SHRIMP and LA-ICPMS U–Pb zircon geochronology of post-tectonic granitoid intrusions in NW of Central Iberian Zone

Geocronologia por SHRIMP e LA-ICPMS em granitóides pós-tectónicos no NW da Zona Centro-Ibérica

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Abstract: Resolving time differences between successive magmatic pulses in granitoid plutons is often a difficult task. High-precision ages, obtained by U-Pb SHRIMP in Lavadores granite, provide evidence that the crystallization of this granite happened at 294±3 Ma which is more precise that a previous age obtained by conventional U-Pb method on multigrain zircon fractions by isotopic dilution (298±11 Ma). In Madalena and Castelo do Queijo granites a zircon geochronological study was performed using a LA-ICPMS. However, there are only a few concordant zircons due the presence of analysis with high amounts of common lead. The Concordia ages yield 307±8 Ma and 303±4.6 Ma for Castelo do Queijo and Madalena granites, respectively. Given the high discordance of zircons, weighted average diagrams were used for all analyses, obtaining a mean of 296±11 Ma for the Castelo do Queijo granite and 296±3 Ma for the Madalena granite. Although these means are not precise ages, they are more in agreement with geological and structural data than the Concordia ages.

Keywords: SHRIMP, LA-ICPMS, Zircon, Granites, Geochronology.

1. Introduction

Plutonic rocks often record a complex mode of formation, involving multiple mantle and crustal sources, mingled and mixed liquids, episodic fractionation of major and accessory mineral phases during solidification, as well as post-magmatic hydrothermal alteration. Attributing precise timing to some of these processes is often not an easy task. U-Th-Pb geochronology is becoming an increasingly important tool in many aspects of Earth science research because technical developments have provided opportunities to improve precision and accuracy, enhance spatial resolution, and acquire data more efficiently. Some of the most exciting advances in geochronology are being driven by SHRIMP (Sensitive High Mass Resolution Ion Microprobe) and by Laser Ablation Inductively Coupled Plasma Mass Spectrometry (LA-ICPMS).

This work focuses on the first dating of zircons from Madalena and Castelo do Queijo granites by LA-ICPMS and a new high-precision SHRIMP U–Pb analyses that were performed on igneous zircons from the Lavadores granite.

2. Geological setting

The granite rocks selected for this study are located in the west boundary of the Central Iberian Zone, in Northern Portugal. At the coast line of the Porto-Vila Nova da Gaia, several late-variscan granites outcrop, such as the Castelo do Queijo granite and Lavadores-Madalena pluton with an orientation NW-SE that according to Ribeiro (2013) correspond to the trending, in this sector, of one of the most important Variscan structures, the Porto-Tomar Shear Zone. The Castelo do Queijo is a small outcrop at seaside of the city of Porto, and is intrusive in the gneisses-migmatites from the Foz do Douro Metamorphic Complex (Noronha & Leterrier, 2000). At Vila Nova da Gaia, outcrops the Lavadores-Madalena granitic pluton, which is an intrusive body in a gneiss-migmatite (metatexites and...
The TEMORA zircon (417 Ma, Black which is used as a concentration standard (238 ppm U). measuring the SL13 zircon (Claoue-Long structure, inclusions, fractures and physical defects were Country, Spain. Prior to isotopic analysis, the internal (LA-ICPMS) at the Geochronology and Isotope SHRIMPTOOLS software (available from www.ugr.es/~fbea), specifically developed for IBERSIMS 3. Analytical techniques A classic mineral separation procedure has been applied to concentrate minerals suitable for U-Th-Pb dating using the facilities available at Department of Geosciences, Environment and Spatial Planning, Porto University. Zircon was separated from < 250 μm sieved fractions using standard heavy liquids and magnetic techniques. Zircon grains were carefully handpicked under a binocular microscope and embedded in epoxy mounts. The grains were then hand-grounded down to expose their central portions and polished. The Sensitive High Resolution Ion Microprobe SHRIMP II Ile/mc instrument of the IBERSIMS lab (Granada University) was used to determine zircon age in a sample selected from the Lavadores granite. Hand-picked zircons from the studied sample, several grains of the TEMORA and one grain of SL13 zircons standards, plus a few grain of the GAL zircon were casted on a 3.5 cm diameter epoxy mount (megamount), polished and documented using optical (reflected and transmitted light) and scanning electron microscopy (secondary electrons and cathodoluminescence CL). After extensive cleaning, mounts are coated with gold (80 μm thickness) and inserted into the SHRIMP for analysis. All calibration procedures were done on the standards included on the same mount. Mass calibration was done on the GAL zircon (ca. 480 Ma, very high U, Th and common lead content; Montero et al., 2008). Analytical sessions started measuring the SL13 zircon (Claoue-Long et al., 1995), which is used as a concentration standard (238 ppm U). The TEMORA zircon (417 Ma, Black et al., 2003), used as isotope ratios standard, was then measured every 4 unknowns. Data reduction was done with the SHRIMPTOOLS software (available from www.ugr.es/~fbea), specifically developed for IBERSIMS by F. Bea.

U-Th-Pb geochronology of zircon from Madalena and Castelo do Queijo granites was conducted by in situ laser ablation inductively coupled plasma mass spectrometry (LA-ICPMS) at the Geochronology and Isotope Geochemistry facility, SGIker-University of the Basque Country, Spain. Prior to isotopic analysis, the internal structure, inclusions, fractures and physical defects were identified by backscattered electron (BSE) imaging using a SEM, FEI QUANTA 400FEG ESEM (CEMUP), and a JEOL FXA-8500F (LNEG) scanning electron microscope. The analyses involved the ablation of minerals in ca. 60 μm thick petrographic sections with an UP213 frequency quintupled Nd:YAG based laser ablation system (NewWave) coupled to a Thermo Fisher Scientific X series 2 quadrupole based ICP-MS instrument with enhanced sensitivity through a dual pumping system. Ablation spot diameters of 30 μm and 25 μm associated to repetition rates of 5 Hz and laser fluence at the target of ca. 5 J/cm² were used for zircon dating. The ablated material was carried into helium and then mixed with argon, before injection into the plasma source.

Data were corrected in both cases for U-Pb and Th-Pb fractionation occurring during laser sampling and for instrumental mass discrimination (mass bias) by standard bracketing with repeated measurements of GJ-1 zircon standard (Jackson et al., 2004). Repeated analyses of GJ-1 zircon standards treated as unknowns were used to control the reproducibility and accuracy of the corrections. For each analysis, the time resolved signal of single isotopes and isotope ratios was monitored and carefully inspected to verify the presence of perturbations related to inclusions, fractures, mixing of different age domains or common Pb. Data reduction was carried out with the Iolite software package (Paton et al., 2011). The data were processed with ISOPLOT software (Ludwig, 2001) for Concordia plots, weighted average and Concordia age calculations.

4. Results and conclusions The zircons from the Lavadores granite range from big, equant to elongate, entire or fragments of euhedral grains, ranging in size from 110 to 240 μm. CL images show typical igneous zircons consisting in a light or dark usually unzoned core rimmed with oscillatory zoning. U contents are very high ranging from 379 to 2600 ppm; moderated Th contents range from 61 to 1067 ppm. The Th/U ratios, present values between 0.25 and 1.55, are in the normal range in zircons of magmatic origin, implying the absence of a Pb loss event (e.g. Castro et al., 2011 and reference therein). There is not any evidence in the studied zircons of any old inherited zircon grain. Twenty four of the 31 analyzed spots (discordance < 5%) formed a well-grouped cluster on the Wetherill plot that yield a Concordia age of 294±4 Ma (Fig 1). This age is more precise that a previous one obtained by conventional U–Pb method on multigrain zircon fractions by isotopic dilution, 298±11 Ma (Martins et al., 2011).

In Madalena and Castelo do Queijo granites a zircon geochronological study was performed using the LA-ICPMS methodology. The zircon samples from the Madalena and Castelo do Queijo granites are about 150–220 μm in length, most of which are long or short columnar (subhedral to euhedral) in shape. The internal structures of most zircons in both granites are very similar and show typical magmatic characteristics. Generally, they consist of an unzoned to weakly zoned core surrounded by a rim of euhedral regular
fine oscillatory zoned zircon in the BSE images. Zircons with inherited cores were not detected.

Both granites present high U contents in the range of 380 to 3060 and 839 to 2750 ppm for Castelo do Queijo and Madalena granites, respectively. The Th contents are very similar in both granites ranging from 262 to 960 ppm. Common Pb is rather high, whereas the Th/U ratio is mostly moderate to fairly high (0.2–0.9), typical of magmatic zircons.

Twenty five points in eleven zircon crystals from the Castelo do Queijo granite and forty three points in eighteen zircons from Madalena granite have been analysed. Most of the analytical points were selected within oscillatory zoned external parts of grains in order to determine the main magmatic event. A few points, however, were placed in cores or central parts of crystals, to test the possible presence of inheritance. All analysis with high quantity of common lead, and those with high analytical errors (higher than 10% in ratios) have been excluded from our interpretation (nine points from Castelo do Queijo and eleven from Madalena). Then, results with discordance (Meinhold et al., 2010) lower than 5% have been used to construct the Concordia diagram and to obtain the Concordia age. The Concordia age for the Castelo do Queijo is 306.7±8 Ma (Fig. 2a) and for Madalena is 303.4±4.6 Ma (Fig. 3a). However, in both samples there are only a few concordant zircons, which makes these ages problematic. Given these significantly discordances we decided to construct a weighted average diagram including all the analyses where the mean ages obtained is 296±11Ma (Fig. 2b) and 296±3Ma (Fig. 3b) for the Castelo do Queijo and Madalena granites, respectively. Although we are aware that these ages can’t be considered precise, we assume that both granites are post-tectonic as the Lavadores granite based on field relationships and structural data.

Fig. 1. Concordia diagram showing results of SHRIMP zircon analyses from the Lavadores granite.

Fig. 1. Diagrama Concordia com o resultado das análises de zircão por SHRIMP do granito de Lavadores.

Fig. 2. (a) Concordia diagram showing results of LA-ICPMS zircon analyses from the Castelo do Queijo granite; (b) Weighted Average diagram and age distribution for the 11 magmatic zircons analyzed.

Fig. 2. (a) Diagrama Concordia com o resultado das análises de zircão por LA-ICPMS do granito do Castelo do Queijo; (b) Idade média obtida nos 11 zircões analisados.
Fig. 3. (a) Concordia diagram showing results of LA-ICPMS zircon analyses from the Madalena granite; (b) Weighted Average diagram and age distribution for the 18 magmatic zircons analyzed.

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