Sm-Nd isotope whole rock and garnet from the southwestern Grenvillian Oaxacan Complex, Mexico: a review of garnet closure temperature and structural implications.

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ABSTRACT

The Oaxacan Complex represents the largest outcrop of late Mesoproterozoic age, granulite-facies metamorphic rocks in Mexico. The southwestern Oaxacan Complex was studied using the Sm-Nd isotopic system in whole-rocks and garnets. The use of garnet for dating granulites is a good approach because garnet is usually a rock-forming mineral in this facies, it strongly prefers heavy over light rare earth elements, and its closure temperature is close to the range of the granulite facies. Since the studied garnets display resorbed borders, they are interpreted as pre- or syn- Grenvillian-age granulitic peak. Their closure temperatures, calculated using the Dodson equation and the Nd diffusion coefficients, are 720–770ºC (5–30ºC/my). The whole-rock Sm-Nd evolution lines intersect the depleted mantle model at ca. 1.4–1.7 Ga, although two paraderivate samples intersect at 2.1–2.2 Ga, suggesting a protolith from an older continental crust. In nearby outcrops, Sm-Nd isochron garnet-whole rock ages follow a certain NW-SE trend, displaying two hypothetical age groups: old ages (954–976 Ma) and young ages (907–920 Ma). The younger age garnet's group display Ti and LREE-MREE rim enrichments with respect to their cores that imply diffusional resetting processes. Folding and faulting structures suggest that these two age groups correspond to different structural levels during the early cooling history of the Oaxacan Complex.

Keywords: Grenvillian orogen, Oaxacan Complex, Sm-Nd, whole-rock, garnet, garnet closure temperature, cooling histories.

INTRODUCTION

Granulites are metamorphic rocks that form under high-temperature (HT, 700–900 ºC) and ultrahigh-temperature (UHT > 900 ºC) conditions at the lower crust and upper mantle depths (Harley 1989). The use of garnet (Grt) for dating granulite is a good approach because Grt is usually a major rock-forming mineral in this metamorphic facies, it strongly prefers the heavy over the light rare earth elements (REE), and it has a closure temperature (*Tc*) that is close to the range of granulite facies metamorphic temperature. Sm-Nd isotopic system is a potential chronometer to decipher the tectonometamorphic processes involved in the evolution of metamorphic orogens (Van Breemen and Hawkesworth 1980; Griffin and Brueckner 1980; Morrissey et al. 2011; Baxter et al. 2017) and provides information about the crustal genesis and crustal residence time of the protolith before metamorphism, the time when the material first separated from reservoirs and the mixing of crustal sources during orogenic events (e.g., Schaaf et al. 1994; Kovalenko 2008; Duchene et al. 2012). The study of the pressure-temperature-time (P-T-t) paths of metamorphic rocks (Spear 1993; Stowell et al. 2003; Gasco et al. 2013), integrating petrological, geochemical, structural, or geochronological data, is an effective tool to reconstruct geophysical and tectonic models of the crust (Rötzler et al. 2004; Du et al. 2014). Applying tectonic models to the evolution of metamorphosed crust (deformation, uplift, and subsequent erosion) requires an understanding of the metamorphic rocks' cooling history (e.g., Flowers et al. 2006; Wintsch et al. 1999).

The idea that different minerals close to the diffusive loss of certain radiogenic daughter elements (such as Ar, Sr, Pb, He) at different temperatures ('blocking temperatures') was suggested by Jäger (1967), an observation soon understood in the context of the T-dependent character of diffusion (Watson and Baxter 2007). The *Tc* concept of diffusive species was first formalized and analytically solved by Dodson

(1973), who found a *Tc* equation as a function of the cooling rate (*dT/dt*) and the Arrhenius diffusion parameters for each mineral and diffusant. Dodson (1973) assumed monotonic cooling following ~1/T to solve the equation. However, *Tc* depends on the cooling rate, grain size, strain rate, and fluid presence or absence (Villa 1998; Thöni 2003; Smit et al. 2013); and the system can be reset to different degrees by a non-monotonic cooling history (Dempster et al. 2008). The sub-microscopic complexities of many minerals raise doubts about the validity of the Dodson assumption that the macroscopic mineral grain radius and the lattice diffusivity determine the characteristic timescale for diffusive losses (Watson and Baxter 2007). Moreover, for the Sm-Nd system, the diffusion profiles of some REEs and their mutual relationships are not fully understood (Kelsey and Powell 2011). Grt constitutes a group of minerals in which Dodson's assumptions can be applied after appropriate rock and crystal selection. Data about the Sm-Nd *Tc* in Grt ranges from ca. 600 to 900ºC: 600 ± 30ºC (Mezger et al. 1992), 700–500ºC (Humphires and Cliff 1982), 650–700ºC (Carlson 2012), ~700ºC (Baxter and Scherer 2013), 690–780ºC (Maboko et al. 2001), > 700–750ºC (Hensen and Zhou 1995), > 850ºC (Jagoutz 1988) and 900ºC (Cohen et al. 1988).

The basement of eastern and southern Mexico is made up of late Mesoproterozoic (Stenian to Tonian)-age granulite-facies metamorphic rocks, which together constitute the core of an inferred microcontinent named Oaxaquia (Ortega-Gutiérrez et al. 1995). Late Mesoproterozoic paleogeographic reconstructions locate Oaxaquia interacting with the north-western margin of the Amazonian craton in a magmatic back/forearc active margin (Ortega-Gutiérrez et al. 2018). Grenvillian basement rocks in Mexico occur as isolated crustal units in the state of Tamaulipas (Novillo Gneiss; Ortega-Gutiérrez 1978a), the state of Hidalgo (Huiznopala Gneiss; Lawlor et al. 1999) and the state of Oaxaca (Oaxacan Complex; Solari et al. 2003, and Guichicovi Complex; Weber and Hecht 2003).

In the state of Chiapas, there are late Mesoproterozoic components that have been mostly reworked by both Ordovician and Permian magmatism and metamorphism (Cisneros de León et al. 2017; Weber et al. 2018) (Figure 1a).

Literature data of Sm-Nd and Lu-Hf from Oaxaquia displays the following ranges: Sm-Nd Grt-whole rock (WR) ages from 1403 ± 140 to 900 ± 2 Ma; Nd $T_{DM(WR)}$ (1 Ga) from 1.37 to 1.77 Ga, Nd $T_{DM(WR)}$ (1.2 Ga) from 0.80 to 2.02 Ga; $\varepsilon Nd_{(WR)}$ (1 Ga) from −15.7 to +6.2; and $\varepsilon Nd_{(WR)}$ (1.2 Ga) from −4 to +4.3 (Patchett and Ruiz 1987; Lawlor et al. 1999; Weber and Köhler 1999; Schulze 2011; Schulze et al. 2016); $\varepsilon Hf_{(Zrn)}$ (1 Ga) from +2.2 to +5.7; and Hf $T_{DM(Zrn)}$ (1.2 Ga) from 1.58 to 1.72 Ga (Weber et al. 2010; Weber and Schulze 2014; Weber et al. 2018; Solari et al. 2020). Sm-Nd and Lu-Hf T_{DMS} suggest oceanic juvenile sources for most igneous precursors and the derived sediments (Ortega-Gutiérrez et al. 2018). Moreover, older crustal components are assimilated in metaigneous rocks and older detrital sources for metasedimentary rocks up to 1.78 Ga (Weber and Schulze 2014; Solari et al. 2014). Lu-Hf and Sm-Nd data indicate that the orthogneisses of the Candelaria unit and the massive anorthosites (Cisneros de León et al. 2017) are isotopically similar to those of the typical late Mesoproterozoic Oaxaquia rocks (Weber et al. 2018).

The Oaxacan Complex (OC) represents the largest outcrop belt (ca. 6600 km^2) of late Mesoproterozoic age, granulite-facies metamorphic rocks in Mexico (Figure 1b). In the northern OC, polyphasic deformation and metamorphism in granulite facies were dated between 1004 and 978 Ma (Solari et al. 2003). Metamorphic peak conditions in the northern OC are inferred to be 836 ± 25ºC and 0.76 ± 0.16 GPa (Ramirez-Salazar 2015); ~813ºC and ~0.86 GPa in the southern OC (Schulze et al. 2016) and 825–875ºC and 0.80–1.0 GPa in the southwestern OC (study area) (Culí et al. 2019).

The main goals of this work are: 1) the study of the Sm-Nd isotopy from southwestern OC rocks to make inferences about the origin of the protoliths; 2) the use of the Grt-WR Sm-Nd isochron's ages spatial distribution to investigate the southwestern OC orogen geometry; and 3) the calculation of the *Tc* range for the OC Grt to compare the obtained results with the bibliographic values.

MATERIALS AND METHODS

GEOLOGICAL SETTING AND COLLECTED SAMPLES

The study area is located between Ayoquezco de Aldama and San Baltazar Loxicha (centre and southern Oaxaca State, México) (Figure 1c). The OC lithological units from the study site have been divided into two main groups. Group 1 (G1) is constituted by semipelitic granulite, quartz-feldspathic gneiss, amphibolite, calc-silicates, pegmatite, anatectic marble, orthogneiss, minor quartzite, mafic granulite, and scarce ultramafic granulite. Group 2 (G2) is constituted by intermediate and basic orthogneiss (mostly metagabbro and some rocks belonging to the charnockitic series) and migmatite, intruded by minor pegmatites. The general foliation trend is around N 330 – N 345. Outcrop-scale ductile deformation consists of a pervasive foliation not evident in all lithologies, a metamorphic lineation that is sometimes observed at a decametric scale and folds that range from tens of meters to a few centimeters. Certain lithotypes such as post-orogenic pegmatite and anatectic marble do not display any deformation in some outcrops. Geothermobarometric studies of the region that involve Grt (Culí et al. 2021) define an NNW-SSE or NW-SE trend and display various P-T features, so different structural levels of the orogen are inferred to be present at the same erosion level. Brittle deformation is represented by both normal and lateral faulting.

Eleven samples were collected. Samples belonging to G1 correspond to two quartz-felspathic paragneisses (LCOx10 and LCOx77), three basic orthogneisses (LCOx60, LCOx83b, and LCOx108), one para-amphibolite (LCOx73) and two ultramafic granulites, ortho (LCOx35) and para derivate (LCOx123a) (Culí et al. 2020). Samples belonging to G2 correspond to one intermediate (LCOx98) and two basic orthogneisses (LCOx15 and LCOx45) (Figure 2, Supplementary Table 1).

ANALYTICAL METHODS

Petrographic studies were carried out in thin sections using a Zeiss Axio Imager A2.m optical microscope at Laboratorio Nacional de Geoquímica y Mineralogía, UNAM.

Samples were crushed using a steel jaw crusher, processed further in a steel mortar, and then split into grain size fractions by sieving. Grt crystals were manually separated using a stereomicroscope to ensure the purity of the concentrate, which was 100% pure except for invisible inclusions.

WR trace element analyses were performed at Actlabs (Ontario, Canada). The used technique employs a lithium metaborate/tetraborate fusion. The resulting molten bead is digested in a weak nitric acid solution. Analyses were made by Inductively Coupled Plasma Mass Spectrometry (ICPMS). The precision of trace elements analyses expressed as coefficient of variation (CV) and limit of detection (LOD) are reported in Supplementary Table 7b.

Grt were analyzed with electron probe micro-analyzer wavelength-dispersive spectrometry (EPMA-WDS) at the Centre Científic i Tecnològic, Universitat de Barcelona (CCiTUB). Analyses were performed on a JEOL JXA-8230 electron microprobe with five wavelength-dispersive spectrometers (WDS) and a silicon-drift detector EDS. To achieve the best lateral resolution, spot analyses were carried out with an accelerating voltage of 15 kV, a beam current of 10 nA, standard counting times of 10 s, and a focused beam. Background positions were carefully adjusted. Analytical standards used were: hematite (Fe, LIF, Kα); rutile (Ti, PET, Kα); periclase (Mg, TAP; Kα); rhodonite (Mn, LIF, Kα); Al_2O_3 (Al, TAP, Kα); Cr_2O_3 (Cr, PET, Kα); diopside (Si, TAP, Kα); wollastonite (Ca, PET, Kα); orthoclase (K, PET, Kα); barite (Ba, PET, Kα) and albite (Na, TAP, Kα).

Grt trace elements concentrations were measured using a Photon Machines Analyte G2 193 nm ArF excimer laser equipped with a HelEx two-volume sample cell coupled to a Thermo iCAP Q ICPMS at the LA-ICPMS Laboratory of the Earth Observatory of Singapore, Nanyang Technological University, Singapore. Using a square spot size of 40 x 40 µm, a 30 s ablation was carried out at a 10 Hz pulse repetition rate with a fluence of 3.5 J/cm^2. The ablated material was transported in He with a total flow rate of 1 L min^{-1} and mixed with ca. 0.7 L min^{-1} Ar ~ 10 cm upstream from the torch. The plasma was sustained at 1550 W, and the system was tuned to maximize sensitivity while keeping ThO/Th < 1%. The Trace_Elements_IS data reduction scheme (Woodhead et al. 2007) of Iolite v 3.6 (Paton et al. 2011) was used to reduce the data, using standard-sample bracketing with USGS basaltic glass GSD-1G as the primary calibration standard plus internal standardization, with Si concentrations determined by EPMA. Reproducibility of secondary reference materials (BCR-2G and BHVO-2G) ranged from 2 to 7% relative standard deviation for elements with concentrations > 0.1 ppm, and the measured concentrations of these reference materials were within 10% of the GeoReM preferred values (Jochum et al. 2005). Isotopic determinations of Sm, Nd and Sr were carried out at Laboratorio Universitario de Geoquímica Isotópica (LUGIS) of the Instituto de Geofísica, Universidad Nacional Autónoma de México. The Grt crystal concentrates of each sample were cleaned with very dilute (1N) 3HCl + 1HNO_3, put in an ultrasonic bath for 5 minutes, and then cleaned and set again in an ultrasonic bath with deionized water (Milli-Q) for another 5 minutes. Between 80 and 150 mg of the dried sample (Grt or WR) were used for isotopic analyses. Pre-spiked WRs were dissolved in 7 ml hydrofluoric acid and a few drops of perchloric acid, unspiked Grts in HF + HNO_3 in Teflon® bombs at 90-100 ˚C. After 48 h, solutions were evaporated. The dry residues were dissolved in HCl. Clear Grt solutions were split into two aliquots. One aliquot was spiked with an ^{84}Sr-^{145}Nd-^{149}Sm tracer to determine concentrations by isotope dilution. The unspiked aliquot was used for isotopic compositions. Sr and REE separation was performed using quartz glass columns filled with DOWEX® 50Wx12 cation exchange resin. Sm and Nd were separated with different columns, consisting of Teflon® powder coated with hydrogen di-ethylhexyl-phosphate (HDEHP).

Nd isotope analyses were performed with a Thermo Scientific Triton Plus Thermal Ionization Mass Spectrometer (TIMS) equipped with nine adjustable Faraday collectors and five ion counters. In contrast, Sm isotopes were measured with a Finnigan MAT 262 TIMS that has a fixed central cup and seven adjustable Faradays. Samples were loaded as chlorides on double rhenium filaments and measured as metallic ions in static mode. Every run consisted of 30 isotope ratios for Sm and 70 for Nd. Total procedure blanks during the run of these samples were 0.31 ng for Nd. The Sm-Nd isochron calculations for Grt-WR pairs were done using Isoplot 4 (Ludwig 2008). The laboratory values used for standardization were NBS987 = 0.710252±0.000013 and standard Nd la Jolla = 0.511847±0.000003. The bibliographic values of these standards are NBS987 = 0.71034±0.00026 (NIST 2007) and Nd La Jolla = 0.511858±0.000007 (Lugmair and Carlson 1978).

TDM ages have been calculated using a single-stage model (Liew and Hofmann 1988) (Eq. 1), using the Depleted Mantle (DM) isotopic ratios $^{147}Sm/^{144}Nd = 0.2128$ and $^{143}Nd/^{144}Nd = 0.513089$.

$$T_{DM} = \frac{1}{\lambda} ln \left[1 + \frac{\left(\frac{^{143}Nd}{^{144}Nd}\right)_m - \left(\frac{^{143}Nd}{^{144}Nd}\right)_{DM}}{\left(\frac{^{147}Sm}{^{144}Nd}\right)_m - \left(\frac{^{147}Sm}{^{144}Nd}\right)_{DM}} \right] (1)$$

The Grt *Tc* has been calculated using the Dodson (1973) equation (Eq. 2). The procedure for assigning values to each variable is described below.

$$T_c = \frac{E/_R}{ln\left[\frac{ART_c\frac{D_0}{r^2}}{E\frac{dT}{dt}}\right]} (2)$$

The main parameters involved in this equation are D_0, the pre-exponential factor; *E*, the activation energy; *r*, the characteristic diffusion radius; *A*, the diffusion geometry; *dT/dt*, the cooling rate; and *R*, the ideal gas constant. *Tc* appears on both sides of the equation, so it must be solved by iteration starting from an approximate initial T value. The Arrhenius law is used to calculate D_0 and *E* from experimental diffusion data. If these values are known for a given mineral and diffusant, it is possible to obtain a value for the diffusion coefficient (*D*) at any temperature within experimental limits (Eq. 3).

$$D = D_0 \exp\left(-E/_{RT}\right) (3)$$

The Grt bibliographic values of D_0 and *E* include their composition as a variable to be considered (P and activation volume can also be included). The Grt end-member composition used for *Tc* calculations in this work is shown in Supplementary Table 4.

The experimental literature values of D_0 and E for neodymium (Nd) diffusion in Grts are scarce. Only three bibliographic references have been found, and they are shown in Supplementary Table 2. This table displays that log(D_0) values from Coughlan (1990) and Cherniak (1998) are similar, Carlson (2012) values are different, while the E from Coughlan (1990) compared with the two previous authors is very distinct. The experiments of Cherniak (1998) and Carlson (2012) were performed on Grts of very different compositions, the Grts from the former being an artificial substance; however, the equality of E values could indicate that the P of the experiments (ca. 1 atmosphere) controls the diffusion in the range of 700 to 1400ºC regardless of the composition. On the other hand, the experiment of Coughlan (1990) gives a very low E similar to a feldspar. The experiment was performed at 0.1 GPa, which could imply higher diffusion at higher P, but this does not make sense since, in general, at higher P, diffusion will be slower (higher E). In this work, the values of Coughlan (1990) and Carlson (2012) have been used for Grt *Tc* calculations.

The samarium diffusion values have also been used in this work for two main reasons: 1) to contrast them with the REE diffusion values in the range of Sm-Nd atomic numbers since there is too much scattering in the bibliography, and 2) to verify that the Sm diffusion was not too high, since this could generate very anomalous ages in the case of heating (the presence of daughter isotopes without parent isotopes). The E and *Do* values of Carlson (2012) (Supplementary Table 2) have been used for Grt *Tc* calculations because they are the most suitable for T conditions inferred for the southwestern OC (Culí et al. 2019; 2021), and the studied Grts' composition (Supplementary Table 4). On the other hand, the values of Van Orman et al. (2002) have also been used to compare both results.

Magnesium (Mg) diffusion parameters have also been used since some previous authors (e.g., Cohen et al. 1988) found that the Nd diffusion in Grts behaves as Mg. *E* and *Do* values for Mg diffusion have been taken from Chakraborty and Rubie (1996) (Supplementary Table 2) because the conditions in which these authors carried out the experiment are more realistic in the context of the OC, but the obtained *Tc* values of these authors, are in the range of ca. 800–1000ºC, which seems unrealistic. For this reason, we have taken the values of Perchuk et al. (2009) (Supplementary Table 2) since the Grt composition used in their experiment most closely resembles the Grt composition studied in this work.

A spherical diffusion geometry (*A*) has been assumed (Eq. 4) because all directions have equal diffusion due to the Grt cubic lattice.

$$\frac{\partial C_i}{\partial t} = D_i\left[\frac{\partial^2 C_i}{\partial r^2} + \frac{2\partial C_i}{r\,\partial r}\right] \quad (4)$$

Mineral grain sizes have been measured using a petrographic microscope or in hand samples using a ruler. Note that if resorption has occurred, it does not affect the *Tc* because the cooling occurred after the resorption. Therefore, the calculated *Tc* applies to the post-resorption diameter of each crystal.

A range of Grt *Tc* has been calculated to account for the range of Grt sizes in some samples. The range of cooling rates in Precambrian orogens is ca. 2ºC/Ma in cratonic regions to 100ºC/Ma in active subduction zones (e.g., Nakajima et al. 1990; Möller et al. 1995; Willigers et al. 2002). Two values of 5 and 30ºC/Ma have been used for the calculations (*dT/dt*) because the range of these values probably includes the real value, and it is consistent with Keppie and Ortega-Gutiérrez (2010).

RESULTS

Eleven samples with high-grade metamorphic assemblages (granulite and high amphibolite metamorphic facies) (Supplementary Table 1) were selected for Sm-Nd isotopy, ten of them for Sm-Nd Grt-WR geochronology (Supplementary Table 4) and eight for garnet *Tc* calculations (Supplementary Table 5).

PETROGRAPHY

The metamorphic peak mineral assemblage of the studied samples is represented by anhydrous minerals such as Opx, Cpx, Grt, Pl, Fsp (perthitic), and Qz (all mineral abbreviations are according to Whitney and Evans 2010). These minerals are often accompanied—in textural equilibrium—by high Fe-Ti Amp, highly pleochroic Bi, and Fe-Ti oxides. In some cases, especially in the orthogneiss lithotype, Amp and Bi display an intergranular texture because they fill spaces between the grains of the anhydrous minerals. This fact indicates high metamorphic conditions that were probably close to the metamorphic peak. The ultramafic granulite lithotype includes an orthopyroxenite mainly composed of Opx, Grt, and Amp and a clinopyroxenite mainly constituted by poikiloblastic Cpx (about 90% of the rock) with Scp, Fsp, Cal, and Hc inclusions. The mineral association of each sample is shown in Supplementary Table 1. Grt is a main phase in nine of the eleven studied samples. Its inclusions correspond to Qz, Opx, Cpx, Amp, Pl, Zrn, and Ap (Figure 3). All Grts used in Sm-Nd geochronology display suballotriomorphic or allotriomorphic morphologies (Figure 4). Mineral resorbed borders often exhibit irregular morphologies, such as embayments, which contrast with sharp original crystal growth faces (Baxter et al. 2017). All studied Grt display resorbed borders, so their nucleation was during prograde metamorphism (Florence and Spear 1993).

GARNET GEOCHEMISTRY

Microprobe analyses of the studied Grt are shown in Supplementary Table 3. Microprobe analyses of Grt lithotypes have been classified according to their end members (Skinner 1956; Hawthorne 2002). Rim-core-rim Grt profiles of main end members from samples LCOx10, LCOx35, LCOx49, LCOx77, LCOx98, and LCOx108 are completely or mostly flat (Supplementary Figure 1). This feature is typical of high-T terranes in which the major element diffusion in Grt is effective, so compositional variations are not observed in the Grt crystal (Carlson and Schwarze 1997). Note that only some significant Fe or Mg variations are observed at the Grt rims from LCOx35 and LCOx98 samples. This variation is related to the fact that these Grt rims are in contact with Bi or Px, i.e., mineral phases that integrate Fe and Mg in their structural formula. The main end member of all Grt is almandine (Alm), except for the LCOx49 Grt, whose main end member is pyrope (Prp), and the second is Prp or grossular (Grs), except for LCOx49 Grt, whose second end member is Alm (Figure 5).

The distribution of the trace elements, especially REEs, can be a useful tool in interpreting Grts geochronology Sm-Nd and Lu-Hf isotopic systems. Fractionation between a Grts crystal and the rock matrix can result in the incorporation of HREE+Y in the Grt cores and a smooth decrease of these elements towards their rims (Anczkiewicz et al. 2007; Kohn 2009). REEs patterns normalized to CI chondrite (Sun and McDonough 1989) from the studied Grt are shown in Supplementary Figure 2. Grts from LCOx10, LCOx15, LCOx77, and LCOx83b samples are enriched in HREE+Y in their cores with respect to their rims (the green line above the blue line, Supplementary Figures 2a, 2b, 2c, and 2e, respectively). The LCOx49 Grt core is depleted (Supplementary Figure 2d), and the LCOx98 Grt sample has equal HREE+Y core and rim concentrations (Supplementary Figure 2f). Referring to the MREE and LREE, the fractionation between the Grt and the matrix may result in either a slight increase towards the rims of the Grt crystals or no zoning (Baxter et al. 2017). Grts from the LCOx49 and LCOx98 samples are enriched in HREE-MREE at their rims with respect to their depleted cores (the blue line above the green line, Supplementary Figures 2d and 2f, respectively). Grt from the LCOx10, LCOx77, and LCOx83b samples are mostly depleted or equal in HREE-MREE at their rims with respect to their cores (the blue line below the green line, Supplementary Figures 2a, 2c, and 2e, respectively).

Sc, Ti, V, Cr, Ni, and Zn patterns normalized to CI chondrite (Sun and McDonough 1989) from the studied Grt are shown in Supplementary Figure 3. Grt from the LCOx10, LCOx77, and LCOx83b samples display Ti lower concentrations at their rims with respect to their cores (the green line above the blue line, Supplementary Figures 3a, 3b, and 3e, respectively). Grt from the LCOx15, LCOx49, and LCOx98 samples display similar concentrations of Ti at their rims and cores (Supplementary Figures 3b, 3d, and 3f, respectively). All Grt show greater or equal Cr concentrations at their rims with respect to their cores (the Grt from the LCOx77 sample cannot be evaluated because its Cr concentrations are below the detection limit, Supplementary Figure 3c). Grt from LCOx10, LCOx15, and LCOx98 samples display low concentrations of Ni on their rims relative to their cores (Supplementary Figures 3a, 3b, and 3f, respectively), and Grt from the LCOx49 and LCOx83b samples have slightly higher concentrations of Ni in their cores relative to their rims (Supplementary Figures 3d and 3e). These facts and their implications are evaluated in the discussion section. Trace element analyses of the studied Grts are shown in Supplementary Table 6.

If the composition of Grt and its WR is compared, it is possible to observe that the Grts are enriched in HREEs, whereas LREE and MREE are much or less depleted in Grt compared to the WR (Figure 6). This trend is expected due to the well-known preference of HREE elements for Grt (e.g., Lesnov 2012). LCOx10 WR (Qz-Fsp paragneiss) displays a large positive Eu anomaly, LCOx77 (Qz-Fsp paragneiss) displays a moderate positive Eu anomaly, and orthogneisses display an Eu flat patterns. All samples have Pl among their main mineral phases except LCOx77, which displays a moderate positive Eu anomaly and has Pl as a minor phase (Supplementary Table 1). The trace element analyses of the WR samples are shown in Supplementary Table 7.

Sm-Nd ISOTOPY

In the age vs. $^{143}Nd/^{144}Nd$ isotopic evolution diagram (Figure 7), most analyzed WR samples intersect the depleted mantle (DM) between 1.4 and 1.7 Ga. The LCOx77 sample (Qz-Fsp paragneiss) intersects the DM at 2.2 Ga, and the LCOx73 sample (amphibolite) intersects it at about 2.1 Ga. Figure 7 also shows data from the OC, Novillo Gneiss, and Huiznopala Gneiss from Ruiz et al. (1988), which intersect the DM between ca. 1.15 and 1.7 Ga, and data from the Guichicovi Complex from Weber and Köhler (1999), which intersects the DM between ca. 1.1 and 1.75 Ga. The chondritic uniform reservoir (CHUR) is also plotted as a reference, but the intersection of the sample evolution with the CHUR is not adequate for most Earth samples, as was already shown by Zindler and Hart (1986). This fact can also be observed in our data, which intersects the CHUR at impossible ages as young as 100 Ma. The WR data of Sm (ppm), Nd (ppm), $^{143}Nd/^{144}Nd$, and $^{147}Sm/^{144}Nd$ from the analyzed samples are shown in Supplementary Table 8.

The T_{DM} calculated range with a single-stage model is 1.43 – 2.12 Ga. The εNd range calculated at 0 Ga is between −12.5 and +7.2, and the εNd calculated at 1 Ga is between −3.9 and +5.3 (Supplementary Table 8).

GARNET WHOLE ROCK, Sm-Nd ISOCHRONES

The isotopic data of Sm-Nd Grt-WR isochrons are shown in Supplementary Table 4 and Supplementary Table 8. The obtained ages display one standard deviation between 21 and 30 Ma based on years-long averages from the Sm/Nd ratio of standards, giving a probably overestimated 2% error. A two standard deviations calculations plot of all ages suggests that all these can be equivalent. However, the data are plotted in the isochron diagram of Provost (1990), which maximizes the dispersion of the data in the two coordinates and explicitly shows the age of any drawn isochron on the right axis. Errors in both coordinates are plotted as two almost superposed bars. In this diagram, two hypothetical age groups seem to be present: 1) between 954-976 Ma and 2) between 907-920 Ma (Figure 8). Apart from that, the structure inferred by Culí et al. (2021) in the same region based on Grt composition and Grt geothermobarometry implies that the dated samples are located at different structural levels of the orogen. A statistical t-Student test of the mean equality from the two age groups indicates that the means are different at 99% probability. The implications of these facts are evaluated in the discussion section.

GARNET Tc RESULTS

The Grt *Tc* results were obtained using the Sm, Nd, and Mg diffusion coefficients from the literature. They span from ~630 ºC to ~970 ºC for the diameters and cooling rates described in this work. It has been explained above that the diffusion coefficient of Sm, Nd, and Mg can be used as they show very similar behavior in Grt. Nevertheless, the range of T is large and somewhat unconvincing for the higher temperatures. The obtained results are shown in Supplementary Table 5. These can be summarized as follows: Diffusion coefficients of Nd from Coughlan (1990) (5ºC/Ma→634ºC, 30ºC/Ma→700ºC) and Carlson (2012) (5ºC/Ma→807ºC, 30ºC/Ma→838ºC), Sm from Van Orman et al.

(2002) (5ºC/Ma→854ºC, 30ºC/Ma→971ºC) and Carlson (2012) (5ºC/Ma→809ºC, 30ºC/Ma→841ºC) and of Mg from Perchuk et al. (2009) (5ºC/Ma→690ºC, 30ºC/Ma→734ºC). These results are evaluated in the discussion section.

DISCUSSION

GARNET COMPOSITION

All studied Grts display suballotriomorphic or allotriomorphic textures (Figure 3 and Figure 4), so from a textural point of view, all studied Grts have been reabsorbed, so they are pre- or syn- Grenvillian-age granulitic metamorphic peak. This fact implies that after the metamorphic peak, they never again reached an equal or higher T that reabsorbed them since no thermal event is reported above the granulite peak temperature of 825–875ºC for the southwestern OC (Culí et al. 2019; 2021). The studied Grts do not display significant zoning in the major element profiles (Supplementary Figure 1); therefore, the metamorphic granulitic T peak was high enough to homogenize the Grt main composition.

The first Grt main end member is Alm, common in granulitic terrains (e.g., Harley and Kelly 2007; Marsh et al. 2012; Guevara et al. 2017). The second main end member is Prp or Grs. This variation in Mg (Prp) and Ca (Grs) content relates to the WR chemistry that hosted them. For example, Grt from ultramafic granulite (LCOx35) displays the highest Prp content, while amphibolite (LCOx73) Grt displays the highest Grs content (Figure 5). The fractionation between Grt and the rock matrix (Rayleigh) gives Grt characteristic elemental profiles. These profiles are visible in three Grt samples: LCOx10, LCOx15, and LCOx77 (Supplementary Figure 2). Therefore, Grts from different structural levels of the study site (Culí et al. 2021) cannot be differentiated by their HREE+Y enrichment in their cores with respect to their rims because these samples are located at both northern and southern study sites within the same P-T sectors. However, the fractionation between the Grt and the matrix can sometimes be observed in the LREE and MREE profiles, which slightly increase towards the rims of the Grt crystals (Baxter et al. 2017). Grt belonging to the southern part of the study site (LCOx15, LCOx49, and LCOx98 samples) are enriched in LREE-MREE in their rims with respect to their cores, but Grt belonging to the northern part of the study site (LCOx10, LCOx77, and LCOx83b) do not fulfill this condition. Hence, there are geochemical differences between Grts from the northern and southern parts of the study site. Although classical geothermobarometric studies of these Grt point to similar P-T conditions (except the LCOx98 sample), Grt from the northern part have older ages than Grt from the southern part (Figure 9).

The main processes that modify the Rayleigh REE fractionation profiles in Grt—and in some cases major element profiles—are the following: a) diffusion-limited REE uptake, which causes a steep chemical potential 'halo' to form around the early-growing crystal (the displaced core peaks in the Grt profiles) (Moore et al. 2013); b) resorption or recrystallisation, which generates HREE + Y enrichment at the Grt rims (Lanzirotti 1995; Pyle and Spear 1999); c) the breakdown of major or accessory rock-forming phases, which results in the liberation of MREE and LREE generating intermediate peaks (not centred at the core or rims) of these elements in the Grt crystals (Skora et al. 2006); d) changes in the kinetics of Grt growth, which generate HREE + Y enrichments at the Grt rims (Hickmott and Spear 1992); e) overprint zoning, in which pre-existing matrix heterogeneities or former accessory phases can be retained as overprint zoning in the chemistry Grt, generating submillimetric elemental Y and Cr patterns that define internal foliations within the Grt crystals (Martin 2009); f) the infiltration of trace element-rich fluid, which generates annular geometries that correspond to very marked ring-shaped zoning of certain elements such as Ca and Mn (Moore et al. 2013). The dated OC Grts have no foliations or rotation evidence (Figure 4) and display flat major element profiles

(Supplementary Figure 1): hence, the overprint zoning and infiltration of trace element-rich fluids can be ruled out. The Grt from the LCOx98 sample is the only which is enriched in Dy and Lu in its rims (Supplementary Figure 2). According to Gatewood et al. (2015), this feature denotes reabsorption or recrystallization processes, although processes related to changes in the kinetics of Grt growth cannot be completely discarded. Grt from this sample is more likely to exhibit geochemical features related to reabsorption/crystallization because the OC metamorphic peak is in granulite facies and all dated Grts display resorption textures. Apart from that, this is precisely the sample that displays different P-T features based on geothermobarometric studies. Discovering the specific process that generates these REE profiles in Grt goes beyond the scope of this work, but the fact that some differences exist regarding the LREE and MREE between the Grts from the northern and the southern part of the study area (Supplementary Table 3) is in accordance with other facts that are described below and allow us to differentiate them. If variations of the transition metals at the rims and cores of the studied Grt are examined (Supplementary Figure 3), Grt from northern and southern parts that define two hypothetical age groups can be differentiated by examining Ti (in ppm). Grt from the northern part shows Ti depletion on their rims with respect to their cores. Grts from the southern part display equal or slightly Ti amounts on their rims with respect to their cores (Supplementary Table 3). The mineral association observed in the studied samples contains accessory ilmenite, which includes Ti as a main component (Ilm, $FeTiO_3$, Supplementary Table 1). The rest of the analyzed transition metals (Cr, Ni) cannot be linked to any other phase as a main component in the studied samples. This fact indicates that it is consistent with using Ti as a petrogenetic indicator in the studied samples. The LCOx10 sample from the northern part (Figure 9) does not display Fe-Ti oxides in its mineral association, consistent with its Grt having the lowest Ti concentrations.

WHOLE ROCK AND Grt Sm-Nd

Most southwestern OC WR samples intersect the depleted mantle (DM) in the age vs. $^{143}Nd/^{144}Nd$ isotopic evolution diagram at ca. 1.4–1.7 Ga. The samples from the Novillo Gneiss and the Huiznopala Gneiss intersect between ca. 1.15–1.7 Ga (Ruiz et al. 1988), and the samples from the Guichicovi Complex intersect it between ca. 1.1–1.75 Ga (Weber and Köhler 1999) (Figure 7). The LCOx77 sample (Qz-Fsp paragneiss) intersects the DM at 2.2 Ga, and the LCOx73 sample (amphibolite) intersects it over 2.1 Ga. These ages suggest a protolith with an old continental crust influence compared with the rest of the samples, probably with some contribution from Amazonia, according to Weber et al. (2008). The amphibolite LCOx73 sample crops out as intercalations of lenticular morphology in the paragneiss unit, and it intercepts the DM at the same age as the LCOx77 sample. Moreover, LCOx73 sample Ni (ppm) and Cr (ppm) concentrations point to a para-amphibolite, according to Leake (1964). To infer an orthoderivate origin for this amphibolite would imply that it corresponds to a rock extracted from the mantle (a basic melt) and then emplaced in a very old crust. These facts have not been reported in the Oaxacan domain by any author. On the other hand, Grt from the LCOx73 and LCOx77 samples are the only ones that have higher Sps content than Prp (Supplementary Table 6). The above facts point to a sedimentary origin for the amphibolite LCOx73 sample.

T_{DM} has been calculated with a single-stage model. The calculated T_{DM} range is 1.43 – 2.12 Ga (Supplementary Table 8). The results obtained are in accordance with the previous work on Oaxaquia done by Patchett and Ruiz (1987) and Weber and Köhler (1999). Southwestern OC εNd, calculated at 1 Ga, displays a range of −3.9 to +5.28, which is consistent with the nature of the protoliths of the studied samples (Supplementary Table 1) as well as with the OC and Oaxaquia realm (Patchett and Ruiz

1987; Weber and Köhler 1999; Schulze 2011; Schulze et al. 2016). The value of +5.28 corresponds to a basic orthogneiss (LCOx83b sample). This sample displays very positive εNd values, whether calculated at 0 or 1 Ga. Thus, the mantle nature of this rock is undeniable, and it seems that cortical processes are not as evident in it as in the other samples (Supplementary Table 8).

GARNET CLOSURE TEMPERATURE

Literature sources use different methodologies to calculate Grt *Tc*: a) Cohen et al. (1988) infer a Grt *Tc* > 850ºC and establish the Nd diffusion in Grt from the study of relict textures. An upper limit for the Nd diffusion in Grts of Prp composition is given, and the authors mention that it is not much greater than the diffusion of Mg in Grts of Prp-Alm composition; b) Jagoutz (1988) infers a *Tc* > 850ºC. He establishes the *Tc* in Grts for Nd diffusion, comparing the diffusion behavior of this element with the Grt-Cpx geothermometer; c) Maboko (2001) infers a *Tc* of 690–780ºC. He calculated the Grt *Tc* using the Sm-Nd ages obtained, the published maximum metamorphic T, and cooling rates; d) Baxter and Scherer (2013) infer a Grt *Tc* ~700ºC. They establish this *Tc* by modeling the T range necessary to reset the age of Grts between 5 and 95% for a given grain size, T, and the t. We use the Dodson (1973) equation to establish *Tc* in OC Grts, although some published works challenge applying Dodson's equation to natural systems (e.g., Villa 1998; Ganguly et al. 1998; Watson and Cherniak 2003). The main limitations are diffusion near the *Tc* and hydrothermal alteration, including recrystallization. Grts are not prone to easy alteration in medium-low P-T conditions. Neither secondary phases nor recrystallization is observed in the studied samples, so Dodson (1973) assumptions can be applied cautiously. In this work, we use the Grt diffusion coefficients of Nd from Coughlan (1990) and Carlson (2012), Sm from Van Orman et al. (2002), and Carlson (2012), and Mg from Perchuk et al. (2009) (Supplementary Table 7). This choice was made because the Grt compositions from these different experiments cover the OC Grt composition, and in some cases, the experimental conditions also cover the range of P-T inferred for the southwestern OC: P = 0.80–1.0 GPa, T = 825–875ºC (Culí et al. 2019; 2021). The average Grt *Tc* obtained for Nd diffusion with *dT/dt* = 5 is 720ºC and with *dT/dt* = 30 is 769ºC, using diffusion coefficients from Coughlan (1990) and Carlson (2012); the Grt *Tc* average obtained for Sm diffusion with *dT/dt* = 5 is 831ºC and with *dT/dt* = 30 is 879ºC, using diffusion coefficients from Van Orman et al. (2002) and Carlson (2012) (Supplementary Table 7). In this work, *Tc* data obtained from the Sm diffusion is higher than the data obtained from Nd and Mg, which are similar (Supplementary Table 8). The T peak inferred for the southwestern OC is 825–875ºC (Culí et al. 2019): this implies that the *Tc* obtained from Nd diffusion, 720–769ºC (*dT/dt* = 5–30º/my), is the most realistic because the *Tc* obtained from Sm diffusion is in the range of the metamorphic peak temperature, 831–879ºC (*dT/dt* = 5–30º/my).

STRUCTURAL IMPLICATIONS

The Sm-Nd Grt-WR isochron ages obtained follow a certain NW-SE trend according to Culí et al. (2021) and display two hypothetical age groups: old ages (954–976 Ma) from the northern part (LCOx77, LCOx83b, and LCOx10) and young ages (907–920 Ma) from the southern part (LCOx15, LCOx49, and LCOx98). The enhanced isochron diagram of Provost (1990) (Figure 8) shows that the two age groups, although equivalent at two standard deviations of the analytical errors, have isochrons grouped in two clusters except for only one sample (LCOx77) that shows a middle age.

Note that all mentioned samples are close to the sectors where folded structures and NW-SE lineations have been traced (Figure 9). As mentioned above, geochemical features from southern Grt that belong to the hypothetical young isochrons age group suggest diffusional resetting, so these rocks reached the Grt *Tc* (~720–770ºC) later than the hypothetical northern old group, i.e., they cooled more slowly.

This fact implies two possible scenarios: 1) the young age Grt were located in deeper parts of the Zapotecan orogen, although at some time before the Zapotecan orogeny, the Grts of both groups were at the same cortical level, which is why they present the same P-T conditions (Culí et al. 2021), or 2) a post-Zapotecan thermal event (907-920 Ma) reset some Grts. This event is explained by the reactivation of major tectonic boundaries during the Tonian post-orogenic gravitational collapse and was recently inferred in southwestern Mexico by Weber et al. (2020) in southern Chiapas.

In the first scenario, Grts belonging to the young age group were exhumed from deeper parts of the orogen during the Tonian (at ~920 Ma) due to a probably folded structure in a ductile regime or faulting associated with the traced lineations at the study site. Grt from the young age group display similar Ti concentrations on their rims and at their cores (while those of the old group are depleted in Ti on their rims with respect to their cores). In general, the young Grt display equal or high LREE and MREE concentrations in their rims with respect to their cores (while the old group Grt displays the opposite). Grt from the young age group cooled more slowly near the T range of the *Tc* than those of the old group, so they reacted at their rims, resorbing Ti and LREE-MREE. There is a difference of ca. 60 Ma between the time the young and old Grt reached their *Tc*. This thermal configuration could be related to the Zapotecan metamorphic peak and a folded/faulted structure, the first in a ductile regime and the second in a ductile-fragile. It should be noted that some works in the literature reported rocks that have been at 800–650 ºC for 50 Ma (Hauzenberger et al. 2015).

Regarding the second scenario related to the Oaxaquia realm, a post-Zapotecan thermal event (907-920 Ma) was recently inferred in southwestern Mexico by Cisneros de León et al. (2017) in southern Chiapas. The reactivation of major tectonic boundaries explains this event during Tonian gravitational collapse. A Tonian (~920 Ma) metamorphic overprint is suggested in Oaxaquia by recrystallized zircon crystals in a banded gneiss from Chiapas (Weber et al. 2018). Nevertheless, note that rocks from Chipas studied by, e.g., Cisneros de Leon et al. (2017) and Weber et al. (2018) are not rocks under granulite facies. They never underwent Grenvillian (Zapotecan) metamorphism. If they belong to Oaxaquia, somehow, they never experienced such high-grade conditions.

Regarding the second scenario outside the Oaxaquia realm, in southwestern Baltica, the Valhalla accretionary orogen is defined by two episodes of siliciclastic sedimentation and magmatic activity: 1) 1025–980 Ma and 2) 920–870 Ma, punctuated by tectonothermal events associated with subduction and crustal thickening at 980–920 Ma and 840–730 Ma (Cawood et al. 2010). Although some events were dated at 920 Ma in southwestern Baltica, these correspond to an exterior accretionary orogen inferred to be regional. However, both 920–907 Ma and 976–954 Ma Sm-Nd Grt-WR isochron ages are present at the OC study site. In the Colombian outcrops of the Amazon craton, Sm-Nd Grt-WR isochron ages in the Garzón massif are 935 ± 5 and 925 ± 7 Ma (Cardona et al. 2010), and in the Santander Massif, they are 971 ± 5 Ma (Ordónez-Carmona et al. 2006). In this case, the age range coincides with data from southwestern OC, but different events are not reported. It should be noted that the minimum apparent distance between young and old Grt in southwestern OC is ca. 2 km (Figure 9). Thus, this is a small-scale, not a regional-scale feature. Therefore, the possibilities implied by the second scenario are ruled out.

Based on the above, it is inferred that in southwestern OC, there are outcrops that belong to different structural levels of the orogen. This fact is deduced from the existence of Grt-bearing rocks that have been T near their Grt *Tc* for some tens of Ma, which are in contact with Grt-bearing rocks that had cooled more quickly by folding and faulting related to the structural processes that occurred in the lower-medium crust. This fact fully agrees with the orogen structure that Solari et al. (2013) observed for the northern OC. However, with the available data, a post-Zapotecan thermal event (920-910 Ma) cannot be completely ruled out. This event could be related to the diachronic rise of isotherms after a continent-continent collision and variable diffusional resetting. On the other hand, the idea that both processes described above (different structural levels and diffusional resetting) may have occurred simultaneously cannot be discarded either.

Finally, a summary of the most important information that can be synthesized from this study and previous works (Culí et al. 2019, 2021) is shown in Figure 10. The proto Oaxaquia oceanic arc is constituted between 1500–1270 Ma. The initial Nd isotope ratio intersects the DM between 1400–1700 Ma, meaning the crust-forming magma is initially separated at these ages, although two paragneiss samples intersect the DM at 2100–2200 Ma indicating a contribution from an old continental crust (Figure 10a). The T_{DM} range obtained is 1280–1650 Ma, probably related to oceanic juvenile sources (Figure 10a and Figure 10b). At ca. 1200 Ma, proto-Oaxaquia is located within a peri Amazonia back-arc basin (Figure 10c). Between 1100 and 1080 Ma, proto-Oaxaquia is attached to the Amazonian continental margin. At these times, the Olmeca event generates partial melting and migmatization in the older parts of proto Oaxaquia (Figure 10d). At ca. 990 Ma, a continent-continent collision occurred between northern Amazonia and southern Baltica craton, corresponding to the Grenvillian (Zapotecan) orogeny (Figure 10e). In the southwestern Oaxacan Complex, peak metamorphic conditions are 825-875ºC and 0.8-1.0 GPa (Culí et al. 2019; 2021). After ~980 Ma, the orogen is exhumed and cooled down. Grt reached their *Tc* between 770ºC and 720ºC (dT/dt=30ºC/Ma and 5ºC/Ma, respectively) (Figure 10f).

CONCLUSIONS

The southwestern OC metamorphic peak mineral assemblages are represented by anhydrous minerals such as Opx, Cpx, Grt, Pl, Fsp (perthitic), and Qz. Southwestern OC samples intersect the depleted mantle (DM) evolution line at ca. 1.4–1.7 Ga. One Qz-Fsp paragneiss sample intersects the DM at 2.2 Ga, and one para-amphibolite sample intersects it at 2.1 Ga. These ages suggest a protolith with an old continental crust influence, probably with some contribution from Amazonia. The studied Grts are allotriomorphic to suballotriomorphic, displaying resorbed borders, so they are pre- or syn- Grenvillian-age granulitic metamorphic peak. The Grt main end member is almandine (Fe), followed by pyrope (Mg) or grossular (Ca). This variation in Mg and Ca content is generated by the different lithotypes that hosted the Grts. The Grts studied do not display significant major element zoning because the granulitic metamorphic peak temperature was high enough to homogenize their composition. The *Tc* of the southwestern OC Grts is 720–770ºC (*dT/dt* = 5–30); it was calculated using the Dodson equation and the Nd diffusion coefficients from Coughlan (1990) and Carlson (2012). In the southwestern OC, Sm-Nd Grt-WR isochron ages follow a certain NW-SE trend displaying two hypothetical age groups: old ages (954–976 Ma) and young ages (907–920 Ma), the latter being located in the middle of this trend and close to the sectors where folded structures are traced. There are some NW-SE lineations located between the two age groups. Grts from the young Sm-Nd isochron group display rim enrichments of Ti and LREE-MREE with respect to their cores, implying diffusional resetting processes. From the available data, it is inferred that in southwestern OC, two groups of Grts cooled at different rates in nearby outcrops. The occurrence of folding and faulting suggests that these two groups correspond to different structural levels that have been detected by their Sm-Nd Grts-WR ages, although the existence of a post-Zapotecan thermal event (~910 Ma) cannot be completely ruled out with the available data.

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DECLARATION OF INTEREST STATEMENT

Conflict of interest: the authors declare no conflict of interest.

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