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(U-Th)/He Ages from the Fluorite Mineralization of the Tanguá Alkaline Intrusion

Idades (U-Th)/He das Mineralizações de Fluorita da Intrusão Alcalina de Tanguá

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Abstract

The Tanguá Alkaline Suite formed as a result of Mesozoic to Cenozoic magmatic events that affected the part of the South American Platform in Brazil's southeast. The alkaline intrusion is composed of alkali-feldspar syenite with nepheline (pulaskites and umptekites), syenite (with or without the presence of pseudo-leucite), varied nepheline syenites (foyaites, micro-foyaites etc.) and alkaline magmatic breccias. The intrusion contains important fluorite mineralization, in the form of NE-SW oriented veins, which were economically extracted in recent decades at the Emitang Mine. This paper focuses on the analysis of these centimetric crystals of fluorite which vary in color from (yellow, to white and purple.) associated with mineralization of Tanguá Body. Geochronological dating by (U-Th)/He of fluorite mineralization showed a wide range of ages, with the oldest age of 74 ± 3 Ma (Late Cretaceous) and the youngest of 0.11 ± 0.02 Ma (Late Pleistocene). Most ages are concentrated between 25 and 8 Ma (Miocene). The oldest age (74.0 ± 3 Ma) is associated with hydrothermal fluids percolating at the time of intrusion of the Tanguá Body. The other ages represent episodes of percolation of hydrothermal fluids and consequent growth of fluorite crystals. Of note is a set of Miocene ages that can be associated with regional tectonic events. This phase of flurite growth may be associated with the reactivation of basement faults and structures in the Tanguá region and the circulation of hydrothermal fluids associated with intrusion of younger dikes. The youngest ages may be associated with neotectonic reactivation of faults during neotectonic events.
Keywords: Tanguá Alkaline Intrusion; fluorite mineralization; (U-Th)/He Ages; Mesozoic to Cenozoic magmatism

Resumo

A Suíte Alcalina de Tanguá é representante das manifestações magmáticas meso-cenozoicas que ocorreram na Plataforma Sul-Americana na região sudeste brasileira. O corpo alcalino é constituído de álcali-feldspato sienitos com nefelina (pulaskitos e umptekitos), sienitos (com ou sem presença de pseudo-leucita), nefelina sienitos variados (foiaitos, micro-foiaitos etc) e brechas magmáticas alcalinas. Destacam-se ao longo da intrusão importantes mineralizações filoneanas de fluorita de orientação NE-SW exploradas economicamente nas últimas décadas na Mina Ermitang. As datações geocronológicas pelo método (U-Th)/He das fluoritas associadas a estas mineralizações apresentaram uma grande variação de idades, sendo a idade mais antiga de 74 ± 3 Ma (Cretáceo Superior) e a mais jovem de 0,11 ± 0,02 Ma (Pleistoceno Superior). A idade mais antiga (74,0 ± 3 Ma) é associada aos fluidos hidrotermais percolantes no momento da intrusão do Corpo de Tanguá. Porém a maior parte das idades concentram-se principalmente no intervalo 25 a 8 Ma (Mioceno), e, , provavelmente representam o principal estágio de mineralização da fluorita. Este estágio pode estar associado a reativações de antigas falhas/lineamentos presentes na região do Corpo Alcalino de Tanguá e eventuais percolações de fluidos hidrotermais associados a intrusões de diques que são observados ao longo da intrusão. As idades mais jovens poderiam representa os últimos estágios tectônicos registrados pela fluorita, e também,estariam possivelmente associadas à reativação neotectônica de falhas.
Palavras-chave: Intrusão Alcalina de Tanguá; mineralização fluorita; Idades (U-Th)/He; magmatismo Mesozoico Cenozoico

1 Introduction

Within Brazil, Mesozoic and Cenozoic magmatism is represented by basaltic and rhyolitic lava flows, tholeiitic dykes and by alkaline (plugs and stocks**;** Almeida, 1976, 1986; Almeida *et al.,* 1996; Thomaz Filho & Rodrigues, 1999). These magmatic manifestations are associated with two large events which affected the South American platform and according to Mota & Geraldes (2006) may be classified in the following way: early Cretaceous tholeiitic magmatism of the Paraná-Etendeka magmatic event, associated with the break-up of Gondwana and with the opening of the South Atlantic Ocean; and later alkaline magmatism linked to Cenozoic uplift of the Serra do Mar and with the development of the Continental Rift of Southeastern Brazil and associated basins. Published geochronogical data indicates that the earlier tholeiitic magmatism peaked at 132 Ma with ages concentrated between 119 and 139 Ma (Almeida *et al.,* 2013). This tholeiitic magmatism has been linked to the early evolution of southeastern Brazil's offshore basins.

Within the Mantiqueira Province of southeastern Brazil, this tholeiitic magmatism was followed by Late Cretaceous to Cenozoic alkaline magmatism, represented by a alkaline intrusions, which are composed of mainly syenites, nepheline syenites and their textural variations.A number of these intrusions are aligned along the NWW-SSE trending Poços de Caldas - Cabo Frio magmatic alignment (Almeida, 1983,1986; Thomaz Filho & Rodrigues, 1999; entre outros) or Cabo Frio Magmatic Alignment Province **(**Almeida, 1991; Riccomini *et al.,* 2004), as shown in Figure 1.

Riccomini *et al.* (2004) showed that numerous alkaline bodies within the central portion of the Mantiqueira Province occur along the edge of Neoproterozoic shear zones which were reactivated as normal or strike-slip faults from the Paleogene onwards, leading to the development of the Continental Rift of Southeastern Brazil (CRSB), as defined by Riccomini (1989). Absolute dating of these Paleogene to recent reactivations is generally only possible when movement is accompanied by the intrusion of igneous rocks. When this is not the case, relative dating is resorted to, through the comparison of fossil records in adjacent sedimentary basins. However, recent advances in low temperature dating techniques has allowed are fining of the timing of the alkaline magmatism and cenozoic to recent reactivations of Neoproterozoic shear zones in southeastern Brazil.

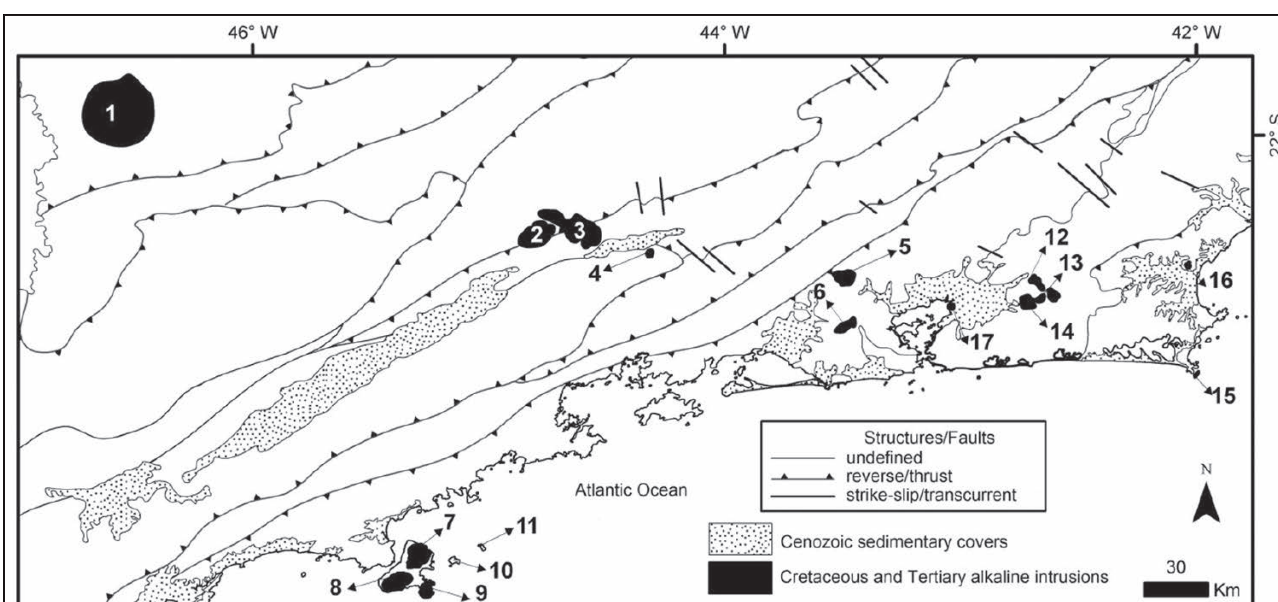

Figure 1 Poços de Caldas - Cabo Frio Magmatic alignment. 1- Poços de Caldas; 2- Passa Quatro; 3- Itatiaia; 4- Morro Redondo; 5- Tinguá; 6-Mendanha; 7- Serraria; 8- São Sebastião; 9- Mirante 10- Ilhas dos Búzios; 11- Ilha de Vitória; 12- Soarinho; 13- Rio Bonito; 14- Tanguá; 15- Ilha de Cabo Frio; 16- Morro de São João; 17- Itaúna.

Dating by the (U-Th)/He technique has been significantly improved during the last ten years and its recent use in flourite thermocronology of is particularly promising (Evans *et al*., 2005 a, b; Pi *et al*., 2005: Tritlla & Levrese, 2006). Fluorite records the last moments of circulation of mineralizing hydrothermal fluids, as such, ages obtained by this method represents the minimum age of hydrothermal activity. Evans *et al* (2005a) has suggested that this method can be used to detect different stages of fluorite mineralization.

This paper presents the results of (U-Th)/He dating of fluorites from the Tanguá Alkaline Intrusion, one of the alkaline intrusions that lies along the Poços de Caldas - Cabo Frio alignment. The fluorite mineralization from Tanguá includes economically important lode deposits (*e,g* the Emitang Mine) and according to Becker *et al.* (1997) these deposits are associated with country rock alteration by fluids of meteoric origin penetrating to depth along faults and fractures. These solutions were heated at depth, resulting in the leaching of the silica, fluorine and calcium from the country rock, which were then precipitated as fluorite and chalcedony when the fluids ascended to the surface. However, fluorite mineralization may also be associated with the circulation of hydrothermal fluids during the initial intrusion of the syenites of the Tingua Alkaline, or associated with hydrothermal fluids associated with the later emplacement of trachyte and phonolite dykes and sills, and possible re-activation of basement faults.

Becker *et al.* (1997) indicated that the lode deposits of Tanguá present several generations of fluorite, as such the (U-Th)/He method is an useful tool for identifying and dating the different phases of mineralization.

2 Methodology: (U-Th)/He Thermochronology

Evans *et al.* (2005a) characterizes (U-Th)/He thermochronology is based on the determination of the relative accumulation of radiogenic ^{4}He produced from U and Th decay Evan *et al.* (2005a). The daughter He is retained until the mineral is heated to a temperature at which its structure changes and allows He to escape. Gradual cooling allows minerals lattice to trap ^{4}He, with the ''closure temperature'' (hereafter assumed to be equivalent to a cooling rate of 10 °C/Ma) varying from one mineral to another (Evans *et al.,* 2005a), and thus this method is a powerful tool that can be used to track the low temperature thermal history of an area. Farley (2002) indicates that while apatite is the most common mineral utilized for (U–Th)/He thermochronology studies, other minerals are also used routinely (e.g. titanite, zircon, monazite).

Some minerals, such as apatite, hematite and fluorite, present low closure temperatures of between 70 ºC to 150 ºC, making them useful for the dating of low temperature events.

(U-Th)/He dating of fluorite is a relatively new technique with great potential **(**Evans *et al*., 2005a and b; Pi *et al*., 2005; Tritlla & Leverese, 2006), as the low closure temperature of 90 ºC, makes fluorite thermochronology the ideal method for dating both magmatic and post magmatic low temperature events associated with the circulation of hydrothermal fluids.

This paper presents the results of (U-Th) / He dating of fluorite mineralization related to the Tanguá Alkaline Intrusion. The samples were collected from the Emitang Mine (Figure 2) and represent different types of ores mainly separated by their color (purple, yellow and white).

The ages presented in this work are of flourite mineralization related to the Tanguá Alkaline Intrusion with samples taken from the Emintang Mine. Flourite crystals were separated by their colour (purple, yellow and white) and analysed by Alphachron and ICP-MSat the John de Laeter Centre for Isotope Research in Perth, Western Australia. by using Alphachron to extract ^{4}He and ICP-MS to analyze for other isotopes.

^{4}He was thermally extracted by heating individual crystals in micro melting pots of niobium, using a 1064 mm Nd-YAG laser. The liberated gas was spiked with ^{3}He and the ^{4}He abundances were determined by isotopic dilution on the Alphachron's Quadrople mass spectrometer (QMS) which is calibrated daily against a ^{4}He standard, resulting in less

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than 1% uncertainty in ^{4}He measurements. After He extraction the fluorite crystals were dissolved using a concentrated hydrofluoric acid (HF) heated for 48 hours at 240ºC in pressure dissolution vases). After HF evaporation, hydrochloric acid (HCl) was added and the samples were pressurized again (200ºC for 24 hours). After this two-acid treatment, the U and Th content of the degassed fluorites were determined by isotopic dilution on a ICP-MS calibrated against ^{235}U and ^{230}Th standards.

3 Tanguá Alkaline Suite

In Brazil's southeast, a number of alkaline plugs, stocks and associated dykes and sills intrude the precambrian rocks of the Mantiqueira Province. The Alkaline Suite of Tanguá being one of them. The Tanguá intrusion (Tanguá Alkaline Suite) is one of these alkaline bodies, located close to the city of Tanguá, to east of the Guanabara Bay within Rio de Janeiro state (Figure 1). Another two alkaline bodies, the The Soarinho and Rio Bonito intrusions, are also located nearby (Figure 2). The Tanguá intrusion covers an area of 60 km^2, is approximately oval in shape and was intruded within Neoproterozoic metasediments (paragneiss, quartzites and schists) and orthogneiss (deformed granitoids). The intrusion contains important fluorite mineralization in the form of NE-SW orientated veins, which were economically extracted in recent decades.

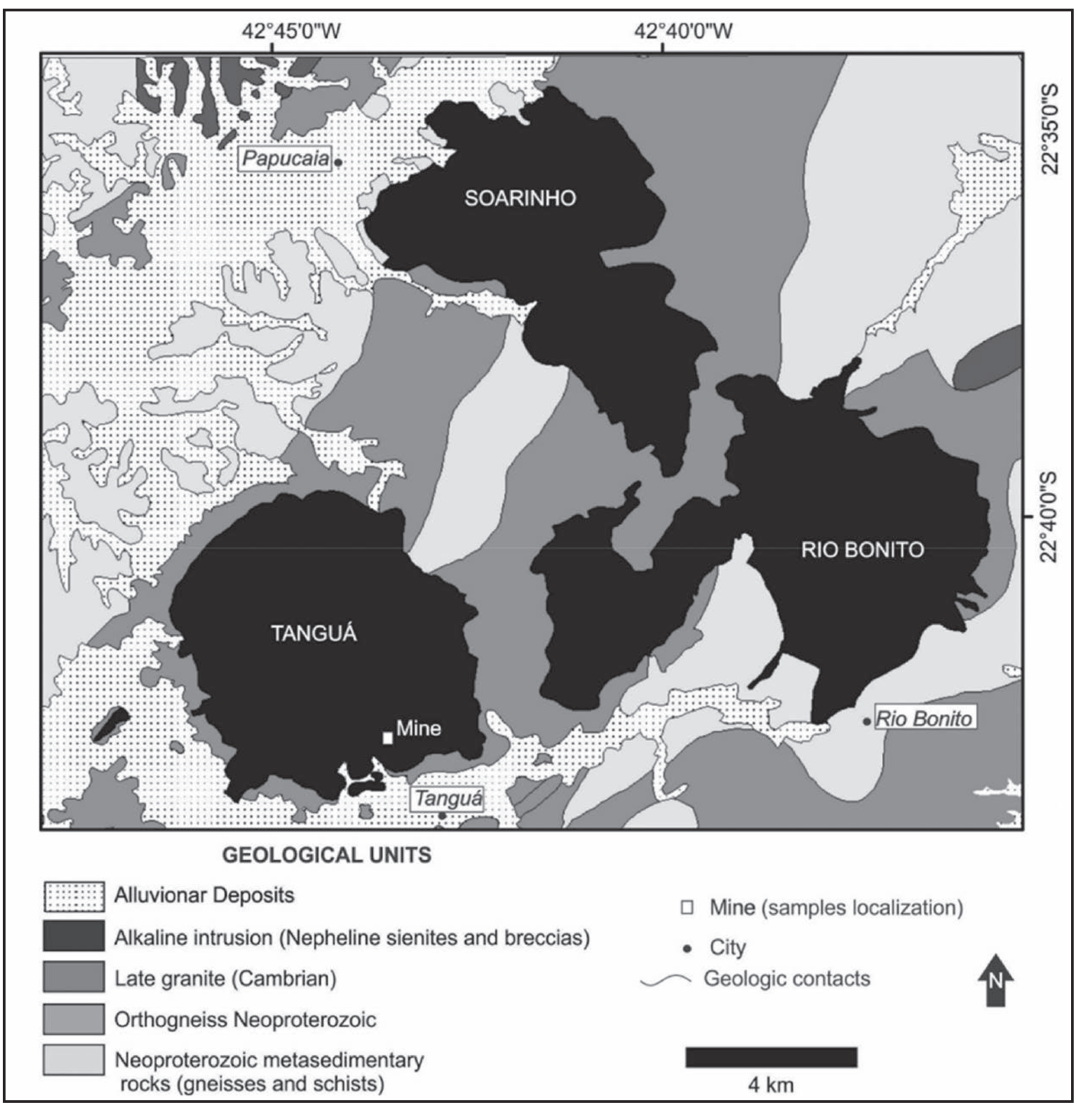

Figure 2 Simplified geology map of the Tanguá, Rio Bonito and Soarinho Intrusions.

Previous studies of the Tanguá Alkaline Suite includes mapping of the intrusive, and petrographic and metallogenic studies (e.g. Valença,1980; Coelho,1987;, Martins *et al.*,1982; Becker *et al.*,1997; Pinho,2007; Souza *et al.*,2008 e Motoki *et al*.,2010). These studies identified the presence of plutonic (syenites, nepheline syenites and alkali feldspar syenites), subvolcanic (mainly dykes) and volcanoclastic rocks (lapilites).

The Tanguá Alkaline Intrusion has very similar geology to both the Rio Bonito and Soarinho intrusives. In the field, the alkaline rocks occur commonly as widely spaced blocks and boulders, spheroidal in shape due to exfoliation weathering. The Tanguá intrusion is bordered by coarse-grained biotite orthogneiss, which displays both porphyroblastic and porphyroclastic textures with large (5 cm) porphyroclasts of K-feldspar and a highly weathered cataclastic gneiss at the contact. The intrusion itself is defined by alkali feldspar syenites with nepheline (pulaskites and umptekites), syenites (with or without pseudo-leucite), nepheline syenites and alkaline magmatic breccias.

According to Valença (1980), the Tanguá Alkaline Intrusion present a concentric pattern of variation in composition, with pseudo-leucite bearing syenites in the centre of the intrusion in contrast with the lack of this mineral in the syenites that outcrop on the edge of the intrusive body. The geochemistry of these syenites,that are in contact with the orthogneissic country rocks, is characterised by smaller modal amounts of nepheline than those in the central portions of the intrusive body.

Becker *et al.* (1997) mapped trachyte and phonolite dykes and sills, which are porphyritic in places and intrude mainly the orthogneissic basement rocks but also intrusive within the syenites of the main intrusive body. Becker *et al.* (1997) also observed that these dykes and sills often contain angular to rounded fragments of trachyte, phonolite and the occasional fragment of syenite or gneiss, ranging in size from a few millimeters to several centimeters. The dominant strike of these dyke is NE-SW and they range in thicknesses from 20 centimeters to tens of meters. A series of fluorite bearing veins, often parallel to the dykes previously described, occur within and in the vicinity of the Tanguá alkaline intrusive body. These veins have a maximum extension of 6 meters and are less than 1m in thickness, and are accompanied by intense hydrothermal alteration of the wall rocks (Figure 3A, B and C). According to Becker *et al.* (1997), this alteration is characterized by silicification and argillic alteration close to the veins, with more widespread sericitization and chloritization further afield. The cited author also described the veins as being characterized by the presence of mainly fluorite and chalcedony with associated pyrite, kaolinite, and or carbonated minerals.

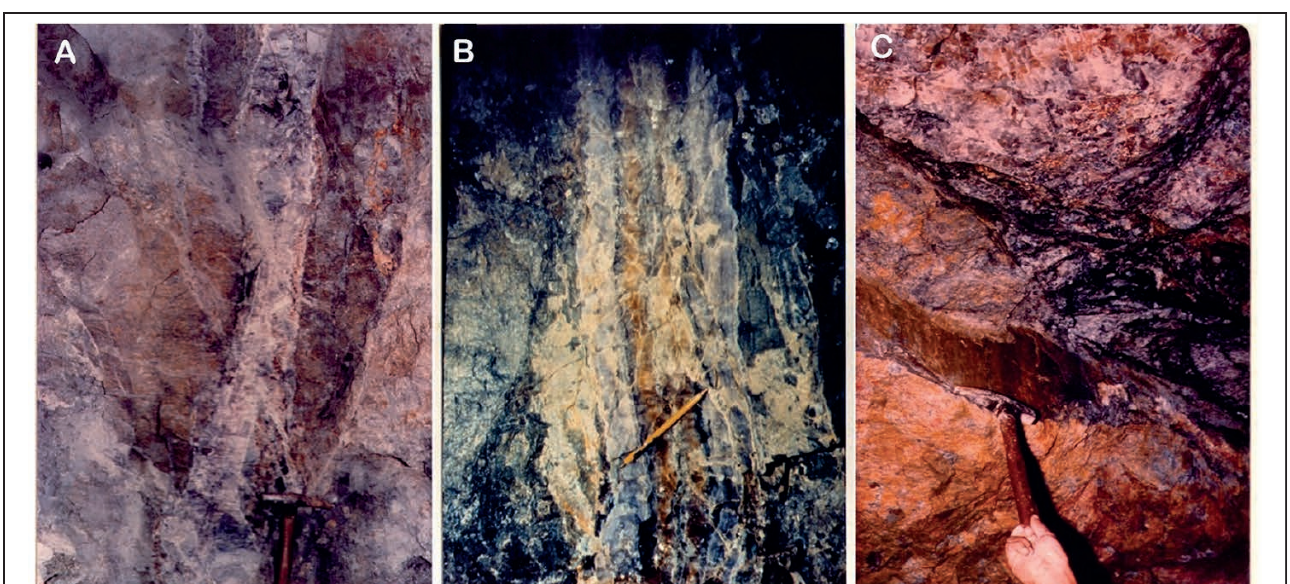

Figure 3 Fluorite ore veins observed in excavations made by EMITANG mining. A. Sub-metric fluorite veins within highly altered country rock; B. Brecciated and fractured, centimetric veins of fluorite; C. Submetric fluorite vein of fluorite extracted by the mine.

4 Results

4.1 Mineralogical Characterization of the Fluorite

In this study, 10 fluorite samples were submitted for analysis; these samples were collected from fluorite-silica veins that cut across syenites of the Tanguá Alkaline Intrusion. These veins are concordant with NE-SW trending trachyte dykes at the Emitang Mine, located close to the southeastern margin of the Tanguá intrusion.

The average thickness of sampled veins was 30cm and they present a brecciated appearance close to the surface, with limonite and manganese oxide staining and cementing.. In some places these iron and manganese oxides were the principal cements between fragments of fluorite and chalcedony.. The veins occur associated with shear zones, which range in direction from N50E to N70E. In hand samples, the fluorite presents a range of colors from purple, yellow, green and white (Becker *et al.,* 1997). The fluorite crystals are strongly fractured principaly along the cleavage planes, and often present rounded to elongate cavities in the form of geodes with microscopic crystals of fluorite. Pyrite, sericite, iron oxides and kaolinite were also occur as secondary minerals.

The field observations of Becker *et al.* (1997) suggest five phases of fluorite mineralisation characterized by structural relations and variations in color (Table 1). The same study characterised three different ranges of homogenization temperature of the flourite fluid inclusions, suggesting at least three different mineralization events.

The majority of the fluorite samples dated in the present study by the (U-Th)/He method were characterized by centimeter long, homogeneous crystals (Figure 4A and B). However, some crystals display zonation at their margins suggesting multiphase crystal growth (Figure 4C). In this case the homogeneous center was analyzed, whilst in other sample the larger (>1.5cm) and most homogeneous crystals (>1.5cm) were preferentially selected for analysis.. An attempt was also made to analyze the largest range of crystal colors (white, yellow and pink) and ore types from the Emitang Mine. And in a number of samples, fluorite crystals of different colors were analyzed separately (e.g. sample TGA 01 L, TGA 01 M and TGA 01 R).

PHASE MINERAL	PRE-I oldest vein	I lodes oblique PRE-I	II cut lodes I	III lode late	IV lode late	V geodes
Chalcedony	■	■	■	■	■	■
Pink Fluorite		■				■
White Fluorite	■	■	■	■		
Yellow Fluorite	■	■	■	- - -	■	■
Green fluorite			■			
Pyrite				■		
Kaolinite					■	■

Table 1 Mineral paragenesis of the fluorite deposits within Tanguá Alkaline Suite. Modified of Becker *et al.* (1997)

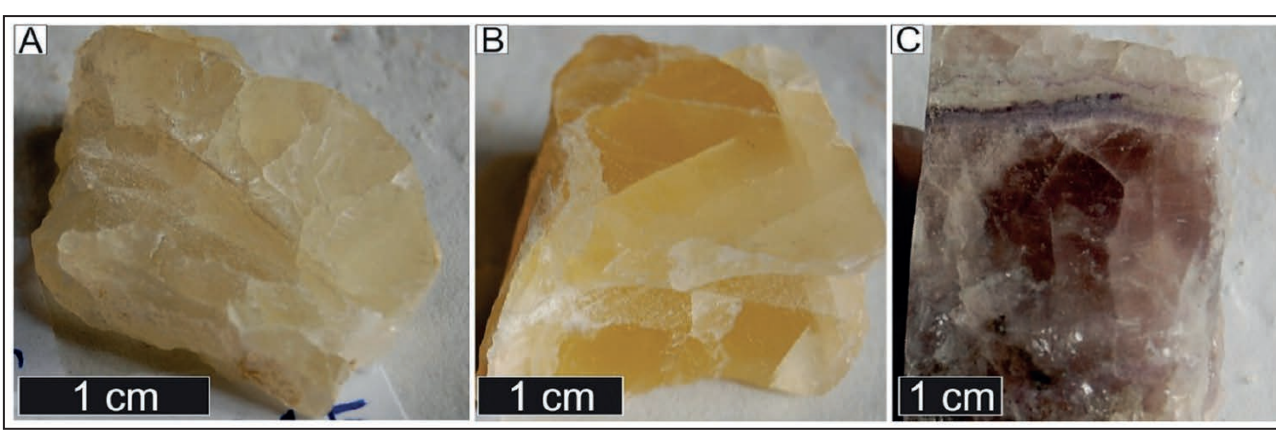

Figure 4 Selected samples of vein fluorite dated by the (U-Th)/He method. (A) Sample TG01 F - homogeneous, subeuhedral, clear to white crystals. (B) Sample TG01 G - homogeneous, subeuhedral, yellow white crystals with prominent cleavage plains. (C) Sample TG01 P white and purple crystal with zonation along the upper margin consistent with multiphase crystal growth.

4.2 U-Th/He dating

The results of the (U-Th)/He thermochronology of the fluorite veins associated with the Tanguá Alkaline Suite are presented in Table 2 below. Ages were calculated using the equations/methodology of Evans *et al.* (2005b). As shown in Figure 5 the calculated ages varied significantly from an oldest age of 74 ± 3 Ma (Upper Cretaceous) to a youngest age of 0.11 ± 0.02 Ma (Upper Pleistocene), suggesting several different phases of fluorite growth associated with the percolation of hydrothermal fluids.

Sample	Color	Crystal size (cm)	^{4}He (nmol/g)	Th/U	Ages (Ma)
TAG01 F	white	3	0.030	4.55	8.00 ± 0.83
TAG01 G	yellow	3	0.184	2.78	74.1 ± 2.8
TAG01 L (a)	white	2	0.001	3.45	0.33 ± 0.05
TAG01 L (b)	white	2	0.001	5.71	0.11 ± 0.02
TAG01 L (c)	white	2	0.002	0.90	1.86 ± 0.15
TAG01 M (a)	pink	1.0	0.038	5.25	12.59 ± 1.38
TAG01 M (b)	pink	1.0	0.032	3.77	13.55 ± 1.33
TAG01 P	pink	1.5	0.125	3.58	32.8 ± 1.2
TAG01 R (a)	white	2	0.185	0.79	21.2 ± 0.83
TAG01 R (b)	white	2	0.079	1.04	25.8 ± 1.55

Table 2 U-Th/He Thermochronology fluorite veins cutting across the Tanguá Alkaline Intrusion.

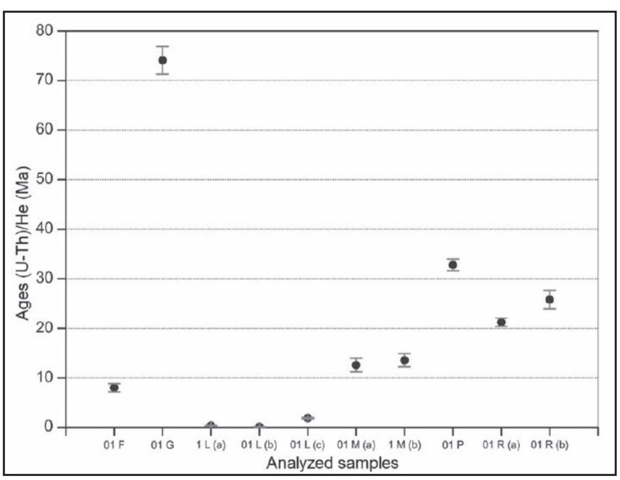

Figure 5 Graphic showing variation in (U-Th)/He of fluorite mineralization associated with the Tanguá Alkaline Suite.

The oldest age (74 ± 3 Ma) is the closest to the Rb-Sr whole rock age of 66.8 Ma obtained by Motoki *et al.* (2010). It is also very close to published K-Ar ages (83.9-69.7 Ma) in alkali feldspar from the nearby Rio Bonito Alkaline Intrusion (Thomaz Filho & Rodrigues, 1999). This older age (74 ± 3 Ma) was obtained in yellow fluorites which suggests that they represent the first mineralization event, in agreement with the mineral paragenesis of Becker *et al.* (1997**)**. Thus, we assume that this age of 74 ± 3 Ma represents the minimum emplacement age of the syenites of Tanguá Alkaline Suite, and the associated circulation of hydrothermal fluids during and immediately after emplacement.

The remaining ages are considerably younger (>40 Ma) than the oldest age, and are concentrated within the Miocene between 25 and 8 Ma. These ages were obtained from flourite crystals of varying colors (pink and white). We suggest that these ages are related to the circulation of hydrothermal fluids incorporated contemporaneous with the intrusion of phonolite and trachyte dykes and sills, within the earlier syenites which form the bulk of the Tanguá Intrusion. As such, this interpretation is in agreement with Becker *et al.* (1997), where the authors suggested that the principal fluorite mineralization occurred during Miocene, associated with the intrusion of alkaline dykes and sills and contemporaneous cataclastite formation and brecciation due to the reactivation of basements faults and structures in the Tanguá region.

Younger ages (1.86 - 0.11 Ma) were also obtained from the sample TAG01 L (a, b & c), suggesting repeated reactivation of basement structures and the circulation of hydrothermal fluids within and adjacent the Tanguá intrusion until as recent as 110.000 years ago.

5 Discussions and Conclusions

The U-Th)/He ages of fluorite veins of the Tanguá Alkaline Suitevaried significantly from an oldest age of 74 ± 3 Ma (Upper Cretaceous) to a youngest age of 0.11 ± 0.02 Ma (Upper Pleistocene), suggesting several different phases of fluorite growth and of hydrothermal fluid percolation. The oldest age of 74 ± 3 Ma (TAG 01 G), was obtained from yellow fluorite crystals and possibly represents the circulation of hydrothermal fluids during and immediately after emplacement of the Tanguá Alkaline Suite.

The other ages, are concentrated mainly within the Miocene (25-8 Ma), and are assumed to represent the main phase of mineralization. A genetic link has been proposed between the fluorite mineralization, hydrothermal alteration and the intrusion of the phonolite and trachyte dykes and sills during the reactivation of basement faults and structures in the Tanguá region.

The Miocene ages obtained from purple and white fluorite veins, have significant tectonic implications as the principal phase of fluorite mineralization can be related to one of the tectonic events described by Riccomini *et al* (2004) for the evolution the Continental/ Rift of Southeastern Brazil. These authors proposed that a sinistral trans-extensional tectonic regime was active during the Miocene, with NW-SE extension. This lead to the development of small pull apart subbasins such as within the São Paulo Basin with the deposition of the Itaquaquecetuba Formation. Paleontological evidence suggest a Lower Miocene for this formation (Arai & Yamamoto, 1995). Therefore, principal phase of fluorite mineralization within the Tanguá Alkaline Suite may be associated with circulation of hydrothermal fluids percolating along faults that were reactivated during this tectonic regime in the Miocene. This hypothesis is supported by the correlation between the NE-SW orientation of the fluorite veins parallel to the direction of maximum compression during the Miocene, as defined by Riccomini *et al.* (2004).

The youngest ages (1.86 - 0.11 Ma) reflect continued reactivation of basement structures during a later tectonic stage in the late Pleistocene to Holocene, which according to Riccomini et al (2004) was characterized by E-W to WNW-ESE.

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