

# U-Pb geochronology of rutile: deciphering the cooling history of the Oaxacan Complex granulites, southern Mexico

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## ABSTRACT

Rutile (TiO<sub>2</sub>) is a heavy mineral, commonly found as accessory in many lithologies, such as basic igneous rocks, high-grade metamorphic units, as well as a detritus in sedimentary clastic rocks. Its chemical composition is sensitive to the crystallization environment, allowing a characterization of either metabasic or metasedimentary protoliths in metamorphic rocks. Thanks to the capability to accept U in its crystalline network, at least in metasedimentary, high-grade protoliths, rutile can be dated by U-Pb geochronology. Furthermore, its closure temperature of ca. 600 °C for the U-Pb system makes rutile a suitable chronometer, complementary to zircon, to unravel provenance and exhumation paths in both sedimentary siliciclastic cover and basement units. Besides, the Zr-in thermometer allows for a very precise calculation of the rutile crystallization temperature.

In the example case presented here, focused on granulite facies units of the Grenvillian Oaxacan Complex (OC), rutile crystallisation took place in the range 808–873 °C. Data for different localities indicate that cooling and exhumation after the Zapotecan granulite facies event (ca. 990 Ma) was heterogeneous among the different tectonic slices that constitute the OC. Cooling occurred in the central sector (Nochixtlán-Oaxaca) right after the granulite peak, with fast cooling rates of ca. 40 °C/Ma. To the north and south, the cooling to ca. 600 °C was much slower, with calculated cooling rates of ca. 3 °C/Ma for the northern OC outcrops in Coatepec (Puebla) to ca. 6 °C/Ma south of Ejutla (Oaxaca). This can be related to a combination of factors, such as an early collapse of some sectors of the orogen, a change of conditions in the subducting plate, or more in general, to a sudden change in the geodynamic conditions during the Zapotecan orogeny and Amazonia-Baltica amalgamation.

Key words: rutile; granulite metamorphism; U-Pb geochronology; chemical composition; Oaxacan Complex; Mexico.

## RESUMEN

El rutilo (TiO<sub>2</sub>) es un mineral pesado, accesorio común en muchas litologías tanto ígneas básicas como metamórficas de alto grado, o bien

presente como detrito en rocas sedimentarias siliciclásticas. Tiene unas importantes características petrogenéticas, como son la composición química sensible al ambiente de formación y una marcada y diferente afinidad en protolitos metabásicos y metasedimentarios. Gracias a su capacidad para aceptar U en su red cristalina, al menos en los protolitos metasedimentarios de alto grado metamórfico, el rutilo puede datarse por medio de geocronología por U-Pb. Además, siendo la temperatura de cierre para el sistema U-Pb en rutilo de aproximadamente 600 °C, el rutilo se convierte en un mineral muy útil para caracterizar el enfriamiento en rocas de alto grado metamórfico, complementario al zircón, para entonces poder descifrar procedencia y eventos de exhumación tanto en rocas sedimentarias siliciclásticas de cobertura, como en unidades del basamento. El termómetro de Zr en rutilo permite también un cálculo muy preciso de la temperatura de cristalización del rutilo.

En el ejemplo presentado en este trabajo, que se enfoca al estudio de algunas unidades granulíticas colectadas en el Complejo Oaxaqueño del sur de México, la cristalización del rutilo ocurrió en el rango 808–873 °C. Las rocas metamórficas estudiadas, colectadas en diferentes localidades, permiten ver que el enfriamiento y exhumación del Complejo Oaxaqueño fue de hecho heterogéneo después del pico metamórfico Zapoteco (ca. 990 Ma) en las diferentes unidades tectónicas que lo componen. En la parte centro-norte (Nochixtlán-Oaxaca) el enfriamiento fue muy rápido, con una tasa de ca. 40 °C/Ma, mientras las porciones norte y sur enfriaron a una tasa mucho menor, de ca. 3 °C/Ma en el norte (al este de Coatepec, Pue.) y ca. 6 °C/Ma en el sur (entre Ejutla y Mihauatlán, Oax.). Esto puede deberse a una combinación de factores, como un colapso temprano de porciones del orógeno, un cambio de condiciones en la placa en subducción o, más en general, a un cambio repentino de las condiciones geodinámicas durante las primeras fases de la amalgamación de Rodinia.

Key words: rutilo; metamorfismo granulítico; geocronología U-Pb; composición química; Complejo Oaxaqueño; México.

## INTRODUCTION

The characterization of heavy minerals in siliciclastic rocks is a fundamental tool in modern geosciences, because it can unravel age, distributions, isotopic and chemical composition, facilitating the

study of high-grade terrains, their tectonic evolution and exhumation history, or even yielding important constraints to provenance analysis (e.g., Morton and Hallsworth, 1999; Keppie et al., 2004; Schulze et al., 2004; Garzanti, 2016). The continuous improvement, and availability, of microanalytical techniques made the quantification of the composition of heavy minerals a standard procedure in many laboratories, taking advantage of reduced time and analytical costs. While zircon is considered the king of the heavy minerals used in provenance studies, thanks to its abundance as well as its chemical and mechanical strength (Park et al., 2010; Ortega-Flores et al., 2013; Spencer et al., 2014), in recent years other minerals, also quite abundant in detrital rocks, have been increasingly used as chemical and isotopic tracers. In particular, apatite and rutile have received a lot of interest in the geoscientific community (e.g., Luvizotto and Zack, 2008; Triebold et al., 2012; Abdullin et al., 2015; O'Sullivan et al., 2018; Rösel et al., 2019), because both serve as excellent petrologic witnesses, as well as geochronometers with closure temperatures lower than that of zircon in the U-Pb isotopic system (ca. 350–550 °C for apatite according to Cochrane et al., 2014, and ca. 550–600 °C for rutile, according to Cherniak, 2000 and Kooijman et al., 2012; compared with zircon, in excess of 900 °C, Cherniak and Watson, 2000). In high-grade metamorphic rocks, rutile provides excellent information on the post-peak exhumation history when U-Pb cooling ages are combined with thermometric calculations, based on the equilibrium between Ti and Zr (Watson et al., 2006; Tomkins et al., 2007).

In this paper we present new microanalytical data for rutile, obtained on separates from samples belonging to the Grenvillian, granulite-facies Oaxacan Complex in southern Mexico (Keppie et al., 2003; Solari et al., 2003, 2004b, 2014, 2020; Weber and Schulze, 2014), as an example of how the information extracted from this mineral can improve the understanding of high-grade rocks, their behavior during metamorphism, and exhumation history.

**GEOLOGIC SETTING**

The Oaxacan Complex (OC hereafter; Figure 1) constitutes the largest basement exposure of granulite facies, Grenvillian-age rocks in Mexico. Together with other minor known outcrops of similar rocks, it constitutes the framework of the Oaxaquia microcontinent, a Proterozoic piece that makes up the backbone of Mexico, as we know it today (Ortega-Gutiérrez et al., 1995; 2018). In more detail, the ca. 1.3–0.95 Ga OC is constituted by an assemblage of (mostly) metaplutonic and, in minor amount, metasedimentary rocks, all metamorphosed under granulite-facies conditions. High-grade metamorphism (ca. 750–800 °C and 7.5–8.5 kbar, Mora and Valley, 1985; Solari et al., 2004b; also see a review in Ortega-Gutiérrez et al., 2018) occurred during the Zapotecan Orogeny (Solari et al., 2003), at ca. 990 Ma. An igneous sequence of ca. 1.2 Ga (locally, up to 1.4 Ga), is either interpreted as indicative of an intra-oceanic arc or a rift (Ortega-Gutiérrez et al.,

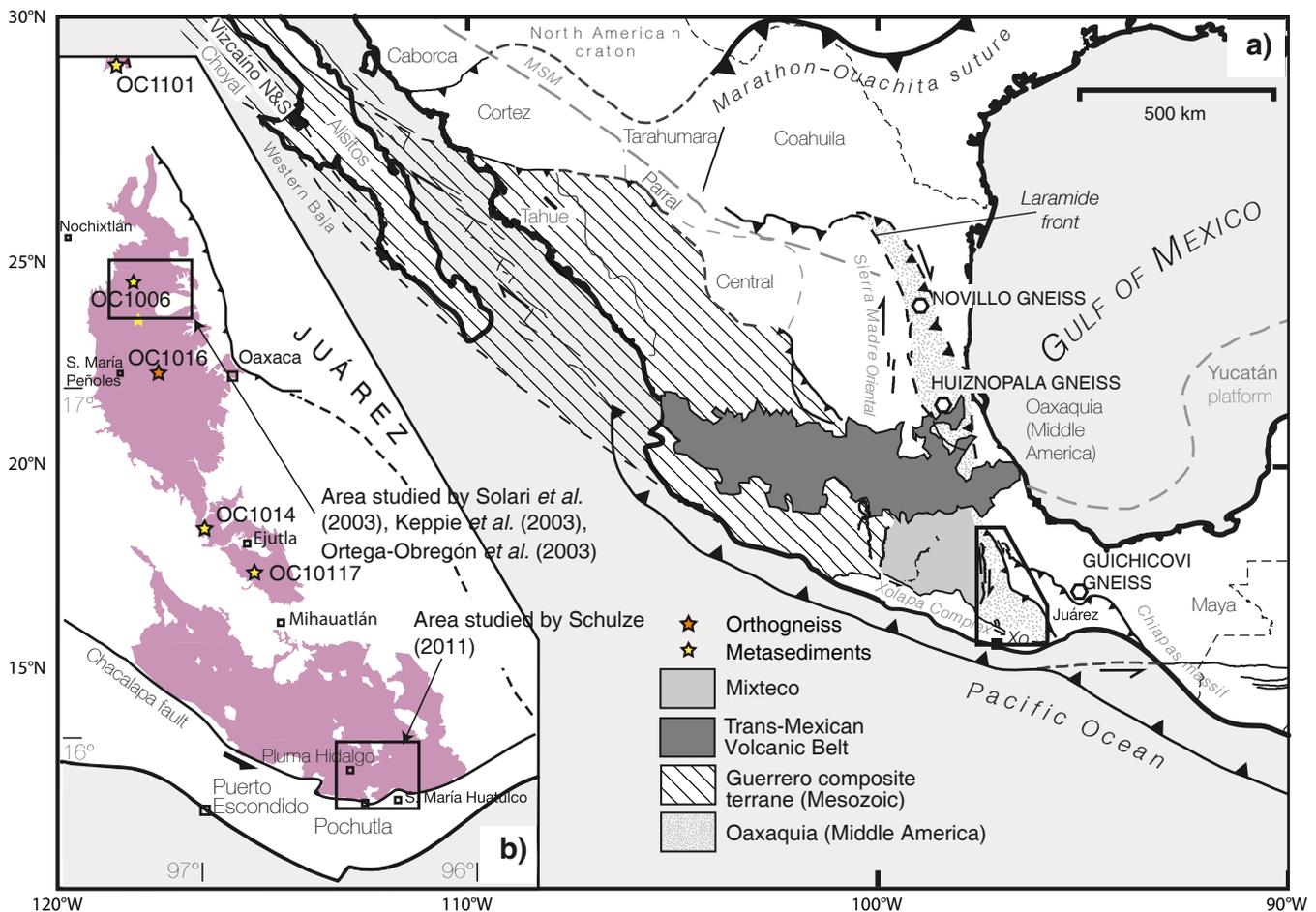


Figure 1. a) General overview of the geology of Mexico, including main subdivision in tectonic domains. b) Schematic map of the Oaxacan Complex, southern Mexico, with sample localities. Modified after Solari et al. (2014).

1995; Keppie *et al.*, 2001 and 2003; Keppie and Dostal, 2007; Solari *et al.*, 2014; Weber and Schulze, 2014; Ortega-Gutiérrez *et al.*, 2018). A younger igneous pulse, dated at around 1.02 Ga, makes up an AMCG (anorthosite-mangerite-charnockite-granite) suite (Keppie *et al.*, 2003). After the Zapotecan metamorphic peak, several post-tectonic, granitic to carbonated pegmatites intruded high-grade rocks of the OC. Their ages span from *ca.* 980 to *ca.* 965 Ma (Solari *et al.*, 2003; Schepetilnikova *et al.*, 2015), being related to magmatism generated by decompressional melting during or right after the Zapotecan event. The tectonic limits of the OC are: to the east, the Sierra de Juárez Mylonitic Complex as a tectonic limit with the Cuicateco terrane (Alaniz-Álvarez *et al.*, 1994, 1996); to the west, the Caltepec fault zone with the Acatlán Complex (Elías-Herrera and Ortega-Gutiérrez, 2002; Elías-Herrera *et al.*, 2007); to the south, the Chacalapa fault system against the Mesozoic-Tertiary Xolapa Complex (Tolson, 2005); to the north it is buried underneath Cenozoic continental deposits (Dávalos-Álvarez *et al.*, 2007) and Quaternary continental deposits associated to the development of the Trans-Mexican Volcanic Belt.

Whereas the Mesoproterozoic to Neoproterozoic evolution of the OC has been tackled in several papers, the post-Zapotecan geologic history is known only locally. For instance, Solari *et al.* (2004a) studied some shear zones in the northern OC, describing their tectonic evolution and geologic significance during the Phanerozoic (mostly associating them with the geologic evolution from the Late Triassic to the Early Cretaceous, as a result of plate re-accommodation during the break up of Pangea and the opening of the Gulf of Mexico). Moreover, Keppie *et al.* (2004) reported some Tonian cooling ages (mostly by Ar-Ar), associating them to the switching between steep and flat-slab subduction and subsequent thermal relaxation of the lithosphere, and slow cooling regime during Cryogenian time that lead to the Oaxaquia breakup during the Ediacaran time (*e.g.*, González-Guzmán *et al.*, 2016). The construction of cooling curves would enormously benefit from more data obtained by different methods, and in minerals with different closure temperatures, collected at different tectonic levels, to correctly define the timing of exhumation and, eventually, the differences in the tectonic regime from one to another locality. The use of rutile thus helps to close one of those gaps, generating data that fit to characterize the post-Zapotecan exhumation of the Oaxacan Complex.

## SAMPLE DESCRIPTION

After screening by petrography in thin section, as well as in heavy mineral concentrates, five samples were selected (geographic locations are illustrated in Figure 1).

The overall strike of the main OC structures bears, mostly, ENE-WSW, gently plunging toward NNW (Solari *et al.*, 2003 and 2004a). At least for the northern OC, a stack of three tectonic sleeves was recognized and described by Solari *et al.* (2003). New field observations, not yet fully documented, suggest the continuation of piling up of tectonic sleeves along the whole OC. As an example for the existence of such tectonic sleeves that represent different depths in the crust, Schulze (2011) calculated for the southernmost OC sector of Pluma Hidalgo, higher metamorphic pressures of *ca.* 10 kb, possibly representative of a deeper tectonic slice when compared to the northern sectors studied by Solari *et al.* (2003). The samples selected for this work, which mostly scatter along the NW-SE length of the whole OC, are thus representative of different depths of those geologic units, now brought to the eroded topographic surface, but originally in an unknown, relative position.

Sample OC1101 is from a garnet-bearing granulite gneiss cropping out along the Federal Highway 190, along an EW Tertiary normal fault (Santa Lucia fault), in the uppermost tectonic slice described by Solari

*et al.* (2003). It is made up of large (up to 5 mm) garnet, almost completely altered pyroxene, perthitic feldspar, quartz and plagioclase. In practice, it resembles an original, quartzo-feldspathic sediment, such as a sandstone or a lithoarenite. Rutile, zircon and apatite are accessory minerals. Rutile is generally tabular to needle shaped, dark-brown colored, ranging in size up to 500  $\mu\text{m}$  in length and up to 80  $\mu\text{m}$  in width; some crystals show twinning (*e.g.*, Figure 2a).

Sample OC1006 is a banded and foliated granulitic gneiss, found south of Asunción Nochixtlán, constituting the host rock for charnockitic (*ca.* 1135 $\pm$ 12 Ma, Solari *et al.*, 2020) and syenitic (*ca.* 1221 $\pm$ 8.2 Ma, Solari *et al.*, 2020) intrusions. It bears quartz, perthitic feldspar, plagioclase (often sericitized), garnet, and sillimanite. The abundant aluminum-rich minerals are suggestive of a metasedimentary origin. Rutile is found either as euhedral crystals, or as inclusion needles in quartz. Their shapes are generally more elongated than in the previous sample, with an up to 1:13 elongation ratio (Figure 2b and 2c), ranging up to 1000  $\mu\text{m}$  in length and up to 80  $\mu\text{m}$  in width. Rutile is light colored, ranging from pale brown to honey brown.

Sample OC1014 is from a granulitic gneiss that occurs NW of Ejutla, whose paragenesis is indicative of a sedimentary protolith. It is made up of quartz, perthitic feldspar, plagioclase, abundant garnet, up to 7 mm in size, together with abundant graphite and secondary biotite. Rutile is an abundant accessory mineral, normally light to moderately brown colored, and up to 800  $\mu\text{m}$  in length and up to 80  $\mu\text{m}$  in width.

Sample OC1016 is from a granulitic orthogneiss cropping out East of Santa María Peñoles. It is faintly banded, and made up of quartz, micro-perthitic feldspar, and plagioclase. Some mafic minerals are completely replaced by an assemblage of oxide (magnetite and ilmenite), biotite and tremolite, tentatively indicating the original presence of pyroxene. Rutile is an accessory mineral together with zircon and minor apatite. It ranges from euhedral, short prismatic to stubby shapes, up to 800  $\mu\text{m}$  in length, to subhedral shapes, in some cases less than 200  $\mu\text{m}$  in length (Figure 2d). The maximum width in the observed crystals is 80  $\mu\text{m}$ . It is also clearly darker than in the previous samples, ranging from dark brown to almost opaque, probably as a consequence of a higher iron content.

Sample OC1017 is a granulitic gneiss from an outcrop along the Federal Road 190, halfway from Ejutla to Mihauatlán. It contains abundant garnet, meso-perthitic feldspar, quartz, graphite, and sillimanite. Rutile and zircon are common accessory minerals. In this sample rutile is prismatic, sometimes with pyramidal terminations, ranging in size up to 1200  $\mu\text{m}$  in length and with a maximum width of 80  $\mu\text{m}$ , mostly reddish-brown colored; only few of the observed crystals are dark brown.

## METHODOLOGY

### Sample preparation

After the petrographic analysis aimed to confirm the presence of rutile, the selected samples were crushed and heavy minerals were separated using conventional techniques (*e.g.*, Solari *et al.*, 2007). The heavy minerals concentrate was further passed through a Frantz magnetic separator to eliminate magnetic minerals (with currents up to 2 A). The final concentrate of non-magnetic heavy minerals consisted of zircon, rutile, apatite, and eventually titanite. Among those phases, rutile is the only dark mineral, so hand picking was pretty straightforward under the binocular microscope. Approximately 120–150 rutile grains from each sample, representative of all morphologies, color and size, were selected and casted in a ring mount with epoxy resin, polished with sandpaper, and then imaged under a binocular microscope prior to analysis.

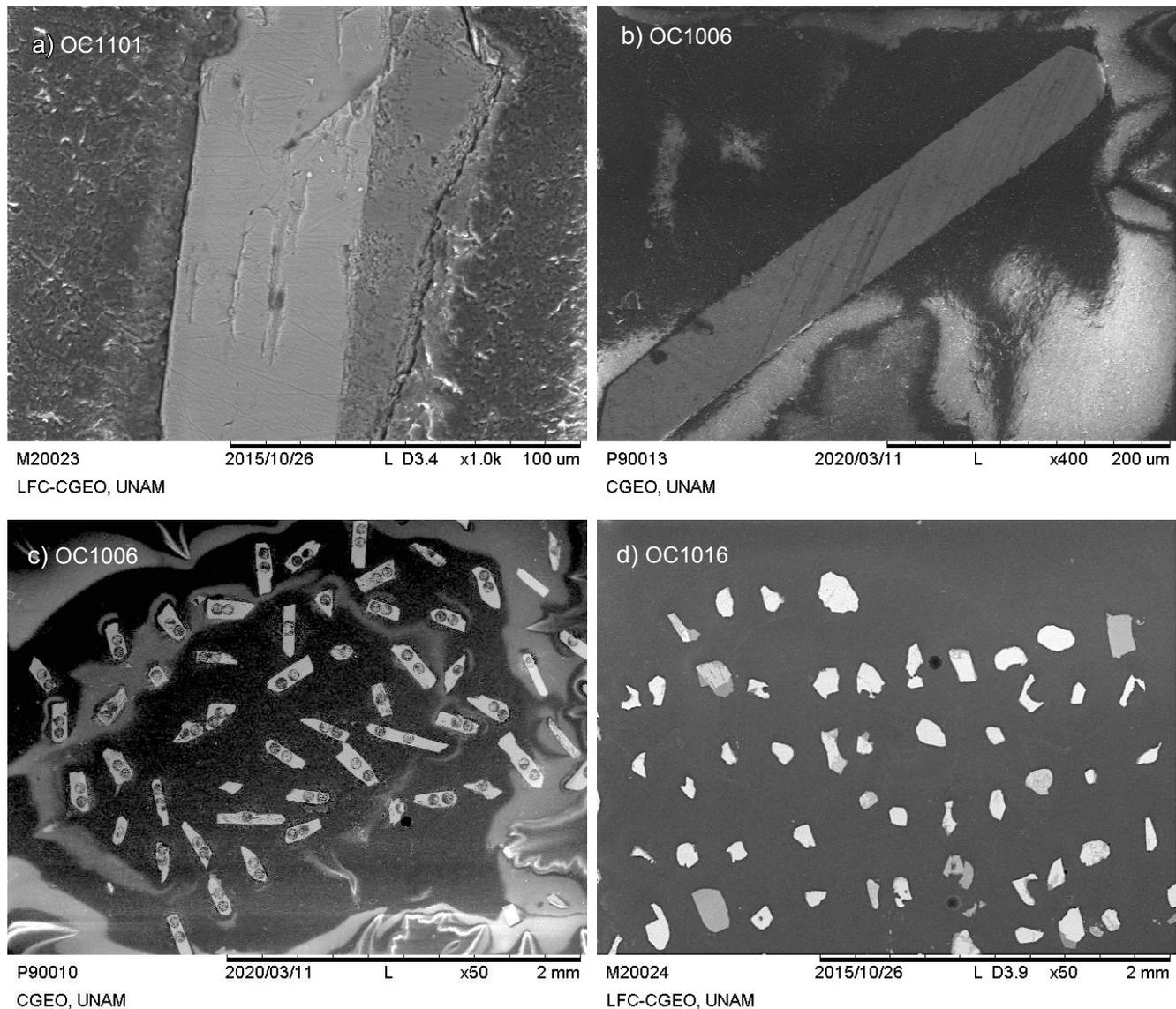


Figure 2. Scanning electron microscope (SEM) photomicrographs of some selected and mounted rutile crystals. The scale is indicated in each image. a) A twinned rutile crystal belonging to sample OC1101. b) A needle-shaped rutile crystal belonging to sample OC1006. c) Post-ablation, enlarged view of some mounted crystals belonging to sample OC1006. d) Pre-ablation image of some selected rutile crystals belonging to sample OC1016.

### Rutile U-Pb analyses

U-Pb analyses were performed by laser ablation inductively-coupled plasma mass spectrometry (LA-ICPMS) employing a Thermo ICap Qc quadrupole ICPMS, coupled with a Resolution M050 excimer laser workstation, located at Laboratorio de Estudios Isotópicos, Centro de Geociencias, Universidad Nacional Autónoma de México (UNAM). A “squid” signal homogenizer was employed right after the ablation cell, before the ablated material entered the plasma. 350 ml/min of He were used as a carrier gas together with *ca.* 800 ml/min of Ar, mixed downstream with 4.5 ml/min of N<sub>2</sub>. A frequency of 5 Hz was utilized during this work, with a constant on-target fluence of 5 J/cm<sup>2</sup>, monitored at the beginning and end of each analytical session with an external energy monitor and systematically employing, throughout the entire duration of this study, an analytical spot of 60 μm. Background was acquired for 15 s, whereas the laser fired for 30 s, for a total sweep time of 0.530 ms, in which all isotopes of interest were acquired, simultaneously, for chemistry and geochronology (see

Tables S1 and S2 in the supplementary material). We did not measure the depth of holes in rutile. However, comparative studies performed on zircons (Bernal *et al.*, 2014) with the same instrument allow us to estimate the penetration depth for rutile in approximately 12–15 μm during the 30 s of analysis. Two rutile standards were employed for this work: R-10 as primary standard, whereas R-19 acted as secondary (control) standard. The accepted ages for the R-10 and R-19 standards are 1090±5 Ma and 493±5 Ma, respectively (Luvizotto *et al.*, 2009; Zack *et al.*, 2011).

To take into account the modeled down-hole fractionation observed in the primary rutile standard, the data reduction was performed using the Iolite 3.5 package (Paton *et al.*, 2010; 2011), making use of the UComPbine data reduction scheme of Chew *et al.* (2014). The calculated age uncertainties corresponded to a two-standard error. Concordia diagrams for all the analyzed samples are plotted using IsoplotR (Vermeesch, 2018). Since rutile incorporates, at the moment of its crystallization, a variable amount of common Pb, this must be

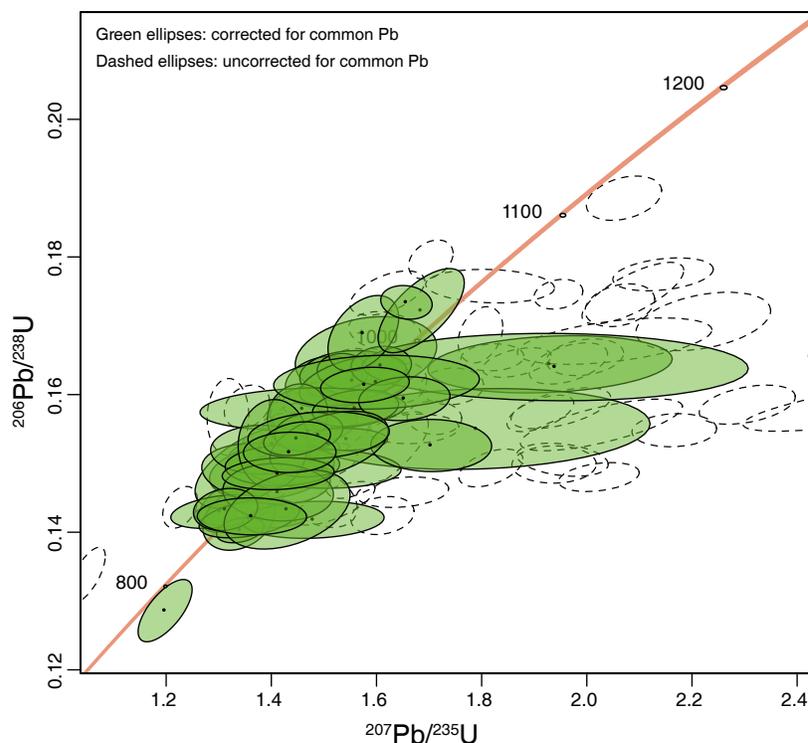


Figure 3. Example of an unknown rutile sample, with data uncorrected (dashed ellipses) and corrected (green ellipses) for common Pb using the methodology explained in the text. Ellipses represent 2-sigma uncertainties.

carefully evaluated to perform a proper correction of the isotope ratios. Fortunately, rutile does not incorporate virtually any Th in its crystal-line network (e.g., Table S2). The common Pb can be thus calculated monitoring  $^{208}\text{Pb}$ , since  $^{208}\text{Pb}$  is formed by decay from  $^{232}\text{Th}$  but, in the absence of the radioactive isotope, all the contained  $^{208}\text{Pb}$  can be considered “common”. Therefore, the correction can be properly applied to the unknown samples (e.g., Figure 3). In general, we analyzed 50–60 rutile crystals from each sample. Finally, by monitoring the signal of particular major and trace elements, we were able to detect any anomaly, such as the presence and ablation of mineral inclusions that could affect the geochronological and chemical data. In such cases, the signal integration in Iolite was manually adjusted or the analysis was rejected. A further filtering was done on discordance, eliminating those analyses which were outside the -5 to 30 % range of concordance (uncolored ellipses in Figure 4).

The secondary standard, measured over the course of these analytical sessions, yielded a concordia age of  $495.9 \pm 5.9$  Ma, in agreement with the aforementioned accepted age (Figure 5). The NIST-SRM 610 glass standard was also analyzed in each experiment (at the beginning, at the end and every 30 unknown analyses) to allow recalculation of trace and REE element concentrations in rutile crystals, employing  $^{49}\text{Ti}$  as internal standard. The reproducibility of NIST standard glasses by LA-ICPMS is very good, with relative standard deviations in general better than 1 % (e.g., Solari *et al.*, 2010, 2015).

## RESULTS

### Rutile U-Pb Geochronology

Once corrected for common Pb, most of the rutile analyses yield concordant or slightly discordant results (Figure 4). All the samples have a unimodal distribution (Figure 4, insets), with apparent  $^{206}\text{Pb}/^{238}\text{U}$

ages straddling the concordia in the 779–1031 Ma range (sample OC1101, Figure 4a), in the 917–1114 Ma range (sample OC1006, Figure 4b) or in the 911–1167 Ma range (sample OC1014, Figure 4c). The sample OC 1016 yields rutile analyses clustering in the 850–1009 Ma range, although few grains have younger ages, down to 779 Ma (Figure 4d). Finally, the sample OC1017 shows a main age cluster at 910–1026 Ma (Figure 4e).

### Rutile trace-element composition and Zr-in thermometry

Common trace elements were measured in the studied rutile crystals on the same analyzed spot. Detrital rutile chemistry in younger, siliciclastic rocks, reflects its original formation environment. The characterization of the chemical composition in rutile crystallizing in basement rocks, such as the case of the OC, can thus draw important data to be used reference in the study of younger sediments (e.g., Zack *et al.*, 2004; Triebold *et al.*, 2012; Rösel, 2019; Martini *et al.*, 2020). In general, the studied OC rutile grains fall in the metapelitic field of the Cr vs. Nb diagram proposed by Zack *et al.* (2004), characterized by high Nb and Cr values (compare OC samples in Figure 6a, with three examples of rutile belonging to mafic rocks from Mexico and Guatemala). The concentration of most of the chemical elements measured in OC rutile is variable (examples in Figure 6b to 6e, all analyzed elements in Table S2). The elemental distribution is, in several cases, similar from one sample to the other, with some elements enriched, especially in the OC-1016 sample (quartzofeldspathic in composition), such as Mo, Zr, Hf, for instance. U is more or less equally distributed, with median concentrations ranging from 23 to 63 ppm (Figure 6e). Finally, it is important to underline the Zr-in thermometer behavior of the studied rutile crystals (according to the formulas of Tomkins *et al.*, 2007): they define median crystallization temperatures in the 808–873 °C range, with the sample OC-1016 recording the highest values among the studied samples (Figure 6f).

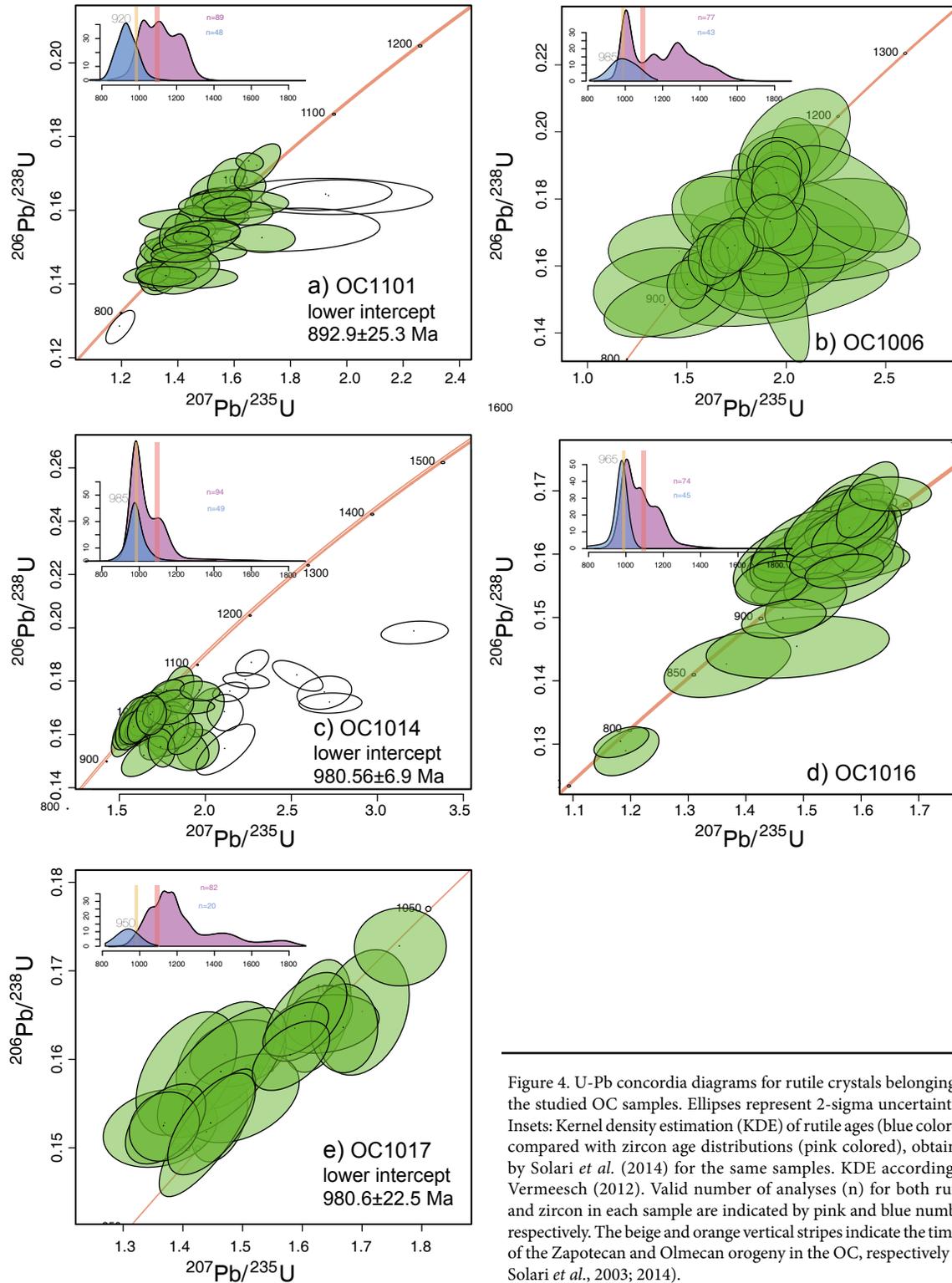


Figure 4. U-Pb concordia diagrams for rutile crystals belonging to the studied OC samples. Ellipses represent 2-sigma uncertainties. Insets: Kernel density estimation (KDE) of rutile ages (blue colored) compared with zircon age distributions (pink colored), obtained by Solari *et al.* (2014) for the same samples. KDE according to Vermeesch (2012). Valid number of analyses (n) for both rutile and zircon in each sample are indicated by pink and blue number, respectively. The beige and orange vertical stripes indicate the timing of the Zapotecan and Olmecan orogeny in the OC, respectively (*cf.* Solari *et al.*, 2003; 2014).

**DISCUSSION**

**Rutile closure temperature and Pb diffusion**

As briefly stated in the introduction, rutile Pb closure temperatures are calculated according to different parameters and techniques. Recent studies have modified the initial findings of Mezger *et al.* (1989), who

calculated a Pb closure temperature of *ca.* 400 °C, to higher values of *ca.* 600–630 °C (Cherniak, 2000; Kooijman *et al.*, 2010 and 2012; Smye and Stockli, 2014). The diffusion studies performed by the former authors make also evident that Zr-in-rutile thermometry allows the calculation of near-metamorphic peak rutile crystallization temperatures, especially in granulite facies rocks (*e.g.*, Kooijman *et al.*, 2012). It is also clear that

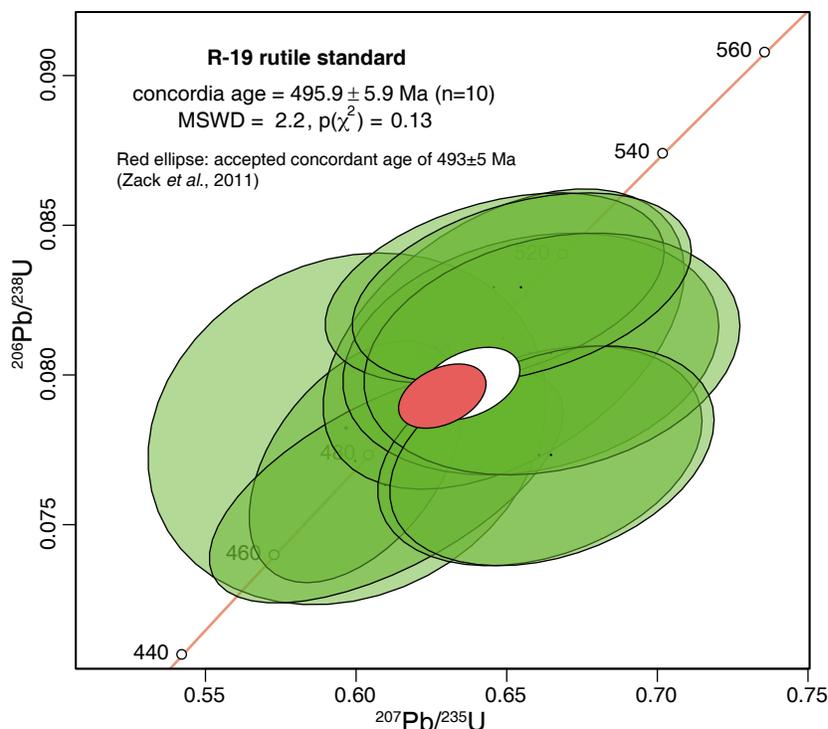


Figure 5. U-Pb concordia diagram for data of the secondary standard R-19 measured over the course of this study. Ellipses are at 2-sigma uncertainty.

Pb diffusion in rutile is completely decoupled from Zr concentrations and distribution, making it impossible to correlate Zr-in-rutile temperatures with U-Pb ages. In this study we did not perform X-Y or depth profile analyses (*cf.* Smye and Stockli, 2014), so the age or concentration calculations are an average of the material sampled in the analyzed spot (60  $\mu\text{m}$  in diameter and around 12–14  $\mu\text{m}$  in depth, see also Bernal *et al.*, 2014). Because the size of the studied rutile crystals (see Sample Description and Figure 2) and the granulite facies metamorphism that affected the OC rocks, whose temperature exceeded the Pb closure temperature reported above, we assume that most of the crystals were reset during the Zapotecan orogeny (*ca.* 990 Ma, Solari *et al.*, 2003) and, thus, the calculated ages reflect the cooling through the Pb blocking temperature of *ca.* 600  $^{\circ}\text{C}$ . Although we do not discard the existence of eventual inherited rutile grains (for instance, some of the older rutile in sample OC1006, Figure 4b), for the present work in which we discuss their cooling after granulite metamorphism, we consider the maximum kernel density estimation (KDE hereafter) rutile distribution (insets in Figure 4) and the difference in time and temperature with the granulite metamorphic peak. The concordia lower intercept could give a more reliable age of the cooling event (see, for instance, Mezger and Krogstad, 1997). However, the age distribution of some of the studied samples (*e.g.*, sample OC1006 and OC1016, Figures 4b and 4d, respectively) does not allow the calculation of a lower intercept in the concordia diagram. It is important to underline, however, that for those samples in which the lower intercept can be calculated (remainder samples in Figure 4), ages obtained are within error of the KDE maximum distribution. We are thus confident that the KDE maximum distribution is a reliable parameter that can be considered as the best approximation to the post-metamorphic peak cooling age for the studied samples.

#### Oaxacan Complex cooling curves

Combining the analytical results obtained in this research on rutile grains belonging to the OC, some interesting interpretations can be

drawn. Chromium and Ni concentrations of the analyzed rutile crystals suggest a sedimentary protolith for the analyzed granulite rocks. This is important because it is sometimes complex to understand the protolith origin of granulite rocks (see, for instance, Solari *et al.*, 2014, 2020).

The rutile KDE age distributions (Figure 4) allow a comparison with the detrital zircon grains of the same samples that were previously analyzed by Solari *et al.* (2014). Zircon, which is a mineral with a U-Pb closure temperature at about  $>900$   $^{\circ}\text{C}$ , shows during magma crystallization or eventually, as is the case of OC, during the granulite-facies metamorphism, a variable age distribution in KDE diagrams that reflect either the protolith (age distributions older than *ca.* 1.0 Ga) or metamorphic crystallization age (age distribution of *ca.* 990 Ma). Arc-forming events (*ca.* 1.15–1.2 Ga, Keppie *et al.*, 2001 and 2003; Weber *et al.*, 2010; Weber and Schulze, 2014) are followed by two tectonothermal pulses, namely the Olmecan and Zapotecan Orogeny at *ca.* 1100 and *ca.* 990 Ma, respectively (Solari *et al.*, 2003; Solari *et al.*, 2014). While the Olmecan is mostly related to a yet-to-be understood, local migmatization event, the Zapotecan orogeny is the granulite-facies metamorphic event synchronous throughout Oaxaquia (see, for instance, Lawlor *et al.*, 1999; Weber and Köhler, 1999; Solari *et al.*, 2003 and 2014; Cameron *et al.*, 2004; Weber and Schulze, 2014). While zircons yield multimodal distributions, witnessing the magmatic pulses in which they crystallized, or eventually, the granulite-facies metamorphism that allowed zircon recrystallization, rutile show unimodal distributions, reflecting their systematic U-Pb reset during granulite facies metamorphism at *ca.* 990 Ma. The age difference of the rutile main distribution from the Zapotecan event reflects the timing of cooling from 808–873  $^{\circ}\text{C}$  (rutile crystallization temperature according to the Zr-in thermometer, Figure 6F) to *ca.* 600  $^{\circ}\text{C}$  for the U-Pb closure temperature for rutile. The calculated cooling rates from the metamorphic peak temperature and age indicate, from N to S (being sample OC1101 the northernmost and sample OC1017 the southernmost ones, Figure 1), a range from *ca.* 3  $^{\circ}\text{C}/\text{Ma}$  in the northernmost OC outcrops, to *ca.* 6  $^{\circ}\text{C}/\text{Ma}$  in the south.

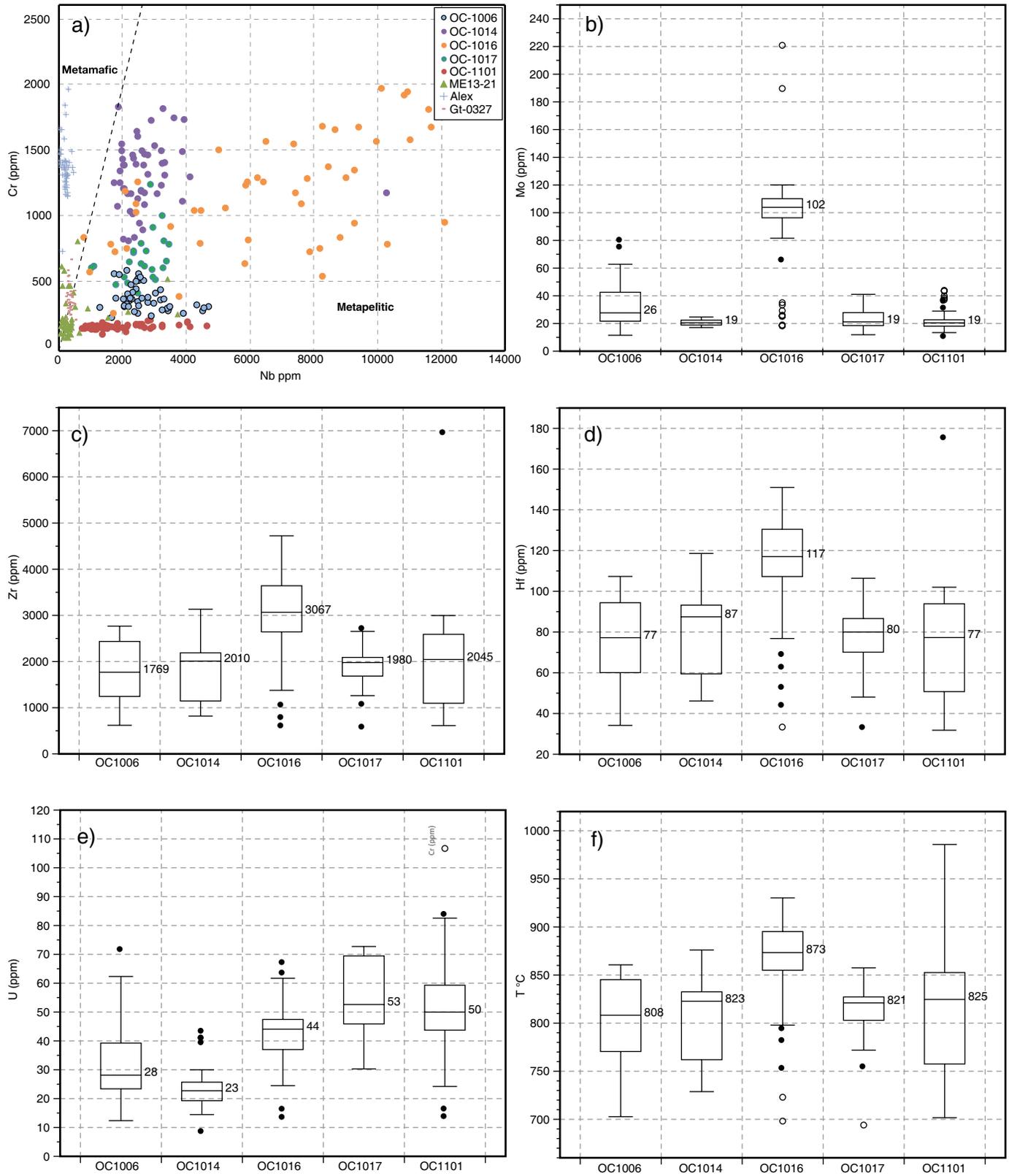


Figure 6. Geochemical composition of analyzed rutile crystals from metapelitic samples of the Oaxacan Complex. a) Cr vs. Nb classification diagram of Zack *et al.* (2004). Rutile composition of three metabasic samples are included for comparison: ME13-21 is a Permian garnet-bearing amphibolite belonging to the Caltepec fault zone, Mexico (*e.g.* Elías-Herrera *et al.*, 2007); Alex is a rutile sample belonging to late Precambrian-early Paleozoic anorthosite-gabbro sequences in Chiapas, Mexico (Cisneros de León *et al.*, 2017); Gt-0327 is a Cretaceous mafic eclogite from the Sierra de Chuacús, Guatemala (Ortega-Gutiérrez *et al.*, 2004). b) to e) box plots with the median value (number close to each box) of some of the elements analyzed in rutile for each OC sample. Concentrations are in ppm. f) Rutile temperature, in Celsius degrees, calculated using the Tomkins *et al.* (2007) Zr-in thermometer. The number close to each box corresponds to the calculated median temperature.

The sample OC1006 collected just south of Nochixtlán (Oax.) shows a maximum distribution at 985 Ma, that almost overlaps with analyzed zircon grains in the same sample (*cf.* Solari *et al.*, 2014), indicative of a very fast cooling rate calculated with *ca.* 42 °C/Ma. Similarly, sample OC1014 shows a cooling rate of *ca.* 45 °C/Ma. Sample OC1016 presents an intermediate cooling rate of about 11 °C/Ma and two of the analyzed crystals show an even slower cooling rate of 1.5 °C/Ma (with ages of *ca.* 800 Ma, Figure 4D). It is possible that these grains were preserved as inclusions in garnet, where they are often observed in thin section, thus allowing such slow cooling rate.

Few published data are available to decipher the cooling history of the granulite basement in Mexico and, among those, rutile has not been employed yet. In the first study integrating cooling ages, Keppie *et al.* (2004) interpreted the whole OC as having cooled through one unique curve with an intermediate cooling rate of *ca.* 8 °C/Ma since the granulite metamorphism occurred at *ca.* 945 Ma, and then through a slower cooling rate of *ca.* 2 °C/Ma to expose the OC to the surface at about 710–760 Ma. Those events were associated to a tectonic switching from steep to flat slab subduction, and to thermal relaxation of the lithosphere, respectively. Our data are indicative of a more complex history, in which rutile plays an important role in identifying a heterogeneous cooling in the OC, which is indicative of the existence of several tectonic blocks, or slices, which behaved independently during the exhumation, right after the Zapotecan event. The shear zones previously characterized by Solari *et al.* (2003) in the northern OC are probably only some of the tectonic boundaries between those slices, mostly reactivated during the complex tectonics that affected the Mexican basement during the Phanerozoic (*e.g.*, Solari *et al.*, 2004a) and, based on the current rutile cooling ages, possibly acted already after the Zapotecan metamorphic peak, during the initial cooling and exhumation. Similar conclusions were recently drawn by Abdullin *et al.* (in press), whose data indicate that the heterogeneity characterized the OC cooling and exhumation even during the Mesozoic.

## CONCLUSIONS

The application of rutile thermometry and U-Pb geochronology on crystals belonging to some metamorphic units of the Grenvillian Oaxacan Complex, southern Mexico, demonstrates that the exhumation after the Zapotecan granulite facies event (*ca.* 990 Ma) was heterogeneous among the different tectonic slices that compose the OC.

While this is a first approximation to U-Pb analysis in rutile, which further deserves a deeper study to better characterize the Pb diffusion during high-grade metamorphism, this example illustrates how combining the dating of different minerals with different closure temperatures (in this case, rutile and zircon, but the example holds true also when including apatite and micas, among others), collected in well-defined tectonic slices, is fundamental to construct meaningful cooling curves through geologic time in basement rocks.

Moreover, the chemical distribution of some of the analyzed rutile trace elements allow a further characterization, which can be useful in provenance analyses of younger sedimentary sequences in which rutile can be found as detrital heavy mineral (*e.g.*, Ortega-Flores *et al.*, 2018; Martini *et al.*, 2020) due to its sensitivity to discriminate between protoliths.

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## SUPPLEMENTARY MATERIAL

Supporting supplementary tables S1 y S2 can be found at the journal web site <<http://rmcg.unam.mx/>>, in the html version of this paper.

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