

1 **Microstructural Evolution of Granitic Stones Exposed to Different Thermal Regimes**  
2 **Analysed by Infrared Thermography.**

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7 **ABSTRACT**

8 Detailed knowledge of the behaviour of rocks under thermal stress is essential in a variety of  
9 fields such as the exploitation of oil and mineral resources, the geothermal sector, the  
10 storage of radioactive liquid waste, or even CO<sub>2</sub> capture and storage.

11 Granites are widely studied and exploited in these fields, and they show different reactions  
12 to high-temperature and thermal cycles due mainly to their high mineralogical and textural  
13 heterogeneity. One of the features that influences the most the thermal response is the  
14 porosity.

15 The objective of this study is to evaluate the influence of porosity when these rocks are  
16 exposed to different thermal treatments. For that purpose, experiments were carried out on  
17 four granitoids selected by their similar crystal size, but with variable mineral proportion and  
18 porosity values, ranging from 1 to 6%. Two kinds of tests were performed: i) progressive  
19 heating cycles from 90 °C to 130 °C to determine the critical threshold for thermal damage;  
20 ii) thermal fatigue with cycles of heating-cooling up to 200 °C.

21 The porosity and the water transport phenomena of the samples were characterised before  
22 and after each cycle by the monitoring of capillary water uptake coupled with infrared  
23 thermography. This technique allowed to follow the capillary fringe migration during the test  
24 and the evolution of the cooling rate index. The direct assessment of the damage was  
25 carried out by mercury injection porosimetry, optical polarising microscopy, and scanning  
26 electron microscopy.

27 The combination of all the results permitted to establish a link between the evolution of  
28 temperature and the modification of porous networks in granitoids. Microcracks appeared

29 distinctly at a temperature between 90 °C to 130 °C for high porosity granitoids whose  
30 Quartz/Feldspar ratio was close to 1. For higher temperatures, the low porosity granitoids  
31 develop microcracks from the first heating cycle. The porosity then showed a stronger  
32 impact on thermal behaviour than the effect of the mineralogy. The results obtained from  
33 infrared thermography allowed to detect the strong variations in the microstructure.

34 Keywords: Thermal damage; Granites; Critical threshold; Thermal fatigue; Microcracks;  
35 Infrared thermography

## 36 1\_INTRODUCTION

37 The effect of temperature on rock structures has been widely studied because of its  
38 common presence in many geological applications. Examples may be found in the field of  
39 ores, hydrocarbons, storage of nuclear waste or CO<sub>2</sub>, as well as in the geothermal resources,  
40 in which the energy is recovered thanks to the circulation of fluid through its porosity (faults,  
41 fractures and matrix) (Bai et al., 2018; Pandey et al., 2017; Parnell, 1988; Witherspoon et al.,  
42 1980). In the latter, the injection of cold water into hot rock is used to increase the transfer  
43 properties of the neighbouring rocks and causes a slow cooling of the peripheral rock mass  
44 (Isaka et al., 2018). These heating and cooling cycles alter the intact rock and thus, influence  
45 the fluid flow and in some occasions the stability of the well (Kumari et al., 2017). The  
46 temperature of geothermal systems can be low temperature like 80 °C, but the global  
47 average temperature is around 200 °C (Breede et al., 2013; Olasolo et al., 2016). Granites are  
48 also prospected for geological disposal of radioactive waste, the containers used in deep  
49 storage can expose the surrounding rock masses to temperatures likely to degrade the rock  
50 microstructure (Chen et al., 2017a). For the storage of high-level radioactive waste, the  
51 temperature in the canister surfaces must not exceed 100 °C (Hoekmark and Faeth, 2003).  
52 The temperature also has an impact on deep tunnels which can generate thermal stress on  
53 rocks surrounding tunnels, most of which do not exceed 130 °C (Chen et al., 2018).

54 The flow properties and the mechanical strength of the rocks are directly influenced by their  
55 microstructures. Recent studies have been conducted at pore-scale to characterise the  
56 physical parameters of the rock such as porosity, permeability, or elastic properties for  
57 natural strain (Chaki et al., 2008; Staněk and Géraud, 2019). The influence of the  
58 temperature is not yet well studied. Unravelling these physical parameters and the

59 microstructures of the rock allows the understanding and the extrapolation to a large-scale  
60 system.

61 Granite is a material showing high mechanical strength with low matrix porosity and  
62 heterogeneous mineralogy, which makes it also very sensitive to the effects of temperature  
63 (Heuze, 1983). The differential mineral dilation, in most cases anisotropic (Berest and  
64 Vouille, 1988; Vázquez et al., 2011) may develop a microcracking (intergranular and  
65 intragranular) from a certain temperature threshold (Géraud et al., 1992). This critical  
66 temperature generates significant changes in physical properties. In general, microscopic  
67 observations show that microcracking often follows preferential directions, such as cleavage  
68 planes and crystal boundaries in rocks with pre-existing microcracks, because less energy is  
69 needed to generate microcracking (Gómez-Heras et al., 2006). The ratio Quartz/Feldspar  
70 (Qz/F) plays a determinant role in the microstress development, and consequently in the  
71 decay of granite (Sajid and Arif, 2015; Sousa, 2013; Tuğrul and Zarif, 1999; Vázquez et al.,  
72 2015).

73 During heating procedures with gradual temperatures, most of the studies conclude that the  
74 temperature threshold corresponding to the beginning of microcracks development is about  
75 120-130 °C (Darot et al., 1992; Guo et al., 2018; Lin, 2002). Geothermal systems operate on  
76 the principle of repetition of heating-cooling, and consequently, it becomes crucial to control  
77 the influence of the fatigue on the microstructure of the rock. In general, thermal fatigue in  
78 rocks leads to the propagation of pre-existing microcracks over cycling. Few works were  
79 done on the influence of fatigue thermal cycles on granite structures (Freire-Lista et al.,  
80 2016; Lin, 2002).

81 The main objective of this study is to assess the evolution of the granite pore network when  
82 these rocks are exposed in a geothermal system. Works on the thermal effects in relatively  
83 low geothermal temperature ranges (<200 °C) is scarce, as well as on damage during its long-  
84 term operation period. For that purpose, four granites, with similar mineralogy and crystal  
85 size but with different porosity values that ranged from 1 to 6%, were tested. Two different  
86 experiments were performed to determine the rock microcrack threshold by progressive  
87 heating cycles from 90 °C to 130 °C (EXP1) and to evaluate the thermal fatigue after five  
88 cycles at 200 °C (EXP2).

89 The samples were characterised in their healthy state and after each experiment by  
90 destructive and non-destructive methods such as Capillary Water Uptake Tests (CWUT),  
91 Infrared Thermography (IRT) monitoring, Mercury Injection Porosimetry (MIP), Optical  
92 Polarising Microscopy (POL), and Scanning Electron Microscopy (SEM).

## 93 2\_MATERIALS

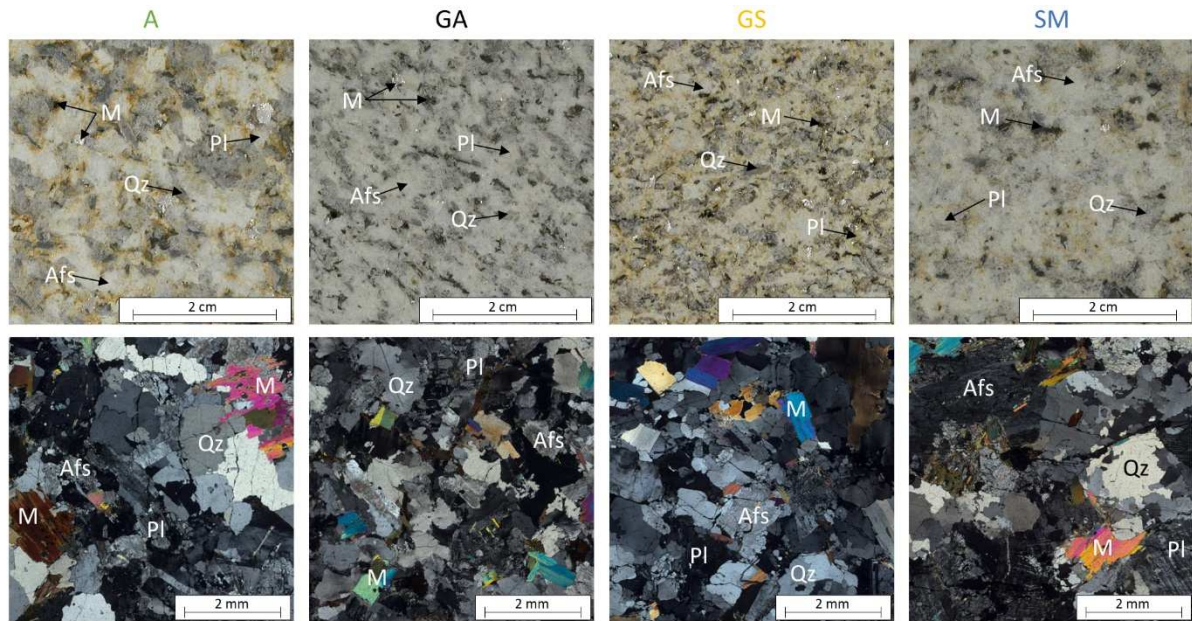
### 94 2.1 Geological settings

95 Four types of granite were chosen due to their similar mineralogy and crystal size but their  
96 difference in alteration degree and consequently in porosity. Their commercial names are  
97 Albero (A), Gris Alba (GA), Golden Ski (GS), and Silvestre Moreno (SM). They all come from  
98 the Iberian peninsula. The orientation of the (XYZ) axes was defined by the main crack  
99 system and the orientation of the mica plane in the quarry (Vázquez et al., 2011).

100 The four studied granites belong to the group of peraluminous syn- and post-kinematic  
101 granites. This group includes granites temporally related to processes of Hercynian crustal  
102 anatexis (Farias et al., 1987; Vera, 2004).

### 103 2.2 Granite description

104 The studied granites are shown in figure 1, their main petrographic characteristics are  
105 introduced in table 1. The petrographic characterisation (mineral proportion) was made with  
106 POL and crystal size was measured on the photographs at the macroscopic scale (Vázquez et  
107 al., 2018).



108  
 109 Figure 1: Macroscopic and microscopic photographs (Polarised light optical microscopy) of  
 110 the studied granite: Albero (A); Gris Alba (GA); Golden Ski (GS); Silvestre Moreno (SM). (Qz:  
 111 quartz; Afs: alkali feldspar; Pl: plagioclase; M: mica).

112 Table 1: Main characteristics of the selected granitoids: Trade name, Mineral proportion,  
 113 IUGS classification (Le Maitre, 2002), Qz/F ratio (Qz: Quartz; F: Alkali feldspar + Plagioclase)  
 114 and crystal size (Vázquez et al., 2018).

Granite	Composition (%)					IUGS classification	Qz/F ratio	Macroscopical crystal size (mm)				
	Qz	Afs	Pl	M	Qz			Afs	Pl	M	Average	
A (Albero)	35	10	30	25	Granodiorite	0.88	5	5	6	4	5	
GA (Gris Alba)	23	37	23	17	Monzogranite	0.37	5	5	4	2	4	
GS (Golden Ski)	47	20	20	13	Monzogranite	1.18	4	4	4	2	4	
SM (Silvestre Moreno)	45	20	20	15	Monzogranite	1.13	4	5	7	4	5	

115 Qz: quartz; Afs: alkali feldspar; Pl: plagioclase; M: mica.

116 Albero (A): It is a homogeneous granodiorite with medium-fine crystal size (5 mm). It has the  
 117 lowest alkali feldspar content among the four granites studied and a high proportion of mica  
 118 (25%) with a similar proportion of muscovite and biotite. This granitoid is characterised by  
 119 open transgranular microcracks.

120 Gris Alba (GA): It is a homogeneous monzogranite with a fine crystal size (4 mm). It has  
 121 anhedral minerals and the boundaries between the quartz crystals are irregular. The  
 122 proportion of muscovite/biotite minerals is about 2:1. The intergranular microcracks  
 123 observed in this granite are located at the mica edges.

124 Golden Ski (GS): It is a homogeneous monzogranite with a fine crystal size (4 mm). Quartz  
125 and feldspars are subhedral and muscovite is euhedral. The muscovite has the largest crystal  
126 size and plagioclase the smallest. Its quartz content is higher than that of feldspars. Pre-  
127 existing microcracks are intergranular and are present in plagioclases. GS also has open  
128 transgranular microcracks.

129 Silvestre Moreno (SM): It is a homogeneous fine sized monzogranite (5 mm). These minerals  
130 are subidiomorphic. After GS, this granite has the highest quartz proportion of the granitoids  
131 studied and proportions of feldspars and plagioclase are similar (20%). Plagioclases are the  
132 minerals with the largest size (up to 7 mm). As with A and GS, this granite has an initial  
133 alteration highlighted by intra-, inter- and trans-granular microcracks (Vazquez et al., 2018).

134 An important parameter regarding the mineralogy is the Qz/F ratio. GA has a ratio  $Qz/F <$   
135  $0.5$ , namely a granite rich in feldspar. Meanwhile, A, GS and SM have a similar proportion of  
136 both mineral types with values between 0.88 and 1.18. Porosity is the main differencing  
137 parameter between these rocks. GA is considered as a fresh granite (porosity  $<2\%$ , (Vázquez  
138 et al., 2018)), with a grey pale colour. The granites A, GS, and SM show a yellow colour that  
139 indicates the presence of clays due to previous weathering and consequently a higher voids  
140 volume than the fresh rock. These three granites exhibit large intra-, inter-, and trans-  
141 granular microcracks, giving them high water porosity for the granite range as well as high  
142 capillary transfer.

## 143 3\_METHODODOLOGY

### 144 3.1 Heating set up



















145 Two types of heating-cooling tests (EXP1 and EXP2) were performed on 2 samples of each  
146 granite with a rectangular prism shape of 10 mm × 40 mm × 40 mm in dimension. In  
147 addition, smaller samples of 10 mm × 10 mm × 15 mm (8 samples per granite type for EXP1  
148 and 3 samples per granite type for EXP2) were also tested at the same time for further  
149 destructive analyses as MIP (Ritter and Drake, 1945), SEM (Fan et al., 2017). Two thin  
150 sections per granite were produced in the initial state and after heating to 200 °C for  
151 observations under POL (Freire-Lista et al., 2016; Jin et al., 2019).

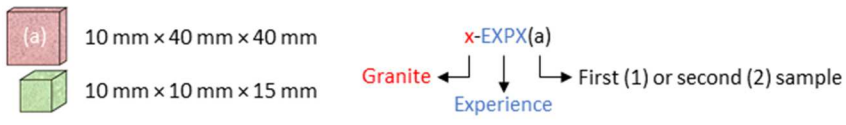
152 - EXP1: this test was conceived to determine the microcrack threshold of each granite.  
153 For this aim, 5 cycles of heating-cooling at an increasing temperature of 90 °C, 100 °C, 110  
154 °C, 120 °C and 130 °C were undertaken. A climatic chamber "Vötsch VC3" ensured a low  
155 heating and cooling rate of 1 °C.min<sup>-1</sup> to avoid the microcracks formation due to a high-  
156 temperature gradient within the sample (Chaki et al., 2008; Dwivedi et al., 2008; Homand-  
157 Etienne and Houpert, 1989; Reuschlé et al., 2006; Takarli and Prince-Agbodjan, 2008). The  
158 target temperature was maintained for 2 hours to assure that the whole sample was  
159 completely heated with homogeneous temperature distribution (Chaki et al., 2008; Kumari  
160 et al., 2017; Yin et al., 2015). It has been shown that heating time is also an important aspect  
161 of the consequences of the heat treatment. The longer temperature duration in the  
162 treatment, the greater the damage, but it is emphasised that the main thermal damage  
163 occurs within the first 2 hours (Tang et al., 2019). The microstructure evolution was assessed  
164 by CWUT and MIP after each cycle, SEM observations were also done at the initial and the  
165 final states.

166 - EXP2: this second test aimed at knowing the effect of the thermal fatigue in  
167 microcracked rocks. For this purpose, a repetition of 5 heating cycles up to 200 °C was  
168 performed. The heating process was carried out with a muffle furnace "Thermo scientific led  
169 M 110" with a heating gradient of 5 °C.min<sup>-1</sup> to promote microcracking (Ding et al., 2016;  
170 Huang et al., 2017; Kumari et al., 2017; Li et al., 2019; Shao et al., 2015). The samples were  
171 thermally stabilised after 2 hours at 200 °C. For cooling, the specimens were left in the  
172 furnace to undergo slow cooling at a rate of 0.5-1 °C.min<sup>-1</sup>. CWUT was monitored by IRT  
173 after every cycle on one of the samples. SEM and POL observations were carried out on fresh  
174 rocks and after the first cycle (200(1) °C).

175 The different cycles will be designated by the reference (Ref.) given in table 2. The two size  
176 categories are represented by the red and green samples. From now on, the followed  
177 sample (red) will be called by the abbreviation of the granite (x), the corresponding  
178 experience, and the number of the sample (a), i.e x-EXPX(a).

179 Table 2: Specification of the measurement conditions for samples subjected to the two-heat  
180 treatments (EXP1 and EXP2). The red samples were used continuously through each EXP,  
181 while the green ones were used only once, as the techniques used were destructive.

EXP1					EXP2					
Cycle	Ref.	CWUT	MIP	SEM	Cycle	Ref.	CWUT	IRT	MIP	SEM
0	Initial state				0	Initial state			-	-
1	90 °C			-	1	200(1) °C				
2	100 °C			-	2	200(2) °C	 		-	-
3	110 °C	  x-EXP1(1) x-EXP1(2)		-	3	200(3) °C	  x-EXP2(1) x-EXP2(1)	 x-EXP2(1)	-	-
4	120 °C			-	4	200(4) °C			-	-
5	130 °C				5	200(5) °C				-



182

183 Ref.: reference; CWUT: capillary water uptake tests; MIP: mercury injection porosimetry;  
 184 SEM: scanning electron microscopy; IRT: infrared thermography.

185 **3.2 Evaluation methods**

186 Microstructural characteristics have been evaluated on the fresh rocks and the samples after  
 187 heating as described in table 2.

188 MIP was undertaken before and after the experiments with a Micromeritics Autopore IV  
 189 9500 on samples of 10 mm × 10 mm × 15 mm size: one sample per cycle and granite for  
 190 EXP1 and one sample per granite after one and 5 cycles (200(1) °C and 200(5) °C) for EXP2.  
 191 Thus, a total of 32 samples were measured. Mercury injection pressures ranged from 0.004  
 192 to 228 MPa, giving corresponding pore access radii of 180 to 0.003 μm, respectively.

193 A total of 12 samples were cut with an approximate size of 10 mm × 10 mm × 15 mm to be  
 194 tested and studied directly under SEM to avoid the creation of additional microcracks from  
 195 sawing after the heating. Observations under POL (Olympus BX51) and SEM (Hitachi TM-  
 196 1000) were done on each granite and after being tested at 130 and 200(1) °C.

197 **3.2.1 Capillary water uptake tests (CWUT) and infrared thermography (IRT) monitoring**  
 198 **experimental setup**

199 For both experiments, capillary kinetics were measured on samples after each cycle to  
 200 assess the modifications of the porous network due to thermal stresses. The capillary

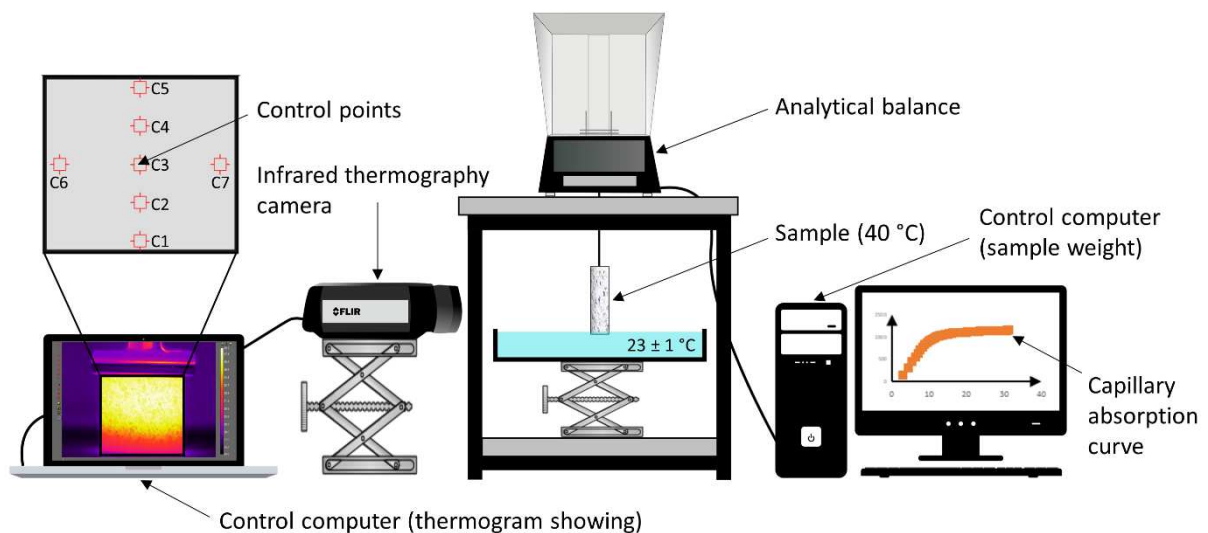


201 coefficient (C) was calculated based on the NF EN 15801 (2010) standard. After each heating  
202 cycle, the granites were thermally stabilised at 40 °C for 2h. Then, the samples were  
203 extracted from the climatic chamber and immediately submitted to a capillarity test with  
204 water at  $23 \pm 1$  °C, for both experiments. These tests were performed on two samples of  
205 each granite with dimensions of 10 mm × 40 mm × 40 mm and the same samples were used  
206 for every cycle within the same experience. The samples were suspended from an electronic  
207 precision balance, with a readability of 0.1 mg, using a hook and put into contact with  
208 distilled water from their bottom face with a fringe of about 1 mm (Fig. 2). The weight was  
209 automatically recorded every 10 seconds on a control computer. The temperature of the  
210 room was kept at  $23 \pm 1$  °C. The test was carried out for 1 hour, time enough for all the  
211 granites to reach the stabilisation of the water uptake. Capillary kinetics are usually  
212 characterised by two phases (Hammecker et al., 1993; Hammecker and Jeannette, 1994).  
213 The first phase is the progressive filling of the free porosity by the capillary forces of the  
214 water without external pressure applied. The slope of this curve that corresponds to the  
215 coefficient C ( $\text{g}\cdot\text{m}^{-2}\cdot\text{s}^{-1/2}$ ) (Roels et al., 2000) and depends on the porous network. The second  
216 phase, slower, begins after the break of slope and corresponds to the filling of the porous  
217 network by diffusion of water in the air.

218 The coefficient C was calculated for each sample, per cycle, to assess the water kinetic  
219 evolution and consequently the variations in the porous network. This test was carried out in  
220 two samples to verify the repeatability. For heterogeneous rocks with low porosity, the  
221 coefficients evolution of the two samples of each granite assessed individually were more  
222 relevant than an average and standard deviation to avoid smoothing of the mean and no  
223 longer perceive the changes.

224 Only for EXP2, IRT monitoring was used to evaluate the microcracking simultaneously to  
225 CWUT (Fig. 2). The IRT camera used is a FLIR SC655, operating in wavelengths between 7.5  
226 and 14  $\mu\text{m}$ . The detection temperature of the camera is comprised between -40 and 150 °C  
227 with a sensitivity of 0.1 °C. The detector is an uncooled array of microbolometers. The used  
228 IRT camera can build images using infrared radiation. The image size is 640 × 480 pixels, with  
229 a noise signal of about 40 mK. The recorded signal, called thermosignal (TS), depends on  
230 temperature and emissivity and is expressed in isothermal units (IU).

231 The cooling kinetics of the samples from 40 °C to room temperature was followed by IRT  
 232 after each heating cycle. The thermal images show that the water spreading into the  
 233 microstructure and propagating according to the physical characteristics of the material,  
 234 leading to a possible evaluation of the porosity (Ludwig et al., 2018). The bottom part of the  
 235 sample was cooled by the water rising by capillarity. The upper part of the sample was not  
 236 affected by the capillary forces and was used to monitor the cooling by the air.



237  
 238 Figure 2: Experimental setup of infrared thermography (IRT) monitoring of a sample  
 239 submitted to capillary absorption.

240 The temperature contrast between the rock and the water facilitated the IRT image  
 241 assessment. This procedure made it possible to observe the progress of the water on the  
 242 different samples since the rise of the water caused a cooling of the rock and different  
 243 emissivity of the two media. In the experimental setup, the risks associated with the  
 244 environmental variations were minimised. The temperature and humidity were monitored  
 245 with a thermometer and a hygrometer. The experimental device was placed in a watertight  
 246 tank which allowed to minimise evaporation phenomena as much as possible. Tests were  
 247 performed in the darkness without artificial light.

248 The IRT camera was configured to record the thermal images at a rate of 1 frame every 10  
 249 seconds for 30 minutes and was activated 1 minute after the start of the experiment to  
 250 avoid the wide signal variations during the calibrations due to the initial setup. Each image or  
 251 thermogram of the whole face of each sample acquired during the experiment was  
 252 processed by the FLIR RESEARCHIR software<sup>®</sup>. Each pixel of this thermogram corresponds to

253 a specific temperature value. The interface allows placing control spots of 3 × 3 pixels on the  
254 thermogram (C1 to C7, Fig. 2). Five monitoring spots were placed vertically on the sample  
255 area, spaced 8 mm. Two spots were placed on the left and right side, centred vertically to  
256 observe the possible lateral variations. The "Temporal Plot" function allows us to have the  
257 evolution of the thermo-signal as a function of the acquisition time for each control spot.

258 To quantify the cooling rate, we used the cooling rate index (CRI) representing the  
259 temperature variation per unit of time. The CRI10 (calculated for the first 10 minutes of the  
260 test) was calculated according to the equation (1) on the 7 control spots of the thermograms  
261 and at each cycle. CRI10 index was described for the first time by Pappalardo et al. (2016) in  
262 the survey of rock masses as a reliable and best-suited index for the indirect quantification of  
263 porosity. According to Mineo and Pappalardo (2016), the cooling curves of a rock show their  
264 major evolution during the first 10 minutes, so that this interval was the one chosen for the  
265 CRI analysis. This parameter is inspired by Newton's law of cooling, which states that the  
266 heat loss of a body is proportional to the temperature difference between the sample and  
267 the environment. This law confirms that cooling is faster in the first minutes of the  
268 experiment due to the higher temperature difference between the sample and the air or the  
269 water. A fractured rock would cool down faster than a non-fractured one. Thus, this index  
270 allows to compare the temperature variation as a function of time of the 4 granites during  
271 heat treatment cycles and indirectly the microcrack evolution.

$$272 \quad \text{CRI10} = \Delta T / \Delta t = (T_{10} - T_0) / (t_{10} - t_0) \quad (1)$$

273 Where  $\Delta T$  is the variation in temperature between initial temperature ( $T_0$ ) and temperature  
274 after 10 minutes ( $T_{10}$ ) and  $\Delta t$  is the duration between the two temperature values, i.e. 10  
275 min. High CRI10 values corresponded to a fast cooling and higher cracking than low values  
276 that represented a slow cooling.

277 The low porosity of the granites together with the evaporation and the air-environment  
278 makes it difficult to distinguish accurately the water limit of the capillary fringe with the  
279 naked eye and on the IRT images. The SURFER software® was used to facilitate the  
280 visualisation of the CRI10 and the capillary fringe on the surface of the samples. This  
281 software transformed the CRI10 data on grids in 2D by, in this case, the kriging method.  
282 Figure 3a shows the schematic distribution of CRI10 on the surface of a sample. The X and Y

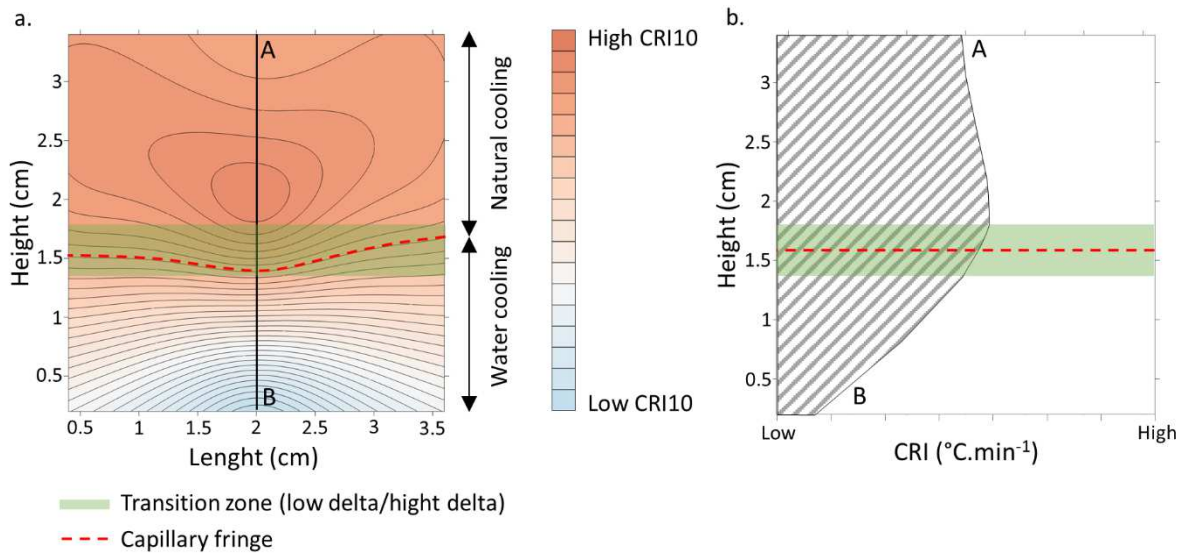
283 axes of the grids represented the dimensions of the sample in centimetres. For a time-lapse  
284 of 10 min, the placement of the isolines made it possible to detect the thermal contrast  
285 between wet and dry areas.

286 It has been considered 2 heat transfers: the air cooling and the water cooling. In both cases,  
287 the sample was at a temperature of 40 °C and the water and air about 20 °C lower.

288 - The air cooling on the top of the sample was quantified thanks to the C5 spot (the  
289 highest on the sample). Within the 10-minute time-lapse the capillary fringe of the  
290 water was not able to reach the C5 spot so that it did not influence the natural  
291 cooling of the rock at this point. In addition, this control spot was the closest to the  
292 upper surface, in which the temperature equilibrium was reached faster. Thus, the  
293 CRI10 (C5) values corresponded to the air-cooling velocity of the rock.

294 - The water cooling of the bottom of the sample was produced by CWUT. The bottom  
295 part showed slower cooling rates than the upper part due to its fast temperature  
296 decrease during the first-minute contact with the water. The diffusion of water in the  
297 sample resulted in isolines of low value, horizontal and close together (Fig. 3a). The  
298 water cooling of the bottom of the sample was quantified by the C1 spot. Thus, the  
299 CRI10 (C1) measurement corresponded to the equilibrium research between the rock  
300 and the water temperature.

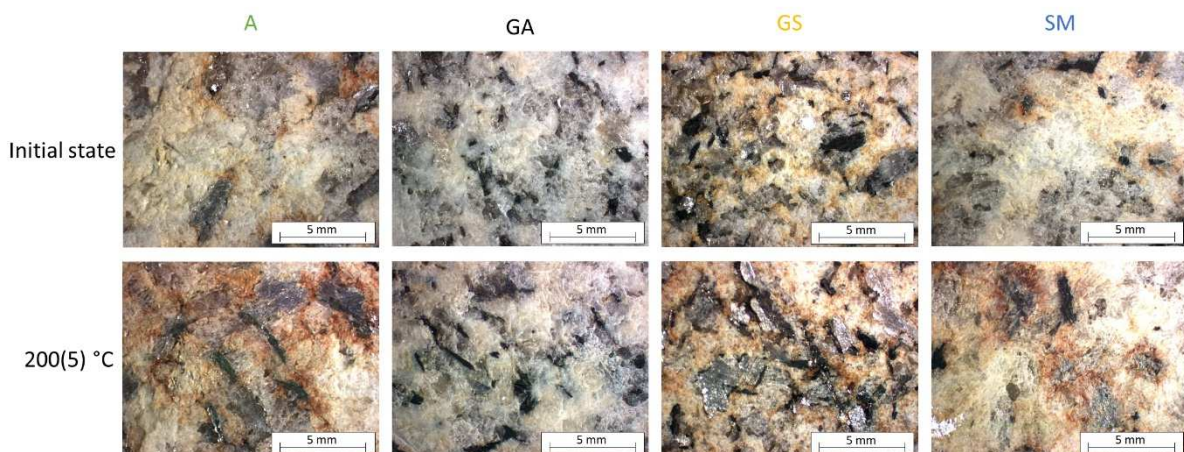
301 The vertical AB profile centred horizontally on the surface of the sample showed that CRI10  
302 increase from bottom to top until reaching a plateau (Fig. 3b). The break-in slope indicated  
303 the transition zone between the cooling by water and the natural cooling. The height of the  
304 capillary fringe was adjusted using isolines. This limit was represented by the red dashed line  
305 in figure 3. The evolution of this height during the different thermal cycles corresponded to  
306 changes in the CWUT and thus in the microstructure of the rock. This parameter was used as  
307 comparative values between the cycles.



308  
 309 Figure 3: (a) Schematic representation of the cooling rate index (CRI10) on the surface of the  
 310 samples. (b) CRI10 values along the vertical profile AB. The red dotted line is positioned in  
 311 the centre of this area. Its position is refined thanks to the isolines on the surface of the  
 312 sample. Cooling rate index (CRI10) representing the temperature variation per unit of time  
 313 (10 min).

## 314 4\_RESULTS

315 The observations with the naked eye did not reveal any microcracks on the samples after the  
 316 two treatments. The colour of A, GS, and SM has changed slightly with an accentuation of  
 317 the reddish tone on the surface, mainly concentrated in the crystal boundaries at 200(5) °C  
 318 due to the iron oxidation of clays (Vázquez et al., 2016). Clay minerals and phyllosilicates are  
 319 more sensitive to heat and can be destabilised and undergo several transformations at high-  
 320 temperatures (Hajpál and Török, 2004). Figure 4 shows the samples before and after EXP2.



321  
 322 Figure 4: Sample surfaces before and after 5 cycles of heat treatment of EXP2. Albero (A);

323 Gris Alba (GA); Golden Ski (GS); Silvestre Moreno (SM). Initially, the yellow-brown colour is  
 324 due to feldspar weathering to clay minerals. A red colour change at crystal boundaries was  
 325 observed at 200(5) °C due to the iron oxidation of those clays.

## 326 4.1 Microstructural analysis

### 327 4.1.1 Mercury intrusion porosimetry (MIP)

328 The analysis was carried out on the 4 granites before heat treatment and after each cycle of  
 329 EXP1 (90, 100, 110, 120, and 130 °C) as well as for the first and last cycle of the EXP2 (200(1)  
 330 °C and 200(5) °C). The porosity (%) measured by MIP of the samples after EXP1 and EXP2 are  
 331 shown in table 3.

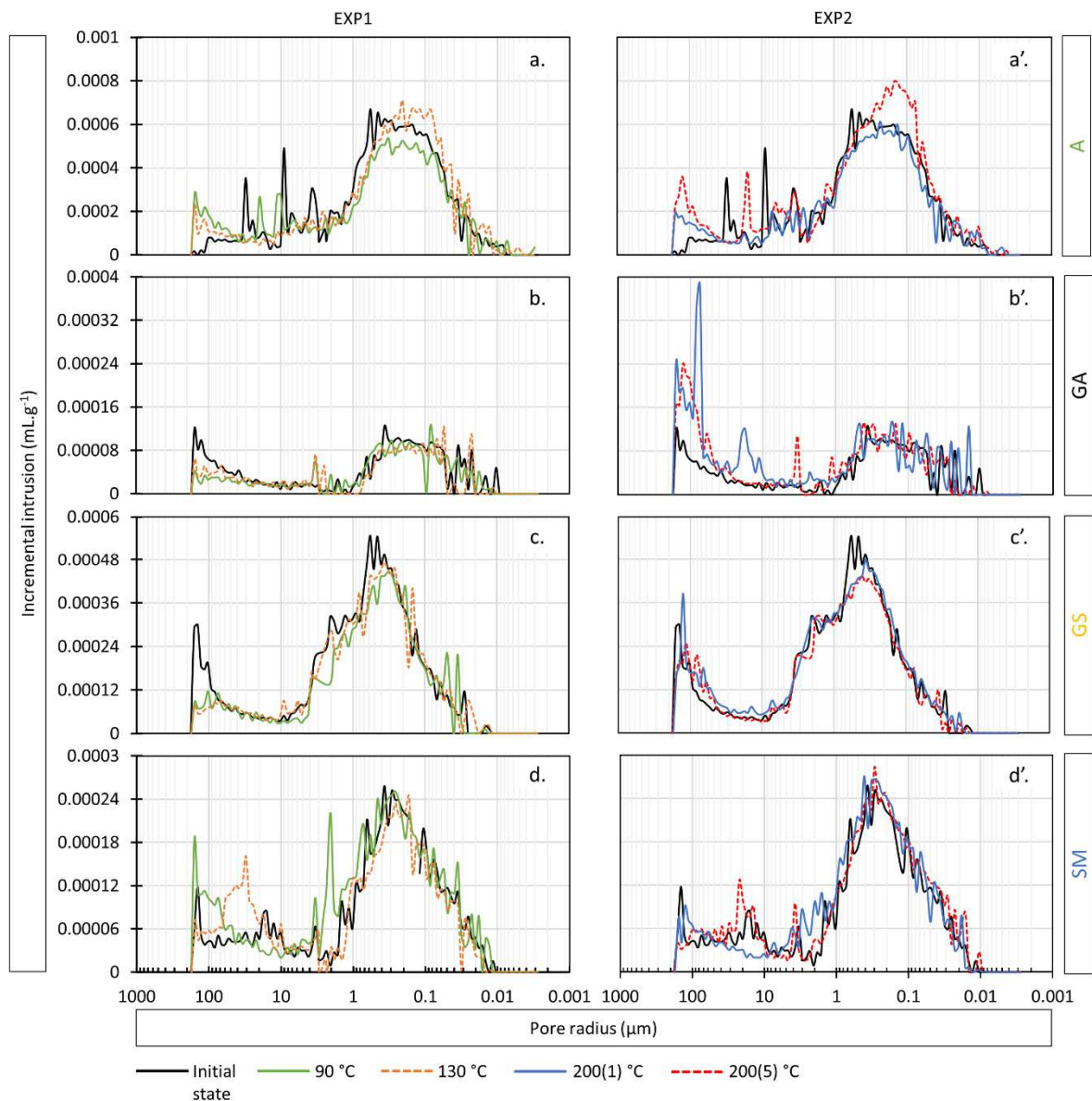
332 Table 3: Total porosity determined by MIP (%) of the 4 granites before and after treatment  
 333 at different temperatures. Underlined values mean lower values than the fresh rock, in bold  
 334 the higher values and bold red the first higher value stated as the microcracking threshold.

	EXP1						EXP2	
	Initial state	Cycle 1 90 °C	Cycle 2 100 °C	Cycle 3 110 °C	Cycle 4 120 °C	Cycle 5 130 °C	Cycle 1 200(1) °C	Cycle 5 200(5) °C
A	5.43	<u>5.03</u>	<u>4.72</u>	<u>4.81</u>	<u>3.76</u>	<b>5.58</b>	<u>5.15</u>	<b>6.56</b>
GA	1.05	<u>0.87</u>	<u>0.95</u>	<u>0.96</u>	<u>0.95</u>	<u>0.80</u>	<b>1.59</b>	<b>1.39</b>
GS	3.77	<u>3.19</u>	<u>3.08</u>	<u>3.68</u>	<b>3.93</b>	<u>3.42</u>	<b>3.97</b>	<u>3.56</u>
SM	1.97	<b>2.30</b>	<b>1.98</b>	<b>2.44</b>	<b>2.25</b>	<b>2.01</b>	<b>2.12</b>	<b>2.16</b>

335

336 Through EXP1, three of the granites (A, GA, GS) experienced firstly a decrease in porosity.  
 337 Three values (in bold red), at 130 °C for A, 120 °C for GS, and 90 °C for SM were higher than  
 338 the initial porosity and the preceding value. GA had not shown any increase in its porosity  
 339 during EXP1.

340 Through EXP2, the general behaviour was an increase in porosity with high-temperatures,  
 341 although value fluctuations indicated that crystal and microcracks adjustment was still taking  
 342 place.



343  
 344 Figure 5: Pore access size distribution curves for EXP1 and EXP2. The curves show the  
 345 granites tested at room temperature (initial state), 90 °C, 130 °C, 200(1) °C and 200(5) °C. (a-  
 346 a') Albero, (b-b'), Gris Alba, (c-c') Golden Ski, (d-d') Silvestre Moreno.

347 The initial MIP curves of the four granites (Fig. 5) can be divided into two parts: i) a main  
 348 distribution between approximately 0.01 μm and 1-10 μm with unimodal shape; ii) and a  
 349 heterogeneous distribution of the higher pore radii access.

350 EXP1:

351 A showed a main pore distribution in the range of 0.007 to 3 μm (Fig. 5a). The other well-  
 352 defined subfamily with a heterogeneous distribution showing peaks at 5 and 31 μm. There  
 353 were variations between MIP yield curves for samples at the initial state and stressed at 90

354 °C and 130 °C. The main part of the curve showed a volume diminution at 90 °C then  
355 recovery at 130 °C. The heterogeneous peaks found at greater pore sizes vanished after  
356 heating.

357 GA is the lowest porous material. The main family curve is observed between 0.01 and 1 µm  
358 with the value of higher intensity at 0.4 µm (Fig. 5b). The second family corresponded to a  
359 pore access radius from 31 to 157 µm. The pore peak of the last family (about 100 µm)  
360 decreased from the first cycle. The last cycle was marked by the apparition of a threshold  
361 family around 1-3 µm. The porous volume associated with the mean threshold family slightly  
362 decreased with temperature and redistribution of peaks for values under 0.1 µm took place.

363 GS had a mean threshold family between 0.01 and 10 µm and a heterogeneous family with a  
364 mode at 143 µm (Fig. 5c). The curve of the sample heated to 90 °C exhibited a volume  
365 reduction for the threshold between 0.2 and 3 µm, slightly recovered at 130 °C.  
366 Furthermore, the large decrease of the pore volume associated with high pore radius sizes  
367 was observed from the first heating cycle.

368 SM showed its main pore family dispersed between about 0.01 and 2 µm (Fig. 5d). An  
369 increase in porosity at 90 °C was marked by the appearance of a peak centred at 2 µm radius  
370 and a gradual increase of the pore volume thereof. At 130 °C, the main distribution did not  
371 differ from fresh results, while the pore volume accessible through threshold in the range  
372 10-100 µm increases.

373 EXP2:

374 For A, the first episode of heating at 200 °C (200(1) °C) induced a general reduction of the  
375 pore volume distributed through all the curve, although a redistribution was observed in  
376 pores greater than 34 µm (Fig. 5a'). At the end of EXP2, A showed an increase in porosity of  
377 21% (up to 6.56%). On pores with an access radius greater than 1 µm, the shift of some  
378 peaks and the volume increase towards a larger radius (110 µm) indicated a widening of the  
379 microcracks during the fifth heating cycle (200(5) °C).

380 For GA, a development of heterogeneities with heating was observed in the main threshold  
381 family (Fig. 5b'). After the first cycle at 200 °C, significant pore radius microcracks formation  
382 centred at 18 and 74 µm appeared, the latter softened during the fifth heating cycle. It was



383 mainly the large accesses of the distribution that were concerned. Porosity values were in  
384 agreement with this increase.

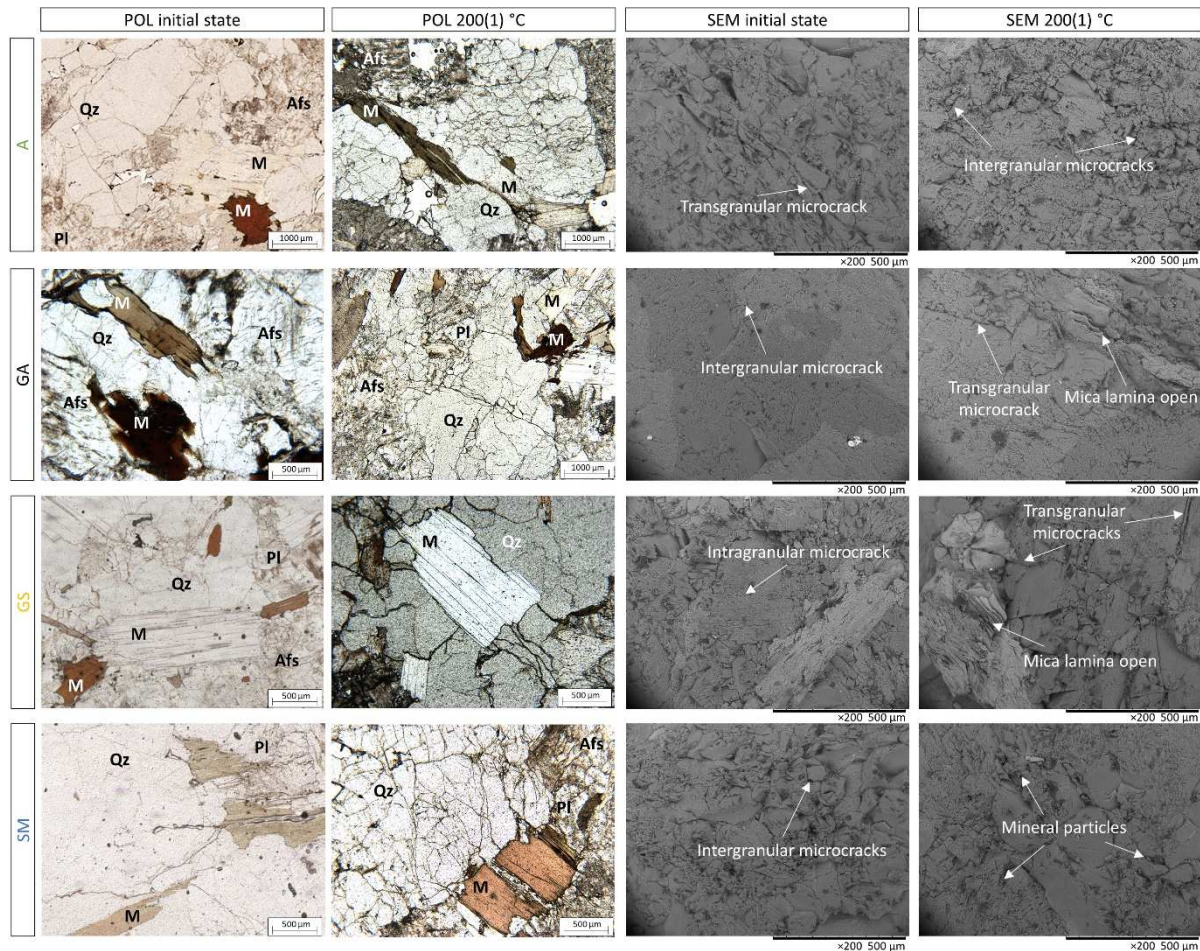
385 For GS, there was a decrease in the main area family, between 0.4 and 0.7  $\mu\text{m}$  at 200(1)  $^{\circ}\text{C}$   
386 and between 0.2 and 0.7  $\mu\text{m}$  at 200(5)  $^{\circ}\text{C}$  (Fig. 5c'). At 200(1)  $^{\circ}\text{C}$ , a peak between 6 and 126  
387  $\mu\text{m}$  was formed and then softened at 200(5)  $^{\circ}\text{C}$ .

388 For SM, the distribution was very irregular over the entire pore radius range after the first  
389 heating cycle, then returned to close to the initial state at the end of the test (Fig. 5d'). The  
390 porosity volume only increased slightly, also according to the curve variations.

#### 391 4.1.2 Optical polarising microscopy (POL) and scanning electron microscopy (SEM)

392 The pre-existing microcracks present naturally in the granites were enhanced by the thermal  
393 effects. The observation and comparison of the microscopic images of the fresh and the  
394 heated up to 130  $^{\circ}\text{C}$  granites revealed little change, while at 200(1)  $^{\circ}\text{C}$ , the variations were  
395 slight although evident enough for description. Figure 6 shows microscopy images (POL and  
396 SEM) of fresh and thermally damaged granite, 200(1)  $^{\circ}\text{C}$ .

397 After heating, each mineral reacts differently to heat treatment. Thus, new-formed  
398 microcracks were observed on the surface of quartz crystals in A but especially in the less  
399 weathered granites GA and SM. Mica sheets were slightly raised due to thermal expansion  
400 and exhibited microcracking and a higher relief in GA and GS. Mineral particles were  
401 detached on the surface of the alkali feldspar crystals in SM.



402  
 403 Figure 6: Optical polarising microscopy (POL) and scanning electron microscopy (SEM)  
 404 illustrating the mineralogy (Qz: quartz; Afs: alkali feldspar; Pl: plagioclase; M: mica) and the  
 405 microcracking of the four granites before and after one thermal treatment at 200 °C (200(1)  
 406 °C). Albero (A), Gris Alba (GA), Golden Ski (GS), and Silvestre Moreno (SM).

407 For A, the heated samples (200(1) °C) showed a crystal surface with a rougher appearance in  
 408 SEM observations. Intragranular microcracks were already present in the quartz crystals and  
 409 clearly developed when compared to the POL observations of a heated and fresh rock. The  
 410 mica presented a slight opening. Microcracks had an average aperture of more than 10 μm  
 411 after heating and did not present any particular orientation.

412 For GA, before the thermal treatment, the POL observations showed predominantly an  
 413 intergranular microcracks network. The POL observations at 200(1) °C showed an increase of  
 414 intergranular microcracks between the Afs-Pl limit, thus propagating microcracking along the  
 415 boundaries of adjacent crystals. Notable intragranular microcracks were also present in  
 416 quartz and alkali feldspar crystals. After one cycle at 200 °C, the pre-existing microcracks  
 417 became larger and slightly wider in diameter in SEM observations. Some intergranular

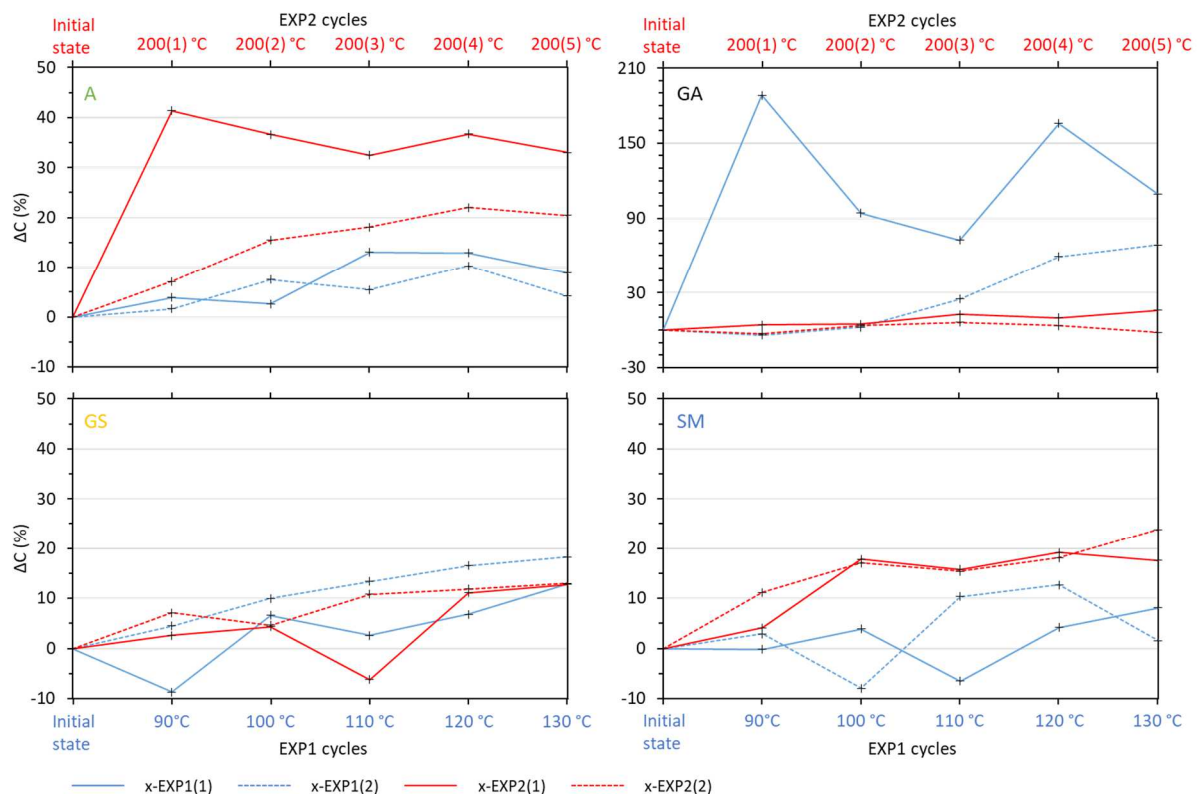
418 microcracks are connected to long transgranular microcracks. The thermal expansion led to  
 419 visible damage to the GA structure, especially on mica which had an increase in their  
 420 roughness.

421 For GS, the POL observations showed a slight opening of mica. The SEM observations  
 422 revealed that GS was marked by an initial alteration with a visible transgranular  
 423 microcracking. This granite had pores larger than 30  $\mu\text{m}$  in diameter. After heating, many  
 424 new microcracks had appeared especially in quartz crystals. Cleavage of mica was also  
 425 altered, showing irregularities.

426 For SM, after treatment, the POL observations showed the development of an intragranular  
 427 microcracking through quartz crystals as well as the advanced deterioration of mica. SM  
 428 exhibited great diversity in the size and nature of the microcracks. There was also a  
 429 detachment of microparticles on the surface of the pre-fractured sample at 200(1)  $^{\circ}\text{C}$ .

#### 430 4.1.3 Capillary coefficient (C)

431 The curves of figure 7 represent the evolution of the capillary coefficient (C) concerning the  
 432 cycles of the 2 experiments.



433  
 434 Figure 7: Evolution of the capillary coefficient (C) during the 2 heat treatments (EXP1 in blue  
 435 and EXP2 in red). The full line corresponds to sample 1 and the dotted line to sample 2.

436 In general, for both experiments, the coefficient C increased with the thermal cycles. For A-  
437 EXP2(1) and GA-EXP1(1), the evolution was non-linear, influenced by a large variation during  
438 the first heating cycle. The redistribution of microcracks during the different experiments  
439 was noticeable by the non-linear variations of the coefficient C during cycles: some isolated  
440 low values and large variability of values between samples of the same granite. The initial  
441 heterogeneity of the sample may explain the differences between the acquired data.

442 EXP1:

443 Overall, the samples experienced an increase in their coefficients C after heating. For A, the  
444 initial coefficients C of the two samples A-EXP1(1) and A-EXP1(2) were 24.9 and 20.1  $\text{g}\cdot\text{m}^{-2}\cdot\text{s}^{-1/2}$   
445 respectively. At, 90 °C the variation was not very evident. The maximum values were  
446 reached at 110 °C for sample A-EXP1 (1) with a coefficients C of 28.2  $\text{g}\cdot\text{m}^{-2}\cdot\text{s}^{-1/2}$  and at 120 °C  
447 for the other (A-EXP1 (2)) with a coefficient C of 22.2  $\text{g}\cdot\text{m}^{-2}\cdot\text{s}^{-1/2}$ .

448 For GA, the initial coefficients C of the two samples GA-EXP1(1) and GA-EXP1(2) were 1.8  
449 and 2.1  $\text{g}\cdot\text{m}^{-2}\cdot\text{s}^{-1/2}$  respectively. This unaltered granite had the highest variability of  
450 coefficients C during the cycles. The increase was high from the first cycle for the first sample  
451 (GA-EXP(1)) with a coefficients C of 5.08  $\text{g}\cdot\text{m}^{-2}\cdot\text{s}^{-1/2}$ , while the C increase was gradual up to 130  
452 °C for the second sample GA-EXP(2) up to 3.67  $\text{g}\cdot\text{m}^{-2}\cdot\text{s}^{-1/2}$  at the end of the treatment.

453 For GS, the initial coefficients C of the two samples GS-EXP1(1) and GS-EXP1(2) were 21.9  
454 and 19.0  $\text{g}\cdot\text{m}^{-2}\cdot\text{s}^{-1/2}$  respectively. At 90 °C, there was a decrease for GS-EXP1(1) and no  
455 important change for the GS-EXP1(2). The maximum value was observed at 130 °C with  
456 values of 24.7 and 22.6  $\text{g}\cdot\text{m}^{-2}\cdot\text{s}^{-1/2}$  for GS-EXP1(1) and GS-EXP1(2) respectively.

457 For SM, the initial coefficients C of the two samples SM-EXP1(1) and SM-EXP1(2) were 9.7  
458 and 10.7  $\text{g}\cdot\text{m}^{-2}\cdot\text{s}^{-1/2}$  respectively. The trends in this granite with the increasing temperature  
459 were irregular, with a sharp decrease at 110 °C for SM-EXP1(1) and at 100 °C for the second  
460 one. The maximal coefficients C was observed at 130 °C and 120 °C with values of 10.5 and  
461 10.8  $\text{g}\cdot\text{m}^{-2}\cdot\text{s}^{-1/2}$  for SM-EXP1(1) and SM-EXP1(2), respectively.

462 EXP2:

463 The general trends after this test were a gradual increase in the C coefficient.

464 For A, an increase of C was measured on both samples. The first sample (A-EXP2(1))  
465 increased after the first cycle from 13.2 to 18.7  $\text{g.m}^{-2}.\text{s}^{-\frac{1}{2}}$  then nearly stabilised over the other  
466 four cycles. The coefficient C of the second sample (A-EXP2(2)) gradually increased until the  
467 fifth cycle (200(5) °C) from 21.0 to 25.3  $\text{g.m}^{-2}.\text{s}^{-\frac{1}{2}}$ .

468 For GA, the overall trend was an augmentation up to the third cycle (200(3) °C). The  
469 coefficients C gained from 4.8 to 5.4  $\text{g.m}^{-2}.\text{s}^{-\frac{1}{2}}$  for GA-EXP2(1) while it remained without  
470 variation for the other sample.

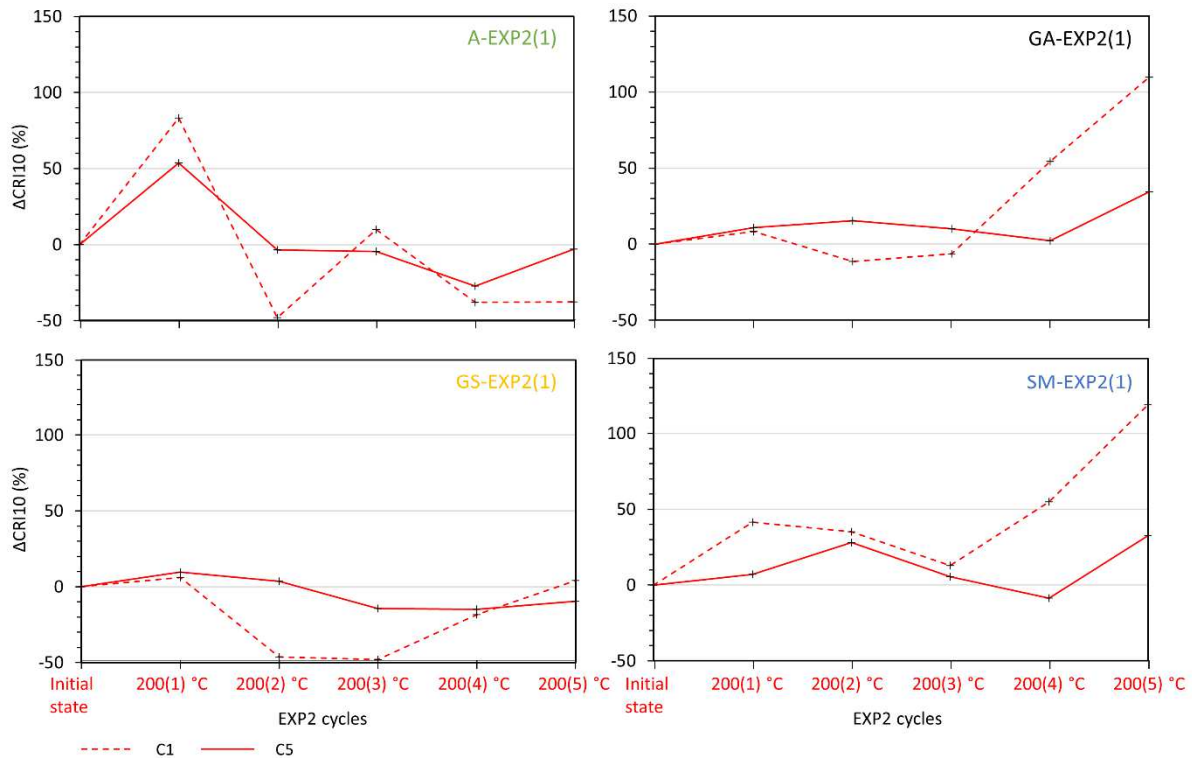
471 For GS, the capillary absorption of both samples changed similarly, except for the decrease  
472 in GS-EXP2(1) in the third cycle (200(3) °C). Both samples showed a final increase of the  
473 same magnitude compared to their initial state.

474 For SM, the two samples showed a notable increase either during the first or second cycle.  
475 The coefficients C then remained stable around 13-14  $\text{g.m}^{-2}.\text{s}^{-\frac{1}{2}}$  until the last cycle.

#### 476 4.2 Infrared thermography (IRT) monitoring of sample cooling

477 In EXP2, the CRI10 evolution of the air cooling was measured at the level of the control spot  
478 5 (C5) and the water cooling at the level of control spot 1 (C1) (Fig. 2 and 8). The changes of  
479 CRI10 values were not linear through the cycles. However, the main trend revealed clearly a  
480 strong decrease of the CRI10 in A and a slight one in GS, while GA and SM experimented an  
481 increase.

482 The initial values of CRI10 (C5) were 0.76  $^{\circ}\text{C}.\text{min}^{-1}$  for A and 0.82  $^{\circ}\text{C}.\text{min}^{-1}$  for GS. GA and SM  
483 have intermediate values of 0.77  $^{\circ}\text{C}.\text{min}^{-1}$  and 0.78  $^{\circ}\text{C}.\text{min}^{-1}$ , respectively. The initial CRI10  
484 (C1) was lower in the low porosity GA and SM (0.25  $^{\circ}\text{C}.\text{min}^{-1}$ ) and higher in the high porosity  
485 A and GS (0.42  $^{\circ}\text{C}.\text{min}^{-1}$ ).



486  
 487 Figure 8: Evolution of the CRI10 measured at the top of the sample (air cooling-spot C5) and  
 488 the bottom (water cooling-spot C1) through EXP2.

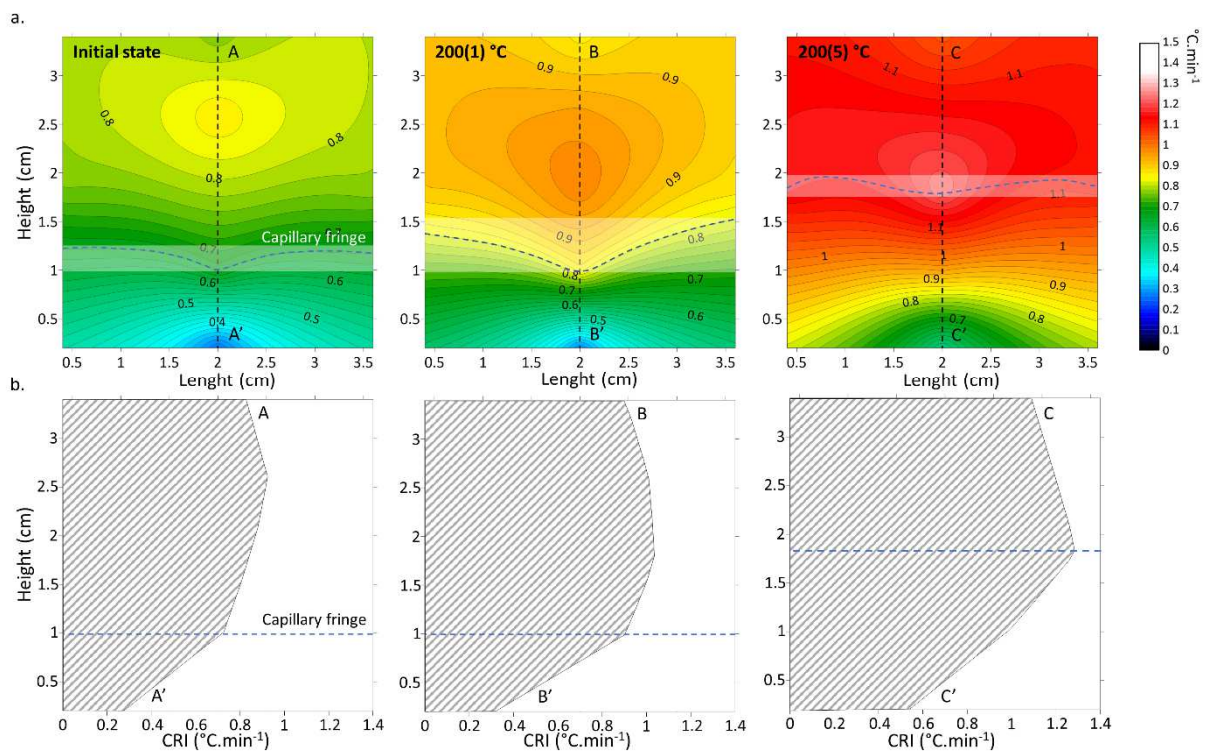
489 For A, the values through the cycles were the most heterogeneous. It was observed that the  
 490 air cooling (C5) accelerated from the first heating cycle with an increase of about 50% to the  
 491 value of  $1.17 \text{ }^\circ\text{C}\cdot\text{min}^{-1}$ . The CRI10 measured in the lower part of the sample (C1) also  
 492 identified an increase of about 80% and reached a value of  $0.76 \text{ }^\circ\text{C}\cdot\text{min}^{-1}$ . The following cycle  
 493 showed a return to the original values of the CRI10 cooled down by air (C5) although there  
 494 was a decrease of about 30% for the cycle at  $200(4) \text{ }^\circ\text{C}$ . Water cooling showed a decrease to  
 495  $0.22 \text{ }^\circ\text{C}\cdot\text{min}^{-1}$  in the cycle  $200(2) \text{ }^\circ\text{C}$ , returns to the origin in the  $200(3) \text{ }^\circ\text{C}$  cycle, and a  
 496 decrease to  $0.26 \text{ }^\circ\text{C}\cdot\text{min}^{-1}$  (about 40%) in the last 2 cycles.

497 For GA, the final state showed a clear progression compared to the initial state. Whether for  
 498 the top or the bottom of the sample, the CRI10 did not exceed variations of 15% during the  
 499 first three cycles. The air cooling that took place at the top of the sample increased by about  
 500 30% with a final value of  $1.03 \text{ }^\circ\text{C}\cdot\text{min}^{-1}$ . It was during the fourth cycle that the water cooling  
 501 increased to  $0.39 \text{ }^\circ\text{C}\cdot\text{min}^{-1}$  and during the last cycle, the CRI10 was twice its initial value with  
 502 a value of  $0.53 \text{ }^\circ\text{C}\cdot\text{min}^{-1}$ .

503 For GS, variations in the air cooling were small. The most important changes were identified  
 504 from cycle 3 marked by a decrease of about 10-15%. Water cooling decreased by about 50%  
 505 ( $0.22\text{ }^{\circ}\text{C}\cdot\text{min}^{-1}$ ) in cycle 2 and 3 and by 20% ( $0.34\text{ }^{\circ}\text{C}\cdot\text{min}^{-1}$ ) in cycle 4.

506 For SM, air cooling increased in cycle 2 and 5, with an increase of 30%. The CRI10 in the  
 507 lower part (C1) showed an increase of more than twice the initial value up to  $0.55\text{ }^{\circ}\text{C}\cdot\text{min}^{-1}$  in  
 508 the last cycle.

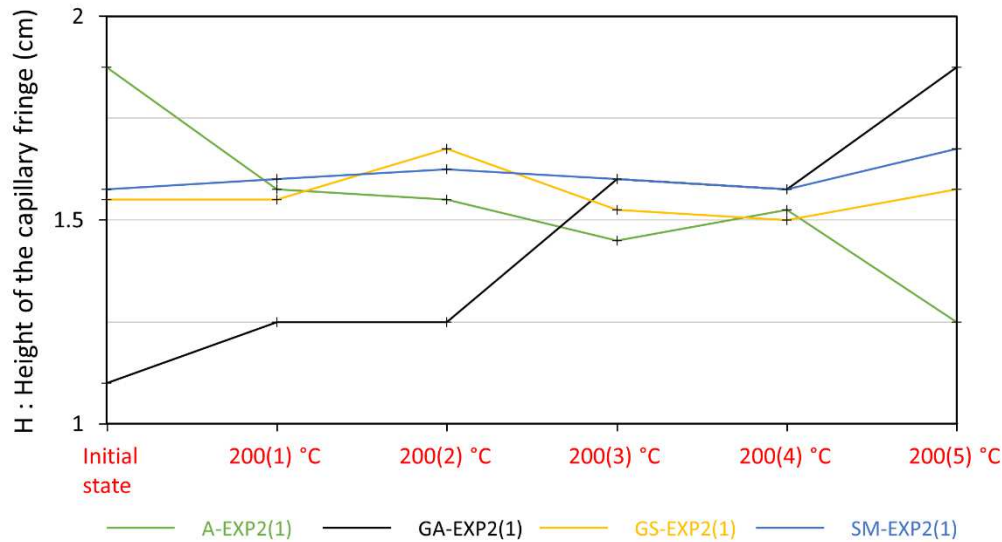
509 The mapping of the CRI10 distribution on the sample surface showed the air-water cooling  
 510 contrast that was allowed to detect the capillary fringe. The example of GA is shown in figure  
 511 9 in which a clear elevation of the capillary fringe was measured through the cycles. For the  
 512 other granites, the change in the height of the capillary fringe (H) during EXP2 is shown in  
 513 figure 10.



514 Figure 9: Example of CRI10 mapping (a) and associated profiles (b) obtained by infrared  
 515 thermography (IRT) monitoring on GA before and after one thermal cycle at  $200\text{ }^{\circ}\text{C}$  (200(1)  
 516  $^{\circ}\text{C}$ ) and five thermal cycles (200(5)  $^{\circ}\text{C}$ ): the dot blue line corresponds to the transition zone  
 517 and is assumed to correspond to the position of the capillary fringe.

519 Before the thermal treatment, the capillary height of A was about 1.8 cm. For GA, this height  
 520 was about 1 cm while GS and SM were in between.

521 A was marked by a general decreasing trend while for GA, the capillary fringe showed a  
 522 gradual rise from approximately 1.0 cm to 1.8 cm after the last cycle. For SM and GS, the  
 523 height of the fringe showed little variation and remained stable.



524  
 525 Figure 10: Height of the capillary fringe after the first 10 minutes (H) of capillary absorption  
 526 of the four granites before and after thermal cycles at 200 °C (EXP2).

## 527 5\_DISCUSSION

528 The exposition of granitic rocks to high-temperatures causes an expansion of the rock-  
 529 forming minerals and a microcrack development above a specific temperature threshold  
 530 (Argandoña et al., 1985; Géraud et al., 1992; Gómez-Heras et al., 2006; Menéndez et al.,  
 531 1999). This thermal behaviour depends mainly on two factors, mineralogy and porosity  
 532 (Benavente et al., 2006; Freire-Lista et al., 2016; Houpert and Homand-Etienne, 1986;  
 533 Miskovsky et al., 2004). Many researchers agree that the microcrack thermal threshold of  
 534 granitoids often begins at a temperature of around 100-200 °C (tab. 4).

535 Table 4: Some previous researches on the thermal microcracking threshold.

Author	Granite	Porosity (%)	Threshold (°C)
(Chen et al., 2017b)	Fujian Province (China)	-	200-400
(Darot et al., 1992)	La Peyratte (France)	-	125
(Argandoña et al., 1985)	Gondomar granodiorite (Spain)	1.37	110-115
(Dwivedi et al., 2008)	Indian granite	0.8	65
(Fan et al., 2017)	Zhejiang Province (China)	-	200-400
(Gautam et al., 2018)	Jalore granite (India)	0.115	300



(Géraud et al., 1992)	Massif de la Borne-Pyrénées-Vendée (France)	0.48-2.3	50-200
(Guo et al., 2018)	Granodiorite (China)	1.32	120-150
(Homand-Etienne and Houpert, 1989)	Senones and Remiremont granite (France)	-	200-400
(Jansen et al., 1993)	Lac du Bonnet Granite (Canada)	0.24	80
(Jin et al., 2019)	Shandong province (China)	-	400-500
(Kumari et al., 2017)	Strathbogie batholith (Australia)	1.16	400
(Lin, 2002)	Inada granite (Japan)	0.75	100-125
(Liu and Xu, 2014)	Qinling granite (China)	-	100-200
(Meredith and Atkinson, 1985)	Westerly granite (New England)	1	100-200
(Shao et al., 2015)	Strathbogie granite (Australia)	0.463	200-400
(Singh et al., 2015)	Bundelkhand granite (India)	-	400
(Sun et al., 2015)	Jining, Shandong (China)	0.88	300-400
(Takarli and Prince-Agbodjan, 2008)	-	0.68	105-200
(Yang et al., 2017)	Shandong province (China)	0.828	300-400
(Yin et al., 2015)	Laurentian granite (Canada)	0.64	100-250
(Yu et al., 2015)	Dandong Liaoning Province (China)	-	100
(Zhao and Feng, 2019)	Lu gray granite (China)	-	200-300

536 Before reaching the thermal threshold, the rock experimented a pore redistribution, defined  
537 by an opening and closing of the pre-existent microcracks or the development of new ones.  
538 (Géraud et al., 1992) observed the opening of pre-existent biotite and quartz boundaries  
539 between 50 and 100 °C. New microcracks were found between 60 °C and 90 °C by Guo et al.  
540 (2018) and Jansen et al. (1993) showed microcracking in their granite at temperatures above  
541 80 °C. Dwivedi et al. (2008) observed pre-existing microcracks widening at 65 °C, followed by  
542 a closure at temperatures between 100 °C and 125 °C. Lin (2002) observed a microcracks  
543 widening threshold at temperatures between 100 °C and 125 °C. Most of the studies found a  
544 thermal threshold from 100 °C (Argandoña et al., 1985; Yu et al., 2015). Takarli and Prince-  
545 Agbodjan (2008), suggested an increase of microcracking from 105 and 200 °C, according to  
546 also to many studies indicating that the critical temperature of granite microcracking takes  
547 place at higher temperatures, between 200 and 300 °C (Meredith and Atkinson, 1985; Sun et  
548 al., 2015; Yin et al., 2015).

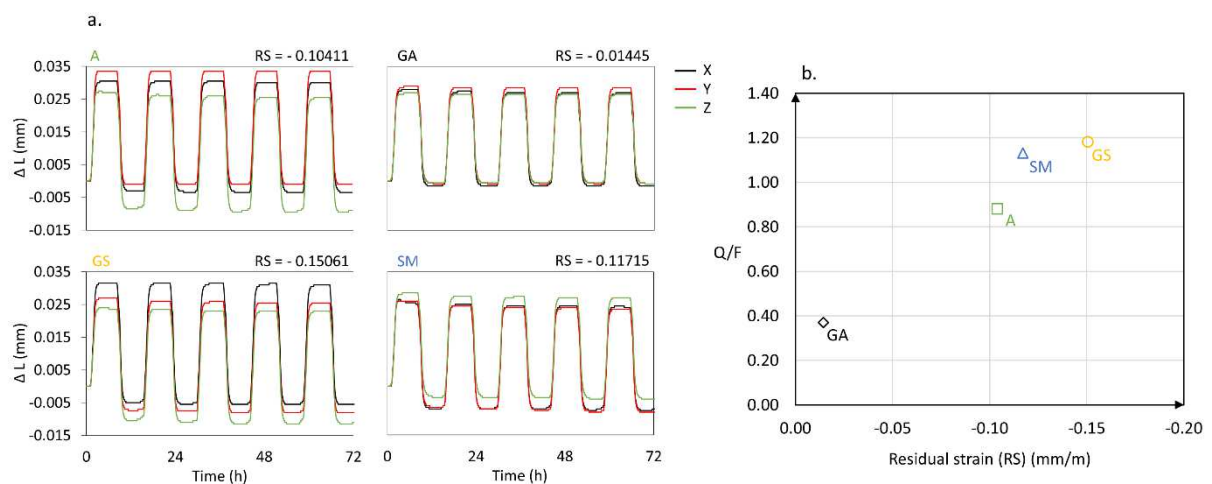
549 Thermal fatigue is an important failure mechanism in granites. Freire-Lista et al. (2016) and  
550 Gómez-Heras et al. (2006) carried out several thermal cycles of continuous heating and  
551 cooling up to 105 °C to granitoids and showed that the thermal cyclic effect causes the  
552 generation of new microcracks and the fusion of pre-existing ones. Lin (2002) performed 5 to  
553 9 cycles at the same peak temperature (from 200 to 600 °C) and observed that at a

554 temperature of 200 °C all the microcracks are produced during the first two heating/cooling  
555 cycles and from 300 °C thermal microcracking continues developing.

### 556 -The effect of mineralogy in granite behaviour

557 One of the main reasons attributed to the generation of microcracks on poly-mineral rocks is  
558 the mismatching of the thermal expansion coefficients of different mineral crystals (Gómez-  
559 Heras et al., 2006). The thermal expansion of quartz is anisotropic ( $a_{11} = 14 \times 10^{-6} \cdot K^{-1}$  and  $a_{33}$   
560  $= 9 \times 10^{-6} \cdot K^{-1}$ ), unlike feldspar with a low and isotropic linear thermal expansion ( $a_{11} = 4.5 \times$   
561  $10^{-6} \cdot K^{-1}$  and  $a_{33} = 4.5 \times 10^{-6} \cdot K^{-1}$ ) (Vázquez et al., 2015). Minerals dilate with temperature but  
562 sometimes they do not recover the original state after cooling, and the rock shows residual  
563 strain.

564 The linear thermal expansion measurements at 90 °C performed by Vázquez et al. (2015,  
565 2011) on the same granites of this study help to understand the behaviour of these granites  
566 during those tests (Fig. 11a). The four studied granites showed a permanent contraction  
567 after the first heating-cooling cycle, more notable for the weathered granites A, GS and SM.  
568 The direct relation between the Qz/F ratio and the residual strain of granites is shown in  
569 figure 11b. Higher quartz content leads to higher residual strain independently of the  
570 porosity of the samples.



571  
572 Figure 11: (a) Thermal dilation curve ( $\Delta L$ ) of the four granites submitted to heating-cooling  
573 cycles over a range of 20 to 90 °C. Residual strain (RS) expressed in mm/m. (b) Relationship  
574 between the Qz/F ratio and average residual strain of A, GA, GS, and SM (Vázquez et al.,  
575 2015, 2011, 2010).

576 **-The effect of initial porosity in granite behaviour**

577 The initial porosity and the microcrack density can influence the thermal alteration of the  
578 granites. Tuğrul and Zarif (1999) found that the influence of textural characteristics on  
579 physical properties seems more important than mineralogy, and Vázquez et al. (2018) stated  
580 that only for sound granitoids with porosity under 2%, mineralogical features influence the  
581 rock behaviour. The mineral expansion of high porosity rocks intrudes on the voids and  
582 reduces the porosity (Géraud et al., 1992). When the rocks are held at the temperature the  
583 cracks generated at this temperature remain closed. During cooling, microstructural  
584 modifications can appear (Homand-Etienne and Houpert, 1989). If the thermal stress does  
585 not reach the microcracks threshold, the minerals will contract during cooling without  
586 generating microcracks. If the thermal stress exceeds the cohesion between the grains, the  
587 minerals by contracting generate microcracks (Dwivedi et al., 2008). The microcrack  
588 geometry (aperture, connection, tortuosity) may play a more important role in the hydric or  
589 thermal properties than the porosity volume itself. For this reason, granite detailed study of  
590 the pore distribution was carried out from MIP results. The threshold was defined for each  
591 granite and the pore access separating the macroporous and microporous domains was  
592 defined according to figure 12, at 3 µm for A, 1µm for GA, 10 µm for GS and 2 µm for SM, as  
593 mentioned in section 4.1.1. That facilitated to understand in which microcrack size domain  
594 the modifications observed in the total porosity took place.



595  
596  
597

Figure 12: Size distribution of the voids. On the top of each column, the porosity by MIP is detailed.

### 598 EXP1: Determination of granite thermal threshold

599 The low initial porosity of SM within the weathered granites, its high quartz (Qz) and mica  
600 content (Vázquez et al., 2015) and its residual strain suggest that the temperature required  
601 for a first closure of the pores could be less than 90 °C. Regarding the porosity values (MIP)  
602 of table 3, SM is the granite that showed firstly an important increase of porosity considered  
603 as a thermal threshold. This occurred at 90 °C and produced mainly an increase in pore  
604 access radii over 2 μm (Fig. 12). The capillary coefficient C of the samples varied slightly  
605 without a clear trend, although the closure of the pores at 100-110 °C took place that  
606 indicated the continuation of the redistribution phase. Beyond these temperatures, the  
607 coefficient C increased slightly. Heating to 110-120 °C would mark the new microcrack  
608 phase. This conclusion is in agreement with the increase in the connected porosity (> 2 μm)  
609 at 110 °C (Fig. 12) that may lead to a more obvious microcracking.

610 In the second most weathered granite, GS, at 90 °C and 100 °C the total porosity and the  
611 radii larger than 10 μm decreased (Fig. 12) in agreement with its residual strain, high Qz/F

612 ratio (Fig. 11) and the variations in the capillary coefficient (Fig. 7). Porosity by MIP indicates  
613 that the microcrack threshold was produced at 120 °C although the created space allowed a  
614 new closure during the last cycle. Capillary water uptake showed a progressive increase from  
615 100 °C that implies the continuous fissuration in the tested samples.

616 The most weathered granite, A, showed contrary results in MIP and capillary coefficient.  
617 From the first heating cycle, a porosity around 100  $\mu\text{m}$  appeared, that favouring the  
618 progressive increase in water uptake by capillarity. Porosimetry showed a progressive  
619 closure up to 120 °C focused on the smaller pores and microcracks that do not affect greatly  
620 the capillary forces, explained by the quartz expansion in the existent fissures. A new  
621 microcracking was generated at 130 °C in the pore family under 3  $\mu\text{m}$ , so that the  
622 temperature range did not allow any pre-existing microcracks widening but possibly  
623 increased the open porosity thanks to the connection of previously closed discontinuities.

624 The results of the capillary water uptake tests of GA were not exactly coincident with MIP  
625 values. The coefficient C depends not only on the porosity volume but on the width and  
626 interconnection between microcracks. The water rises faster when the connections are  
627 larger, more numerous, and more uniformly distributed in the sample. MIP showed a  
628 microcrack closure through all the test explained by the low  $Q_z/F$  ratio (Vázquez et al., 2015).  
629 The slight expansion of the feldspar occupies the existent fissures and microcracks initially  
630 isolated, observed in the intergranular boundaries by microscopic methods, have merged to  
631 increase the vertical connections.

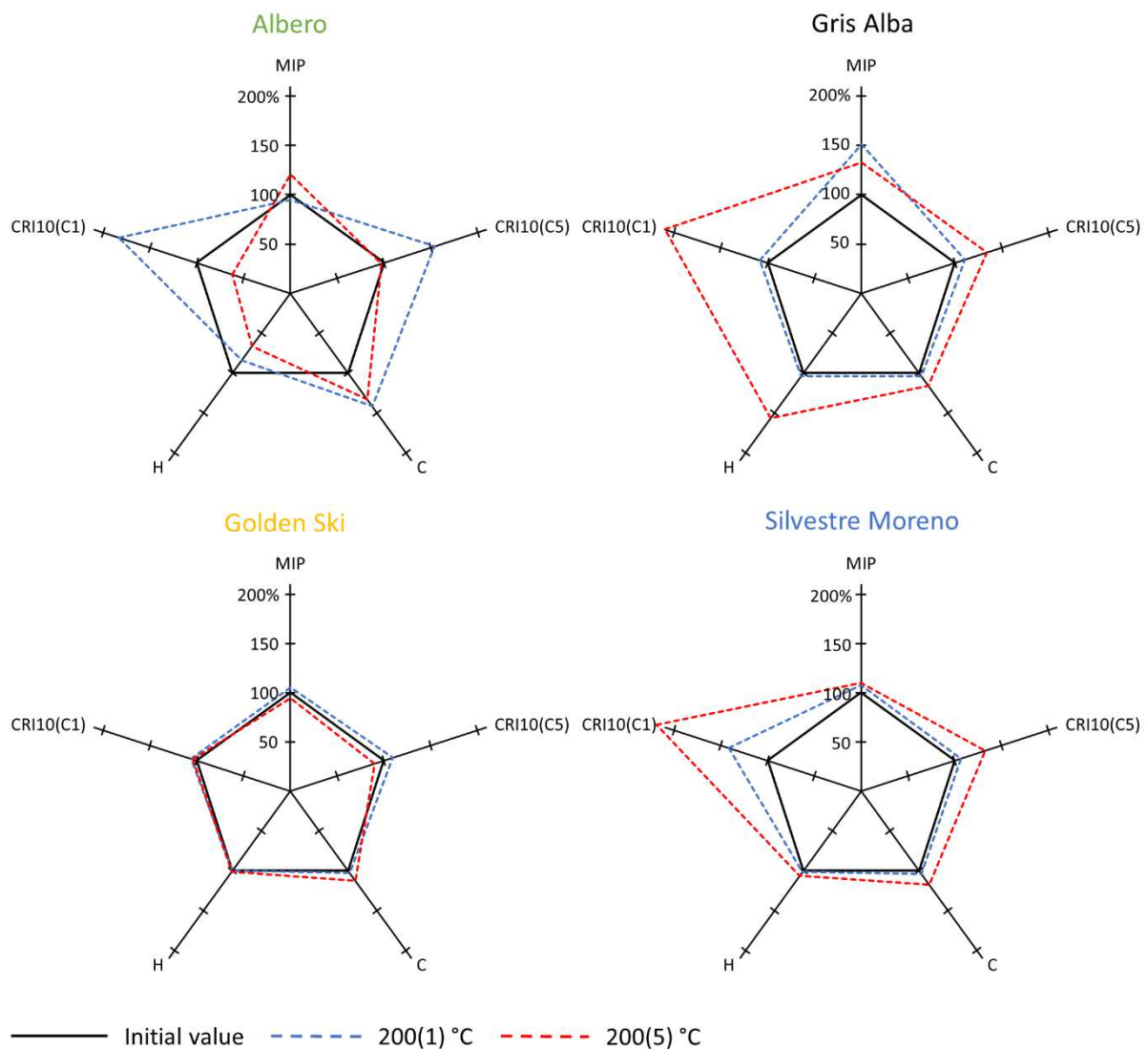
## 632 **EXP2: Effect of thermal fatigue at 200 °C**

633 According to the porosity value change (tab. 3), some rocks experimented a microcracking at  
634 200 °C observed under SEM.

635 Granites with lower initial porosity (GA and SM) showed the greatest microcracking increase  
636 (Fig. 13), in agreement with Simmons and Cooper (1978). The thermal fatigue in GA and SM  
637 leads to a progressive increase of microcracking measured by the different techniques. The  
638 damage caused by the first treatment at 200 °C confirmed a pore size augmentation beyond  
639 1  $\mu\text{m}$  (Fig. 12) that favoured a faster cooling (CRI). In GA, for the two tested samples, the  
640 coefficient C had a positive progression with temperature due to new connections, in

641 agreement with the increase in the height of the capillary fringe. SM showed good C  
 642 repeatability for both samples with also a positive progression. The rest of pore-related  
 643 properties showed an increase, which confirms an increase in microcracking with the  
 644 thermal cycles.

645 High porosity and high mica content allowed a mineral expansion and contraction without a  
 646 catastrophic failure of the rock. Thus, in the case of GS, that despite slight variations through  
 647 the fatigue cycles, it can be said that the initial and final state of these granites remained  
 648 similar (Fig. 13). As the number of cycles increased, the porous network of GS has  
 649 reorganised, avoiding major modifications. The small crystal size and the mica and clay  
 650 content favoured the mineral adjustment.



651  
 652 Figure 13: Diagram of the parameter evolution during EXP2: mercury intrusion porosimetry

653 (MIP), the cooling rate index (CRI10 (C1); CRI10 (C5)) to spot C1 and C5, the capillary  
654 coefficient (C) and the height of the capillary fringe detected with an infrared thermography  
655 camera (H). Except for MIP, the parameters were measured on the same samples.

656 A, showed the development of large microcracks that influence the CRI and caused a  
657 decrease in the height of the capillary fringe, while MIP remained constant. The capillary  
658 coefficient C increased as a result of the widening of the pre-existing intergranular  
659 microcracking observed with SEM. After the fatigue test, the bigger microcracks closed  
660 possibly by mineral expansion or/and clay remobilisation placed on the edge of pore walls,  
661 which did not, however, prevented the continuous circulation of water (Robert, 2004).

## 662 6\_CONCLUSION

663 The heterogeneity of low porosity granites induces more dispersed results, while the trends  
664 are more similar for high porosity ones. The creation of microcracks and the thermal  
665 expansion of minerals are two contradictory phenomena in the evolution of porosity, leading  
666 to heterogeneous thermal behaviour. Several parameters are involved in the microcracking  
667 evolution with temperature. Thus, for a target temperature, some techniques may indicate  
668 an increase in the microcracks while others do not vary or show the contrary. High porosity  
669 granites fluctuate between microcracking closure and aperture, with some strong variations  
670 that can be considered as a thermal threshold between 90-130 °C. Low porosity granites do  
671 not show a thermal threshold at temperatures under 130 °C and only a microcrack closure is  
672 measured. However, that is also influenced by the low Qz/F ration of this rock.

673 Thermal fatigue at a temperature of 200 °C shows the evolution of existent and/or  
674 generated microcracks. The lower porosity granites present an evident and progressive  
675 microcracking development from the very first heating cycle. Capillary coefficient increases  
676 with the repetition of the thermal cycles regardless of the affected pore family. The high  
677 porosity granites continue to show a redistribution of pre-existing microcracks through all  
678 the test and no signs of new microcracking.

679 Regarding the studied rocks, porosity shows a stronger impact on the thermal behaviour  
680 than the mineralogy. Nevertheless, for similar Qz/F ratios, the lower porosity granites reach  
681 the thermal microcracking threshold earlier. Quartz allows to interpret the closure of the

682 microcracking of granites with high porosity due to its higher thermal expansion. In addition,  
683 the low quartz content in the low porosity granite leads to a microcrack closing without  
684 microstress development, at low temperature. Mica accommodates the mineral expansion  
685 reducing microstresses and the consequent microcracking.

686 The IRT camera allows to calculate the cooling rate index. Water cooling is more important  
687 for granites with high initial porosity. The air cooling remains similar between the different  
688 granites. This parameter gives more confident results on the formation of greater  
689 microcracks. The image treatment provides a qualitative interpretation of the migration of  
690 the capillary fringe. The results show a good correlation with the capillary coefficient,  
691 validating this method. These preliminary experiments confirm the utility of the IRT camera  
692 in the monitoring of the thermal behaviour of the granites that will help in the field of  
693 geothermal energy and nuclear waste storage.

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