



Nanorocks: a 10-year-old story

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Abstract

The presence of nanogranitoids (crystallized melt inclusions showing a granitic *s.l.* composition) in regionally metamorphosed migmatitic and granulitic terranes was documented for the first time in 2009. This important finding represented a hallmark event for crustal petrologists, demonstrating that the pristine composition of anatectic melts may be preserved and recovered in small objects hosted in peritectic minerals of high-grade metamorphic rocks. Many further occurrences were documented in rocks worldwide, from diverse geodynamic settings, from the Archean to Miocene, and these inclusions turned out to have diverse composition (from silicatic to carbonatitic). Therefore, we propose “nanorocks” as a comprehensive name for these crystallized melt inclusions. In the last decade, many studies demonstrated their utility in characterizing mechanisms of melting and in tracking crustal magma evolution. Although scholars are now able to easily recognize nanorocks in high-grade metamorphic rocks, only a few research groups remelt them by means of experimental devices because of a time-consuming preparation. However, when cutting-edge techniques are applied in addition to more routine ones, nanorocks do open new perspectives in crustal petrology.

Keywords Nanorocks · Nanogranitoids · Migmatites · Granulites · Crustal melting

1 Definition and evolution of terminology

Rocks are generally made of aggregates of minerals (Fig. 1a, b). However, in some high-grade metamorphic rocks such as migmatites and granulites, some peculiar minerals called peritectic phases may contain tens to hundreds of “rocks” too (Fig. 1c). The latter occur as very tiny (mostly < 20 μm) aggregates of crystals with micrometric (few μm) or sub-micrometric (hundreds of nm) grain size, often showing equigranular, hypidiomorphic-to-allotriomorphic texture (Fig. 2a, b). Here, we propose “nanorocks” as a comprehensive name for these small objects in peritectic minerals of high-grade metamorphic rocks and, in this section,

we present the different types of nanorocks described in the literature.

In most occurrences, the main micro- to nano-minerals are silica polymorphs (quartz, tridymite, and cristobalite), NaAlSi₃O₈ polymorphs (albite, kumdykolite), KAlSi₃O₈ polymorphs (kokchetavite and K-feldspar), biotite, and muscovite. Sometimes, granophyric-to-nano-granophyric intergrowths of quartz and feldspars may be present (Cesare et al. 2011; Ferrero et al. 2012). Considering (1) that these textural features are all typical of common plutonic rocks such as granites and (2) the micro- to nano-scale grain size, the cryptocrystalline aggregate found within these inclusions in peritectic minerals was initially named “nanogranite” (Cesare et al. 2009). Being trapped during incongruent melting reactions, nanogranites represent a unique case study where anatexis (i.e., partial melting of the continental crust) is recorded and can be observed in situ, in its early stages.

An experimental protocol based on a piston-cylinder apparatus resulted in the complete re-homogenization of nanogranites (Bartoli et al. 2013a, b, 2016a, b). After remelting experiments at high-temperature and pressure conditions and, after quenching, nanogranites generally contain a homogeneous glass which can be chemically characterized for major and trace elements and volatiles (Bartoli et al.

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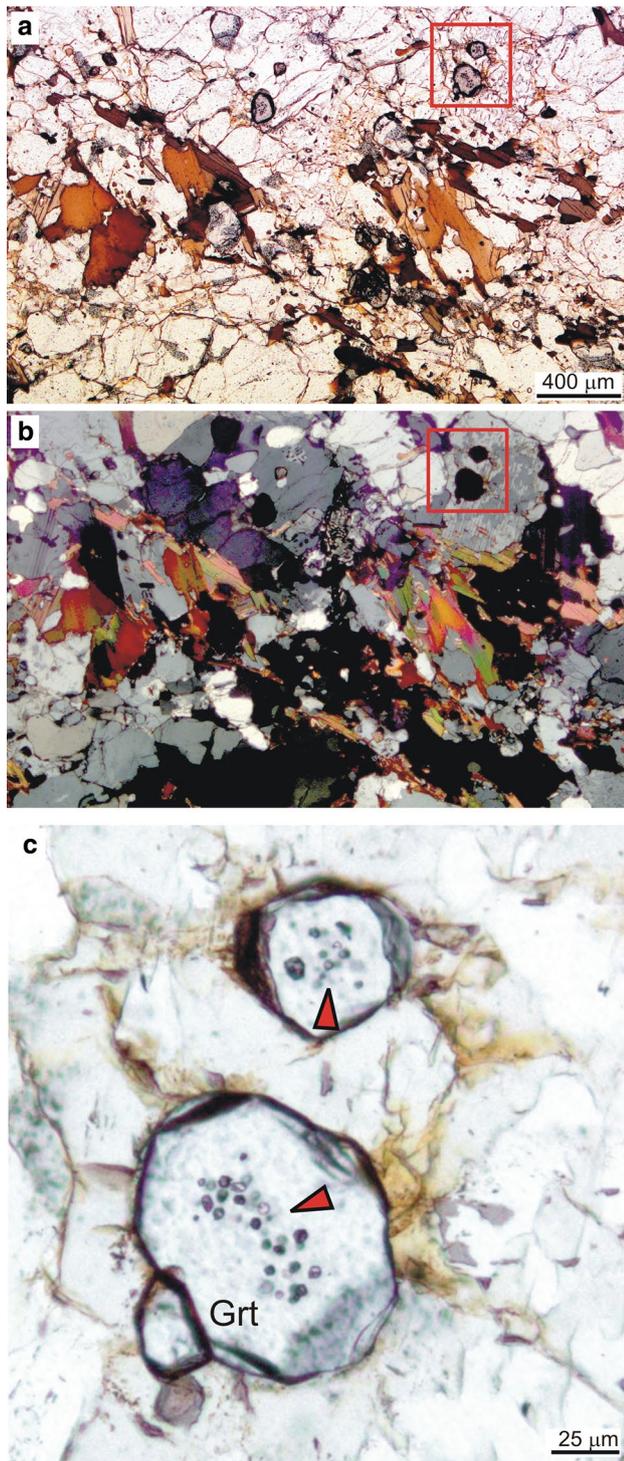


Fig. 1 Photomicrographs of migmatitic rocks from Ronda (S Spain). **a, b** Fine-grained mesocratic matrix composed of quartz, plagioclase, K-feldspar, biotite, sillimanite, and garnet; plane-polarized light and crossed polars, respectively. Square: area enlarged in **c**. **c** Close-up of garnet crystals where clusters of melt inclusions are present (red arrows); plane-polarized light. Parisatto et al. (2018) performed a synchrotron X- μ CT study and detected up to 500 melt inclusions within a single tiny garnet crystal from Ronda migmatites. Mineral abbreviations after Kretz (1983)

2014). The fact that successfully and completely remelted nanogranites yielded leucogranitic compositions (see Bartoli et al. 2013b) has represented a solid proof of their origin: they can be reasonably interpreted as former droplets of anatectic melt (i.e., melt inclusions) which have crystallized into a cryptocrystalline aggregate during cooling (Cesare et al. 2011).

After the first discovery of nanogranites in 2009 within granulites from the Kerala Khondalite Belt (India), many further occurrences were documented. Melting experiments and detailed microchemical studies have revealed the existence of nanorocks showing different compositions. For instance, Carosi et al. (2015) proposed the term “*nanotonalites*” for melt inclusions hosted in peritectic garnet of Kali Gandaki migmatites (Nepal) (Fig. 2c). Granodioritic and trondhjemitic melts were also reported in these rocks (Cesare et al. 2015; Bartoli et al. 2019) as well as in migmatites and granulites from Ronda (Spain; Acosta-Vigil et al. 2016) and Ivrea Zone (Italy; Carvalho et al. 2019), and in clinopyroxenites (Borghini et al. 2018). As a consequence of the melt compositional diversity, Cesare et al. (2015) and Bartoli et al. (2016b) proposed to adopt the term “*nanogranitoids*” to refer to crystallized melt inclusions in high-grade metamorphic rocks. Recently, nanogranitoids have been described in mafic–ultramafic rocks (i.e., Ferrero et al. 2018a; Borghini et al. 2018, 2019).

Surprisingly, Ferrero et al. (2016a) described polycrystalline inclusions with calcite, clinocllore, and white mica, coexisting with nanogranitoids in garnet from the Bohemian Massif (Oberpfalz area, central Europe). Piston-cylinder experiments successfully remelted nanogranitoids, but produced also calcite re-crystallization to microcrystalline aggregates and vesiculation. Considering the sub-micrometric internal assemblage and the bulk composition, calcite-rich inclusions were named “*nanocarbonatites*” (Ferrero et al. 2016a) (Fig. 2d). This so-far unique association of carbonatitic and silicate melts along with CO_2 -bearing fluids has been explained as the possible result of triple immiscibility during synchronous melting of two different protoliths (metapelites and metacarbonates) closely associated in space (see also Ferrero et al. 2018b; Ferrero and Angel 2018).

Dokukina et al. (2014) reported an interesting occurrence in rocks from Gridino area (Russia). Here, eclogitized olivine gabbro-norite is intruded by a granitoid rock containing kyanite and garnet within a quartzo-feldspathic matrix (\pm clinopyroxene and orthopyroxene). This rock has been classified as enderbite, a term belonging to the charnockite series. This enderbite has been interpreted as the result of mixing between a tonalitic melt and small proportion of residual material. Zircon grains from enderbite may contain polycrystalline inclusions ($< 20 \mu\text{m}$) of omphacite, phengite, biotite, and quartz, which were named “*nanoenderbite*” by Dokukina et al. (2014) (Fig. 3). They are likely to represent

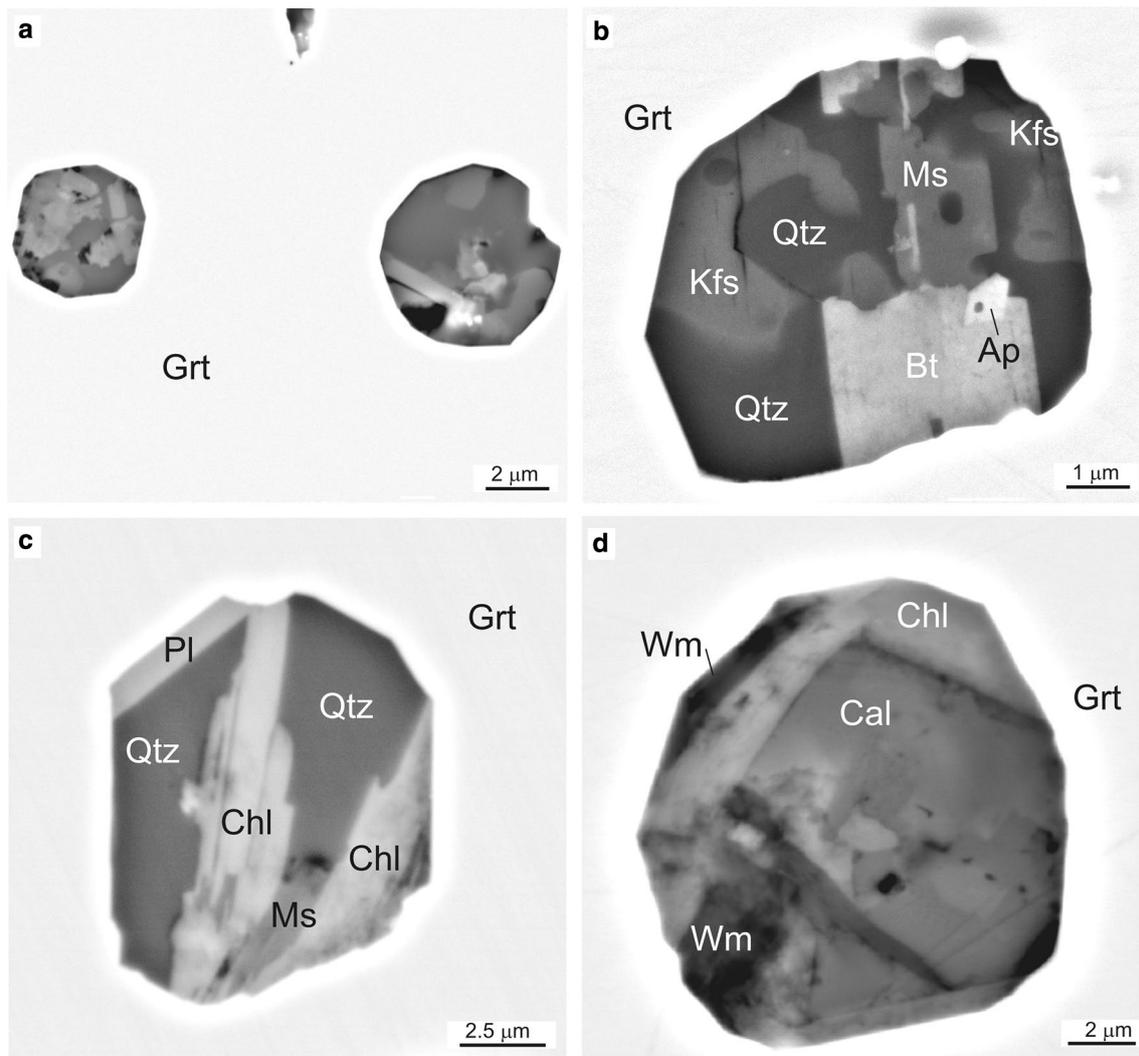


Fig. 2 SEM-backscattered (BSE) images of melt inclusions with the typical negative crystal shape. **a, b** Nanogranitoids from Ronda migmatites (Spain; see also Bartoli et al. 2013c, 2015, 2016a). **c** Nanogranitoid inclusion from Kali Gandaki migmatitic gneisses (Nepal). Phase equilibria modeling demonstrated that the formation of chlorite does not require the infiltration of external fluids; rather,

it can form in the subsolidus during cooling (see Bartoli et al. 2019). **d** Nanocarbonatite from Bohemian Massif; image courtesy of S. Ferrero (see also Ferrero et al. 2016a, b, 2018b; Ferrero and Angel 2018). Minerals were identified by means of EDS spectra and Raman spectroscopy

the crystallization product of a former melt, whereas the host zircon is interpreted to mark the upper age limit for the eclogite facies metamorphism.

Polycrystalline inclusions have been also described in zircons from charnockites of the Gruf Complex (central Alps). In this case, inclusions are composed of quartz, biotite, orthopyroxene, and garnet, resembling the mineralogy of the host rock (Galli et al. 2012). Because their size can reach 3–4 μm, these inclusions could be named “nanocharnockites”, even though a detailed study would be needed to characterize their microstructures.

An important point that should be highlighted is that all the terms reported above refer to minute-sized inclusions

(1) derived from crystallization of a silicatic (with the exception of the nanocarbonatites of Ferrero et al. 2016a) anatectic melt and (2) containing diverse solid phases (sometimes along with minor H₂O or CO₂ bubbles; see Bartoli et al. 2013b). Therefore, the term “nanorocks” should not be considered synonymous of “multiphase inclusions” which, instead, contain solid phases and larger proportions of fluid/void; the latter represent former fluid inclusions (additional details in Tacchetto et al. 2019 and Bartoli 2020).

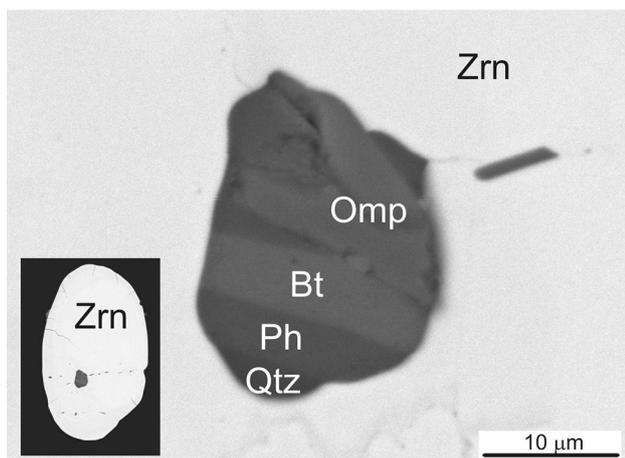


Fig. 3 SEM-BSE image of nanoenderbite (modified from Dokukina et al. 2014). Image courtesy of K. A. Dokukina

2 Nanorock occurrences

Although many nanorock occurrences have been documented so far (see Table 1 in Cesare et al. 2015), their experimental remelting and chemical characterization has been basically carried out by only two research groups at Padua (Italy) and Potsdam (Germany) universities (Fig. 4). Indeed, experimental remelting of nanorocks is time-consuming

because of the trial-and-error nature of the experiments and the very small size of these objects. For the sake of completeness, it should be noted that anatectic melt inclusions from the Kokchetav Massif were remelted at ANU (Australia) (Stepanov et al. 2016).

The extensive geochemical database of melt inclusions in high-grade metamorphic rocks comprises more than 700 major- and 300 trace-element analyses. Regarding volatiles, the data set consists of about 200 and 50 measurements of H_2O and CO_2 , respectively, by either SIMS, NanoSIMS, or Raman spectroscopy.

To date, the analyzed nanorocks come from five continents (Europe, Asia, America, Africa, and Antarctica) (Fig. 4) and are related to different geological and geodynamic scenarios such as extensional and collisional settings, up to extreme conditions such as ultra-high temperature (UHT) and ultra-high pressure (UHP) (Fig. 4). For very recent discoveries, the interested reader is referred to Liu et al. (2020a, b), Borghini et al. (2020), and Carvalho et al. (2020). Geochemical modeling using nanorocks is crucial to reconstruct crustal magma evolution (see Bartoli et al. 2016b, 2019; Bartoli 2020). In addition, composition of melt inclusions in migmatites and granulites may give us a wealth of information on mechanisms and kinetics of anatexis (Acosta-Vigil et al. 2010, 2012, 2016; Barich et al. 2014; Carvalho et al. 2019).

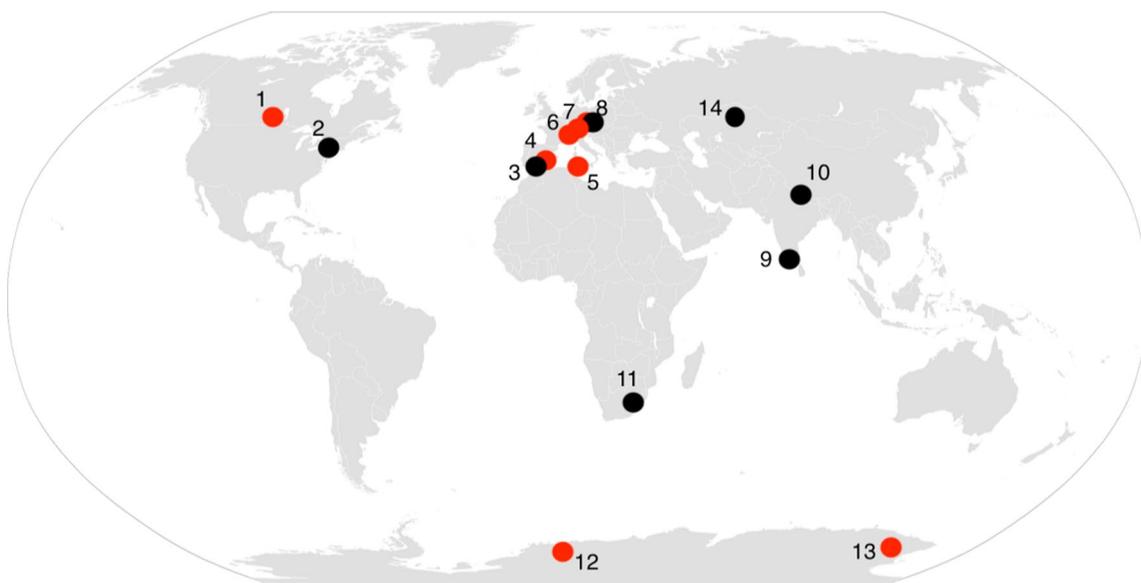


Fig. 4 World map of nanorock occurrences. Only experimentally remelted nanorocks and preserved glassy melt inclusions are reported here. The red dots denote localities where nanogranitoids and CO_2 - and COH-bearing multiphase inclusions coexist. The black dots denote, instead, localities where nanorocks occur alone. 1: Tacchetto et al. (2019); 2: Ferrero, unpublished; 3: Bartoli et al. (2013a) and Acosta-Vigil et al. (2016); 4: Acosta-Vigil et al. (2010, 2012, 2017);

5: Ferrero et al. (2014); 6: Carvalho et al. (2019); 7: Carvalho et al. (2020); Gianola, unpublished; 8: Borghini et al. (2018); Ferrero et al. (2018b) and references therein; 9: Cesare et al. (2009); 10: Bartoli et al. (2019); 11: Bartoli, unpublished; 12: Ferrero et al. (2018a); 13: Ferri, unpublished. 14: Stepanov et al. (2016). Many localities in the Bohemian Massif contain nanogranitoids and the interested reader is referred to Ferrero et al. (2018b) for more details

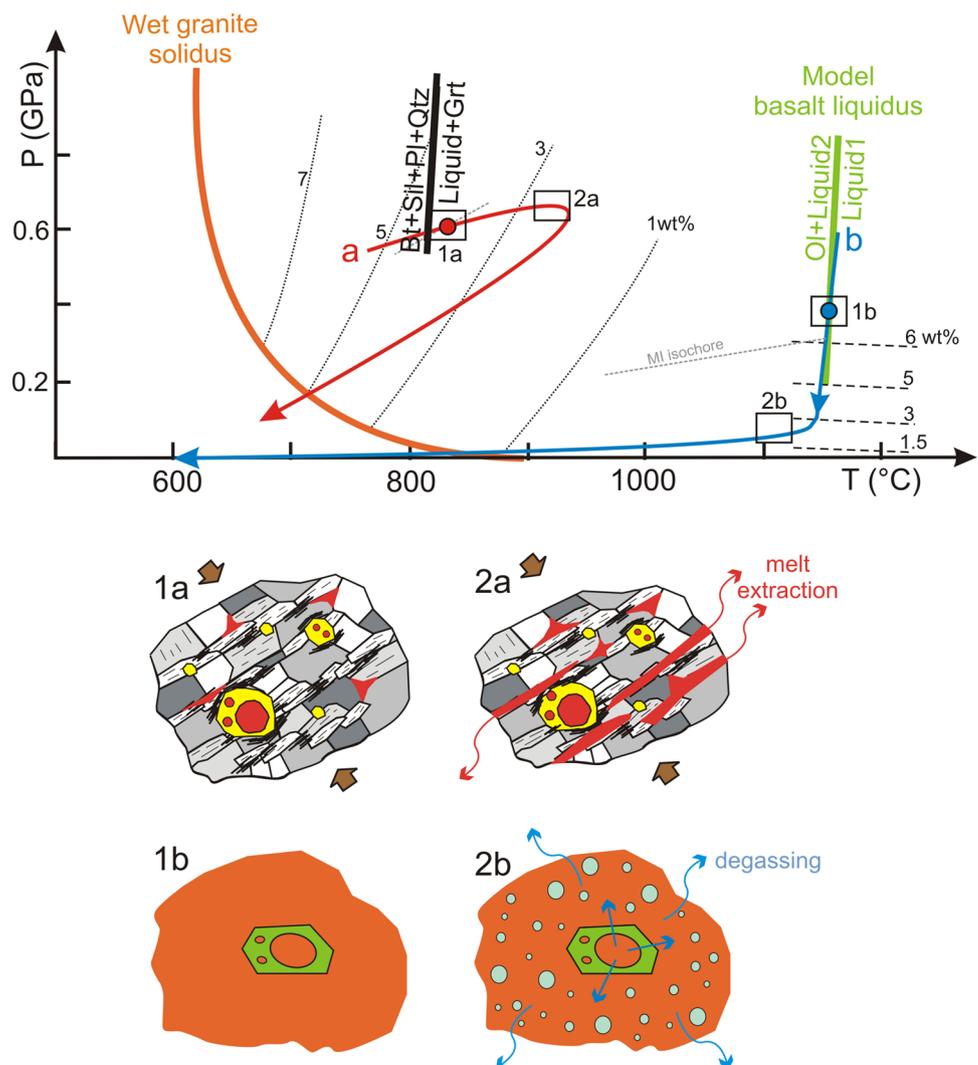
3 Different trapping modes: implications for H₂O budget of melt inclusions

Melt inclusions have long been studied in volcanic and plutonic rocks, as well as in mantle xenoliths (e.g., Frezzotti 2001; Bodnar and Student 2006; Webster and Thomas 2006; Audetat and Lowenstern 2014; and references therein). These melt inclusions (hereafter “classic melt inclusions”) are generally trapped during crystallization of magmas, i.e., along the cooling path where the host crystal is crystallizing from the melt that is being entrapped (path b in Fig. 5). In other words, classic melt inclusions are samples of magma from which the host mineral was crystallizing. Conversely, a diverse entrapment process occurs in high-grade metamorphic rocks. Here, incongruent melting reactions create the necessary conditions for the entrapment of anatectic melt along the heating path (path a in Fig. 5). Indeed, these reactions are

characterized by formation of solid phases (the peritectic mineral) along with melt (Fig. 5). Being the solid product of melting reactions in a wide P–T–X range, garnet is the most common host of melt inclusions in migmatites and granulites (Cesare et al. 2015).

The two contrasting modes of trapping (during crystallization of magmas vs. during incongruent melting) along with the contrasting P–T paths commonly observed in the two settings have diverse implications for the H₂O content of the trapped melt (Fig. 5). After considering the equation of Brodkey and Hershey (2003) for mass transport by diffusion, Bartoli et al. (2014) argued that the likely driving forces for H₂O diffusion from/to melt inclusions at supra-solidus conditions are gradients of H₂O concentration and pressure. In this sense, melt inclusions in volcanic rocks are the most suitable system for diffusional re-equilibration and H₂O loss, as also demonstrated by experimental studies (e.g., Chen et al. 2011; Gaetani et al. 2012; Portnyagin et al. 2008). This unavoidable evolution is related to the (1)

Fig. 5 Two contrasting scenarios of melt inclusions entrapment (modified from Bartoli et al. 2014) and their consequence on H₂O content of trapped melt. Path a: melt entrapment within peritectic garnet during incongruent melting reactions in crustal rocks. Path b: melt entrapment in olivine phenocryst during basaltic magma crystallization. Dashed black lines refer to H₂O solubility in basaltic melts. Dotted black lines represent the liquidus curves for the haplogranite eutectic or minimum melt composition at specified H₂O contents interpreted to predict the minimum H₂O content of granitic melts. The cartoon reflects boxes in the P–T diagram. See text for details



high-temperature conditions ($> 1000\text{ }^{\circ}\text{C}$) of the surrounding magma which favor element diffusivities in phenocrysts, (2) pressure gradients developed during (often very) rapid near-isothermal decompression of associated magma, and (3) volatile concentration gradients which are continuously established between the melt within inclusions and the external degassing magma (Fig. 5) (see also Bartoli et al. 2014).

All the above conditions are generally not applicable in a migmatitic and granulitic terrane. Following Bartoli et al. (2014), the main differences are: (1) melt inclusion-bearing peritectic minerals are present in a predominantly solid matrix, because matrix melt is extracted when, under differential stress conditions, a critical threshold of $\approx 7\text{ vol.}\%$ is reached (Rosenberg and Handy 2005) (Fig. 5), (2) the lower temperatures resulting in much lower (up to 5–7 orders of magnitude) hydrogen diffusivities in silicate minerals, and (3) the lack of significant pressure gradients between rock matrix and melt inclusions (Fig. 5). In addition, nanorock-bearing garnet porphyroblasts are commonly much larger (a few mm up to several cm) than lava phenocrysts, resulting in longer diffusion distances between melt inclusion and mineral boundary. Therefore, it is not surprising that the equilibration times regarding hydrogen and H_2O between melt inclusions and the surrounding melt are much greater (> 10 order of magnitudes) in high-grade metamorphic terranes compared to plumbing magmatic systems (Bartoli et al. 2014). Rather, intense decrepitation and/or fluid infiltration along subsolidus retrograde path may affect H_2O budget of nanorocks, but these processes leave clear microstructural evidence (see Fig. 15 in Cesare et al. 2015), and the modified nanorocks can be easily identified and discarded.

An additional criterion to identify nanorocks that have not been modified by intense decrepitation and fluid leakage is the presence of kumdykolite and kokchetavite (polymorphs of $\text{NaAlSi}_3\text{O}_8$ and KAlSi_3O_8 , respectively; Ferrero and Angel 2018). Indeed, Ferrero et al. (2016b) identified these phases within preserved nanorocks from Bohemian Massif, whereas these polymorphs were not present in coexisting melt inclusions with cracks reaching the host-matrix boundary.

4 The unexpected: coexistence of anatectic melt and COH fluid in the deep crust

The systematic investigation of crystallized melt inclusions by laser microRaman spectroscopy (e.g., Ferrero et al. 2016b) has led to the recent finding of carbonate-bearing inclusions coexisting with nanogranitoids in the same clusters within garnet. The pioneering work by Tacchetto et al. (2019) on such associations has also made use of the serial slicing technique at the FIB-SEM (Fig. 6), which allowed a 3D characterization of these small objects, with

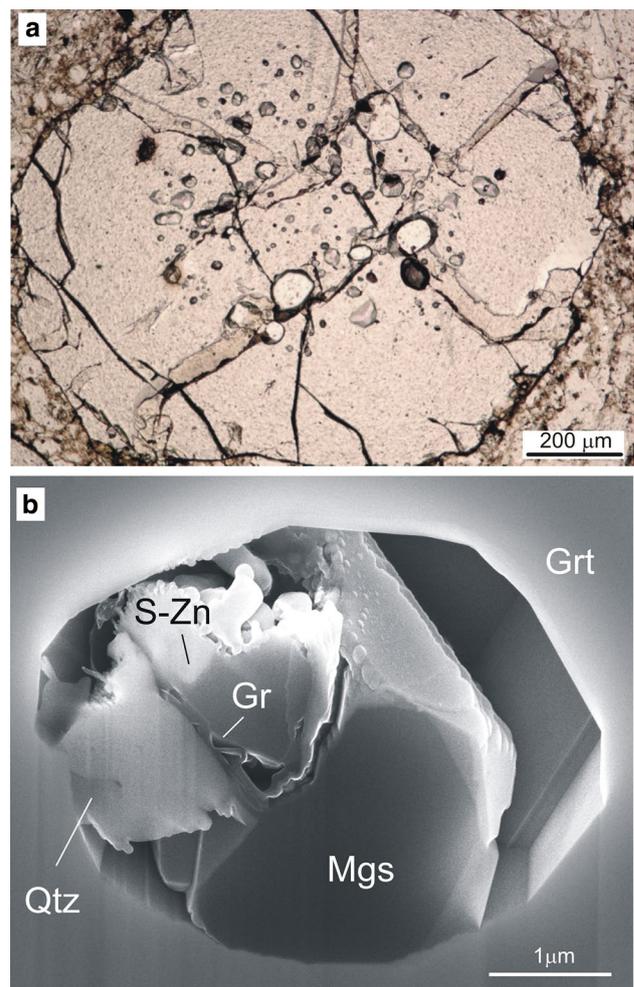


Fig. 6 **a** Garnet from felsic granulite from the Athabasca Granulite Terrane, containing nanorocks, CO_2 -bearing multiphase inclusions, and quartz grains. **b** SEM-BSE image of solid phases in an inclusion exposed by FIB-SEM technique (modified from Tacchetto et al. 2019). The void was occupied by low-density CO_2 as demonstrated by Raman spectroscopy. Image courtesy of T. Tacchetto

quantification of volume and composition of all phases in them, including the fluid component. Due to the large volume ($> 40\%$, Tacchetto et al. 2019) occupied by a CO_2 -rich fluid phase, these inclusions cannot be interpreted as former carbonatitic melts such as in the study by Ferrero et al. (2016a), but as multiphase inclusions as defined above. More recent investigations (e.g., Carvalho et al. 2019; Safonov et al. 2020) and a reassessment of literature data (see Carvalho et al. 2020 and references therein) show that in high-grade rocks, these multiphase inclusions, as well as their association with nanogranitoids, are much more common than expected.

The primary multiphase inclusions (Fig. 6b) contain the systematic association of Fe–Mg(–Ca) carbonates, one or more Si–Al phase (Al_2SiO_5 , corundum, pyrophyllite, and

kaolinite) and a CO₂-dominated fluid ($\pm \text{H}_2\text{O} \pm \text{CH}_4 \pm \text{N}_2$) displaying a much lower density than expected for the P–T conditions of formation of the host garnet. Based on these characteristics, Carvalho et al. (2020) have proposed an interpretation of these inclusions (and a reinterpretation of other occurrences from the literature) as former carbonic fluid inclusions that reacted during cooling with the host garnet. In fact, balanced reactions that form carbonates, pyrophyllite, and/or kaolinite can be proposed between garnet and a fluid containing CO₂ ± H₂O. This process of step-daughter mineral formation (as described by Roedder 1984) also explains the decrease of fluid density within inclusions (e.g., Heinrich and Gottschalk 1995; Tacchetto et al. 2019). The variable CO₂/H₂O ratio in the primary fluid determines the type and proportions of step-daughter phases forming in the multiphase inclusions, with the amounts of kaolinite and pyrophyllite relating to the aqueous component of the carbonic fluid. The frequent occurrence of carbonate-bearing primary multiphase inclusions in garnet and, conversely, the rarity (or absence) of definitively primary carbonic fluid inclusions in the same host have led Carvalho et al. (2020) to conclude that carbonic fluid inclusions entrapped at high-T conditions are not preserved when hosted in garnet, because they are expected (and observed) to re-equilibrate by production of step-daughter assemblages.

The coexistence of nanogranitoids and multiphase inclusions in microstructural positions that support a primary entrapment indicates that during anatexis and garnet growth, there was a situation of melt–fluid immiscibility: the melt could not dissolve all the fluid, because of its significant carbonic component (Tamic et al. 2001), and a free fluid phase was therefore present. Given the widespread association of these multiphase inclusions with nanogranitoids (occurrences reviewed by Carvalho et al. 2020), the phenomenon of melt–fluid immiscibility in migmatites and granulites appears to be quite common, albeit overlooked. Indeed, COH fluid–melt immiscibility during anatexis is far from unexpected, especially in graphitic metasedimentary protoliths. From a theoretical point of view the occurrence of graphite necessarily implies the presence of carbonic components in a fluid, even if produced by dehydration reactions (Connolly and Cesare 1993). In turn, this creates the conditions for the fluid not to be totally miscible with a granitic anatectic melt. Before the recent findings in migmatites and granulites, these theoretical predictions have been confirmed by the studies of fluid–melt immiscibility in cordierite and garnet from graphitic anatectic enclaves at El Hoyazo and Mazarrón, Spain (Cesare et al. 2007; Ferrero et al. 2011) and in garnet at la Galite, Tunisia (Ferrero et al. 2014). Note that in the latter study, the anatectic rock does not contain graphite. In fact, immiscibility can occur also in non-graphitic rocks, whenever a carbonic fluid—either internally or externally derived—is present.

5 Conclusions and perspectives

Cesare et al. (2011) concluded the first review paper on nanogranitoids writing “we believe that many occurrences of MI have been overlooked because they simply were not searched for, and that they will be uncovered by careful re-investigation of migmatite and granulite samples worldwide.” After a decade, we can say that this sentence was true. Nanorocks occur within rocks worldwide and their presence has been only recently disclosed in world-renowned anatectic terranes (such as the Ivrea-Verbano Zone and the Bohemian Massif), which have been previously investigated through many approaches (see Ferrero et al. 2018b; Carvalho et al. 2019).

Nanorocks are a novel subject of research and are becoming a matter of considerable scientific interest. After a 10-year-old story, a compositional database of primary anatectic melts, entirely based on nanorocks, is now available, even though the accurate remelting and geochemical characterization of these small objects is far from being routine. However, we believe that the rapid improvement of analytical techniques will have overcome the problems related to their small size. In particular, owing to the common coexistence of nanorocks and COH fluid inclusions, finding a method to distinguish quickly and certainly between them is essential and represents the next step in this research field.

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Compliance with ethical standards

Conflict of interest We declare that there are not potential conflicts of interest.

Ethical approval We declare that my manuscript complies to the Ethical Rules applicable for this journal.

Human and animal rights We declare that my research does not involve humans, human embryos, animals and processing of personal data and does not have direct military use.

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