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Microstructures of melt inclusions in anatectic metasedimentary rocks

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Abstract

The recent finding of crystallized and glassy melt inclusions (MI) in high-grade, partially melted metapelites and metagraywackes opened up the possibility to investigate anatectic processes through the MI study technique. The present work expands the study of Cesare et al. (2009; 2011) by focussing on three case studies: Khondalites (India), Ronda migmatites (Spain) and Barun Gneiss (Nepal Himalaya). The results of a detailed microstructural investigation are here reported, along with some new microchemical data on MI bulk composition.

16 MI were trapped within peritectic garnet and ilmenite during their growth, and are therefore primary 17 inclusions. Inclusions are generally isometric and very small in size, mostly $\leq 15 \mu m$, and only rarely $\leq 30 \mu m$. 18 In most cases inclusions are crystallized ("nanogranites"), and contain a granitic phase assemblage with 19 quartz, feldspars and one or two micas (depending on the case study), often along with accessory phases (mainly Zr, Ap, Rt). Besides nanogranites, partially crystallized inclusions are locally abundant, and also 20 21 glassy inclusions, generally very small ($\leq 8 \mu m$) may occur in the same cluster. After entrapment, inclusions 22 underwent limited microstructural modifications, such a diffuse shape maturation, locally necking-down 23 processes, and decrepitation (mainly in Barun gneisses), which, however, did not influence their bulk 24 composition. Re-homogenized nanogranites and glassy inclusions (where present) always show a 25 leucogranitic and peraluminous melt, whose composition varies depending on the case study and it is 26 generally consistent with the results of partial melting experiments on metapelites and metagraywackes.

Anatectic MI should therefore be considered as a new and important tool to understand the partial melting
 processes, providing further constraints to anatectic melt composition in metasedimentary rocks.

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1) Introduction and aim of the study

Melt inclusions (MI) represent a powerful tool to gain data on several processes of the Earth system, from 31 32 magma behavior during eruptions to the emplacement of plutons in the upper crust (Roedder, 1984; Student 33 & Bodnar, 1996; Webster, 2006). The abundance of MI in magmatic minerals was already recognized in the 34 XIX century by H.C. Sorby (1858), who published the first study of MI from various types of igneous rocks 35 (and also from some artificial products, such as slags), supported by a detailed petrological characterization. 36 Many of the considerations he made on this topic are still valid, e.g. the same approach and assumptions 37 applied to the interpretation of fluid inclusions (FI) in minerals may apply to MI (see also Roedder, 1984; 38 Bodnar & Student, 2006). Despite the long-lived interest in MI, a large amount of data became available only 39 with the spread of new in-situ microanalytical techniques. In particular, the development of electron 40 microprobes allowed the characterization of major and minor elements (e.g. Anderson, 1974; Melson, 1983), 41 while more recently trace elements can be quantified using laser ablation ICP-MS also (e.g. Halter et al., 42 2002). Moreover, infrared spectroscopy, ion microprobe and new methods for analyses of hydrous glasses 43 make now possible to characterize and quantify also volatiles within the enclosed melt (see Sobolev, 1996 44 and related references). MI bulk composition determined by these means is likely to reflect magma chemistry 45 at depth (e.g. Roedder, 1979; Sobolev, 1996; Frezzotti, 2001).

Crystal phases that nucleate and grow in (or along with) a magma have the chance of enclosing portions of the melt present at the moment of their growth (Roedder, 1984). When metamorphic rocks undergo partial melting with incongruent reactions, droplets of melt may be trapped as inclusions by the peritectic phase(s) that is nucleating at the same time the melt is produced (Cesare, 2008). Petrologists' chance to study those small portions of trapped melt mainly resides on their preservation during their retrograde path. Unfortunately, between the partial melting and the exhumation on the Earth's surface, high-grade 52 metamorphic rocks generally reside for a long time at P-T conditions likely to produce changes in the 53 peritectic phases, e.g. retrograde reactions, re-crystallization, with subsequent erasure and/or modifications of 54 the MI possibly present.

55 Although it is intuitive that peritectic phases may trap anatectic MI, no clear and natural evidence was reported until the finding of primary glassy MI in metapelitic enclaves (Cesare et al., 1997) from the 56 57 Neogene Volcanic Province (NVP), Spain. These MI did not undergo the effects of a slow cooling, because 58 the enclaves were rapidly brought up to the surface. This case of frozen "migmatites-in-progress" preserved 59 many of the features they had at depth, allowing the verification of many of the current ideas on the 60 mechanisms of partial melting, derived by the petrological characterization of classic migmatites (Cesare, 2008 and references therein). Glassy inclusions from the NVP are believed to contain the anatectic melt 61 62 produced during the prograde path of the rock at T > 700 °C and preserved by rapid cooling, as confirmed by 63 microstructural and compositional characterization (see Cesare et al., 2007; Ferrero et al., 2010 for major 64 elements; see Acosta-Vigil et al., 2007, 2010 for major and trace elements). Proof of the occurrence of anatectic MI also in classic migmatitic terrains came with the finding of mainly crystallized MI within 65 Khondalites from Kerala Khondalite Belt (KKB, India), reported by Cesare et al., (2009). These MI occur in 66 67 peritectic garnets, and show a bulk composition consistent with that expected from the partial melting of 68 metapelites at T approaching 900°C. The generally granitic phase assemblage, the sub-um grain size often 69 observed and the presence of igneous microstructures led to the choice of the name "nanogranites" (Cesare et 70 al., 2009) for these inclusions.

The reported case studies demonstrate that it is possible to apply the MI study techniques to the characterization of anatectic processes in migmatites and granulites, a fundamental point in the understanding of the crustal differentiation trough time (Brown & Rushmer, 2006). The composition of the anatectic melt is commonly investigated by focusing on migmatites, in particular on the leucosome produced by the crystallization of the anatectic melt. However, several processes, such as cumulus phenomena, entrainment of xenocrysts and fractional crystallization, may change its composition. This fact makes any estimation based on the chemical composition of leucosomes highly questionable (Sawyer, 1996, 2008; Marchildon and Brown, 2001). To overcome the problems listed above, the experimental approach became in the last decades a well established tool in partial melting investigation on metapelites and metagraywackes (e.g., Thompson 1982; Vielzeuf & Holloway 1988; Carrington & Harley 1995; Gardien et al. 1995; Stevens et al. 1997; Patiño-Douce & Harris 1998), since they are the most fertile (melt-producing) lithologies in the middle crust. However, also in this case it must be considered that experimental conditions cannot entirely reproduce those at which partial melting takes place in nature, in terms of both a_{H2O} and bulk rock composition.

84 The finding of anatectic MI in migmatites gives us now the chance to study anatectic melt in situ within 85 natural samples. In this case, the real challenge becomes the small size of the anatectic MI. Inclusions in 86 KKB are $\leq 30 \ \mu m$ in diameter, and contain an aggregate of quartz, biotite, K-feldspar and plagioclase, close or beyond the resolution limits of many analytical instruments. Cesare et al. (2009) showed that these MI can 87 88 be thoroughly characterized by using high resolution electron microscopes and microprobes. Not 89 surprisingly, these inclusions strongly resemble the sketches of Sorby (1858) of MI, author's "stone cavities", 90 in plutonic rocks, although Sorby's inclusions are generally larger. Both anatectic and plutonic MI are 91 partially/totally crystallized (or devitrified) (Frezzotti et al., 1992), as expected for melt droplets in slowly 92 cooled rocks, and are small in size, see e.g. MI in Erzegebirge granites which show mean diameter 10 µm 93 (Thomas et al., 1996).

94 The two case studies previously reported, MI in Spanish enclaves and in Khondalites, represent the first 95 detailed characterization with modern high-resolution techniques. Two previous cases of MI in migmatitic 96 rocks are briefly reported in literature. Tomilenko and Chupin (1983; see also Touret and Olsen, 1985) 97 discuss small ($\leq 10 \mu m$ across) silicate inclusions in high grade terrains, containing an unknown silicate phase 98 along with crystals, and often a shrinkage bubble. However, since they occur in guartz and plagioclase within 99 the leucosome, these MI probably did not form during prograde melting, and therefore do not contain the 100 original anatectic melt produced during the prograde history of the rock. In the second case, Hartel et al. 101 (1990) reports crystallized MI in garnet from felsic migmatites collected in the western central Sulawesi,

Indonesia. They are commonly crystallized MI, 2-10 µm across, with dodecahedral shape and a roughly granitic composition -from microchemical data obtained by using a defocalized analytical beam and subtracting the estimated host contribution. Such inclusions were interpreted as containing melt produced by biotite dehydration melting, although compositional data are limited due to the MI average size.

This study aims to present and discuss anatectic MI features in greater detail with respect to the previous 106 107 works of Cesare et al. (2009; 2011). With reference to three different case studies of migmatitic-granulitic 108 terrains, Khondalites (KKB, India), Ronda migmatites (Betic Cordilleras, SE Spain) and Barun Gneiss (Higher Himalavan Crystalline or HCC, Nepal), the emphasis of this paper is primarily microstructural. The 109 110 chosen case studies are high-grade metasedimentary rocks which experienced different P-T evolutions. New microstructural and microchemical data on anatectic MI in Khondalites and in Ronda migmatites are 111 112 presented and discussed in this paper, along with a detailed study of MI in Barun gneisses, firstly reported by 113 Groppo et al., (submitted). The characterization of the host minerals, the MI microstructures and the internal 114 crystallization microstructures of the phases was performed by using both optical and FEG microscopes. The bulk composition of the trapped phase was then investigated by re-melting crystallized inclusions and 115 116 analyzing them with an electron microprobe using a very narrow electron beam.

117 The three selected case studies are the first studied from a continuously-growing list of findings from 118 different geological settings, including Ivrea-Verbano Zone (T.Ewing, Pers. Comm.), Adirondacks mountains (Henriquez & Darling, 2009), Massachusettes (J.Thomson. Pers. Comm.), Argentera Massif 119 120 (Ferrero), Ulten zone (Braga & Massonne, 2008) and La Galite archipelago (R. Braga, Pers, Comm.). Many of the listed case studies are within intensively studied terrains, where anatectic MI have been evidently 121 122 overlooked until now. Moreover, the length of the case study list by itself suggests that anatectic MI are less rare than what expected and may then be present in many high-grade terrains, in spite of the fact that they 123 124 were (almost) never reported in literature until now.

125

126 **2) Geological setting and petrography**

127 The first case study consists of garnet-sillimanite-graphite gneisses, named khondalites, from KKB in southern India, that underwent anatexis at HT/UHT conditions during the PanAfrican event (Chacko et al., 128 129 1996). At the outcrop scale these rocks are stromatic diatexites, coarse to medium in grain size (Fig. 1a). The 130 melanosome consists of large garnet and cordierite porphyroblasts, ≤ 2 cm across, coarse-grained sillimanite, biotite, ilmenite, green spinel and a small amounts of feldspar, along with flakes of graphite. The leucosome 131 consists of guartz and massive feldspar porphyroblasts, up to 5 mm, and locally plagioclase is abundant. 132 133 Melanosome may be distinguished in Bt-poor and Bt-rich types even at thin section scale, possibly reflecting 134 a non homogeneous protolith with a more or less refractory assemblage. The Bt-rich melanosome shows a spaced foliation, due to iso-orientation of coarse-grained sillimanite and biotite, while the Bt-poor 135 melanosome is not internally foliated, but defines a mineralogical banding through interfingering with 136 137 leucosome domains. These migmatites experienced a counter-clockwise PT evolution, with a partial melting 138 event, corresponding to the metamorphic peak, around 900°C (Braun et al., 1996; Shabeer et al., 2005,) and 139 0.6-0.8 GPaGPa (Cenki et al., 2002), followed by an almost isobaric cooling down to 800°C (Nandakumar & Harley, 2000) and then an isothermal decompression to 0.55 - 0.3 GPa (Santosh, 1986; Chacko et al., 1987). 140 141 Ronda migmatites are located at the western part of the Internal Zone of the Betic Cordillera (SE Spain) 142 and underlie the Ronda peridotites. The migmatites range from metatexites (Fig. 1b), diatexites to mylonitic 143 migmatites towards the contact with the overlying peridotites (Acosta-Vigil et al., 2001; Esteban et al., 144 2008). The hot thrusting of the peridotites over a metasedimentary sequence produced a dynamothermal 145 aureole during the Alpine Orogeny (Tubía & Cuevas, 1986; Tubía et al., 1997; Platt & Whitehouse, 1999). However, SHRIMP studies on zircons (Sánchez-Rodríguez, 1998; Acosta-Vigil, in preparation) have yielded 146 147 both Hercynian and Alpine ages, suggesting the existence of two different anatectic events separated in time. The peridotite overthrusting and HT metamorphism occurred during decompression in a syn-collisional 148 149 tectonic setting (Tubía et al., 1997; Platt et al., 2003). The outcrops consist of stromatic metatexite, nebulitic 150 or deformed diatexites and mylonitic migmatites; the melanocratic portion contains garnet, biotite, 151 sillimanite, graphite, ilmenite, plagioclase and quartz. Leucosomes in metatexites and mylonites consist of quartz, plagioclase, K-feldspar and rare biotite and garnet. Ronda migmatites underwent a partial melting event at 700-800 °C and 0.5-0.8 GPa (Torres-Roldán, 1983; Tubía & Cuevas, 1986; Tubía et al., 1997); the temperature and pressure of metamorphic peak increase from metatexites to mylonitic migmatites. The T peak was followed by rapid cooling (400-200 °C/Ma, Platt et al. 2003; Esteban et al. 2004).

Barun Gneiss (Bordet, 1961) occur at the base of the HHC at the higher structural levels of the Himalayan 156 belt, in Eastern Nepal. This granulitic/migmatitic belt, which extends toward the east in Sikkim and Bhutan 157 158 (e.g. Goscombe et al., 2006 and references therein) underwent high grade metamorphism and partial melting during the Alpine-Himalayan orogeny (28-16 Ma, Viskupic & Hodges, 2001). At the outcrop scale Barun 159 gneisses present a discontinuous planar foliation, defined by mm-thick layers of melanosomes alternating 160 161 with elongated leucosome domains, up to 1 cm in thickness. Two different samples, 07-19 and 07-36, were 162 investigated in this work, both of them with mineralogical and microstructural features very close to those of 163 sample 07-35 studied in detail by Groppo et al. (submitted). The fine-grained melanosome consists of small-164 sized biotite, sillimanite and plagioclase plus quartz, with locally abundant garnet porphyroblasts, ≤ 5 mm 165 across, wrapped by the foliation. Garnet occurs both as subhedral poikiloblasts (sample 07-36) and skeletal porphyroblasts (sample 07-19). Kyanite may also occur as a relic, rimmed by a thick plagioclase corona. 166 167 Leucosome levels consist of quartz, K-feldspar and plagioclase. Based on the petrogenetic modeling, Barun 168 gneisses underwent a clockwise PT path, with nearly isobaric heating up to 800-860°C at 0.8 GPa with partial melting, followed by a near isothermal decompression down to 0.65 GPa, when the crystallization of 169 170 the melt is believed to have taken place, and by a slightly decompressional cooling along a less steep 171 retrograde path (Groppo et al., submitted).

The presence of melt in the investigated rocks may be inferred based on the occurrence of several microstructural evidences. Besides the presence of leucosome domains (Fig. 1c), pseudomorphs of meltfilled pores (Holness and Sawyer, 2008) commonly occur. They mainly consist of quartz and plagioclase that crystallize as grains with a cuspate-lobate shape, mimicking the original geometry of the pore they were confined in, often at the contact between MI-bearing garnet porphyroblasts and leucosome domains (Fig. 1c177 h). In Barun gneisses melt pseudomorphs commonly occur within large polymineralic inclusions of Qtz+Pl+Bt in garnet, described in detail by Groppo et al. (submitted). They usually consist of films and shells 178 of plagioclase surrounding rounded quartz or biotite, with locally biotite growing from inclusion walls 179 toward the inner part of the volume. Similar features suggest those aggregates were originally large pockets 180 of melt, \leq 300 µm wide. However, such a large size is likely to favour the entrapment of crystal phases 181 already present in the rock, and moreover they are never fully enclosed in the garnet. Their composition is 182 183 therefore modally unconstrained, not allowing estimation of the melt bulk chemistry. Further witnesses to the 184 former presence of melt are symplectites (Vernon, 2011), due to garnet retrograde reaction with the melt, e.g. the quartz-cordierite symplectites often observed in khondalites or the biotite+plagioclase symplectites in the 185 186 Barun gneisses.

187 Although some of the microstructures reported above represent reliable indications for the former presence 188 of melt in the investigated rocks, none of them allows for the composition of the anatectic melt to be 189 retrieved. This target was accomplished below, using the novel approach based on MI.

190

3) Methods

192 The study of anatectic MI has been performed on about 50 thin and doubly-polished thick sections. The 193 thickness of the doubly-polished sections is variable and ranges 100-250 µm depending on the samples. In some cases, garnets were separated from crushed thick sections. The characterization of the MI features, 194 195 phase assemblage and crystallization microstructures was carried out using different instrumental devices. Petrographic characterization was carried out with optical microscope both in transmitted and reflected light, 196 197 in order to verify the occurrence of MI also in opaque minerals, i.e. Fe and Ti oxides. Crystallized minerals 198 and chemistry of the components were investigated by secondary electrons (SE) and back-scattered electrons 199 (BSE) imaging, semi-quantitative energy dispersive X-ray spectroscopy (EDS) analyses and X-ray mapping. Data were collected with different scanning electron microscopes (SEM): 1) a CAM SCAN MX2500, 200 201 equipped with LaB6 cathod, at the Department of Geosciences, University of Padova; 2) a Jeol JSM-6500F

202 thermal Field Emission Scanning Electron Microscope (FESEM), available at the High Temperature/High 203 Pressure Laboratory, I.N.G.V. (Istituto Nazionale Geofisica e Vulcanologia), Rome; 3) a FEI Quanta 600 FEG, equipped with a Bruker EDX-Silicon Drifted Detector, available at the Nanoscale Characterization and 204 Fabrication Laboratory, I.C.T.A.S. (Institute for Critical Technology and Applied Science), Virginia Tech, 205 USA; 4) a SEM Zeiss equipped with FEG Gemini and a Silicon Drifted detector at CNR-IENI, Padova. BSE 206 images were acquired at variable magnifications and variable accelerating voltages, commonly 8 to 15 kV 207 depending on the used machine. Elemental X-ray maps were acquired at 20 and 15kV accelerating voltage 208 and at variable magnifications, in the range 5000-6000X, depending on the MI size, with resolution of 209 500X375 pixel. 210

MI re-homogenization was obtained with different techniques. A LINKAM TS1500 high temperature and 211 212 ambient pressure stage, in an inert atmosphere of He to prevent sample oxidation, was used to re-melt 213 anatectic MI in khondalites and Barun gneisses at the Fluid Inclusion Laboratory, Earth Science Department-214 University of Parma, Italy. Inclusions were re-homogenized within doubly polished chips of garnet. A temperature correction factor was applied, after having determined a calibration curve with different 215 216 standards of K₂Cr₂O₇, Ag and Au with melting T respectively of 398°C, 962°C and 1064°C. The accuracy of measurements is ±15°. KKB samples were heated up to 500°C with a rate of 50°C/min, followed by a 2 217 218 hours stop. Then MI were heated at 40°C/min up to the beginning of melting, i.e. when crystallized 219 inclusions under observation showed some modifications of the internal crystal boundaries. The samples 220 were then kept at this T for 15-30 min to allow a complete homogenization, and quenched afterwards in liquid nitrogen to avoid as much as possible glass re-crystallization. Homogenization T values obtained 221 trough this approach is ~1040°C. A different ramp was selected for Barun samples, since the ramp of KKB 222 samples caused diffuse MI decrepitation. After being heated up to ~550°C, the sample was brought to 223 224 ~750°C, with stops of 10 min every 100°C, and up to 830°C (observed melting T) with 5 min stops every 25°C. The heating rate applied during the entire experiment is 50°C/min. Homogenized MI were then 225 selected by optical microscope investigation, and exposed to the surface through a careful polishing process, 226

where possible done by hand on abrasive sheets coated of Al₂O₃ and SiC with variable grain-size, to impose less stress to the sample surface. MI in Ronda migmatites were re-homogenized at high pressure with a piston cylinder apparatus at Laboratorio di Petrologia Sperimentale, Dipartimento di Scienze della Terra, Università di Milano. The experimental conditions were 700 °C, 0.5 GPa and 24 h.

The MI microchemical composition was determined both in glassy inclusions and in homogenized 231 inclusions using two different Jeol JXA 8200 Superprobes at the High Temperature/High Pressure 232 233 Laboratory, I.N.G.V., Rome, and at the Dipartimento di Scienze della Terra, Università di Milano. Used analytical conditions were selected depending on the machine and the case study, always using a 15 kV 234 accelerating voltage and a focused beam with size of 1 µm. Analytical parameters (beam current and 235 236 acquisition time) vary as follow: 1) in Ronda migmatites, 2 nA and 10 s on the peak and 5 on background; 2) 237 in Barun samples 6 nA, 5 s on peak and 2 on the background; 3) in KKB samples 3.5 nA and 10 s on peak 238 and 5 on background.

239 Na is known to migrate out of the electron beam excitation volume, and this may render the EMP data 240 unrepresentative of the analyzed melt. Therefore, according to Morgan & London (2005), correction factors for Na, Si, Al and K were calculated from the ratio between analyzed and known concentrations of the 241 242 correspondent oxides within different glass standards, selected to be as close as possible in composition to 243 each of the analysed sample (in particular as H₂O contents). The used standards are respectively a 10 wt% H₂O bearing glass (LGB 5; Behrens & Jantos, 2001), a 6.6 wt% H₂O-bearing melt (Morgan & London, 244 2005) and a 5.5 wt% H₂O glass from Acosta-Vigil et al. (2003). The Na loss was estimated as 23% relative 245 246 for Ronda migmatites, 8% in Barun samples and none in khondalites. Hand-by-hand selection of analytical points allowed to verify the real position of the analytical beam, avoiding instrumental drift. 247

248

4) Microstructural and chemical characterization of anatectic melt inclusions

250

4.1) Occurrence and optical features

Anatectic MI have been recognized, so far, mainly within garnet in the three investigated cases (Fig. 2a,f), although they locally occur also in ilmenite in metatexites from Ronda (Fig. 3).

MI-bearing garnets have different features and size. In khondalites, garnets form large anhedral 253 porphyroblasts (<2 cm wide) located in the melanocratic portion of the diatexite, often not in touch with 254 leucosome because sheltered by cordierite overgrowths. The garnet in the leucosome are always MI-free. In 255 metatexites from Ronda, garnet crystals are generally subhedral and small (<200 µm across; fig. 2b,e), 256 257 surrounded by leucosome that may form melt pseudomorph microstructures around garnets (Fig. 1e,f). In 258 more foliated samples, e.g. mylonites at the contact with Ronda peridotites, MI-bearing garnets are more rare. The rare MI-bearing ilmenites occur as anhedral crystals (100-200 µm across) in the rock matrix (Fig. 259 260 3). In Barun gneisses MI occur both in poikyloblastic and skeletal garnets, ≤ 1 cm across (Fig. 2c,f).

261 The abundance and the microstructural distribution of MI in garnet vary depending on the case study. In 262 Barun gneisses MI are less abundant than in the other two cases, and show no preferred microstructural 263 position in skeletal garnets, while they mainly occur at the periphery of the crystal in poikiloblastic garnets, 264 where mineral inclusions and polymineralic inclusions are absent. Locally MI are clustered, forming groups of tens of inclusions, often characterized by similar size. Where garnet is relatively mineral inclusion-free, as 265 266 in khondalites and Ronda migmatites, MI may form clusters of tens to hundreds of inclusions, locally with a 267 spherical geometry. Clusters do not have preferential location in anhedral porphyroblasts, such as in KKB samples (Fig. 2a,d) while in subhedral to euhedral hosts, i.e. in Ronda metatexites, they occur at the core 268 (Fig. 2b,e), leaving a more or less narrow MI-free rim (similarly to what reported in garnets from El Hoyazo 269 enclaves, Acosta-Vigil et al., 2007). In ilmenite MI occur both as isolated and grouped inclusions with no 270 preferred microstructural position (Fig. 3). Locally the MI cluster may touch the host boundary, if the crystal 271 has been partially replaced by some retrograde mineral, e.g. biotite on garnet as in Ronda migmatites. No 272 273 proofs of secondary entrapment, such as linear arrays of MI, were observed.

The observed spatial arrangement as cluster is a strong constraint to the primary nature of MI with respect to the host crystal (Roedder, 1984), i.e. they were trapped when garnet was growing. Both in KKB and 276 Ronda samples MI are evenly distributed within the clusters. However, if the average mutual distances 277 among adjacent MI are evaluated, they show a different "degree of packing" with the average varying from 278 $\leq 15 \,\mu$ m in Ronda samples to 80-100 μ m in the khondalites (Fig. 2d,e).

Within clusters, mineral inclusions of size comparable to MI, i.e. $\leq 15 \mu$ m, often occur. They mainly consist of accessory minerals: apatite, rutile, zircon are widespread, while spinel occurs only in khondalites. Depending on the case study also quartz, plagioclase, rarely graphite and fibrolite needles may be present. As already observed in El Hoyazo enclaves by Acosta-Vigil et al. (2007), MI generally do not occur in the surroundings of large mineral inclusions (>50 µm), e.g. in Barun samples the mineral inclusion-rich core is poor in MI with respect to the rim. Similar occurrence is visible in poikiloblastic garnet in khondalites, which coexist at the thin section scale with MI-bearing garnets, completely free of mineral inclusions.

286 Investigated inclusions appear as small dots that might be misinterpreted at a first glance as defects of the 287 polished surface, in the case of very small (<10 µm) inclusions. However a careful microscope investigation 288 shows that they are three-dimensional objects confined within the thin-section (fig. 2d-f). Two different types of inclusions may be distinguished at optical investigation. The first type consists of dark-brownish 289 290 inclusions, containing a polymineralic aggregate as confirmed by the presence of different birefringent 291 crystals at crossed polars (Fig. 4a,b,e,f,i,l). This inclusion type represents the majority of the reported MI in 292 the investigated case studies, e.g. 85% in khondalites (Cesare et al., 2009). The second type of inclusions is 293 partially transparent in transmitted light and isotropic at crossed polars, always with a single birefringent 294 mineral (Fig. 4c,d,g,h). Raman spectroscopy, SEM and EMP investigations (Cesare et al., 2009, 2011) 295 showed that this inclusion type contain glass along with one or more accessory phases. Glassy inclusions in Ronda metatexites are more rare than in KKB samples, and totally absent in Barun samples. 296

The shape is isometric in both inclusion types (Figg. 2;3;4;5) and the average size is \leq 50 µm. As general observation, smallest (as average size) inclusions occur in Ronda garnets, ~5 µm, while they are slightly larger (~15 µm) in khondalites, where the MI size population define a Gaussian distribution (Cesare et al., 2009). MI size in Ronda ilmenites ranges from 1.5 to 20 µm. In Barun gneisses MI range from few µm up to 301 50 μ m, and only the smallest ones, with average size ~8-10 μ m, show a regular isometric shape, while larger MI have an irregular shape and are characterized by the presence of offshoots, containing birefringent 302 303 minerals. The last MI type represents the most part of the MI observed in these samples. Their offshoots are 304 generally many times in length the MI diameter, with irregular distribution around the MI. Locally they may 305 connect two or more inclusions if close to each other (Fig. 4m,n). Similar features, although less common, 306 are also observed in khondalites MI, where some inclusions have one or two "tails" roughly opposite in 307 orientation and shorter in length, usually \leq MI diameter, with respect to Barun Gneiss samples (Fig. 4g). 308 Besides isometric and highly-irregular inclusions, locally tubular inclusions are observed (Fig. 2c,d), more abundant in Barun gneisses. In few cases polycrystalline inclusions are acicular with diameter ~5-6 µm and 309 310 length $\leq 100 \,\mu\text{m}$, where melt adhered to elongated phases, such as rutile needles.

311

312 **4.2) Microstructures and phase assemblage**

The isometric shape, visible under optical microscope observation, corresponds to a more or less developed negative-crystal shape when MI are investigated at the FESEM (Fig. 5; see also Cesare et al., 2011). This shape is generally developed in smallest inclusions, i.e. $\leq 20 \ \mu m$ (Fig. 5a,b,e,h), unless the presence of accessory minerals touching the MI walls (Fig. 5d). Larger inclusions are more irregular, although they commonly have planar boundaries.

Within polycrystalline inclusions, the FESEM investigation shows different phase assemblages in different 318 case studies. Both crystallized inclusions in Barun (Fig. 5b,c;6a) and Ronda garnets (Fig. 5d,e) may contain 319 320 quartz, plagioclase, biotite and muscovite, and in the latter case study also minor K-feldspar may occur, while within ilmenites crystallized inclusions contain guartz, biotite and plagioclase. Various accessory 321 322 minerals are commonly present: in particular, rutile is abundant in mylonitic migmatites at Ronda (Fig. 5d), 323 while zircon is more common in Himalayan samples. In khondalites instead MI do not contain muscovite, and the phase assemblage consists of quartz, K-feldspar and biotite, with minor Na-rich plagioclase (Fig. 324 5a,f). Apatite and spinel are the most common accessories. 325

326 Generally the largest grains within crystallized inclusions consist of subhedral to euhedral grains of micas, \leq 5 µm in size (Fig. 5b,c), and they are likely to be the first phases to have crystallized. Locally muscovite 327 328 grows in continuity with biotite (Fig. 5c;6a). Both phases are often easily distinguishable in SE and BSE images because of the basal cleavage and the irregular surface, due to the polishing process (Fig. 5f). 329 330 Usually K-feldspar and plagioclase form subhedral to anhedral crystals after Bt and/or Ms growth, with size variable from hundreds of nanometers up to few microns, depending on the inclusion, while quartz often 331 332 occurs as interstitial phase. Igneous microstructures, such as granophyric intergrowths, are widespread 333 especially in khondalites and Barun Gneiss samples. In the first case study, quartz may form angular-wedge like shape crystals ("micrographic" shape) crystallizing along with K-feldspar (Fig. 6b). Locally K-feldspar, 334 335 associated with biotite, occurs as worm-like grains embedded in large quartz grains (Fig. 5f). Quartz may 336 crystallize as cuneiform rods along with plagioclase in Barun samples (Fig. 5c). Elongated quartz rods are 337 moreover visible within plagioclase in khondalites (Fig. 11a in Cesare et al., 2011). Pseudomorphs of melt 338 filled pores, reported at the thin section scale, occur also inside the MI, at the tens-of-nanometers scale 339 (Cesare et al. 2009; 2011). The same accessory phases visible along MI in clusters occur also inside the 340 inclusions, often indented into the host mineral (Fig. 4h). Some MI show a diffuse nanoporosity, due to the 341 higher density of the crystallized phases with respect to the original melt. At Ronda, this nanoporosity is 342 filled with H₂O (M.L.Frezzotti, Pers. Comm.), suggesting a hydrous melt as confirmed by microchemical data (see below). 343

Investigated MI show however a variable degree of crystallization, from total (nanogranites) to none (glassy inclusions). Fully glassy inclusions contain an homogeneous glass, and occurs along with the same accessory phases found in nanogranites (Fig. 5h). Their shape is often a negative crystal shape, more developed in khondalites samples with respect to the Ronda ones.

The abundance of partially crystallized inclusions is variable. While they represent the largest portion of MI in Ronda migmatites, in KKB and Barun Gneiss samples most MI consist of nanogranites. Partially crystallized inclusion are indistinguishable from nanogranites at the optical microscope because of the small 351 size: the presence of a residual melt along with crystal phases is revealed by FESEM investigation and elemental mapping. This amorphous phase may occupy different percentages of MI area, i.e. 20 to 60% in 352 353 khondalites. Residual melt is generally characterized by a diffuse nanoporosity, mainly localized at the 354 interface melt/crystal phases (Fig. 5g), and occurs along with a set of mineral phases quite constant in each case study. One or two micas are often present, supporting the inference they are the first phases to 355 crystallize. In detail, in khondalites residual melt occurs along with Bt+Kfs+Qtz (Fig. 5g), while in Barun 356 357 migmatites the melt-coexistent assemblage is Bt+Ms+Pl+Qtz. In Ronda samples partially crystallized 358 inclusions generally contain Ms+Bt+Qtz along with residual melt, although, more rarely, Na-rich plagioclase may be the only crystal phase present along with residual melt. The melt presence has been also confirmed 359 360 by the EMP analyses and the elemental maps collected both on nanogranites and on partially crystallized MI. 361 If compared, elemental distributions are different between the two different inclusion types. In Khondalites 362 Na, Ca and Cl are mainly partitioned in the residual melt (Fig. 7), while in melt absence, i.e. within 363 nanogranites, the same elements show the highest concentration respectively in plagioclase, apatite and 364 biotite. Similarly, in Barun samples the residual melt is characterized by the highest concentration of Na and Ca, and minor amount of K, preferentially partitioned in micas. Often the residual melt shows cuspate-lobate 365 366 geometries, like films surrounding some minerals (Fig. 7), resembling the pseudomorphs of melt-filled pores 367 observed at larger scale in the investigated rocks. These microstructures may be actually regarded as the natural precursors of the melt pseudomorphs, since they still contain glass instead of crystallized phases. 368

Irregularly shaped MI in Barun gneisses present offshoots filled with biotite and quartz (Fig. 8b), while in Khondalites only biotite crystallize in them (Figg. 5g,h). Generally their length is \leq MI radius in KKB samples, and >> MI radius in Barun Gneiss samples. In case of developed negative crystal shape, offshoots usually occur at the corners of the inclusions outward, locally defining a radial arrangement (Fig. 8a). Along with negative crystal shaped and irregular inclusions, in Barun samples necking down phenomena are visible, such as fully crystallized inclusions with hourglass shape, interpreted as a former elongate inclusion that underwent an incomplete separation in two different volumes of melt, now both crystallized (Fig. 8b).

4.3) Microthermometry on nanogranites

The large variability in microstructures and grain size within nanogranites, coupled with their small diameters, does not allow a reliable estimate of their modal composition based on image analysis. Only glassy inclusions may be analyzed directly by EMP means, but they are rare (or absent, e.g. in Barun samples), and with size often below analytical limits. Re-heating experiments have been then performed to re-melt nanogranites, restoring their original homogeneity and allowing to perform EMP analyses on the enclosed melt (Lowenstern, 2003).

After re-melting experiments it has been observed that only MI in Ronda migmatites, reheated at high 384 385 pressure by using a piston cylinder apparatus, reached a complete, melt + vapor homogenization (Fig. 9a), 386 commonly observed in re-homogenization experiments performed on igneous MI. Experiments on KKB and 387 Barun samples were instead performed at ambient P conditions with high-temperature stage, and the re-388 melted nanogranites always contain one or more bubbles along with a homogeneous melt (Fig. 9b). After 389 experimental runs, the MI population in re-heated samples consists of fully re-homogenized inclusions and 390 partially re-melted inclusions, containing still relics of the original phase assemblage and characterized by the 391 occurrence of decrepitation cracks. Generally larger nanogranites in each samples did not undergo 392 homogenization, and after re-heating they appear darker and often with microcracks.

Our investigation focused on the first type of inclusions, after a careful selection performed via optical and 393 394 electron microscope to verify the homogeneity of the melt under analysis. Homogenized inclusions maintain 395 both the isometric shape, locally also negative-crystal, and solid inclusions observed in original nanogranites, i.e. accessory minerals. In samples re-heated with heating stage, $\leq 10 \mu m$ long cracks, filled with melt, are 396 often visible also in fully homogenized inclusions. In khondalites and Barun gneisses these cracks are often 397 398 dark at optical observation, due to the precipitation of cubic Fe-oxides grains. The same mineral phase may 399 occur also in limited embayments on the MI walls. Locally, a crystal phase similar in composition to a high-400 T orthopyroxene (e.g. Sato, 2004) occur in the KKB samples, likely to be considered a product of garnet-melt 401 interaction due to overheating during HT experiments. Decrepitated inclusions commonly show multiple 402 bubbles because of the major vesciculation induced by MI depressurization after microcracks nucleation 403 (Lowenstern,1995). Microchemical data were collected on selected inclusions, avoiding those with 404 embayments and significant amounts of Fe-oxides crystallized within decrepitation cracks.

405

406 **4.4**) Microchemical characterization

407 Mean EMP data (Table 1) show that homogenized inclusions contain a leucogranitic melt, with $SiO_2 \ge 68$ 408 wt%, Al₂O₃ ~12-13 wt%, variable K₂O and Na₂O, FeO ≤ 3 wt% and CaO, MgO < 1 wt%. Fluids content was 409 estimated by EMP difference, and it is variable depending on the case study. The enclosed melt is always 410 peraluminous, as testified by the ASI index ≥ 1.15 . The average maficity of the melt (total iron and 411 magnesium contents expressed as atomic Fe+Mg) is generally low, and increases from 0.026 in Ronda 412 samples to 0.056 in khondalites.

413 In Ronda migmatites, where MI were homogenized at T= 700°C via piston cylinder, the enclosed melt shows the lowest SiO₂ and the highest volatiles contents (respectively ~ 67 wt% and ~ 8 wt%) among the 414 investigated inclusions. K₂O is only slightly higher than Na₂O (4 wt% vs. 2.5 wt%), even after Na correction 415 416 (see "methods" paragraph). Re-homogenized MI have a bulk composition very similar to the preserved 417 glassy inclusions in not re-heated samples. Nanogranites from Barun gneisses, re-homogenized via heating stage at ~830°C, have higher SiO₂, ~74 wt%, and a lower fluid content, ~2.5 wt% and K₂O > Na₂O, 5 wt% 418 419 vs. 2 wt%. In khondalites the enclosed melt has similar SiO_2 , while fluid content is lower, <1 wt%, and $K_2O >> Na_2O$, 7 vs. 1 wt%. In the latter case the average composition is comparable to those of the preserved 420 glassy inclusions in terms of alkalis contents, confirming the ultrapotassic nature of the trapped melt (Cesare 421 et al., 2009), and fluid content (or EMP totals), while SiO₂ and FeO are respectively lower and higher with 422 423 respect to glassy inclusions.

According to CIPW normative values, the analyzed melts have similar Qz (~40), while Ab and Or are variable, with Or from similar to much higher than Ab, depending on the protolith; An is always low, ≤ 4 and 426 every analyzed melt is corundum-normative. When plotted in a Ab-Qz-Or ternary (Fig. 10) data from each case study define a single cluster, which include the correspondent glassy MI compositions in the case of 427 428 Ronda migmatites and khondalites. MI compositions plot always above the cotectic curve, in the Qz-bearing field composition, at variable distance from the "minimum melt" composition expected to represent the 429 lowest T composition (at P= 0.5 GPa and a_{H2O} = 0.5; Johannes & Holtz, 1996) of a haplogranite melt (see fig. 430 10). Among the reported data, Ronda melt compositions are the closest to the "minimum", although with 431 432 higher Qz component. Barun melts are characterized by the highest normative An value among the 433 considered datasets. Their compositions plot in an intermediate position among Ronda migmatites and khondalites. The latter case study show instead the melt with the highest mean Or, ~4-5 times the Ab value, 434 which plot close to the Qz-Or join, very far from the "minimum melt" composition. 435

436

437 5) Discussion

5.1) Occurrence

The microstructural study confirmed that investigated anatectic MI are primary, i.e. they were trapped 439 440 during peritectic mineral growth, as testified by their zonal arrangement (Sobolev & Kostyuk, 1975). 441 Anatectic MI have been recognized in garnet and, less commonly, in ilmenite. Both minerals have a large 442 stability field, and are generally more resistant to deformation and microcracking (because of hardness and 443 absence of cleavages) with respect to another common MI-host phase such as quartz, and MI enclosed in 444 them are therefore likely to be preserved during the post-peak trajectory. Among the peritectic phases in the 445 metapelitic system, also spinel may be considered as a reliable host for anatectic MI. Glassy MI containing anatectic melt were reported to occur also in andalusite within crustal xenoliths from Mazarrón, NVP (Cesare 446 et al., 2003) and from Crd-bearing rhyolites of Lipari (Cesare et al., 2011; Di Martino et al., 2011), providing 447 448 further constraints to the Al_2SiO_5 triple point. Also a mineral phase such as sillimanite, although commonly a 449 reactant in partial melting reactions in metapelitic migmatites, if in excess during partial melting reactions may re-crystallize as prismatic crystals possibly entrapping the anatectic melt, as locally visible in 450

451 khondalites (E. Salvioli-Mariani, Pers. Comm.). In El Hoyazo enclaves also plagioclase and biotite contain 452 MI, besides garnet and spinel, because both peritectic products of a rapid, disequilibrium melting reaction 453 involving a low grade metapelitic protolith (Cesare and Maineri, 1999). This occurrence suggests that also 454 those phases, commonly regarded as reactants under partial melting conditions, may trap melt in peculiar 455 geological settings characterized by high heating rates, and should be therefore targeted during investigation 456 for anatectic MI.

457 Mineral inclusions of size comparable to MI, mainly accessories (Ap, Rt, Zr), often visible in the investigated inclusions, show features, e.g. large size and mineralogical variability, which rule out the 458 possibility they are daughter phases. These mineral phases are often indented in MI walls, suggesting they 459 460 were first trapped in the growing host mineral, and then acted as clinging surfaces for the melt, similarly to what happens in igneous rocks (Roedder, 1984). Analogous occurrence was reported in migmatites from the 461 462 Western Adirondacks Highlands by Henriquez and Darling (2009), where crystallized inclusions in peritectic 463 garnet, interpreted as nanogranites, always contain or cling to a zircon grain. This occurrence suggests that a fine grain-sized protolith containing abundant "inert" accessory phases (graphite, rutile, apatite, zircon, 464 465 ilmenite) is more likely to favour the MI formation during anatexis.

466

467 **5.2) Post entrapment changes**

468 Shape maturation

The negative-crystal shape typically found in anatectic MI is not a primary feature, as confirmed by experiments on fluid inclusion formation in different minerals and artificial crystalline compounds (Maze et al., 1981; Sisson et al., 1993). At HT conditions, such a shape is likely to develop trough the host redistribution in a brief span of time, e.g. in less than one year for MI in quartz (e.g. Clocchiatti, 1975; Manley, 1986). This "shape maturation" is commonly observed both in experimental and in natural MIbearing rocks (Skirius et al., 1990; Lowenstern, 1994) and it represents a process trough which the surface free energy is lowered by modifying the inclusion morphology (Roedder, 1984; Lowenstern, 1995). The 476 lowest energy configuration corresponds to a negative-crystal for a melt inclusions, whose shape varies 477 depending on the host crystalline structure, e.g. in garnet-hosted inclusions this means an isometric shape 478 with rombododecahedral geometry. In MI under investigation shape maturation is likely to predate the 479 beginning of melt crystallization, since mineral phases in them often grow on planar walls.

Another example of "shape maturation" is the necking down of former elongate MI, already reported in El Hoyazo enclaves (Acosta-Vigil et al. 2007) and locally observed in Barun samples. The same process may be regarded as the responsible for the occurrence of MI with a well developed negative-crystal shape and a very short mutual distances ($\leq 1 \mu m$), both in Barun and Ronda samples (see e.g. fig. 14b in Cesare et al., 2011).

484

485 Decrepitation

In Barun samples, the most part of MI presents offshoots. Similar microstructures were reported by Stöckhert et al. (2009) in UHP inclusions in garnet of the quartzofeldspathic rocks of Saxonian Erzgebirge, Germany, and interpreted as microcracks produced by MI decrepitation, afterwards in-filled with crystal phases. The presence of offshoots with radial arrangements, often observed in decrepitated FI, support this hypothesis. Moreover, results of re-equilibration studies (see e.g. Bodnar, 2003) show how larger FI are more likely to undergo microcracking, and this is confirmed in Barun case where only smallest MI are preserved.

492 Decrepitated inclusions are extremely abundant in Barun samples, and offshoots also occur in khondalites. The two case studies differ as retrograde P-T-t trajectory, counterclockwise in khondalites and clockwise in 493 494 Barun gneisses. In the first case a T decrease precedes the drop of the confining pressure, and this largely 495 lowered the MI internal pressure (because of the steep slope of the MI isochore, Lowenstern, 1994), limiting therefore the magnitude of the differential pressure experienced by the MI, and subsequently the extent of 496 decrepitation phenomena. On the contrary, in the second case study depressurization took place after the 497 498 partial melting event, likely with no appreciable T (and subsequent internal P) decrease, and caused therefore 499 decrepitation of a larger amount of MI.

A further difference is related to the common offshoots length, minor in khondalites with respect to Barun samples. In the first case the observed length, < MI radius, corresponds to what expected when MI decrepitate because of the building up of the differential pressure (Tait, 1992). Conversely, in Barun MI the major length of the offshoots, often >> MI radius, may suggest that, besides overpressurization, MI decrepitation was enhanced also by some other factors, most likely a deformational phase, during the retrograde path, as also suggested by the abundance of microcracks in MI-bearing garnets.

Since preserved and decrepitated inclusions have the same phase assemblage, it may be inferred that there were no external fluid infiltrations neither in Barun nor in KKB samples, although decrepitation likely caused the leakage of (at least) part of the fluids from the inclusion. In khondalites, the occurrence of biotite, probably the first mineral phase to precipitate, within the offshoots suggest that decrepitation took place before the beginning of the melt crystallization. It is also possible that decrepitation triggered crystallization, as often recognized in acqueous solutions-bearing FI, where pressure drop causes a solubility drop that leads to daughter phases precipitation (Manning, 2004).

513

514 **5.3**) Re-melting the nanogranites

MI-bearing sample from Barun gneisses and khondalites were re-heated under continuous optical observation, until the crystalline assemblage melted completely. The melting T is interpreted as the minimum T of crystallization of the melt, and is generally assumed as to its trapping temperature (Frezzotti, 2001). Melting temperatures obtained in this work are consistent in the case of Barun gneisses, for which an anatectic event at 800-860°C at 0.8 GPa was proposed (Groppo et al., submitted), while for Khondalites homogenization was reached only at T>1000°C, a temperature not consistent with those reported in literature, ~900 °C (Shabeer et al., 2005).

In case of Ronda migmatites, homogenization was obtained through piston cylinder method, and the application of a confining pressure reduced MI decrepitation (and subsequent volatile loss) and Fe oxides formation. Generally the piston cylinder approach gives better results in term of percentage of fully rehomogenized and not decrepitated MI in the clusters. The abundance of preserved MI in the investigated samples is the basic requirement for the choice of this method, although the use of the heating stage remains useful at the preliminary phase of the investigation, since it represents a less time-consuming method to verify if crystallized inclusions may be actually re-melted. In case of less favorable sample the heating stage may however represent the only feasible approach, e.g. for Barun gneisses case study, given the scarcity of preserved inclusions.

531 FESEM investigation showed that, after homogenization, re-melted MI mainly show linear boundaries and, locally, the negative-crystal shapes already observed in nanogranites. This fact suggests that within 532 investigated anatectic MI, or at least a part of them, no host dissolution took place on re-heating. This a 533 534 remarkable difference with respect to what observed in MI study from igneous rocks, where host re-melting 535 is actually expected, in order to bring back the melt to its original composition. In fact, it is normally 536 observed that, during cooling, host crystallizes on MI walls, with variable amounts depending on the case study (e.g. Frezzotti, 2001). The lack of host-crystallization in anatectic MI is easier to be verified when they 537 538 occur in host minerals which do not belong to the phase assemblage expected for a nanogranite, e.g. garnet. 539 Vice-versa, in case of MI trapped in peritectic plagioclase or K-feldspar, both phases are likely to be also 540 crystallization products of the enclosed anatectic melt, and the resultant overgrowth on MI walls may result 541 hardly distinguishable from the original host, mainly because of the investigation scale.

542

543 **5.4**) Compositions of the anatectic melt

The bulk composition obtained by EMP analyses is peraluminous and leucogranitic, with variable alkali contents depending on the case study. In the Ab-Qz-Or ternary diagram the collected datasets define three different clusters, whose positions is always in the Qz-bearing field, at variable distance from the "minimum melt" composition (Johannes & Holtz, 1996).

548 From a general point of view, experimental data on haplogranitic system and natural rocks represent a 549 valid frame to handle the compositional datasets made available by the investigation of anatectic MI. For 550 example, when the different melts are compared, the average normative Ab, decreasing from ~25 to 10 in 551 Ronda and KKB melts respectively, is negatively correlated with the increasing temperature of formation, in 552 agreement with higher melting temperatures reported in literature for Na-poor haplogranite melts (e.g. 553 Johannes & Holtz, 1996). Also volatile contents are consistent with the inferred conditions of partial melting, in fact MI from Ronda, likely to have been trapped at ~700 °C (Tajcmanova et al., 2011) show the highest 554 amount of fluid (~8 wt%), while KKB melts, generated at T>900, are almost dry. In Barun and KKB 555 556 samples, however, the inferred fluid contents are likely to represent minimum values, because of the 557 widespread evidence of decrepitation and vesciculation among re-melted MI after heating runs.

The average FeO and Na₂O of KKB inclusions is similar to the values reported in literature for peraluminous granites (Droop et al., 2003) produced by partial melting of natural rocks at 900°C and 0.5 GPa, suggesting therefore that the relatively high FeO and low Na₂O contents can be a primary feature of the anatectic melt. That being so, both the objections raised by Clemens (2009) whether or not the trapped phase was anatectic melt and on the quality of the geochemical data in Cesare et al. (2009), in particular about the applied Na-loss correction, can be considered definitely untenable.

The maficity index slightly increases with the melting temperature and is always low and comparable with a pristine S-type leucogranite compositions, e.g. subvolcanic bodies in the Cape Granite Suite reported by Stevens et al. (2007). Investigated melts should be therefore considered as examples of the "starting" magma which, migrating upward and incorporating variable amounts of peritectic phases (Stevens et al., 2007), will be emplaced at shallower crustal levels forming S-type granitic bodies with large compositional variability (Villaros et al., 2009a;b).

An important point that needs to be addressed is whether or not the trapped melt is representative of the bulk anatectic melt in the investigated rocks. Acosta-Vigil et al., (2007, 2010) reported the occurrence of disequilibrium phenomena in anatectic MI from El Hoyazo enclaves, but also showed how disequilibrium affects only the concentrations of the compatible trace elements and not those of major and incompatible trace elements. In the present study, microchemical data on major elements in anatectic MI are therefore 575 likely to be considered as representative of the bulk composition of the original anatectic melt, also in terms 576 of Na content, after the application of correction factors (if needed) calculated using the appropriate glass 577 standards (see "methods").

578 Re-melted MI compositions are comparable with the correlated glassy MI (Fig. 10; Table 1), supporting the hypothesis that clusters contain inclusions of the same melt, despite the different degree in crystallization 579 revealed by the microstructural investigation. This evidence, coupled with their texturally primary nature, 580 581 rules out the possibility, proposed by Clemens (2009), that glassy inclusions may represent later-infiltrated silica-rich fluids with respect to nanogranites. Moreover, silica-rich fluids may occur in UHP terrains (e.g. 582 Stöckhert et al., 2009; Malaspina et al., 2006) but they commonly have a phase assemblage characterized by 583 584 hydrous mineral phases, i.e. phyllosilicates and amphiboles, very different with respect to the nanogranite 585 assemblage. Moreover, the fluid content in investigated anatectic MI is 2 -10 wt%, notably lower than the 586 fluid in common silica-rich inclusions, e.g. 25-50 wt% (Frezzotti et al., 2007).

587 Since the enclosed melt composition is comparable, the different behavior on crystallization is unexpected. 588 The most reliable hypothesis involves a pore size effect on the phase nucleation in the melt, based on a 589 statistical study of MI size in khondalites (8 and 13 µm for preserved glassy and nanogranite inclusions 590 respectively, Cesare et al., 2009), suggesting that phase solubility (or the threshold supersaturation) is higher 591 in smaller pores (Putnis et al., 1995). This is related to the highest interfacial energy of the crystals in smaller pores with respect to larger ones, which promotes and accelerate crystal dissolution in melt volumes with 592 593 diameter $\leq 10 \text{ }\mu\text{m}$ (Immanuel et al., 2009). The occurrence of glassy MI in Ronda migmatites with size often equal to, or larger than, nanogranites is however puzzling and cannot be explained only with the pore size 594 595 effect, but rather with the heterogeneous distribution of nucleation sites in inclusions with a threshold diameter (Cesare et al., 2011). 596

599 Melt inclusions occur in garnet, and locally also in ilmenite, within partially-melted metasedimentary rocks from Ronda migmatites, Barun gneisses and KKB. Data here presented along with previous works 600 601 (Cesare et al., 2009; 2011), support the hypothesis that investigated inclusions formed during peritectic phase 602 growth and contain the melt generated by crustal melting. The microstructural study revealed the occurrence of a leucogranitic phase assemblage formed by quartz, feldspar(s), biotite and/or muscovite \pm accessories 603 within completely crystallized MI (nanogranites). A peculiar feature is represented by the variable degree of 604 605 crystallization within the same cluster, where nanogranites coexist with partially crystallized and glassy 606 inclusions. Normally MI show post-entrapment changes such as shape maturation, resulting in a more or less developed negative crystal shape, and necking down phenomena, which however did not modify the original 607 608 melt composition. No external fluid infiltration is visible in the decrepitated inclusions, which show the same 609 mineralogical assemblage of preserved nanogranites.

610 Microchemical data on re-melted anatectic MI are considered representative of the enclosed melt, since the Na-loss was corrected by using standard glasses of compositions similar to the analyzed melts. The resultant 611 612 composition is generally peraluminous and leucogranitic, as suggested by experimental melting of metapelites and metagraywackes. The similarity of melts in different inclusions (in the same case study) rules 613 614 out the presence of phenomena such as boundary layer effects and local disequilibrium, implying therefore 615 that the investigated melt can be considered as representative of the original anatectic melt. Compositional data give further support to the hypothesis that MI with different crystallization degrees contain the same 616 617 melt, and subsequently glassy inclusions did not form after the nanogranites (as suggested by Clemens, 2009) 618 but at the same time. The microstructural investigation showed that melt entrapment, with subsequent MI formation, is more probable in fine grained protoliths, as suggested by the abundance of small-size trapped 619 620 minerals. So far inclusions have been recognized to occur in minerals more resistant to deformation, e.g. 621 garnet and ilmenite, and because of the similar features also spinel may be regarded as a suitable host for anatectic MI. 622

MI may be also regarded as valuable microstructural evidence for the presence of melt, especially because, being sheltered by the host phase, they are likely to survive retrograde modifications re-crystallization and also further metamorphic cycles, which commonly obliterate other melt witnessing microstructure.

626 Anatectic melt inclusions represents a powerful approach to the characterization of the original melt 627 produced by partial melting within natural samples, before it undergoes modifications commonly observed in leucosomes. Furthermore, they allow the comparison between melt in natural rocks and experimental 628 629 datasets, already available on natural samples and synthetic compositions. The study of anatectic MI poses 630 severe technical problems, and requires the use of very high-resolution techniques to obtain valuable microstructural and microchemical data. However, now available technologies allow us to face and overcome 631 632 these challenges, moving further our comprehension of the partial melting processes in metapelites and 633 metagraywackes, and subsequently the evolution of an important portion of the continental crust during orogenic cycles. 634

635

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878 **Figure captions**

Figure 1: Field aspect of the investigated rocks and microstructural evidences of the former presence of 879 880 melt in the MI-bearing investigated rocks. (a) Coarse-to-medium grain sized khondalites outcropping at 881 Koliakkode quarry, KKB. *Camera cap* = 8 cm; (b) Fine-grained metatexites at Ronda. *Pen length* = 8 cm; (c) Thin section aspect of khondalites, plane-polarized light. On the left part of the image, Qtz+Kfs bearing 882 leucosome is visible. Red box: location of MI cluster; (d) Quartz crystallized as a melt pseudomorph in 883 884 khondalites (white arrow), at crossed polars; (e) Subhedral garnet from Ronda metatexites surrounded by 885 plagioclase crystallized as a melt pseudomorph (white arrow) at crossed polars; (f) Same image of (e) at plane-polarized light, MI occur at the core of the garnet; (g) Detail of a skeletal garnet from Barun gneisses at 886 887 crossed polars, with plagioclase forming a melt pseudomorph (white arrow); (h) Same image of (g) at 888 crossed polars.

889

Figure 2: Examples of garnet hosts, with MI cluster positions (*red boxes*), in plane-polarized light. (a) Large anhedral porphyroblasts in Bt-rich melanosome in khondalites; (b) Subhedral garnet porphyroblasts in Ronda metatexites; (c) Skeletal garnet porphyroblasts in Barun gneisses; d) Close-up of MI cluster in khondalites; (e) Close-up of MI cluster in Ronda samples; (f) Close-up of MI occurrence in Barun gneisses. *White arrows:* two different clusters of MI characterized by different average size.

895

Figure 3: Ilmenite grain containing primary MI with azonal arrangement, SEM image.

897

Figure 4: Photomicrographs of anatectic melt inclusions. (a) and (b) Parallel and crossed polars images of a crystallized inclusion in khondalites; (c) and (d) Parallel and crossed polars images of crystallized and glassy MI coexisting in the same cluster; the single birefringent mineral within the glassy MI is an accessory mineral, most likely an apatite; (e) and (f) Parallel and crossed polars images of a crystallized inclusion with large birefringent phases in Barun gneisses; (g) Crystallized inclusions with symmetrically developed 903 offshoots in khondalites, plane-polarized light; (h) Glassy inclusion in khondalites with a rutile needle 904 indented in the host garnet, plane-polarized light; (i) and l) Parallel and crossed polars images of a 905 crystallized inclusion in Ronda metatexites, (m) and (n) Parallel and crossed polars images of two 906 crystallized inclusions in Barun gneisses, connected by an offshoots filled with birefringent minerals (*white* 907 *arrow*). In lower portion of the picture a preserved crystallized inclusion is visible.

908

909 Figure 5: BSE images of anatectic melt inclusions. (a) Nanogranite in khondalites, with a well developed 910 negative crystal shape; (b) Nanogranite in Barun gneisses with a perfectly developed negative crystal shape; (c) Nanogranite with offshoots from Barun gneisses, with cuneiform-shaped quartz intergrown with 911 plagioclase; (d) Crystallized and partially crystallized MI with large rutile needles, likely to have favored the 912 913 melt entrapment, in Ronda mylonitic migmatites; (e) Typical occurrence of nanogranite in Ronda 914 metatexites; (f) large nanogranite with offshoot in khondalites. Red box: K-feldspar elongated grains in quartz; (g) Partially crystallized MI in khondalites. Red dashed line: interface residual melt-crystallized 915 phases; (h) Typical occurrence of glassy MI in khondalites, characterized by a perfectly developed negative 916 917 crystal shape, with a trapped mineral.

918

Figure 6: X-rays elemental maps on anatectic MI. (a) Large nanogranite from Barun gneisses, the same of fig. 3e, with the typical phase assemblage, and a trapped apatite grain; (b) Nanogranite in khondalites with a micrographic intergrowth of quartz + Kfeldspar.

922

Figure 7: X-rays map of a partially crystallized MI in khondalites, with Na, Ca and Cl showing the same
distribution. *Red arrow:* cuspate-lobate shape of the residual melt around a biotite grain.

925

Figure 8: (a) Nanogranite from Barun gneisses, characterized by offshoots with radial arrangement typical
of decrepitated inclusions. *White arrow:* offshoots with crystallized phases within; (b) Hourglass MI which

underwent a partial necking down in Barun gneisses. *Black arrow:* point of development of the bottle neckthat precedes the complete separation.

930

Figure 9: SEM images of re-homogenized nanogranites. (a) Fully homogenized nanogranite in garnet from Ronda migmatites; (b) Homogenized nanogranite from Barun gneisses, characterized by multiple shrinkage bubbles (*white arrows*).

934

Figure 10: Mean compositions of anatectic MI reported in a CIPW Ab-Qz-Or diagram (see legend). Cotectic curves and minimum melting points for 0.5, 1 GPa are from Johannes & Holtz (1996) for a haplogranite composition at a_{H2O} = 0.5 are reported for comparison. *Black symbols:* re-homogenized nanogranites; *gray symbols:* preserved glassy inclusions, where present. *Dashed/dotted lines:* distributions of the retrieved compositions for each case study.

940

941 **Table 1:** EMP analyses of homogenized and preserved glassy MI (where present), reported for 942 comparison. All Fe considered as Fe^{2+} .





Fig.2



Fig.3



Fig.4











Fig.7



INGV

Fig. 9

COMPO 10.0 KV X8000 1 µm WD 8.1mm



					Barun gneisses										
		На	omogen	ized M	Τ		Gle	assy in	clusion	S					
	1	1 2 3 4 5 Aver.		13 1		6	Aver.	5_1	5_7	9_1	9_2	Aver.			
SiO ₂	68,62	67,23	67,67	70,21	70,18	68,78	68,42	71,70	68,95	69,69	73,37	71,25	73,57	76,90	73,77
TiO ₂	0,26	0,00	0,00	0,15	0,01	0,08	0,00	0,00	0,24	0,08	0,21	0,02	0,07	0,08	0,09
Al_2O_3	11,39	11,07	11,28	11,31	12,03	11,42	11,57	12,16	11,63	11,78	12,97	14,85	12,40	11,38	12,90
FeO	1,82	1,86	1,55	1,45	1,27	1,59	1,26	1,27	1,07	1,2	2,70	2,93	2,25	2,14	2,51
MnO	0,12	0,18	0,21	0,11	0,06	0,14	0,19	0,03	0,04	0,09	0,31	0,48	0,11	0,12	0,25
MgO	0,18	0,07	0,26	0,01	0,07	0,12	0,06	0,05	0,10	0,07	0,67	0,63	0,41	0,42	0,53
CaO	0,52	0,53	0,42	0,29	0,45	0,44	0,31	0,25	0,60	0,39	0,79	1,51	0,73	0,38	0,85
Na ₂ O	2,58	2,42	2,56	3,02	3,14	2,74	3,37	2,96	2,93	3,09	1,89	1,86	2,12	1,87	1,94
K ₂ O	4,18	4,31	3,72	3,75	4,05	4,00	4,05	4,07	4,45	4,19	4,47	5,71	3,97	5,30	4,86
P_2O_5	0,11	0,76	0,63	0,00	0,22	0,35	0,04	0,00	0,49	0,18	0,04	0,04	0,00	0,00	0,02
Cl	n.a.	n.a.	n.a.	n.a.	n.a.	n.a	n.a.	n.a.	n.a.	n.a	n.a.	n.a.	n.a.	n.a.	n.a
Total	89,78	88,45	88,30	90,28	91,48	89,66	89,27	92,48	90,50	90,76	97,42	99,27	95,64	98,59	97,73
Fluids by diff.	10,22	11,55	11,70	9,72	8,52	10,34	10,73	7,52	9,50	9,24	2,58	0,73	4,36	1,41	2,27
ASI	1,17	1,15	1,25	1,18	1,16	1,18	1,10	1,25	1,08	1,14	1,38	1,24	1,36	1,20	1,29
Fe+Mg*	0,028	0,030	0,031	0,020	0,020	0,026	0,022	0,019	0,015	0,019	0,056	0,063	0,042	0,041	0,050
CIPW norn	native min	erals													
Cor	2	3	2	2	3	3	1	2	2	2	4	3	3	2	3
Qz	35	37	36	35	36	36	31	37	34	34	40	32	42	43	39
Ab	20	22	26	27	23	23	28	25	25	26	16	16	18	16	16
Or	25	22	22	24	24	24	24	24	26	25	26	34	23	31	29
An	0	0	1	1	0	0	1	1	0	1	4	7	4	2	4

 Table 1 (continua)

					Glassy inclusions											
	12_1	12_8	10_7	9_1	9_2	7_5	7_6	6_1	6_3	4_5	4_11	Aver.	246	8_1	8_3	Aver.
SiO ₂	70,87	74,11	69,24	77,02	76,28	75,84	76,81	75,92	72,93	72,08	72,42	73,96	77,27	77,91	77,98	77,72
TiO ₂	0,00	0,27	0,18	0,00	0,10	0,02	0,11	0,20	0,11	0,18	0,00	0,11	0,00	0,13	0,00	0,04
Al_2O_3	14,45	13,86	14,90	12,62	11,11	11,93	11,32	11,71	14,12	12,99	13,41	12,95	11,96	11,90	11,84	11,90
FeO	3,18	2,94	4,13	2,73	3,12	2,70	2,16	2,38	2,47	3,14	4,39	3,03	0,91	0,97	1,15	1,01
MnO	0,06	0,07	0,07	0,01	0,08	0,00	0,06	0,00	0,00	0,00	0,06	0,04	0,00	0,00	0,11	0,04
MgO	0,72	0,42	0,90	0,67	0,40	0,43	0,62	0,99	0,91	0,66	0,45	0,65	0,02	0,02	0,00	0,01
CaO	0,48	0,70	0,88	0,55	0,54	0,11	0,33	0,57	0,54	0,51	0,63	0,53	0,03	0,00	0,06	0,03
Na ₂ O	1,41	1,02	1,24	0,51	0,70	0,80	1,30	1,07	1,24	1,48	1,38	1,10	1,02	0,98	0,91	0,97
K ₂ O	8,19	7,04	6,24	6,66	6,36	7,14	6,24	5,83	7,60	6,23	6,43	6,72	7,56	7,27	7,96	7,60
P_2O_5	0,04	0,10	0,00	0,02	0,02	0,17	0,00	0,00	0,00	0,02	0,02	0,03	n.a.	0,12	0,19	0,15
Cl	0,36	0,21	0,34	0,08	0,14	0,09	0,18	0,20	0,37	0,44	0,33	0,25	n.a.	n.a.	n.a.	n.a
Total	99,76	100,74	98,12	100,86	98,85	99,23	99,12	98,88	100,29	97,73	99,53	99,37	98,77	99,29	100,21	99,47
Fluids by diff.	0,24	-0,74	1,88	-0,86	1,16	0,77	0,88	1,12	-0,29	2,27	0,47	0,63	1,23	0,71	-0,20	0,53
ASI	1,20	1,31	1,43	1,39	1,23	1,29	1,19	1,29	1,26	1,29	1,29	1,29	1,21	1,26	1,16	1,21
Fe+Mg*	0,063	0,049	0,078	0,055	0,053	0,048	0,045	0,055	0,056	0,058	0,073	0,056	0,013	0,012	0,018	0,014
CIPW norm	native mine	rals														
Cor	2	4	4	4	2	3	2	3	3	3	3	3	2	3	2	2
Qz	31	42	40	50	50	46	47	50	37	42	41	37	42	46	46	42
Ab	14	10	13	5	7	7	12	11	12	15	14	9	9	7	9	8
Or	56	48	47	45	43	47	41	40	51	44	45	40	45	47	45	45
An	2	3	4	3	3	0	2	3	3	2	3	2	0	0	0	0

Khondalites

	Ronda migmatites								Barun gneisses						Khondalites																
		Homogenized MI Glassy inclusion					\$	Homogenized MI						Homogenized MI										Glassy inclusions							
	1	2	3	4	5	Aver.	13	1	6	Aver.	5_1	5_7	9_1	9_2	Aver.	12_1	12_8	10_7	9_1	9_2	7_5	7_6	6_1	6_3	4_5	4_11	Aver.	246	8_1	8_3	Aver.
SiO_2	68,62	67,23	67,67	70,21	70,18	68,78	68,42	71,70	68,95	69,69	73,37	71,25	73,57	76,90	73,77	70,87	74,11	69,24	77,02	76,28	75,84	76,81	75,92	72,93	72,08	72,42	73,96	77,27	77,91	77,98	77,72
TiO ₂	0,26	0,00	0,00	0,15	0,01	0,08	0,00	0,00	0,24	0,08	0,21	0,02	0,07	0,08	0,09	0,00	0,27	0,18	0,00	0,10	0,02	0,11	0,20	0,11	0,18	0,00	0,11	0,00	0,13	0,00	0,04
Al_2O_3	11,39	11,07	11,28	11,31	12,03	11,42	11,57	12,16	11,63	11,78	12,97	14,85	12,40	11,38	12,90	14,45	13,86	14,90	12,62	11,11	11,93	11,32	11,71	14,12	12,99	13,41	12,95	11,96	11,90	11,84	11,90
FeO	1,82	1,86	1,55	1,45	1,27	1,59	1,26	1,27	1,07	1,2	2,70	2,93	2,25	2,14	2,51	3,18	2,94	4,13	2,73	3,12	2,70	2,16	2,38	2,47	3,14	4,39	3,03	0,91	0,97	1,15	1,01
MnO	0,12	0,18	0,21	0,11	0,06	0,14	0,19	0,03	0,04	0,09	0,31	0,48	0,11	0,12	0,25	0,06	0,07	0,07	0,01	0,08	0,00	0,06	0,00	0,00	0,00	0,06	0,04	0,00	0,00	0,11	0,04
MgO	0,18	0,07	0,26	0,01	0,07	0,12	0,06	0,05	0,10	0,07	0,67	0,63	0,41	0,42	0,53	0,72	0,42	0,90	0,67	0,40	0,43	0,62	0,99	0,91	0,66	0,45	0,65	0,02	0,02	0,00	0,01
CaO	0,52	0,53	0,42	0,29	0,45	0,44	0,31	0,25	0,60	0,39	0,79	1,51	0,73	0,38	0,85	0,48	0,70	0,88	0,55	0,54	0,11	0,33	0,57	0,54	0,51	0,63	0,53	0,03	0,00	0,06	0,03
Na ₂ O	2,58	2,42	2,56	3,02	3,14	2,74	3,37	2,96	2,93	3,09	1,89	1,86	2,12	1,87	1,94	1,41	1,02	1,24	0,51	0,70	0,80	1,30	1,07	1,24	1,48	1,38	1,10	1,02	0,98	0,91	0,97
K ₂ O	4,18	4,31	3,72	3,75	4,05	4,00	4,05	4,07	4,45	4,19	4,47	5,71	3,97	5,30	4,86	8,19	7,04	6,24	6,66	6,36	7,14	6,24	5,83	7,60	6,23	6,43	6,72	7,56	7,27	7,96	7,60
P_2O_5	0,11	0,76	0,63	0,00	0,22	0,35	0,04	0,00	0,49	0,18	0,04	0,04	0,00	0,00	0,02	0,04	0,10	0,00	0,02	0,02	0,17	0,00	0,00	0,00	0,02	0,02	0,03	n.a.	0,12	0,19	0,15
Cl	n.a.	n.a.	n.a.	n.a.	n.a.	n.a	n.a.	n.a.	n.a.	n.a	n.a.	n.a.	n.a.	n.a.	n.a	0,36	0,21	0,34	0,08	0,14	0,09	0,18	0,20	0,37	0,44	0,33	0,25	n.a.	n.a.	n.a.	n.a
Total	89,78	88,45	88,30	90,28	91,48	89,66	89,27	92,48	90,50	90,76	97,42	99,27	95,64	98,59	97,73	99,76	100,74	98,12	100,86	98,85	99,23	99,12	98,88	100,29	97,73	99,53	99,37	98,77	99,29	100,21	99,47
Fluids by diff.	10,22	11,55	11,70	9,72	8,52	10,34	10,73	7,52	9,50	9,24	2,58	0,73	4,36	1,41	2,27	0,24	-0,74	1,88	-0,86	1,16	0,77	0,88	1,12	-0,29	2,27	0,47	0,63	1,23	0,71	-0,20	0,53
ASI	1,17	1,15	1,25	1,18	1,16	1,18	1,10	1,25	1,08	1,14	1,38	1,24	1,36	1,20	1,29	1,20	1,31	1,43	1,39	1,23	1,29	1,19	1,29	1,26	1,29	1,29	1,29	1,21	1,26	1,16	1,21
Fe+Mg*	0,028	0,030	0,031	0,020	0,020	0,026	0,022	0,019	0,015	0,019	0,056	0,063	0,042	0,041	0,050	0,063	0,049	0,078	0,055	0,053	0,048	0,045	0,055	0,056	0,058	0,073	0,056	0,013	0,012	0,018	0,014
CIPW norm	native mine	erals																													
Cor	2	3	2	2	3	3	1	2	2	2	4	3	3	2	3	2	4	4	4	2	3	2	3	3	3	3	3	2	3	2	2
Qz	35	37	36	35	36	36	31	37	34	34	40	32	42	43	39	31	42	40	50	50	46	47	50	37	42	41	37	42	46	46	42
Ab	20	22	26	27	23	23	28	25	25	26	16	16	18	16	16	14	10	13	5	7	7	12	11	12	15	14	9	9	7	9	8
Or	25	22	22	24	24	24	24	24	26	25	26	34	23	31	29	56	48	47	45	43	47	41	40	51	44	45	40	45	47	45	45
An	0	0	1	1	0	0	1	1	0	1	4	7	4	2	4	2	3	4	3	3	0	2	3	3	2	3	2	0	0	0	0