

The composition of nanogranitoids in migmatites overlying the Ronda peridotites (Betic Cordillera, S Spain): the anatectic history of a polymetamorphic basement

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Abstract

The study of the composition of primary melts during anatexis of high-pressure granulitic migmatites is relevant to understand the generation and differentiation of continental crust. Peritectic minerals in migmatites can trap droplets of melt that forms via incongruent melting reactions during crustal anatexis. These melt inclusions commonly crystallize and form *nanogranitoids* upon slow cooling of the anatectic terrane. To obtain the primary compositions of crustal melts recorded in these nanogranitoids, including volatile concentrations and information on fluid regimes, they must be remelted and rehomogenized before analysis. A new occurrence of nanogranitoids was recently reported in garnets of mylonitic metapelitic gneisses (former high pressure granulitic migmatites) at the bottom of the prograde metamorphic sequence of Jubrique, located on top of the Ronda peridotite slab (Betic Cordillera, S Spain). Nanogranitoids within separated chips of cores and rims of large garnets from these former migmatites were remelted at 15 kbar and 850, 825 or 800 °C and dry (without added H₂O), during 24 hours, using a piston cylinder apparatus. Although all experiments show glass (former melt) within melt inclusions, the extent of rehomogenization depends on the experimental temperature. Experiments at 850-825 °C show abundant disequilibrium microstructures, whereas those at 800 °C show a relatively high proportion of rehomogenized nanogranitoids, indicating that anatexis and entrapment of melt inclusions in these rocks was likely close to 800 °C. Electron microprobe and NanoSIMS analyses show that experimental glasses are leucogranitoid and peraluminous, though define two distinct compositional groups. Type I corresponds to K-rich, Ca- and H₂O-poor leucogranitic melts, whereas type II represents K-poor, Ca- and H₂O-rich granodioritic to tonalitic melts. Type I and II melt inclusions are found in most cases at the cores and rims of large garnets, respectively. We tentatively suggest that these former migmatites underwent two melting events under contrasting fluid regimes, possibly during two different orogenic periods. This

study demonstrates the strong potential of melt inclusions studies in migmatites and granulites in order to unravel their anatectic history, particularly in strongly deformed rocks where most of the classical anatectic microstructures have been erased during deformation.

Keywords: Melt inclusions; nanogranitoids; migmatites and granulites; mechanisms of crustal anatexis; Betic Cordillera

Introduction

Recent studies have demonstrated that melt inclusions (MI) constitute a new and powerful tool to investigate crustal anatexis (Cesare 2008; Cesare et al. 2009, 2011, [2015](#)). Studies of MI in crustal anatectic enclaves found within peraluminous dacites of SE Spain have shown that these trapped droplets of melt, now solidified to glass due to rapid ascent and extrusion, can provide precise information on the composition of primary anatectic melts during, and on the mechanisms of, crustal anatexis (Cesare et al. 1997, 2003; Acosta-Vigil et al. 2007, 2010, 2012a). In fact, MI do represent a window into the suprasolidus prograde evolution of anatectic rocks (Acosta-Vigil et al. 2010).

Former MI were known to exist in deeply subducted, ultrahigh-pressure (UHP) crustal crystalline rocks, where they have been named as melt, polyphase, or multiphase inclusions (e.g. Hwang et al. 2001; Stockhert et al. 2001; Ferrando et al. 2005; Korsakov and Hermann 2006; Gao et al. 2012; Frezzotti and Ferrando 2015). However, detailed microstructural and, particularly, geochemical studies on these small data repositories in slowly cooled crystalline rocks, have only developed after the recent discovery of MI in low-to-medium P anatectic terranes (Cesare et al. 2009), building up on the previous studies of glassy MI of anatectic enclaves (Cesare et al. 1997, 2003; Cesare 2008; Acosta-Vigil et al. 2010, 2012a). Thus, detailed studies of MI in anatectic terranes have shown the great potential of this new approach, providing precise information on the primary compositions of melt from a

particular terrane and lithology, including volatile concentrations and clues on the fluid regime during partial melting (Cesare et al. 2009, [2015](#); Bartoli et al. 2013a, 2014; Ferrero et al. 2015). Most of the MI in crystalline rocks, however, appear today crystallized to a granitic assemblage due to slow cooling at depth, with grain size commonly $<1 \mu\text{m}$; Cesare et al. (2009) named them as *nanogranites*. A previous and fundamental step for the detailed studies on these crystallized MI has been the development and further refinement of appropriate methodologies to rehomogenize them successfully under high P, in order to prevent MI decrepitation and interaction with the host mineral and matrix rock (Bartoli et al. 2013b; see also Malaspina et al. 2006; Perchuk et al. 2008). After application of this experimental methodology and subsequent analysis of several occurrences of nanogranites, Cesare et al. ([2015](#)) have observed that the studied MI have bulk compositions varying from granitic to granodioritic, trondhjemitic and tonalitic, and hence they have renamed them as *nanogranitoids*.

Barich et al. (2014) have documented the presence of crystallized MI in Grt from strongly deformed, former high P granulitic migmatites overlying the Ronda peridotite slab, in the Betic Cordillera of S Spain (hereafter mineral abbreviations after Kretz 1983). Despite the mylonitic microstructure of these former migmatites (hereafter referred to as [mylonitic](#) gneisses, after Barich et al. 2014), the microstructural study of MI has shown that they are present throughout the entire (up to 15-20 mm in diameter) Grt porphyroclasts, whose compositional profiles and mineral inclusion systematics attest for a polymetamorphic history (Barich et al. 2014, and references therein). Our study focuses on the experimental remelting of these MI, and the subsequent analysis of the major element compositions of the glass (quenched melt). Along with providing further constraints on the anatectic history and metamorphic evolution of the crustal units overlying the Ronda peridotite, this research constitutes an effort towards exploring the applications of the detailed study of MI in

migmatites and granulites, and building up a composition database of MI in anatectic terranes, particularly for rocks such as these high P granulites. The latter are transitional between the two thermal environments registered by low-to-medium P and high P to UHP granulites, and are thought to form during continental collision at the base of thickened continental crust, or during subduction of the crust into the mantle (e.g. O'Brian and Rötzler 2003; Brown 2007). Since both environments are important regarding the investigation of crustal genesis and differentiation processes (e.g. Vielzeuf et al. 1990; Schmidt et al. 2004; Hermann and Spandler 2008; Hacker et al. 2011; Sawyer et al. 2011; Brown 2013), high P granulites are particularly relevant rocks for the study of MI.

Geological setting

The Betic Cordillera in southern Spain and Rif in northern Morocco constitute a roughly E-W trending arcuate orogenic belt known as the Gibraltar arc (Fig. 1). This belt formed during the N-S to NW-SE collision between Eurasian and African plates and the westward migration of the so-called Alborán continental lithospheric domain, from Early-Middle Eocene to Early Miocene times (Andrieux et al. 1971; Balanyá and García-Dueñas 1987; Sanz de Galdeano 1990; Platt et al. 2013). The Alborán domain represents the hinterland of this orogen, and is made of mostly supracrustal metamorphic rocks initially organized in a complex stack of nappes. In the Betic Cordillera, these nappes have been grouped within two major complexes: Maláguide on top, and Alpujárride at the bottom (Platt et al. 2013, and references therein) (Fig. 1A). The metamorphic grade of the Alpujárride units increases from east to west in the orogen, such that in the western Betics granulitic migmatites occur at the base of the sequence and appear spatially associated with slices of subcontinental mantle peridotites (i.e. the Ronda peridotites; Lundeen 1978; Obata 1980; Van der Wal and Vissers 1996; Garrido et al. 2011; Précigout et al. 2013). Thus, in the vicinity of the Ronda

peridotites, crustal rocks show systematically the highest metamorphic grade and extensive melting (Loomis 1972; Torres-Roldán 1981, 1983; Balanyá et al. 1997; Tubía et al. 1997, 2013; Argles et al. 1999; Acosta-Vigil et al. 2001, 2014; Platt et al. 2003; Esteban et al. 2008; Barich et al. 2014). This contribution focuses on MI in Grt of mylonitic, former high P granulitic metasedimentary migmatites from the Jubrique Alpujarride unit. Jubrique constitutes a complete though strongly thinned crustal section (≤ 5 km) ranging from carbonates and low-grade phyllites at the top, to schists towards the middle, and to stromatic metasedimentary migmatites and [mylonitic](#) gneisses (former high P granulitic metasedimentary migmatites) at the bottom (Fig. 1a; Loomis 1972; Torres-Roldán 1981; Balanyá et al. 1997; Platt et al. 2003; Barich et al. 2014). All rocks in the sequence are affected by a penetrative foliation parallel to the lithological contacts, that may appear folded at the dm to m scale. The Ronda peridotites constitute a slab of subcontinental mantle up to 5-8 km thick (Ludeen 1978; Balanyá et al. 1997; Torné et al. 1992; Précigout et al. 2013). The [mylonitic](#) gneisses are in contact with the underlying Ronda peridotites along a high temperature ductile shear zone; this contact is parallel to the mylonitic foliation developed in both crustal and mantle rocks, and to the penetrative foliation and lithological contacts in the sequence (Balanyá et al. 1997; Platt et al. 2003; Garrido et al. 2011; Précigout et al. 2013).

The age of deposition of carbonates and phyllites of Jubrique is Permo-Triassic and hence these rocks were deformed and metamorphosed during the Alpine orogeny (e.g. Zeck et al. 1989; Platt et al. 2013). Conversely, schist, migmatites and [mylonitic](#) gneisses are pre-Carboniferous and represent a polymetamorphic basement affected by at least the Variscan and Alpine orogenies (Montel et al. 2000; Whitehouse and Platt 2003; Rossetti et al. 2010; Massonne 2014). Rocks from all levels in the crustal section seem to record nearly isothermal decompression paths, from 1.4-1.2 kbar to 0.6-0.4 GPa at 750-850 °C in the case of the [mylonitic](#) gneisses located at the contact with the Ronda peridotites (Torres-Roldán

1981; Argles et al. 1999; Platt et al. 2003; Barich et al. 2014). The HP-HT event has been related to crustal thickening of the Alborán domain. The main foliation in the rocks postdates HP-HT assemblages (e.g. Grt-Ky-Rt) and predate LP-HT assemblages (e.g. Crd-Sil-Ilm), and hence has been associated with the ductile thinning of the sequence. In this interpretation, Jubrique would represent a thinned and stretched remain of the Alpine collisional thickened crust (Torres-Roldán 1981; Balanyá et al. 1997; Argles et al. 1999; Platt et al. 2003).

[Mylonitic](#) gneisses at the bottom of the crustal sequence were above their solidus during part of their metamorphic evolution. Previous studies have concluded that partial melting occurred during decompression and in the stability field of sillimanite (Argles et al. 1999; Platt et al. 2003). Recent studies focused on the MI, however, have shown that melt was present in the system both at the peak (HP-HT stage) and post-peak (LP-HT) conditions, and that most Grt in the rock grew in the presence of melt (Barich et al. 2014).

Petrography of [mylonitic](#) gneisses and microstructures of melt inclusions

This study deals with the remelting and rehomogenization of MI in garnets of the [mylonitic](#) gneisses of Jubrique –equivalent to the lower gneiss series of Loomis (1972), granulites of Argles et al. (1999) and garnet gneiss of Platt et al. (2003). They constitute a ≈300-500 m-thick sequence of dark, Grt-rich and rather massive rocks, except for the presence of frequent mm-to-cm (rarely dm) Grt-bearing leucocratic bands (former leucosomes) that defines the main foliation of the rock, S_p (Figs. 2a-b). Garnet is present in both leucocratic and mesocratic to melanocratic bands. Thin, Grt-absent and Bt-Crd-bearing leucocratic veins, intruded into the gneisses apparently under ductile-to-fragile conditions, are frequent and crosscut the main foliation at high angle (Barich et al. 2014).

Garnets from sample JU-8 of the [mylonitic](#) gneisses (Fig. 1) were selected as the starting material of this experimental study, due to their abundance in MI (Figs. 2c, d). The

microstructures of these rocks have been described in detailed by Barich et al. (2014) and references therein; we summarize below the most relevant features. [Mylonitic](#) gneisses are mostly mylonitic rocks made of abundant to frequent Grt, Qtz, Pl, Kfs, Ky, Sil and Crd, scarce to rare Bt, and accessory Spl, Gr, Ap, Rt, Ilm, Zrn and Mnz. They show a fine-grained ($\approx 20\text{-}200\ \mu\text{m}$) matrix of $\text{Qtz}+\text{Pl}+\text{Kfs}+\text{Als}\pm\text{Crd}$, that includes porphyroclasts of Grt, Ky and Kfs. Based on microstructural criteria, these minerals have been grouped into three main assemblages. Cores of large ($\approx 5\text{-}20\ \text{mm}$ in diameter) Grt and their unoriented inclusions of Ky, Rt, Pl, Bt, Qtz and melt represent the oldest, peak and pre- S_p high P mineral assemblage ($\approx 850\text{-}800\ ^\circ\text{C}$, $1.4\text{-}1.2\ \text{GPa}$). Rims of large Grt and small ($\leq 3\ \text{mm}$) Grt in the matrix, together with their inclusions of Sil, Ilm and melt, and oriented Sil, Qtz, Pl, Kfs and Ilm in the matrix, constitute a pre-to-syn- S_p , medium-to-low P post-peak assemblage. Undeformed coronas of Sil or $\text{Spl}+\text{Pl}\pm\text{Crd}\pm\text{Kfs}$ around relicts of oriented Ky in the matrix, and of $\text{Crd}+\text{Qtz}+\text{Bt}+\text{Ilm}\pm\text{Kfs}\pm\text{Spl}\pm\text{Pl}$ replacing Grt rims, constitute post- S_p assemblages formed at $\approx 800\text{-}750\ ^\circ\text{C}$ and $0.6\text{-}0.4\ \text{GPa}$. Leucocratic bands, parallel to S_p (Figs. 2a, b), are granitic and composed of Qtz, Kfs, Pl and accessory Grt, Sil and Ilm, with relict Ky and Rt. Although deformed under ductile conditions, they still show some microstructures indicating the former presence of melt, and record melt-rich domains segregated before or during development of S_p . Biotite-Crd bearing leucocratic veins crosscutting S_p are undeformed, mostly made of Qtz and Pl (i.e. tonalitic or trondhjemitic), have subhedral microstructure, and hence contrast in composition and microstructures with respect to former leucosomes.

Melt inclusions appear scattered from core to rim of large and small garnets (Figs. 2c, d). Due to the large proportion of Grt, presence of MI throughout the entire crystal and very small amount of Bt, Grt has been interpreted as a peritectic mineral formed together with the melt during Bt-breakdown melting reactions (see Barich et al. 2014). Most of the MI correspond to *nanogranitoids*, i.e. totally crystallized polycrystalline inclusions (Cesare et al.

2009, [2015](#)). They are isometric, often have negative crystal shapes, range between ≈ 5 to 200 μm in diameter and have a mean size of $\approx 30\text{-}40$ μm (Figs. 2b, c). Melt inclusions in the cores of large Grt occur in the vicinity of single inclusions of Ky and Rt, whereas MI at the rims of large Grt, or in small Grt, are often associated with inclusions of Sil, Ilm and relict Ky and Rt. In general, MI towards the cores of large Grt are smaller ($\approx 5\text{-}15$ μm) with respect to those at the rims (commonly ≥ 20 μm), where some of the crystallized minerals can be identified by optical microscope (Figs. 2c, d). Totally to partially crystallized MI are composed of daughter Qtz, Pl (albite to bytownite), Kfs, ternary feldspar, Bt, Ms, rare calcite, and trapped crystals of Ky (\pm Spl), Gr, Phl, Zrn, Mnz, Rt, Ilm and Ap (Fig. 2e). Crystals of Ky are present within most of the MI, and represent the main solid inclusion that favored the entrapment of MI during Grt growth; they are mostly anhedral and, in MI at the rims of large Grt appear partially replaced by a low-Zn, hercynitic Spl. Ilmenite occurs only within MI located at the rim of large Grt, whereas Rt, Zrn and Mnz have been found throughout the entire host. Offshoots around MI have been observed in a few cases; they are filled with daughter minerals and do not necessarily show a radial distribution. Conversely, MI are commonly affected by late fractures crosscutting the entire Grt. Some small rounded cavities can be interpreted as micro- to nano-porosity; Raman micromapping conducted in similar microstructures from other MI study (Bartoli et al. 2013a) strongly suggest that they were filled with fluid dissolved in the former hydrous melt that exsolved upon crystallization. Some fluid inclusions, of possible primary origin, are spatially associated with MI in clusters within Grt.

Experimental and analytical procedure

We have used doubly polished $\approx 200\text{-}300$ μm - and ≈ 3 mm-thick sections to obtain single MI-bearing Grt chips ($\approx 2 \times 2$ mm) and fragments (up to $\approx 4 \times 4$ mm) containing complete and,

except for the potential presence of late fractures affecting Grt, totally sealed MI. Remelted nanogranitoids come from four different thick sections of sample JU-8, from which several Grt crystals were selected. Garnet chips were separated by cutting manually under a binocular microscope. To investigate potential variations of MI composition with microstructural location, fragments of Grt were collected from three different microstructural positions, including cores of large (≈ 6 mm) Grt, rims of large Grt, and small (≈ 1 -2 mm) single Grt (Table 1).

Piston-cylinder experiments

Remelting experiments of MI contained within fragments of Grt followed the methodology described by Bartoli et al. (2013b), and were conducted using a single-stage, piston-cylinder apparatus at the Laboratory of Experimental Petrology, Dipartimento di Scienze della Terra (Università di Milano, Italy). Chips and fragments of Grt were loaded into Au capsules with external diameter of 3 mm and 5 mm, respectively, together with powdered silica to isolate Grt pieces from each other. No water was added to the capsules, which were crimped and sealed by arc welding. We conducted four experiments (AB1 to AB4, Table 1), each of them containing either three 3 mm capsules or one 5 mm capsule embedded in a low friction, 22 mm assembly composed of a inner MgO plug and capsule container-salts, a graphite heater and a outer NaCl sleeve, 5 mm wall thickness. Capsules were accommodated within the crushable MgO-salt and a thermocouple was positioned above the samples at about the midpoint of the furnace assembly. Experiments were run for 24 hours at a constant pressure of 1.5 GPa and at temperatures of 850, 825 or 800 °C. The temperature range was chosen based on previous thermobarometric estimates of peak and post-peak metamorphic conditions (Loomis 1972; Torres-Roldán 1981; Argles et al. 1999; Platt et al. 2003; Barich et al. 2014), whereas the temperature sequence (starting with 850 °C and continuing down to 825 °C and 800 °C) was dictated by the experimental results (see below). We chose the highest pressure

value obtained in most of the previous thermobarometric studies, 1.5 GPa, in order to maintain a pressure on the Grt fragments equal or larger than the internal pressure in the remelted MI, and therefore to prevent MI decrepitation and volatile loss upon heating. Temperature was controlled by K-type (chromel-alumel) thermocouples and is considered accurate to ± 5 °C (Ferri et al. 2009). Using a Johannes-type piston cylinder, pressure can be directly measured by a load cell built into the frame (Johannes 1973) and is considered to be accurate to ± 0.043 GPa (~~Molina and Poli 2000~~ Schmidt 1992). Calibration was performed against the reaction albite = jadeite + quartz (Johannes et al. 1971) and no friction was observed compared to values obtained from load cell as a result of the large volume NaCl salt sleeve used. During each run, pressure was first increased to 1.5 GPa and then the assembly was isobarically heated at a constant heating rate (≈ 50 °C/min) until the target temperature was reached. After 24 h of run time, experiments were quenched isobarically by turning off power to the heater, at a rate of ≈ 50 °C/sec down to ≈ 50 °C. The confining pressure was released after quenching. Capsules were mounted in epoxy, and the inclusions in the Grt were exposed gradually by manual polishing, using first 4000- to 80- μm grits sizes, and 5- and 1- μm diamond suspensions for the final polishing stage.

Analytical techniques

Back-scattered electron (BSE) imaging and semi-quantitative energy dispersive spectroscopy (EDS) of the analyzed remelted MI were carried out using a CAM Scan MX2500 Scanning Electron Microscope (SEM) equipped with LaB₆ cathode, at the Dipartimento di Geoscienze of the Università di Padova (Italy), and a QUANTA 400 environmental SEM equipped with EDAX EDS (ultrathin window) and Li(Si) detectors, at the Centro de Instrumentación Científica (CIC) of the Universidad de Granada (Spain).

The major element composition of glass in remelted MI was analyzed with Jeol JXA 8200 and Cameca SX-50 Electron Microprobes (EMP) at the Dipartimento di Scienze della

Terra (Università di Milano) and Dipartimento di Geoscienze (Università di Padova), respectively. To minimize alkali loss and changes in major elemental ratios, two analytical conditions were used as recommended by Morgan and London (1996, 2005). Sodium, K, Al and Si were analyzed first (and concurrently in the case of the Jeol JXA 8200), using a beam current and beam diameter of 2 nA and 1 μm , respectively, and accelerating voltages of 15 kV (Jeol JXA 8200) or 20 kV (Cameca SX-50). The elements Fe, Mn, Mg, Ti, Ca, P, F and Cl were analyzed afterwards with 15-kV, 20-nA, 1- μm (Jeol JXA 8200) or 20-kV, 20-nA, 1- μm (Cameca SX-50) beams. Counting times were 10 s on peak for all elements (except Fe, Mg and F with 20 s), and 10 s (Cameca SX-50) or 2 s (Jeol JXA 8200) on background. Matrix reduction used the PAP correction algorithm (Pouchou and Pichoir 1985). Analyses were corrected using anhydrous and hydrated haplogranite glasses of known composition as secondary standards (Morgan and London 2005). The standards were analyzed at the start and end of each analytical session using similar working conditions. Sodium loss was estimated as $\approx 20\%$ relative during these analyses. Initial estimates of water concentrations in glass were calculated by the difference of electron microprobe totals from 100% (Table 2). Morgan and London (1996) and Acosta-Vigil et al. (2003) showed that using the above analytical methods on granitic glasses, the accuracy of H_2O by difference is better than $\pm 10\%$ relative for H_2O concentrations in the range of 2-10 wt%.

After a detailed optical and SEM investigation of the experimental run products to check for MI homogeneity and absence of cracks in the host Grt, we identified 26 remelted nanogranitoids within 8 Grt crystals appropriate to conduct a detailed determination of H_2O concentrations in experimental glasses. Analyses of H_2O were performed by Nano Secondary Ion Mass Spectrometry (NanoSIMS) using the Cameca NanoSIMS 50 at the Muséum National d'Histoire Naturelle of Paris. Polished experimental capsules with MI exposed on the Grt surface and standard glasses were mounted in In to reduce H background in the

analysis chamber (Aubaud et al. 2007). Melt inclusions were identified through images of $^{28}\text{Si}^-$, $^{39}\text{K}^{16}\text{O}^-$ and $^{56}\text{Fe}^{16}\text{O}^-$ secondary ions. For each of the analyses, we first performed a pre-sputtering step on a $3 \times 3 \mu\text{m}^2$ surface area for 2 minutes with a 400 pA primary Cs^+ beam to remove the gold coating, surface contamination and to reach a steady state sputtering regime. Then a primary beam of 37 pA was used for data acquisition. Data were acquired by rastering a $3 \times 3 \mu\text{m}^2$ surface area and collecting only ions from the inner $1 \times 1 \mu\text{m}^2$ (beam blanking mode) to reduce surface contamination. Each analysis is a stack of 200 cycles, a cycle being 1.024 s long. $^{16}\text{OH}^-$ (used as a proxy for H_2O), $^{28}\text{Si}^-$, $^{39}\text{K}^{16}\text{O}^-$ and $^{56}\text{Fe}^{16}\text{O}^-$ were recorded simultaneously in multicollection mode. We checked that the $^{16}\text{OH}^-/^{28}\text{Si}^-$ ratio was stable during MI analyses. Secondary ions were collected by electron multipliers with a dead time of 44 ns. Mass resolution was set to 8000 to resolve any mass interference on the selected ions. Several of the analyzed MI were large enough to conduct replicated analyses. Three leucogranitic glasses with well-known H_2O concentrations, varying between ≈ 0 -7 wt%, were used for NanoSIMS calibration (Fig. 3): glass DL reported in Acosta-Vigil et al. (2003) with $\text{H}_2\text{O}=6.5$ wt%; glass LGB1 from Behrens and Jantos (2001) with $\text{H}_2\text{O}=4.9$ wt% (uncertainties in these analyses are $\pm 13\%$ relative); and the almost anhydrous glass B from Morgan and London (2005) with $\text{H}_2\text{O}=300 \pm 42$ ppm. Data corrections using the aforementioned calibration, and error calculations, were performed using the R program (Graybill 1976). Although errors combine counting statistic and uncertainty of the calibration curve (Fig. 3), the errors reported in Table 3 are dominated by the latter, which corresponds to a prediction interval at 68%. During the NanoSIMS sessions, the vacuum in the analysis chamber remained between 2.5 and 5×10^{-10} Torr. The detection limit for water was around 650 ppm.

The precise hydrogen concentrations of the three reference samples used in the calibration curve reported above were measured by Elastic Recoil Detection Analysis

(ERDA). This method has been used as a reference for H determination in various materials, including geological samples with a large range of H₂O concentrations from several wt% to hundreds of ppm in nominally anhydrous minerals (Raepsaet et al. 2008; Aubaud et al. 2009; Bureau et al. 2009; Withers et al. 2012). Analyses were conducted at the Nuclear Microprobe of CEA Saclay (Khodja et al. 2001), using the 3.75 MV Van de Graaff single stage accelerator from HVEE which delivered a 3 MeV ⁴He⁺ incident beam with a size of 12 x 3 μm² and a current of 950 pA, that impinged the target at a grazing angle of 15°. Ejected H atoms were collected in a silicon barrier detector placed at 30° from the incident beam and at 50 mm from the surface of the sample, protected against the backscattered He ions by a 15 μm Al filter. Homogeneous regions of analysis, excluding cracks or bubbles in the glass, were selected by mapping the major components using Particle Induced X-Ray emission (PIXE) and backscattered helium ions spectroscopy (Rutherford Backscattering, RBS).

Results

Microstructures of remelted nanogranitoids

All exposed MI from the experimental runs (850 to 800 °C) show glass (former melt). The extent of melting, however, is variable regardless of temperature and microstructural location of MI. Exposed remelted MI vary in size from ≈5 to 200 μm and, in addition of glass, they commonly have Als and, less frequently Gr, Zrn, Rt, Sp and Mnz crystals. The large size of the crystals compared to the MI, and the low solubility of these minerals in granitic melts (e.g. Watson and Harrison 1983; Montel 1993; Acosta-Vigil et al. 2003; Stepanov et al. 2012; Boehnke et al. 2013), indicates that they are solid inclusions (i.e. were already present during the entrapment of the MI) and hence are not expected to dissolve into the melt during the experiments (Table 1). In fact, these minerals are commonly present in most of the starting material, i.e. the unmelted MI, sometimes with clear microstructures indicating their

accidental nature (Fig. 2e, and Barich et al. 2014). Among the potential daughter minerals (i.e. those that commonly constitute major or minor minerals in granitoid rocks), Bt and Qtz are the most commonly present in the studied remelted MI (Table 1). Hereafter, “rehomogenized MI” refers to MI that after experimental remelting: (i) have negative crystal (or at least regular) shape with no decrepitation cracks; (ii) show no reaction with the host Grt, such as recrystallization of the Grt or crystallization of new minerals at the MI-Grt interface; and (iii) is formed by glass \pm accidental minerals, with no reactions between accidental minerals and melt, and no coexisting vapor –i.e. bubbles (Danyushevsky et al. 2002; Audetat and Lowenstern 2013). Conversely, “remelted MI” refers to MI having glass but lacking some or all of the above characteristics.

The first experiments, conducted at 850 °C, showed few rehomogenized MI and abundant disequilibrium microstructures (Figs. 4a-d). Melt inclusions failed to rehomogenize as attested by the presence of: (i) frequent daughter crystals; (ii) reaction between accidental Als and melt to form St (Als is never rimmed by St in the starting materials, see Barich et al., 2014); (iii) irregular MI walls; and (iv) thin (\approx 1-10 μ m) recrystallized Grt domains at the Grt-MI boundary, indicated by variation of grey tone in BSE images with respect to the rest of the starting Grt (Table 1). Within MI, euhedral rims of St grow on, and replace Als after reaction with the melt, which may have produced a change in the composition of the originally trapped melt. This reaction, together with the presence of recrystallized Grt, decrepitation cracks and irregular MI boundaries, suggest that these inclusions were overheated.

Experiments conducted at 825 °C also show a low proportion of apparently rehomogenized MI and abundant disequilibrium microstructures as those described above, i.e. persistence of daughter minerals, reaction of Als+melt to St, irregular MI walls, presence of offshots and recrystallization of Grt at the Grt-MI boundaries (Figs. 4e-h, Table 1). No

clear differences between microstructures in remelted MI at the cores and rims of large Grt crystals have been observed, and hence 825 °C (at 1.5 GPa) is not the appropriate homogenization temperature for MI at any of the microstructural locations.

Remelted nanogranitoids at 800 °C show a much closer approach to equilibrium (Figs. 4i-l). With respect to the higher T experiments, the proportion of apparently rehomogenized MI increases (up to ≈35 %), Als appears stable and is not replaced by St, walls of MI are mostly regular and many of them show negative crystal shapes, and offshots are nearly absent (Table 1). No microstructural differences have been observed among the different locations, except for the case of MI within small Grt that seem somewhat further from equilibrium compared to MI at cores and rims or large Grt.

Composition of glass in remelted nanogranoids

We have conducted a total of 172 EMP glass analyses on 81 remelted/rehomogenized MI, from which 94 of the analyses have been initially used to investigate the composition of anatectic melt; the rest of analyses show clear signals of contamination by either the host Grt or minerals present within the MI, with e.g. $\text{SiO}_2 < 60$ wt%, $\text{Al}_2\text{O}_3 > 20$ wt% and $\text{FeO}_t + \text{MgO} + \text{TiO}_2 \geq 7$ wt%. These 94 analyses are shown in Fig. 5new, where the rough negative correlation between NaO_2 and ASI, and the very low NaO_2 concentrations and very high ASI values of some analyses seem to indicate severe alkali loss during electron microprobe analyses. Hence we have only selected about 70 analyses showing $\text{NaO}_2 \geq 2$ wt% and $\text{ASI} \leq 1.5$ (see Acosta-Vigil et al. 2003 for ASI values of granitic melts at equilibrium with a variety of frequent peraluminous minerals in crustal rocks) to calculate the mean glass compositions of Table 2 and plot in diagrams of Figs. 5 and 6. These analyses include glasses from both remelted and rehomogenized MI, and have moderate to strongly peraluminous granitoid compositions [$\text{ASI} \approx 1.05-1.50$, mean=1.31; $\text{ASI} = \text{mol. Al}_2\text{O}_3 / (\text{CaO} + \text{Na}_2\text{O} + \text{K}_2\text{O})$], with mostly high SiO_2 ($\approx 62-74$ wt%, mean=66 wt%), low $\text{FeO} + \text{MgO} + \text{TiO}_2$ ($\approx 1-4$ wt%,

mean=[2.4 wt%](#)), and variable CaO, alkalis and H₂O concentrations ([reported values are on a hydrous basis](#); Table 2). They distribute into two distinct compositional groups. **Type I MI** show low concentrations of CaO (≈ 0.3 - 1.0 wt%, [mean= \$0.66\$ wt%](#)), variable though mostly low H₂O (≈ 2 - 8 wt%, mean=[6](#) wt%; H₂O by difference), higher K₂O (\approx [2.0](#)- 6.5 wt%, [mean= \$4.5\$ wt%](#)) and K# [mean=[0.58](#); K#=mol. K₂O/(K₂O+Na₂O)] and lower Mg# [≈ 0.30 ; Mg#=mol. MgO/(MgO+FeO_i)] (Fig. 5). These glasses are granitic in composition (Fig. 6a) and, in Qtz-Or-Ab normative pseudoternary diagrams, they plot in the vicinity of H₂O-undersaturated haplogranitic eutectics [and Qtz-Or cotectic lines](#) (Fig. 6b) [as](#) most of the previously analyzed glassy MI and rehomogenized nanogranitoids reported in the literature (Cesare et al., [2015](#); [Bartoli et al. in press](#)). **Type II MI** have moderate to high CaO (≈ 1.5 - 3.5 wt%, [mean= \$2.44\$ wt%](#)), high H₂O (8 - 15 wt%, mean= 12 wt%; H₂O by difference), lower K₂O (≈ 1.0 - 2.5 wt%, [mean= \$1.72\$ wt%](#)) and K# (≈ 0.35) and higher Mg# (≈ 0.40). These glasses are granodioritic, trondhjemitic and tonalitic in composition (Fig. 6a) and, unlike most of the previously analyzed natural or experimental glasses coming from the partial melting of metasedimentary rocks, they plot far from the haplogranitic eutectics and surprisingly close to the Qtz-Ab sideline of the normative Qtz-Or-Ab pseudoternary diagram (Fig. 6b). In addition, they define a broad trend parallel to the Qtz-Ab [join](#).

When glass within large single MI is analyzed [in](#) two or more locations, it shows a rather homogeneous composition, and hence each of the analyzed remelted and rehomogenized MI pertains to either type I or type II. The two compositional groups of MI are unrelated to the location of glass either in remelted or rehomogenized MI. Instead, [and although there is still some degree of uncertainty](#), these groups [appear to be](#) mostly related to the microstructural location of the MI. Thus, type I is mostly formed by MI coming from the cores of large Grt, whereas type II is mostly constituted by MI present at the rims of large Grt and within small Grt ([see below](#)).

The H₂O concentrations of glass in remelted and rehomogenized MI determined by NanoSIMS span a wide range of values between \approx 3-14 wt% (Table 3). Figure 7 compares glass H₂O concentrations measured by NanoSIMS and estimated by EMP-difference. For the majority of analyzed MI (\approx 60 %) the two techniques provide quite similar results, as differences between both methods are \leq 30% relative, in good agreement with results from previous analyses of MI by NanoSIMS (Bartoli et al. 2014). However, a group of analyzed MI shows concentrations of H₂O by difference much higher (\geq 40 % relative) than those by NanoSIMS (Fig. 7). This discrepancy has been mainly observed in MI located in the proximity of large Grt cavities produced during sample polishing. Because the ¹⁶OH⁻ and ²⁸Si⁻ signals were rather constant during analysis of MI away from these cavities, but variable during analysis of MI close to the cavities, we ascribe the large differences between H₂O concentrations by both methods to NanoSIMS instrumental instability associated with the presence of Grt cavities. Nevertheless, the NanoSIMS analyses confirm the EMP H₂O estimates, and in particular the high H₂O content of type II MI.

Discussion

P-T conditions of anatexis at Jubrique

The microstructures of the experimental run products show that the frequency of disequilibrium features increases from 800 °C to 825-850 °C (Table 1, Fig. 4). Disequilibrium features include: (i) the presence of rather irregular and ragged boundaries between MI and host Grt, as opposed to straight, negative crystal shape boundaries; (ii) the change in composition of the host Grt at the boundaries with MI with respect to the starting composition, indicated by the variation in grey tones of BSE images and implying Grt-melt chemical interaction during the experiment (e.g. Perchuk et al., 2008); (iii) the destabilization of accidental Als to form St at the contact with melt; (iv) the presence of offshots, suggesting

decrepitation; and (v) the presence of abundant partially resorbed daughter minerals. Except for the presence of daughter minerals and rare offshots, none of these features have been observed in the natural starting material (Barich et al. 2014). Concomitantly, there is a decrease in the proportion of apparently rehomogenized MI with increasing temperature, from 20-35% at 800 °C to 15-25% at 825 and 850 °C. In the only previous systematic study on the remelting of nanogranitoids from anatectic terranes, Bartoli et al (2013b) have found that the proportion of rehomogenized MI at the P-T conditions of anatexis of the rocks was \approx 30-40%. They found equilibrium features in the rehomogenized MI such as negative crystal shape, absence of any daughter granitic minerals or bubbles, and presence of glass with typical leucogranitic compositions similar to those produced in experimental studies on crustal anatexis. The MI studied by Bartoli et al. (2013a, 2013b) are much smaller (\approx 5-15 μ m across) than those studied from Jubrique. Considering the much larger size of MI in Jubrique, and the increase in rehomogenization timeframes with MI size found in previous studies on the remelting of crystallized MI (e.g. Thomas et al. 1996; Thomas and Klemm 1997; Bodnar and Student 2006; Zajacz et al. 2008), we conclude that: (i) the proportion of 20-35 % rehomogenized MI found at 800 °C is relatively high and likely large enough for the glass analyses to be representative of the trapped melt compositions; and (ii) the temperature of 800 °C is likely close to that of anatexis and MI entrapment in the studied rocks.

We have not considered variations in P during this experimental study. Among all the previously reported peak P for these rocks, including classical thermobarometric and phase equilibria modeling calculations (Loomis 1972; Torres-Roldán 1981; Balanyá et al. 1997; Argles et al. 1999; Platt et al. 2003; Barich et al. 2014; Massonne 2014), we have chosen the upper value of 1.5 GPa provided by most of these studies, expecting that this imposed external P on the Grt chips would prevent decrepitation of included MI due to increase in their internal P upon heating (e.g. Bartoli et al. 2013a). Following most of the previous

petrologic studies, this P would correspond to that of generation of Grt cores (Loomis 1972; Torres-Roldán 1981; Argles et al. 1999; Platt et al. 2003; Barich et al. 2014). These experiments, conducted at a single P, cannot provide precise constraints on the P of anatexis of the host former migmatites. However, experimental results support that the P of anatexis should have been similar to or lower than 1.5 GPa, given that a relatively large number of studied MI did not decrepitate and rehomogenized at a combination of expected P-T anatectic conditions from previous thermobarometric studies, i.e. 800 °C and 1.5 GPa. It is unclear yet if departures of remelting experimental P above that of anatexis and MI entrapment would produce Grt-melt chemical interactions and hence lack of rehomogenization. Scarce experimental results on this issue suggest that departures of >0.5 GPa produce interaction of host Grt with melt and crystallization of new minerals (Ferrero et al. 2015). Nevertheless, further detailed experiments considering variations in P are required to investigate the effect of P on MI rehomogenization.

Significance of glass compositions in remelted nanogranitoids

The major element concentrations of analyzed glasses define two compositional groups corresponding to leucogranites (type I) and [granodiorites, trondhjemites and tonalites \(hereafter granodiorites-to-tonalites; type II\)](#) (Table 2, Figs. 5, 6). Compared to the leucogranitic, granodioritic-to-tonalitic glasses represent the majority of the EMP analyses (80%), found within most (75%) of analyzed remelted/rehomogenized nanogranitoids. [Considering the 850 °C and, in particular, 800 °C experiments \(as the latter show the highest proportion of homogenized MI of all conducted experiments\), virtually all of granodioritic-to-tonalitic glasses](#) come from remelted/[rehomogenized](#) MI located at the rims of large Grt or within small Grt, interpreted to record the latest stages in the evolution of the migmatites (Loomis 1972; Torres-Roldán 1981; Argles et al. 1999; Platt et al. 2003; Barich et al. 2014; Massonne 2014); [whereas all](#) of leucogranitic glasses come from remelted/rehomogenized

MI located at the cores of large Grt, interpreted to record the first documented stages in the evolution of these rocks (op. cit.) (Figs. new# and 6a). However, analyzed remelted/rehomogenized MI in the 825 °C experiments shed some doubts on the previous statement, as most glasses analyzed in a fragment of apparently large Grt core (AB3-Core8) are granodioritic-to-tonalitic, whereas half of glass analyses from a fragment of apparently large Grt rim are leucogranitic (AB2-Rim8; see Table 2 and Fig. 6a). Garnet in the studied rocks are zoned with respect to the major elements, with large Grt cores having generally lower Ca concentrations (CaO \approx 1.0-2.5 wt%) with respect to large Grt rims or small Grt (CaO \approx 1.5-5.0 wt%; Barich et al. 2014, and Fig. new#). To investigate the presence of granodioritic-to-tonalitic MI and leucogranitic MI apparently at the cores and rims, respectively, of large Grt used in the 825 °C experiments (Table 2), we have analyzed the composition of garnet at the very contact with these MI, and compared with the composition of garnets at the contact with MI in the 850 °C and 800 °C experiments (Table 3) and garnets analyzed from the starting rock JU-8 (Table new#, Fig. new#). The major element compositions of these garnets are not expected to have been modified by diffusion during the short 24-h remelting experiments (e.g. Caddick et al. 2010). We found that all garnets next to MI in the 825 °C experiments show intermediate compositions between previously analyzed garnet cores and rims (CaO \approx 2.0-2.5 wt%), whereas garnets in the 850 °C and 800 °C experiments show either high CaO concentrations when they are next to type II MI, or low CaO when next to type I MI. Hence we conclude that an explanation for the apparently contradictory information provided by glass analyses in the 825 °C experiments is that garnet fragments used in these experiments were mixed core-rim domains.

Leucogranitic compositions have been so far the most frequently reported in (i) bulk rock analyses of leucosomes in metasedimentary/metagranitic migmatites (e.g. Sawyer 1996, 2008; and references therein), (ii) glass analyses from the relatively few reported studies of

glassy MI in metasedimentary/metagranitic anatectic enclaves and rehomogenized nanogranitoids in migmatites and granulites (Cesare et al. 1997; 2003; 2009; 2011, [2015](#); Acosta-Vigil et al. 2007; Ferrero et al. 2012, 2014, 2015; Bartoli et al. 2013a, 2013b, [in press](#)), and (iii) glass analyses in experimental studies on the fluid-absent anatexis of metasedimentary rocks (e.g. Vielzeuf and Holloway 1988; Patiño Douce and Johnston 1991; Montel and Vielzeuf 1997; Patiño Douce and Harris 1998; Schmidt et al. 2004; Hermann and Spandler 2008). The latter tell us that leucogranitic melts are [typically](#) produced [by](#) the dehydration melting of micas in metasedimentary/metagranitoid rocks, at moderate-to-low H₂O activities, within a wide range of mostly crustal P-T ($\approx 750\text{-}900$ °C, 0.1-1.3 GPa), but also at mantle conditions (up to 1000 °C, 2.5-5 GPa) (op. cit.).

Compared to leucogranitic, granodioritic-to-tonalitic compositions are less frequently reported in bulk rock analyses of leucosomes and/or dikes/veins from metasedimentary anatectic terrains (e.g. Whitney and Irving 1994; García-Casco and Torres-Roldán 1996; Newton et al. 1998; Sawyer 2008; Morfin et al. 2013; Barich et al. 2014). A single study of rehomogenized MI in Grt from metasedimentary migmatites at the base of the Greater Himalayan Sequence (Kali Gandaki, Nepal) has recently reported tonalitic glass compositions (Carosi et al. 2015). Experimental granodioritic-to-tonalitic, CaO-rich, #K-low melts have been obtained during the H₂O-saturated partial melting of metasedimentary rocks at $\approx 675\text{-}775$ °C and 0.6-1.4 GPa (Patiño Douce and Harris 1998; García-Casco et al. 2003; Ferri et al. 2009). [Accordingly, some of the studies of metasedimentary migmatites \(e.g. Whitney and Irving 1994\) and the recent study on the rehomogenization of MI by Carosi et al. \(2015\) have interpreted that granodioritic-to-tonalitic compositions represent primary melts formed during H₂O-saturated anatexis. However, granodioritic-to-tonalitic melts have also been produced during experimental partial melting of intermediate-to-basic rocks under a wide range of conditions: H₂O-saturated to fluid-absent, \$\approx 775\text{-}1150\$ °C and 0.5-3.2 GPa](#)

(e.g. [Helz 1976](#); Rutter and Wyllie 1988; Hacker 1990; Rushmer 1991; Rapp et al. 1991; [Wolf and Wyllie 1994](#); Rapp and Watson 1995; [Skjerlie and Patiño Douce 2002](#); Schmidt et al. 2004; [Laurie and Stevens 2012](#); [Quian and Hermann 2013](#)).

Implications for the geodynamic evolution of lower continental crust in the Betic Cordillera

The majority (75%) of the analyzed remelted/rehomogenized MI have granodioritic-to-tonalitic glass, and most of the reported analyses (Table 2, Figs. 5, 6) come from apparently rehomogenized MI showing either glass or glass plus solid inclusions such as Als or Zrn. We notice that, in addition to granodioritic-to-tonalitic MI, Barich et al. (2014) have described the presence of tonalitic veins crosscutting the main foliation of [mylonitic](#) gneisses, emplaced/segregated during the late history of these rocks. Based on experimental work (see above), one interpretation is that granodioritic-to-tonalitic melts at Jubrique may represent exotic liquids formed during the HP partial melting of a deeper intermediate-to-mafic source, and later segregated and intruded into metasedimentary rocks of upper crustal levels. However, the presence of granodioritic-to-tonalitic melt as inclusions within one of the major mineral components of these rocks, i.e. Grt, rather indicate [that these veins were produced during](#) anatexis of the host rock [with](#) growth of peritectic Grt. In addition, the existence of tonalitic veins is compatible in composition and timing with the presence of granodioritic-to-tonalitic MI at the rims of large garnets and within small garnets, recording the late magmatic history of these former migmatites. Hence we conclude that granodioritic-to-tonalitic glasses represent primary compositions of melt formed at some point during the anatectic history of these rocks, and particularly during partial melting under H₂O-rich fluid-present conditions (see above, and compare with Patiño-Douce and Harris 1998; García-Casco et al. 2003; Ferri et al. 2009).

Remelted/rehomogenized MI showing glass with leucogranitic composition are much less abundant, and their significance and meaning is more difficult to ascertain. On one hand,

they may record former melts produced during partial melting of host migmatites under fluid-absent conditions (see above), and hence compositions of former MI may be compatible with two different melting scenarios of their host polymetamorphic metasedimentary rocks. On the other hand, leucogranitic glasses may result from some other processes, such as (i) the incomplete remelting of the MI, as MI were not rehomogenized in all cases; or (ii) the entrapment of a compositionally heterogeneous melt present in the partially melted rock, i.e. analyzed glasses represent natural compositions controlled by the kinetics of melting, and not by chemical equilibrium at constant P-T-X. We discuss below these two interpretations.

Although some leucogranitic glasses come from the analysis of partially crystalline MI (Fig. 4l), none of these partially remelted MI show any visible Ca-rich daughter mineral whose dissolution could produce, upon complete rehomogenization, a compositional shift towards Ca-rich, K-poor concentrations. In addition, the first melt fractions formed upon heating of a granitoid assemblage (i.e. the nanogranitoid) are expected to have the highest H₂O concentrations, as opposed to the lowest H₂O concentrations shown by leucogranitic glasses. Moreover, some of the leucogranitic glasses come from rehomogenized MI (Fig. 4j). Hence, and although the total number of rehomogenized leucogranitic MI is small, we conclude that leucogranitic melts have not been artificially produced during the experiments due to incomplete remelting of MI, but were present at some point during the anatexis history of the studied migmatites.

The trapping of a heterogeneous melt during a single, Grt-producing melting event in the migmatites is an explanation for the presence of leucogranitic and granodioritic-to-tonalitic melt compositions. The current experimental study has been conducted using a single rock sample (JU-8) but several (≈ 10) Grt crystals coming from a decimetric fragment of that sample. Distances between the studied Grt in this rock, therefore, are at least in the range of a

few cm. Melt interconnection in partially melted metasedimentary rocks is expected to occur at the temperatures registered in the migmatites (≥ 800 °C; Laporte et al. 1997; Clemens 2006, and references therein). However, the interconnected melt is likely to be compositionally heterogeneous due to the sluggish diffusion of Si and Al in melt (Acosta-Vigil et al. 2006a, 2012b). Thus, melt in the vicinity of dissolving quartz crystals will be enriched in SiO₂ with respect to that close to feldspars, which will have higher Al₂O₃ concentrations (Acosta-Vigil et al. 2006b). Conversely, H₂O concentrations and, particularly, the ratio of alkalis throughout an interconnected melt network will show much lower concentration gradients, due to fast diffusion of these components in melt (Acosta-Vigil et al. 2002, 2006a, 2006b; Morgan et al. 2008). These diffusion systematics of granite melts can explain the linear trend shown by granodioritic-to-tonalitic glass compositions in the normative Qtz-Or-Ab pseudoternary diagram, i.e. [the presence of](#) melts with very similar alkali ratios, but variable Si/Al ratios [\(compare with Fig. 10 of Acosta-Vigil et al. 2006b\)](#). However, they cannot explain the coexistence of granodioritic-to-tonalitic and leucogranitic melts, constituting two well-defined clusters in the Qtz-Or-Ab and Harker diagrams, and characterized by contrasting H₂O concentrations and alkali ratios (Figs. 5, 6).

The above evidence, together with the distribution of leucogranitic MI at the cores of large Grt and granodioritic-to-tonalitic MI at the rims of large Grt or within small Grt (Table 2 [and Fig. new#, though considering some degree of uncertainty in this statement](#)), may suggest that former migmatites at Jubrique represented by the [mylonitic](#) gneisses underwent two melting events under contrasting fluid regimes. The first anatexis event occurred under fluid-absent and low a_{H₂O} conditions, and was recorded by MI at the cores of large Grt. A second partial melting event took place in the presence of H₂O-rich fluids and was recorded by MI at the rims of large Grt and in small Grt of the matrix. Several geochronological studies have shown that basement rocks in the studied area of the Alpine Betic Cordillera still

preserve Variscan minerals and/or fabrics (Acosta 1998; Sanchez-Rodríguez 1998; Zeck and Whitehouse 1999, 2002; Acosta-Vigil et al. 2014; Sánchez-Navas et al. 2014). In particular, studies of the [mylonitic](#) gneisses of Jubrique have shown that Grt cores likely formed during the Variscan orogeny, whereas Grt rims and the matrix of the rock may have crystallized during the Alpine (Whitehouse and Platt 2003; Massonne 2014; see also Montel et al. 2000; Rossetti et al. 2010). We tentatively suggest that the two reported glass compositions may reflect the anatexis of the host migmatites during two different orogenic events. Variscan anatexis formed the cores of large Grt and their leucogranitic MI, likely during the fluid-absent melting of Bt at ≈ 800 °C and 1.4-1.2 GPa. Alpine anatexis [would have](#) produced the growth of Grt rims on previous Variscan Grt and formed new small Grt in the matrix, together with the trapped granodioritic-to-tonalitic MI. This occurred during H₂O-rich fluid-present melting of the rock at similar T but lower P conditions (≈ 800 °C and 0.8-0.6 GPa), and associated with an incongruent melting reaction involving Grt growth.

Significance for melt inclusion studies, [and](#) crustal melting and differentiation

Cesare et al. (1997) and Acosta-Vigil et al. (2007, 2010) have documented variations in the composition of glass (former melt) in metasedimentary anatectic enclaves (El Hoyazo, S Spain) as a function of microstructural location. Thus, glassy MI in Pl have different composition from glassy MI in Grt, which are also different in composition from matrix glass. Acosta-Vigil et al. (2007, 2010, 2012a) have interpreted these variations as reflecting the evolution of melt composition during prograde anatexis and, on this basis, have provided information on the nature and mechanisms of anatexis in the enclaves during the prograde path, including melting reactions, fluid regimes, degree of melt homogeneity and extent of melt-residue equilibration. Later on, and during the novel studies of glassy and remelted MI in migmatites and granulites, Bartoli [et al. \(2015\)](#) have documented variations in the composition of MI in Grt, this time as a function of the structural location of the host

[quartzo-feldspathic](#) migmatite in the anatectic sequence of Ojén (Ronda, S Spain), and in turn of the T of formation. Thus, MI in Grt of lower T metatexites have [lower FeO and H₂O concentrations and #K values compared](#) to MI in Grt of higher T diatexites [which, coupled with a thorough microstructural and petrologic work, was interpreted to reflect the evolution of primary anatectic melt along the prograde anatectic path](#). The current study shows that MI composition may vary as a function of its microstructural location within a single mineral in the rock, i.e. Grt cores versus Grt rims. All the above indicates that MI compositions may vary systematically and at different scales: within a single mineral, among different minerals in the same rock, and among crystals of a single mineral present [in a particular protolith](#) throughout a migmatitic sequence showing variations in T of formation. Hence, as in the studies of MI in anatectic enclaves (Acosta-Vigil et al. 2010), detailed investigation of MI in migmatites and granulites can supply information on the evolution of melt composition during the anatectic history of the rocks, as well as on the nature and mechanisms of the process of partial melting (see also Cesare et al. [2015](#)).

Recently, Aranovich et al. (2014) have discussed the potential role of the mantle as a source of extra heat and fluids to drive anatexis at deep crustal levels, melt ascent, and in turn differentiation of the continental crust. Among the problems raised by these authors against a pure [closed-system \(except for the extraction of granitic liquids\)](#), fluid-absent incongruent melting model for anatexis and crustal differentiation, there is the presence of non-granitic – e.g. tonalitic– leucosomes in migmatites. The presence of low H₂O concentration leucogranitic MI at the cores of Grt in [the studied](#) former migmatites suggests that [supracrustal rocks such as metapelites were brought to](#) the bottom of a thickened continental crust [where they](#) partially melted under fluid-absent conditions. These observations are in accordance with a fluid-absent melting model for anatexis and, in the case of melt extraction and ascent, crustal differentiation. However, based on the experimental results of Patiño

Douce and Harris (1998), García-Casco et al. (2003) and Ferri et al. (2009), granodioritic-to-tonalitic MI at the rims of Grt also indicates that anatexis at mid-to-lower levels of an average continental crust took place in the presence of an H₂O-rich fluid (see also Carosi et al. 2015). Aranovich et al. (2013, 2014) have proposed that [strongly saline \(Cl-rich\), H₂O-bearing fluids \(brines\) coming from a variety of mechanisms \(e.g. metamorphic fluids enriched in salts by loss of H₂O during hydration reactions, or the crystallization and degassing of basaltic magmas; see also Yardley and Graham 2002\)](#) are important agents for open-system metamorphism and anatexis of deep crustal levels. The analyses of glass reported in this contribution show very low proportions of halogens and high concentrations of H₂O (Table 2). [Glasses show H₂O concentrations at or close to saturation at the inferred P or melting, ≈0.8-0.6 GPa. Also, they have virtually no F, and Cl concentrations \(0.10-0.15 wt%\) are much lower than the saturation values obtained in experimental granite melts coexisting with brines reported by Aranovich et al. \(2013\) \(0.17-0.71 wt%\) or Safonov et al. \(2014\) \(0.24-1.63 wt%\). In addition, melt compositions produced during melting of a granite assemblage in the presence of brines at or near the solidus correspond to K-rich metaluminous granites \(Aranovich et al. 2013; Fig. #b\), and not to peraluminous granodiorites, trondhmemites or tonalites. Increasing the proportion of melt will displace this composition towards that of the bulk rock \(blue symbol in Fig. b\). Hence we conclude that compositions of the granodioritic-to-tonalitic glasses do not support anatexis due to the presence of saline, but H₂O-rich fluids.](#)

Based on theoretical grounds [and the inferred temperatures and initial H₂O concentrations of high level granitoid magmas](#), Clemens and Watkins (2001) have concluded that the processes of crustal melting, genesis of granitoid magmas and crustal differentiation occurs in the absence of excess pervasive fluid. However, the only direct available method to actually measure in situ the proportion and nature of volatiles in primary crustal melts, and

hence to obtain precise information on the fluid regime during crustal anatexis, is the detailed study of MI (Cesare et al. 2011, [2015](#); Bartoli et al. 2013a, 2014). And the current investigation tells us that H₂O-rich fluid-present partial melting did occur in metapelites of the middle-to-lower continental crust of the Ronda area. In fact, recent studies on anatexis are beginning to stress the importance of water-present melting in the continental crust (Sawyer 2010; Weinberg and Hasalovà 2015). Another issue, beyond the scope of this contribution, is the origin of the fluids. Crustal rocks in general, and metapelites in particular, have only a very low proportion (<0.1 wt%) of free H₂O at temperatures slightly below their solidus, due to the strong reduction of porosity during prograde regional metamorphism (Yardley 2009). Hence, H₂O-rich fluid-present anatexis seem to indicate the influx of external fluids into the deep continental crust. Although previous investigations have provided some ways to introduce hydrous fluids of crustal origin into deep continental crust rocks (e.g. Brown 2010; Sawyer 2010; Weinberg and Hasalovà 2014), the mechanisms of fluid infiltration during high-grade metamorphisms are not sufficiently understood yet (Brown 2013).

Concluding remarks

Electron microprobe and NanoSIMS analyses of experimental glass in remelted and rehomogenized nanogranitoids within Grt suggest that former migmatites located at the bottom of the Jubrique crustal unit (Betic Cordillera, S Spain), and in contact with the underlying Ronda peridotite slab, underwent two melting events under contrasting fluid regimes. In both cases Grt constituted a peritectic mineral that trapped droplets of the primary anatectic melt. Water, however, was either provided by the fluid-absent incongruent melting of micas (perhaps Ms, surely Bt) during the first anatectic event (represented by leucogranitic MI at the cores of large Grt), or possibly introduced in the system as an external fluid during the second anatectic event (represented by granodioritic to tonalitic MI at the

rims of large Grt). Nevertheless, further detailed studies are necessary to confirm the systematic distribution of leucogranitic and granodioritic-to-tonalitic MI at the cores and rims of large Grt crystals, respectively. This contribution demonstrates the potential of detailed studies of MI in migmatites and granulites for the investigation of crustal anatexis and continental crust generation and differentiation (see Cesare et al. [2015](#)).

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Figure captions

Figure 1. Geological maps of the Betic-Rif orogen and the studied area in the western Betic Cordillera of S Spain (modified from Balanyá et al., 1997; including data from Martín-Algarra, 1987; Sanz de Galdeano and Andreu, 1995; Mazzoli and Martín-Algarra, 2011; Tubía et al., 2013). The location of the studied sample JU-8 is shown as a yellow star.

Figure 2. (a, b) Field appearance of the studied [mylonitic](#) gneiss (a, former migmatites; white arrow shows a cm-thick former leucosome; dark and light grey arrows show Kfs and Grt porphyroclasts, respectively; the hammer is 29 cm long) and a dm-thick leucocratic band parallel to the main foliation of the rock (b, former leucosome; grey and white arrows show Grt crystals and schlierens, respectively; the coin is 25 mm across; modified after Fig. 2c of Barich et al., 2014). (c, d) Plane-polarized light photomicrographs of small crystallized MI (c), mostly found towards the cores of large Grt crystals and in the vicinity of single Ky and Rt inclusions; and large crystallized MI (d), generally found towards the rims of large Grt crystals, and spatially associated with single Sil and Ilm inclusions. Although most of the inclusions in (c) and (d) correspond to crystallized MI, red arrows show those most clearly distinguishable. White arrows in (d) shows inclusions of Sil needles. Notice that, in the case of the large MI, individual minerals are clearly visible and some of them can be identified under the optical microscope. This is not the case of the small MI, whose polycrystalline nature is clearly visible under cross-polarized light (small inset in Fig. 2c, representing an enlargement of two of the MI shown in Fig. 2c), though minerals cannot be identified under the microscope. (e) Backscattered electron (BSE) scanning electron microscope (SEM) image of a large crystallized MI in Grt (modified after Fig. 6g of Barich et al., 2014). Notice the indentation of Gr within the MI walls (white arrow), indicating the accidental nature of this mineral in this MI.

Figure 3. NanoSIMS calibration curve determined for the analytical session during which the experimental glasses in remelted and rehomogenized MI were analyzed. This linear

calibration is based on the H₂O concentrations measured by Elastic Recoil Detection Analysis (ERDA) on the reference glasses B, LGB1 and DL. OH/Si stands for ¹⁶OH⁻/²⁸Si⁻ determined by NanoSIMS. Replicates on each standard are reported. The spread shows the reproducibility during the analytical session. See text for details.

Figure 4. BSE-SEM images of remelted and rehomogenized nanogranitoids in several microstructural locations, after quenching of the 850 °C (a-d), 825 °C (e-h) and 800 °C (i-l) experiments. 850 °C and 825 °C experiments show a low proportion of rehomogenized MI (d, h), and abundant disequilibrium microstructures such as frequent partially dissolved daughter crystals (a, c, f, g), reaction between accidental Als and melt to form St (b, e), irregular MI walls (a, b, c, g), presence of offshots (b, f) and recrystallized Grt at the boundary with the MI (a, g). 800 °C experiments show a larger number of rehomogenized MI (i-k), but also remelted MI (l).

Figure 5. Harker diagrams of analyzed glasses in remelted and rehomogenized nanogranitoids. The complete EMP glass dataset (≈80 analyses, see Table 2) includes analyses affected by some contamination from host Grt and/or trapped minerals (shown by somewhat higher FeO, MgO and TiO₂ concentrations, and ASI values) and extensive Na loss (manifested by values of ASI>1.5 after correction for Na loss). These values have not been considered when calculating mean concentrations (Table 2), and the corresponding analyses have not been included in Figs. 5 and 6. Dark and light grey areas represent the compositional domains corresponding to type I and type II MI, respectively. The bulk rock compositions of the studied [mylonitic](#) gneiss and the thick leucocratic band shown in Fig. 2b (former leucosome) are shown in blue and red symbols, respectively. Notice that the former leucosome contrasts in composition with respect to any of the analyzed MI. In particular, the leucosome is nominally anhydrous and show much higher FeO+MgO+TiO₂ concentrations (≈6 wt%).

Figure 6. Anorthite-Or-Ab (a) and Qtz-Or-Ab (b) pseudoternary normative diagrams (in wt%) for the analyzed glasses in remelted and rehomogenized nanogranitoids. Dark and light grey areas, and blue and red symbols, as in Fig. 5. Notice that although the analyzed leucosome have Qtz-Or-Ab proportions similar to type I MI, leucosome and MI are different in composition (Fig. 5).

Figure 7. Comparison between H₂O concentrations estimated by the difference method (100-electron microprobe totals) and measured by NanoSIMS on experimental glass from the same MI.