First finding of native Nickel in cumulates of the Canindé Domain, Brazil

Primeira descoberta de Níquel nativo em cumulatos do Domínio Canindé, Brasil

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ABSTRACT

Studies of the Fe-Ti oxides and Cu-Ni sulfides mineralizations of the Canindé Domain, Sergipe state, Brazil, are being carried out by our research group. These studies, which are aimed at mineralogical and geochemical characterization of these occurrences, involved until now field work, transmited and reflected light microscopy and scanning electron microscopy (SEM). In the field the Fe-Ti oxides (magnetite-ilmenite with spinel) cumulates occur mainly as small blocks in the contact between the Complexo Gabróico Canindé unit and the Novo Gosto-Mulungu formation. Through SEM analysis it was possible to identify native nickel (Ni) grains as inclusions in spinel of the magnetitic cumulates. This was the first time that native nickel was discovered in the Canindé Domain and in Brazil and has the additional scientific interest of being one of the extremely rare occurences of native Ni in the world, as only five have been described worldwide, according to our best knowledge. It also determines a necessary re-evaluation of the mineralizations of the Canindé Domain, of the geochemical significance of this occurrence for its genesis, the implications it has in the mineralizations modelling and economical potential.

Keywords: Nickel. Canindé Domain. Fe-Ti cumulates.

RESUMO

Estudos das mineralizações de óxidos de Fe-Ti e de sulfetos de Cu-Ni do domínio de Canindé, Sergipe, Brasil, estão sendo realizados pelo nosso grupo de pesquisa. Esses estudos, que visam à caracterização mineralógica e geoquímica dessas ocorrências, envolveram até agora trabalhos de campo, microscopia de luz transmitida e refletida e microscopia eletrônica de varredura (MEV). No campo, os cumulatos de óxidos de Fe-Ti (magnetita-ilmenita com espinela) ocorrem principalmente como pequenos blocos no contato entre a unidade Complexo Gabróico Canindé e a formação de Novo Gosto-Mulungu. Através da análise MEV, foi possível identificar grãos de níquel nativo (Ni) como inclusões em espinela dos cumulatos magnetíticos. Esta é a primeira vez que níquel nativo é descoberto no Domínio Canindé e no Brasil e tem o interesse científico adicional de ser uma das extremamente raras ocorrências de Ni nativo no mundo, já que apenas cinco foram descritas em todo o mundo, de acordo com nosso conhecimento. Também determina uma reavaliação necessária das mineralizações do Domínio Canindé, do significado geoquímico desta ocorrência para a sua gênese, as implicações que tem na sua modelagem e o potencial econômico destas mineralizações.

Palavras-chave: Níquel. Domínio Canindé. Cumulatos de Fe-Ti.

1 Introduction

The Canindé Domain (CD), located in the state of Sergipe, has been recognized as an area with potential for Fe-Ti and Cu-Ni since the late 70's (Silva Filho *et al.*, 1979).

Our ongoing research project in this area has the objectives of improving the knowledge of the metalogenesis in the CD, through mineralogical and chemical characterization of the ore minerals.

While conducting scanning electron microscopy (SEM) analysis of samples of spinel-magnetite-ilmenite cumulates we observed micrometer sized grains of native nickel as inclusions in spinel. The aim of the present paper is to describe this occurrence of native nickel in the CD.

The occurrence of native nickel is extremely rare (Nickel, 1959; Challis, 1975; Hudson & Travis, 1981; Bai *et al.*, 2000; McDonald *et al.*, 2010), and we haven't found any reference to it occurring in spinel of magnetitic cumulates. Correia de Brito *et al.* (2005) have described awaruite ($Ni_{2-3}Fe$) occurring in magnetitite pods in Rio Jacaré sill, Bahia. Some authors cite the occurrence of native nickel, and other metal alloys like awaruite, resulting from serpentinization, hydrothermalism and other reconcentration linked processes undergone by peridotite type rocks (Nickel, 1959; Ramdohr, 1969; Challis, 1975; Hudson & Travis, 1981; Radhakrishna *et al.*, 1982; Klein & Bach, 2009; McDonald *et al.*, 2010).

2 Geological setting

The main geotectonic province in northeastern Brazil is the Borborema Province, which is a set of crustal blocks with different ages, origin and evolution, amalgamated during the Brazilian Orogeny, spreading across 800 km from the state of Rio Grande do Norte to the state of Sergipe.

The main geotectonic system of the Borborema Province in the state of Sergipe is the Sergipano Orogenic System (SOS) (Conceição *et al.*, 2015). The SOS is characterized by metassedimentary terrains with granitic plutonism amidst extensive transcurrent shear zones (D'el-Rey Silva, 1995, Oliveira *et al.*, 2010). It is subdivided into eight geotectonic domains of which the Canindé Domain is the northernmost one (Figure 1).

Fig. 1. – A: State of Sergipe geographical position in Brazil; B: Sergipe's geotectonic–stratigraphic domains andstudy area rectangle (adapted Teixeira *et al.*, 2014); and C: geological sketch of studyarea with sites of occurrences of Fe-Ti cumulates as rolled blocks(adapted from Santos & Souza, 1988, and Teixeira *et al.*, 2014).
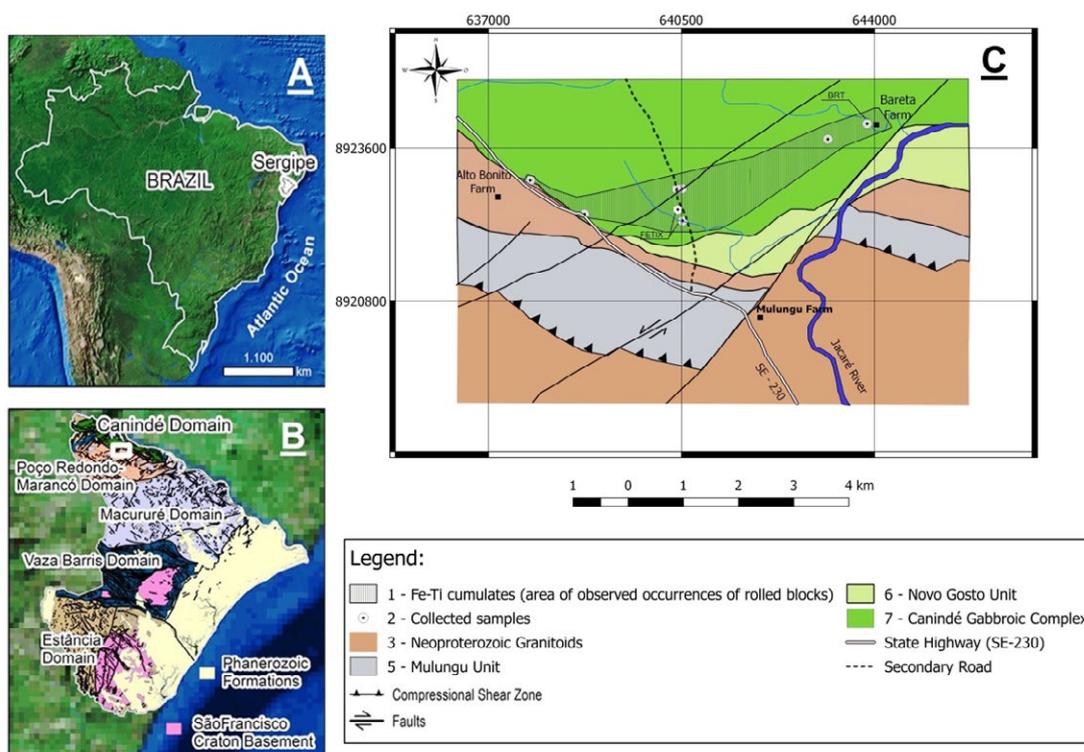
2.1 Canindé Domain (CD)

Occupying a NW-SE strip about six to fifteen kilometres wide parallel to the São Francisco river, the CD (Figure 1), consists of polydeformed and sheared metavolcano-sedimentary rocks of the Canindé Complex (CC), intruded by a differentiated gabbroic body which is the Canindé Gabbroic Complex (CGC) (Santos *et al.*, 2001; Oliveira *et al.*, 2010).

Fe-Ti oxides and Cu-Ni sulphides occurrences were identified in the CC and the CGC by the Brazilian Mineral Resources Research Company (CPRM) (Silva Filho *et al.*, 1979; Tesch *et al.*, 1980; Santos & Souza, 1988; Seixas & de Moraes, 1996; Santos *et al.*, 1998, 2001).

The CC is a set of metavolcanic and metasedimentary rocks, first described and individualized by Silva Filho *et al.* (1979) in the following units: Mulungu, Novo Gosto, Gentileza and Garrote.

The CGC and the Neoproterozoic granitoids (formerly known as the Garrote Unit) occur in the nucleous of the CD.

The classification of these units has been maintained until today, with the exception of the Garrote Unit, which is shown in Figure 1 as Neoproterozoic granitoids.

In the Novo Gosto Unit megafolds of interbedded limestones and quartzites are topographicaly predominant. Calcissilicatic rocks, phyllites, gneisses and pyroclastic breccias occur very localized. The Mulungu Unit, in addition to rock types similar to the Novo Gosto Unit, has lenses of talcified meta-ultramafic rocks, mylonitic granitoids and acid metavolcanic schists. Oliveira *et al.* (2010) groups these two units as Novo Gosto-Mulungu.

The parageneses found in the Canindé Complex units are indicative of amphibolite facies metamorphism with the presence of andalusite and cordierite. Green schist facies retrograde metamorphism is predominant in the sheared zones.

The CGC (Oliveira *et al.*, 2010) is composed of plutonic rocks mainly of gabbro composition, where most of the Fe-Ti and Cu-Ni mineralizations are found. It occurs as a strip of around five kilometres width and a length of approximately forty kilometers, parallel to the São Francisco River (Figure 1), between the village of Niterói and the town of Canindé de São Francisco. Smaller bodies occur intruded in the supracrustal rocks of the CC or as megaxenolithes in granitoids. Their contacts are intrusive or by ductile shear zones, especially with respect to CC units. According to Santos

et al. (2001), the CGC has a compositional range from gabbros, to norites, anorthosites, troctolites and other ultramafic rocks, sometimes with cumulus textures, indicative of magmatic differentiation processes. The paragenesis of these rocks indicates an average degree of metamorphism of the amphibolite facies to epidote-amphibolite, with located retrogressive

metamorphism of the green schist facies. Fine-grained gabbros occur predominantly in the periphery of the main body, whilecoarse-grained leucogabros are more frequent in the region northwest of Poço Redondo. Troctolites occur in the form of pockets related to leucogabros. Souza & Santos (1988) suggest that the major occurrences of Cu-Ni sulphides are associated with troctolites. The Fe-Ti cumulates are present near the southern contact of the main massif. Previous works described these occurrences as subsurface layers and thick lenses in the gabbro and also as loose rolled blocks in the ground (Tesch *et al.*, 1980; Seixas & de Moraes, 1996).

Santos & Souza (1988) concluded that this complex was generated from the partial fusion of two materials, one toleiític, low-K, and another alkaline. Bezerra *et al.* (1992) make an analogy with sinorogenic intrusions, while Oliveira & Tarney (1990) propose intracontinental rifting and anorogenic magmatism. Van Schmus *et al.* (1997) obtained a model-age of 940My, by the Sm/Nd method, for the gabbroic rocks. Oliveira *et al.* (2010) making a synthesis of the various data and hypotheses interprets the Canindé Domain as a rift sequence, probably having evolved in a ocean basin because of the presence of amphibolite in Novo Gosto unit interleaved with marble lenses and relics of pillow basalts.

3 Methodology

Field trips to the study-region were made to colect geological data and samples for analysis. Outcrops of the rocks that contain Fe-Ti oxides are very scarce and the samples mostly occur as detached rolled blocks in the ground, which is corroborated by other authors (Tesch *et al.*, 1980; Seixas & de Moraes, 1996, Santos *et al.*, 2001).

The collected samples were chosen and prepared for optical microscopy and scanned electron microscopy (SEM-EDS). The samples of Fe-Ti cumulates are referenced FETIX and BRT. Polished thin sections were prepared by the Brazilian Geological Service in Bahia (CPRM-Bahia).

The thin sections were studied using an Olympus model BX41 transmitted light optical microscope with coupled digital camera Olympus SC30 for microphotography using *Cell^B* software. For reflected light microscopy we used an *OPTON* model TNP-09NT.

Scanning electron microscopy was performed using a *Tescan* model Vega3 with secondary electrons and back-scattered electrons detector for imaging and an *Oxford Instruments* energy dispersive spectrometer model *x-Act*, for content analysis. Coating of the samples was made with a Quorum (Q150R ES).

During SEM analysis vacuum conditions were of $1,5 \times 10^{-3}$ Pa. The beam current was of 15 Kv, tension 20 nA and diameter was in most cases about 800 to 850 mµ. Mean counting time for EDS was of 80 seconds.

4 Results

Thin sections referenced FETIX-1, FETIX-2 and BRT were observed by polarizing microscope enabling to identify the major minerals present as magnetite, ilmenite, spinel (pleonaste) and corundum. The texture of this rock is a typical magnetitic cumulate texture as described by Ramdohr (1969) which can be observed in Figures 2 and 3. Cumulus phases are magnetite and ilmenite crystals, with ilmenite frequently intergrown in trellis texture in the magnetite (similar to Ramdohr, 1969, p. 900) (Fig. 4). Spinel crystals complete the matrix which has a homogenous isotropic fabric with some pores. The proportions are estimated to be in the mean of about 50% magnetite+ilmenite to 50% spinel. The magnetite crystals are usually subidiomorphic to allotriomorphic. There is minor intercumullus magnetite and ilmenite. Most of the remaining space is occupied by subidiomorphic spinel crystals (Figs. 2 and 3). The opaque minerals as well as the intergrowth phenomena between magnetite and ilmenite, described as trellis texture, were also observed in reflected light microscopy. Spinel is mostly of the variety pleonaste (Table 1), has green color, or is otherwise colorless to olive with brownish rims, and is usually subhedral (Figs. 2 and 5). Crystal size ranges from 0,11mm up to 0.45 mm in well-developed crystals. Its contacts with the opaque minerals tend to be straight. The spinel crystals often show exsolutions (most probably of iron oxides) in {111} planes (Fig. 5), similar to what is presented in Ramdohr (1969, p. 892-893). As acessory minerals there occurs: anhedral corundum filling gaps in the intercumulus, having rough edges with other opaque minerals, and bluish color; ocasionally, a redish mineral observed in reflected microscopy which

probably is hematite, was formed in magnetite crystal rims'; and clinochlore which is anhedric, white and with anomalous interference colors, and occurs filling fractures mostly in the intercumulus.

During the SEM analyses other minerals of much smaller size were identified as: zircon, monazite, baddeleyite, and native nickel.

Fig. 2. – FETIX-1 sample image observed under transmitted light microscopy obj. 4x in plane polarized light. Cumulatic texture of magnetite-ilmenite-spinel. Spinel is the colourless to pale green mineral with brownish rims; Magnetite-ilmenite the black opaque. Minor phases are mainly corundum, clinochlore and zircon.
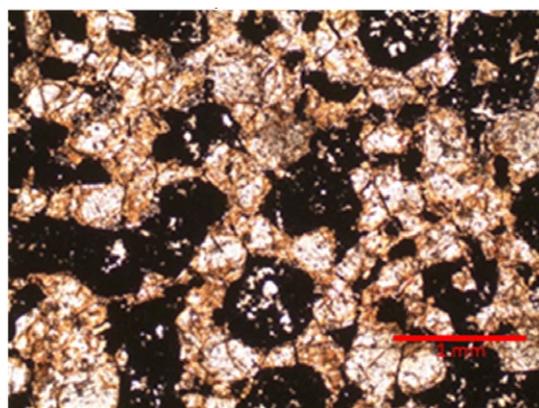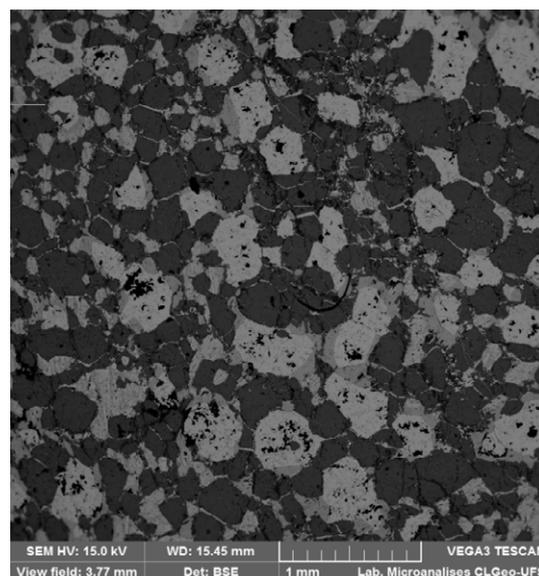

Fig. 3. – View of cumulatic texture in sample FETIX-1. Back-scattered electrons SEM image.

Zircon, monazite and baddeleyite occur mainly in the intercummulus or occasionally inclosed in spinel. The native nickel grains were observed inclosed in spinel. Backscattered electron images showed

brilliant small grains inclosed in spinel (Figures 6 and 7). EDS analyses (Figure 8) of these grains revealed a surprising result of close to 90% nickel. The analyses were redone in folowing SEM sessions months apart always with the same results. The native nickel grains in spinel are usually very small, with maximum size of 8 x 4 μm (Figure 7) and much smaller (~1 μm). They appear anhedric and with rounded edges, and sit deep inside spinel crystals. In the case of the grain shown in Figures 6 and 7, it does not sit directly in a major fracture of the spinel crystal, but quite close to some, like the small one on the upper left corner of the crystal (Fig. 7), and is quite inside the spinel without reactive rims or other indications of disequilibrium.

Fig. 4. – Trellis texture of exsolution of ilmenite in magnetite. BSE image from SEM. Light grey – magnetite; dark grey – ilmenite; dark – spinel.
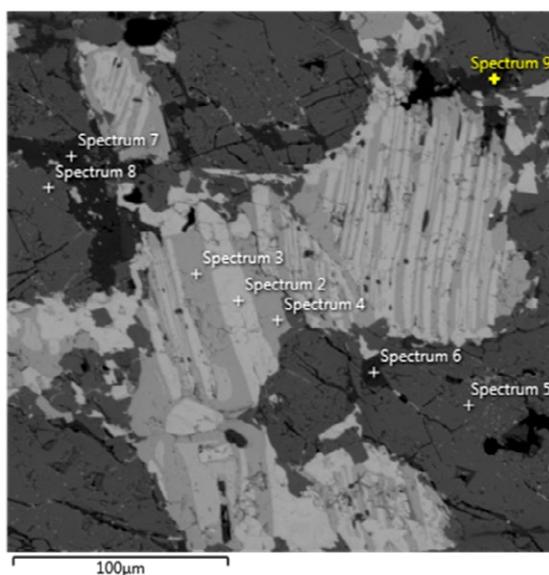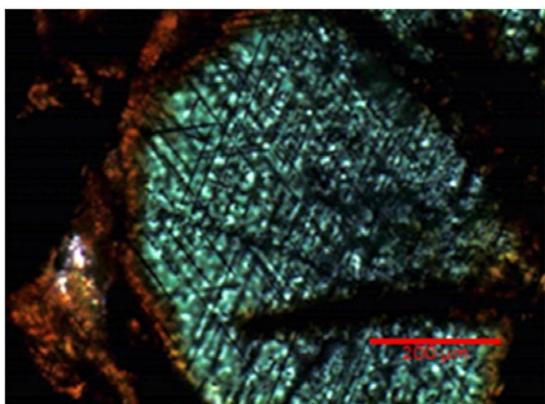

Fig. 5. – Spinel observed in plane polarized light obj. 40x in transmitted light microscopy of sample FETIX-2 exhibiting exsolutions of Fe oxides in {111} planes.

SEM analysis also aided in establishing the following paragenesis: magnetite - ilmenite- spinel - magnetiteII?-ilmeniteII? - baddeleyite – zircon, corundum and clinochlore. It is difficult to place the native nickel in this paragenesis, but we believe it occurs as an exsolution of spinel in a post-magmatic stage, as we discuss below.

Table 1. – Analytical data in Ox% for spinel pleonaste, maximum, minimum, mean and standard deviation for 26 analysis.

Ox%	Max	Min	Mean	Sd
Al	81.90	31.50	61.70	11.11
Fe	63.00	5.20	20.86	9.54
Mg	31.90	3.40	13.29	4.87
Ce	5.50	5.50	5.50	-
La	2.80	2.80	2.80	-
Nd	2.40	2.40	2.40	-
P	4.50	4.50	4.50	-
Si	28.40	1.00	14.70	19.37
Ti	24.80	0.40	7.64	7.56
Zr	0.70	0.70	0.70	-
Mn	0.80	0.80	0.80	-

Fig. 6. – Backscattered electrons image of native nickel particle (marked with circle) enclosed in spinel surrounded by magnetite and ilmenite, in FETIX-1 sample. Mag – magnetite; Spl – spinel; Ilm – ilmenite.
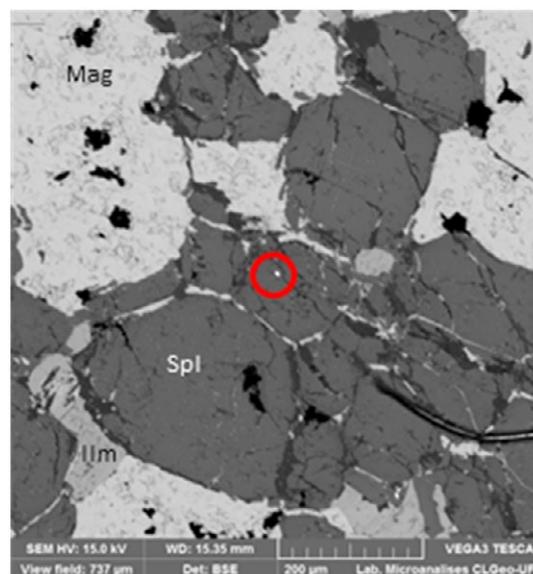
Fig. 7. – Backscattered electrons image of native nickel particle in FETIX-1 sample. Noticeable are the shape, size and edges of the Ni particle.
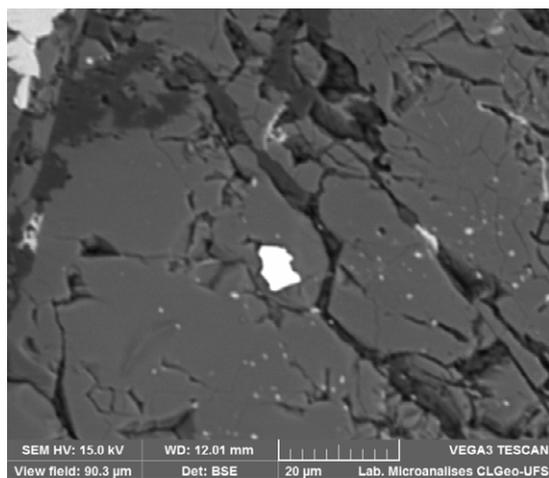

Table 2. – Analytical data in wt% for native Ni, maximum, minimum, mean and standard deviation for 29 analysis.

Wt%	Max	Min	Mean	Sd
Ni	92.15	89.94	91.21	0.49
Fe	5.87	4.30	4.78	0.37
Zn	4.72	2.68	3.94	0.53
S	0.22	0.21	0.22	0.00
Mo	0.64	0.54	0.59	0.07

In total twenty-nine analyses by EDS (Fig. 8) were conducted and the analyses revealed Ni and Fe as the main elements present (Table 2). In most of the analyses several other elements were detected, and there is most certainly a contamination due to the surrounding or nearby minerals (spinel and clinochlore) due to the grain size. The conspicuous presence of Si, O, Mg, and Al could be related to interaction with clinochlore present in fractures of the spinel or, in the case of Al, Mg, and O, with the enclosing spinel itself. Thus, the high values for Al (4,9 wt%) initially found are, we believe, the result of influence from the enclosing spinel due to the Ni particle size, and were discarded. Br and F are possibly contaminants or result from peak identification problems, for example of Al or Fe (Newbury, 2009). Mo, S, and Zn, were retained due to their chalcophile/siderophile nature. We thus proceeded to eliminate those elements which we considered foreign to the mineral, and recalculated the values. Table 2 shows mean and standard deviation

data for the twenty-nine analysis made, which shows Ni with mean 91.21 wt%, with a maximum of 92.15 wt% and a minimum of 89.94 wt%, and standard deviation of 0.49. The other elements occur as minor (Fe 4.78 wt%, Zn 3.94 wt%) or trace presences (Mo 0.04 wt%, S 0.03 wt%).

Fig. 8. – Spectrum of the SEM-EDS analysis point 14 of the native nickel.
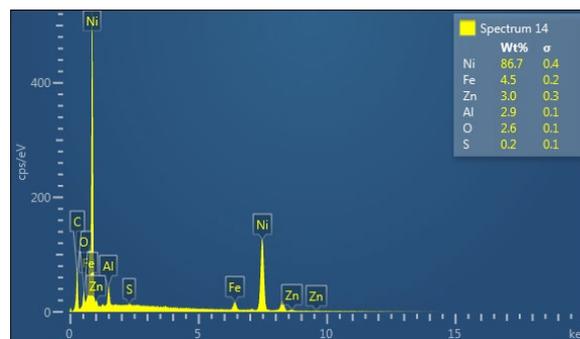
Stoichiometry of the analyses results make us consider this mineral as native nickel because, the other chemically closest mineral, awaruite, has a much lower Ni and much higher Fe content in the analyses described in the literature (Radhakrishna *et al.*, 1982; Anthony *et al.*, 1990; Klein & Bach 2009).

5 Discussion

The presence of native nickel in the Canindé magnetitic cumulates presents a conundrum in relation to its origin. Some works (Nickel, 1959; Ramdohr, 1969; Eckstrand, 1975; Radhakrishna *et al.*, 1982; Bai *et al.*, 2000; Klein & Bach, 2009; McDonald *et al.*, 2010) refer the origin of awaruite (Ni_3Fe), a mineral compositionally similar to native nickel, as a product of serpentinization of ultramafic rocks such as peridotites. Others refer to native nickel of primary origin (Van Roermund *et al.*, 2000), or resulting from hydrothermal alteration of Ni sulphides (Ramdohr, 1969; Hudson & Travis 1981; Bai *et al.*, 2000; Correia de Brito *et al.*, 2005; McDonald *et al.*, 2010), or also of native nickel flakes generated by detrital reconcentration of previously decomposed awaruite (Challis, 1975).

Detrital concentration of native nickel flakes, as the by-product of altered serpentinites which carried awaruite, is not the present case, so this hypothesis can be safely discarded.

We thus end up with three hypothesis:

The native nickel grains are of primary origin – this would imply fO_2 below or close to Ni metal saturation conditions, which depend on fO_2, p, T and Fe-Ni content. The proto-magmatic nature of the Fe-Ti cumulates as well as the fact that the native nickel grains are deep inside the core of spinel crystals support this hypothesis. The fact that there is no reactive rim in the observed Ni grains, indicates that no late stage re-equilibration occurred, which questions a primary origin for these native nickel grains, as they would most certainly be high pressure phases subject to reequilibration (Van Roermund et al., 2000). The native nickel grains result from retrograde metamorphic processes - The igneous origin of most Fe-Ti layered deposits is widely accepted (Charlier et al., 2015). But it is not uncommon that retrograde metamorphic processes rework these layers (Amcoff & Figueiredo, 1990). Retrograde metamorphic hydration (serpentinization) has a strongly reducing nature which favours the occurrence of native metals (Nickel, 1959; Klein & Bach, 2009). In this case, native nickel is frequently the product of oxidation or desulfidation of a precursor phase (Ramdohr, 1969; McDonald et al., 2010). In our samples we observed the characteristic trellis textures of magnetite-ilmenite exsolution (Fig. 5) which could either represent a true exsolution resulting from a simple cooling of a solid solution in a late magmatic stage (Ramdohr, 1969; Amcoff & Figueiredo, 1990), or a process of exsolution by oxidation in which a rise in the fO_2 causes destabilization, which could also affect the exsolution of Ni rich phases in spinel (Nickel, 1959; Figueiredo, 2000), bearing in mind we also observed exsolutions in spinel, believed to be mostly of iron oxides, along {111} planes (Fig. 7). We think a combination of a higher grade facies (amphibolite to granulite) followed by lower grade facies (lower serpentinization greenschist), or a combination of these similar to a hornfels facies of metamorphism, could have affected these rocks. Further on the alteration path, the peripheral decomposition of spinels is evidenced by zoning (brownish zones at crystal rims), presence of corundum peripheral to spinel crystals, and later formation of clinochlore in a final hydrothermal phase. Reducing regimes of low fS_2, fO_2, pH, in a possibly Ni enriched spinel, would promote native nickel as a stable phase. The observations and analyses made in the studied samples do not permit us to be conclusive about this hypothesis.

Lastly, the native nickel grains are the result of hydrothermal activity – this process, which would imply a precurssor sulphide or alloy phase, has been described by some authors (Ramdohr, 1969; Hudson & Travis, 1981; Bai et al., 2000; McDonald et al., 2010). The presence of possibly Ni-rich sulphides (pyrite and chalcopyrite) in the Fe-Ti cumulates of CGC has been previously reported (Seixas & de Moraes, 1996; Santos et al., 2001), but we have not found any of them. Ni rich sulphides (pyrite, pyrrhotite, pentlandite, violarite, chalcopyrite) do occur in the CGC gabbros adjacent to the Fe-Ti cumulates (Damasceno, 2017).

Hydrothermalism surely played a part at a later stage but we have not found the mineral assemblages one would expect from this process to generate native nickel.

Observing that: the native nickel crystals occur inclosed in spinel; the shape of the native nickel crystal with rounded solid edges and no reactive rims were observed; the observed trellis textures of magnetite-ilmenite exsolution probably represent a process of exsolution by oxidation, which could also affect the exsolution of Ni-rich phases; later hydrothermal reactions occurred, evidenced by the presence of clinochlore; and the mineral assemblages present;

We thus presume that these native nickel grains are the result of events which occurred during the final stages of the genesis of the present cumulate, after the magmatic stage, and as exsolutions facilitated by retrograde metamorphism and coupled with prevailing hydrothermalism. We believe this composite hypothesis to be substantiated, given the mineral paragenesis, the chemical analyses, the observed textures and habits, and the similarities with findings described by other authors.

Deposits which have similar, although not identical, features have been described in the northeastern region of Brazil, like the Rio Jacaré Sill in Bahia state or the Serrote da Laje deposit in Alagoas state (Amcoff & Figueiredo, 1990; Figueiredo, 1992; Correia de Brito et al., 2005), but no native Ni has been identified in these deposits, only awaruite (Ni_3Fe) in the Rio Jacaré Sill. The particular paragenesis of the CGC Fe-Ti cumulates does not seem to be exactly ascribable to other Fe-Ti deposits worldwide (Figueiredo, 1995; Charlier et al., 2015).

6 Conclusions

The Fe-Ti cumulates of the CGC have a rich mineralogy with the presence of some rare minerals. We have been able to identify amongst others native nickel grains which occur embedded in spinel. This occurrence is notable for being, according to our

knowledge, one of only five other cited worldwide for native Ni and the first one in Brazil. The Ni grains have a composition almost exclusively of Ni with minor Fe content. The genesis of these native nickel crystals is not yet precisely ascertained, although we presume as a reasonable hypothesis that they are the result of exsolution from spinel facilitated by retrograde metamorphism and hydrothermal processes. Further work on these ores is being carried out which will contribute to a better understanding of this and other questions on the Fe-Ti cumulates of the Canindé Domain.

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