces ranges in a rather narrow span of values (416-450°C). Almost 65% of all temperature data are lower than 500°C and the class interval of highest frequency is 400-500°C; the mean temperature is 444°C and it falls into this class interval.

The comparison between the temperature data of biotite from pegmatite and those of biotite from the pegmatite-hosting metamorphic rocks shows that the latter category of biotites crystallized at temperatures of about 150°C higher than biotites from pegmatite bodies.

Conclusions

The issues emphasized in the previous paragraph led to the following conclusions:

1) At least in the case of the biotites from barren pegmatites (feldspar and/or muscovite-rich pegmatites), the geothermometer of Henry et al. (2005) provides data of limited credibility; in this particular case, getting accurate data on temperature requires a much more complex approach, based on the use of several geothermometers.

2) The temperature values based on chemical data obtained through the XRF and electron microprobe techniques are also problematic, as long as the amounts of Ti frequently exceed 0.22 apfu.

3) Even though the absolute data of temperature determined by the geothermometer of Henry et al. (2005) can be subjected to criticism, the wide range of temperatures determined for the biotite from pegmatites is obvious. It proves the multistage character of the mineralogical associations of pegmatites, which can be produced either by successive stages of one single phase (i.e. pegmatitic, pneumatolytic, and hydrothermal) or the successive development of the three genetic phases.

4) It is highly probable that the biotites submitted to the present study have formed during different genetic stages, such as pegmatitic, pneumatolytic, or hydrothermal.

5) More accurate conclusions regarding the wide range of temperature data would need to take into account whether the pegmatite bodies have a zoned character and, if so, what the sampled zone of the pegmatite body is. However, even though this information would be available, the problem remains difficult to solve, because most of the pegmatite bodies from the Carpathian Pegmatite Province show no zonation or, when zoned, the range of pegmatite body sizes is so wide that an accurate comparison, based exclusively on mineralogical associations, is difficult.

The obviously higher temperature of biotites from the metamorphic rocks, than that of biotites from pegmatites, suggests that the metamorphic processes develop enough temperature to generate fluids of pegmatite composition; this fact could be in agreement with the theory of genesis of some pegmatite bodies through metamorphic differentiation.

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