



Physical and Chemical properties of Fluid and Melt Inclusions of the Lagoa Real Uraniferous Albitites (Brazil)

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Abstract

Data of melt and fluid inclusions obtained by LA-ICP-MS and microthermometry techniques represent an important investigation complement to understand geological processes which took place in Lagoa Real uraniumiferous albitites (Brazil). Melt inclusions found in augite of preserved magmatic portion (syenite) of the albitites are richer in Na, Al and Ti than augite itself. These elements belonged to the magma when augite crystallized and were further used by albite (Na and Al) and uraniumiferous titanite (Ti) during syenite crystallization processes. There is radiogenic Pb in the magmatic augite structure, which reveals the previous presence of U in the syenitic magma. Primary fluid inclusions in magmatic augite of the albitites contain Na, denoting once more its presence in original magma. The formation of andradite from augite during shear events that generated the metamorphosed syenite (uraniferous albitite) was certified by the ICP-MS signals and uranium released by magmatic titanite (U source mineral) during the 1.9 Ga metamorphism was recorded in the fluid inclusions found in andradite, mineral that was formed in this same metamorphic event which recrystallized titanite crystals. Such uranium was responsible by precipitation of the disseminated uraninite found inside andradite.

Keywords: Fluid and melt inclusions, Lagoa Real albitites, microthermometry, LA-ICP-MS.

Resumo

Dados físico-químicos de inclusões fluidas e fundidas obtidos por LA-ICP-MS e microtermometria representam importante complemento de investigação na compreensão dos processos geológicos que ocorreram nos albititos uraníferos de Lagoa Real (Brasil). Inclusões fundidas encontradas em augita da parte magmática preservada (sienito) dos albititos são mais ricas em Na, Al e Ti que a própria augita. Estes elementos pertenciam ao magma quando a augita cristalizou-se e foram utilizados pela albite (Na e Al) e titanita uranífera (Ti) durante os processos de cristalização do sienito. Há Pb radiogênico na estrutura da augita magmática, o que revela a presença de U no magma sienítico original. Inclusões fluidas primárias em augitas magmáticas dos albititos contêm Na, denotando mais uma vez sua presença no magma original. A formação de andradita a partir da augita magmática durante os eventos de cisalhamento que geraram os sienitos metamorfisados (albititos uraníferos) foi certificada pelos sinais do ICP-MS e o urânio cedido pela titanita magmática (mineral-fonte de U) durante o metamorfismo de 1,9 Ga foi registrado nas inclusões fluidas encontradas na andradita, mineral que se formou neste mesmo evento metamórfico que recrystalizou a titanita. Tal urânio foi responsável pela precipitação da uraninita disseminada que é encontrada no interior da andradita.

Palavras-Chave: Inclusões fluidas e fundidas, albititos de Lagoa Real, microtermometria, LA-ICP-MS.

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1. Introduction

Plant et al. (1999) point out that many uranium rich provinces are related to evolved felsic igneous rocks intruded in the crust, not only anorogenically, but also during the final stages of orogenesis. Furthermore, according to Bonin (1987), during the late orogenic stages, ductile shear fault zones control the site of emplacement of felsic magmatic provinces, some of them with associated uranium. Uranium mineralization at Lagoa Real is found as finely disseminated (micro to millimetric) uraninite associated with mafic minerals (mainly garnet) belonging to discontinuous tabular bodies of albitites located along shear zones (Geisel Sobrinho, 1981; Ribeiro et al., 1984; Costa et al., 1985; Turpin et al., 1988; Lobato & Fyfe, 1990; Cordani et al., 1992).

Geochronological studies related to Lagoa Real uraniumiferous albitites have been done by Chaves et al. (2007). LA-ICP-MS U-Pb dating of zircon and uraninite of the albitites revealed protolith (uraniferous sodic syenites) crystallization and simultaneous deformation around 1.9 Ga along shear zones active during the final stages of the Orosirian Orogeny. Disseminated uraninite found inside garnet initially crystallized during 1.9 Ga metamorphism from U released by uraniumiferous titanite of the syenite. This article aims to show that physical and chemical properties of fluid and melt inclusions from uraniumiferous albitites, specially the presence of Na, Al, Ti, and U in the original syenitic magma, can support this geological scenario.

2. Geological setting of the Lagoa Real Uranium Ore Bodies

The Lagoa Real region is located in the central-southern part of São Francisco Craton (Fig. 1). The basement of this region is formed of Archean/Paleoproterozoic granulitic, migmatitic, and gneissic

rocks, which belong to the Paramirim and Gavião blocks (Inda & Barbosa, 1996). The Ibitira-Brumado volcanosedimentary unit is found in the area and comprises amphibolites, banded iron formations, gneisses, metacherts, marbles, and schists. Mascarenhas (1973) interpreted this unit as a Lower Paleoproterozoic greenstone belt.

The Paleoproterozoic Lagoa Real Granitic-Gneissic Complex covers an area larger than 2,000 km² of the Paramirim Block and includes granitoid bodies, gneisses, albitites and amphibolites. Differently from Chaves et al. (2007), Maruéjol (1989) assigned the genesis of the uranium-bearing albitites to metamorphism and progressive deformation of the 1.75 Ga anorogenic São Timóteo Granite along shear zones, where a episyenitization process took place under the influence of uranium and sodium-rich hydrothermal fluids.

Another important geological unit in the region is the Espinhaço Supergroup, comprising sandstones, conglomerates, siltstones, shales, quartzites and schists overlying a sequence of 1.7 Ga rhyolites and rhyodacites. This supergroup is related to a basin developed during Upper Paleoproterozoic rifting event. Tertiary covers and Quaternary alluvial sediments complete the geological scenario of this region (Fig. 1).

According to Almeida (1977) and Cordani & Brito Neves (1982), the geological and tectonic context of the Lagoa Real region is part of the evolution of the São Francisco Craton and of successive geological cycles: Jequié (Archean - with orogenic event around 2.7 Ga), Transamazonian (Paleoproterozoic – with Orosirian orogenic event between 2.05 and 1.8 Ga), and Brasiliano (transition Neoproterozoic/Phanerozoic - with orogenic event around 0.54 +/- 0.1 Ga). During the latter cycle, Archean gneissic basement overthrusts metasedimentary rocks of the Espinhaço Supergroup and therefore N-S regional thrust faults are found

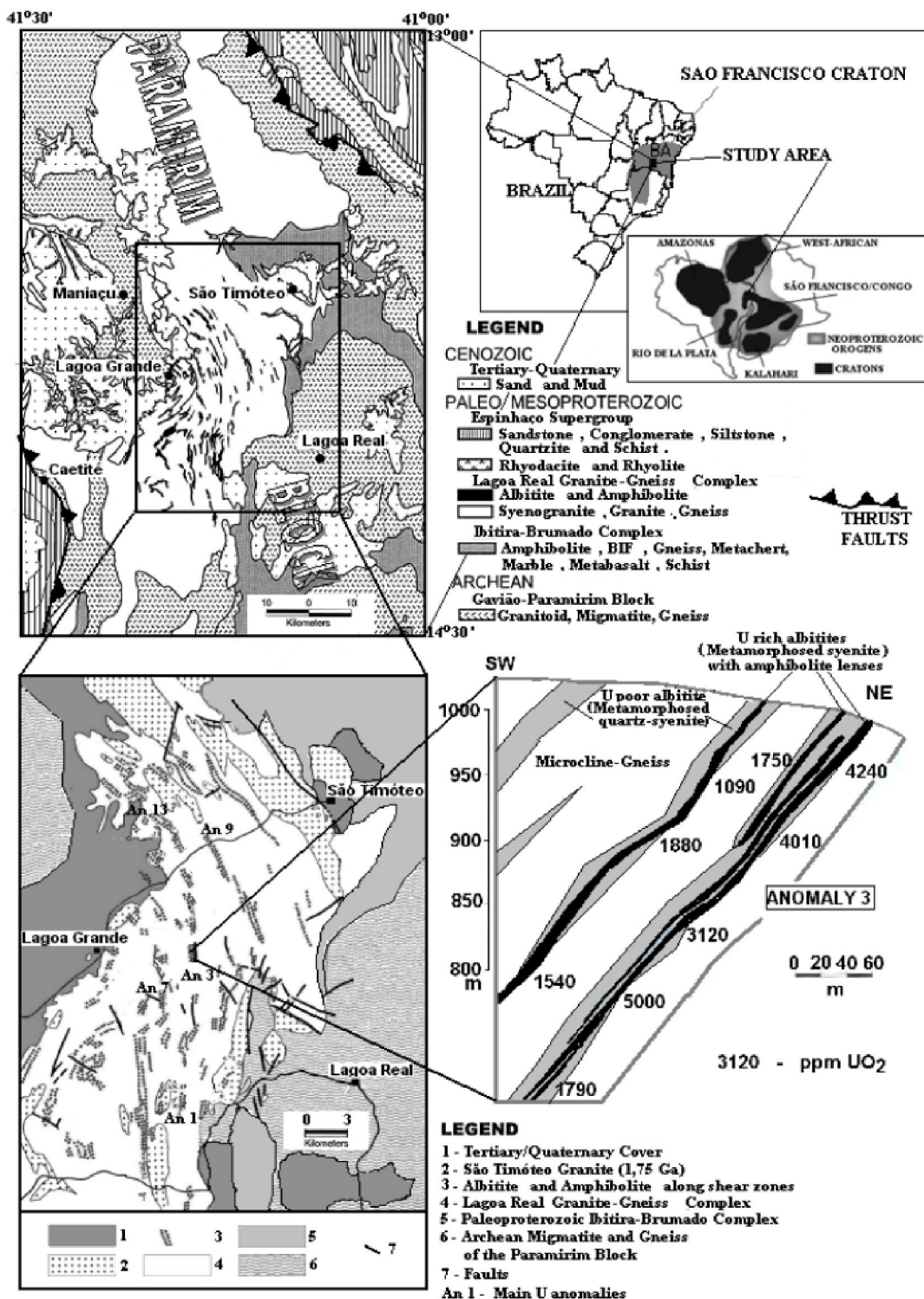


Fig. 1 - Geological setting of the Lagoa Real uraniferous albitites, Bahia (BA-Brazil). Modified from Pascholati et al. (2003) and Costa et al. (1985).

in the Paramirim Block (Caby & Arthaud, 1987).

As already mentioned, uranium mineralization at Lagoa Real is found as finely disseminated (micro to millimetric) uraninite

associated with discontinuous tabular bodies of albitites located along shear zones. According to Costa et al. (1985), most bodies trend N40E to N30W and dip 30° to 90° to the southwest or northwest, with the exception of the northern most

deposits, which dip to the east, and those situated in the central part of the region, which are almost vertical. Each body has maximum length of 3 km and average width of 10 m (max. 30 m). Mineralization extends up to 850 m below the surface as shown by drill cores. Bodies contain one or more mineralized levels, which may be interrupted in places. The contacts between mineralized levels with host gneissic rocks is transitional or eventually abrupt. Amphibolites often occur along tabular bodies of albitites with the same structural trends and are also affected by shear zones.

Reasonably assured reserves of the Lagoa Real Uraniferous Province are presently 94,000 tons of UO_2 and 6,700 tons of UO_2 are inferred reserves (CPRM/CBPM, 2003). Figure 1 inset shows a representative albitite vertical section, which presents UO_2 contents in ppm for some mineralized levels of the anomaly 3. These contents are similar to the main uranium anomalies of the province (between 1000 and 5000 ppm).

3. Methodology

In order to understand the physical and chemical properties of the fluid and melt inclusions associated with the Lagoa Real uranium mineralization, the following initial steps were undertaken: (1) field work for geological survey and sample collecting from Cachoeira Mine pit (anomaly 13) and drill-core rocks from anomalies 1, 3, 7, and 9 of the Lagoa Real Uraniferous Province (Fig. 1); (2) preparation of polished thin sections in Sample Laboratory of Center for Development of Nuclear Technology (CDTN); (3) mapping of fluid and melt inclusions in some mineral phases in Fluid Inclusions and Metallogenesis Laboratory (LIF) of CDTN. A Leica DMR-XP microscope was used.

After that, microthermometric studies were carried out in LIF by using Chaixmeca heating/freezing system stage adapted to Leica DMR-XP microscope. Finally, analyses of the chemical contents of fluid and melt inclusions in some minerals of the paragenesis associated to the uranium mineralization of Lagoa Real were performed by using

the LA-ICP-MS technique (Laser Ablation Inductively Coupled Plasma Mass Spectrometry, reported by Sylvester, 2001) with standard NIST 610 Glass Reference Material.

The coupled Laser Ablation (Cetac/Geolas-Pro - operating wavelength 193 nm, energy density $40J/cm^2$ with spot size of 20 micrometers) and ICP-MS (Thermo/Element2 - sensitivity 1×10^9 cps/ppm In, mass resolution 600, 8000, 20,000 FWHM, magnetic scan speed m/z 7 \rightarrow 240 to 7 $<$ 150 ms, signal stability better than 2% over 1 hour) instruments used in this study are installed at the Memorial University of Newfoundland, St. John's - Canada.

4. Petrography, Metamorphic reactions, and Uraninite formation

After Chaves et al. (2007), the term albitite represents two distinct petrographic types. Both are rich in albite, as the name indicates, and are closely related to ductile shear zones. The first one - study target - is a metamorphosed syenite without quartz but with associated uranium mineralization. The second one is a metamorphosed quartz-syenite. The second type differs from the first one due to its quartz, higher potassic feldspar content and lower volume of accessory minerals, and rare associated uranium mineralization. The mineralogy is nearly the same for both petrographic types.

The explanation for the quartz-free rocks being syenites and not hydrothermal albitites (epysyenites) as previously suggested by Marujol (1989) was found during micropetrographic studies developed by Chaves et al. (2007), which revealed a strong anisotropy in the metamorphic foliation generated during ductile shear development. There are portions of the rock where the texture and the mineralogy of magmatic stage are preserved, including antiperthites and zoned augites. Other ones mix magmatic and metamorphic textures and many others have exclusively granoblastic texture. Therefore, the transformation of the magmatic stage minerals during metamorphism up to their complete recrystallization is evident. Besides the recrystallized minerals, new ones also resulted from metamorphic

reactions. In preserved portions of the magmatic stage both quartz and features resembling silica dissolution were not found. Since quartz is absent in these portions, there is no reason to believe that quartz-rich São Timóteo Granite underwent sodic metasomatism to generate albitites.

In portions that preserve the magmatic stage, albite (50% to 70% of rock volume), iron-rich augite (20% to 40%) and some microcline (up to 15%) are found as essential members and this composition classifies the rock as syenite. Accessory minerals of the magmatic portions are dark brown uraniferous titanite (titanite crystals with high uranium concentrations have been reported by Gregory et al., 2005 and Pimentel et al., 1994, and can be understood by the replacement between Ti^{4+} and U^{4+} , which have similar ionic radius), allanite-Ce, magnetite, fluor-apatite, zircon, fluorite, and apophyllite. Magmatic calcite is sometimes present, which can be found inside undeformed augite crystals.

In a first metamorphic stage, under high-grade amphibolite facies, not only hastingsite, but also andradite (Ca and Fe^{3+} garnet) resulting from iron-rich augite transformation appeared. At that moment, andradite became part of the metamorphosed syenite, rock formed through recrystallization of iron-rich augite, albite, microcline (+/- calcite), and accessory minerals. During recrystallization, iron-rich augite became aegirine-augite (more sodic) and albite became oligoclase (slightly more calcic). The association between oligoclase and andradite reveals high pressure metamorphism common to ductile shear zones (Yardley, 1989). Magnetite was replaced by hematite. Therefore, the mineralogical transformations reveal the rising of Fe^{3+} during metamorphism.

Uraninite, whose uranium derives essentially from magmatic uraniferous titanite, was also formed. It was precisely from the transformations imposed on U-rich titanite, not only during metamorphism related to shear processes but also during hydrothermal events, that uranium became available in both reduced (tetravalent uranium) and more

mobile oxidized forms (as uranyl ions – UO_2^{+2} , in which uranium is hexavalent) to take part in chemical reactions which led to the formation of uraninite.

Epidote and biotite appeared during a second metamorphism event. They partially replaced the minerals formed during the initial metamorphism. This paragenesis indicates re-equilibrium established under new temperature and pressure conditions, less intense than the ones that formed garnet during the initial metamorphism. It is interesting to notice that uraninite crystals are found inside epidote and biotite and also probably have precipitation related to Redox processes (both Fe^{2+} and Fe^{3+} appear in biotite while in epidote only Fe^{3+}). Uraninite precipitation inside these minerals, eventually with involvement of calcite, should have occurred under these new metamorphic conditions between greenschist and amphibolite facies.

5. Study of Fluid and Melt Inclusions by LA-ICP-MS and Microthermometry: Results and Discussions

Chemical content of fluid and melt inclusions in some minerals of the paragenesis related to the uraniferous mineralization of Lagoa Real was qualitatively analysed by LA-ICP-MS (Laser Ablation Inductively Coupled Plasma Mass Spectrometry). This technique has proven to be extremely effective in chemical studies of fluid and melt inclusions in minerals (Heinrich et al., 2003). The way of graphic interpretation of the ICP-MS signals of figures below is simple: from background, when signal of each chemical element is amplified initially it means that laser ablated host mineral. After that, when signal amplifies once more for any element it means that laser ablated melt or fluid inclusion, which is rich in such element.

The presence of iron-rich augite in the magmatic stage of metamorphosed syenites (uraniferous albitites) is confirmed by melt inclusions as well as by zoned structure found in some augite crystals. Melt inclusions are colorless with two-phases (vapour-V and solid-S) or pale brown monophasic (solid-S). The ICP-MS signals show that melt inclusions are richer in Na, Al and Ti than augite itself (Fig. 2).

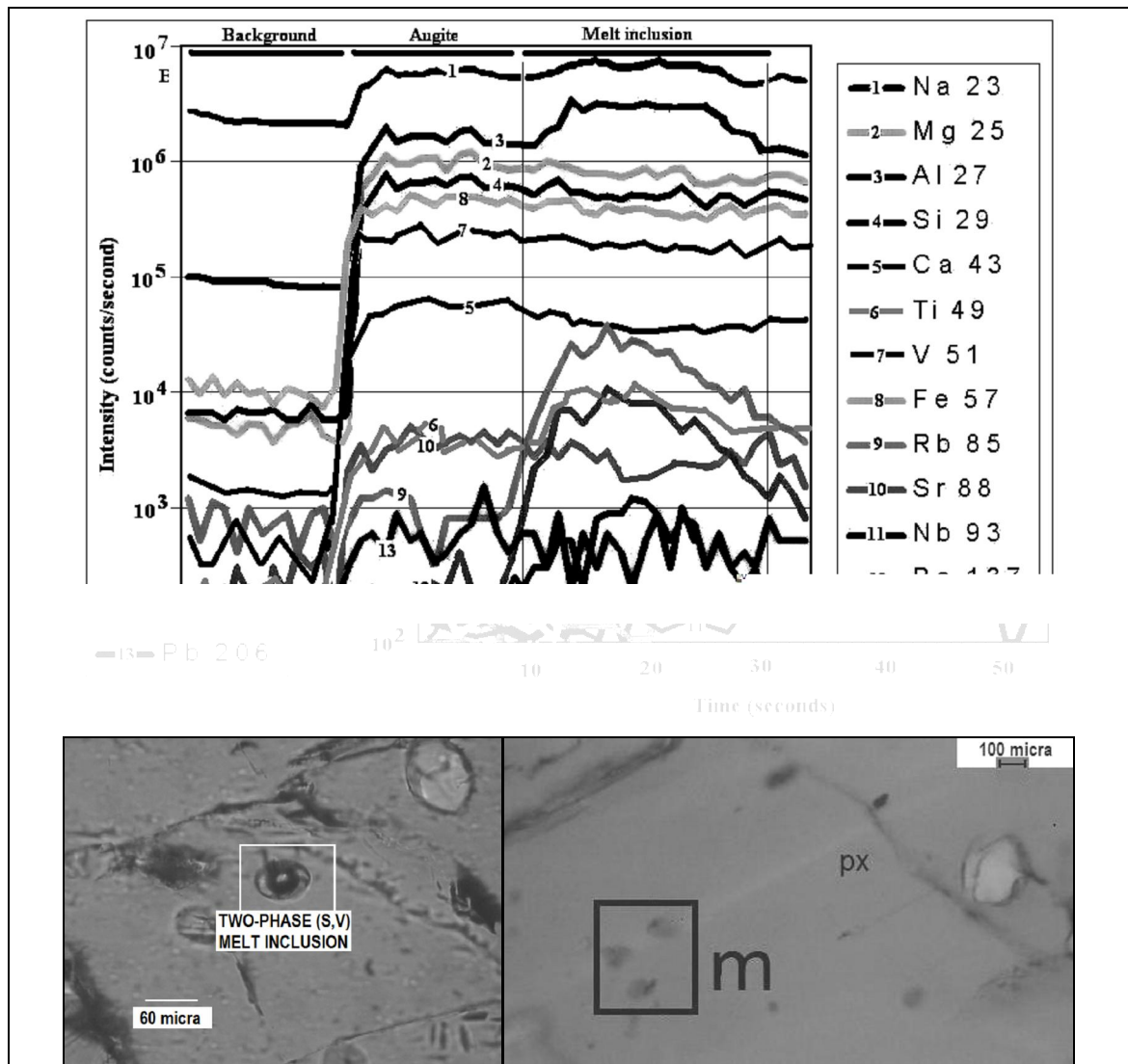


Fig. 2 - ICP-MS signals of magmatic iron-rich augite of syenite (uraniferous albitite) and of one of its melt inclusions, which shows two-phases (V - vapour and S - solid, see left photomicrograph). Pale brown monophasic melt inclusions (m) are also shown (px = augitic pyroxene, see right photomicrograph). Logarithmic intensity scale.

These elements belonged to the magma when augite crystallized and were further used by albite (Na and Al) and uraniferous titanite (Ti) during syenite crystallization processes. The melt inclusions also contain Nb, Rb, and Ba, which are incompatible in the main silicate minerals of the rock. It is interesting to note that the content of Ca and Sr is smaller in melt inclusions than in augite itself because these elements are compatible in the augite structure. There is some radiogenic lead in the augite structure, which reveals the previous presence of U in the syenite magma.

To understand the magmatic fluids, the primary three-phase fluid inclusions were analysed (solid crystalline phase-S, vapour phase-V, and aqueous phase-L) in augite crystals of the magmatic stage of the uraniferous metamorphosed syenite (uraniferous albitite). The diagram in Figure 3 shows that the primary fluid inclusion in magmatic iron-rich augite of the albitite contains Na, Rb, and Ba (as incompatible Rb and Ba did not fit in syenite minerals, they remained in the fluid phase). Microthermometric studies point to a very low eutectic temperature, between -69.7°C and -62.6°C (Tab. 1), which is probably caused by the presence

of Rb and Ba in this complex saline system, certainly of magmatic origin. Fluorine (inferred from the presence of fluorite in rock), rubidium, and barium would cause intense solvation with water molecules, resulting in very low eutectic temperatures.

Fluid inclusions in garnet and recrystallized apatite were also analysed in order to determine the fluids of the metamorphic stage that generated the metamorphosed syenites and their andradite with included uraninite. The fluid inclusions in garnet normally have either four phases (L, V, and two S – dark-orange and colorless) or three phases (L, V, and either colorless S or dark-orange S) and recrystallized apatite has either two phases (V-L) or one phase (L). It is worth pointing out that apparent solid (remnant melt?) inclusions were identified in recrystallized apatite.

The formation of andradite from iron-rich augite during shear events that generated the

metamorphosed syenites is certified by the ICP-MS signals presented in Figure 4. The elements Si, Ca, Ti, V, Fe, Na, Mg, Al, and Sr that make part of the structure of the magmatic augite were found in the structure of the garnet, with the exception of Mg and Na, which passed to the fluid phase (they did not enter in andradite structure). Relative to the fluid inclusions in garnet (Fig. 4), besides Mg and Na, it was also found Rb, Ba, U (U235 and U238) and associated radiogenic Pb.

Rb and Ba once more remained in the fluid phase, as they are incompatible in the andradite structure. According to microthermometry, the eutectic temperature between -53.5°C and -51.6°C is similar to that obtained from secondary fluid inclusions in augite crystals (Tab. 1) and is not far away from that of the magmatic stage of augite, probably indicating slight aqueous dilution of magmatic fluids in the metamorphic environment (an almost closed system?) yielded during 1.9 Ga shear event shown in Figure 5.

Tab. 1 - Microthermometric data of fluid inclusions in augite, andradite and apatite from Lagoa Real metamorphosed syenite (uraniferous albitite).

=1	AUGITE				ANDRADITE		APATITE	
	Primary fluid inclusions (yielded during magmatic stage)		Secondary fluid inclusions (yielded during metamorphic stage)		Fluid inclusions yielded during metamorphic stage		Fluid inclusions yielded during metamorphic stage	
	Initial ice-melting temperature ($^{\circ}\text{C}$)	Final ice-melting temperature ($^{\circ}\text{C}$)	Initial ice-melting temperature ($^{\circ}\text{C}$)	Final ice-melting temperature ($^{\circ}\text{C}$)	Initial ice-melting temperature ($^{\circ}\text{C}$)	Final ice-melting temperature ($^{\circ}\text{C}$)	Initial ice-melting temperature ($^{\circ}\text{C}$)	Final ice-melting temperature ($^{\circ}\text{C}$)
1	-64.0	-12.0	-52.5	-12.2	-53.0	-12.0	-49.5	-9.0
2	-65.4	-11.5	-52.3	-15.0	-52.1	-11.6	-51.4	-8.4
3	-63.8	-11.7	-52.7	-11.5	-52.0	-11.9	-51.7	-10.7
4	-62.6	-11.4	-55.0	-11.0	-52.1	-11.6	-50.0	-10.1
5	-64.4	-11.1	-50.9	-11.2	-53.5	-9.3	-49.9	-11.6
6	-65.5	-13.1	-52.2	-11.5	-53.1	-11.7	-52.4	-9.5
7	-64.2	-12.1	-52.0	-12.0	-51.7	-13.3	-51.0	-8.9
8	-66.2	-12.1	-55.0	-11.2	-52.5	-13.0	-53.7	-9.2
9	-69.7	-11.4	-54.9	-13.0	-51.6	-9.6	-50.2	-10.0

Uranium released by magmatic titanite (U source mineral) during the 1.9 Ga metamorphism was recorded in the fluid inclusions found in andradite, mineral that was formed in this same metamorphic event which recrystallized the titanite crystals. Therefore, this uranium was responsible for precipitation of the disseminated uraninite found inside andradite. Furthermore, the dark-orange saturation crystal found inside the fluid inclusions of andradite

(Fig. 4) probably contains radiogenic Pb from U of uranyl ions of the saturation crystal.

The material released during one of the laser ablations in recrystallized apatite produced the ICP-MS signals presented in Figure 6. The laser reached the apatite, two fluid inclusions, and a probable remnant solid inclusion. P, Ca, Sr, V, and the rare earths (La, Ce, Nd, Sm) make part of apatite. The rare earth elements and thorium are the main

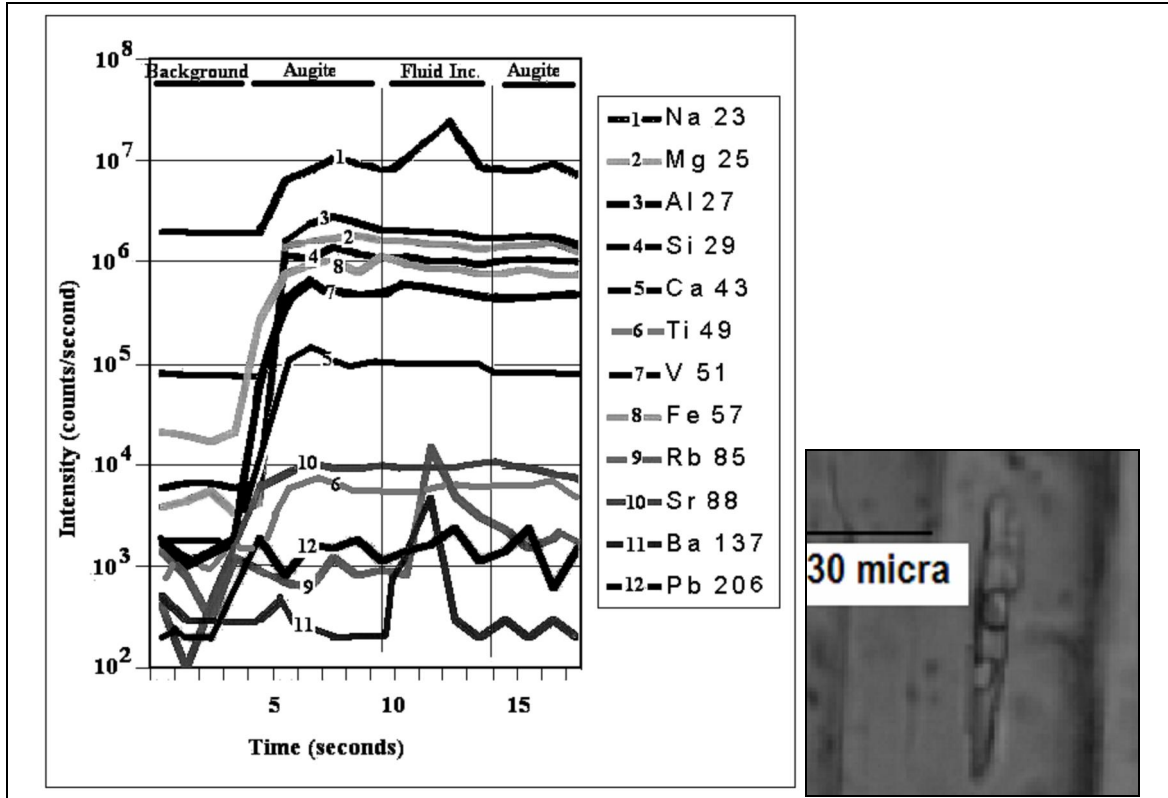


Fig. 3 - ICP-MS signals of magmatic iron-rich augite of the metamorphosed syenite (uraniferous albitite) and of one of its primary three-phase fluid inclusions (photomicrograph inset). Logarithmic intensity scale.

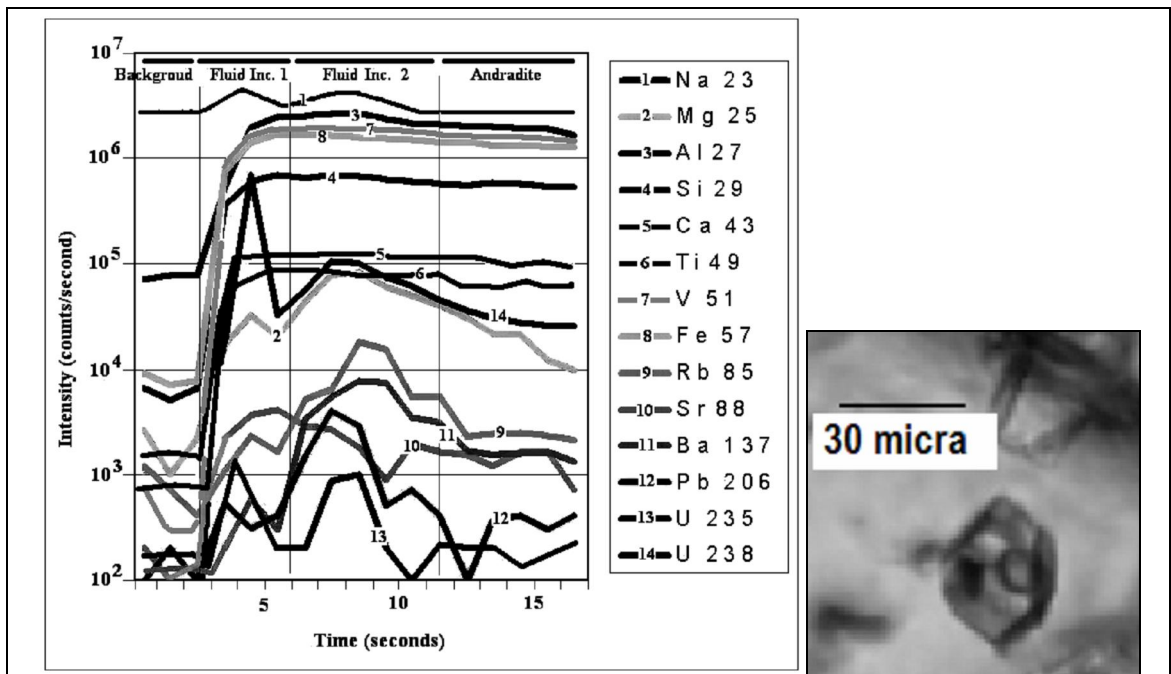


Fig. 4 - ICP-MS signals for andradite garnet of the metamorphic stage that generated the metamorphosed syenites (uraniferous albitites) and two of its fluid inclusions. One representative four-phase fluid inclusion (2 solid phases – one of them is dark-orange and the other one is colorless, 1 liquid phase, and 1 vapour phase, see photomicrograph inset) in andradite is shown in photo. Logarithmic intensity scale.

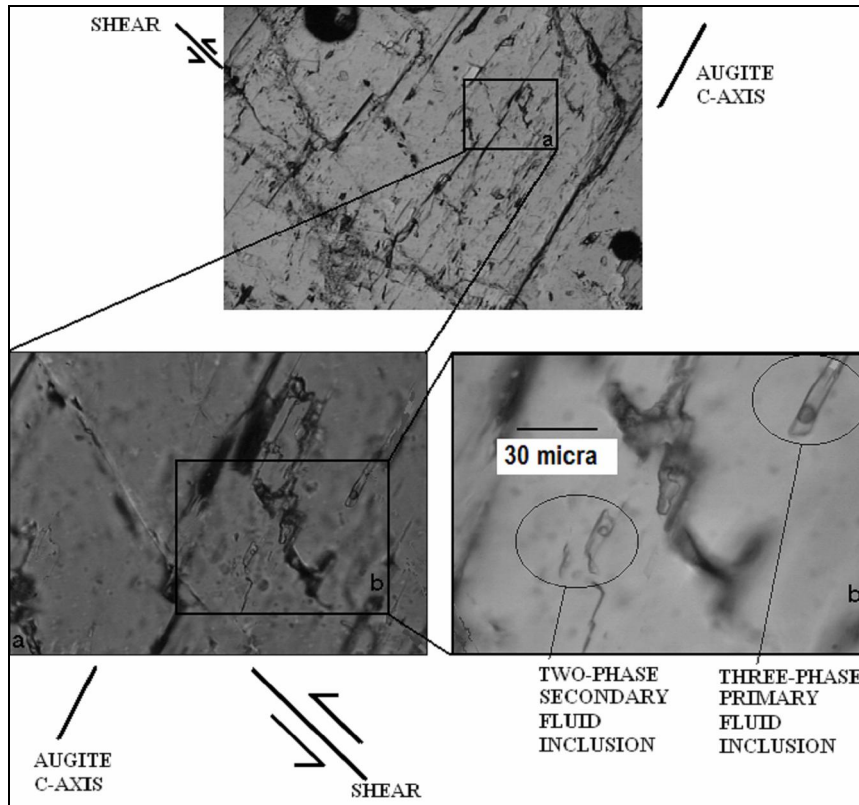


Fig. 5 - Primary and secondary fluid inclusions in augite partially affected by shear stress. The primary ones represent magmatic fluids and the secondary ones represent metamorphic fluids. The orientations of the black lines indicate shear and crystallographic c axis of augite, respectively.

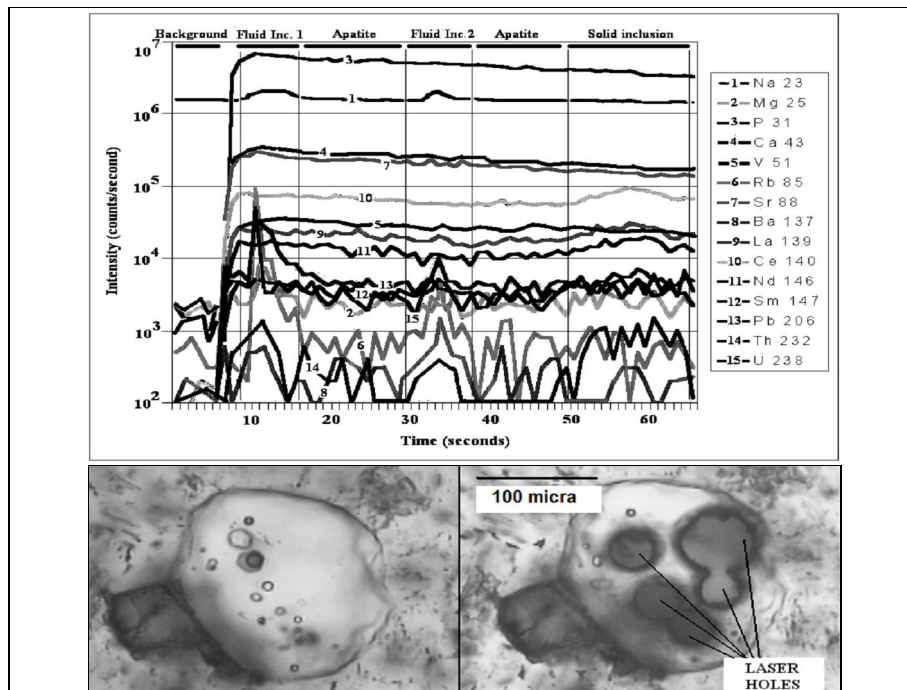


Fig. 6 - ICP-MS signals for recrystallized apatite of the metamorphic stage that generated the metamorphosed syenites (uraniferous albitites). The ICP-MS signals of two of its fluid inclusions and of a solid inclusion are also presented. Monophase and two-phase fluid inclusions in recrystallized apatite from a metamorphosed syenite before (left) and after (right) laser ablation are shown (photomicrograph inset). Logarithmic intensity scale.

constituents of the solid inclusion. The content of the fluid inclusions in recrystallized apatite is the same as those of the garnet fluid inclusions and eutectic temperatures between -53.7°C and -49.5°C are also the same (Tab. 1), which reveals that andradite was crystallized in the same metamorphic event responsible for apatite recrystallization.

6. Conclusions

Data of melt and fluid inclusions obtained by LA-ICP-MS and microthermometry techniques represent an important investigation complement to understand geological process which took place in Lagoa Real albitites. Melt inclusions found in magmatic augite are richer in Na, Al and Ti than augite itself. These elements belonged to the magma when augite crystallized and were further used by albite (Na and Al) and uraniferous titanite (Ti) during syenite crystallization processes. There is some radiogenic Pb in the augite structure, which reveals the previous presence of U in the syenite magma. Primary fluid inclusions in magmatic iron-rich augite of the albitite contain Na and, once more, it seems to be evident that sodium was in the original magma and this fact explains the composition of albitites, making the argument of sodic metasomatism unnecessary to explain albitite petrogenesis.

The formation of andradite from iron-rich augite during shear events that generated the metamorphosed syenites was certified by the ICP-MS signals and uranium released by magmatic titanite (U source mineral) during the 1.9 Ga metamorphism was recorded in the fluid inclusions found in andradite, mineral that was formed in this same metamorphic event which recrystallized titanite and apatite crystals. Such uranium lead to precipitation of disseminated uraninite found inside andradite. According to microthermometry of fluid inclusions in andradite, the eutectic temperature is similar to that obtained from secondary fluid inclusions in augite crystals and is not far away from that of the magmatic stage of augite, probably indicating slight aqueous dilution of magmatic fluids in the metamorphic environment (an almost closed system?) yielded during 1.9 Ga shear event.

Compositions and properties of studied melt and fluid inclusions seem to sustain the model proposed by Chaves et al. (2007) for the crystallization, metamorphism, and uranium mineralization of Lagoa Real albitites. However, melt and fluid inclusions in a large number of albitite minerals must be found and investigated to provide additional support to this model.

Acknowledgments

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