

CALCITE-GRAPHITE ISOTOPE GEOTHERMOMETER APPLIED TO ESTIMATE METAMORPHIC PEAK CONDITIONS IN MARBLES FROM THE BORBOREMA PROVINCE, NORTHEASTERN BRAZIL

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ABSTRACT – The chemical composition of marbles is a limiting factor for common mineral geothermometry. But carbon isotope fractionations occur in the system carbonate-CO₂-graphite-CH₄ both at low and high temperatures. Thus, the fractionation of the ¹³C and ¹²C isotopes between calcite and graphite has become a useful geothermometer for a large range of temperatures. In this work, the results of the calcite-graphite geothermometer are reported to estimate the temperature of the metamorphic peak of the Itajubatiba region, Borborema Province, Northeastern Brazil. Calibration curves were used that are valid for a range of temperatures (0-700 °C and 400-680 °C) that include those (572-627 °C; mineral geothermometry in mica schists) considered as the peak of the regional metamorphism. The temperatures (560-656 °C) obtained for the metamorphic peak are coherent with those previously known from the regional scenario. The agreement between the temperatures found and those already established indicates that calcite and graphite are in isotopic equilibrium, as corroborated by the intimate physical contact between both minerals. The ¹³C/¹²C isotopic ratios of calcite and graphite are a good tool to estimate metamorphic peak temperatures in marbles, and therefore can be applied as a geothermometer in other regions.

Keywords: ¹³C and ¹²C isotope ratios, calcite-graphite geothermometer, phlogopite-bearing marble, Itajubatiba region, Borborema Province.

RESUMO – J.A. Souza Neto, J.M. Legrand, Ph. Sonnet - *Geotermômetro isotópico calcita-grafita aplicado para estimar condições de pico metamórfico em mármore da Província Borborema, nordeste do Brasil.* Os mármore não possuem uma composição que permita se utilizar geotermômetros minerais comuns. Por outro lado, o fracionamento de isótopos de carbono no sistema carbonato-CO₂-grafita-CH₄ ocorre tanto em baixa quanto em alta temperatura. Dessa forma, o fracionamento dos isótopos ¹³C e ¹²C entre a calcita e a grafita tem se tornado um geotermômetro usual em amplo intervalo de temperaturas. Neste trabalho, os resultados do geotermômetro calcita-grafita são apresentados e tiveram como objetivo a estimativa da temperatura do pico metamórfico da região de Itajubatiba (Província Borborema, nordeste do Brasil). Foram utilizadas curvas de calibração válidas para o intervalo de temperaturas (0-700 °C e 400-680 °C) que inclui aquelas consideradas para o pico do metamorfismo regional (572-627 °C; geotermometria mineral em mica xistos). As temperaturas obtidas (560-656 °C) são coerentes com as do metamorfismo regional. Esta coerência sugere que a calcita e a grafita estão em equilíbrio isotópico, como atesta a íntima relação de contato observada entre estes dois minerais. As razões isotópicas ¹³C/¹²C da calcita e grafita revelam ser uma ferramenta apropriada para se estimar as temperaturas do pico metamórfico em mármore, e podem ser aplicadas como geotermômetro em outras áreas.

Palavras-chave: Razões isotópicas ¹³C e ¹²C, geotermômetro calcita-grafita, flogopita mármore, Região de Itajubatiba, Província Borborema.

INTRODUCTION

Marbles have a particular composition that it is not useful for the application of common mineral geothermometers (Yardley, 1989). Generally, metamorphic conditions in these rocks are broadly

evaluated using the P-T or P-T-XCO₂ stability curves for their mineral parageneses, which normally contain calcite (or dolomite) and other common minerals such as talc, tremolite, diopside, forsterite, and wollastonite

(Winkler, 1979; Yardley, 1989). The difficulty to have a well calibrated mineral assemblage for the application of mineral geothermometry is a known characteristic of the marbles.

The isotope geothermometry is based on the partitioning of two stable isotopes of an element between two mineral phases, and its general principle is that the isotopic fractionation for the isotope exchange between minerals is a function of the temperature, as in a formula $1/T^2$. The condition to apply an isotope geothermometer is isotopic equilibrium. Isotope exchange equilibrium should be established during reactions whose products are in chemical and mineralogical equilibrium. The most important characteristic of the isotopic exchange is the pressure insensitivity of isotope partitioning. This represents a considerable advantage relative to other geothermometers that exhibit pressure dependence. The study of carbon isotope fractionations in the system carbonate-CO₂-graphite-CH₄ is important not only under low-temperature conditions, but also at high temperatures. Thus, the fractionation of the ¹³C and ¹²C

isotopes between calcite and graphite has become a useful geothermometer applied to a large range of temperatures, even higher than 800 and 900 °C (Hoefs, 2009).

In order to evaluate the isotopic equilibrium between calcite and graphite, the texture and surface patterns of graphite can be observed. Crystalline graphite, in hexagonal or semi-hexagonal crystals, with a polished surface and high reflection, indicates isotopic equilibrium and thus preserves the metamorphic peak conditions. On the other hand, less crystalline forms, irregular crystals, showing rough surface (overgrowth in early graphite), suggest isotopic disequilibrium after metamorphic peak conditions (Wada & Suzuki, 1983).

In this work the results of the calcite-graphite geothermometer (¹³C/¹²C) are reported to estimate the temperature of the metamorphic peak of the Itajubatiba region, Borborema Province, Northeastern Brazil. This geothermometer was applied using samples of the phlogopite-bearing marble occurring in the investigated area.

GEOLOGICAL SETTING OF THE ITAJUBATIBA REGION

The Itajubatiba village is situated about 35 km southwest from the town of Patos, in the state of Paraíba. At Itajubatiba there is a skarn gold deposit that was discovered in the 1940's and was mined over a thirty-year period, with a total production of approximately 5 tons of gold. The ore grade ranged from 0.5 to 2.0 ppm and the highest grade reached 6.3 ppm (Lins & Scheid, 1981; Rebouças, 1985).

The Itajubatiba skarn deposit is located north of the Patos lineament, where continental scale W-E-trending strike-slip shear zones dominate the tectonic features. These shear zones with associated folds and thrusts were developed during transpressional tectonics that were active during the Brasiliano/Pan-African orogeny, around 600 Ma (Archanjo & Bouchez, 1991; Caby et al., 1991; Corsini et al., 1991, 1992, 1996; Vauchez et al., 1995).

The age of the metamorphic peak was estimated by the ⁴⁰Ar/³⁹Ar isotopic geothermochronology in the Patos lineament region as 544 ± 3 Ma at 550-650 °C, and the retrograde metamorphism as 505 Ma at 400-500 °C (Figueiredo, 1992).

The stratigraphic positioning of the geologic units is particularly difficult in the region of Itajubatiba, due to the intense tectonic perturbation. Thus, a stratigraphic positioning has been proposed, based on a regional geological correlation (Souza Neto, 1999). The lithologies are grouped (from the oldest to the youngest) into marble, schist, ortho-derived migmatites, and

alkaline igneous rocks. Marble and schist are correlated to the supracrustal rocks of the Seridó Group (Jucurutu and Seridó formations, respectively), of Mesoproterozoic and Neoproterozoic age, respectively (Van Schmus et al., 2003). The ortho-derived migmatites and igneous rocks are correlated to the widespread neoproterozoic magmatism occurring in the Borborema Province (Souza Neto, 1999; Souza Neto et al., 2008). These last two rock units show a higher migmatization degree in the Itajubatiba region, if compared to their equivalents in the Seridó metapelitic belt. The reason is that the Patos lineament zone has probably exposed deeper rocks with a higher metamorphic grade.

At Itajubatiba region, the southern portion of the area is dominated by schist, while migmatites are the main rocks present in the northern portion. The alkaline intrusions are highly elongated in the area. Skarns occur in the northern part of the area as beds and lens within the marble, at the contact margins of a meta-syenogranite intrusion, and within this intrusion itself (endoskarn). The marble is described in detail below based on samples from outcrops and drill cores.

MARBLE

Marble occurs as isolated lenses and blocks 500 to 800 m long within ortho-derived migmatites of the Itajubatiba area. In the geological profiles reconstructed by drill core descriptions in the area of the Itajubatiba

mine, marble beds occur as tectonic bodies 2 to 250 m thick, shear bounded, within ortho-derived migmatites. Marble is either phlogopite- or olivine-bearing.

Phlogopite-Bearing Marble

The phlogopite-bearing marble presents the paragenesis calcite + phlogopite + tremolite + talc + graphite + apatite. Chlorite has been formed at the expense of phlogopite. Phlogopite-bearing marble has a fine to medium-grained and lepidogranoblastic texture.

Phlogopite-bearing marble contains paragneiss, which occurs as centimetric interlayered lenses and pockets (*boudins*), 15 to 150 cm wide, and up to 10 m long, within the phlogopite-bearing marble. It is composed of the following assemblage: alkali feldspar + plagioclase + phlogopite + quartz + titanite + allanite + apatite + tourmaline + opaque minerals. Actinolite and chlorite also occur and were formed at the expense of the phlogopite. Paragneiss presents a fine to medium-grained and lepidogranoblastic texture.

In the contact between paragneiss and marble, a diopside-rich band (up to 3 cm wide) was developed. Diopside is also present disseminated within the paragneiss, mainly near the diopside outer zone of skarns. Quartz and quartz-actinolite veins (up to 2 cm wide) orthogonally crosscut the paragneiss lenses.

Olivine-Bearing Marble

Olivine-bearing marble occurs within the marble beds, mainly at the periphery of the skarns, between skarns and the phlogopite-bearing marble. They constitute the outer zone of the skarns. Olivine-bearing

marble is composed of the assemblage calcite + olivine + talc + hercynite + apatite + allanite. Serpentine occurs within this marble and was formed by replacement of olivine. Humite-clinohumite partly replaces olivine crystals. Actinolite-tremolite, clinocllore and white mica also occur and probably were formed at the expense of phlogopite. Phlogopite breakdown is attested by the progressive decrease in the phlogopite amount, or its total absence, going from phlogopite-bearing marble towards the adjacent olivine-bearing marble. Magnetite, pyrrhotite, pyrite, chalcopyrite and hematite occur as well within olivine-bearing marble. Olivine-bearing marble shows a fine to medium-grained and granoblastic texture.

Serpentine-rich pockets (0.5 to 1 mm in diameter) consist of a typical texture shown by olivine-bearing marble. These pockets also have clinocllore, hercynite and magnetite, and enclose remnants of olivine and phlogopite. Locally pockets of pargasite-tschermakite (olivine pseudomorphs ?) also occur and contain hercynite inclusions and an aureole of clinocllore. In this case, pargasite-tschermakite is the amphibole also found in the amphibole-rich zone of the adjacent skarn.

Near the skarn contact, titanite is present as an accessory phase in the olivine-bearing marble, and calcite shows an alteration feature (in bands), where it has a dirty aspect with many small inclusions (<10 µm) of opaque minerals. Late tremolite-filled micro-veins can crosscut the olivine-bearing marble in this position. The metasomatic process taking place in the skarn formation is probably responsible for titanite, altered calcite, and tremolite formation.

CALCITE-GRAPHITE GEOTHERMOMETER STUDY

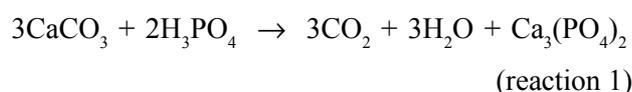
SAMPLE AND ANALYTICAL PROCEDURES

Two representative samples of the phlogopite-bearing marble occurring in the Itajubatiba region were used to obtain the analyses of carbon and oxygen isotopes to be used in the calcite-graphite geothermometer. The mineralogical compositions of the investigated samples are shown in Table 1.

The samples (mineral concentrates) were prepared from hand specimens, which had been broken and washed before crushing. Calcite and graphite were concentrated from 250-500 µm (and 500-1,000 µm in some more coarse samples) grain sizes by magnetic separation and handpicking under a binocular magnifier. Later, calcite concentrates were cleaned in ethanol, dried at 60 °C in an oven, and crushed (<100 µm) in an agate mortar. Graphite samples were crushed in agate with ethanol to clean and remove impurities present

between the sheets of graphite. Afterwards, these samples were also dried in an oven at 60 °C.

Isotopic analyses of calcite and graphite samples were performed at the *Unité de Recherches en Biogéochimie des Isotopes Stables (URBIS)*, University of Liège, Belgium. CO₂ for isotopic analyses was extracted from calcite by reaction under vacuum with 100 % orthophosphoric acid (H₃PO₄) at 25 °C for 16-20 h in two-legged sealed reaction vessels (McCrea, 1950). CO₂ is produced by the reaction:



or



TABLE 1. Carbon isotope analyses of calcite and graphite and calculated temperature using the calcite-graphite geothermometer for the phlogopite-bearing marble from Itajubatiba, Borborema Province, Northeastern Brazil.

Samples	Mineralogical composition (in decreasing order of abundance)	$\delta^{13}\text{C}_{\text{V-PDB}} (\text{‰})$		$\Delta^{13}\text{C}_{\text{calcite-graphite}}$	Temperature ($^{\circ}\text{C}$)	
		Calcite	Graphite		Bottinga (1969)	Wada & Suzuki (1983)
AL 192/1 (outcrop)	cal, phl, tr, tlc, gr, ap	- 0.7	- 7.3	6.6	560	516
SIT 8/9 (drill core, 66m deep)	cal, phl, tr, tlc, gr, ap, ttn	- 1.7	- 7.5	5.8	656	556

Mineral abbreviations (according to Kretz (1983) and Spear (1993)): ap - apatite, cal - calcite, gr - graphite, phl - phlogopite, tlc - talc, tr - tremolite, ttn - titanite.

Sample aliquot varying from 10 to 30 mg and 5 ml of acid were used in this reaction. Cryogenically purified CO_2 was then analyzed on a Micromass Optima Isotope Ratio Mass Spectrometer (IR-MS) fitted with a dual inlet, and the results were expressed relative to V-PDB (Vienna Peedee Bellemnitte, d^{13}C) and V-SMOW (Vienna Standard Mean Ocean Water, d^{18}O) - Table 1. Internal and international reference materials were analyzed at the same time as the samples, and each measure is the average of 10 d measurements that were obtained by alternating absolute isotopic ratio measurements for the standard and for the sample. Some sample analyses were replicated. Results for calcite were calibrated against reference materials prepared and provided by the International Atomic Energy Agency, i.e. IAEA-CO-1 (from a slab of Carrara marble, Italy, provided by IMEG Viareggio, with $\text{d}^{13}\text{C}_{\text{V-PDB}} = 2.48 \text{‰}$ and $\text{d}^{18}\text{O}_{\text{V-SMOW}} = 28.38 \text{‰}$) and IAEA-CO-8 (from a carbonatite from Kaiserstuhl, Germany, provided by the Geologische Landesamt, Freiburg, with $\text{d}^{13}\text{C}_{\text{V-PDB}} = -5.75 \text{‰}$ and $\text{d}^{18}\text{O}_{\text{V-SMOW}} = 7.53 \text{‰}$).

Carbonic gas (CO_2) from graphite was obtained by combustion and reduction of the samples in a Fisons NA 1500 NC Elemental Analyzer. After chromatographic separation, the gas was driven under a helium stream into the mass spectrometer using a continuous flow interface. Results for graphite were calibrated against intercomparison materials IAEA-CH-6 (sucrose, $\text{d}^{13}\text{C}_{\text{V-PDB}} = -10.4 \text{‰}$) and IAEA-N-1 ($(\text{NH}_4)_2\text{SO}_4$, $\text{d}^{15}\text{N}_{\text{AIR}} = 0.4 \text{‰}$). Cyclohexanone-2, 4-dinitrophenylhydrazone ($\text{C}_{12}\text{H}_{14}\text{N}_4\text{O}_4$, provided by Fisons Instruments, Italy) with 51.81 wt% of C and 20.15 wt% of N, was used as a standard for C-N composition. With this procedure, a precision of $\pm 0.02 \text{‰}$ and a reproducibility of 0.26 ‰ could be achieved.

RESULTS AND DISCUSSION

The calcite-graphite pair was calibrated by various

authors and by different approaches (e.g. theoretical calculations, experimental determinations in laboratory and calibration on an empirical basis; Table 2). A notorious good agreement exists among the various calibration curves, especially in the temperature range from 600 to 800 $^{\circ}\text{C}$ (Kitchen & Valley, 1995).

In this study, the calibration curves of Bottinga (1969) and Wada & Suzuki (1983) were used, because they are valid for a range of temperatures (0-700 $^{\circ}\text{C}$ and 400-680 $^{\circ}\text{C}$, respectively), which include those (572-627 $^{\circ}\text{C}$) considered for the peak of the regional metamorphism of the Seridó meta-pelitic belt (Lima, 1986; Luiz-Silva, 1995; Cunha de Souza, 1996). Temperature calculations were done by interpolation using the values of the Bottinga's curve and using the equation that describes the curve of Wada & Suzuki (1983). The results obtained with the calcite-graphite geothermometer for the phlogopite-bearing marble from the Itajubatiba region are shown in Table 1.

The temperatures obtained (560-656 $^{\circ}\text{C}$) for the metamorphic peak of the Itajubatiba marble, using the Bottinga's calibration of the calcite-graphite geothermometer, are rather coherent with those previously established (572-627 $^{\circ}\text{C}$) for the metamorphic peak of the Seridó meta-pelitic belt using mineral geothermometers in mica schists (Lima, 1986; Luiz-Silva, 1995; Cunha de Souza, 1996). Temperatures (516-556 $^{\circ}\text{C}$) calculated using the calibration of Wada & Suzuki (1983) are slightly lower in relation to the other calibration used. The agreement between the results found in the present study and the previously known metamorphic temperatures indicates that calcite and graphite of the investigated marble are in isotopic equilibrium. This can be corroborated by the intimate physical contact between calcite and graphite as observed in thin sections (Figure 1), which reflects a textural equilibrium between both minerals.

TABLE 2. Curve equations for the main calibrations of the calcite-graphite geothermometer and their range of temperatures.

Curve equations*	Range of temperature calibrated (°C)	References
$\Delta^{13}\text{C}_{\text{calcite-graphite}} = 25.419 - 0.066363T + (5.5468T^2/10^5)$ (K)	0 - 700	Bottinga (1969)
$\Delta^{13}\text{C}_{\text{calcite-graphite}} = 0.00748 \times T + 8.68$ (°C)	610 - 760	Valley & O'Neil (1981)
$\Delta^{13}\text{C}_{\text{calcite-graphite}} = 5.6 \times (10^6/T^2) - 2.4$ (K)	400 - 680	Wada & Suzuki (1983)
$\Delta^{13}\text{C}_{\text{calcite-graphite}} = 3.56 \times (10^6/T^2)$ (K)	650 - 850	Kitchen & Valley (1995)

* T temperature in Kelvin (K) and in °C.

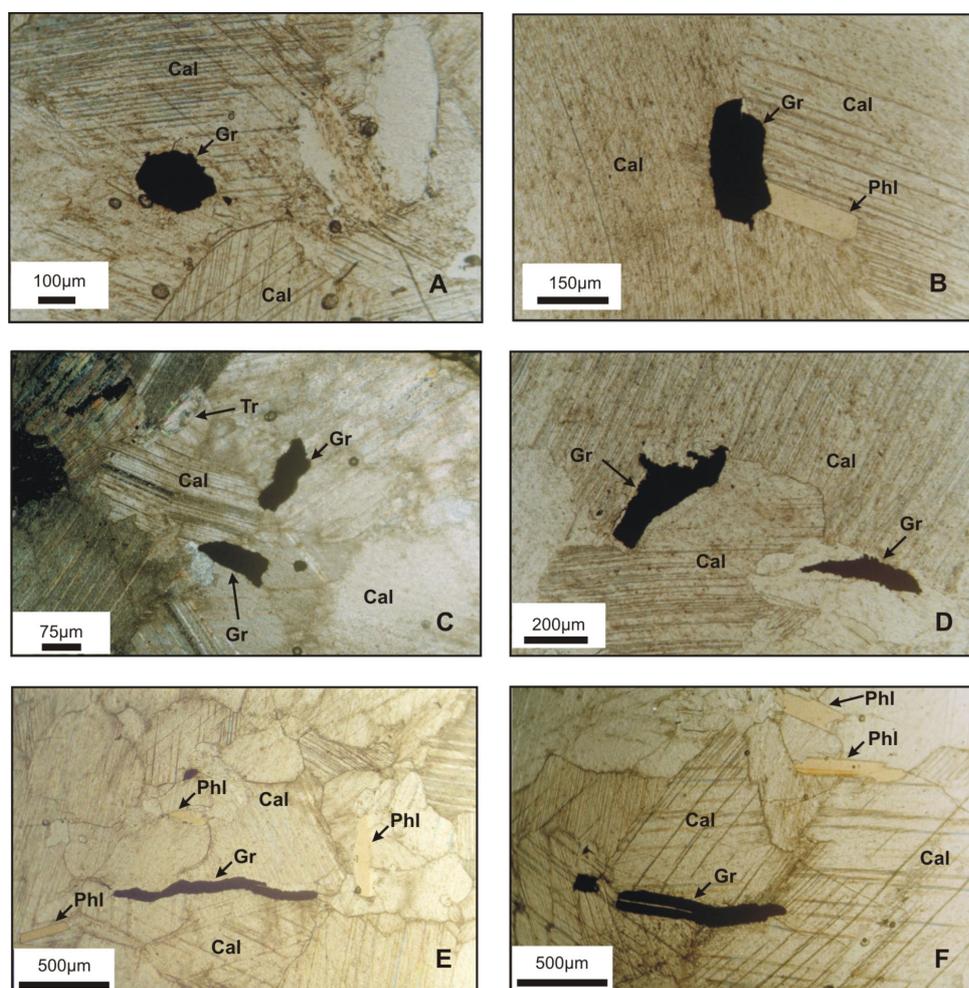


FIGURE 1. Photomicrographs showing graphite textures in the phlogopite-bearing marble from Itajubatiba, Borborema Province, northeastern Brazil. (A) Partial hexagonal flake of graphite (Gr) in textural equilibrium with calcite (Cal). Sample AL10, transmitted and natural light. (B) Graphite (Gr) flake in textural equilibrium with calcite (Cal) and phlogopite (Phl). Sample AL192-C2, transmitted and natural light. (C) Partial hexagonal and irregular flakes of graphite (Gr) in textural equilibrium with calcite (Cal). Marble matrix also shows tremolite (Tr, on the top left). Sample AL10C, transmitted and plane-polarized light. (D) Partial hexagonal flake of graphite (Gr, on the left) in textural equilibrium with calcite (Cal), and a subeuhedral, irregular, needle-shaped graphite crystal (Gr, on the bottom right). Sample AL192-C2, transmitted and natural light. (E) Thin, needle-shaped graphite (Gr) crystal flexured between calcite (Cal) crystals. Marble matrix also shows phlogopite (Phl, on the center, on the right, and on the bottom left). Sample AL10, transmitted and natural light. (F) Needle-shaped graphite (Gr) crystal flexured between calcite (Cal) crystals. Marble matrix also exhibits phlogopite (Phl, on the top right). Sample AL10, transmitted and natural light.

CONCLUSIONS

This work reveals that calcite-graphite pairs seem to preserve isotopic equilibrium, and thus record peak metamorphic temperatures in the marbles of the Itajubatiba area, because the temperatures obtained for the metamorphic peak using the calcite-graphite geothermometer are in agreement with those calculated in previous studies for regional metamorphism using

mineral geothermometers. Among the calibration curves used, the Bottinga curve shows the best estimate of peak metamorphic temperatures. The $^{13}\text{C}/^{12}\text{C}$ isotopic ratios of calcite and graphite reveal to be a reliable tool to estimate metamorphic temperatures using marbles, and thus can be applied as a geothermometer in other marble units.

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