

Into the High to Ultrahigh Temperature Melting of Earth's Crust: Investigation of Melt and Fluid Inclusions within Mg-Rich Metapelitic Granulites from the Mather Peninsula, East Antarctica

Zhao Liu¹, Bruna B. Carvalho², Wancai Li³, Laixi Tong^{1,*}, Omar Bartoli², Longyao Chen⁴, Qinghe Yan⁵ and Haobo Wu¹

¹State Key Laboratory of Continental Dynamics, Department of Geology, Northwest University, Xi'an 710069, China

²Dipartimento di Geoscienze, Università di Padova, Via Gradenigo 6, Padua 35131, Italy

³CAS Key Laboratory of Crust–Mantle Materials and Environments, School of Earth and Space Sciences, University of Science and Technology of China, Hefei 230026, China

⁴Key Laboratory of Paleomagnetism and Tectonic Reconstruction of Ministry of Natural Resources, Institute of Geomechanics, Chinese Academy of Geological Sciences, Beijing 100081, China

⁵School of Resource Environment and Earth Science, Yunnan University, Kunming 650091, China

*Corresponding author. Telephone: +86 029-88302312. E-mail: tonglx@nwu.edu.cn

Abstract

Precise constraints on the compositions of melts generated by anatexis under ultrahigh temperature (UHT) conditions are critical for understanding processes of partial melting and differentiation of the Earth's crust. Here we reveal geochemical and physical signatures of anatectic melts preserved as nanogranitoids (i.e. crystalized melt inclusions) within sapphirine-bearing UHT metapelitic granulites from the Mather Peninsula, East Antarctica. Their coexistence with high–Al orthopyroxene as inclusions in garnets strongly suggests that the investigated melts were at least partially UHT in origin. The nanogranitoids are enriched in SiO₂ (69.9–75.6 wt.%), strongly peraluminous (ASI values = 1.2–1.6) and potassic to ultrapotassic (Na₂O + K₂O = 7.1–9.5 wt.%, K/Na = 2.2–9.3). When compared to the granulitic restite, the melts are enriched in Li, Cs, Rb, Ta, Sm, Nd, Zr, U and Pb, and depleted in Ce, Th, Ba, Sr and Nb. Their geochemical characteristics are consistent with biotite–dehydration melting in the absence of plagioclase. Our calculation results indicate that these hot crustal melts have low densities of 2.47 ± 0.07 g/cm³, low viscosities of $10^{4.9 \pm 1.2}$ Pa·s and high heat production values of ~ 2.8 μW/m³. Therefore, such melts are mobile and susceptible to be extracted from the source, and consequently their flow and removal from the deep crust may greatly affect the chemical and thermal structure of the continental crust.

Secondary C–O–H fluid inclusions within garnet and orthopyroxene have also been detected. These inclusions contain magnesite, pyrophyllite, corundum, with or without residual CO₂. The minerals within the fluid inclusions are interpreted as stepdaughter minerals, which were produced by the reaction of the fluid with its host. The metamorphic timing of the investigated rocks is still a matter of debate. Zircon U–Pb dating results obtained in this study suggest that the metapelitic granulites may have undergone two separated thermal events at ~ 1000 and ~ 530 Ma, respectively. The presence of fluid inclusions indicates that fluid infiltration and Pan–African reworking may have played an important role in obscuring chronological information of the early thermal scenario in poly-metamorphic terranes.

Key words: East Antarctica; Mather Peninsula; C–O–H fluid; Melt inclusions; UHT metamorphism

INTRODUCTION

Ultrahigh temperature (UHT) metamorphism is the most thermally extreme type of crustal metamorphism, with peak temperatures exceeding 900°C at moderate pressures (7–13 kbar) (Harley, 1998a, 2008), with dT/dP values of 66–200°C/kbar (Harley, 2021). UHT metamorphism is now recognized as a relatively common rather than anomalous process of the Earth's crust due to (1) its wide age distribution ranging from Archean to Quaternary (e.g. Brown, 2007; Pownall *et al.*, 2014; Harley, 2021; Zhang *et al.*, 2022); and (2) its extensive exposures around the world (>70 localities; Harley, 2021; Lei & Xu, 2018) related to various geodynamic processes. For example, UHT metamorphism may occur in tectonic backgrounds associated with (1) lithospheric extension

and underplating of mantle-derived magmas as a result of slab rollback (e.g. Pownall *et al.*, 2014), mantle plume activity (e.g. Tong *et al.*, 2014a; Liu *et al.*, 2020); (2) oceanic ridge subduction (e.g. Santosh *et al.*, 2012), and inversion and thickening of hot back-arc (e.g. Brown, 2007); (3) long-lived orogenic self-heating by radioactive elements (e.g. Clark *et al.*, 2011; Zhang *et al.*, 2022); or (4) a combined contribution from several driving forces (Huang *et al.*, 2019, 2021).

High temperature (HT) to UHT metamorphism of crustal rocks inevitably produces anatectic melts at depth, and their loss yields a refractory lower crust, whereas ascent and emplacement of granitic melts at shallower levels create a more silicic upper crust (e.g. Brown, 2007, 2013; Sawyer *et al.*, 2011). Furthermore,

it may result in decrease of the rock strength and variations in bulk compositions of the granulitic rocks (e.g. Rosenberg & Handy, 2005; Diener & Fagereng, 2014; Cavalcante *et al.*, 2016). However, interpretations of melting mechanisms of crustal rocks remain a long-standing challenge, which have been traditionally tracked with the aid of (1) geochemistry of granites and leucosomes (e.g. Zeng *et al.*, 2005; Gao *et al.*, 2017; Wolf *et al.*, 2019), (2) phase equilibria modeling at specific P - T conditions (e.g. White *et al.*, 2004, 2014; Wei, 2016; Huang *et al.*, 2021), and (3) melting experiments on natural rocks under variable P - T - X_{H_2O} conditions (refer to Gao *et al.*, 2016 and references therein). Furthermore, a recent progress is to conduct in situ observations and analyses on nanogranitoid inclusions (partially to totally crystallized melt inclusions preserved in peritectic phases; named after Cesare *et al.*, 2015) and their glassy counterparts (e.g. Cesare *et al.*, 2009, 2015; Acosta Vigil *et al.*, 2010, 2012; Ferrero *et al.*, 2012, 2016, 2018, 2021a, 2021b; Bartoli *et al.*, 2013a, 2013b, 2014, 2016, 2019; Gao *et al.*, 2014; Borghini *et al.*, 2018, 2023; Ferrero & Angel, 2018; Carvalho *et al.*, 2019, 2023a; Ferri *et al.*, 2020; Bartoli & Carvalho, 2021; Gianola *et al.*, 2021; Tacchetto *et al.*, 2021; Higashino & Kawakami, 2022). Thus far, melt inclusions have been only recognized in a few UHT metapelitic terranes, including (1) the Ivrea Zone (NW Italy) (Carvalho *et al.*, 2019), (2) the Kerala Khondalite Belt (India) (Cesare *et al.*, 2009; Ferrero *et al.*, 2012), (3) the Gruf Complex (European Central Alps) (Gianola *et al.*, 2021), (4) the Lützow-Holm Complex (East Antarctica) (Kawakami & Motoyoshi, 2004; Hiroi *et al.*, 2019; Suzuki & Kawakami, 2019; Carvalho *et al.*, 2023a), (5) the Sør Rondane Mountains (East Antarctica) (Higashino & Kawakami, 2022), and (6) the Chinese Altai orogen (NW China) (Liu *et al.*, 2020). It is worth noting that the occurrence of nanogranitoids in UHT rocks does not imply automatically that they reflect the UHT crustal melts because melts can be trapped during the HT prograde metamorphism (refer to Gianola *et al.*, 2021). A better understanding of potential effects of UHT metamorphism on the differentiation and dynamic evolution of the crust demands that the hottest crustal melts are chemically characterized, in particular for their major and trace elements.

The Mather metapelitic granulites from the Rauer Islands (East Antarctica) are a world-renowned poly-metamorphic UHT lithology, with peak P - T conditions of 8.5–12 kbar and 950–1050°C (Harley & Fitzsimons, 1991; Harley, 1998b, 2016; Tong & Wilson, 2006; Kelsey *et al.*, 2007; Harley *et al.*, 2009; Clark *et al.*, 2019; Chen *et al.*, 2023). In this contribution, we conduct an integrated investigation of microstructural features, major and trace element contents of nanogranitoid inclusions (NIs) preserved within garnet porphyroblasts. Data collected in this work are fundamental to the understanding of melting regimes of crustal rocks under UHT conditions, and their contribution to crustal differentiation and orogenic evolution. Secondary fluid inclusions (FIs) in peritectic phases are also characterized to reveal the nature of post-peak infiltrated fluid, and to evaluate its potential role in resetting the U-Pb clock of zircons.

GEOLOGICAL SETTING

The Antarctic continent is separated by the Transantarctic Mountains into two distinct geological units, namely East Antarctica and West Antarctica. East Antarctica is one of the largest cratons around the world, documenting more than 3.5 billion years of evolutionary history (Harley *et al.*, 2013). The Prydz Belt was recognized as a tectonic mobile belt in East Antarctica that has been extensively affected by the Pan-African (~530 Ma)

thermal event (e.g. Hensen & Zhou, 1997; Harley *et al.*, 1998; Kelsey *et al.*, 2003, 2007; Harley & Kelly, 2007; Wang *et al.*, 2007, 2008, 2022; Liu *et al.*, 2009, 2021; Grew *et al.*, 2012; Tong *et al.*, 2014b, 2019). The tectonic nature of the Prydz Belt remains debated and there are two main schools of thought; one considers that it represents a Pan-African suture zone associated with the final assembly of East Gondwana (e.g. Zhao *et al.*, 1995; Hensen & Zhou, 1997; Fitzsimons, 2003; Kelsey *et al.*, 2007; Liu *et al.*, 2021; Wang *et al.*, 2022), and the other views it as a Pan-African intraplate orogen related to intracontinental reworking (e.g. Phillips *et al.*, 2007; Wilson *et al.*, 2007; Tong *et al.*, 2014b, 2019; Ren *et al.*, 2016).

The Rauer Islands are located on the eastern margin of the Prydz Belt, with the Brattstrand Bluff and Larsemann Hills to the southwest, and the Vestfold Hills to the northeast. The Rauer Islands were thought to be texturally correlated with the Rengali-Eastern Chats Province, forming parts of the Indo-Antarctica supercontinent (e.g. Sawant *et al.*, 2017). Archean and Proterozoic crustal components have been recognized from the Rauer Islands (Kinny *et al.*, 1993). The Proterozoic domain comprises metamorphosed 1420–1000 Ma mafic-intermediate-felsic intrusive rocks (Kinny *et al.*, 1993; Liu *et al.*, 2021). The early Neoproterozoic (i.e. Grenville aged; 1000–900 Ma) and late Neoproterozoic/Cambrian (i.e. Pan-African aged; 590–500 Ma) ages have been identified from metamorphic rocks in this region (e.g. Kinny *et al.*, 1993; Kelsey *et al.*, 2007; Liu *et al.*, 2021). The Archean domain predominantly consists of tonalitic to granitic orthogneisses with three age clusters of 3470–3270, 2840–2800 and ~2550 Ma (Kinny *et al.*, 1993; Harley *et al.*, 1998; Hokada *et al.*, 2016; Liu *et al.*, 2021). This domain also contains Fe- or Mg-rich layered mafic complexes that were subjected to a granulite facies metamorphism in the Cambrian (Harley *et al.*, 1998; Harley & Kelly, 2007; Chen *et al.*, 2023). Although Pan-African ages have been widely recognized in the Archean domain (e.g. Kelsey *et al.*, 2003, 2007; Wang *et al.*, 2007; Hokada *et al.*, 2016; Clark *et al.*, 2019; Liu *et al.*, 2021), it is still unclear whether it was also involved in the Grenville-aged tectono-thermal event.

The Rauer Islands consist of two paragneiss successions, namely the Filla and the Mather Paragneisses (Fig. 1a; Harley & Fitzsimons, 1991). These two paragneiss successions are mainly distributed on the Proterozoic and Archean crustal domains, respectively. Deposition ages of the Filla Paragneiss have been constrained to be Mesoproterozoic (Sims *et al.*, 1994; Liu *et al.*, 2021). This unit was considered to have experienced a prominent metamorphic event at 1030–970 Ma and a high-grade reworking at 575–510 Ma (e.g. Kinny *et al.*, 1993; Kelsey *et al.*, 2007; Liu *et al.*, 2021). The Mather Paragneiss typically comprises magnesian and aluminous garnet-orthopyroxene-sillimanite-bearing metapelite, orthopyroxene-sillimanite metaquartzite, magnesian garnet-orthopyroxene metapelite and garnet-bearing mafic granulite (Fig. 1b; Harley *et al.*, 1995; Harley, 1998b). This suite usually occurs as thin and laterally discontinuous horizons from the Mather Peninsula to the Short Point in the eastern Rauer Islands (Hokada *et al.*, 2016). Mather Paragneiss preserves strong evidence for UHT metamorphism, with exposures of sapphirine-bearing metapelitic granulites on the Mather Peninsula (e.g. Harley, 1998b; Tong & Wilson, 2006) and the Torckler Island (Harley *et al.*, 2009), with peak P - T conditions of 8.5–12 kbar and 1000–1050°C (e.g. Harley, 1998b, 2016; Tong & Wilson, 2006). The sapphirine-bearing UHT gneisses preserve inherited zircon ages of 2800–2400 Ma (e.g. Wang *et al.*, 2007; Hokada *et al.*, 2016), and are hosted by ~3267 Ma tonalitic orthogneisses (Hokada *et al.*, 2016). However, timing of the UHT metamorphism is still a matter of debate.

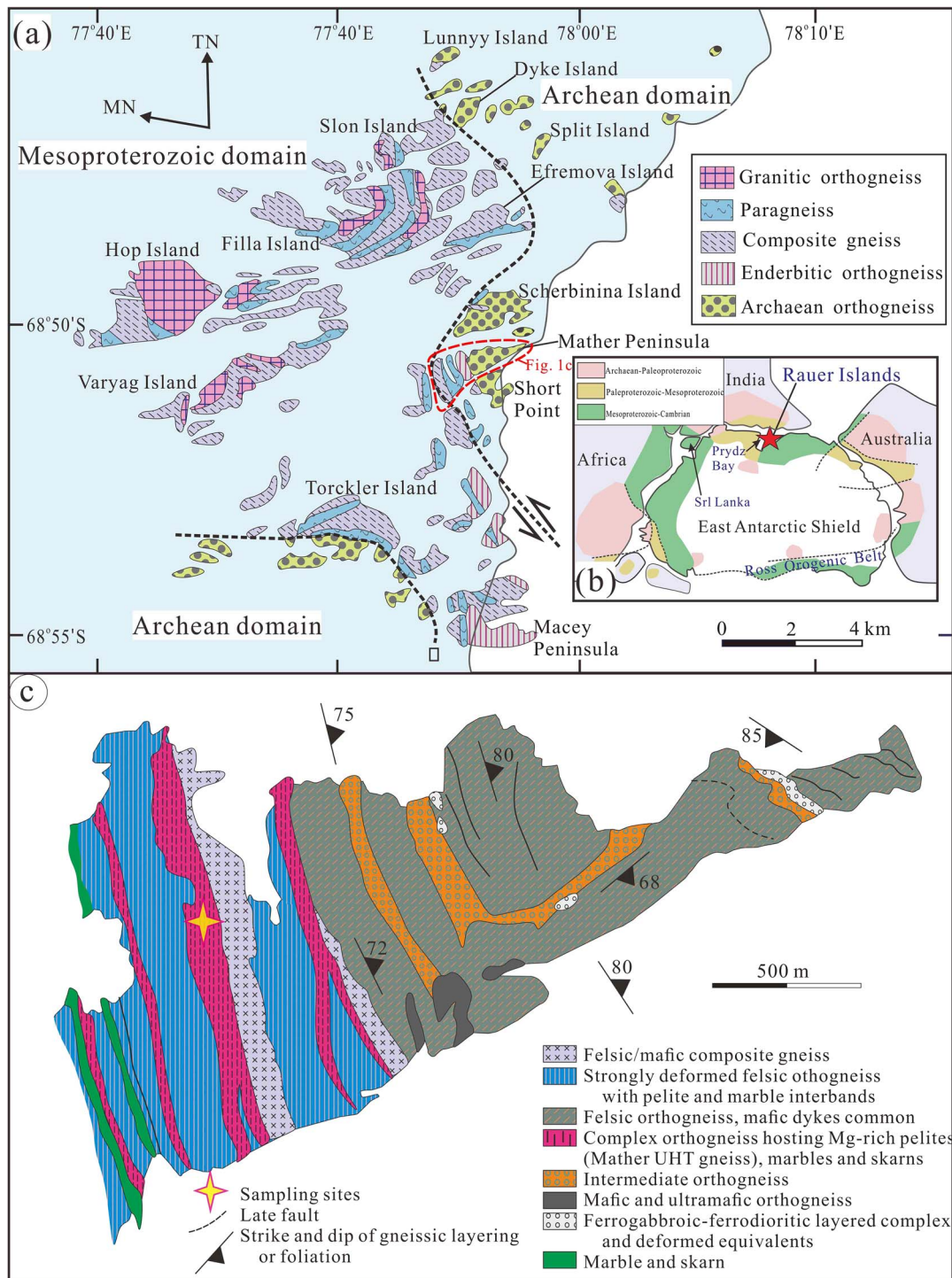


Fig. 1. (a) Simplified geological map of the Rauer Islands, East Antarctica (after Harley & Fitzsimons, 1991; Tong & Wilson, 2006; Tong *et al.*, 2021). The black dashed line represents the boundary between Mesoproterozoic and Archean crustal components. (b) Location of the Rauer Islands in the reconstructed Gondwana supercontinent at ~500 Ma (modified after Fitzsimons, 2000; Wilson *et al.*, 2007; Hokada *et al.*, 2016). (c) Lithological distribution and sample locality on the Mather Peninsula (after Harley, 1998b; Hokada *et al.*, 2016).

Some researchers argued that the UHT metamorphism occurred at ~1000 Ma and the subsequent decompression and final cooling happened at ~530 Ma (e.g. Tong & Wilson, 2006; Wang *et al.*, 2007) while others considered that the UHT metamorphism occurred in the Cambrian (e.g. Kelsey *et al.*, 2003, 2007; Clark *et al.*, 2019) or prior to 590–580 Ma (Harley *et al.*, 2009; Hokada *et al.*, 2016).

PETROGRAPHY

The granulite sample RG16–80 (68°50'50" S and 77°54'43" E) was collected from the Mather Peninsula in the Rauer Islands, East Antarctica (Fig. 1b). Compositions and mineral assemblages vary considerably at outcrop scale, ranging from quartz-bearing to SiO₂-undersaturated rocks (refer to Harley, 1998b for more details). The studied rocks occur as discrete rafts or

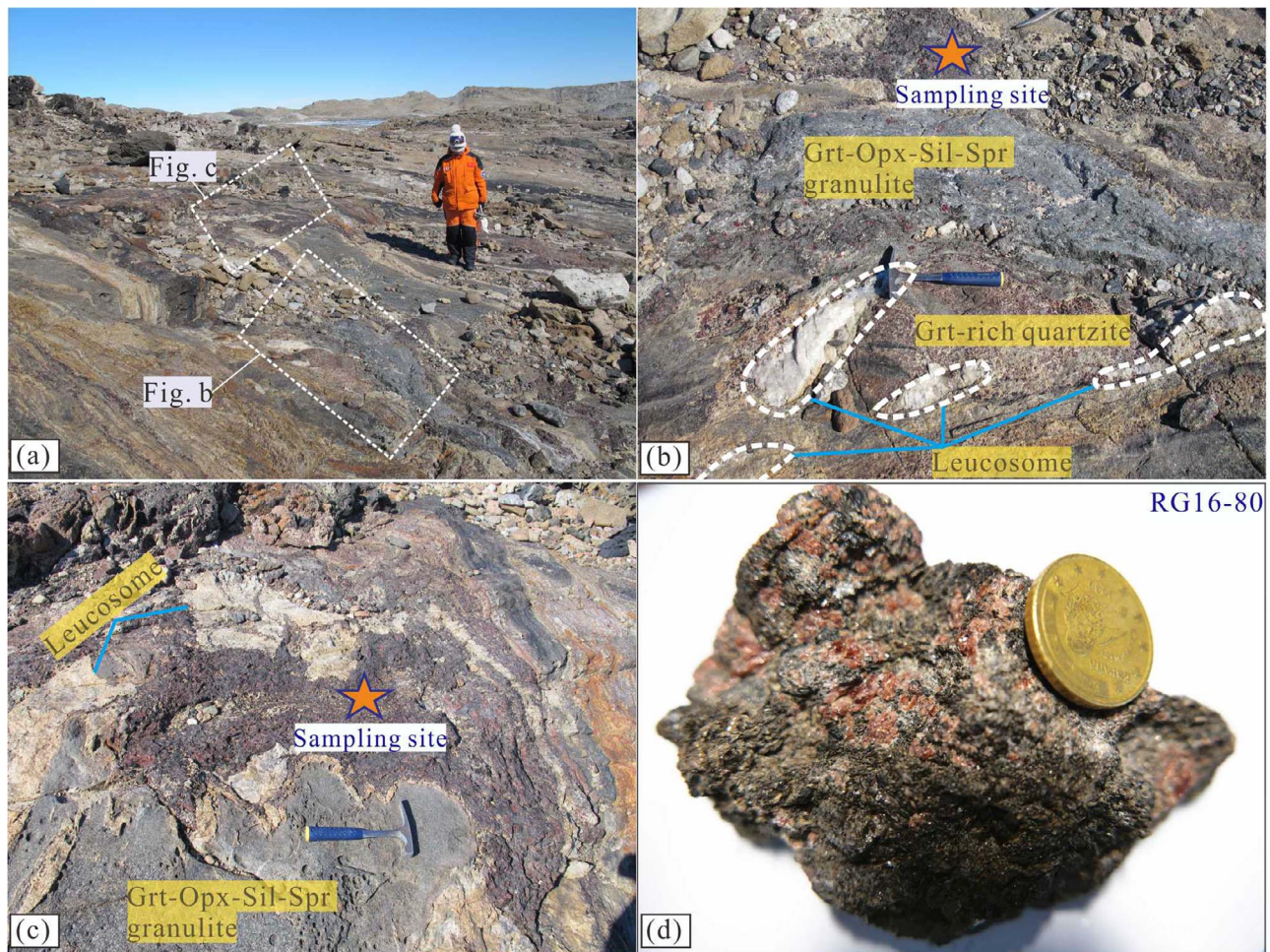


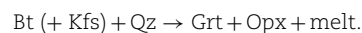
Fig. 2. (a) A field photograph showing the occurrence of UHT granulites from the Mather Peninsula, East Antarctica. (b,c) Enlarged photographs of white boxes in (a) displaying the presence of different lithological components. The sampling sites are shown by orange stars. The hammer for scale is 25 cm in length. (d) A representative hand specimen of the studied rocks. The coin for scale is about 2.4 cm in diameter.

boudins interlayering with Grt–Opx–Sil–Spr granulites, Grt-rich quartzites, Grt–Sil metapelites, two-pyroxene granulites, Opx–Sil–(Per)-bearing and Spr–Opx–Kfs-bearing leucogneisses (Fig. 2). Mineral abbreviations are from Warr (2021). The studied Mg-rich metapelites constitute 5–8% of the outcrop, and are roughly parallel to foliation S6 (Tong & Wilson, 2006), corresponding to the composite high-strain fabric described by Dirks & Wilson (1995). The samples are coarse-grained, with an assemblage of orthopyroxene (35–45%), biotite (25–30%), garnet (~15%), cordierite (7–10%), minor sapphirine, plagioclase, K-feldspar, quartz (<5%), and accessory spinel, ilmenite, apatite, zircon and monazite (Fig. 3).

Garnet porphyroblasts are subhedral to anhedral, with sizes varying from 0.1 to 30 mm. They usually preserve corroded biotite and quartz inclusions in cores. Most orthopyroxene grains are present as porphyroblasts (up to 1.2 cm in diameter), and in rare cases, they may contain platy or rounded biotite inclusions (Fig. 3a, b). The inclusions within garnet cores and orthopyroxene, such as biotite and quartz, are considered as pre-peak (M0) minerals. The cores of orthopyroxene porphyroblasts are considered to have been formed at M0 stage because they contain relatively low Al_2O_3 contents (<6.5 wt.%) compared with the rim (>8.0 wt.%; Op_{x1}) (see details on the section of mineral chemistry), and high Al_2O_3 contents in orthopyroxene usually suggest that they were formed under UHT conditions (e.g. Harley, 1998a).

Sometimes, orthopyroxene with high Al_2O_3 contents (up to 8.85–9.04 wt.%) may occur as inclusions within garnet mantle (Fig. 3a), and both minerals are considered to form at, or close to, peak conditions. Collectively, garnet mantles, orthopyroxene rims, high–Al orthopyroxene inclusions within garnets and biotite porphyroblasts (Fig. 3b) are interpreted to be formed at the metamorphic peak (M1) stage. K-feldspar occurs in very local domains, and is usually 0.5–4.0 mm in size (Fig. 3c). However, it is difficult to determine whether K-feldspar was formed at M0 or M1 stage based on petrographic observations.

The presence of biotite and quartz inclusions within garnets, coupled with the formation of garnet and orthopyroxene porphyroblasts, implies the progress of the following Bt – dehydration melting reaction:



Garnet porphyroblasts are commonly replaced at their margins by the intergrowth of orthopyroxene (Op_{x2}) + cordierite \pm biotite \pm plagioclase \pm spinel (Fig. 3d, e), which are assigned to the post-peak (M2) assemblage. Formation of the intergrowth is considered to result from the following decompression-related reaction:



Intergrowths of sapphirine + cordierite are found in the matrix (Fig. 3f). Occasionally, vermicular low-Al orthopyroxene and

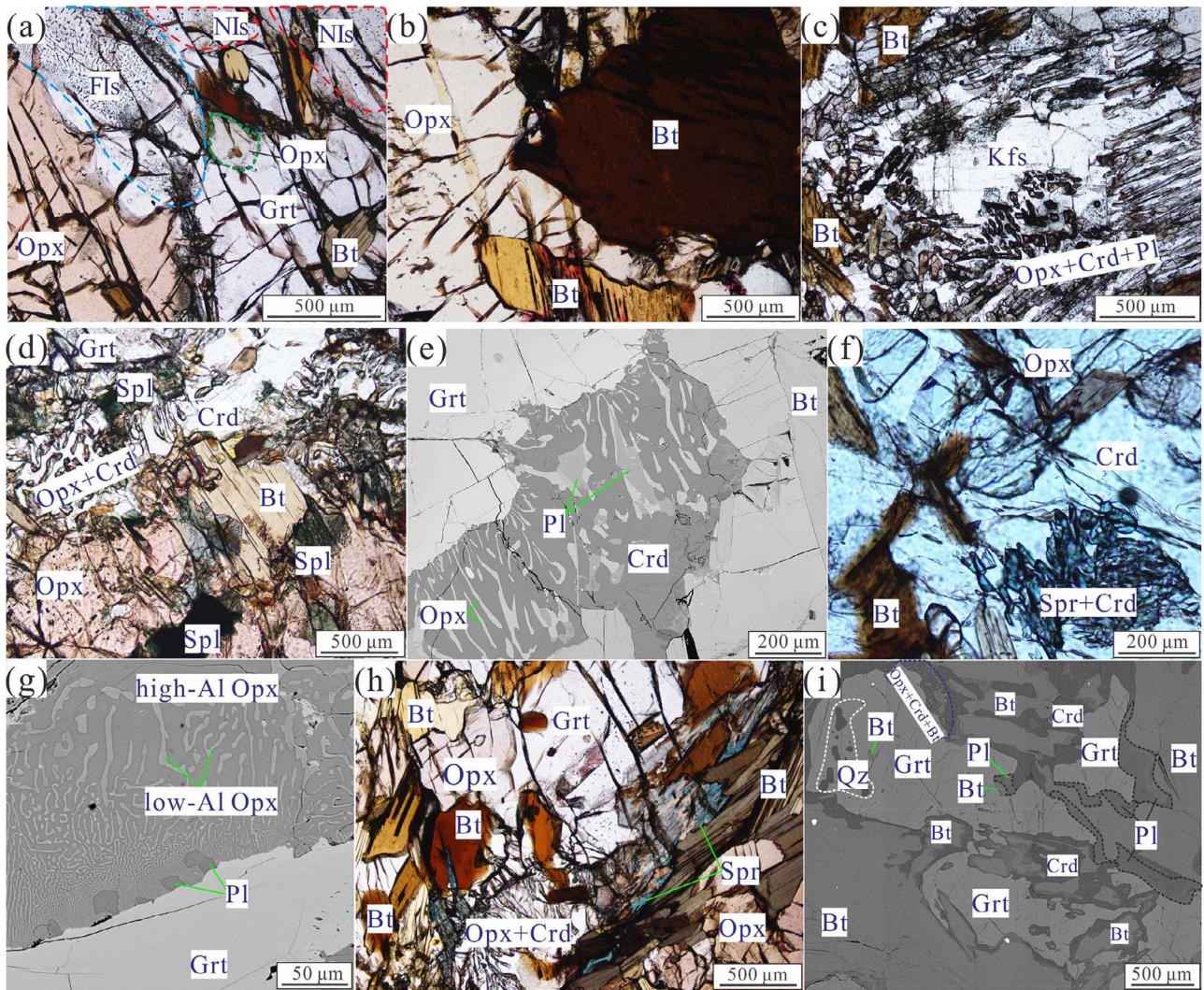


Fig. 3. Plane polarized light photomicrographs and BSE images of the Mather UHT granulite sample RG16-80. (a) Co-existence of garnet and orthopyroxene porphyroblasts. High-Al orthopyroxene inclusion within garnet is outlined by green dashed lines in the figure, which coexists with primary NIs (outlined by red dashed lines). Also shown in this figure are secondary FIs, highlighted with blue dashed lines. (b) Coexistence of biotite and orthopyroxene porphyroblasts, with a biotite inclusion within orthopyroxene. (c) K-feldspar grain rimmed by the intergrowth of orthopyroxene + cordierite + plagioclase. (d) Garnet and orthopyroxene porphyroblasts separated by fine-grained intergrowths of orthopyroxene + cordierite. (e) A BSE image showing the intergrowth of orthopyroxene + cordierite + plagioclase around garnets. (f) Intergrowths of sapphirine + cordierite in the matrix. (g) BSE image showing vermicular low-Al orthopyroxene in the rim of a high-Al orthopyroxene porphyroblast. (h) Biotite + plagioclase symplectites surrounding orthopyroxene. (i) Elongated garnet, cordierite, biotite, plagioclase in strongly deformed domains. Interstitial plagioclase is outlined by dashed black lines while quartz inclusions within garnets are highlighted with white dashed lines.

plagioclase are preserved in the rim of high-Al orthopyroxene porphyroblasts (Fig. 3g). These microstructures are also interpreted to form at the post-peak (M2) stage.

Some medium- to fine-grained sapphirine strips are associated with oriented biotite, which sometimes, may surround orthopyroxene + cordierite intergrowths (Fig. 3h). In strongly deformed domains, elongated garnet, cordierite, biotite, plagioclase, rare orthopyroxene and spinel grains show a similar orientation (Fig. 3i), which may have been formed at the final cooling (M3) stage. The appearance of interstitial plagioclase suggests the presence of former melts (Fig. 3i) (e.g. Holness *et al.*, 2011).

The presence of retrograde biotite and plagioclase is in agreement with progress of the following reaction:



In summary, the Spr-bearing UHT granulites (sample RG16-80) from the Mather Peninsula preserve four-stage metamor-

phic assemblages: (1) M0 stage is represented by the Bt-Qz-bearing inclusions and the cores of garnet and orthopyroxene porphyroblasts; (2) M1 stage is marked by the growth of garnet-orthopyroxene₁-biotite in the presence of melt, with or without the presence of K-feldspar; (3) M2 stage is featured by the development of orthopyroxene₂ + cordierite ± biotite ± plagioclase ± spinel and sapphirine + cordierite intergrowths surrounding the peak garnet or orthopyroxene; and (4) M3 stage is characterized by the growth of cordierite-biotite-plagioclase-bearing assemblages during the crystallization of melts.

ANALYTICAL METHODS

Back-scattered electron imaging, X-ray mapping and EPMA analysis

Analyses of major elements in minerals and back-scattered electron (BSE) imaging were conducted using JEOL JXA-8230 electron

probe microanalyzer (EPMA) at the Department of Geology, Northwest University, Xi'an, China. Additional X-ray mapping of NIs was done using JEOL JXA-8230 EPMA at the Wuhan Sample Solution Analytical Technology Co., Ltd., Wuhan, China. Analytical conditions were: an accelerating voltage of 15 kV, and a beam current of 2×10^{-8} A, a beam diameter of 2 μm . Counting times were 20 s on peaks, with 10 s on backgrounds. Data correction was processed by using a ZAF method. Fe^{3+} was calculated by charge balance. The analysis results are shown in Table 1. X-ray mapping was performed using an accelerating voltage of 20 kV, a beam current of 5×10^{-8} A, a beam diameter of 0.3 μm and a dwell time of 50 ms.

Trace elements in garnet

Trace element analyses of garnets were conducted with LA-ICP-MS at the Wuhan Sample Solution Analytical Technology Co., Ltd., Wuhan, China. Detailed operating conditions of the laser ablation system, the ICP-MS instrument and the data reduction were as same as in Zong *et al.* (2017). Laser sampling was performed using a GeolasPro laser ablation system that consists of a COMPexPro 102 ArF excimer laser and a MicroLas optical system. An Agilent 7700e ICP-MS instrument was used to acquire ion-signal intensities. A 'wire' signal smoothing device was included in this laser ablation system (Hu *et al.*, 2015). Spot size and frequency of the laser were set to be 44 μm and 5 Hz, respectively. Trace element compositions of garnets were calibrated based on various reference materials (BHVO-2G, BCR-2G and BIR-1G) without using an internal standard (Liu *et al.*, 2008). An Excel-based software ICPMSDataCal was used to perform quantitative calibration for trace element analyses (Liu *et al.*, 2008).

Raman measurements

Micro-Raman measurements were performed on representative thick sections at the School of Earth Science and Resources, Chang'an University, using a HORIBA LabRam HR Raman microspectrometer. A frequency-doubled Nd:YAG laser with 532 nm excitation wavelength was used. A petrographic microscope Olympus (50 \times objective) was used to focus the laser on the selected inclusions. Raman spectra in the ranges 100–4000 cm^{-1} were collected with an integration time of 10 s, and three accumulations. Data were processed using LabSpec v5.58.25 and the phases inside the inclusions were identified according to the data from the literature (Frezzotti *et al.*, 2012; Ferrero *et al.*, 2016, 2021b; Carvalho *et al.*, 2020; Gianola *et al.*, 2021).

Re-homogenization of nanogranitoids

Re-homogenization experiments of NIs were conducted following the procedure described in Bartoli *et al.* (2013a), using a piston cylinder apparatus at the CAS Key Laboratory of Crust-Mantle Materials and Environments, University of Science and Technology of China and the QUICKpress non-endloaded piston cylinder available at the Laboratory of Experimental Petrology and Geochemistry, Dipartimento di Geoscienze, Università di Padova. During each run, separated garnet grains were charged into Au capsules with external diameters of 3–5 mm and length of 5–10 mm. They were alternated by powdered silicon dioxide. The experiments were performed under the following conditions: 1000°C and 6 hours, 950°C and 4 hours, 950°C and 2 hours, and 950°C and 1 hour. A fixed pressure of 12 kbar was adopted during the experiments.

Major elements in nanogranitoids

Major element compositions of the re-homogenized NIs were determined using a JEOL JXA 8530F plus EPMA at the CAS Key Lab-

oratory of Crust-Mantle Materials and Environments, University of Science and Technology of China, Hefei, China. During EMPA analysis of hydrous granitic glasses, migration and loss of alkali from the inclusions may happen in the case that small beam sizes and high beam currents are adopted (e.g. Cesare *et al.*, 2015). To minimize the alkali loss, K_2O and Na_2O were determined at first. An anhydrous glass B (Morgan VI & London, 2005) was analyzed to obtain correction coefficients, which were used to correct the data of the unknown glasses. Analytical conditions were set as: a 15 kV accelerating voltage, a 5 nA beam current, variable beam diameters of 2–5 μm and counting times of 10 s on peak and 5 s on backgrounds.

Trace elements in nanogranitoids

Trace elements in NIs were analyzed at the State Key Laboratory for Mineral Deposit Research, Nanjing University, using a Coherent GeolasHD system equipped with a 193 nm excimer laser coupled to a NexION 350 ICP mass spectrometer (Pan *et al.*, 2019). The samples were loaded and analyzed in an in-house rhomb-shaped plexiglass cell of relatively small internal volume to enable fast washout of aerosol. The He-based aerosol was admixed with nebulizer gas via a T-junction prior to injection into the plasma. The ICP-MS was tuned to achieve maximum sensitivity and low oxide production rate by lowering the ThO/Th ratios to <0.5% before analyses. The analyzed inclusions are 2–25 μm beneath the surface to ensure they were representative of the original melts. The NIs close to the surface (2–10 μm in depth) were preferentially selected for analyses. In the rare case of deep-seated inclusions (15–25 μm in depth), the host garnet next to the inclusion was also analyzed and the ablation signal from the same depth interval as the inclusion was integrated, which will minimize the down-hole fractionation of elements during ablation (Zajacz & Halter, 2007). Repetition rate of the laser was increased to 10 Hz, on-sample energy densities of 5 J/cm^2 and spot size of 24 or 32 μm based on the size of NIs. Generally, measurement of each melt inclusion consists of 20–30 s instrumental background followed by 50–80 s ablations depending on the depth of melt inclusion. The glass NIST SRM 610 was employed as external standard. For the analyses of the NIST SRM 610, the spot size and energy density were set as 32 μm and 5 J/cm^2 , respectively. The acquired time-resolved signals were processed using the software SILLS (Guillong *et al.*, 2008). Due to the small size of NIs, part of the host garnet has also been ablated along with target inclusions. Therefore, deconvolution of the resulting mixed garnet-glass signal was applied following the procedures of Halter *et al.* (2002). The Al_2O_3 of the re-homogenized inclusions derived from EPMA was adopted as reference standards for inclusions and garnet hosts, and MgO was used as the matrix-only tracer. Uncertainties and calculated limits of detection increase with the concentrations of elements in the host, thus, some elements which are strongly compatible in the host garnets (e.g. Y, Gd, V, Zn, Sc and heavy rare earth elements–HREEs) are normally not detectable in the inclusions. Some inclusions show anomalously high concentrations of Zr, Th, U, Nb and Sr, suggesting the occurrence of accidental trapped phases (e.g. zircon, monazite, rutile and apatite) (Bartoli *et al.*, 2019). For these inclusions with extremely high contents of Zr, Th, U, Nb and Sr, only Rb, Cs, Ba and Li are reported in Table 4 and discussed in the text.

U–Pb dating of zircon

Conventional magnetic and heavy liquid techniques followed by hand-picking under a binocular microscope were used for separation of zircons from the Mather UHT metapelitic granulites.

Table 1: Representative mineral compositions of the Mather UHT granulites. The major element compositions are shown in wt.%

	Grt-c	Grt-m	Grt-r	Grt ₃	Opx _{inGrt}	Opx-c	Opx-r	Opx _{exsolved}	Opx _{inter}	Opx ₃	Spr _{withCrd}	Spr _{withBt}	Crd _{inter}	Bt _{inGrt}	Bt _{porphy}	Bt _{inter}	Bt-3	Pl _{inter}	Pl ₃	Spl	Pl-in Zrn	Bt-in Zrn	Opx-in Zrn
SiO ₂	40.94	40.51	40.63	39.94	51.14	52.34	49.11	51.07	50.20	50.68	13.27	13.41	49.20	39.71	36.67	36.69	37.48	47.03	55.19	0.04	59.87	38.51	50.28
TiO ₂	0.03	0.06	0.00	0.00	0.04	0.07	0.08	0.11	0.09	0.06	0.05	0.10	0.03	3.47	4.08	4.32	4.12	0.00	0.00	0.04	0.01	3.54	0.05
Al ₂ O ₃	23.23	23.01	23.11	22.38	8.85	6.49	9.16	6.88	7.37	7.67	62.22	61.34	34.03	15.49	16.93	17.13	16.88	33.73	29.04	61.11	24.95	16.96	6.79
Cr ₂ O ₃	0.12	0.10	0.09	0.01	0.10	0.03	0.03	0.07	0.12	0.07	0.32	0.49	0.06	0.09	0.13	0.25	0.10	0.00	0.00	1.28	0.06	0.08	0.11
Fe ₂ O ₃	17.26	18.17	19.73	23.84	12.04	13.06	14.93	15.21	15.24	15.49	7.79	8.51	2.61	5.44	8.25	8.92	8.68	0.24	0.25	21.85	0.03	11.53	17.62
MnO	0.18	0.16	0.26	0.38	0.04	0.03	0.10	0.07	0.08	0.11	0.04	0.06	0.03	0.00	0.02	0.00	0.01	0.02	0.01	0.08	0.06	0.06	0.08
MgO	16.87	15.96	15.00	11.81	27.31	27.61	25.58	26.23	27.00	25.17	16.26	16.70	12.60	21.90	18.24	17.79	18.16	0.00	0.13	12.71	0.00	15.58	24.69
CaO	1.47	1.18	1.63	1.78	0.10	0.09	0.11	0.12	0.09	0.07	0.00	0.02	0.03	0.02	0.02	0.00	0.00	16.46	10.40	0.00	7.15	0.00	0.10
Na ₂ O	0.01	0.03	0.02	0.02	0.05	0.00	0.00	0.03	0.02	0.04	0.02	0.00	0.04	0.43	0.44	0.33	0.41	2.14	5.33	0.08	7.24	0.12	0.01
K ₂ O	0.00	0.00	0.00	0.01	0.00	0.00	0.01	0.00	0.00	0.03	0.00	0.00	0.01	9.46	9.96	9.52	9.85	0.03	0.53	0.02	0.16	8.56	0.00
ZnO	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	1.37	-	-	-
F	-	-	-	-	-	-	-	-	-	-	-	-	-	0.37	0.09	0.29	0.22	-	-	-	-	-	-
Cl	-	-	-	-	-	-	-	-	-	-	-	-	-	0.10	0.12	0.11	0.12	-	-	-	-	-	-
Total	100.11	99.18	100.47	100.17	99.67	99.72	99.09	99.79	100.21	99.39	99.97	100.63	98.63	96.49	94.94	95.34	96.03	99.66	100.87	98.58	99.52	94.95	99.72
O	12	12	12	12	6	6	6	6	6	6	20	20	18	11	11	11	11	8	8	4	8	11	6
Si	2.984	2.995	2.987	3.009	1.815	1.864	1.773	1.834	1.791	1.833	1.578	1.587	4.921	2.802	2.681	2.676	2.710	2.165	2.468	0.001	2.679	2.807	1.826
Ti	0.002	0.003	0.000	0.000	0.001	0.002	0.002	0.003	0.002	0.002	0.004	0.009	0.002	0.184	0.224	0.237	0.224	0.000	0.000	0.001	0.000	0.194	0.001
Al	1.996	2.005	2.003	1.988	0.370	0.272	0.390	0.291	0.310	0.327	8.723	8.561	4.012	1.289	1.459	1.472	1.439	1.831	1.531	1.936	1.316	1.458	0.291
Cr	0.007	0.006	0.005	0.001	0.003	0.001	0.001	0.002	0.003	0.002	0.030	0.046	0.005	0.005	0.008	0.015	0.006	0.000	0.000	0.027	0.002	0.004	0.003
Fe ³⁺	0.028	0.000	0.020	0.000	0.000	0.000	0.060	0.035	0.091	0.005	0.087	0.201	0.044	0.000	0.000	0.000	0.000	0.009	0.009	0.037	0.001	0.000	0.051
Fe ²⁺	1.024	1.123	1.193	1.502	0.357	0.389	0.391	0.422	0.364	0.464	0.688	0.642	0.175	0.321	0.504	0.544	0.525	0.000	0.000	0.456	0.000	0.703	0.484
Mn	0.011	0.010	0.016	0.024	0.001	0.001	0.003	0.002	0.002	0.003	0.004	0.006	0.003	0.000	0.001	0.000	0.001	0.001	0.000	0.002	0.001	0.004	0.002
Mg	1.832	1.758	1.644	1.326	1.444	1.465	1.376	1.404	1.436	1.357	2.882	2.946	1.878	2.303	1.987	1.934	1.957	0.000	0.009	0.509	0.000	1.692	0.004
Ca	0.115	0.093	0.128	0.144	0.004	0.003	0.004	0.005	0.003	0.003	0.000	0.003	0.003	0.002	0.002	0.000	0.000	0.812	0.498	0.000	0.343	0.000	0.004
Na	0.001	0.004	0.003	0.003	0.003	0.000	0.000	0.002	0.001	0.003	0.005	0.000	0.008	0.058	0.063	0.047	0.058	0.191	0.462	0.004	0.628	0.017	0.000
K	0.000	0.000	0.000	0.001	0.000	0.000	0.000	0.000	0.000	0.001	0.000	0.000	0.001	0.852	0.929	0.885	0.909	0.002	0.030	0.001	0.009	0.796	0.000
Zn	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	0.027	-	-	-
F	-	-	-	-	-	-	-	-	-	-	-	-	-	0.08	0.02	0.07	0.05	-	-	-	-	-	-
Cl	-	-	-	-	-	-	-	-	-	-	-	-	-	0.01	0.02	0.01	0.02	-	-	-	-	-	-
Total	8.000	7.997	7.999	7.998	3.998	3.997	4.000	4.000	4.003	4.000	14.001	14.001	11.052	7.911	7.893	7.888	7.893	5.011	5.007	3.002	4.979	7.675	3.999
Alm	34	38	40	50																			
X _{Mg}	0.64	0.61	0.58	0.47	0.80	0.79	0.78	0.77	0.80	0.75	0.81	0.82	0.91	0.88	0.80	0.78	0.79	0.81	0.50	0.53	0.35	0.71	0.73
X _{Aln}																							

Abbreviations: c, core; m, mantle; r, rim; in, inclusions; inter, intergrowth; porphy, porphyroblast; 3, minerals formed at M3 stage.

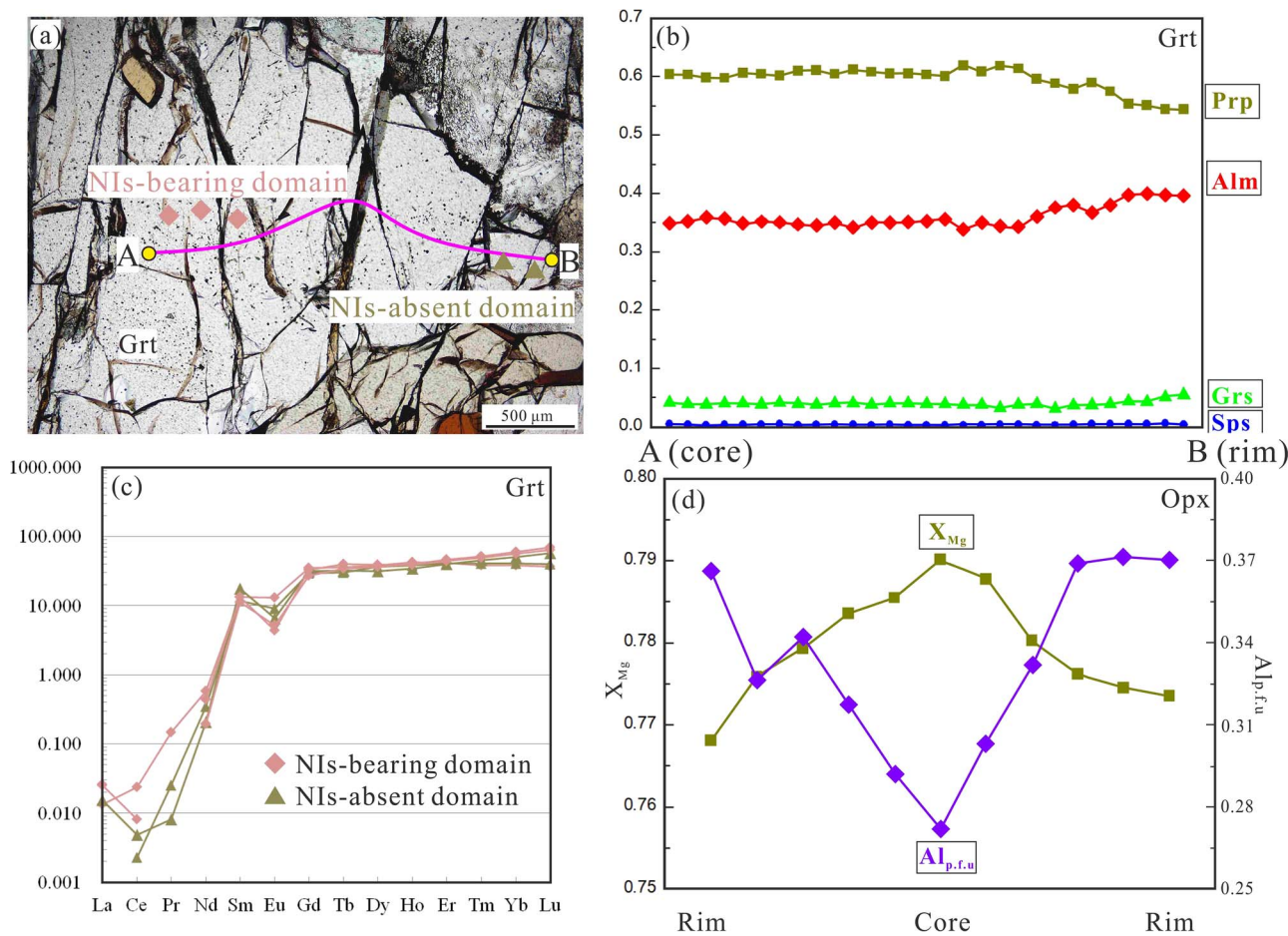


Fig. 4. (a) Garnet grain showing the analysis positions. (b) Compositional zoning profile across garnet from core to rim. (c) Chondrite-normalized REE patterns of garnets for the NI-bearing and NI-absent domains. (d) Compositional zoning profiles across an orthopyroxene porphyroblast.

Morphology and internal structure of the zircons were documented with transmitted, reflected light microphotographs and cathodoluminescence (CL) images. Zircon U–Pb dating was carried out using LA–ICP–MS at the Wuhan Sample Solution Analytical Technology Co., Ltd., Wuhan, China. Detailed operating conditions for the laser ablation system and the ICP–MS instrument and data reduction were described in Zong *et al.* (2017). Laser sampling was performed using a GeolasPro laser ablation system that consists of a COMPexPro 102 ArF excimer laser (wavelength of 193 nm and maximum energy of 200 mJ) and a MicroLas optical system. Each analysis incorporated a background acquisition of approximately 20–30 s followed by 50 s of data acquisition from the sample. Spot size and frequency of the laser for analyses were set to 24 mm and 10 Hz, respectively. Zircon 91500 and glass NIST610 were used respectively as external standards for U–Pb dating and trace element calibrations. In order to monitor the external uncertainties of zircon dating, a zircon standard GJ–1 was alternately analyzed together with the other unknown zircons. Fourteen measurements of the zircon GJ–1 yielded a weighted mean $^{206}\text{Pb}/^{238}\text{U}$ age of 602.1 ± 4.4 Ma (MSWD = 0.22), which is roughly in agreement with the recommended $^{206}\text{Pb}/^{238}\text{U}$ age value of 600.4 ± 0.6 Ma (Jackson *et al.*, 2004). An Excel-based software ICPMSDataCal was used to perform off-line selection and integration of background and analyzed signals, time-drift correction and quantitative calibration for trace element analyses and U–Pb dating (Liu *et al.*, 2010). Programs SQUID 1.0 and ISOPLOT (V.3.0; Ludwig, 1999) were used for data processing.

RESULTS

Mineral chemistry

Garnet porphyroblasts are chemically homogeneous from core to mantle ($\text{Alm}_{34-38}\text{Prp}_{59-62}\text{Grs}_{3-4}\text{Sps}_{0-1}$). Small garnet crystals and garnet porphyroblast rims have slightly lower pyrope contents, with compositions of $\text{Alm}_{40-46}\text{Prp}_{55-56}\text{Grs}_{4-3}\text{Sps}_{0-1}$ (Fig. 4a, b). The garnet relicts from the strongly deformed domain display the lowest magnesium contents ($\text{Alm}_{48-50}\text{Prp}_{46-44}\text{Grs}_{4-5}\text{Sps}_{0-1}$). NI-bearing and NI-absent domains display similar REE patterns in the chondrite-normalized diagram, with enrichments of HREEs and slightly negative Eu anomalies (Fig. 4c).

Orthopyroxene porphyroblasts preserve evident Al-zoning patterns, with y_{Al} ($= \text{Al}^{\text{IV}} = \text{Al}/2$) values increasing from 0.14 ($\text{Al}_2\text{O}_3 = \sim 6.49$ wt.%) in the core to 0.19 ($\text{Al}_2\text{O}_3 = \sim 8.79$ wt.%) in the rim (Fig. 4d). The highest Al_2O_3 content of orthopyroxene detected in the sample is ~ 9.16 wt.%, with corresponding y_{Al} value of 0.19. However, orthopyroxene porphyroblasts show relatively restricted X_{Mg} ($X_{\text{Mg}} = \text{Mg}/(\text{Mg} + \text{Fe}^{2+})$) variations (0.77–0.79). Orthopyroxene inclusions in garnet have Al_2O_3 contents of 8.85–9.04 wt.% ($y_{\text{Al}} = \sim 0.19$) and X_{Mg} values of 0.78–0.80. Orthopyroxene from the orthopyroxene–cordierite symplectites have y_{Al} of 0.14–0.16 and X_{Mg} of 0.75–0.80. Vermicular low-Al orthopyroxene in the rims of some orthopyroxene porphyroblasts displays Al_2O_3 contents of ~ 6.88 wt.% ($y_{\text{Al}} = \sim 0.15$) and X_{Mg} values of ~ 0.77 . Orthopyroxene grains associated with the retrograde biotite display Al_2O_3 contents of 7.67–7.69 ($y_{\text{Al}} = 0.16$ –0.17) and X_{Mg} of 0.74–0.76.

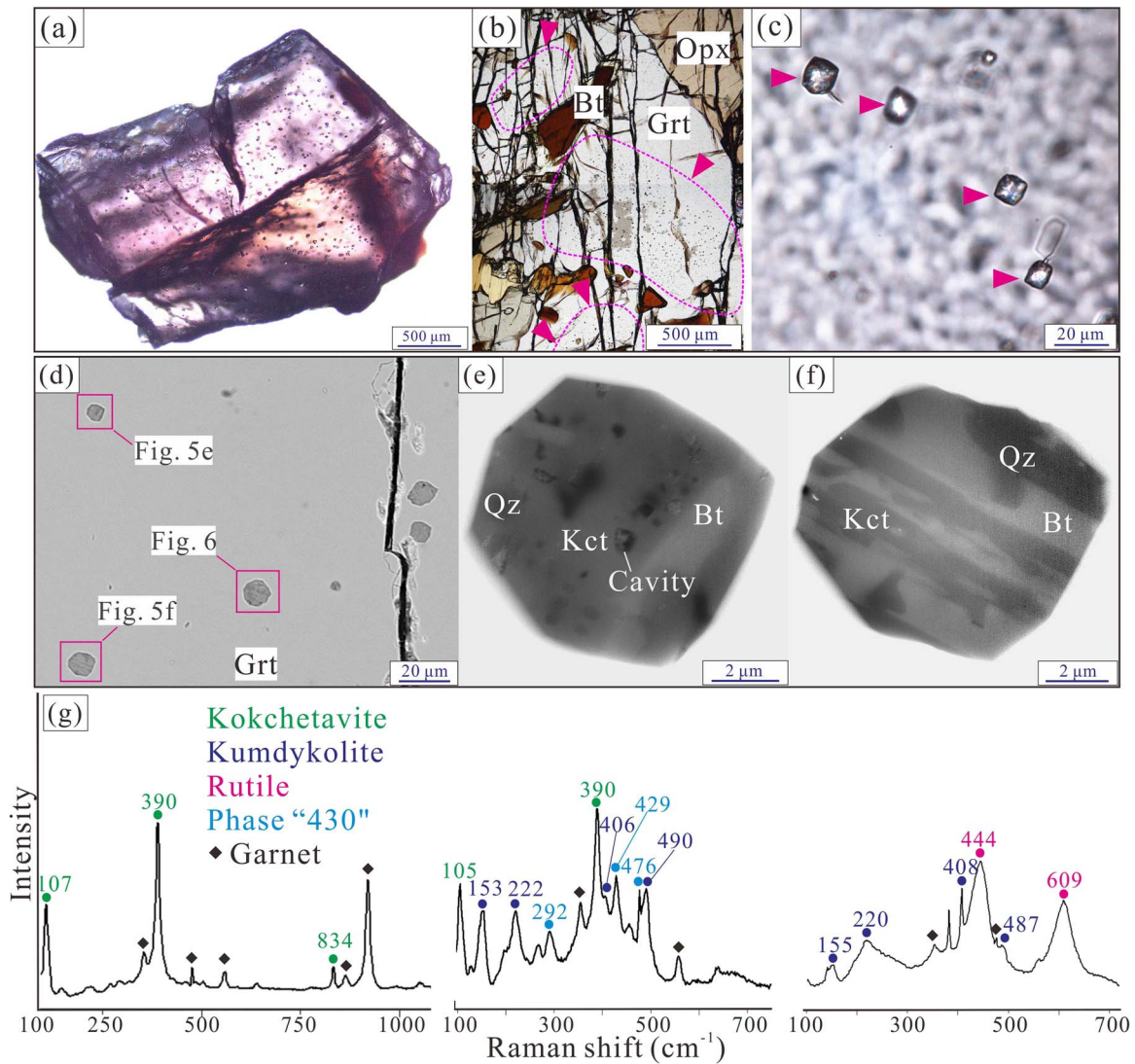


Fig. 5. Plane polarized light photomicrographs and BSE images of NIs within garnet porphyroblasts and Raman spectrum of these inclusions. (a) Garnet crystal mounted on an epoxy resin disc containing abundant NIs. (b,c) Plane polarized photomicrographs showing NIs within peritectic garnets. (d–f) BSE images showing NIs with daughter phases of quartz, biotite and kokchetavite. Some cavities are also observed and might be related to the exsolution of fluid during the retrograde crystallization of melt. (g) Mixed Raman spectrum of NIs and their garnet hosts.

Biotite porphyroblasts show relatively homogeneous compositions, with TiO₂ contents of 4.29–4.61 wt.% and X_{Mg} of 0.79–0.81. The low TiO₂ contents for peak biotite can be partially ascribed to crystal-chemical constraints, which hamper the temperature control on Ti content of biotite (Cesare *et al.*, 2008). Biotite inclusions within garnet have relatively higher X_{Mg} values (0.84–0.87) than those within orthopyroxene (~0.79). However, they show similar TiO₂ content variations ranging from 2.90 to 4.46 wt.%. The retrograde biotite has TiO₂ contents of ~2.63 wt.% and X_{Mg} values of ~0.81. Halogen concentrations in the analyzed biotite crystals are undistinguishable, with F and Cl contents of 0.09–0.37 wt.% and 0.10–0.12 wt.%, respectively.

Sapphirine associated with retrograde biotite has slightly higher X_{Mg} values (0.84–0.86) than that from sapphirine–cordierite intergrowths (0.80–0.82), both of which show indistinguishable Fe³⁺/Fe^T ratios varying from 0.71 to 0.81. Cordierite grains from different positions show restricted X_{Mg} variations from 0.90 to 0.92. Spinel has X_{Mg} of 0.43–0.48 and ZnO contents of 0.64–1.72 wt.%. Plagioclase from the orthopyroxene–cordierite intergrowths is rich in anorthite, with a typical X_{An} (= Ca/(Ca + Na + K))

of ~0.81. However, plagioclase associated with the retrograde biotite has lower X_{An} values of 0.51–0.58.

Microstructures of melt and fluid inclusions

Nanogranitoids are randomly distributed within garnet crystals, but absent in the outer rims (Figs 3a, 4a, 5a). They are also absent in the small garnet crystals from the strongly deformed domains (Fig. 3i). These inclusions show spherical or polyhedral shape, and are 5–15 μm in size (Figs 5b–f, 6). They usually display negative crystal shapes, and may contain nano-scale porosities (Fig. 5e), implying the possible presence of fluid which was dissolved in the former melts and subsequently exsolved upon crystallization (Bartoli *et al.*, 2013b). Coupled Raman analyses and compositional maps suggest that they contain daughter phases of quartz, kokchetavite (KAlSi₃O₈), minor kumdykolite (NaAlSi₃O₈) and biotite (Figs 5g, 6), indicating the former presence of silicate melts. Moreover, an unknown phase with Raman peaks at 292, 429 and 476 cm⁻¹ has also been detected (Fig. 5g). Such phase has been previously recognized within garnet–hosted melt inclusions from the Gruf Complex (Gianola *et al.*, 2021) and the Central Maine

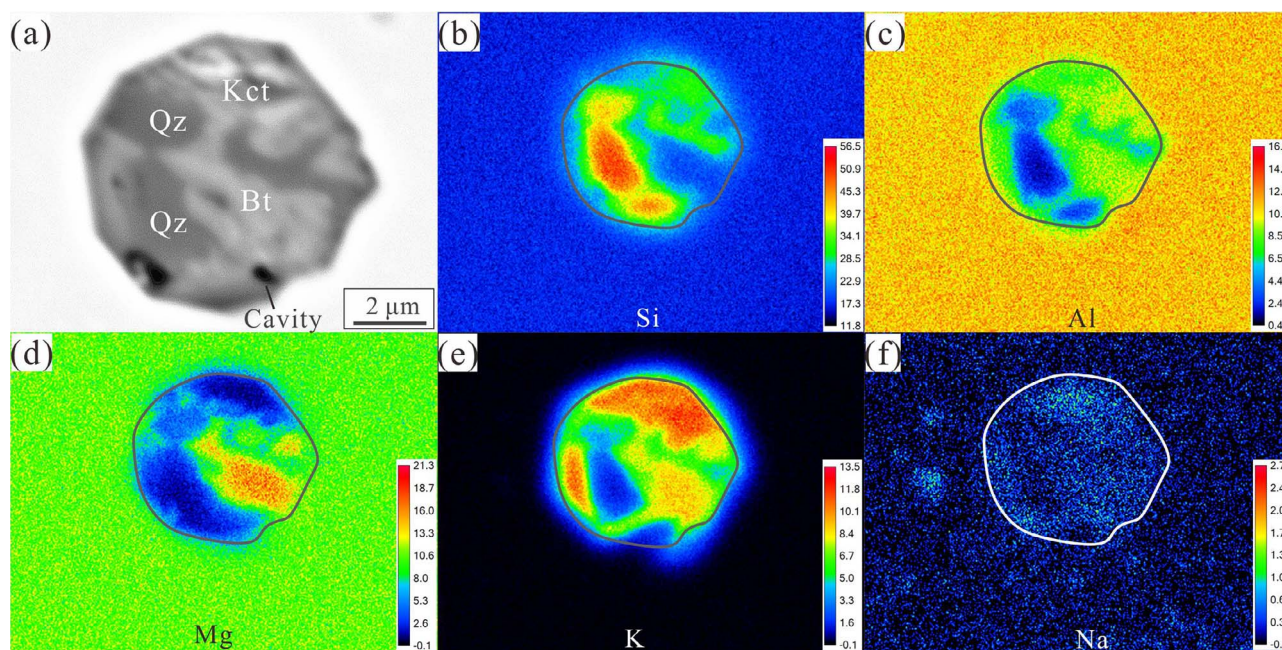


Fig. 6. A BSE image of a nanogranitoid inclusion and corresponding X-ray maps of Si, Al, Mg, K and Na.

Terrane (Ferrero *et al.*, 2021b). This phase was described as phase '430' by Ferrero *et al.* (2021b). Apatite, rutile, zircon and monazite may be present as accidentally trapped phases, as indicated by the relatively large size of trapped phases compared with the size of the melt inclusions, and also by their low solubility in granitic melts (e.g. Bartoli *et al.*, 2019). Daughter biotite crystals in NIs have very low TiO₂ contents of 0.03–0.50, and is characterized by high X_{Mg} values ranging from 0.92 to 0.95, whereas F and Cl contents in biotite are negligible, lower than 0.07 and 0.03 wt.%, respectively. Kokchetavite has a typical composition of Or₉₄Ab₆ and kumdykolite displays compositions of Or₅An₈Ab₈₇. For detailed mineral compositions, please refer to Table 2.

Fluid inclusions are also found within garnet (Figs 3a, 7a, b) and orthopyroxene (Fig. 7c). These fluid inclusions display granular or vermicular shapes, with sizes of 5–40 μm (Fig. 7). They are usually present along healed cracks, suggesting that they are secondary in origin. Some early trapped fluid has reacted with their hosts to produce stepdaughter phases (i.e. their precipitation results from fluid–host interaction; Carvalho *et al.*, 2020) of pyrophyllite, magnesite, corundum and quartz, with or without residual CO₂ (Fig. 7d, e). The measured CO₂ Fermi diad splits range from 102.8 to 105.1 cm⁻¹, corresponding to densities of 0.2–1.0 g/cm³ calculated with the densimeter of Wang *et al.* (2011). Secondary pure CO₂ inclusions may also occur along healed garnet cracks (Fig. 7f), which have a density of ~0.8 g/cm³. Although not detected in this study, the existence of trace amounts of free H₂O cannot be precluded because H₂O-rich liquid may be present as a thin film (<0.1 μm) wetting the walls of the FIs at ambient conditions (e.g. Berkesi *et al.*, 2009; Lamadrid *et al.*, 2014). Other fluid species such as N₂ and CH₄ have not been detected in this study.

Microstructures of re – homogenized nanogranitoids

Experiments at 950°C with run durations of 2–4 hours yielded some homogenized melt inclusions. However, those at 950°C with run duration of 1 hour produced the largest proportion of perfectly re – homogenized inclusions (Fig. 8). Some inclusions show clear

Table 2: Compositions of daughter phases within NIs. The major element compositions are shown in wt.%

	Bt	Kok	Kum
SiO ₂	39.22	75.17	66.34
TiO ₂	0.50	0.00	0.03
Al ₂ O ₃	19.22	11.89	19.5
Cr ₂ O ₃	0.18	0.00	0.01
Fe ^T O	2.88	0.75	0.83
MnO	0.02	0.03	0
MgO	23.88	0.04	1.67
CaO	0.06	0.02	1.46
Na ₂ O	0.73	0.44	8.38
K ₂ O	9.29	10.85	0.71
F	0.00	–	–
Cl	0.02	–	–
Total	95.99	99.21	98.93
O	11	8	8
Si	2.729	3.345	2.934
Ti	0.026	0.000	0.001
Al	1.576	0.624	1.017
Cr	0.010	0.000	0.000
Fe ³⁺	0.025	0.028	0.031
Fe ²⁺	0.142	0.000	0.000
MnO	0.001	0.001	0.000
Mg	2.477	0.003	0.110
Ca	0.004	0.001	0.069
Na	0.098	0.038	0.719
K	0.824	0.616	0.040
F	0.000	–	–
Cl	0.002	–	–
Total	7.914	4.656	4.921
X _{Mg}	0.95		
X _{An}		0.94	0.08

evidence for overheating, such as the occurrences of (1) irregular boundaries between inclusions and garnet hosts (Fig. 8a); (2) secondary minerals (i.e. orthopyroxene) formed during experiments or upon quenching (Fig. 8a–d); (3) new growth of garnet on the

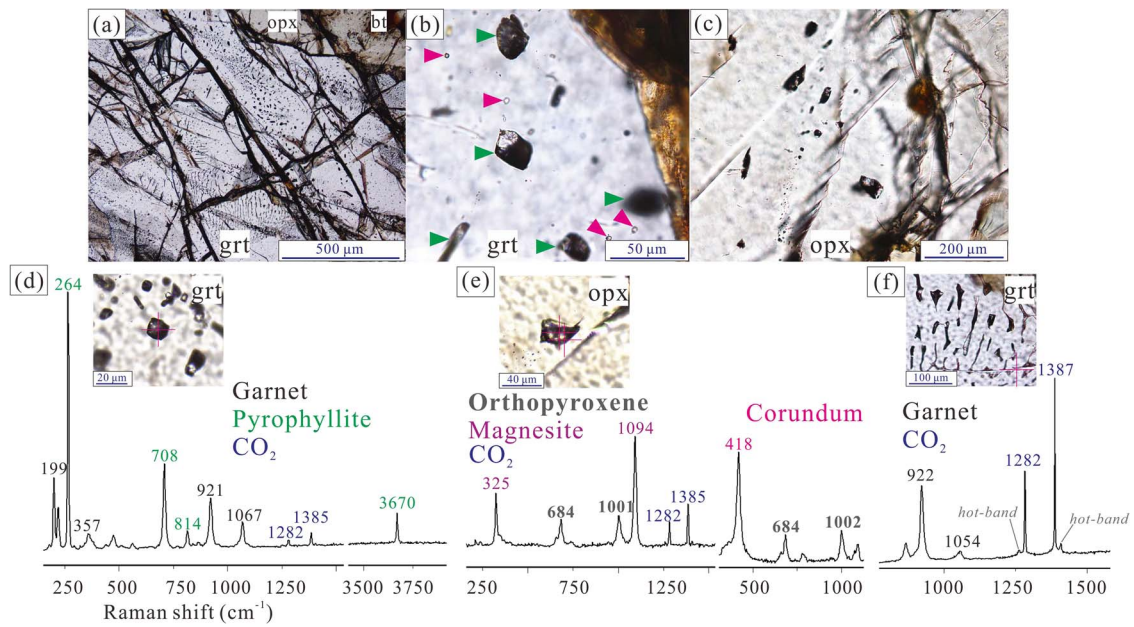


Fig. 7. (a–c) Secondary FIs within garnet and orthopyroxene porphyroblasts. Pink and green arrows respectively point to nanogranitoid and fluid inclusions, where NIs are randomly distributed while FIs are mostly present along the cracks. (d–f) Mixed spectra of FIs and their hosts.

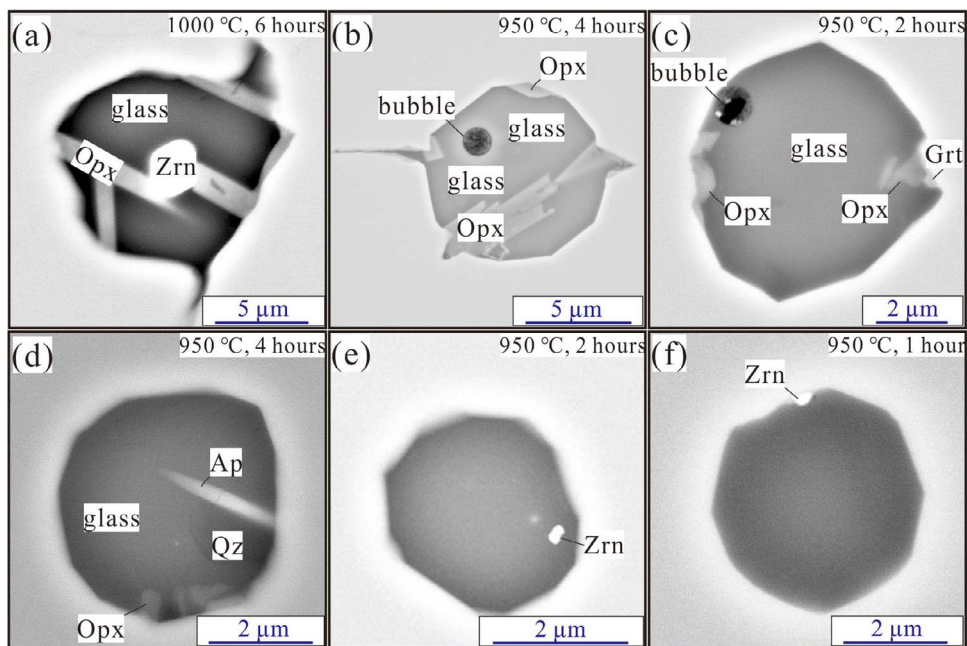


Fig. 8. BSE images of experimentally re-homogenized NIs under 1000°C and 12 kbar with a run duration of 6 hours (a), 950°C and 12 kbar with run durations of 4 hours (b, d), 2 hours (c, e), and 1 hour (f). Perfectly re-homogenized inclusions with well-developed negative shapes are shown in (e) and (f). Other inclusions show clear evidence for overheating (a–d).

inclusion walls (Fig. 8c); and (4) offshoots around melt inclusions formed as a consequence of decrepitations (Fig. 8a, b). The compositions of these inclusions showing interaction with the host have been disregarded. Shrinkage bubbles have also been observed within the overheated inclusions (Fig. 8b, c), suggesting that the fluid may have undergone incomplete dissolution in the melts during the experimental run or, alternatively, it diffused during cooling to form shrinkage bubbles (Lowenstern, 1995; Bartoli *et al.*, 2013a). Some inclusions were not re-homogenized properly during the experiments, as indicated by the coexistence of melts with rounded residual quartz (Fig. 8d). Some melt inclusions still

contain accessory minerals such as apatite, rutile, zircon and monazite (Fig. 8a, d–f). This strongly indicates that these phases are accidentally trapped minerals (Ferrero *et al.*, 2012).

Major element compositions of re-homogenized nanogranitoids

Re-homogenized inclusions are strongly peraluminous ($ASI = [Al_2O_3 / (CaO + Na_2O + K_2O)] = 1.24–1.61$) (Fig. 9) and rhyolitic in composition, with high SiO_2 contents of 69.89–75.61 wt.%, high $Na_2O + K_2O$ contents of 7.08–9.53 wt.% ($K/Na = 2.21–9.31$) and extremely low CaO (< 0.7 wt.%) and $FeO + MgO$ contents (Table 3).

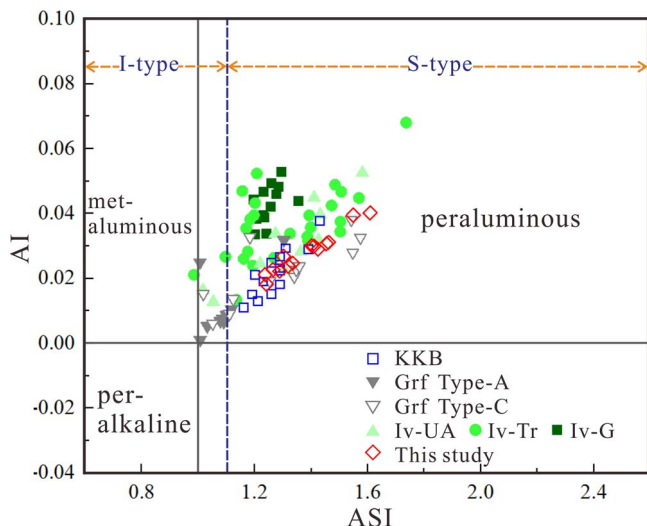


Fig. 9. Plot of aluminum saturation index vs. alkalinity index [$AI = \text{molar } Al_2O_3 - (\text{Na}_2O + \text{K}_2O)$] (from Frost & Frost, 2008). Dotted line separates the compositional fields of I- and S-type granites (from Chappell, 1999). KKB, Kerala Khondalite Belt (India) (Cesare et al., 2009; Ferrero et al., 2012), with peak P-T conditions of 900°C and 6–8 kbar (Cenki et al., 2002; Shabeer et al., 2005); Grf Type-A and -C, type-A or -C granulites from the Gruf Complex (European Central Alps) (Gianola et al., 2021), with peak P-T conditions of 920–940°C and 8.5–9.5 kbar (Galli et al., 2012); Iv, metapelitic migmatites from the Ivrea Zone (NW Italy) (Carvalho et al., 2019), with P-T conditions of 650–730°C and 3.5–6.5 kbar for the UA, and 900–950°C and 11–12 kbar for the G (Ewing et al., 2013; Redler et al., 2013). UA, upper amphibolite facies zone; Tr, transition zone; G, granulite facies zone.

Difference of EMPA totals from 100% suggest that the H_2O contents in the analyzed MIs are 1.49 to 4.24 wt.%, with an average value of 2.79 wt.%.

In the CIPW-normative Qz–Ab–Or diagram (Fig. 10a), the high- SiO_2 , potassic to ultrapotassic melts plot far away from haplogranitic minimum melts, and define an elongated trend paralleling to the Qz–Or sideline, which are consistent with the melt compositions found in other UHT occurrences reported by Cesare et al. (2009), Gianola et al. (2021) and Ferrero et al. (2012). Similar compositions have also been obtained by melting experiments of pelitic protoliths at 5–7 kbar and 900–950°C (Patiño Douce & Johnston, 1991; Droop et al., 2003) (Fig. 10a). In the Ab–An–Or diagram, the NIs show variable Or/Ab ratios and low An contents. They plot in the granite field, and are close to the boundary between granite and Qz–monzonite (Fig. 10b).

Trace element compositions of nanogranitoids

In the average upper continental crust (UCC) normalized diagram (Rudnick & Gao, 2014), NIs from the Mather Peninsula are enriched in Rb, Sm, Ta and depleted in Ba, Pb, Th, Ce and Sr (Fig. 11). Compared with the granulitic restite (i.e. granulites that have experienced partial melting and substantial melt loss), NIs are enriched in Li (5.3–76.9 ppm), Cs (0.9–35.5 ppm), Rb (126.7–1325.0 ppm), Ta (1.4–3.2 ppm), Sm (5.5–27.2 ppm), Zr (84.2–276.5 ppm), Nd (7.2–22.8 ppm), and depleted in Ce (2.4–13.4 ppm), Th (0.4–3.8 ppm), Ba (56.3–565.6 ppm), Sr (5.8–27.6 ppm) and Nb (4.0–15.7 ppm) (Table 4; Fig. 11). The melts have relatively higher U (0.6–9.3 ppm) and Pb contents (3.9–12.8 ppm), and display higher Rb/Sr (7.69–52.08) and Sm/Nd (0.52–1.14) ratios relative to the granulitic restite (Rb/Sr = ~8.56; Sm/Nd = ~0.25). They also contain variable amounts of Sc (9.7–42.0 ppm), V (29.4–146.6 ppm) and Zn (83.3–130.7 ppm), which are discarded in

this study because these concentrations might be affected by contamination from the garnet hosts (Bartoli et al., 2019).

Zircon morphology, U–Pb dates and trace elements

Zircon grains from the Mather UHT metapelitic granulites are usually rounded, and have grain sizes of 80–250 μm . They typically display complex core–mantle–rim structures and occasionally, they may be homogeneous in the CL images (Fig. 12a). The cores are generally 30–120 μm in diameter, and show oscillatory zoning or homogeneous patterns. Most zircon cores are enclosed by dark-grey mantles, which usually show blurred zoning or are unzoned. Sometimes, zircon cores and mantles are enveloped by pale-grey to bright rims (Fig. 12a). A total of 46 analyses of zircon were conducted on 34 grains. Zircon cores yield inherited $^{207}\text{Pb}/^{206}\text{Pb}$ dates of 2623–2088 Ma ($N = 13$; Th/U = 0.28–1.81). Zircon mantles document $^{206}\text{Pb}/^{238}\text{U}$ dates ranging from 1050 to 607 Ma ($N = 27$; Th/U = 0.01–0.09) while the rims record $^{206}\text{Pb}/^{238}\text{U}$ dates varying from 569 to 472 Ma ($N = 6$; Th/U = 0.11–0.34) (Table 5).

Orthopyroxene, biotite, plagioclase, apatite, chlorite, quartz and monazite inclusions were observed within zircons (Fig. 12c). Some trivial inclusions are extensively present along the healed fractures in zircons (Fig. 12b, d). They cannot be analyzed due to their small sizes. However, their appearance resembles Fls reported in literature (Fig. 6d in Li et al., 2018). Thus, these inclusions are suspected to also be Fls. Accordingly, the large variations of U–Pb dates for zircon mantles and rims (Fig. 12e) and Pb loss could result from extensive fluid infiltration and the Pan-African HT–UHT overprinting. Zircon cores are characterized by the presence of low-Mg biotite ($X_{Mg} = \sim 0.71$) and low-Ca plagioclase ($X_{An} = \sim 0.35$) inclusions (Table 1), which were not found in the matrix, suggesting that these cores are detrital in origin. It is worth noting that an orthopyroxene grain enclosed in a detrital zircon (i.e. it shows a near concordant date of ~2381 Ma) shows high Al_2O_3 contents of ~6.79 wt.% (Fig. 12c), indicative of a possible high-grade metamorphism in the Paleoproterozoic.

PHASE EQUILIBRIA MODELING

Phase equilibria modeling for the Mather UHT metapelitic granulite sample (RG16–80) was conducted with Perple_X (v. 6.9.1; Connolly, 2005) and the ds622 thermodynamic data set (Holland & Powell, 2011). Pseudosections were calculated in the 11-component MnO– Na_2O –CaO– K_2O –FeO–MgO– Al_2O_3 – SiO_2 – H_2O – TiO_2 – O_2 (MnNCKFMASHTO) system. This system is considered to provide a realistic approximation to the composition of the samples within which melt and Fe^{3+} can be assessed. The solution model for sapphirine is from Wheller & Powell (2014) and a - x models for other phases are same to those adopted by White et al. (2014). Whole-rock composition of a piece of relatively homogeneous rock ($5 \times 5 \times 3 \text{ cm}^3$), obtained from XRF analysis, was used as the bulk rock composition for the modeling. The formation of fine-grained intergrowths is usually controlled by local effective bulk compositions. In this study, we assume that whole-rock composition obtained from XRF can roughly represent the local effective bulk composition of the symplectite formation. The H_2O contents were adjusted to ensure that the final assemblage is stable just above the solidus (Korhonen et al., 2012). A preliminary T–X(Fe_2O_3) diagram was calculated to evaluate the effect of Fe^{3+} contents on phase assemblage (Figure S1). Based on the predicted peak assemblages across the range of Fe^{3+} contents, a Fe^{3+}/Fe^T value of ~0.16 is finally adopted.

Table 3: Major element concentrations of re – homogenized NIs. The compositions are shown in wt.%

	1	2	3	4	5	6	7	8	9	10	11	12	13	14	ave.	σ	PEM
SiO ₂	73.60	70.36	72.93	69.94	70.99	69.89	73.16	75.61	72.78	74.94	73.14	71.33	73.86	70.48	72.36	1.80	69.33
TiO ₂	0.00	0.05	0.04	0.07	0.03	0.01	0.02	0.00	0.06	0.06	0.00	0.04	0.02	0.03	0.03	0.02	0.00
Al ₂ O ₃	14.28	14.21	15.21	15.28	14.30	14.49	12.70	13.47	13.33	13.05	15.06	14.21	13.89	14.52	14.14	0.76	14.19
Cr ₂ O ₃	0.00	0.04	0.00	0.00	0.00	0.02	0.00	0.01	0.00	0.00	0.00	0.03	0.00	0.02	0.01	0.01	0.00
Fe ^T O	1.10	1.12	1.22	1.11	1.22	1.43	1.03	0.98	1.00	0.99	1.33	1.44	1.11	1.09	1.15	0.15	0.48
MnO	0.00	0.02	0.02	0.04	0.00	0.00	0.00	0.00	0.01	0.02	0.00	0.00	0.02	0.04	0.01	0.02	0.00
MgO	0.54	1.23	0.77	1.34	0.83	0.94	0.39	0.41	0.41	0.41	0.62	1.87	0.58	0.54	0.78	0.42	0.29
CaO	0.35	0.45	0.43	0.46	0.56	0.32	0.36	0.38	0.58	0.41	0.63	0.51	0.39	0.30	0.44	0.10	0.63
Na ₂ O	1.26	0.94	0.99	1.06	1.30	1.39	0.99	0.99	1.05	1.31	1.63	0.74	0.64	0.61	1.06	0.28	1.70
K ₂ O	6.53	8.43	6.50	8.47	6.47	7.52	7.33	6.60	7.15	6.68	5.46	7.40	7.20	8.62	7.17	0.86	8.53
Cl	0.05	0.05	0.06	0.05	0.06	0.04	0.06	0.06	0.06	0.05	0.06	0.04	0.08	0.02	0.05	0.01	0.00
P ₂ O ₅	0.00	0.02	0.00	0.01	0.00	0.00	0.00	0.00	0.00	0.00	0.03	0.00	0.03	0.00	0.01	0.01	0.00
Total	97.71	96.91	98.18	97.84	95.76	96.05	96.04	98.51	96.44	97.92	97.95	97.61	97.80	96.29	97.21	0.89	96.29
H ₂ O by diff	2.29	3.09	1.82	2.16	4.24	3.95	3.96	1.49	3.56	2.08	2.05	2.39	2.20	3.71	2.79	0.89	4.85
ASI	1.46	1.24	1.61	1.30	1.41	1.32	1.24	1.42	1.26	1.29	1.55	1.40	1.45	1.33	1.40	0.11	1.08
Al	0.03	0.02	0.04	0.03	0.03	0.02	0.02	0.03	0.02	0.02	0.04	0.03	0.03	0.02	0.03	0.01	0.01
Na ₂ O + K ₂ O	7.79	9.37	7.49	9.53	7.77	8.91	8.32	7.59	8.21	7.99	7.08	8.14	7.84	9.23	8.23	0.72	10.23
K/Na	3.40	5.90	4.30	5.25	3.29	3.57	4.85	4.38	4.46	3.34	2.21	6.59	7.44	9.31	4.43	1.89	3.31
Qz norm	39.73	29.93	39.93	28.14	36.84	31.02	38.69	43.01	37.99	40.32	40.33	34.53	40.99	32.80	36.75	4.48	25.86
Crn norm	4.61	2.86	5.85	3.65	4.34	3.64	2.57	4.07	2.89	2.98	5.52	4.18	4.50	3.78	3.96	0.94	1.07
Or norm	39.50	51.44	39.15	51.17	39.97	46.31	45.12	39.64	43.86	40.31	32.93	44.83	43.53	52.96	43.60	5.40	52.98
Ab norm	10.95	8.21	8.58	9.19	11.47	12.22	8.76	8.52	9.26	11.36	14.07	6.41	5.52	5.36	9.27	2.45	15.12
An norm	1.76	2.19	2.19	2.23	2.90	1.63	1.89	1.91	3.01	2.08	3.01	2.55	1.81	1.54	2.19	0.48	3.28
Density	2.48	2.47	2.51	2.51	2.42	2.43	2.41	2.51	2.43	2.48	2.50	2.50	2.49	2.44	2.47	0.03	2.30
Viscosity(S96)	10 ^{4.8}	10 ^{4.5}	10 ^{5.0}	10 ^{4.9}	10 ^{4.0}	10 ^{4.1}	10 ^{4.1}	10 ^{5.2}	10 ^{4.3}	10 ^{4.9}	10 ^{4.9}	10 ^{4.8}	10 ^{4.9}	10 ^{4.2}	10 ^{4.6}	10 ^{4.6}	10 ^{5.8}
Viscosity(G08)	10 ^{5.3}	10 ^{4.5}	10 ^{5.6}	10 ^{5.0}	10 ^{4.1}	10 ^{4.1}	10 ^{4.4}	10 ^{6.1}	10 ^{4.6}	10 ^{5.5}	10 ^{5.4}	10 ^{4.9}	10 ^{5.4}	10 ^{4.4}	10 ^{4.9}	10 ^{6.6}	10 ^{5.7}

ASI values are calculated on anhydrous basis. S96, Scaillet et al. (1996); G08, Giordano et al. (2008).
 PEM, melt compositions calculated by phase equilibria modeling at 950 °C with 25 mol% melts reintegrated back into bulk rock compositions.

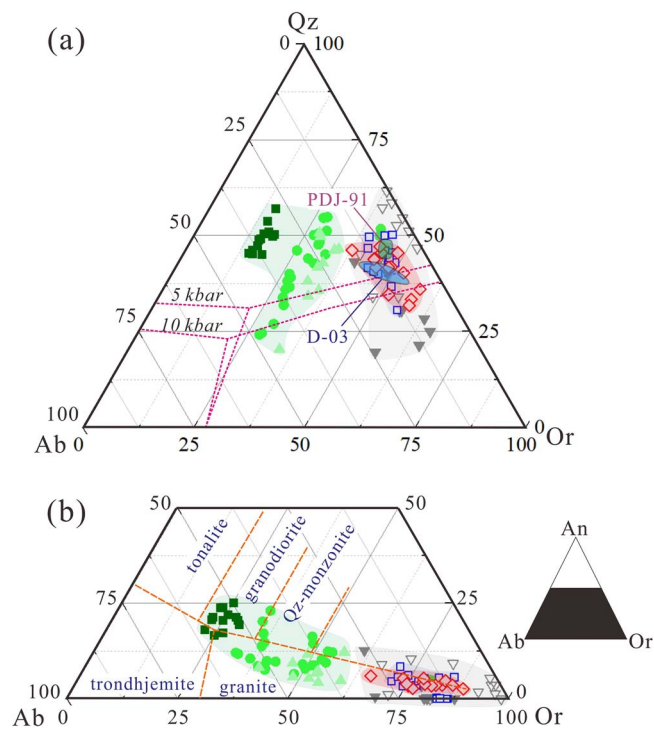


Fig. 10. (a) CIPW Qz–Ab–Or diagram showing normative compositions of analyzed NIs (normalized to 100% on anhydrous basis). Pink dashed lines show the eutectic points and cotectic lines for the subaluminous haplogranite system at $a_{\text{H}_2\text{O}} = 1$ and different pressures (Luth *et al.*, 1964). References to experimental glasses: PDJ91 (shown in green), 950°C and 7 kbar (Patiño Douce & Johnston, 1991); D03 (shown in blue), 900°C and 5 kbar (Droop *et al.*, 2003). (b) An–Or–Ab diagram showing the normative compositions of analyzed NIs (after O'Connor, 1965). For the symbols, please refer to Fig. 9.

Peak and post–peak evolution of the Mather UHT metapelitic granulites

A pseudosection for the rocks was constructed within a P – T window of 3–15 kbar and 750–1200°C. Due to lower diffusion rates of Al^{3+} compared to Fe^{2+} and Mg^{2+} , aluminum contents in orthopyroxene are considered reliable to document the peak temperatures of UHT metamorphism. As shown in Fig. 13a, the pseudosection is contoured by ($X_{\text{Mg}}(\text{Opx}) = \text{Mg}/(\text{Fe}^{2+} + \text{Mg})$) and $y_{\text{Al}}(\text{Opx})$ in order to retrieve peak P – T conditions. The interpreted peak assemblage of garnet + orthopyroxene + biotite + melt is stable over a wide P – T range (>7.5 kbar and $>860^\circ\text{C}$). The measured highest y_{Al} (0.18–0.19) and X_{Mg} (0.775–0.780) in orthopyroxene intersect at 12–13 kbar and 1070–1130°C, which is interpreted as the peak P – T conditions.

The peak minerals usually show breakdown to intergrowths of orthopyroxene₂ + cordierite ± biotite ± plagioclase ± spinel or sapphirine + cordierite at the post–peak stage. As shown in the enlarged P – T diagram ($P = 5.0$ – 9.0 kbar and $T = 800$ – 1000°C) (Fig. 13b), these assemblages occupy a narrow field, constraining the P – T conditions to 6.5–7.0 kbar and 910–950°C (outlined by purple bold lines). Plagioclase from the orthopyroxene–cordierite-bearing intergrowths has a typical X_{An} of 0.80–0.81, consistent with the prediction by the modeling. The inferred final assemblage is featured by later growth of sapphirine, biotite and plagioclase, which is predicted to be stable at a narrow pressure range of 6.5–6.8 kbar, with temperatures varying from 825 to 900°C. This field is bounded by the fluid-absent solidus, rutile-in, garnet- and ilmenite-out curves. The measured X_{Mg} in biotite (~ 0.81) and

X_{An} in plagioclase (0.51–0.58) are consistent with the P – T field, further constraining the cooling path through P – T conditions of 6.5–7.0 kbar and 830–870°C (outlined by black bold lines).

Tracking the pre-peak melting process with melt reintegration

Phase equilibria modeling suggests that the solidus is located between 800 and 870°C (at 3–7 kbar) and the rock produces <15 vol.% melt at the peak stage, which is much lower than the expected values for metapelites. In addition, biotite is predicted to be stable at $>1100^\circ\text{C}$ (Fig. 13a). All these observations are in agreement with the strongly residual character of the bulk rock composition ($\text{SiO}_2 = 44$ wt.%). Thus, this composition obtained from XRF analysis is only appropriate for modeling the peak and post-peak metamorphic evolutions (White *et al.*, 2004; Liu *et al.*, 2020). Here we explore the potential effects of melt loss on the topology during pre-peak evolution by reintegrating a certain amount of melts to the residual composition (Bartoli, 2017, 2019). The simplified chemical systems with respect to natural occurrences (Bartoli & Carvalho, 2021) and the limitations of solution models (White *et al.*, 2011) make it impossible to precisely predict the primary melt compositions of partially melted rocks. As a consequence, NIs are used as proxy of the pristine melts that was present in the system (e.g. Bartoli, 2019). This single-step melt reintegration may be only suitable to reconstruct the probable bulk rock compositions (BRC) at or near UHT conditions as the rock may have undergone several episodes of melt loss (e.g. White *et al.*, 2014; Bartoli, 2017) and the compositions of NIs cannot represent the melt compositions generated at low temperatures.

The presence of trapped rutile grains within NIs are confirmed by Raman analyses (Fig. 5g), but not observed in the matrix or predicted by the phase equilibria modeling (Fig. 13). As shown in Fig. 14a, rutile is expected to appear at $>950^\circ\text{C}$ if >20 mol.% melts are reintegrated back into the rock. This is consistent with their presence as trapped phase within the NIs (Fig. 5g), and is strongly supportive of the entrapment of rutile-bearing melt inclusions at UHT conditions. With 25 mol.% melts reintegrated back into the BRC, the modeling predicts that most melts (~ 40 mol.%) were produced in the temperature range of 850–950°C. The melt reintegration also results in a reduction of the stability field of biotite, which is predicted to disappear at $>950^\circ\text{C}$ (Fig. 14a). The melt composition calculated at 12.5 kbar and 950°C shows slightly higher Na_2O and K_2O contents (Table 3). This divergence between NIs and the calculated melts was also documented in other case studies, and was ascribed to the lack of equilibrium with the residuum or diffusion in the melts, and alternatively, the imperfections of forward phase equilibria modeling (Bartoli & Carvalho, 2021).

The P – T pseudosection reconstructed with a reintegration of 25 mol.% melts into the BRC is shown in Fig. 14b. The most evident changes are expansions of the stability fields of K–feldspar and rutile, which were predicted to be stable during prograde melting. Despite the fact that kyanite/sillimanite is absent in the investigated rocks, kyanite pseudomorphs (Tong & Wilson, 2006) and kyanite relics replaced by sapphirine + quartz (Harley, 2021) have been identified in rocks from the same region, suggesting that the rocks may have been in the kyanite stability field. The quartz–, kyanite–in and plagioclase–out curves suggest that the rocks underwent a nearly isobaric heating from M0 to M1 stage (shown by dashed lines in Fig. 13a). Accordingly, the Mather UHT granulites may have experienced a clockwise P – T path with peak conditions of 12–13 kbar and 1070–1130°C,

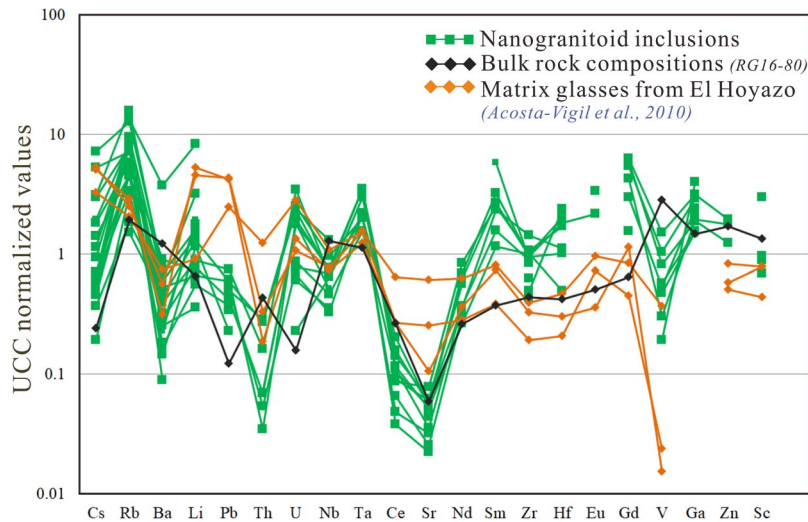


Fig. 11. Trace element compositions of NIs and granularitic restite, normalized to the average upper continental crust (UCC) composition (data from Rudnick & Gao, 2014). Trace elements of matrix glasses within metapelitic enclaves from El Hoyazo (SE Spain) (samples HO-50A, -33A and -54; Acosta Vigil et al., 2010) are also shown for comparison. Note that peak P - T conditions for the metapelitic enclaves from El Hoyazo are $850 \pm 50^\circ\text{C}$ and 5–7 kbar, slightly lower than the investigated rocks in this study.

followed by decompression and cooling to 6.5–7.0 kbar and 910 – 950°C , and finally isobaric cooling (IBC) to solidus conditions ($P = 6.5$ – 7.0 kbar; $T = 830$ – 870°C).

DISCUSSIONS

P - T conditions of melt entrapment and anatexis regime

In East Antarctica, NIs in high-grade metamorphic rocks have been reported from the Lützow-Holm Complex (Spr-bearing UHT metapelitic granulites; Carvalho et al., 2023a), the Dronning Maud Land (ultramafic granulites; Ferrero et al., 2018), the Northern Victoria Land (metagreywackes; Ferri et al., 2020), and the Sør Rondane Mountains (East Antarctica) (Sil-Grt-Bt gneiss; Higashino & Kawakami, 2022). The present study expands the existing database to include the melts produced by UHT metamorphism of the Mg-rich and Sil-absent metapelitic rocks from the Rauer Islands.

The presence of kokchetavite and kumdykolite is confirmed by Raman analyses (Fig. 5g). Kokchetavite has been found within multiphase inclusions in high to ultrahigh pressure (HP-UHP) rocks, which was considered to crystallize from K-rich melts and may exist as a metastable phase (e.g. Hwang et al., 2004, 2013; Ferrero et al., 2016; Borghini et al., 2018, 2023; Gao & Liu, 2018). Kumdykolite was not only present as inclusions within HP-UHP rocks (e.g. Hwang et al., 2009; Kotková et al., 2014; Ferrero et al., 2016; Borghini et al., 2018, 2023; Gao & Liu, 2018), but also found in chondrites (Németh et al., 2013). However, Ferrero et al. (2016) concluded that high pressure is not required for the formation of kumdykolite and kokchetavite, and their presence is thus not an indicator of UHP conditions. More recently, NIs from granulites have also been reported to preserve kokchetavite and/or kumdykolite as daughter phases (Ferrero & Angel, 2018; Carvalho et al., 2019; Gianola et al., 2021). Therefore, both kokchetavite and kumdykolite may have crystallized from HT to UHT melts, and cannot be used to infer the P - T entrapment conditions. According to Ferrero et al. (2016), the presence of feldspar polymorphs indicates that the melt compositions (in terms of major elements and volatiles) are mostly preserved.

Melting during heating is predicted by phase equilibria modeling (Figs 13 and 14). Rutile is present within NIs as an accidentally trapped phase (Fig. 5g). However, it is not observed in the matrix of the rocks. This suggests that rutile existed in the pre-peak stage and became metastable as a consequence of melt extraction and loss. After an integration of $\sim 25\%$ melts into the granularitic restite, phase equilibria modeling predicts that rutile appears when temperature exceeds 950°C at 12.5 kbar (Fig. 14), highly in agreement with the remelting experimental constraints given by NIs at 12.5 kbar. Furthermore, some NIs occur near to high-Al orthopyroxene inclusions ($\text{Al}_2\text{O}_3 = 8.85$ – 9.04 wt.%) within garnets (Fig. 3a). These are strong evidence that at least some NIs investigated in this study were trapped at UHT conditions. Although the rocks studied here have undergone peak temperatures of 1070 – 1130°C , the melts may be produced at or near UHT conditions (850 – 950°C).

The H_2O contents of NIs show variable values ranging from 1.49 to 4.24 wt.% (Table 3), suggestive of heterogeneous distributions of water (Bartoli et al., 2014) or existences of C-bearing phases in some NIs (e.g. Carvalho et al., 2019; Ferri et al., 2020; Gianola et al., 2021). Estimation of H_2O by difference of EMPA totals from 100% usually overestimates the H_2O contents of NIs (e.g. Cesare et al., 2015), and specific reasons for this inconsistency still need further investigations. As shown in Figs 9 and 10, the highly silicic, peraluminous and potassic to ultrapotassic NIs have also been found in peritectic garnets from the Kerala Khondalite Belt (Cesare et al., 2009; Ferrero et al., 2012), the Gruf Complex (Gianola et al., 2021) and the Lützow-Holm Complex (Carvalho et al., 2023a). Moreover, K-rich melts have also been experimentally derived from pelitic starting materials under UHT conditions (e.g. Droop et al., 2003; Patiño-Douce & Johnston, 1991). Ti and Fe^{3+} are expected to stabilize biotite to expand the Bt-dehydration melting to occur at higher temperatures (e.g. White et al., 2014), which likely accounts for the enrichments of K_2O in HT to UHT melts (Gianola et al., 2021). However, it is also evident that melt compositions of HT-UHT granulites from the Ivrea Zone plot away from the K-apex of the triangle (Fig. 10), with low K_2O contents of ~ 2.0 wt.% (Carvalho et al., 2019). The authors argued that the low-K melts may be produced at P - T conditions approaching the Bt-out curves

Table 4: Trace element concentrations of NIs and the granulitic restite from the Mather Peninsula. The trace element compositions are shown in ppm

	80_05	80_07	80_09	80_10	80_12	80_16	80_18	80_21	80_24	80_25	80_27	80_38	80_48	80_03*	80_08*	80_26*	80_13*	80_44*	80_46*	80_47*	80_42*	80_11*	ave.	σ	WRC
Rb	268.8	755.9	387.1	708.9	711.7	676.6	732.7	282.3	385.7	212.2	156.6	126.7	605.0	411.1	798.8	1131.7	1325.0	380.3	489.2	1077.4	402.1	691.9	578.1	312.9	162.4
Cs	0.9	5.6	2.6	9.5	2.8	14.7	4.6	3.2	6.9	1.8	1.8	b.d.l.	26.1	2.3	3.3	b.d.l.	15.3	3.4	9.0	35.5	3.5	2.9	7.8	8.8	1.2
Ba	180.2	203.0	107.6	143.3	279.7	56.3	162.3	114.7	91.1	446.7	332.1	267.5	248.2	159.7	398.3	2364.7	432.8	402.3	565.6	483.7	373.7	275.1	367.7	457.2	769.5
Li	18.8	b.d.l.	13.5	46.5	31.5	b.d.l.	42.7	b.d.l.	27.8	b.d.l.	16.0	b.d.l.	21.5	8.6	76.9	200.8	38.8	b.d.l.	17.8	b.d.l.	33.9	b.d.l.	42.5	47.0	15.6
Pb	7.2	10.4	6.3	b.d.l.	10.1	5.8	b.d.l.	8.9	3.9	9.9	10.1	8.6	12.8	n.c.	n.c.	n.c.	n.c.	n.c.	n.c.	n.c.	n.c.	n.c.	8.5	2.4	2.1
Th	0.7	0.4	b.d.l.	0.8	b.d.l.	b.d.l.	b.d.l.	0.6	b.d.l.	3.8	2.8	3.0	1.7	n.c.	n.c.	n.c.	n.c.	n.c.	n.c.	n.c.	n.c.	n.c.	1.7	1.2	4.5
U	4.8	5.2	2.3	4.9	6.4	1.6	0.6	2.1	1.8	8.7	6.3	6.8	9.3	n.c.	n.c.	n.c.	n.c.	n.c.	n.c.	n.c.	n.c.	n.c.	4.7	2.7	0.4
Nb	9.3	8.7	5.6	6.2	15.7	4.2	7.8	8.4	4.0	14.3	8.4	11.6	8.0	n.c.	n.c.	n.c.	n.c.	n.c.	n.c.	n.c.	n.c.	n.c.	8.6	3.4	15.4
Ta	3.2	3.2	1.4	b.d.l.	b.d.l.	b.d.l.	2.0	1.8	2.0	3.2	b.d.l.	1.8	2.7	n.c.	n.c.	n.c.	n.c.	n.c.	n.c.	n.c.	n.c.	n.c.	2.4	0.7	1.0
Ti	997.5	1052.6	793.0	963.1	904.3	334.2	419.1	470.5	274.1	734.6	654.5	705.3	314.5	n.c.	n.c.	n.c.	n.c.	n.c.	n.c.	n.c.	n.c.	n.c.	662.9	265.1	n.c.
Ce	7.3	10.0	7.0	6.7	5.5	4.1	b.d.l.	3.0	2.4	13.4	5.8	12.8	8.9	n.c.	n.c.	n.c.	n.c.	n.c.	n.c.	n.c.	n.c.	n.c.	7.2	3.4	16.8
Sr	16.6	17.8	11.4	17.0	24.8	8.2	14.1	10.2	7.2	27.6	19.0	17.9	20.0	n.c.	n.c.	n.c.	n.c.	n.c.	n.c.	n.c.	n.c.	n.c.	16.3	5.8	19.0
Nd	7.2	16.7	10.0	b.d.l.	22.8	8.3	b.d.l.	b.d.l.	b.d.l.	16.8	9.7	18.7	15.2	n.c.	n.c.	n.c.	n.c.	n.c.	n.c.	n.c.	n.c.	n.c.	13.9	5.0	7.1
Sm	5.5	12.6	7.4	27.2	11.8	b.d.l.	b.d.l.	b.d.l.	b.d.l.	19.0	11.1	15.3	b.d.l.	n.c.	n.c.	n.c.	n.c.	n.c.	n.c.	n.c.	n.c.	n.c.	13.7	6.4	1.8
Zr	194.9	185.1	172.9	173.9	197.0	84.2	206.3	163.8	121.1	256.0	276.5	182.8	96.2	n.c.	n.c.	n.c.	n.c.	n.c.	n.c.	n.c.	n.c.	n.c.	177.8	47.7	84.9
Hf	10.7	9.7	b.d.l.	b.d.l.	2.6	b.d.l.	9.0	b.d.l.	b.d.l.	12.5	5.9	5.4	12.7	n.c.	n.c.	n.c.	n.c.	n.c.	n.c.	n.c.	n.c.	n.c.	8.6	3.4	2.2
Eu	b.d.l.	2.2	b.d.l.	b.d.l.	b.d.l.	b.d.l.	b.d.l.	b.d.l.	3.4	b.d.l.	b.d.l.	b.d.l.	b.d.l.	n.c.	n.c.	n.c.	n.c.	n.c.	n.c.	n.c.	n.c.	n.c.	2.8	0.6	0.5
Gd	6.3	b.d.l.	11.9	b.d.l.	b.d.l.	17.1	b.d.l.	21.9	b.d.l.	b.d.l.	b.d.l.	23.9	25.1	n.c.	n.c.	n.c.	n.c.	n.c.	n.c.	n.c.	n.c.	n.c.	17.7	6.8	2.6
V	b.d.l.	b.d.l.	52.0	b.d.l.	b.d.l.	29.4	18.6	101.1	55.5	35.8	46.1	79.7	146.6	n.c.	n.c.	n.c.	n.c.	n.c.	n.c.	n.c.	n.c.	n.c.	62.7	38.1	274.7
Ga	b.d.l.	33.2	42.4	25.7	35.7	34.3	70.5	50.7	27.1	18.8	26.9	32.8	55.3	n.c.	n.c.	n.c.	n.c.	n.c.	n.c.	n.c.	n.c.	n.c.	37.8	14.0	25.9
Zn	b.d.l.	83.3	b.d.l.	b.d.l.	b.d.l.	119.8	b.d.l.	b.d.l.	b.d.l.	b.d.l.	b.d.l.	130.7	b.d.l.	n.c.	n.c.	n.c.	n.c.	n.c.	n.c.	n.c.	n.c.	n.c.	111.2	20.3	114.9
Sc	b.d.l.	b.d.l.	12.4	b.d.l.	b.d.l.	b.d.l.	b.d.l.	42.0	b.d.l.	b.d.l.	9.7	13.5	b.d.l.	n.c.	n.c.	n.c.	n.c.	n.c.	n.c.	n.c.	n.c.	n.c.	19.4	13.1	19.0
Trapped phases														Apt	Mnz	Apt;	Mnz	Rt	Rt;	Rt	Rt	Rt			
Rb/Sr	16.21	42.48	33.93	41.79	28.65	82.46	52.08	27.74	53.71	7.69	8.22	7.08	30.25			Zrn							33.3	20.9	8.56
Sm/Nd	0.76	0.76	0.74		0.52					1.13	1.14	0.82											0.8	0.2	0.25
U/Th	6.62	14.47		6.39			3.74			2.27	2.22	2.27	5.47										5.4	3.8	0.09

b.d.l. below detection limit; n.c. not considered; WRC, whole-rock composition; * Nanogrammitoid inclusions with accidentally trapped phases such as zircon, monazite, apatite and rutile.

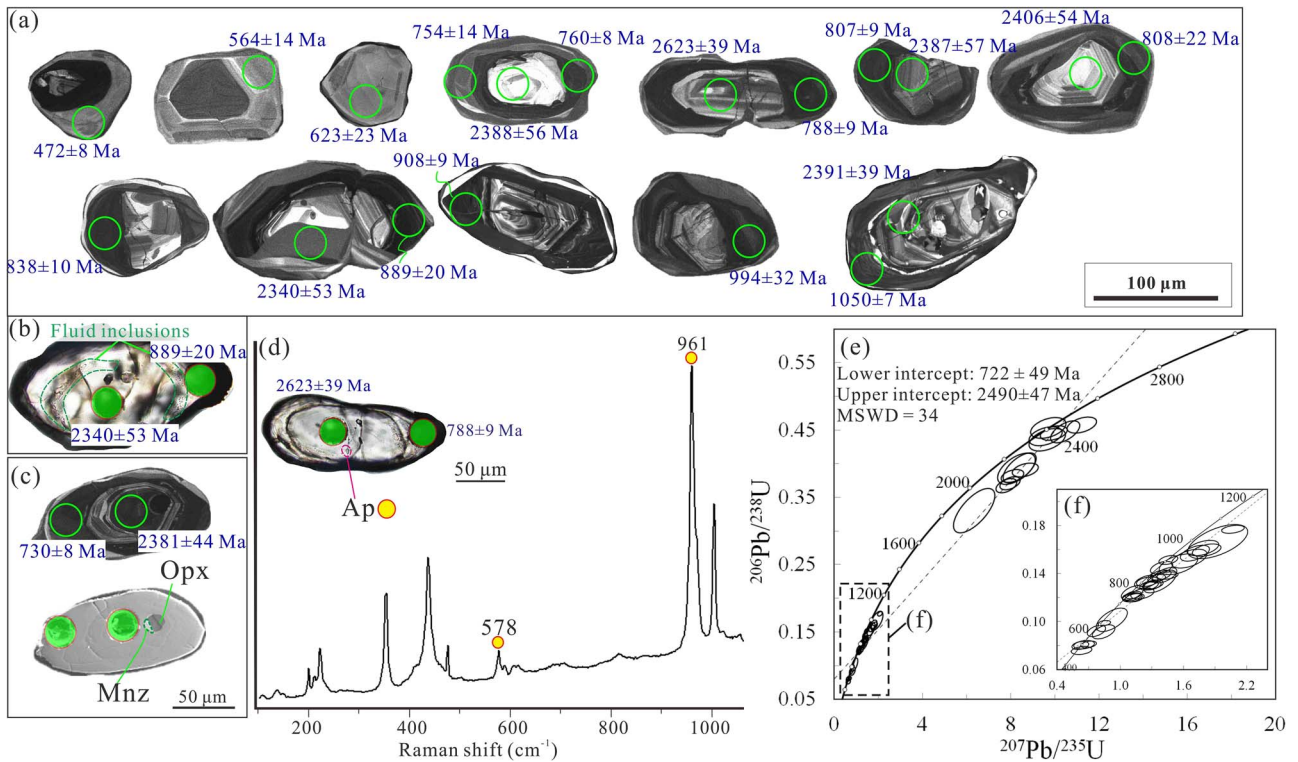


Fig. 12. (a) CL images of representative zircons mounted on epoxy resins. Green circles represent analyzed spots and the nearby blue numbers represent their isotopic dates. (b–d) Photographs showing inclusions within zircons. (e) Concordia diagram showing the results of U–Pb zircon dating. (f) Enlarged concordia diagram of the box in (e).

(Carvalho *et al.*, 2019). Alternatively, participation of plagioclase in the prograde melting reactions may also be responsible for the relatively higher CaO/NaO concentrations in that study. The potassic nature of melts, coupled with phase equilibria modeling (Fig. 14), supports that the melts were produced by Bt–dehydration melting of pelites under HT to UHT conditions (850–950°C).

Our data show that the melts are enriched in Li, Cs, Rb, Ta, Sm, Nd, Zr, U and Pb and depleted in Ce, Th, Ba, Sr and Nb relative to the granulitic restite (Table 4; Fig. 11). Dissolution of zircon or monazite during high temperature melting (Kelsey & Powell, 2011) may contribute to the U, Pb and Zr enrichments in anatectic melts. NIs in this study are depleted in Ce (<12.8 ppm) and Th (<3.8 ppm), which may be ascribed to the shielding of some monazites by porphyroblastic phases (Huang *et al.*, 2021) or disequilibrium melting (Watt & Harley, 1993). It suggests that the melts are likely undersaturated with respect to monazite. Besides, the melts show mild depletion of Ba compared with the restite. K–feldspar is the most important repository of Ba in the rocks and it was still stable during melting, which might reserve appreciable amounts of Ba (Zeng *et al.*, 2005).

The investigated melts contain higher Rb contents than the melts produced through Ms–dehydration melting (Acosta Vigil *et al.*, 2010, 2012; Bartoli *et al.*, 2019), consistent with melts yielded by Bt–dehydration melting because biotite is the major reservoir of Rb (e.g. Acosta Vigil *et al.*, 2010; Wolf *et al.*, 2019). They also show higher Rb contents relative to the matrix glasses from El Hoyazo (Fig. 11), which were considered to be yielded at the onset of biotite–dehydration melting (Acosta Vigil *et al.*, 2010). Moreover, Sr contents are usually low in micas but abundant in plagioclase (Weinberg & Hasalová, 2015), and plagioclase was not involved in the incongruent melting in this study. Thus,

higher Rb/Sr ratios of the investigated NIs can be attributed to Bt–dehydration melting in the absence of plagioclase. On the other hand, although Zeng *et al.* (2005) previously pointed out that Ms/Bt–dehydration melting and dissolution of apatite will produce melts with high Sm/Nd values, the mechanisms that caused elevated Sm/Nd ratios in the melts are still vague. Assuming no isotopic fractionations of Nd and Sr, enrichments of Sm and Rb in anatectic melts are expected to result in higher $^{143}\text{Nd}/^{144}\text{Nd}$ and $^{87}\text{Sr}/^{86}\text{Sr}$ values in melts than the granulitic restite after enough long time.

Physical properties of the hottest crustal melts

The chemical data of NIs allow to peer into physical properties of the melts and therefore, may provide insights into rheology and mobility of the hottest granitic melts and their potential effects on orogenic evolution. Assuming conditions of $P = 12$ kbar and $T = 950^\circ\text{C}$, the average compositions of re-homogenized melt inclusions are considered to have densities of 2.47 ± 0.07 g/cm³, calculated with program DensityX (Iacovino & Till, 2019). These values are slightly higher than previously estimated densities for hydrous granitic melts (~ 2.20 g/cm³; Clemens & Vielzeuf, 1987), but lower than the average density for granites (~ 2.70 g/cm³; Artemieva *et al.*, 2017).

Viscosity of melts may be affected by several potential factors, such as fluid contents (Scaillet *et al.*, 1996; Holtz *et al.*, 2001) and melt compositions (Giordano *et al.*, 2008). At 950°C, applications of the equations of Giordano *et al.* (2008) and Scaillet *et al.* (1996) yield melt viscosities of $10^{4.1}$ – $10^{6.1}$ Pa·s and $10^{4.0}$ – $10^{5.2}$ for the investigated melts, respectively. When considering the average melt compositions, these methods produce results of $10^{4.9 \pm 1.2}$ and $10^{4.6 \pm 0.8}$ Pa·s. As the estimated H₂O contents for the melts based on difference of EMPA totals from 100% usually slightly

Table 5: LA – ICP – MS analysis results for zircons from the Mather UHT granulites

Spot	Th (ppm)	U (ppm)	Th/U	²⁰⁶ Pb/ ²³⁸ U	1σ	²⁰⁷ Pb/ ²⁰⁶ Pb	1σ	²⁰⁷ Pb/ ²³⁵ U	1σ	²⁰⁶ Pb/ ²³⁸ U (Ma)	1σ	²⁰⁷ Pb/ ²⁰⁶ Pb (Ma)	1σ	²⁰⁷ Pb/ ²³⁵ U (Ma)	1σ
Rim															
1	49	145	0.34	0.076	0.001	0.060	0.004	0.633	0.040	472	8	611	157	498	25
2	45	167	0.27	0.080	0.001	0.057	0.003	0.621	0.033	497	8	480	120	490	20
3	40	143	0.28	0.081	0.001	0.059	0.004	0.656	0.040	500	9	565	136	512	25
4	98	437	0.22	0.081	0.001	0.062	0.003	0.699	0.030	502	7	676	96	538	18
5	47	420	0.11	0.091	0.002	0.065	0.003	0.842	0.044	564	14	770	96	620	24
6	57	271	0.21	0.092	0.002	0.061	0.003	0.774	0.035	569	11	657	100	582	20
Mantle															
7	8	877	0.01	0.099	0.001	0.062	0.002	0.848	0.024	607	5	661	66	624	13
8	23	481	0.05	0.101	0.004	0.062	0.003	0.920	0.060	623	23	687	95	662	32
9	18	1126	0.02	0.120	0.001	0.068	0.002	1.136	0.037	730	8	865	65	771	18
10	32	659	0.05	0.120	0.001	0.067	0.002	1.115	0.039	730	8	843	70	761	18
11	18	811	0.02	0.121	0.001	0.066	0.002	1.110	0.037	735	8	1200	71	758	18
12	22	1024	0.02	0.122	0.001	0.066	0.002	1.123	0.031	743	7	817	59	765	15
13	48	524	0.09	0.123	0.002	0.070	0.002	1.210	0.045	750	11	937	67	805	21
14	42	521	0.08	0.124	0.002	0.065	0.002	1.115	0.043	754	14	759	70	760	21
15	31	1178	0.03	0.130	0.002	0.072	0.002	1.301	0.035	788	9	983	55	846	16
16	21	955	0.02	0.130	0.002	0.070	0.002	1.273	0.036	788	9	943	54	834	16
17	42	848	0.05	0.131	0.002	0.072	0.002	1.310	0.040	793	9	981	56	850	17
18	15	821	0.02	0.133	0.002	0.067	0.002	1.245	0.045	806	13	835	61	821	20
19	12	675	0.02	0.133	0.002	0.068	0.002	1.257	0.043	807	9	861	72	827	19
20	43	666	0.06	0.133	0.004	0.073	0.003	1.376	0.063	808	22	1013	70	879	27
21	14	1386	0.01	0.136	0.001	0.071	0.002	1.345	0.035	822	8	955	49	865	15
22	22	825	0.03	0.138	0.001	0.076	0.002	1.448	0.041	836	7	1087	55	909	17
23	10	548	0.02	0.139	0.003	0.073	0.002	1.421	0.047	837	15	1020	54	898	20
24	21	765	0.03	0.139	0.002	0.072	0.002	1.384	0.040	838	10	977	61	882	17
25	24	1183	0.02	0.145	0.002	0.069	0.002	1.391	0.043	873	12	889	57	885	18
26	47	758	0.06	0.148	0.004	0.073	0.002	1.552	0.074	889	20	1013	67	951	30
27	10	1346	0.01	0.151	0.002	0.069	0.002	1.456	0.037	908	9	898	56	912	15
28	20	1432	0.01	0.152	0.003	0.076	0.002	1.654	0.069	915	18	1098	57	991	26
29	23	944	0.02	0.157	0.003	0.078	0.002	1.708	0.056	940	15	1139	57	1012	21
30	26	972	0.03	0.160	0.002	0.081	0.002	1.820	0.055	954	12	1232	56	1053	20
31	27	805	0.03	0.161	0.003	0.078	0.002	1.783	0.062	964	15	1158	55	1039	22
32	19	707	0.03	0.167	0.006	0.078	0.003	1.924	0.116	994	32	1154	64	1090	40
33	21	1131	0.02	0.177	0.001	0.084	0.002	2.069	0.044	1050	7	1292	40	1139	14
Core															
34	151	529	0.28	0.330	0.013	0.129	0.004	6.343	0.383	1840	65	2088	56	2024	53
35	172	219	0.79	0.393	0.009	0.150	0.005	8.258	0.349	2135	44	2340	53	2260	38
36	242	351	0.69	0.394	0.009	0.151	0.004	8.407	0.306	2140	42	2361	48	2276	33
37	58	289	0.20	0.367	0.004	0.152	0.004	7.803	0.193	2017	20	2374	41	2209	22
38	101	144	0.70	0.382	0.004	0.153	0.004	8.080	0.210	2085	18	2381	44	2240	24
39	140	167	0.83	0.453	0.006	0.154	0.005	9.704	0.327	2410	28	2387	57	2407	31
40	216	378	0.57	0.371	0.004	0.154	0.004	7.970	0.188	2035	19	2391	39	2228	21
41	80	140	0.57	0.441	0.006	0.155	0.005	9.582	0.307	2355	26	2406	54	2395	30
42	798	441	1.81	0.391	0.004	0.160	0.004	8.661	0.241	2126	19	2452	46	2303	25
43	233	337	0.69	0.446	0.004	0.160	0.004	9.951	0.249	2376	20	2461	41	2430	23
44	50	49	1.01	0.436	0.007	0.161	0.006	9.656	0.338	2331	31	2465	58	2403	32
45	138	234	0.59	0.448	0.007	0.168	0.004	10.468	0.270	2384	30	2540	38	2477	24
46	118	227	0.52	0.458	0.005	0.177	0.004	11.225	0.273	2429	22	2623	39	2542	23

higher than their real values due to the possible presence of CO₂ (e.g. Cesare et al., 2015), the calculated viscosities may represent the minimum values. Viscosities for the investigated melts are roughly in agreement with the calculation results for melt inclusions within UHT granulites from the Gruf Complex (10^{4.1}–10^{4.8} Pa·s; Gianola et al., 2021). These values also overlap the estimates of granitic melts that were experimentally derived at 5–8 kbar and 700–950°C (10^{4.3}–10^{5.4} Pa·s) (Hess & Dingwell, 1996; Scaillet et al., 1996). However, melts formed at low temperature (~700°C) showed higher viscosities (~10^{6.0} Pa·s) (Bartoli et al., 2013b), and therefore less mobile and result in slower rates of melt segregation

and ascent (Holtz et al., 2001; Brown, 2013; Bartoli et al., 2013b). Clemens et al. (2020) concluded that melting at high temperatures (mostly >850°C) under fluid-deficient or -absent conditions are usually highly mobile, and therefore, the low viscosity melts may facilitate melt extraction, and consequently crustal differentiation and redistribution of heat-producing elements (such as K, Th and U) (Gianola et al., 2021).

The melts are enriched in heat producing elements such as K and U. Despite that the melts are depleted in Th, the heat production rate of Th (0.072 μW/m³) is much lower than that of U (0.265 μW/m³) (Bea, 2012). The capacity for heat production

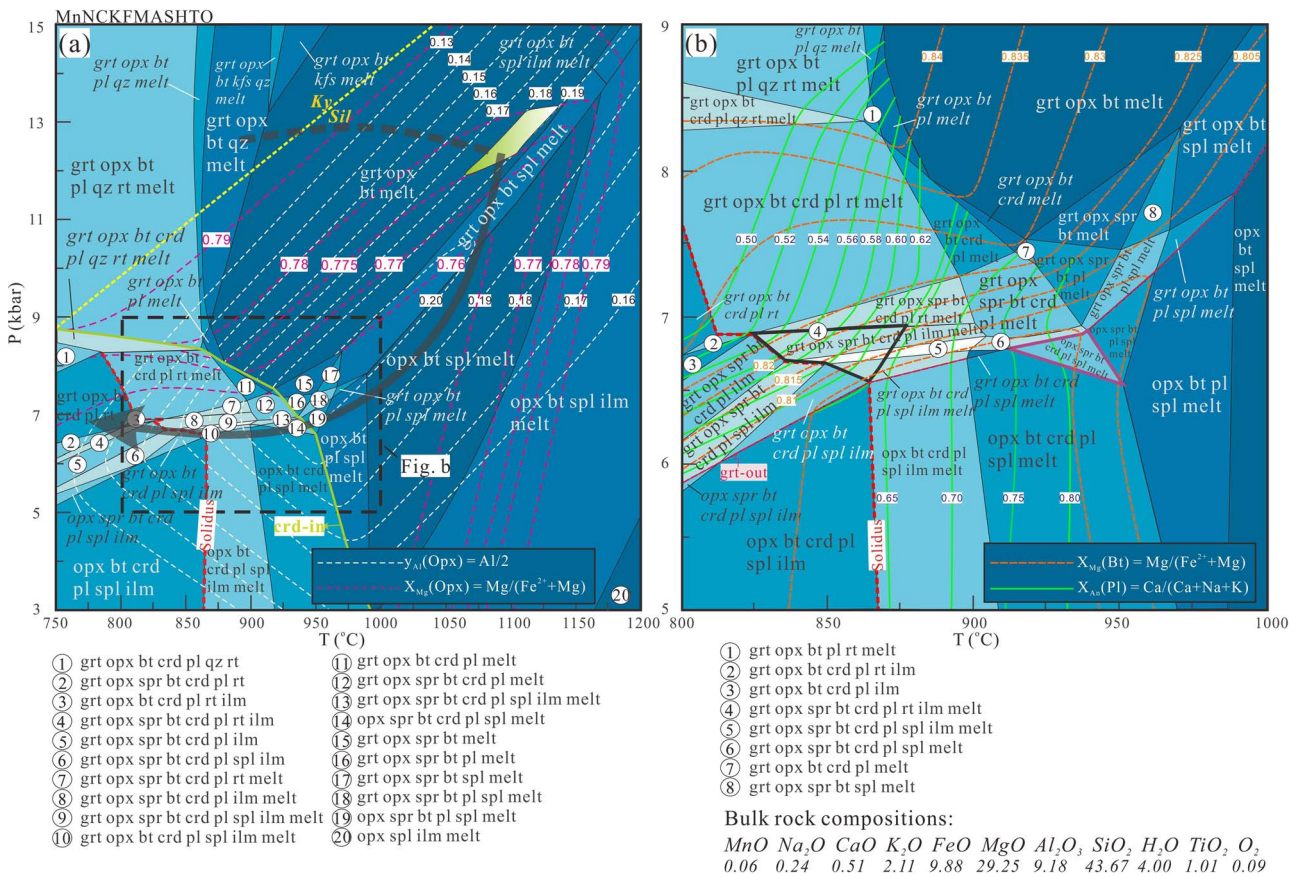


Fig. 13. (a) P-T pseudosection calculated at $X(Fe_2O_3) = Fe^{3+}/Fe^{2+} = 0.35$, with a P-T range of 3–15 kbar and 750–1200°C. The fluid-absent solidus is shown in red dashed curves and the interpreted peak P-T conditions are shown in pale yellow. The grey curves in (a) represent the inferred P-T path. (b) Enlarged P-T pseudosection with a P-T range of 5–9 kbar and 800–1000°C. The constrained P-T fields for the M2 and M3 stage are outlined by purple and black bold lines, respectively.

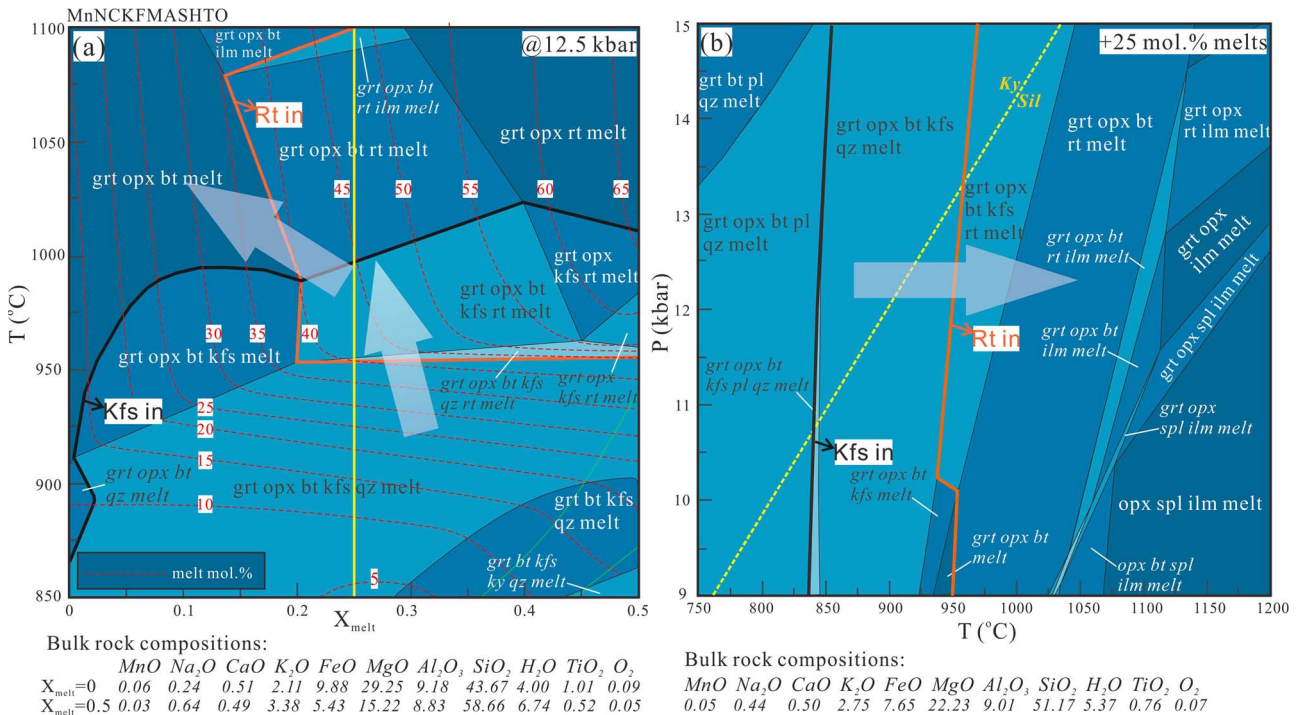


Fig. 14. (a) T-X_{melt} pseudosection showing the pre-peak evolution of the investigated rocks. Average re-homogenized nanogranitoid compositions are adopted as the melt compositions. The pressure is fixed at ~12.5 kbar. (b) Reconstructed P-T pseudosection after 25% melt reintegration into the bulk rock composition.

of the hot melts was determined with the following equation (Rybach, 1988):

$$H = \rho \times (0.0952 \times C_U + 0.0348 \times C_K + 0.0256 \times C_{Th}).$$

Where H is heat production in $\mu\text{W}/\text{m}^3$, ρ is density in g/cm^3 , and C_U , C_K , and C_{Th} are concentrations of radioactive isotopes of U, K, and Th in ppm, wt.% and ppm, respectively.

As calculated above, the density of melts is estimated as $2.47 \pm 0.07 \text{ g}/\text{cm}^3$. Calculated heat production value for the average melt composition is $\sim 2.3 \mu\text{W}/\text{m}^3$. However, the obtained K, U and Th contents of melts cannot represent their real concentrations during the thermal event because of long-standing decay of the parental elements. Accordingly, heat production of average melt compositions was back-calculated to $\sim 530 \text{ Ma}$ based on appropriate decay equations for ^{238}U , ^{235}U , ^{232}Th and ^{40}K . The resultant average value is $\sim 2.8 \mu\text{W}/\text{m}^3$, which is higher compared with bulk heat production in granitic rocks of all ages ($\sim 2.0 \mu\text{W}/\text{m}^3$; Artemieva et al., 2017).

Granitic melts investigated in this study are particularly rich in heat-producing elements (such as K and U), and show densities of $2.47 \pm 0.07 \text{ g}/\text{cm}^3$ and viscosities of $10^{4.9 \pm 1.2} \text{ Pa}\cdot\text{s}$. Such melts are mobile and prone to be extracted from the source, and as a consequence, might be responsible for the formation of large granitic intrusions. Stagnation and flow of such melts in the deep crust provides a potential source at the base of the crust to heat the orogenic hinterlands through radioactive decay of heat producing elements (Clark et al., 2011; Gianola et al., 2021), and may have a great influence on the thermal structure of the continental crust.

Secondary fluid infiltration and its implications for the timing of UHT metamorphism: Open questions

Fluid infiltration is a well-documented phenomenon in the Rauer Islands. For example, Harley (1998b) has suggested the growth of cordierites in Grt-Sil-Qz gneiss was linked with the ingress of CO_2 -rich fluid ($X_{\text{CO}_2} > 0.8$) based on secondary ion mass spectrometry analyses of H_2O and CO_2 in the secondary cordierite. On the contrary, coarser cordierites adjacent to leucosomes show lower X_{CO_2} values of 0.05. Such features indicate that the fluid regime in this region has changed markedly as a consequence of ambient C-O-H fluid ingress. As zircon U-Pb systematics of the layered mafic complex and intrusive tonalites from the nearby Scherbinina Island show clear resetting at $\sim 520 \text{ Ma}$, the fluid ingress was considered to have occurred at 530–510 Ma (Harley et al., 1998; Harley & Kelly, 2007). On the other hand, Liu et al. (2021) reported that hornblende and clinopyroxene in mafic granulites from the Filla Island were partially replaced by actinolite, which was also ascribed by the authors to strong fluid activities during the Cambrian reworking. In this study, we report on the occurrence of multiphase fluid inclusions (i.e. magnesite, pyrophyllite, corundum and CO_2) within orthopyroxene and garnet (Fig. 7), which are considered to be secondary in origin. Such stepdaughter phases were previously recognized in garnet-hosted primary FIs (e.g. Tacchetto et al., 2019; Carvalho et al., 2020, 2023b; Gianola et al., 2021). Here, we confirm that the secondary C-O-H fluid along the healed fractures of garnet and orthopyroxene at high temperatures could not survive neither, and has reacted with its hosts to produce CO_2 and OH-bearing phases during cooling (possibly at 400–600°C; Carvalho et al., 2020). Pure CO_2 inclusions within garnets are also present, which might represent the fluid that infiltrated into the rocks at a very late stage.

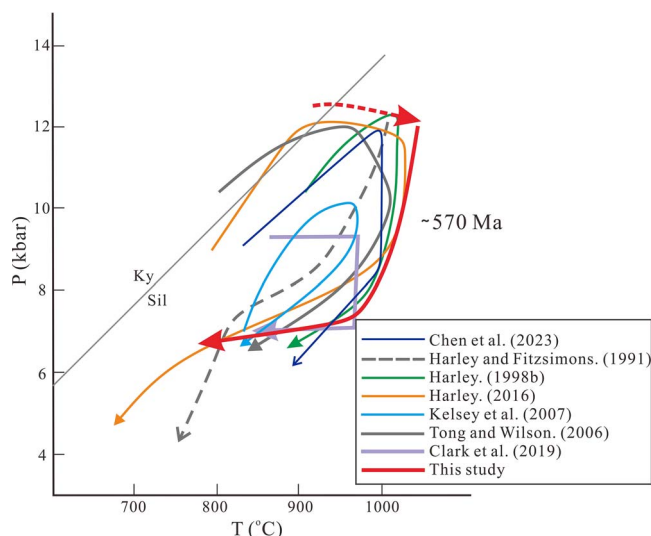


Fig. 15. Suggested P-T path of UHT granulites from the Mather Peninsula, East Antarctica. Also shown are previously retrieved P-T paths for the UHT granulites from the Rauer Islands (modified after Tong et al., 2021).

In regard of the origin of the fluid, several potential sources can be considered. Harley et al. (1998) speculated that the infiltration of CO_2 -rich fluid might have occurred during the decompression as a consequence of emplacements of granitic pegmatites in the Cambrian (Kinny et al., 1993). Additionally, coeval graphite-bearing sediments (e.g. Harley & Fitzsimons, 1991) and forsterite marbles (e.g. Buick et al., 1994; Harley, 1998b) have been reported in the Rauer Islands, and their decarbonation may also be external sources for the fluid. Collectively, an external source for the C-O-H fluid is favored in this study. Fluid infiltration not only lead to possible transfer of fluid-related elements (e.g. Liu et al., 2018), but also caused partial to complete resetting of U-Th-Pb systems in zircon and monazite (e.g. Harley et al., 1998; Harley & Kelly, 2007; Taylor et al., 2014; Das et al., 2021).

As addressed on the section of geological setting, the timing of the UHT metamorphism is still a matter of debate. Constraints on its timing are critical to understanding the tectono-thermal evolution of the Rauer Islands, and even the whole Prydz Belt. Some works proposed that the Prydz Belt represents an early Neoproterozoic ($\sim 1000 \text{ Ma}$) metamorphic complex that was reworked by a Pan-African ($\sim 530 \text{ Ma}$) thermal event (e.g. Wang et al., 2008; Liu et al., 2009, 2014; Tong et al., 2014b, 2019), and the UHT metamorphism in the Rauer Islands occurred at $\sim 1000 \text{ Ma}$ (Tong & Wilson, 2006; Wang et al., 2007). This point of view has not been accepted by other researchers, who argued that this episode of UHT metamorphism may have taken place in the late Neoproterozoic to Cambrian (e.g. Kelsey et al., 2003, 2007; Clark et al., 2019) or prior to $\sim 590 \text{ Ma}$ (Harley et al., 2009; Hokada et al., 2016). Our dating results for zircon mantles show variable dates ranging from 1050 to 607 Ma (Fig. 12e; Table 5). Previous U-Pb dating on Spr-bearing UHT granulites from the same locality also showed similar age spreads from 1000 Ma to 500 Ma (Wang et al., 2007; Hokada et al., 2016). Such scattered ages were previously interpreted as mixed ages, detrital ages, or ascribed to variable effects of the Pan-African thermal event on pre-existing zircons (Hokada et al., 2016; Spreitzer et al., 2021; Wang et al., 2022). Although we cannot preclude the possibility of melt-mediated-coupled dissolution-precipitation of zircons in the Cambrian (Wang et al., 2022), findings of secondary FIs in

peritectic phases (such as garnet, orthopyroxene) and zircons in this study seem to support that the broad range of isotopic dates may be partially caused by extensive C–O–H fluid infiltration at high temperature conditions (>600°C). Till now, all the U–Th–Pb dating on monazite pointed to metamorphic ages of 580–510 Ma (Clark et al., 2018; Hokada et al., 2016; Kelsey et al., 2003, 2007; Liu et al., 2021). Here, we prefer the interpretation that U–Th–Pb clock of monazites has been completely reset as they were more vulnerable to fluid ingress compared with zircons (e.g. Taylor et al., 2014; Das et al., 2021).

Additionally, garnet inclusions within zircon mantles from mafic granulites in the Archean crustal domain indicate that the post-peak decompression and cooling probably occurred at 540–471 Ma (our own unpublished data), roughly consistent with the dates documented by zircon rims in this study (569–472 Ma). If it is the case, the peak metamorphism took place at least prior to ~570 Ma. Hokada et al. (2016) reported the microstructure and CL image of an orthopyroxene-hosted zircon inclusion from the same outcrop, and it documents ages of 2502 Ma in the core and 1254–1148 Ma in the rim. This suggests that this type of zircons has not been affected by the Pan–African thermal event likely due to the shielding effect of orthopyroxene. Five dark-CL mantles show $^{206}\text{Pb}/^{238}\text{U}$ dates of 1050–940 Ma (Table 5), which however, are slightly discordant (Fig. 12f). Up to now, the most concordant ages for the Spr-bearing UHT granulites from the Mather Peninsula were obtained by Wang et al. (2007), which yielded a weighted mean $^{207}\text{Pb}/^{206}\text{Pb}$ age of 995 ± 15 Ma. Nonetheless, their dating results also showed some discordance from 1000 Ma toward 530 Ma (Wang et al., 2007). The present study highlights that C–O–H fluid infiltration and alternatively, melt-mediated-coupled dissolution-precipitation during the Pan–African reworking, have contributed to the partial resetting of U–Th–Pb systems in zircons. Collectively, the P–T framework (Fig. 15) might be linked with two separated thermal events that happened at ~1000 Ma and ~530 Ma, respectively. We propose that the UHT metamorphism and relevant melting most likely occurred prior to ~570 Ma (Harley et al., 2009; Hokada et al., 2016), while the post-peak decompression and the final cooling happened at 570–470 Ma (Fig. 15). However, it is still far from precisely determining the timing of UHT metamorphism in this region, which requires further investigations.

CONCLUSIONS

Phase equilibria modeling suggests that the Spr-bearing UHT granulites from the Mather Peninsula (East Antarctica) have undergone a clockwise P–T path, with peak conditions of 12–13 kbar and 1070–1130°C. Melt and fluid inclusions are characterized in this study. Coupled with chronological dating results, the main conclusions drawn are described below:

(1) Abundant NIs are preserved within garnet, which are usually 5–15 μm in diameter and display typically negative crystal shapes. NIs have crystallized into polycrystalline aggregates, comprising daughter phases of quartz, biotite, kokchetavite, kumdykolite and an unknown phase (phase '430'). Petrographic observations and mineral chemistry suggest that the melts were at least partially yielded at UHT conditions. The NIs were perfectly re-homogenized with a piston cylinder apparatus at ~12 kbar and ~950°C, and show SiO_2 -rich, strongly peraluminous and potassic to ultrapotassic composition.

(2) Compared with the granulitic restite, the NIs are enriched in Li, Cs, Rb, Ta, Sm, Nd, Zr, U and Pb, and depleted in Ce, Th, Ba, Sr and Nb. Melting mechanisms and dissolution of accessory

minerals played a key role in the partitioning of elements between anatexic melts and their granulitic counterparts. High Rb/Sr and Sm/Nd ratios for the melts, coupled with phase equilibria modeling, suggest that the melts were formed by fluid-absent Bt–dehydration melting. These UHT crustal melts have densities of $2.47 \pm 0.07 \text{ g/cm}^3$, viscosities of $10^{4.9 \pm 1.2} \text{ Pa}\cdot\text{s}$ and high heat production values of $\sim 2.8 \mu\text{W/m}^3$. As a consequence, these melts are expected to be mobile and may have played an important role in redistribution of heat-producing elements (such as K and U).

3) Secondary FIs are distributed along healed fractures of the peritectic phases (such as garnet and orthopyroxene), typically comprising magnesite, corundum, pyrophyllite and residual CO_2 ($\rho_{\text{CO}_2} = 0.2\text{--}1.0 \text{ g/cm}^3$). They are interpreted as stepdaughter phases due to interaction between C–O–H fluid and their hosts during post-peak metamorphic evolutions. Pure CO_2 inclusions with a density of ~0.8 are also present, representing the fluid that infiltrated the rock at a very late stage. Ingress of the C–O–H fluid under high temperatures probably resulted in mobility and loss of Pb in zircons, and was likely responsible for the large date span of zircon mantles from 1050 to 607 Ma. Another possible reason is melt-mediated-coupled dissolution-precipitation during the Pan–African reworking. Therefore, the retrieved P–T path might be linked with two separated thermal events happened at ~1000 and ~530 Ma, respectively. Although metamorphic timing for the UHT metamorphism is still debated, we conclude that the peak metamorphism occurred prior to ~570 Ma.

DATA AVAILABILITY

The authors confirm that the data cited in Fig. 11 of this manuscript are available within the the paper of Acosta Vigil et al. (2010).

Supplementary Data

Supplementary data are available at *Journal of Petrology* online.

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