

APPENDIX 2: CIEMAT Mineralogical characterization of fracture fillings: short description of selected samples

With the general knowledge of the system, a more specific selection of samples for more detailed analysis has been made (Figure 10). The selection try to cover the range from sub-surface (around 54 m) to depth (around 464 m). Tablets of selected samples were cut in order to perform thin slices for microscopic studies and in some occasions also thin-polished slices for electronic microprobe, electronic microscopy, scanning electron microscopy and, in specific cases, fluid inclusions, cathodoluminescence analysis and chemical analysis of selected minerals by LA-ICP-MS.

In this section a brief description of samples selected, all of them from boreholes SR-3 and SR-4, is made.

Sample RT-9

Location: borehole SR3 (54.5 m.)

Only core samples and thin sections. No data available.

Visu description: The fracture fillings are mainly composed by carbonate, quartz, apatite and Fe oxyhydroxides. These fracture fillings show a thickness in the range of 1.5 to 5 cm and present straight and net contacts with the granitoid host rock.

Sample RT-8

Location: borehole SR3 (140.5 to 150 m)

Available core samples, thin sections and chemical and mineralogical analysis by SEM, electronic microprobe and ICP-MS (trace elements and REE in the totality of the fracture filling – appendix I-)

This sample corresponds to a fracture filling of carbonate type, where it has being able to distinguish through optic microscopy up to three carbonate generations. The first type (carbonate type 1 described in previous sections) presents big crystals with a spear morphology and zoned according with the chemical composition; the second one (carbonate type 2 described in previous sections) is characterised by a massive aspect made out by alotriomorphic carbonate crystals with a great variety of sizes; finally, the carbonate type 3 is formed by crystals with a mosaic texture which can reach more than 30 microns of size.

Carbonate characterisation and analyses have been done with SEM and electronic microprobe, respectively. The mineral association studied by SEM consist of: carbonates, quartz, iron sulphides and apatite (Figure 11).

In order to perform fracture fillings carbonates chemical analyses through electronic microprobe, three fields from a thin polished slice where the three above mentioned carbonate types were well represented were selected. Then, the obtained results are

detailed, showing the corresponding optic microscopy images for every field selected (Figure 12).

Results of carbonate chemical composition by means of electron microprobe analysis are:

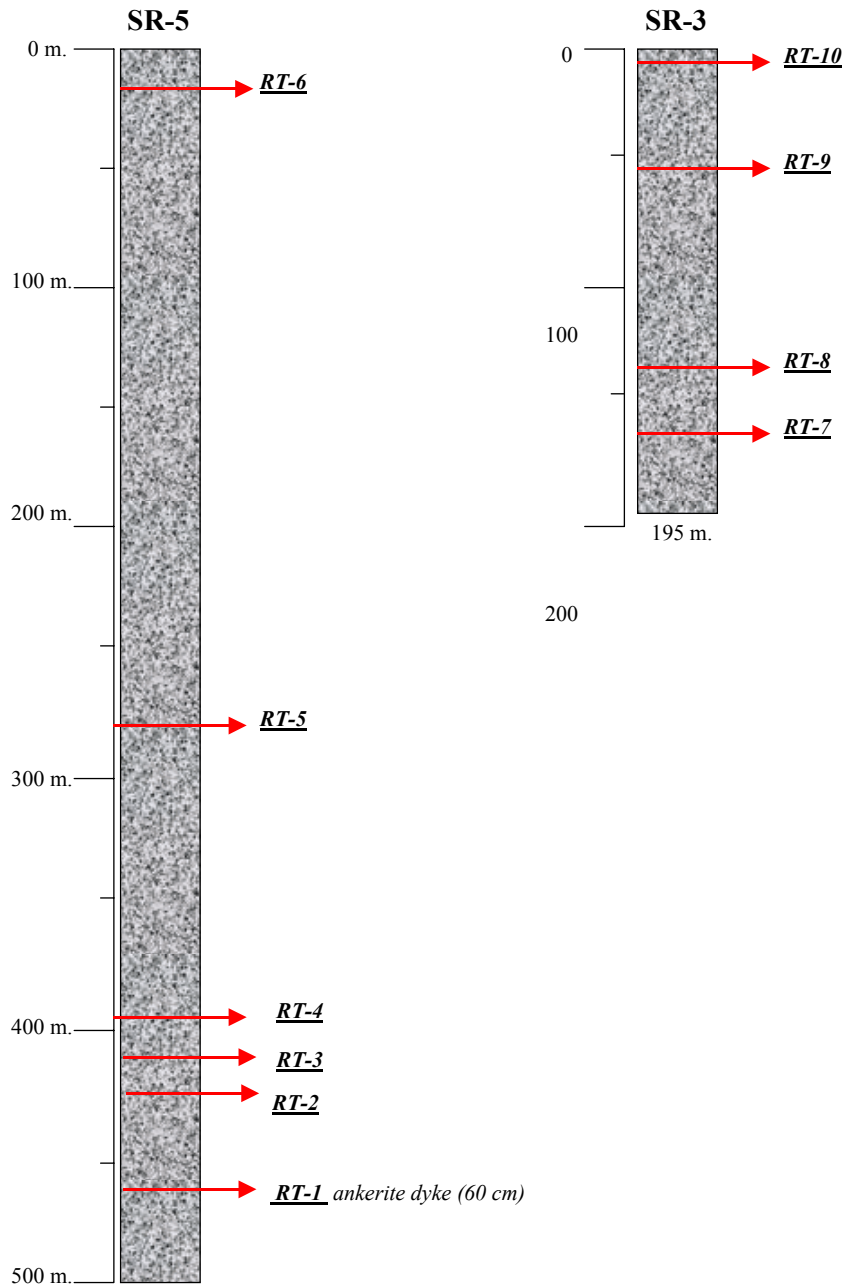
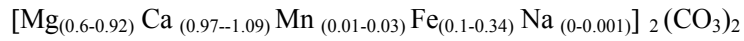


Figure 10. Samples selected for detailed analysis. All of them are from boreholes SR3 and SR5 of Ratones area.

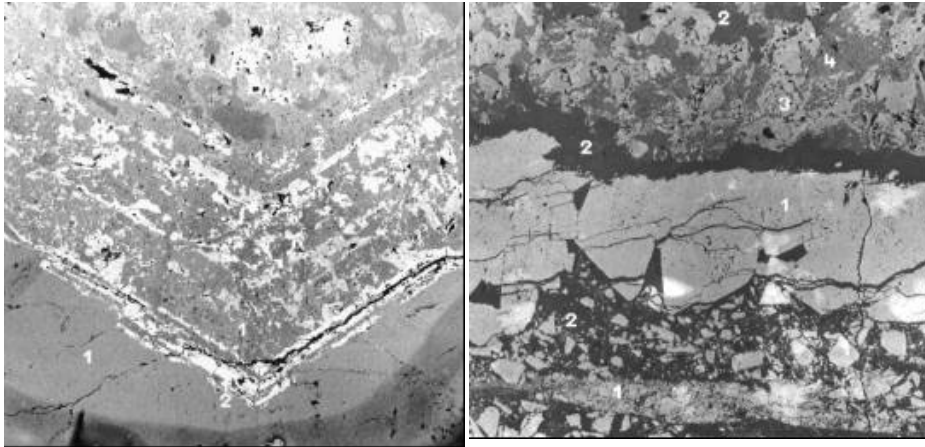


Figure 11. (left) ankeritic carbonates (1) and (2). (right) Apatite (1), quartz (2), and carbonates (3) and (4).

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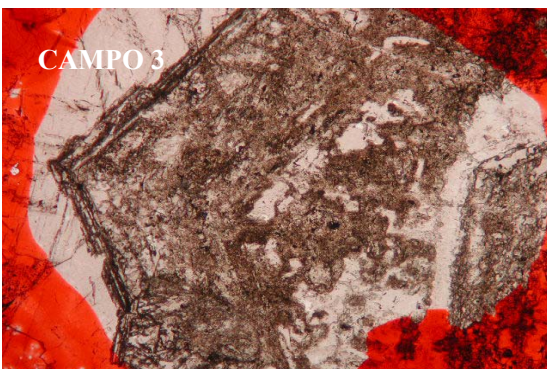
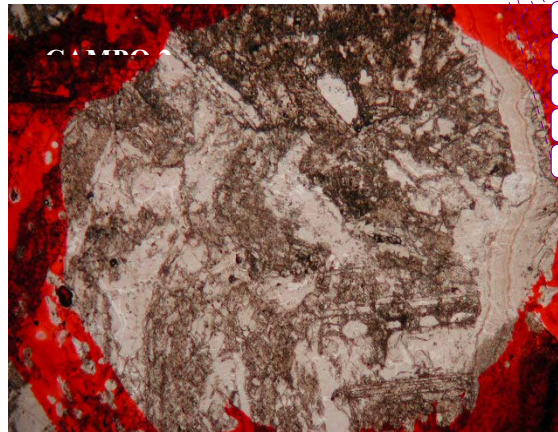
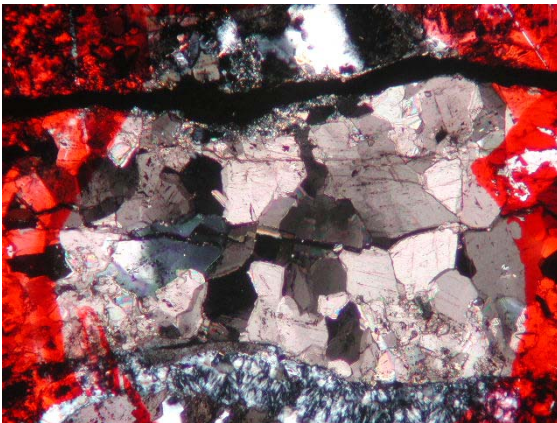
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Figure 12. Thin slice RT-8 filling optical microscopy view of defined fields for carbonate analysis through electronic microprobe.

Sample RT-7

Location: borehole SR3 (168.3 m)

Only core samples and chemical analysis by ICP-MS (trace elements and REE in the totality of the fracture filling – appendix I-)

The fracture fillings are mainly composed by carbonate, chlorite, phyllosilicates (from feldspars alteration) and opaques. These fracture fillings show a milimetric tickness and non-consolidated nature (Figure 13).



Figure 13. Look to magnifying glass of this fracture filling .

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Sample RT-6

Location: borehole SR5 (36.3 m)

Available core sample and thin section

These fracture fillings show a milimetric tickness and they are composed by quartz, Fe oxihydroxydes, carbonate, opaques and host rock minerals -mainly plagioclases, K-feldspars and micas (Figure 14).

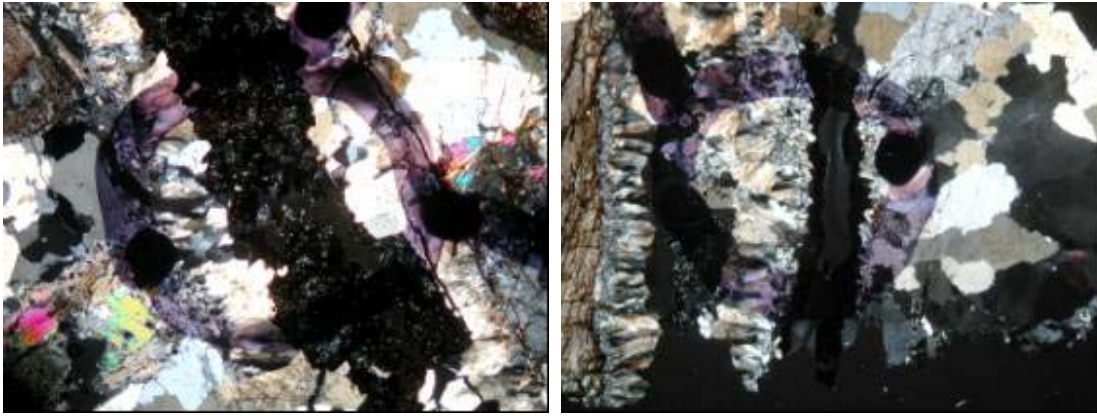


Figure 14. Microphotographs of the fracture filling SR3-36.3.

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Sample RT-5

Location: borehole SR5 (288.8 m)

Available core sample, thin sections and mineralogical and chemical analysis by SEM, electronic microprobe and ICP-MS (trace elements and REE in the totality of the fracture filling – appendix I-)

This sample correspond to a multiepisodic fracture filling of quartz and carbonate with pyrite, apatite, Fe oxyhydroxides and phyllosilicates (Figure 15). Its characterization was carried out by optic microscope, electronic microscope and electronic microprobe

The existence of several phases of Quartz fracture filling with different morphologies can be observed with the optic microscopy (Figure 15). In general terms, it can be distinguished:

1.- A first microcrystalline Quartz generation which is closely located next to fracture walls, and 2.-, a second generation constituted by large drusic Quartz crystals (Figure 16a) where in determined zones they can be seen syntaxial microcrystalline Quartz growths, followed by a third generation of drusic Quartz. After Quartz, carbonate (Figure 15 and 16 b) appears filling in internal fracture zones, as well as existing voids among Quartz crystals, presenting clear signs of dissolution. As far as the Fe oxides and oxyhydroxides is concerned, although they are mainly concentrated on the fracture walls, they also appear distributed on the rest of the fracture filling forming small veins filling in the existing voids.

The observation of this sample through SEM has allowed to complete and characterise the mineral association of the filling with the presence of inherited minerals from the granite, i.e. Biotite, Chlorite and Apatite (Figure 16c) and neo-formed minerals as the Smectite (Figure 16e) as well as LREE phosphates bearing uranium traces (Figure 16f).

Figure 14. Microphotograph of the fracture filling SR5-288.84

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With respect to opaque minerals, it has been observed pyrite that keeps its typical cubic morphology and also Ag sulphides of high temperature (Figure 16d). Finally, it is worth to highlight the presence of organic matter in specific zones of the sample which has also been described in other anatexia granites.

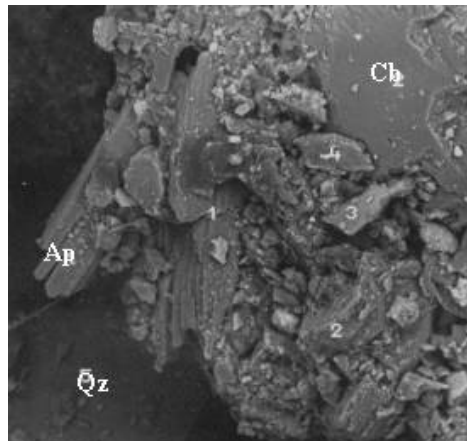
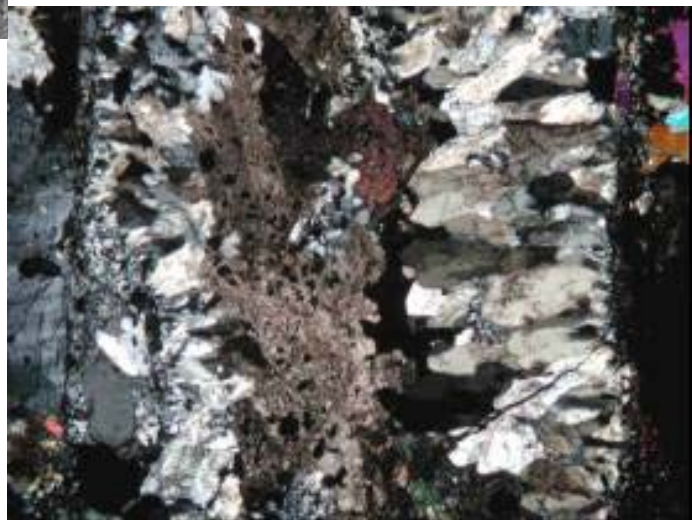


Figure 15. This microphotograph (SEM) shows one section of fracture filling SR5- 288.85. We can observe carbonates (Cb and 3), apatite (Ap and 1), quartz (Qz), biotite (3) and chlorite (4).



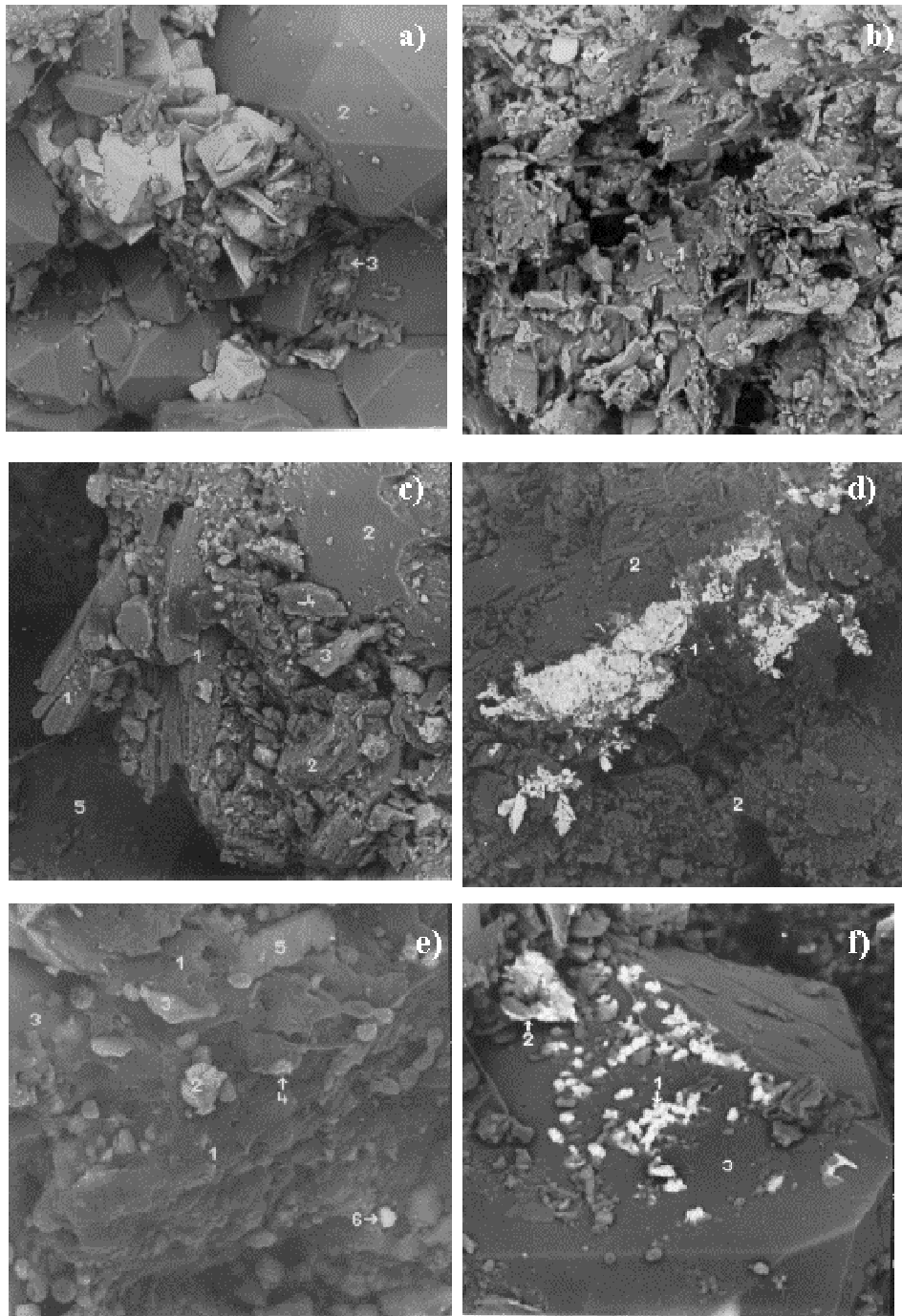


Figure 16: a) Pyrite(1) and Quartz (2), b) carbonates (1) and Pyrite with arsenic traces(2), c) Apatite(1), Fe and Mn carbonates(2), Biotite(3), Chlorite(4) and Quartz (5), d)Argentite(1) and Smectite with carbonates(2), e) bitumen (1), carbonates (2), Quartz (3), Gypsum(4) and Pirite(6), f) LREE phosphates with Ca and U (1) and Quartz(3).

Chemical analyses of carbonates from this filling have been made through electronic microprobe.

In Figure 17 it can be seen the optical microscopy image corresponding to the field where analytical determinations were carried out as it shows the biggest proportion of carbonates with regard to the whole polished thin slice.

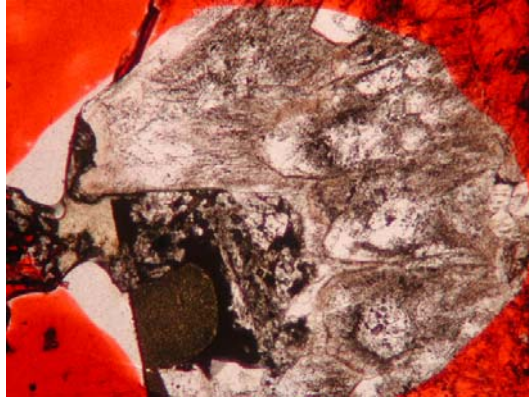


Figure 17. Optical microscopy image from the selected field to carry out chemical analyses by means of electronic microprobe.

Results of carbonate chemical composition: next compositions to sideritic pole of the sideritic-magnesite serie $[\text{Mg}_{(0.7-0.82)} \text{Mn}_{(0.04-0.06)} \text{Fe}_{(1.05-1.24)} \text{Na}_{(0.001)}]_2 (\text{CO}_3)_2$

Sample RT-3 and 2

Location: borehole SR5 (419.15 m -RT-3- and 423.2 m -RT-2)

Available core samples and thin sections.

These samples have the same kind of fracture filling: a multiepisodic fracture filling of quartz, carbonate and apatite, with Fe oxyhydroxides, pyrite, opaques and micas – moscovite neoformed and inhereted biotite- (Figure 18). The carbonates shows several textures. On the other hand, to emphasize the large development of apatite crystals.

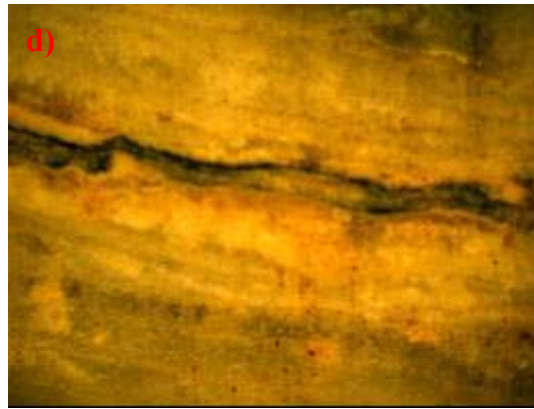
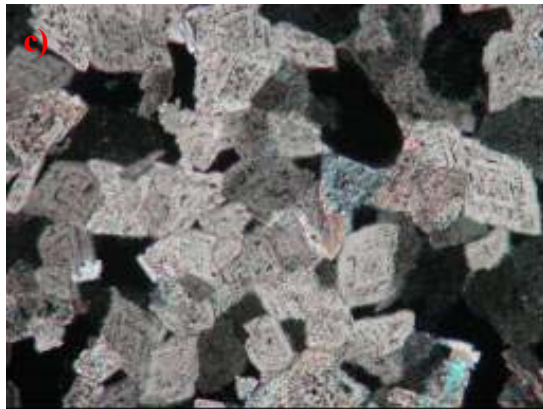
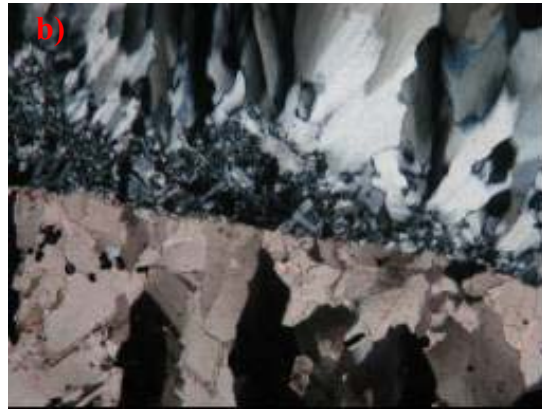
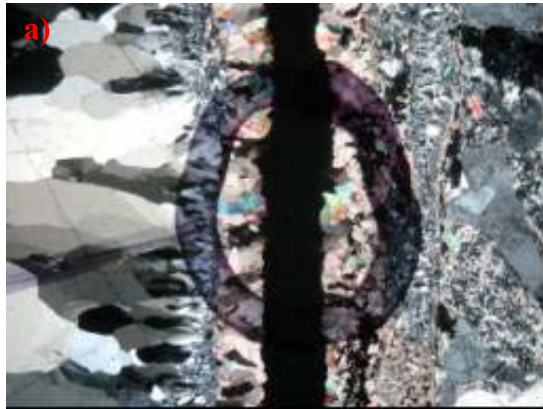


Figure 18. a) View of the fracture fillings (optical microscope) from thin sections: b) and c) carbonates textures d) Visu photograph e) microphotograph of the carbonate, the quartz and the apatite in one section of this fracture filling.

Sample RT-1

Location : borehole SR5 (463-9 m) – Figure 19 –

Available core samples, thin sections and analysis by α -spectrometry, cathodoluminescence and fluid inclusions.

It is possible to recognize a multi-episodic fracture filling, with the following episodes:

- 1) Represented by major macrocrystalline and euhedral to subeuhedral ankerite mineralisation.
- 2) Comprises phyllosilicates, feldspar, quartz and fluorapatite filling veins and the contact between the ankerite from (1) and the granite.
- 3) The “youngest” mineralisation, possibly formed from “recent” groundwaters: calcite and dolomite mineralisation. Measurements of $^{230}\text{Th}/^{234}\text{U}$ indicate an age around 2×10^5 years (just one sample, so large uncertainty on it) (Figure 20). This carbonates are scarce but of main importance for this study. They are growing in pores and microfissures (Figure 21).



Figure 19. Core from borehole SR5. The white zone corresponds to an ankeritic dyke at around 463 m depth.



Figure 20. Examples of carbonate crystals studied in the ankeritic dyke (≈ 464 m depth). The age is measured from α -spectrometry.

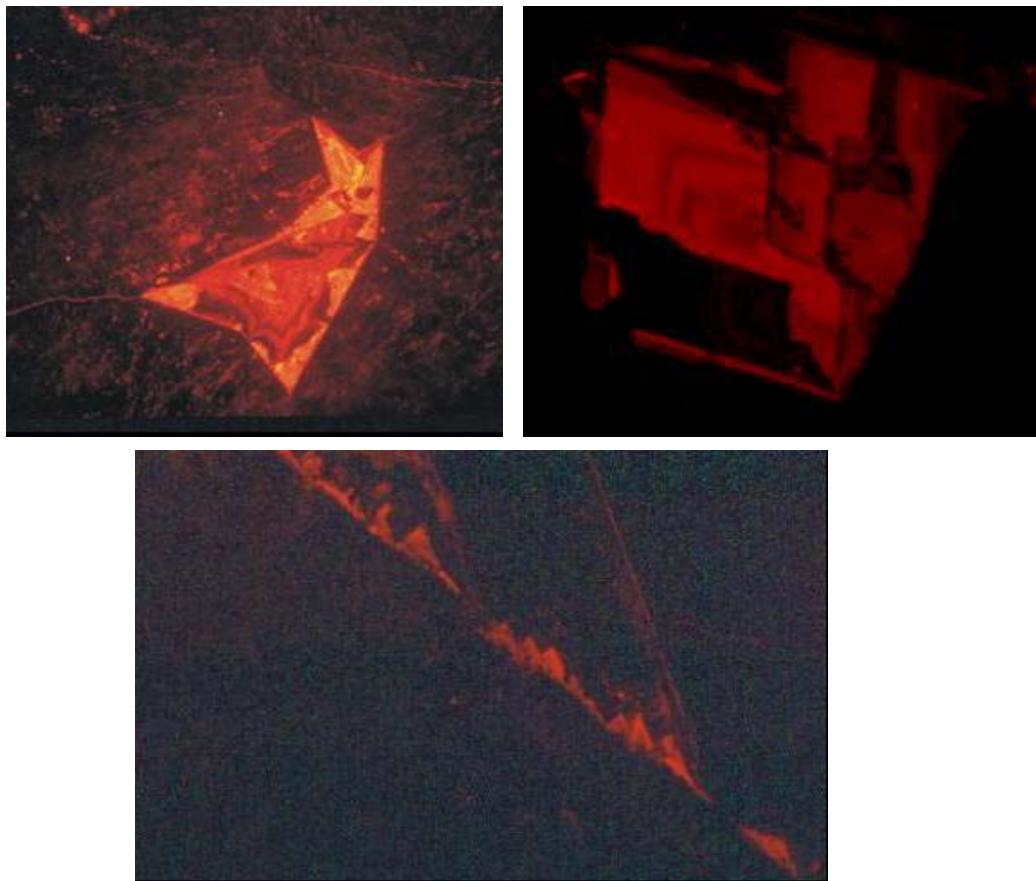


Figure 21.. Examples of “recently-formed” calcite filling pores and microfissures. Top left and right are typical zoned calcite crystals in pores from a thin section.