

**Anatectic melt inclusions in ultra-high temperature  
 granulites**

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1           **Title: Anatectic melt inclusions in ultra-high temperature granulites**

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## 22 **Abstract**

23 Partial melting up to ultra-high temperature (UHT) conditions is one of the major processes  
24 for the geochemical differentiation and reworking of the mid- to lower continental crust, with  
25 relevant implications on its rheological behaviour. UHT granulites from the Gruf Complex  
26 (European Central Alps) display garnet and sapphirine porphyroblasts containing a variety of  
27 primary melt inclusions. Typically, melt inclusions in garnets occur as glassy and  
28 polycrystalline inclusions (i.e. nanogranitoids), the latter often organized in mm-sized  
29 clusters associated with primary fluid inclusions. Nanogranitoids are characterized by an  
30 elliptical faceted shape, with variable sizes ranging from 2 to 115  $\mu\text{m}$ , while glassy inclusions  
31 show negative crystal shapes that usually never exceed 15  $\mu\text{m}$  in diameter and present  $\text{CO}_2$ -  
32 rich shrinkage bubbles. The characteristic mineral assemblage observed in nanogranitoids  
33 consists of quartz, biotite, muscovite, plagioclase, K-feldspar, kokchetavite and rarely  
34 aluminosilicates. Glassy and re-homogenized melt inclusions are peraluminous and rhyolitic  
35 in composition, with  $\text{SiO}_2 = 69\text{-}80 \text{ wt}\%$  and  $\text{Na}_2\text{O}+\text{K}_2\text{O} = 5\text{-}12 \text{ wt}\%$ . Commonly, the  
36 analysed melt inclusions have very high  $\text{K}_2\text{O} (>6 \text{ wt}\%)$  and very low  $\text{Na}_2\text{O} (<2 \text{ wt}\%)$   
37 contents, indicative for potassic to ultrapotassic melts. Measured  $\text{H}_2\text{O}$  contents of the melts  
38 range from 2.9 to 8.8 wt%, whereas  $\text{CO}_2$  concentrations are between 160 and 1738 ppm.  
39 Accordingly, calculated viscosities for re-homogenized melt inclusions vary between  $10^4$  and  
40  $10^5 \text{ Pa}\cdot\text{s}$ . Related primary fluid inclusions mainly contain  $\text{CO}_2$ , with rare occurrence of CO  
41 and  $\text{N}_2$ , and are often associated with quartz, as well as different carbonates and  
42 phyllosilicates. It is assumed that the source for the carbonic fluid was external and probably  
43 related to the degassing lithospheric mantle. Consequently, it is argued that anatexis was  
44 initially triggered by incongruent dehydration melting reactions involving biotite breakdown  
45 and proceeded in the presence of an externally derived COH-bearing fluid. The coexistence  
46 of COH-bearing fluid and melt inclusions indicates that partial melting occurred under

47 conditions of fluid-melt immiscibility. Potassic to ultrapotassic melt inclusions in UHT  
48 granulites suggests that lower crustal anatexis may play a significant role in the redistribution  
49 of heat-producing elements (such as  $K_2O$ ), potentially influencing the thermal structure of the  
50 continental crust.

51

52 **KEYWORDS:** Fluid inclusions; Garnet; Granites; Gruf Complex; Nanogranitoids

53

## 54 1. INTRODUCTION

55 Buoyant andesitic continental crust (e.g. Rudnick, 1995; Taylor & McLennan, 1985) is a  
56 distinctive feature that differentiates the Earth from all the terrestrial planets of the Solar  
57 System. Although the upper levels of continents can be easily studied and sampled, the  
58 structure and chemical composition of the lower continental crust remain mostly enigmatic,  
59 due to its inaccessibility for direct observations. Rock xenoliths entrained in lavas and  
60 tectonically exposed metamorphic terranes, characterized by mineral assemblages that record  
61 lower crustal pressures (i.e. 5-15 kbar), are the only opportunities to obtain useful  
62 petrological information on the deep crust. A large number of such exposed terrains (e.g. the  
63 Napier Complex in Antarctica, the Eastern Ghats granulite belt in India; see also Kelsey &  
64 Hand, 2015) has provided a strong evidence that the lower continental crust can attain  
65 extreme thermal regimes on a regional scale ( $>775^\circ\text{C}/\text{GPa}$ ; e.g. Harley, 2008; Kelsey & Hand  
66 2015; Brown & Johnson, 2018). Under these conditions, rocks are subjected to ultra-high  
67 temperature (UHT) metamorphism, in which peak temperatures exceed  $900^\circ\text{C}$  at typical  
68 pressures of 7-13 kbar (Harley, 1998), with important implications for the geochemical  
69 differentiation, reworking and rheology of the continental lower crust (Brown, Korhonen, &  
70 Siddoway, 2011; Jamieson, Unsworth, Harris, Rosenberg, & Schulmann, 2011). UHT  
71 metamorphism is typically preserved in Mg-Al-rich metapelitic granulites (Brandt, Will, &

72 Klemm, 2007; Harley, Hensen, & Sheraton, 1990; Harley, 2008; Kelsey, 2008; Santosh,  
73 Tsunogae, Li, & Liu, 2007), whose diagnostic paragenesis is characterized by the presence of  
74 sapphirine+quartz, Al-rich orthopyroxene+sillimanite and osumilite-bearing mineral  
75 assemblages (Harley, 1998; Kelsey, 2008). Progressing towards UHT conditions, the lower  
76 crust buffers the increasing temperature undergoing extensive partial melting, with the  
77 production of felsic magma that may then segregate from its source, leaving behind a more  
78 mafic and less fertile residuum (Clemens & Stevens 2016; Sawyer 1994; Sawyer, Cesare, &  
79 Brown, 2011; Vielzeuf & Holloway, 1988; White & Powell, 2002). Although S-type granites  
80 and granitic leucosomes are the two most prominent products of crustal anatexis, they do not  
81 provide the pristine chemical composition of the felsic anatectic melt because they are  
82 affected by fractional crystallization, crystal accumulation, chemical contamination and  
83 presence of xenocrysts (Carvalho, Sawyer, & Janasi 2016; Marchildon & Brown 2001;  
84 Milord, Sawyer, & Brown, 2001; Sawyer, 2008; Sawyer et al., 2011; Stevens, Villaros, &  
85 Moyen, 2007). Consequently, initial anatectic melt compositions have been mainly deduced  
86 from experimental studies (e.g. Clemens, Holloway, & White, 1986; Le Breton & Thompson,  
87 1988; Montel & Vielzeuf, 1997; Stevens, Clemens, & Droop, 1997) and thermodynamic  
88 calculations (Grant, 2009; Holland & Powell, 2001; White, Stevens, & Johnson, 2011).  
89 Another compelling method to determine the composition of crustal melts during the early  
90 stages of anatexis is represented by the study of primary melt inclusions entrapped in  
91 peritectic minerals during incongruent melting of metapelitic rocks (Acosta-Vigil, Cesare,  
92 London, & Morgan, 2007; Acosta-Vigil et al., 2016; Bartoli et al., 2013; Bartoli, Acosta-  
93 Vigil, Tajčmanová, Cesare, & Bodnar, 2016; Cesare, Marchesi, Hermann, & Gómez-  
94 Pugnaire, 2003; Cesare, Ferrero, Salvioli-Mariani, Pedron, & Cavallo, 2009; Ferrero et al.,  
95 2012; Ferrero, Wunder, Walczak, O'Brien, & Ziemann, 2015; Ferrero et al. 2019). Primary  
96 melt inclusions (MI) in high-grade metamorphic rocks typically show variable degrees of

97 crystallization, ranging from glassy to fully crystallized, the latter also referred as  
98 *nanogranitoids* (Cesare, Acosta-Vigil, Bartoli, & Ferrero, 2015). Since MI represent droplets  
99 of an early crustal melt, they preserve precious information on its primordial composition,  
100 before the melt is subjected to any chemical change due to magmatic differentiation.  
101 At present, only few studies have approached a detailed investigation of primary melt and  
102 fluid inclusions at, or near, UHT conditions (e.g. in the Kerala Khondalite Belt, Cesare et al.,  
103 2009 and Ferrero et al., 2012, in the Athabasca granulite terrane, Tacchetto et al., 2019 or in  
104 the Ivrea-Verbano Zone, Carvalho et al. 2019) and therefore the pristine composition of both  
105 anatectic melts and fluids in UHT rocks is largely unconstrained. In order to overcome this  
106 paucity, we examined primary melt and fluid inclusions in peritectic minerals observed in  
107 UHT granulites of the Gruf Complex (Central Alps). The aim of this study is to provide a  
108 microstructural framework and geochemical characterization of anatectic melts and fluids and  
109 evaluate their role during crustal reworking processes associated to HT-UHT metamorphism.

110

## 111 **2. GEOLOGICAL SETTING**

112 The Penninic units of the Alpine nappe stack in the Central Alps are characterized by mineral  
113 isograds, isotherms and isobars that have a concentric shape, defining the so-called  
114 “Leontine Metamorphic Dome” (Todd & Engi, 1997; Wenk, 1955). The Gruf Complex is a  
115 ~12 x 10 km migmatitic body across Switzerland and northern Italy, located in the south-  
116 eastern part of the Lepontine Dome (see Figure 1). To the west the Gruf Complex is  
117 delimited by the Adula Nappe, whereas to the north is surrounded by the mafic-ultramafic  
118 bodies of the Chiavenna Unit and by the crystalline rocks of the Tambo Nappe. To its eastern  
119 and southern margin, the Gruf Complex is intruded by the Bergell pluton, a tonalitic to  
120 granodioritic calc-alkaline intrusive body that formed between 33 and 30 Ma (Gianola et al.,  
121 2014; Oberli, Meier, Berger, Rosenberg, & Gieré, 2004; Samperton et al., 2015; von

122 Blanckenburg, 1992). The southern extremity of the Gruf Complex, and part of the Bergell  
123 intrusion, are then cut by the 24 Ma old Novate granite (Liati, Gebauer, & Fanning, 2000), a  
124 strongly peraluminous S-type granitic stock composed by a swarm of dykes that presumably  
125 derived from the partial melting of the surrounding gneissic units (von Blanckenburg, Früh-  
126 Green, Diethelm, & Stille, 1992). The dominant lithologies of the Gruf Complex are  
127 migmatitic orthogneisses, paragneisses and micaschists (Galli et al. 2011), which underwent  
128 upper amphibolite facies migmatization (Bucher-Nurminen & Droop 1983; Nagel, De  
129 Capitani, & Frey, 2002) between 34 and 29 Ma (Galli et al. 2012). These rocks are also  
130 associated with irregular tabular bodies of charnockites and leucogranites (Galli et al. 2011).  
131 Sapphirine-bearing granulites occur as schlieren in charnockites and as residual enclaves in  
132 both migmatitic orthogneisses and charnockites (Galli et al. 2011). According to Galli et al.  
133 (2011), granulites and charnockites formed at ultra-high temperature conditions of  $T = 920-$   
134  $940^{\circ}\text{C}$  and  $P = 8.5-9.5$  kbar, while migmatization of the surrounding gneissic and metapelitic  
135 lithologies took place at  $\sim 740^{\circ}\text{C}$  and  $6.5-7.5$  kbar (Galli, Le Bayon, Schmidt, Burg, &  
136 Reusser, 2013).

137

### 138 **3. ANALYTICAL TECHNIQUES AND EXPERIMENTAL METHODS**

139

140 Back-scattered electron (BSE) images and semi-quantitative energy dispersive spectroscopy  
141 (EDS) analyses of MI were carried out with a CamScan MX2500 Scanning Electron  
142 Microscope (SEM), at the Department of Geosciences of the University of Padova (Italy).  
143 Micro-Raman spectroscopy of MI was conducted with a HORIBA Jobin-Yvon LabRAM HR  
144 800, equipped with a Nd:YAG laser ( $\lambda = 532$  nm, laser power on sample  $\sim 2-3$  mW), at the  
145 Institute of Earth and Environmental Science, University of Potsdam. Raman spectra were  
146 acquired between  $100$  and  $4000$   $\text{cm}^{-1}$  with an acquisition time of 20 s during three spectral

147 accumulations. The typical setup also included a grating of 300 lines/mm, a slit width of 100  
148  $\mu\text{m}$  and a confocal hole set at 200. Spectral resolution is considered to be of  $\sim 10\text{ cm}^{-1}$ ,  
149 compared to available literature and databases.

150 The chemical composition of garnet was measured with a CAMECA SX50 electron  
151 microprobe (EPMA), equipped with four wavelength dispersive spectrometers (WDS), at the  
152 C.N.R.-I.G.G. (Consiglio Nazionale delle Ricerche – Istituto di Geoscienze e Georisorse),  
153 Department of Geosciences, University of Padova. Analyses were carried out using a  
154 focussed beam with a current of 20 nA and an accelerating voltage of 15 kV. Counting times  
155 for each element were 10 s on peak and 5 s for the background.

156 Major element composition of glass and minerals in the re-homogenized and glassy MI were  
157 determined with a JEOL JXA-8200 Superprobe, equipped with five WDS, at the Department  
158 of Earth Sciences (University of Milano, Italy), employing an acceleration voltage of 15 kV,  
159 a beam current of 5 nA and beam diameter of 1  $\mu\text{m}$ . Acquisition times were 10 s on peak and  
160 5 s for background, measuring Na and K first to avoid diffusional losses. Alkali  
161 concentrations were corrected using conservative factors obtained by the analysis of hydrous  
162 and anhydrous leucogranitic glasses of known composition. In particular, the standards  
163 employed in this study were a 10.1 wt%  $\text{H}_2\text{O}$ -bearing glass (LGB 2; Behrens & Jantos,  
164 2001), a 5.5 wt%  $\text{H}_2\text{O}$ -bearing glass (DL; Acosta-Vigil, London, Morgan, & Dewers, 2003)  
165 and a nearly anhydrous ( $\text{H}_2\text{O} = 300 \pm 42\text{ ppm}$ ) glass (B; Morgan & London, 2005).

166 Single garnet core and rim fragments ( $\sim 1.5 \times 2\text{ mm}$  in size), used for the re-homogenization  
167 experiments of the nanogranitoids, were manually separated by cutting from 1.5 mm-thick  
168 double-polished thick sections. Two sets of experiments were performed. In the first set,  
169 garnet chips were charged into Au capsules (external and internal diameter of 3 mm and 2.7  
170 mm, respectively) alternating them with powdered graphite (granulometry  $< 45\text{ }\mu\text{m}$ ). In the  
171 second type of experiments, the same gold capsules were filled with alternating layers of



172 garnet chips and powdered SiO<sub>2</sub>, obtained from tetraethylorthosilicate (TEOS). In both cases,  
173 capsules were crimped and sealed with an arc-welder. Experiments with garnet+graphite  
174 charges were conducted using a single-stage and an end-loaded piston cylinder apparatuses at  
175 the Laboratory of Experimental Petrology, Department of Earth Sciences, University of  
176 Milano. Capsules were placed vertically into MgO spacers and then inserted in a NaCl  
177 sleeve-pyrex (only for end-loaded piston cylinder)-graphite piston cylinder assembly. The  
178 temperature was monitored by a K-type (nickel-chromium / nickel-alumel) thermocouple,  
179 with an estimated accuracy of  $\pm 5^{\circ}\text{C}$  and no corrections for the pressure effects on the  
180 electromotive force were applied. Experiments were performed at 10-12 kbar (i.e. higher than  
181 the calculated peak conditions, in order to prevent decrepitation of the inclusions) between  
182 950°C and 800°C and with variable run durations (from 1 to 10 hours). This allowed to  
183 constrain the interval in which re-homogenization was best attained. All runs were first  
184 pressurized to 10-12 kbar at room temperature and then the assembly was isobarically heated  
185 at a constant heating rate (100 °C/min) until the targeted temperature was achieved. The  
186 experiments were terminated by switching off the power supply, resulting in quenching rates  
187 of  $\sim 40^{\circ}\text{C/s}$ . Capsules were then dissolved in aqua regia and the recovered garnets were  
188 singularly mounted in epoxy resin. Experiments with garnets+quartz charges were performed  
189 at the Institute of Geochemistry and Petrology, ETH Zurich, using a single stage and an end-  
190 loaded piston cylinder apparatuses. The assembly consisted of a talc sleeve, pyrex (only for  
191 end-loaded piston cylinder), a graphite furnace and MgO spacers, in which the Au capsules  
192 were positioned. Temperature was measured by a B-type (Pt<sub>94</sub>Rh<sub>6</sub>-Pt<sub>70</sub>Rh<sub>30</sub>) thermocouple  
193 (accuracy:  $\pm 5^{\circ}\text{C}$ ). Experiments were performed at 12 kbar, 875°C and with a run duration of  
194 10 h. The experimental procedure was the same used for the experiments with  
195 garnets+graphite charges. Although the two set of experiments were performed in slightly

196 different assemblies, it is assumed that this has no relevant influence on the re-  
197 homogenization of the inclusions.

198 H<sub>2</sub>O and CO<sub>2</sub> contents of remelted inclusions were determined by Nano Secondary Ion Mass  
199 Spectrometry (NanoSIMS) employing a Cameca NanoSIMS 50 at Muséum National  
200 d'Histoire Naturelle of Paris, following the approaches of Bartoli, Cesare, Remusat, Acosta-  
201 Vigil, and Poli (2014) and Créon, Levresse, Remusat, Bureau, and Carrasco-Núñez (2018).

202 Polished experimental capsules with quartz-garnet charges and standard glasses were  
203 mounted in indium to lower the hydrogen background in the analysis chamber (Aubaud et al.,  
204 2007), and coated with gold (20 nm thick). Before each analysis, a 5 x 5 µm surface area was  
205 initially pre-sputtered for 150 s with a 170 pA primary Cs<sup>+</sup> rastering beam, in order to remove  
206 the gold coating and reach a sputtering steady-state (Thomen, Robert, & Remusat, 2014).

207 Subsequently, the primary Cs<sup>+</sup> beam was set at 17 pA and scanned over a surface area of 3 x  
208 3 µm. Nevertheless, to avoid surface contamination, only ions from the inner 1 x 1 µm region  
209 were collected with the “beam blanking mode”. Exposed MI were analysed through  
210 collection of secondary ions of <sup>12</sup>C<sup>-</sup>, <sup>16</sup>OH<sup>-</sup>, <sup>28</sup>Si<sup>-</sup> and <sup>56</sup>Fe<sup>16</sup>O<sup>-</sup> in multicollecion mode. Mass  
211 resolving power was set at minimum 5500, enough to resolve interferences on <sup>16</sup>OH<sup>-</sup>. Each  
212 analysis consisted in a stack of 200 cycles, with a duration of 1.024 s for every single cycle.

213 The vacuum in the analysis chamber never exceeded 2.5 x 10<sup>-10</sup> Torr during the entire session  
214 of measurements. H<sub>2</sub>O and CO<sub>2</sub> contents of the glasses were quantified measuring <sup>16</sup>OH<sup>-</sup>/<sup>28</sup>Si<sup>-</sup>  
215 and <sup>12</sup>C<sup>-</sup>/<sup>28</sup>Si<sup>-</sup> ratios, respectively (Bartoli et al., 2014; Créon et al., 2018). These ratios were  
216 converted into concentrations using calibration curves obtained from standards of known  
217 composition. Standards for the determination of H<sub>2</sub>O included two standards used for the  
218 alkali correction (DL and B, see above) plus an additional hydrous glass with 4.3 wt% H<sub>2</sub>O  
219 (LGB1; Behrens & Jantos, 2001). On the other hand, standards for CO<sub>2</sub> consisted in four  
220 trachyandesites (STR 9, 10, 11 and 13) from the Stromboli volcano, experimentally doped in

221 carbon and water by Bureau et al. (2003). Concentrations and uncertainties were calculated  
222 using the R program, following the procedure described in Bartoli et al. (2014) and Thomen  
223 et al. (2014). Average major element and volatile concentrations of the analysed MI are  
224 reported in Table 1.

225

## 226 **4. RESULTS**

227

### 228 **4.1 Mineral assemblages of the UHT granulites**

229 All the investigated samples were collected in the lower part of Val Codera, near the locality  
230 of Bresciadega (see also Galli et al., 2011). Two varieties of UHT granulite with granofelsic  
231 texture were studied (Figure 2). The first type corresponds to type A of Galli et al. (2011) and  
232 consists of a dark, massive granulite containing up to 2 cm large garnet (Grt) and up to 5 mm  
233 sapphirine (Spr) porphyroblasts, associated with a matrix of up to 3 mm-sized orthopyroxene  
234 (Opx), cordierite (Crd), biotite (Bt) and K-feldspar (Kfs). Garnet appears as faint pink,  
235 rounded crystals showing a clear zonation. Numerous mineral inclusions of biotite,  
236 orthopyroxene, sapphirine, plagioclase (Pl), cordierite, rutile (Rt), ilmenite, sillimanite,  
237 tourmaline, apatite (Ap), spinel, staurolite, corundum, monazite and zircon are present within  
238 the porphyroblastic garnets. On the other hand, sapphirine is generally hypidiomorphic,  
239 prismatic and pleochroic (from pale-red to blue), showing sometimes a longitudinal, simple  
240 twinning system.

241 The second type of granulite belongs to type C of Galli et al. (2011). The rock is massive and  
242 melanocratic with a mineral assemblage made of up to 1.5 cm large porphyroblastic garnet,  
243 porphyroblastic orthopyroxene (<1.5 mm), biotite, cordierite, sillimanite and symplectitic  
244 spinel. Sapphirine may be present as symplectitic grains in the rock matrix, or as small (<500  
245  $\mu\text{m}$ ) inclusions within garnet. Other mineral inclusions observed within garnet grains are:

246 biotite, quartz, sillimanite, cordierite, plagioclase, spinel, zircon, apatite, corundum, rutile,  
247 staurolite and tourmaline.

248 In both granulites, melt inclusions (MI) are mainly present in the porphyroblastic garnets,  
249 where they may occur as single, scattered inclusions or may form mm-large clusters. These  
250 clusters are commonly found in the cores of the garnets (Figure 3a,b), but their occurrence  
251 towards to the rims has been also noticed (Figure 3c,d). The presence of different birefringent  
252 crystals under crossed polars (Figure 3e,f) indicates that the majority of MI are  
253 polycrystalline (i.e. nanogranitoids; Cesare et al. 2015). Often, fluid inclusions occur  
254 associated to nanogranitoids within the same clusters. Typically, fluid inclusions show a  
255 distinctive darker appearance and high birefringence (Figure 3g). Moreover, sporadic glassy  
256 inclusions containing shrinkage bubbles have been also observed (Figure 3h). In contrast to  
257 polycrystalline MI, glassy MI occur exclusively as single inclusions dispersed in the garnets,  
258 without forming clusters. Occasionally, glassy MI may occur also close to nanogranitoids. MI  
259 with multiple birefringent phases have been also identified in sapphirine porphyroblasts of  
260 Type A granulites (Figure 3i). The MI in sapphirine are too rare to allow their experimental  
261 re-melting, but they were characterized by Raman spectroscopy.

262

#### 263 **4.2 Micro-Raman characterization of glassy, nanogranitoid and fluid inclusions**

264 Micro-Raman investigations for the glassy inclusions observed in Type A samples show that  
265 the glass is hydrous (see Figure 4a). Furthermore, the single Raman band displayed at 1382  
266  $\text{cm}^{-1}$  (Figure 4a) indicates that some dissolved molecular  $\text{CO}_2$  is also present in the melt  
267 (Brooker, Kohn, Holloway, McMillan & Carroll, 1999; Ni & Keppler 2013). Shrinkage  
268 bubbles in the glass contain  $\text{CO}_2$ , as demonstrated by the presence of the “Fermi-doublet” in  
269 the Raman spectra (see Figure 4a). Densities of  $\text{CO}_2$  in these bubbles (see Table S1),  
270 calculated with the densimeter of Wang et al. (2011), range between 0.5 and 0.8  $\text{g/cm}^3$  ( $\pm 0.3$

271 g/cm<sup>3</sup>). Raman analyses showed that nanogranitoids (Figure 4b,c) contain quartz, muscovite,  
272 phlogopite, K-feldspar, kokchetavite (a polymorph of KAlSi<sub>3</sub>O<sub>8</sub>) and an unknown phase with  
273 a main Raman peak at 430 cm<sup>-1</sup>. A phase with identical spectrum has been identified in  
274 nanogranitoids from other case studies and it is currently under investigation (Ferrero,  
275 unpublished data). Unexpectedly, Raman investigations also showed that some of these  
276 nanogranitoids could retain some CO<sub>2</sub> bubbles (Figure 4b,c), suggesting the existence of  
277 micropores. Nanogranitoids observed in sapphirine crystals from type A granulites (Figure  
278 4d) contain quartz, muscovite, kokchetavite and the same unknown phase with the peak at  
279 430 cm<sup>-1</sup>.

280 Fluid inclusions (FI), present in both granulites, display a fluid fraction as well as a solid  
281 portion, in which several phases were identified. In type A granulites muscovite, Fe-  
282 magnesite, calcite, pyrophyllite, quartz and biotite were the most common phases, besides  
283 CO<sub>2</sub>. FI in type C granulites, in addition to the same phases observed for type A, have  
284 paragonite, magnesite, siderite, phlogopite, N<sub>2</sub> and CO (Figure 4e,f). For one inclusion it was  
285 possible to calculate the relative amount of components in the fluid, applying the method of  
286 Dubessy, Poty, and Ramboz (1989). The obtained molar fractions were 94 mol% for CO<sub>2</sub> and  
287 6 mol% for N<sub>2</sub>. Regarding the carbonate phases, Fe-magnesite in FI of both granulites has  
288 variable Fe content ( $Fe\# = 100 \times Fe/(Fe+Mg+Mn+Ca+Al)$ ), as defined by Boulard, Guyot, &  
289 Fiquet, 2012), typically ranging between  $Fe\# = 20$  and  $Fe\# = 55$ . Calculated CO<sub>2</sub> densities  
290 (Wang et al. 2011) for FI in type A granulites vary between 0.3 and 1.0 g/cm<sup>3</sup> (see Table S1),  
291 a range comparable to that calculated for type C granulites (0.4-0.8 g/cm<sup>3</sup>). Remarkably,  
292 neither in the shrinkage bubbles of glassy inclusions nor in fluid inclusions, H<sub>2</sub>O was  
293 detected.

294

295

### 296        **4.3 Microstructures of nanogranitoids**

297 Detailed observations by SEM allowed to determine the principal microstructural features of  
298 nanogranitoids, as well as their mineral assemblage. Typically, MI of both Type A and C  
299 granulites have an elliptical faceted shape, with major axes ranging between 2 and 115  $\mu\text{m}$   
300 (Figure 5a-f). Nevertheless, for Type A, the size of MI decreases from rims to cores. MI in  
301 the rims have an average size of  $47 \pm 5 \mu\text{m}$ , those found in the intermediate region of the  
302 garnets (i.e. between cores and rims) are  $35 \pm 5 \mu\text{m}$ , while MI found in the cores have an  
303 average size of  $24 \pm 3 \mu\text{m}$ . Together with nanogranitoids with a nearly negative crystal shape  
304 (Figure 5a,b), some MI display natural decrepitation, with offshoots that are arranged radially  
305 around the inclusions (Figure 5c,d). The number of offshoots per inclusion varies drastically  
306 (from 1 to 10), with larger inclusions showing more and usually longer offshoots. The  
307 calculated average length of the offshoots for MI  $<45 \mu\text{m}$  in size is  $6 \mu\text{m}$ , whereas MI bigger  
308 than  $45 \mu\text{m}$  have an average length of the offshoots of  $13 \mu\text{m}$ . Glassy inclusions in Type A  
309 granulites show negative crystal shapes and their size is  $<15 \mu\text{m}$ , with an average diameter of  
310  $8 \mu\text{m}$ . MI in type C granulites also have an elliptical faceted shape and show a polycrystalline  
311 nature. However, in contrast to MI in type A granulites, they have similar sizes from core to  
312 rim and hardly exceed  $40 \mu\text{m}$  in diameter, with an average length of the major axis of  $21 \pm 2$   
313  $\mu\text{m}$ . Also garnets from type C granulites show some decrepitated MI (Figure 5e,f), which  
314 have an average length of the offshoots of  $5 \mu\text{m}$ .

315 In general, the mineral assemblage within nanogranitoids has an inequigranular and  
316 interlobate fabric composed of mainly hypidiomorphic crystals of quartz, biotite, muscovite,  
317 plagioclase, K-feldspar and very rarely aluminosilicates (Figure 5a-f). Sometimes  
318 nanogranitoids clearly display an igneous microstructure defined by intergrowths of quartz,  
319 feldspars and biotite (Figure 5e-f). SEM analyses also highlighted the presence of accessory

320 minerals, typically Cl-bearing apatite and rutile, which are usually more idiomorphic  
 321 compared to the other phases (Figure 5d,e).

322

#### 323 **4.4 Chemical composition of host garnets and minerals in nanogranitoids**

324 In this section we report only chemical analyses of garnet porphyroblasts. For detailed  
 325 chemical analyses of all rock-forming minerals see Galli et al. (2011). Analysed garnets of  
 326 Type A and Type C granulites are almost pure almandine-pyrope solid solutions (see Table  
 327 S2 and Figure S1) and display a zonation marked by pyrope-richer rims (Type A = Alm<sub>40-</sub>  
 328 <sub>52</sub>Prp<sub>43-55</sub>Sps<sub>0-2</sub>Grs<sub>2-6</sub>; Type C = Alm<sub>46-54</sub>Prp<sub>43-52</sub>Sps<sub>0-1</sub>Grs<sub>1-3</sub>) compared to cores (Type A =  
 329 Alm<sub>48-61</sub>Prp<sub>26-46</sub>Sps<sub>1-4</sub>Grs<sub>3-12</sub>; Type C = Alm<sub>53-62</sub>Prp<sub>32-43</sub>Sps<sub>0-1</sub>Grs<sub>3-6</sub>). Nevertheless, Type C  
 330 granulites may also have garnets with cores enriched in pyrope-component (Alm<sub>41-48</sub>Prp<sub>48-</sub>  
 331 <sub>57</sub>Sps<sub>0-1</sub>Grs<sub>2-4</sub>) compared to rims (Alm<sub>42-53</sub>Prp<sub>43-54</sub>Sps<sub>0-1</sub>Grs<sub>2-4</sub>).

332 Nanogranitoids are often too small to allow a precise chemical characterization of their  
 333 mineral assemblage. Nevertheless, some nanogranitoid inclusions in Type A granulites were  
 334 large enough and their single mineral phases were measured (see Table S3). Plagioclases  
 335 show variable  $X_{An}$  [ $An/(An+Ab)$ ] between 0.10 (albite) and 0.49 (andesine). By contrast,  
 336 biotites are Ti-free and characterized by  $X_{Mg}$  [ $Mg/(Mg+Fe^{2+})$ ] ranging from 0.71 to 0.86.  
 337 Other K-rich phases that have been recognized are K-feldspar and white mica, the latter  
 338 displaying a  $X_{Mg} = 0.59$ .

339

#### 340 **4.5 Remelted nanogranitoids**

341 Experiments between 900 and 875°C produced the most homogenized glasses. Remelted MI  
 342 are typically <15 µm in size and display elongated to perfect negative crystal shapes (Figure  
 343 6a-d). Some inclusions, together with glass, display mineral phases such as rutile and apatite  
 344 that indent the walls of the MI (Figure 6c). These phases are interpreted to be accidental

345 minerals trapped during the formation of the inclusions, since they are observed before and  
346 after the re-homogenization experiments. Therefore, they are not daughter minerals (i.e.  
347 phases that crystallized directly from the melt; e.g. Ferrero et al., 2012; Acosta-Vigil et al.  
348 2016). Moreover, few MI may also exhibit bubbles (Figure 6d), suggesting that the fluid  
349 underwent incomplete dissolution in the melt during the experimental run or, alternatively, it  
350 diffused during cooling to form shrinkage bubbles (Lowenstern, 1995). A little fraction of the  
351 analysed MI population is characterized by the presence of unmelted daughter minerals,  
352 testifying incomplete remelting (Figure 6e,f). Typical daughter minerals coexisting with glass  
353 are euhedral to subhedral biotite grains ( $X_{Mg} = 0.82-0.88$  and  $TiO_2 < 0.2$  wt%, see Table S4)  
354 and rounded quartz crystals.

355 Experiments with garnets+quartz charges at 875°C produced substantial amounts of melt in  
356 the matrix outside the garnets (Figure 6g). This melt infiltrated within the garnet crystals and  
357 led to the formation of large melt pockets. The latter ones differ microstructurally from  
358 remelted MI by their size (commonly  $>20$   $\mu m$  in diameter, although few of them have sizes  
359 comparable to remelted MI) and by the striking abundance of bubbles, a feature that indicates  
360 high fluid contents (Figure 6h). The matrix melt derives from the melting of mineral  
361 intergrowths or matrix phases, such as biotite and feldspar, which could not be avoided  
362 during the manual crushing of the garnet chips. Often, a peritectic reaction between the melt  
363 and the garnet produced orthopyroxene or corundum (Figure 6g).

364 Evidence of overheating in the MI during experiments has been observed at all temperatures,  
365 but predominantly at 950°C. This is indicated by the interaction between melt and garnet,  
366 with the formation of peritectic phases such as orthopyroxene (Figure 6i) or gedrite, the  
367 recrystallization of new garnet within the inclusion or changes in the chemical composition of  
368 the garnet around the remelted inclusions. Chemical analyses of peritectic orthopyroxenes  
369 (see Table S4) indicate that they are Al-rich (8.3-10.4 wt% at 950°C and 6.5-9.2 wt%  $Al_2O_3$



370 at lower temperature), with  $X_{Mg} = 0.69-0.73$  (at 950°C) and 0.67-0.84 (at 900-875°C). These  
371 compositions are comparable to those measured in the orthopyroxene porphyroblasts in the  
372 matrix of the granulites (Galli et al. 2011). One analysis of a recrystallized garnet at 950°C  
373 shows that the new garnet is Fe-poorer and Mg-richer ( $X_{Mg} = 0.64$ ) compared to the original  
374 garnet composition ( $X_{Mg} = 0.47$ ). Since at 950°C microstructural evidence for overheating is  
375 dominant, the glasses analysed at these conditions were considered not representative and  
376 therefore disregarded (Figure S2). In the experiments performed at 800°C, MI often display  
377 incomplete remelting and only rare homogeneous glasses could be found. This might indicate  
378 that this temperature corresponds to the lower boundary for remelting experiments.

379

#### 380 **4.6 Chemical composition of glasses**

381 Figure 7a-b and Table S5 report chemical analyses of glasses for remelted inclusions and for  
382 preserved glassy inclusions, all recalculated on anhydrous basis. For what concerns  
383 experimentally remelted MI, analyses that displayed clear evidence for  
384 contamination/interaction with the host garnet, or mixing with matrix melt, were discarded  
385 and are not further discussed (see Figure S2). Glasses are dominantly peraluminous ( $ASI =$   
386  $1.0-1.6$ ,  $[Al_2O_3/(CaO+Na_2O+K_2O)]_{molar}$ ), with only one MI that falls into the peralkaline  
387 field. Natural glassy inclusions have a slightly more restricted range ( $ASI = 1.0-1.1$ )  
388 compared to re-homogenized nanogranitoids. With some exceptions, analysed MI are  
389 corundum-normative ( $Crn = 0.20-4.88$ , see Table S5). Both re-homogenized MI and glassy  
390 inclusions are predominantly rhyolitic in composition, with  $SiO_2 = 68.6-79.7$  wt% and  
391  $Na_2O+K_2O = 5.3-12.0$  wt% (on anhydrous basis), developing a negative correlated trend in  
392 the  $SiO_2$  vs.  $Na_2O+K_2O$  space (Figure 7b). However, melts with a trachytic composition have  
393 been also observed, as shown by one MI from Type A granulite remelted at 875°C. Glassy  
394 inclusions have slightly higher total alkali contents compared to remelted nanogranitoids.

395 Plotted in Harker variation diagrams (Figure 8 a-f), glassy and re-homogenized inclusions  
396 show a clear increase in  $\text{Al}_2\text{O}_3$  and  $\text{K}_2\text{O}$  with decreasing  $\text{SiO}_2$ . By contrast,  $\text{CaO}$ ,  
397  $\text{FeO}+\text{MgO}+\text{TiO}_2$ ,  $\text{Na}_2\text{O}$  and  $\text{Cl}$  display more scattered distributions. Glassy inclusions differ  
398 from re-homogenized nanogranitoids by their low  $\text{CaO}$  (<0.2 wt% vs. 0.2-2.0 wt%) and  
399  $\text{FeO}+\text{MgO}+\text{TiO}_2$  (<1.4 wt% vs. 1.4-3.4 wt%) concentrations. All the analysed melt  
400 inclusions are characterized by a decoupling in the alkali content, with most inclusions  
401 showing  $\text{Na}_2\text{O}$  concentrations <2 wt% and  $\text{K}_2\text{O}$  >6 wt%. Re-homogenized MI have variable  
402 K# ratios [ $\text{K\#} = \text{molar K}_2\text{O}/(\text{K}_2\text{O}+\text{Na}_2\text{O}) \times 100$ ], ranging from 72 to 99 (average:  $87 \pm 4$ ) for  
403 Type C granulites and from 56 to 82 (average:  $71 \pm 8$ ) for Type A granulites. Similarly,  
404 glassy inclusions from Type A granulites have K# between 59 and 87 (average:  $77 \pm 5$ ).  
405  $\text{H}_2\text{O}$  and  $\text{CO}_2$  concentrations of remelted inclusions, determined by NanoSIMS, are reported  
406 in Table 2 and shown in Figure 9.  $\text{H}_2\text{O}$  contents in the analysed MI range from 2.9 to 8.8  
407 wt%, without any clear distinction between garnet cores and rims. Indeed, calculated  
408 averages are  $6.2 \pm 1.3$  wt% (type A) and  $7.1 \pm 3.0$  wt% (type C) for garnet cores, whereas for  
409 garnet rims is  $4.4 \pm 0.9$  (type C). By contrast,  $\text{CO}_2$  concentrations allow to subdivide the  
410 analysed MI into two distinct families, one clustering at ~1200 ppm and the other showing  
411 concentrations <500 ppm. From Figure 9 it is also possible to notice that MI with low  $\text{CO}_2$   
412 concentrations are characterized by a narrower range in  $\text{H}_2\text{O}$  (4.1-5.5 wt%) compared to MI  
413 with higher  $\text{CO}_2$  contents.

414

## 415 **5 DISCUSSION**

### 416 **5.1 Anatexis close to UHT conditions**

417 The mineral assemblage displayed by nanogranitoid inclusions within the peritectic garnet of  
418 the studied granulites indicates that these inclusions represent droplets of pristine felsic melt,  
419 an observation that was also confirmed by the measurement of their chemical composition

420 after remelting experiments. As suggested by Galli et al. (2011) garnet porphyroblasts are  
421 peritectic minerals generated by fluid-absent biotite melting along the prograde heating stage.  
422 Further investigations of Guevara and Caddick (2016) and Oalman, Duesterhoeft, Möller,  
423 and Bousquet (2019) by means of phase equilibria modelling, showed that garnet in the Gruf  
424 granulites grows along the up-temperature path and then starts to be consumed at ~920-950  
425 °C. It follows that MI experimentally re-homogenized at 875-900 °C were trapped while  
426 garnet was growing along the prograde heating path. Therefore, the investigated MI allow an  
427 appraisal of crustal melts and anatexis near to UHT conditions.

428 Granulites from the Gruf Complex are characterized by the presence of glassy and  
429 nanogranitoid inclusions. Their coexistence at the micrometric scale indicates that the melt  
430 behaves differently when it is trapped within pores. Glassy inclusions are on average smaller  
431 than nanogranitoids and therefore their preservation may be explained by inhibition of  
432 crystallization (Cesare et al., 2009) due to the greater supersaturation required by smaller  
433 pores to develop crystals compared to larger ones (Holness & Sawyer, 2008). Alternatively,  
434 lower H<sub>2</sub>O contents and, in turn, higher viscosities in some inclusions could have inhibited  
435 the nucleation with the formation of glass (Bartoli, Acosta-Vigil, & Cesare, 2015). A  
436 different volatile content in glassy and crystallized melt inclusions seems to be supported by  
437 the different electron microprobe totals (Table 1). MI display compositional trends in SiO<sub>2</sub>  
438 and total alkali that may be ascribed to local diffusion in the melt (Acosta-Vigil et al., 2017)  
439 or due to lack of melt interconnection (Bartoli et al., 2019). In general, the chemical  
440 composition of re-homogenized MI is consistent with the mineral assemblage observed in the  
441 nanogranitoids. This can be also observed in the AKF diagram (Figure 10), where analysed  
442 glasses for Type A granulites plot within the triangle defined by the tie-lines of the minerals  
443 measured in the non-remelted nanogranitoids. Although the chemical composition of the  
444 minerals forming MI in Type C granulites could not be measured (Figure 10b), it is assumed

445 that the mineral assemblage should be similar to the one noticed for Type A granulites and  
446 therefore an equivalent triangle for MI in Type C granulites is expected. From Figure 10a it is  
447 also possible to observe that glassy inclusions plot towards the K-apex of the triangle, while  
448 remelted MI tend to be more scattered, with some analyses plotting slightly outside the  
449 triangle defined by the mineral assemblage. In the Qz-Ab-Or normative diagram (see Figure  
450 S3) glassy and re-homogenized inclusions from the Gruf Complex plot close to the Qz-Or  
451 join, considerably away from the haplogranite ternary eutectic. Furthermore, MI also plot at  
452 lower Ab-contents compared to charnockites, leucogranites and S- and A-type granites, but  
453 overlap the field displayed by MI from the Kerala Khondalite Belt.

454 Figure 8 shows that the analysed glasses have markedly different compositions compared to  
455 the charnockites of the Gruf Complex (see Galli et al., 2013), indicating that the charnockites  
456 might have been generated from another metapelitic source or during a melting event  
457 unrelated with the formation of the granulites. Alternatively, charnockites do not represent  
458 true primary melt compositions as expected in rocks crystallized at lower crustal conditions  
459 and/or are slightly modified by the entrainment of peritectic phases such as garnet and  
460 orthopyroxene. By contrast, MI have similar concentrations for CaO, Al<sub>2</sub>O<sub>3</sub> and  
461 FeO+MgO+TiO<sub>2</sub> compared to the leucogranites that intrude the migmatitic paragneisses and  
462 micaschists (Galli et al. 2013). Moreover, MI overlap the trend displayed by the melt  
463 inclusions from the Kerala Khondalite Belt (Cesare et al. 2009; Ferrero et al. 2012), even  
464 though the latter have slightly higher FeO+MgO+TiO<sub>2</sub> and Cl concentrations. On the other  
465 hand, MI from the Gruf Complex have different trends for FeO+MgO+TiO<sub>2</sub>, alkalis, and  
466 partly CaO, compared to A- and S-type granites. This evidence strongly reinforces the  
467 assumption that S- and A-type granites, similar to the Gruf charnockites, do not mirror  
468 primary crustal melts, but liquids modified by a large plethora of mechanisms during their  
469 production and segregation from the source (see INTRODUCTION).

470 MI are characterized by high  $K_2O$  and low  $Na_2O$  concentrations compared to regional  
471 charnockites and leucogranites, but also relative to A- and S-type granites (Figure 8d-e).  
472 However, a decoupled behaviour in alkali contents has been also noticed in the MI from the  
473 Kerala Khondalite Belt (KKB, S-India), in which ultrapotassic rhyolitic melts have been  
474 reported (Cesare et al. 2009; Ferrero et al., 2012). It is generally believed that khondalites  
475 from the KKB originated during crustal anatexis at UHT conditions (Nandakumar and  
476 Harley, 2000; Yu, Santosh, Li, & Shaji, 2019) and therefore it seems that high  $K_2O/Na_2O$   
477 ratios are a distinctive feature of anatectic melts at extremely high crustal temperatures. K-  
478 rich melts have also been experimentally derived by Patiño Douce and Johnston (1991) from  
479 a pelitic starting material. In particular, their experiments between  $P = 7-13$  kbar and  $T =$   
480  $875-1075^\circ C$  give  $K_2O$ -rich and  $Na_2O$ -poor liquids that closely resemble MI from the Gruf  
481 Complex and the KKB. Additionally, Droop, Clemens, & Dalrymple (2003) obtained K-rich  
482 melts ( $K_2O/Na_2O = 4.1-9.5$ ) from partial melting experiments of metapelitic micaschists at  
483  $900^\circ C$  and 5 kbar.

484 In the AKF diagram (Figure 10), analyses for the KKB, as well as the experimental glasses of  
485 Droop et al. (2003) and Patiño-Douce & Johnston (1991), plot towards the K-apex,  
486 overlapping in part the field formed by the MI of the Gruf Complex. The fact that K-rich  
487 melts are observed up to UHT conditions, independently of the pressure, may be explained by  
488 the role of titanium and other elements such as F, Cl and  $Fe^{3+}$  in expanding the stability field  
489 of biotite towards higher temperatures (e.g. Mouri, Guiraud, & Hensen, 1996; Stevens et al.,  
490 1997; Tajčmanová, Connolly, & Cesare, 2009). Consequently, the melting of biotite may be  
491 delayed, compared to the melting of sodic plagioclase, leading to an enrichment of potassium  
492 in the melt.

493 All this evidence suggests that crustal melts produced at the onset of anatexis under high to  
494 ultra-high temperature conditions are dominantly potassic to ultrapotassic, as was also  
495 recognized in previous studies (Cesare et al., 2009; Ferrero et al., 2012).

496

## 497 **5.2 Volatiles in the deep crust**

498 Primary clusters of melt and fluid inclusions occur typically in both garnet cores and rims of  
499 the granulites from the Gruf Complex, indicating that they were entrapped during the same  
500 anatectic event. The coexistence of CO<sub>2</sub> with stepdaughter phases such as carbonates (Fe-  
501 magnesite, siderite and calcite) and phyllosilicates (e.g. pyrophyllite, muscovite and biotite)  
502 within FI strongly suggests that the original fluid had a COH(N) composition. Nevertheless,  
503 the presence of K-rich phases such as muscovite and biotite might also indicate that some of  
504 the FI are instead mixed inclusions (fluid+melt) and therefore part of the H<sub>2</sub>O may derive  
505 from the melt. Similar mixed inclusions were documented in garnet of crustal enclaves  
506 (Cesare, Maineri, Baron Toaldo, Pedron, & Acosta-Vigil, 2007), in anatectic granitoids  
507 (Ferrero, Braga, Berkesi, Cesare, & Laridhi Ouazaa, 2014) and in low-to-medium pressure  
508 migmatites (Ferrero, Wunder, Ziemann, Wälle, & O'Brien, 2016). Although H<sub>2</sub>O as a free  
509 phase has never been demonstrated by Raman spectroscopy within the measured FI of the  
510 Gruf Complex, its occurrence at the nanometric scale cannot be excluded (see also Lamadrid,  
511 Lamb, Santosh, & Bodnar, 2014; Esposito et al., 2016). On the other hand, carbonates and  
512 phyllosilicates are interpreted as re-speciation products after post-entrapment carbonation and  
513 hydration reactions between garnet host and fluid, as it has been suggested also for other  
514 HT/UHT terranes (Ferrero et al. 2014; Tacchetto et al., 2019; Carvalho et al., 2019, 2020).  
515 Hence, the different densities displayed by the FI are a direct consequence of incomplete  
516 reactions, which leave a variable amount of CO<sub>2</sub> in the residual fluid.

517 Raman spectroscopy measurements allowed to confirm the presence of CO<sub>2</sub> and H<sub>2</sub>O in  
518 glassy and nanogranitoid inclusions (see Fig. 4), indicating that both types of inclusions  
519 derived from the similar melt. The ubiquity of pure CO<sub>2</sub>-rich shrinkage bubbles (without any  
520 apparent detectable H<sub>2</sub>O, see discussion above) within glassy inclusions can be explained by  
521 the preferential partition of H<sub>2</sub>O into silicate melt than CO<sub>2</sub> (e.g. Holtz, Johannes, Tamic, &  
522 Behrens, 2001; Tamic, Behrens, & Holtz, 2001). Further investigations by NanoSIMS  
523 enabled the quantification of the minimum volatile component dissolved in this melt. The  
524 dissolved H<sub>2</sub>O measured in the MI displays a wide spectrum between 2.9 and 8.8 wt%, with a  
525 large proportion of the analyses that have >5 wt%. Such values indicate that crustal melts at T  
526 ~900°C can have higher H<sub>2</sub>O contents than commonly assumed for the haplogranitic system.  
527 However, as pointed out by Holtz et al. (2001), this behaviour is to be expected for melts that  
528 markedly differ from eutectic compositions and that involve the breakdown of micas in the  
529 melting reaction, conditions which are both realised for the MI from the Gruf Complex.  
530 Moreover, analogous H<sub>2</sub>O contents have been also observed in MI from the granulites and  
531 upper amphibolite-facies migmatites from the Strona Valley, in the Ivrea Zone (Carvalho et  
532 al., 2019). This may suggest that, at HT/UHT conditions, anatectic rhyolitic melts have  
533 considerable but uneven H<sub>2</sub>O contents, which may strongly affect the viscosity and therefore  
534 the mobility of these magmas. Variable H<sub>2</sub>O contents in anatectic melts were also recognized  
535 by Bartoli et al. (2014) in peritectic garnets of amphibolite-facies migmatites close to the  
536 Ronda peridotite and were interpreted to represent micro-scale buffered compositions due to  
537 heterogeneous domains in the precursor. A similar mechanism might be envisaged also for  
538 the MI of the Gruf Complex, since low and high values sometimes can be found in the same  
539 garnet crystal. Another striking feature of the analysed MI is the marked difference in the  
540 CO<sub>2</sub> contents (Figure 9). MI form two distinct groups, with the highest CO<sub>2</sub> concentrations

541 that are nearly identical to those observed in MI from granulites and amphibolites from the  
542 Ivrea Zone (see also Carvalho et al., 2019).

543 Our results clearly demonstrate that the peritectic garnets of the investigated UHT granulites  
544 grew with coexisting COH-bearing fluids and silicate melts, that, due to the low solubility of  
545 CO<sub>2</sub> in silicate melts (Holtz et al., 2001; Tamic et al., 2001), led to fluid-melt immiscibility.

546 The origin of the COH fluid in high-grade metamorphic rocks is source of debates. The  
547 granulites from the Gruf Complex lack graphite in their mineral assemblage and therefore an  
548 internal origin for carbon seems to be unlikely. However, the absence of graphite in the rocks  
549 does not preclude the possibility that graphite was present in the original mineral assemblage  
550 and was completely consumed before or during anatexis. Another source for carbon may be  
551 represented by carbonaceous sediments interlayered in metapelitic rocks. In the Gruf  
552 Complex, rare, up to 20 x 1 meter lenses of calc-silicates occur all along the contact with the  
553 Bergell tonalite, but never within the migmatitic gneisses that host the UHT granulites (Galli  
554 et al., 2013). Furthermore, scarce, up to 20-meter-thick discontinuous bands of calc-silicates  
555 have been only recognized in the mafic-ultramafic Chiavenna Unit adjacent to the Gruf  
556 Complex (Schmutz, 1976). Thus, a sedimentary source for carbon is not supported by field  
557 observations. Accordingly, we infer that CO<sub>2</sub> was externally introduced in the lower crust,  
558 very likely from a mantle source. This is in agreement with previous studies that suggested  
559 the mantle as a common source for CO<sub>2</sub> in high temperature granulites (e.g. Hoefs & Touret,  
560 1975; Jackson, Matthey, & Harris, 1988; Santosh & Omori, 2008; Touret & Huizenga, 2011).

561 However, we also emphasize that without a rigorous isotopic determination for carbon, the  
562 origin of CO<sub>2</sub> remains uncertain. On the other hand, the minor N<sub>2</sub> amount detected in Type C  
563 granulites is interpreted to derive from the breakdown of NH<sub>4</sub><sup>+</sup>-bearing micas during anatexis  
564 (e.g. Moin, Guillot, & Gibert, 1994; Cesare et al., 2007).



565 For the Gruf Complex, if an external source for CO<sub>2</sub> is assumed, the different CO<sub>2</sub>  
566 concentrations in the MI may be related to small-scale heterogeneities of the fluid  
567 composition within the matrix surrounding the growing peritectic garnets.

568

### 569 **5.3 Rheology of anatectic melt**

570 Granulites from the Gruf Complex represent UHT residues of metapelitic lower crustal rocks  
571 which lost *c.* 40-50% of melt during anatexis (Galli et al., 2011). Partial melting generated  
572 rhyolitic melts that are now preserved as MI within peritectic garnets in the granulites. The  
573 investigation of these MI not only allows to retrieve the chemical composition of anatectic  
574 melts but also to calculate melt density and viscosity, two crucial physical properties to  
575 understand melt rheology and its mobilization through the crust. Assuming a pressure of 9  
576 kbar and a temperature of 875°C, which corresponds to the experimental re-homogenization  
577 temperature, an average melt density of 2.33 g/cm<sup>3</sup> was determined using the program  
578 DensityX by Iacovino and Till (2019). On the other hand, melt viscosities calculated at  
579 875°C range between 10<sup>3.9</sup>-10<sup>4.5</sup> Pa·s, applying the equation of Scaillet, Holtz, Pichavant, &  
580 Schmidt (1996) and between 10<sup>4.1</sup>-10<sup>4.8</sup> Pa·s, using the equation of Giordano, Russell, &  
581 Dingwell (2008). These values are ~2 orders of magnitude lower than viscosities estimated  
582 for nearly anhydrous crustal melts at 900°C (e.g. Hess and Dingwell, 1996) but are consistent  
583 within error with the experimental values obtained by Ardia, Giordano, and Schmidt (2008)  
584 for rhyolitic melts between 5 and 15 kbar at similar water contents (see Figure S4).  
585 Moreover, viscosities for MI from the Gruf Complex overlap the range of viscosities obtained  
586 by Bartoli et al. (2013) for melts in metasedimentary metatexites at the contact with the  
587 Ronda peridotite, in the Spanish Betic Cordillera.  
588 The determination of viscosity and density allows us then to speculate on the ascent velocity  
589 of the melt from its source. In our calculations, melt ascent is approximated as simple porous

590 flow following Turcotte & Schubert (2002). We assume an average melt viscosity of  $10^{4.4 \pm 0.3}$   
591 Pa·s and an average melt density of  $2.33 \text{ g/cm}^3$  as calculated from MI, an average density of  
592  $3.10 \text{ g/cm}^3$  for the lower crust (Hacker, Kelemen, & Behn, 2015) and a grain size for the  
593 matrix of 1-2 mm. For porosities of 8% and 25%, which respectively correspond to the liquid  
594 percolation threshold (LPT) and the melt escape threshold (MET) of Vigneresse, Barbey, &  
595 Cuney (1996), melt velocities range between 5 and 250 mm/year (see Table S6).

596 It has to be stressed that these melt velocities are calculated for a constant temperature of  $T =$   
597  $875^\circ\text{C}$  and are relative to a static, homogeneous solid matrix. Therefore, our calculations give  
598 only a first-order approximation on the ascending behaviour of pristine anatectic melts at the  
599 onset of crustal anatexis approaching ultra-high temperatures. In fact, melting in the lower  
600 crust occurs in a dynamic, heterogeneous environment, where several factors may affect melt  
601 ascent. Ascent velocities of magmas may be strongly increased by deviatoric stresses  
602 (Sawyer 1994) or by the occurrence of high permeability anomalies in the crust, such as large  
603 scale shear zones, which may be able to provide preferred pathways for the ascending melt  
604 due to their high anisotropy. Furthermore, at high melt fractions it is expected that the  
605 pervasively distributed melt might be channelized by porosity waves (e.g. Connolly &  
606 Podladchikov, 2015), resulting in an enhanced melt migration rate. On the other hand, syn-  
607 anatexis exhumation of partly molten crustal portions to upper and colder crustal levels  
608 would decrease the temperature and therefore lower the melt velocity and its ability to be  
609 extracted. Cooling induces a strong increase of melt viscosity but only a limited density  
610 decrease, which is reflected in a dramatic drop of the melt velocity (see Table S6). Since  
611 exhumation favours high cooling rates, high exhumation rates in a dynamic crust will have a  
612 negative feedback on the melt velocity. Hence, at high cooling rates melt extraction would  
613 become less efficient and (at least) part of the produced UHT melt would be likely stuck and  
614 move coupled with the ascending hot crust (Teyssier & Whitney, 2002). Such scenario is

615 expected to become more common at anatexis temperature  $<800^{\circ}\text{C}$  because melt velocities  
616 at  $800^{\circ}\text{C}$  are almost one order of magnitude lower than at  $900^{\circ}\text{C}$  (see Table S6).

617

## 618 **6. CONCLUSIONS**

619 Evidence that the Earth's continental crust has the ability to attain, on a regional scale, ultra-  
620 high temperatures has been widely reported. Under these conditions, middle and lower crust  
621 undergo substantial modifications that have important consequences on their rheological  
622 properties, which, in turn, may largely affect the mass transfer at the crust-mantle boundary.  
623 Peraluminous, K-rich rhyolitic melt inclusions observed in garnet porphyroblasts of UHT  
624 granulites from the Gruf Complex are the result of crustal anatexis, the latter driven by the  
625 breakdown of biotite. The presence of primary melt and fluid inclusions with both a COH  
626 volatile component suggests that anatexis initially started due to dehydration melting  
627 reactions and was promoted by the influx of an external  $\text{CO}_2+\text{H}_2\text{O}$  fluid. Although the source  
628 of  $\text{CO}_2$  cannot be undoubtedly ascertained, we infer that a mantle origin for the carbon is a  
629 more likely explanation. On the other hand, the high  $\text{H}_2\text{O}$  content measured in the melt  
630 inclusions suggests that anatexis melts up to UHT conditions are less dry than commonly  
631 assumed, with relevant implications for the viscosity of anatexis melts. The production of  
632 low density rhyolitic melts in the lower crust may induce large-scale weakening (Rosenberg  
633 and Handy, 2005) that ultimately may lead to the lateral flow of mountain belts (Jamieson et  
634 al. 2011) or to the formation of domes and crustal diapirs (e.g. Gerya, Perchuk, Maresch, &  
635 Willner, 2004; Teyssier & Whitney, 2002). The latter mechanism can be an effective  
636 geological process that may trigger the exhumation of high-grade metamorphic rocks.  
637 Moreover, the potassic to ultrapotassic composition of the analysed anatexis melts may  
638 indicate that crustal anatexis can have a larger impact in the redistribution of heat-producing  
639 elements (such as  $\text{K}_2\text{O}$ ), with important implications for the thermal structure of the

640 continental crust (e.g. Antonelli, DePaolo, Chacko, Grew, & Rubatto 2019; Hacker et al.  
641 2015).

642 This study shows that anatectic melt inclusions provide a fundamental insight into the  
643 comprehension of the melting regime in UHT terranes and the chemical differentiation of the  
644 lower crust.

645

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## 1086 SUPPORTING INFORMATION

1087 Additional supporting information may be found online in the Supporting Information  
1088 section.

1089 **Figure S1.** Garnet zoning in Type A and Type C granulites.

1090 **Figure S2.** Harker diagrams showing the complete set of MI analyses.

1091 **Figure S3.** CIPW Qz-Ab-Or diagram showing normative compositions for the analysed MI  
1092 and reference analyses from the literature.

1093 **Figure S4.** Viscosities of rhyolitic melts vs. H<sub>2</sub>O content.

1094 **Table S1.** Calculated densities of CO<sub>2</sub> within melt and fluid inclusions.

1095 **Table S2.** Major elements profiles for garnets of Type A and Type C granulites.

1096 **Table S3.** Chemical compositions of minerals in nanogranitoid inclusions.

1097 **Table S4.** Composition of peritectic and daughter minerals from experimental runs at  
1098 different P-T-t conditions.

1099 **Table S5.** Chemical composition of re-homogenized and glassy melt inclusions.

1100 **Table S6.** Model parameters and calculated melt velocities

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1105 **CAPTIONS FIGURES TEXT**

1106

1107 **FIGURE 1** Tectonic map of the Central Alps modified after Trommsdorff and Nievergelt  
1108 (1983). Coordinates are given according to the Swiss geographical grid (units in kilometres).

1109

1110 **FIGURE 2** UHT granulites from the Gruf Complex. (a) Massive sapphirine-orthopyroxene-  
1111 cordierite-garnet granulite (Type A) and (b) massive orthopyroxene-cordierite-garnet  
1112 granulite (Type C).

1113

1114 **FIGURE 3** Photomicrographs of the granulites from the Gruf Complex. (a) Garnet  
1115 porphyroblast from a Type A granulite showing a cluster of MI near to the core of the crystal  
1116 (red box). (b) Zoom of the same cluster of MI as in (a). (c) Garnet porphyroblasts from a  
1117 Type C granulite. The red box at the rim of the garnet indicate a cluster of MI. (d) Zoom of  
1118 the same cluster of MI as in (c). (d) Plane-polarized light photomicrograph of MI in garnet.  
1119 (e) Same as (d) under crossed-polarized light, showing multiple birefringent phases within  
1120 the inclusions. (g) Fluid inclusions (FI) and MI within a cluster. (h) Glassy MI containing a  
1121 CO<sub>2</sub> shrinkage bubble. (i) MI in a sapphirine porphyroblast from a Type A granulite.

1122

1123 **FIGURE 4** Raman spectra of nanogranitoids and glassy MI. (a) Glassy MI with a shrinkage  
1124 bubble containing CO<sub>2</sub>. (b) Analysis of a nanogranitoid inclusion (red circle) close to a glassy  
1125 MI showing a shrinkage bubble. (c) Nanogranitoid with a CO<sub>2</sub> bubble. (d) Nanogranitoid in  
1126 sapphirine. (e) Mixed inclusion (fluid + melt) showing CO together with phyllosilicates,  
1127 quartz and Fe-magnesite. (f) Mixed inclusion with phyllosilicates, Fe-magnesite, CO<sub>2</sub> and N<sub>2</sub>.

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1129 **FIGURE 5** Backscattered images of nanogranitoids. Nanogranitoids from Type A (a-d) and  
1130 from Type C (e-f) granulites are shown.

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1132 **FIGURE 6** Backscattered images of experimentally remelted nanogranitoids. (a,b) Remelted  
1133 MI with a well-developed negative shape. (c) Re-homogenized MI showing a trapped rutile  
1134 needle, indenting the wall of the inclusion. (d) MI showing an irregular border and bubbles  
1135 within the glass. (e,f) Partially remelted nanogranitoids, displaying daughter phases (biotite  
1136 and quartz) and evidence of minor overheating (recrystallization of garnet and peritectic  
1137 orthopyroxene). (g) Interaction of matrix melt with a garnet chip, forming peritectic  
1138 orthopyroxene needles. (h) Melt pocket of infiltrated matrix melt within the garnet chip. The  
1139 glass displays abundant bubbles. (i) MI from an experiment at 950°C showing strong  
1140 evidence of overheating (formation of peritectic orthopyroxene and recrystallization of new  
1141 garnet at the wall of the inclusion).

1142

1143 **FIGURE 7** Chemical classification of remelted and glassy MI from the Gruf Complex  
1144 (normalized to 100% on anhydrous basis). Reported are MI analyses of re-melting  
1145 experiments between 800 and 900°C with graphite-bearing (Gr) and quartz-bearing (Qz)  
1146 capsules. (a) Aluminum saturation index vs. alkalinity index. (b) TAS diagram. Analyses for  
1147 charnockites and leucogranites from the Gruf Complex are from Galli et al. (2013), whereas  
1148 MI analyses from the Kerala Khondalite Belt are from Cesare et al. (2009) and Ferrero et al.  
1149 (2012). Data for S-type granites (Chappel & White, 1992; Jung, Hoernes, & Mezger, 2000;  
1150 Kalsbeek, Jepsen, & Jones, 2001; Tulloch, Ramezani, Kimbrough, Faure, & Allibone, 2009;  
1151 Visonà & Lombardo, 2002) and for A-type granites (Castro et al., 2012; de Almeida, de  
1152 Pinho Guimarães, & da Silva Filho, 2002; Florisbal, de Fátima Bitencourt, Nardi, &  
1153 Conceição, 2009; Ghani et al., 2014; Goodenough, Upton, & Ellam, 2000; King, White,



1154 Chappell, & Allen, 1997; King, Chappell, Allen, & White, 2001; McDermott, Harris, &  
1155 Hawkesworth, 2000; Whalen, Currie, & Chappell, 1987; Zhang et al., 2007) are also reported  
1156 for comparison.

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1158 **FIGURE 8** Chemical composition of glasses from experimentally remelted and natural  
1159 glassy inclusions (normalized to 100% on anhydrous basis). Reference analyses are the same  
1160 as in Figure 7.

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1162 **FIGURE 9** H<sub>2</sub>O and CO<sub>2</sub> content measured in remelted nanogranitoid inclusions. Error bars  
1163 are displayed only when bigger than the symbol. Data for MI from the Ivrea Zone (Carvalho  
1164 et al., 2019) are also reported for comparison (black dots = upper amphibolite facies, grey  
1165 squares = transition zone, brown crosses = granulite facies).

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1167 **FIGURE 10** AKF diagram (after Vielzeuf & Holloway, 1988) projected from quartz for MI  
1168 and bulk rocks. (a) Type A granulite. (b) Type C granulite. Also reported for comparison are:  
1169 melt inclusions from the Kerala Khondalite Belt (Cesare et al., 2009; Ferrero et al., 2012),  
1170 experimental glasses (900°C, 5 kbar) of Droop et al. (2003) and experimental glasses (875-  
1171 1075°C, 7-13 kbar) of Patiño-Douce & Johnston (1991).

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1179 **CAPTIONS FIGURES SUPPORTING INFORMATION**

1180

1181 **Figure S1.** Compositional profiles of garnets in Type A and Type C granulites.

1182 **Figure S2.** Harker diagrams showing the complete set of MI analyses. Small symbols

1183 represent disregarded analyses. The line displayed for Al<sub>2</sub>O<sub>3</sub>, FeO and and MgO represents

1184 the tie-line between the host garnet (Type A granulite) and the average composition of the

1185 glassy inclusions (black triangle).

1186 **Figure S3.** CIPW Qz-Ab-Or diagram showing normative compositions for the analysed MI

1187 and other reference analyses from the literature (see text).

1188 **Figure S4.** Viscosities of rhyolitic melts vs. H<sub>2</sub>O content (modified after Ardia et al., 2008).

1189 Viscosities for MI are calculated according to Giordano et al. (2008).

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1           **Title: Anatectic melt inclusions in ultra-high temperature granulites**

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	Type A granulites		Type C granulites	
	BRE-core	BRE-rim (glassy)	GRAG-core	GRAG-rim
No. analyses by EPMA / NanoSIMS	6 / 6	11	21 / 3	6 / 7
Wt%				
SiO <sub>2</sub>	70.08 (2.51)	75.33 (1.26)	74.90 (1.00)	71.93 (1.54)
TiO <sub>2</sub>	0.05 (0.04)	0.00 (0.01)	0.05 (0.04)	0.06 (0.07)
Al <sub>2</sub> O <sub>3</sub>	14.45 (0.88)	12.87 (0.64)	11.75 (0.43)	13.29 (1.19)
FeO	1.67 (0.32)	0.91 (0.05)	1.52 (0.14)	1.93 (0.36)
MnO	0.05 (0.02)	0.05 (0.02)	0.02 (0.02)	0.08 (0.04)
MgO	0.54 (0.24)	0.10 (0.08)	0.71 (0.10)	0.53 (0.23)
CaO	0.94 (0.55)	0.05 (0.02)	0.51 (0.15)	0.55 (0.19)
Na <sub>2</sub> O	1.96 (0.54)	1.66 (0.38)	0.73 (0.27)	0.72 (0.39)
K <sub>2</sub> O	7.31 (1.43)	8.67 (0.82)	6.96 (0.48)	7.95 (1.88)
P <sub>2</sub> O <sub>5</sub>	0.20 (0.18)	0.04 (0.03)	0.12 (0.06)	0.17 (0.15)
Cl	0.04 (0.03)	0.01 (0.00)	0.06 (0.02)	0.12 (0.04)
Total	97.31 (2.33)	99.70 (0.31)	97.33 (0.79)	97.33 (1.57)
H <sub>2</sub> O by NanoSIMS [wt%]	6.20 (1.25)	-	7.05 (3.00)	4.35 (0.92)
CO <sub>2</sub> by NanoSIMS [ppm]	779 (326)	-	1113 (843)	940 (407)
ASI	1.14 (0.13)	1.06 (0.03)	1.24 (0.07)	1.27 (0.19)
K#	71 (8)	77 (5)	87 (4)	87 (7)
Mg#	35 (8)	13 (8)	44 (4)	32 (11)
CIPW norm				
Quartz	27.04	31.56	40.98	34.15
Corundum	2.10	0.76	2.39	2.89
Orthoclase	43.23	51.23	41.13	46.98
Albite	16.59	14.02	6.16	6.13
Anorthite	3.31	0.00	1.73	1.65
Hypersthene	4.43	2.01	4.51	4.91
Ilmenite	0.10	0.01	0.09	0.11
Apatite	0.47	0.10	0.29	0.39

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2 **TABLE 1** Average compositions of remelted and preserved glassy MI. Numbers in  
3 parentheses refer to the standard error of the mean expressed as 2σ. ASI = molar  
4 [Al<sub>2</sub>O<sub>3</sub>/(CaO+Na<sub>2</sub>O+K<sub>2</sub>O)], K# = molar [K<sub>2</sub>O/(K<sub>2</sub>O+Na<sub>2</sub>O)] x 100; Mg# = molar  
5 [MgO/(MgO+FeO)] x 100.

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Analysis	Sample	Approx. size	Homogeneity	H <sub>2</sub> O [wt%]	1 $\sigma$ error	CO <sub>2</sub> [ppm]	1 $\sigma$ error
<i>Type A granulites</i>							
C_MI1_Gr1	BRE17-B13 core	<10 $\mu$ m	Glass	5.52	0.04	460	20
C_MI3_Gr3_1	BRE17-B13 core	$\geq$ 20 $\mu$ m	Glass + bubble	8.27	0.27	1164	33
C_MI3_Gr3_2	BRE17-B13 core	$\geq$ 20 $\mu$ m	Glass + bubble	7.34	0.23	1064	31
C_MI16_Gr4	BRE17-B13 core	$\geq$ 20 $\mu$ m	Glass + 10% crystals + bubble	6.88	0.21	1194	34
E_MI6_Gr2_1	BRE17-D2 core	<10 $\mu$ m	Glass	4.41	0.04	408	17
E_MI6_Gr2_2	BRE17-D2 core	<10 $\mu$ m	Glass	4.76	0.04	387	17
OG16_MI2_Gr1	BRE17-B13 rim	<10 $\mu$ m	Glass + 3% crystals	4.68	0.04	1153	32
<i>Type C granulites</i>							
A_MI1_Gr1	GRAG-P3 core	<10 $\mu$ m	Glass	8.75	0.07	1738	41
A_MI3_Gr2	GRAG-P3 core	<10 $\mu$ m	Glass + bubble	8.34	0.06	1291	33
A_MI2_Gr2	GRAG-P3 core	<10 $\mu$ m	Glass + 3% crystals	4.06	0.04	310	17
F_MI12_Gr1	GRAG-P2 Gr1 rim	10-20 $\mu$ m	Glass + 3% crystals	4.51	0.04	160	15
F_MI1_Gr4	GRAG-P2 Gr1 rim	<10 $\mu$ m	Glass + 2% crystals	2.88	0.04	1569	34
OG18_MI1_Gr4	GRAG-P3 rim	<10 $\mu$ m	Glass	3.11	0.04	1149	28
OG18_MI3_Gr4	GRAG-P3 rim	<10 $\mu$ m	Glass + bubble	3.44	0.04	1064	32
OG18_MI6_Gr1	GRAG-P3 rim	<10 $\mu$ m	Glass + 1% crystals	5.18	0.04	221	15
OG18_MI4_Gr2_1	GRAG-P3 rim	10-20 $\mu$ m	Glass	5.94	0.04	1287	31
OG18_MI4_Gr2_2	GRAG-P3 rim	10-20 $\mu$ m	Glass	5.41	0.04	1132	27

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3 **TABLE 2** H<sub>2</sub>O and CO<sub>2</sub> contents of re-homogenized MI determined by NanoSIMS.

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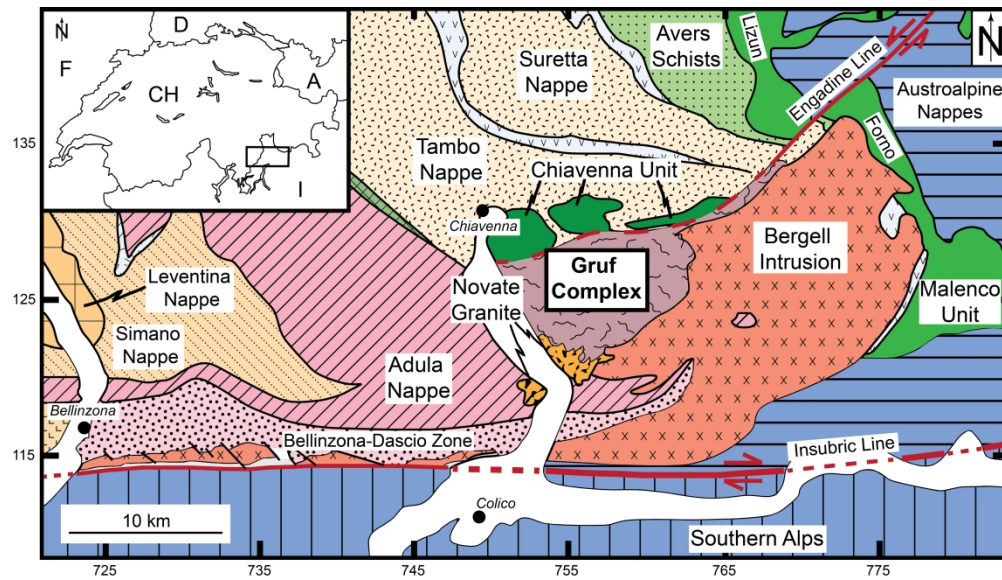


FIGURE 1 Tectonic map of the Central Alps modified after Trommsdorff and Nievergelt (1983). Coordinates are given according to the Swiss geographical grid (units in kilometres).

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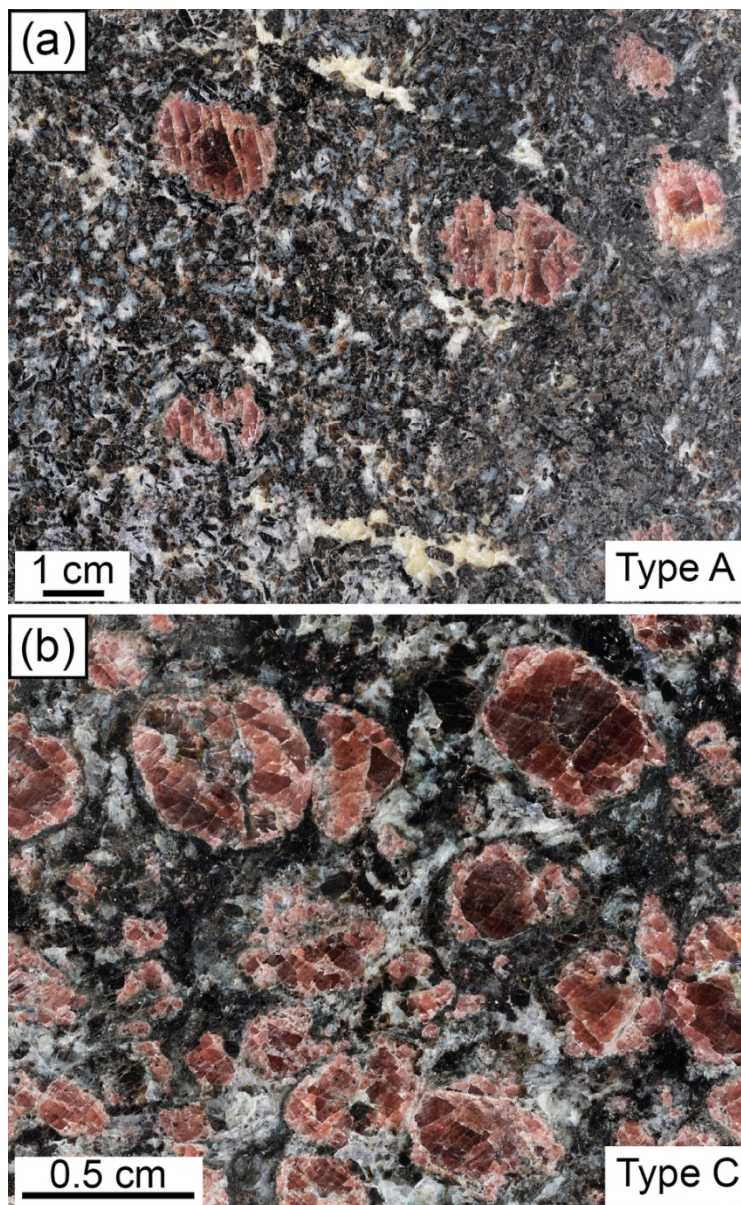


FIGURE 2 UHT granulites from the Gruf Complex. (a) Massive sapphirine-orthopyroxene-cordierite-garnet granulite (Type A) and (b) massive orthopyroxene-cordierite-garnet granulite (Type C).

80x129mm (300 x 300 DPI)



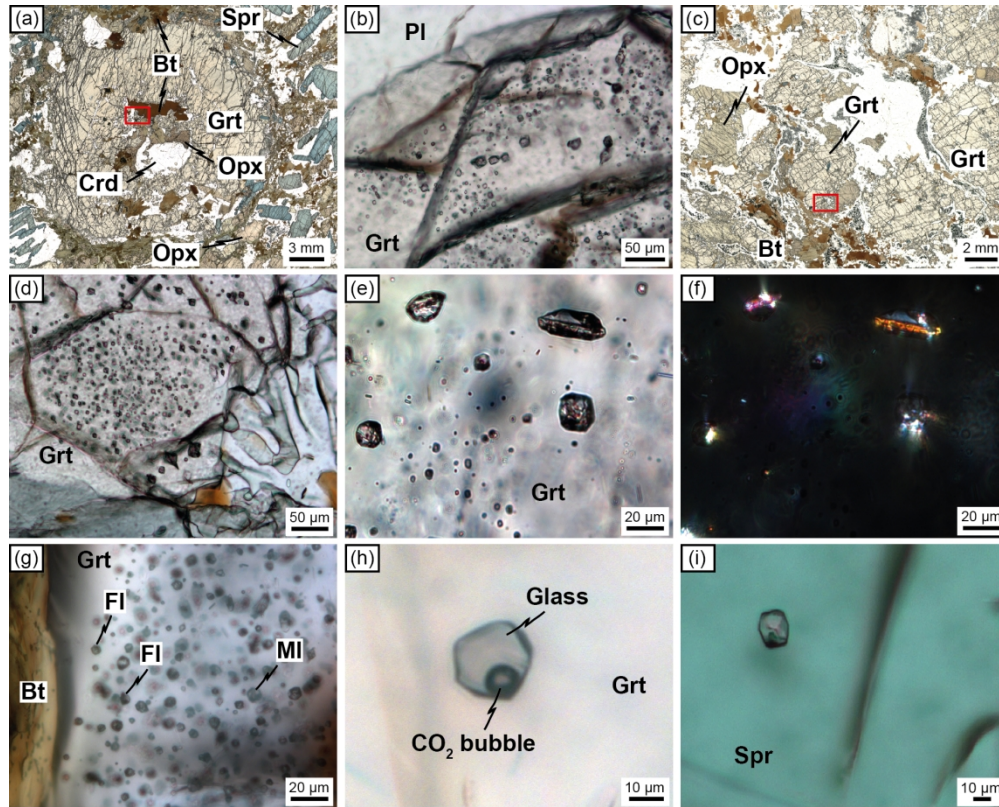


FIGURE 3 Photomicrographs of the granulites from the Gruf Complex. (a) Garnet porphyroblast from a Type A granulite showing a cluster of MI near to the core of the crystal (red box). (b) Zoom of the same cluster of MI as in (a). (c) Garnet porphyroblasts from a Type C granulite. The red box at the rim of the garnet indicate a cluster of MI. (d) Zoom of the same cluster of MI as in (c). (d) Plane-polarized light photomicrograph of MI in garnet. (e) Same as (d) under crossed-polarized light, showing multiple birefringent phases within the inclusions. (g) Fluid inclusions (FI) and MI within a cluster. (h) Glassy MI containing a CO<sub>2</sub> shrinkage bubble. (i) MI in a sapphirine porphyroblast from a Type A granulite.

166x133mm (300 x 300 DPI)



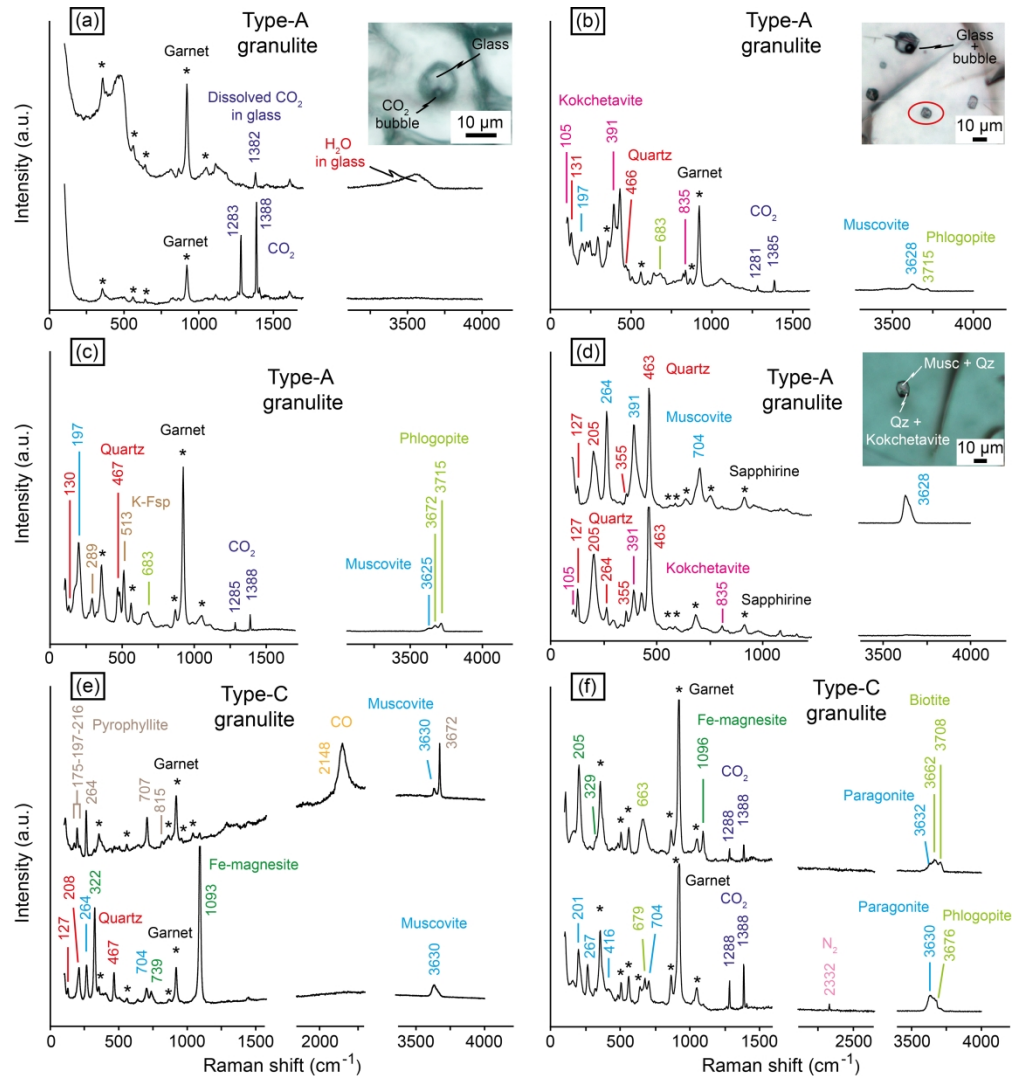


FIGURE 4 Raman spectra of nanogranitoids and glassy MI. (a) Glassy MI with a shrinkage bubble containing CO<sub>2</sub>. (b) Analysis of a nanogranitoid inclusion (red circle) close to a glassy MI showing a shrinkage bubble. (c) Nanogranitoid with a CO<sub>2</sub> bubble. (d) Nanogranitoid in sapphirine. (e) Mixed inclusion (fluid + melt) showing CO together with phyllosilicates, quartz and Fe-magnesite. (f) Mixed inclusion with phyllosilicates, Fe-magnesite, CO<sub>2</sub> and N<sub>2</sub>.

168x180mm (600 x 600 DPI)

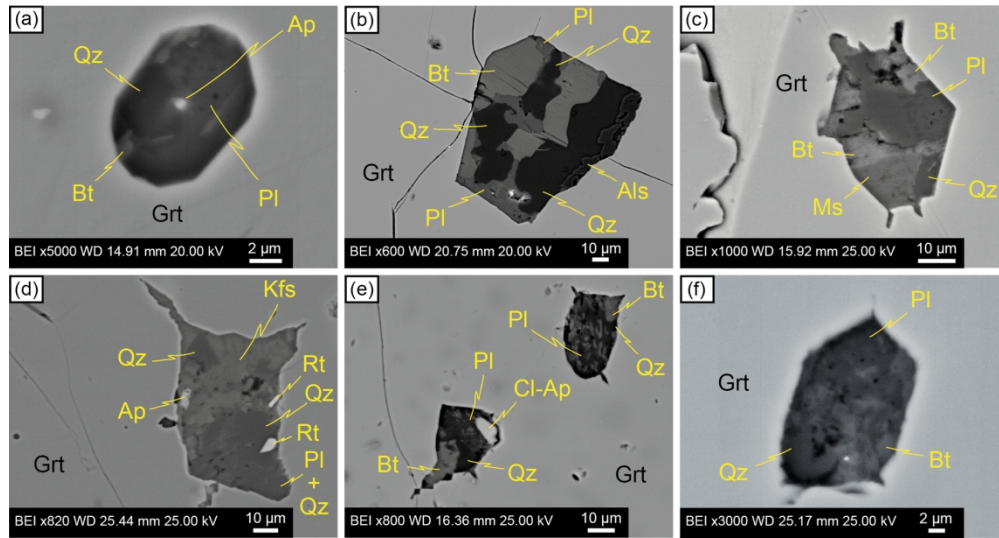


FIGURE 5 Backscattered images of nanogranitoids. Nanogranitoids from Type A (a-d) and from Type C (e-f) granulites are shown.

168x89mm (300 x 300 DPI)

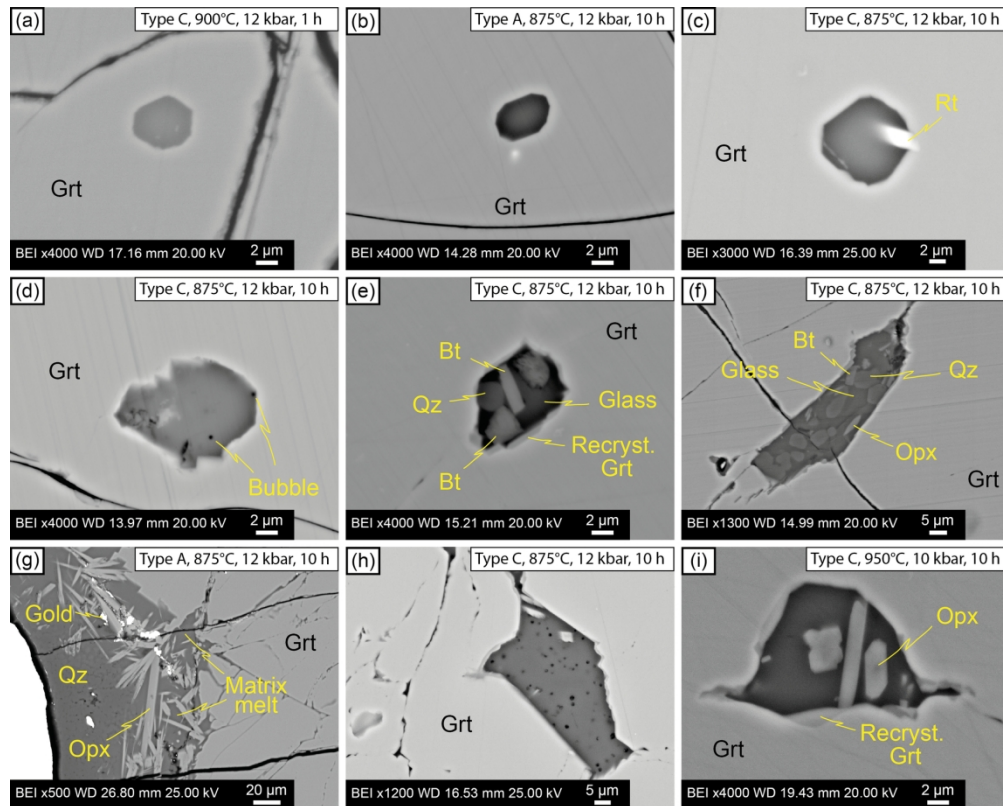


FIGURE 6 Backscattered images of experimentally remelted nanogranitoids. (a,b) Remelted MI with a well-developed negative shape. (c) Re-homogenized MI showing a trapped rutile needle, indenting the wall of the inclusion. (d) MI showing an irregular border and bubbles within the glass. (e,f) Partially remelted nanogranitoids, displaying daughter phases (biotite and quartz) and evidence of minor overheating (recrystallization of garnet and peritectic orthopyroxene). (g) Interaction of matrix melt with a garnet chip, forming peritectic orthopyroxene needles. (h) Melt pocket of infiltrated matrix melt within the garnet chip. The glass displays abundant bubbles. (i) MI from an experiment at 950°C showing strong evidence of overheating (formation of peritectic orthopyroxene and recrystallization of new garnet at the wall of the inclusion).

168x135mm (300 x 300 DPI)

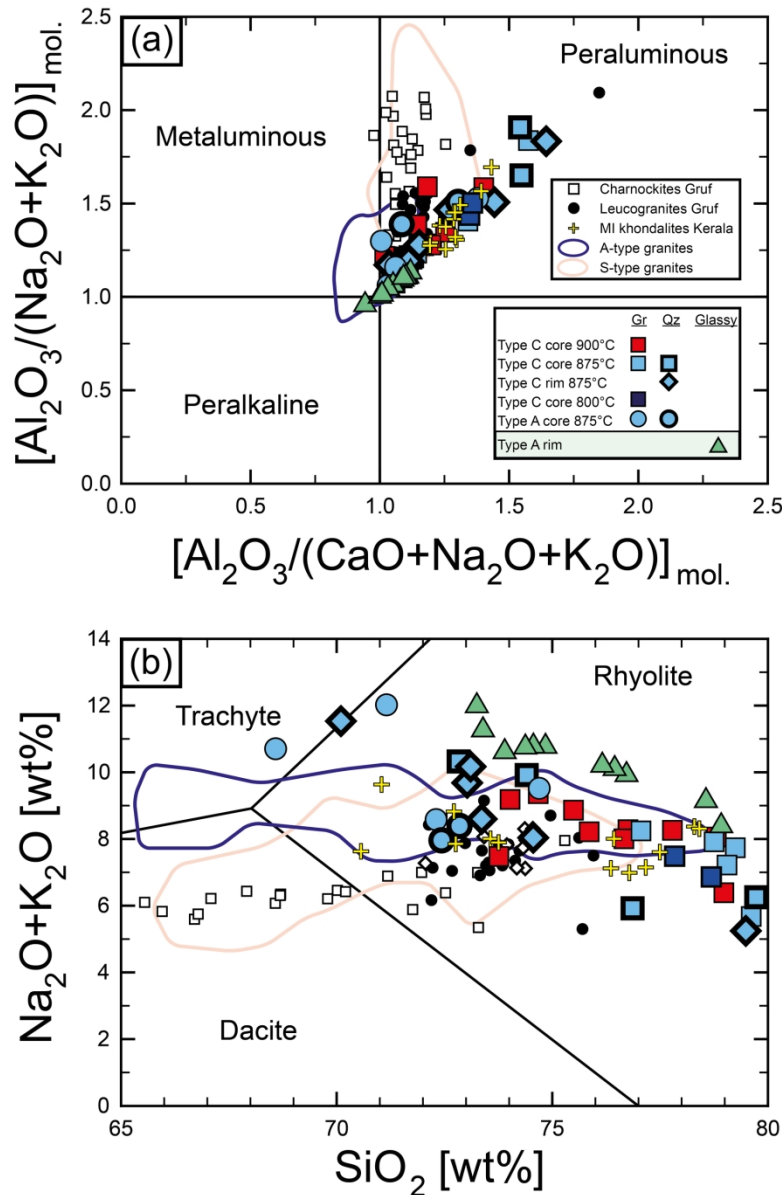


FIGURE 7 Chemical classification of remelted and glassy MI from the Gruf Complex (normalized to 100% on anhydrous basis). Reported are MI analyses of re-melting experiments between 800 and 900°C with graphite-bearing (Gr) and quartz-bearing (Qz) capsules. (a) Aluminum saturation index vs. alkalinity index. (b) TAS diagram. Analyses for charnockites and leucogranites from the Gruf Complex are from Galli et al. (2013), whereas MI analyses from the Kerala Khondalite Belt are from Cesare et al. (2009) and Ferrero et al. (2012). Data for S-type granites (Chappel & White, 1992; Jung, Hoernes, & Mezger, 2000; Kalsbeek, Jepsen, & Jones, 2001; Tulloch, Ramezani, Kimbrough, Faure, & Allibone, 2009; Visonà & Lombardo, 2002) and for A-type granites (Castro et al., 2012; de Almeida, de Pinho Guimarães, & da Silva Filho, 2002; Florisbal, de Fátima Bitencourt, Nardi, & Conceição, 2009; Ghani et al., 2014; Goodenough, Upton, & Ellam, 2000; King, White, Chappell, & Allen, 1997; King, Chappell, Allen, & White, 2001; McDermott, Harris, & Hawkesworth, 2000; Whalen, Currie, & Chappell, 1987; Zhang et al., 2007) are also reported for comparison.



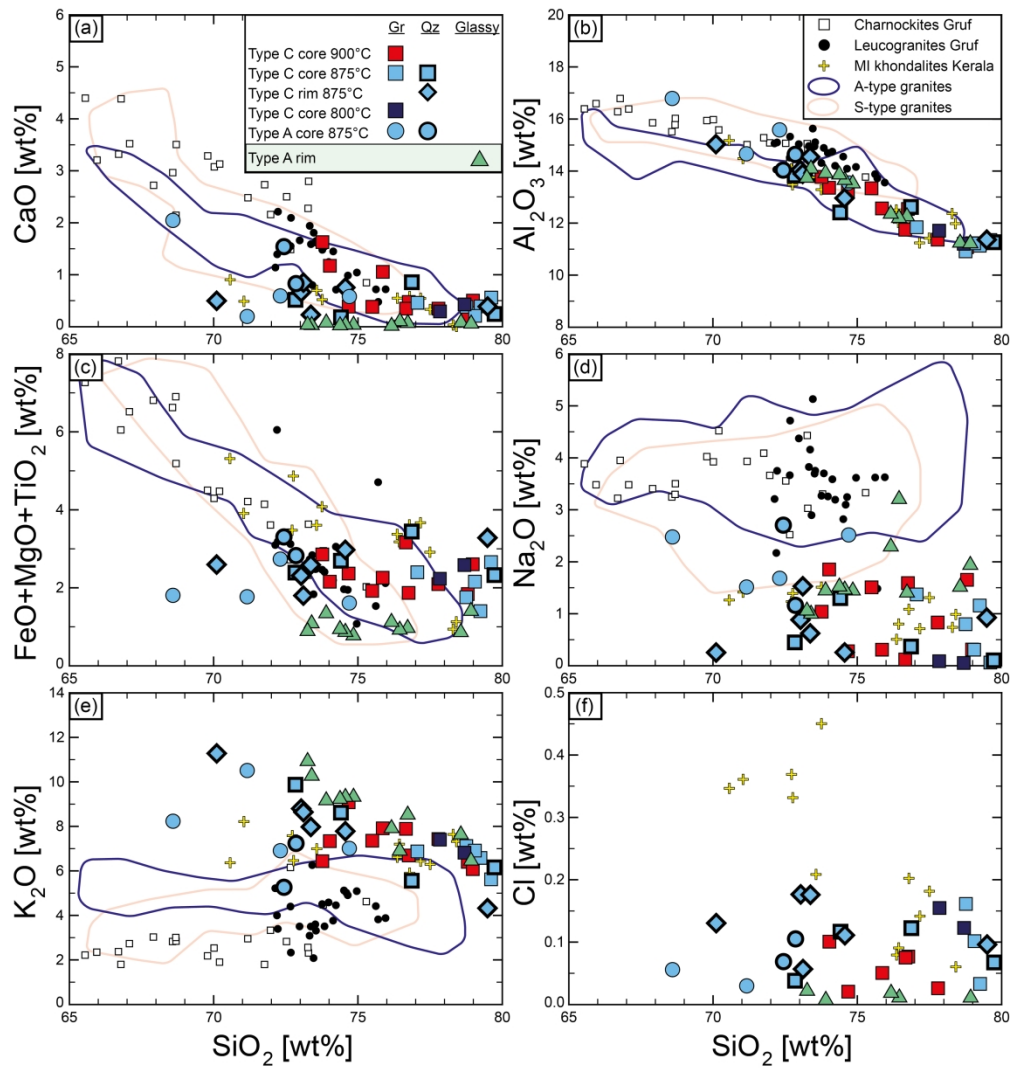


FIGURE 8 Chemical composition of glasses from experimentally remelted and natural glassy inclusions (normalized to 100% on anhydrous basis). Reference analyses are the same as in Figure 7.

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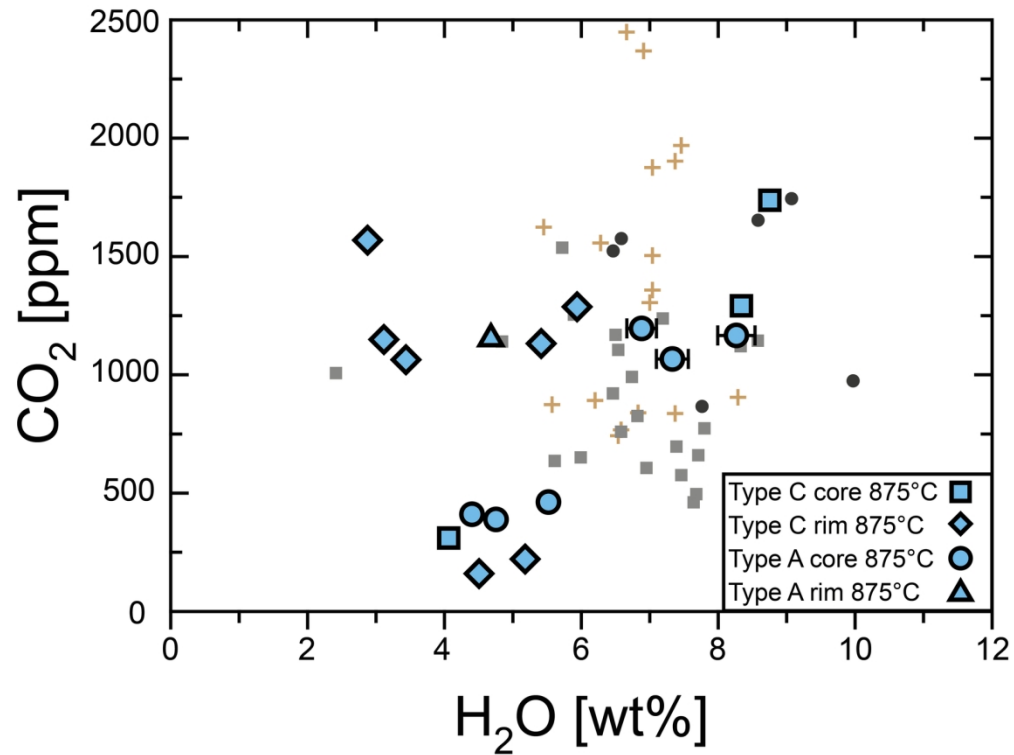


FIGURE 9 H<sub>2</sub>O and CO<sub>2</sub> content measured in remelted nanogranitoid inclusions. Error bars are displayed only when bigger than the symbol. Data for MI from the Ivrea Zone (Carvalho et al., 2019) are also reported for comparison (black dots = upper amphibolite facies, grey squares = transition zone, brown crosses = granulite facies).

78x59mm (600 x 600 DPI)

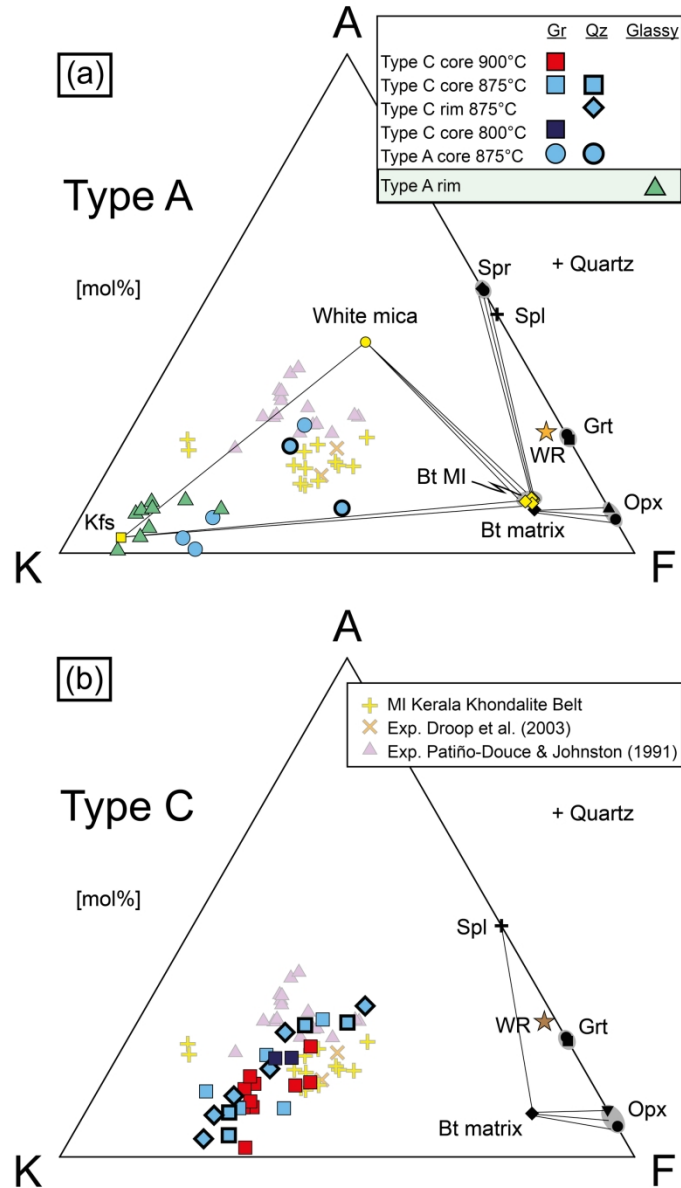
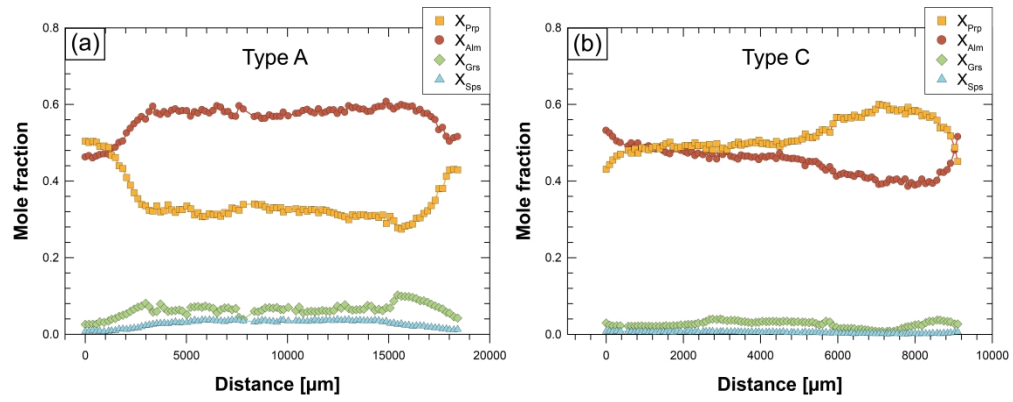


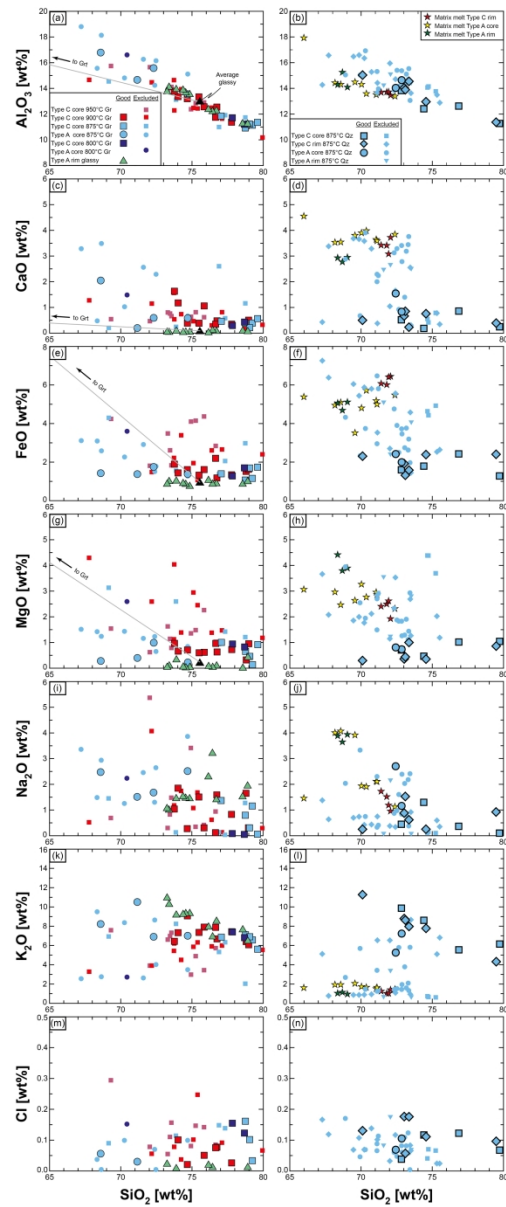
FIGURE 10 AKF diagram (after Vielzeuf & Holloway, 1988) projected from quartz for MI and bulk rocks. (a) Type A granulite. (b) Type C granulite. Also reported for comparison are: melt inclusions from the Kerala Khondalite Belt (Cesare et al., 2009; Ferrero et al., 2012), experimental glasses (900°C, 5 kbar) of Droop et al. (2003) and experimental glasses (875-1075°C, 7-13 kbar) of Patiño-Douce & Johnston (1991).

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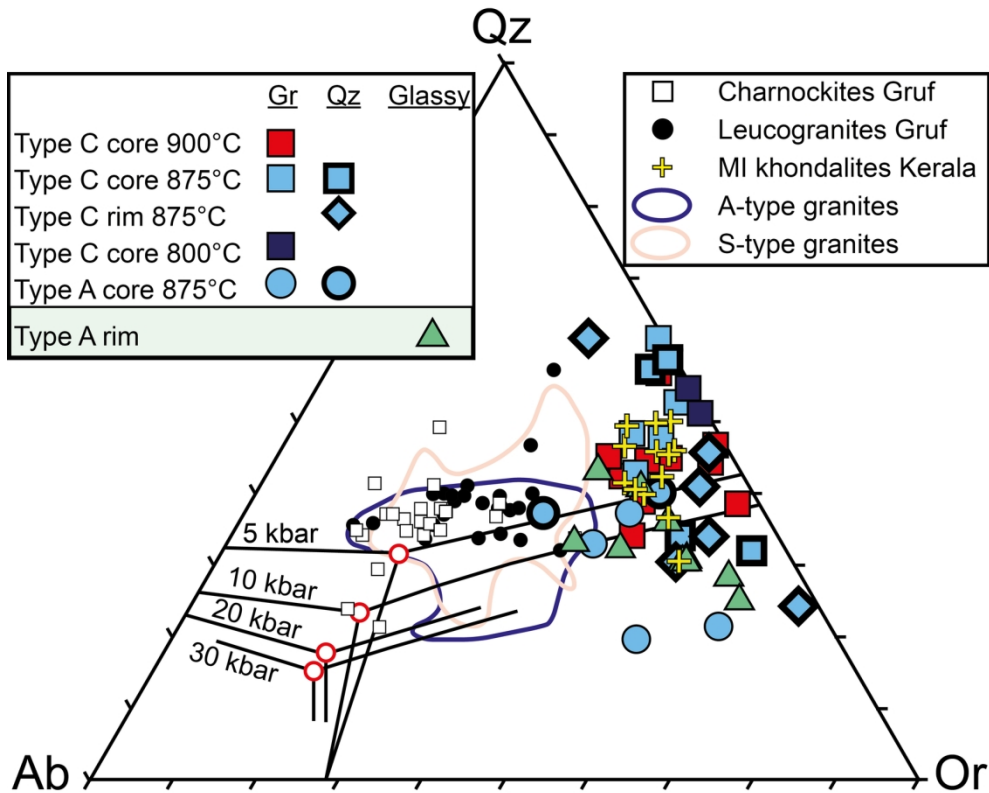




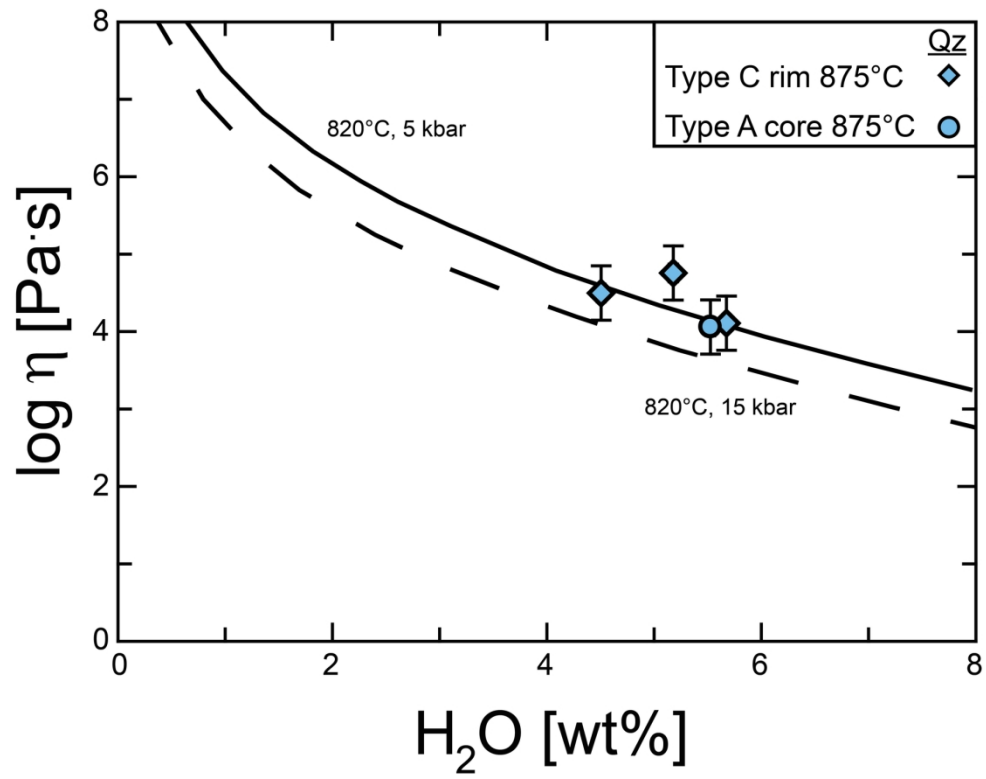
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