Rb-Sr AND SINGLE - ZIRCON GRAIN ²⁰⁷Pb/²⁰⁶Pb CHRONOLOGY OF THE MONESTERIO GRANODIORITE AND RELATED MIGMATITES. EVIDENCE OF A LATE CAMBRIAN MELTING EVENT IN THE OSSA-MORENA ZONE, IBERIAN MASSIF

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ABSTRACT

The Monesterio granodiorite, a small granodioritic body emplaced in a migmatitic complex in the SW of the Olivenza-Monesterio antiform, is a key plutonic body to understanding the relationships among the magmatism, metamorphism, and deformation in the Ossa-Morena Zone, SW Iberian Massif. We dated the granodiorite with the single-zircon stepwise-evaporation $^{207}Pb/^{206}Pb$ method, and the related migmatization event with the Rb-Sr method on leucosomes. Our results indicate that the Monesterio granodiorite crystallised at 510 ± 7 Ma and its protolith had a component with Upper Proterozoic zircons with a minimum age of 1696 Ma. Leucosomes give a Rb-Sr age of 511 ± 40 Ma (MSWD = 1,7) with initial $^{87}Sr/^{86}Sr = 0.70914 \pm 0.00048$. The lower initial $^{87}Sr/^{86}Sr$ of the granodiorite and its calc-alkaline chemistry precludes it from having derived from the same protolith as the migmatites. The existence of different magmatic bodies in the Ossa-Morena Zone with ages clustering around 500-510 Ma reveals the existence of a significant melting event during the Late Cambrian that involved protoliths with very different geochemical and isotopic signatures.

Key words: Geochronology, Pb-Pb Migmatite, Monesterio granodiorite, Ossa-Morena Zone Spain.

RESUMEN

La granodiorita de Monesterio es un pequeño cuerpo emplazado en un complejo migmatítico en el SO del antiforme Olivenza-Monesterio, importante para entender las relaciones entre magmatismo, metamorfismo y deformación en la Zona de Ossa-Morena. Se ha datado la granodiorita por el método de evaporación secuencial de ²⁰⁷Pb/²⁰⁶Pb en cristal único de circón y los leucosomes de las migmatitas circundantes por el método Rb-Sr. Los datos indican una edad de cristalización de la granodiorita de 510 ± 4 Ma y un posible protolito Proterozoico Superior con una edad mínima de ~ 1.700 Ma, obtenida a partir de núcleos heredados de los circones analizados. Los leucosomes dan una edad Rb-Sr de 511 ± 40 Ma, con una relación ⁸⁷Sr/⁸⁶Srin. = 0,70914 ± 0,00048. La relación inicial de ⁸⁷Sr/⁸⁶Sr en la granodiorita (~ 0,7049) es mucho más baja que en los leucosomes, lo que junto con su naturaleza calcoalcalina indica que no derivan del mismo protolito. La existencia en la zona de Ossa-Morena de diferentes cuerpos magmáticos con edades en torno a 500-510 Ma, indica un evento de fusión importante durante el Cámbrico Superior desarrollado sobre protolitos con características geoquímicas e isotópicas muy diferentes.

Palabras clave: Geocronología, Pb-Pb Migmatita granodiorita de Monesterio, Zona Ossa-Morena España.

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Introduction

The tectonometamorphic history of the Ossa-Morena Zone, SW Iberian Massif, is highly controversial due to the superposition of the Cadomian and Variscan deformations (Quesada, 1990, Dallmeyer and Quesada, 1992, Azor *et al.*, 1995) and large discrepancies in available geochronological data. A good example is the Monesterio granodiorite, a small granodioritic body emplaced in a migmatitic complex. Although crucial for understanding the relationships among magmatism, metamorphism and deformation in this zone, the complex has been dated by various methods from \approx 400 to 550 Ma (see below).

Radiometric dating of the Monesterio granodiorite and associated migmatites is certainly complex. The granodiorite is affected by a strong shear zone, has little petrographic or chemical variation, and its primary mineralogy is partially affected by hydrothermal alteration, making it very complicated to obtain reliable data with Rb-Sr or K-Ar methods. In addition, we found that a significant fraction of zircon crystals have inherited cores, so that conventional U-Pb ages of this mineral would give unrealistic mixed ages; besides, monazite and xenotime are often metamict (see Ochsner, 1993). Dating the migmatization event of the surrounding metapelites is also problematic. Isotopic disequilibrium during partial melting (Bea, 1996; Knesel and Davidson, 1996; Tommasini and Davies, 1997) makes it extremely difficult to use coupled mesosome-leucosomemelanosome samples for Rb-Sr isochrons. The low solubility of zircon in low-temperature highly silicic leucosomes (Watson and Harrison, 1983; Watson, 1996), on the other hand, means it is unreliable to use zircons for dating the leucosome segregation.

In this paper we present the results of a study aimed at obtaining the very best ages for the crystallization of the Monesterio granodiorite and the migmatization event that affected its host rock. To do so, we dated the granodiorite with the single-zircon stepwise-evaporation ²⁰⁷Pb/²⁰⁶Pb method (Kober, 1986, 1987). This technique, as it is less sensitive to secondary processes than U-Pb dating, can yield accurate crystallization ages on some zircon populations that display complex discordant patterns, and is still capable of dating different concentric parts of a single zircon grain (e.g. Doughertypage and Foden, 1996; Karabinos, 1997). Due to the abovmentioned lack of solubility of zircon in leucosomes, we dated the migmatization event with the Rb-Sr method.

Geological background and petrography

The Monesterio granodiorite is a small elongated body of $\approx 70 \text{ km}^2$, located in the S of the Olivenza-Monesterio antiform (fig. 1). This structure is a NW-SE km-scale SW-vergent fold, where the basal part of the Ossa-Morena stratigrafic sequence crops out (Azor, 1994). The lowermost formation, called Serie Negra, is composed of a thick sequence of metapelites and metagreywackes with intercalations of amphibolites, black quartzites and rare marbles (Carbalhosa, 1965; Eguiluz, 1987). Its base, where the central part the Monesterio granodiorite is located, is affected by low-P high-T metamorphism that locally produces metatexitic migmatites (Eguíluz et al., 1983; Eguíluz and Abalos, 1992). The contact between the granodiorite and wall-rock metapelitic migmatites is usually sharp and crossed by abundant aplitic dikes, thus suggesting the granodiorite is intrusive. However, when deformation is intense, the contact seems to be more gradual, suggesting the granodiorite might be a subautochthonous body.

The Monesterio granodiorite is a dark grey, medium-grained porphyritic rock with a local gneissic fabric. The main minerals are quartz, oligoclaseandesine (An₂₅-An₃₀), K-feldspar and biotite. Accessory phases are apatite, ilmenite, zircon, monazite, and rare xenotime. The chemical composition is very uniform and corresponds to a moderately peraluminous calc-alkaline granodiorite, with SiO₂ \approx 66 - 69 wt. %, CaO \approx 2 - 3 wt. %, Na₂O+K₂O \approx 6.5 - 7.5 wt. %, Na₂O >= K₂O, and mol. Al₂O₃/(CaO+Na₂O+K₂O) \approx 1.05 - 1.15.

Migmatites consist of metapelitic metatexites. Leucosomes appear as discordant lens-shaped veins seldom larger than 100 x 50 x 30 cm. They are composed of quartz, K-feldspar, albite-oligoclase (An₂-An₁₅), muscovite, and rare cordierite in large prismatic crystals. Biotite is rare, appearing as thin selvages located mostly at the contacts of leucosome veins and therefore interpreted as restitic. Accessory phases are limited to a few grains of ilmenite, apatite, monazite and zircon. After dissolution in HF, leucosomes always leave a residuum (0.05-0.2 wt. %) comprising very minute particles of graphite, suggesting they derived from partial melting of Serie Negra metapelites.

Previous chronological data

Previous attempts to determine the age of the Monesterio granodiorite are detailed below:

(1) Quesada (1990) obtained two Rb/Sr isochrons. The first gave 528 ± 100 Ma, with a MSWD = 6.95 and an initial 87 Sr/ 86 Sr = 0.7058 \pm 29, the second gave 552 ± 72 Ma, with a MSWD = 2.63 and an initial 87 Sr/ 86 Sr = 0.7049 \pm 21.



Fig. 1.—Shematic geological situation of the Monesterio granodiorite within the Olivenza-Monesterio Antiform, Iberian Massif. (Simplified from Eguíluz and Abalos, 1992).

(2) Dallmeyer and Quesada (1992) determined ${}^{40}\text{Ar}/{}^{39}\text{Ar}$ ages on three mineral concentrates. The first was a hornblende concentrate from a mafic xenolith inside the granodiorite and recorded an age of 553.1 ± 6.3 Ma. The other two were muscovite concentrates from mylonitic samples in shear zones within the granodiorite, which gave apparent ages of 458.9 ± 1 Ma and 412.8 ± 1.2 Ma respectively.

(3) Ochsner (1993) applied U-Pb methods on concentrates of monazite, xenotime and zircon from the granodiorite. Six analyses of highly metamict xenotimes gave a discordia line intercepting the concordia curve at 526.8 ± 9.9 / -7.0 Ma. Another six monazite fractions scattered around a linear array intersecting the concordia curve at 521 + 161 / -9 Ma. Five highly metamictic zircon concentrates also scattered around a line that intersects the concordia at 476 + 13 / -17 and 1597 + 256 / -230 Ma.

(4) Ordoñez-Casado *et al.* (1997) dated the outermost parts of zircons from the surrounding leucosomes with SHRIMP, obtaining an average age of 524 ± 7 Ma.

Samples

Zircons for chronology were collected from the least deformed and unaltered outcrop of the granodiorite, located in the southern part of the pluton, near the village of Monesterio. It is a biotite granodiorite with abundant large zircon crystals that were concentrated using conventional techniques of heavy liquids and magnetic separation. Zircon crystals appear either as long or short prisms with poorly developed pyramidal faces that, in most cases, correspond to P1 and P2 morphotypes (Pupin, 1980). They are usually pale yellow to brownish, turbid, translucent or opaque, and some of them have small dark inclusions. Selected zircons had a size about 250 x 150 µm. Cathodoluminescence studies showed that crystals with a rounded anhedral core are relatively common. We selected six unbroken idiomorphic zircon grains for analysis. Rb-Sr dating of leucosomes was performed on four specimens. Three are whole-rock samples collected from a large, coarse-grained, almost pegmatitic leucosome vein. From the fourth sample, which is an adjacent medium-grained small vein, whole-rock and three mineral concentrate —plagioclase, K-feldspar and muscovite— were analysed separately.

Analytical procedure

Single-zircon ²⁰⁷Pb/²⁰⁶Pb stepwise-evaporation and Rb-Sr isotope analyses were done at the University of Granada with a SEM-RPQ multicollector Finnigan MAT 262 Mass Spectrometer with a double-filament ion source arrangement.

Zircon grains were mounted on canoe-shaped Re filaments and heated until the Pb beam intensity was sufficient and common Pb emission (monitored by ²⁰⁴Pb signal) low enough. Then Pb was collected on the ionization filament for 20-30 min and afterwards analysed in 5 blocks with 7 scans per block. Data were acquired by peak hopping with the 206-204-206-207-208 mass sequence, using a secondary electron multiplier (SEM) as detector. The 204/206 mass-ratio was monitored to detect and, if necessary, correct for common Pb. Once the analysis was finished, a new analytical cycle (hereafter called step) started by heating the zircon on the evaporation filament to a higher temperature than in the previous step and analysing on the ionization filament as before. The procedure continued until all the Pb is exausted from the zircon. The number of steps depends on the size and Pb content of each zircon. Measurements with ²⁰⁴Pb/²⁰⁶Pb higher than 0.8 % at the 2 σ level were rejected. Factors for common Pb correction were calculated by iteration from the ²⁰⁴Pb/²⁰⁶Pb and ²⁰⁴Pb/²⁰⁷Pb ratios provided by the model of Stacey and Kramers (1975) at the calculated age until convergence to a constant value. Mass fractionation was corrected by multiplying by $\sqrt{(207/206)}$. Standard Errors for each step were calculated according to the formula:

 Table 1.—Isotopic data from single-zircon evaporation of the Monesterio granodiorite.

Zircon	Step	²⁰⁴ Pb/ ²⁰⁶ Pb	²⁰⁷ Pb/ ²⁰⁶ Pb (corrected)	SE % (2σ)	AGE (Ma)	Age Error
Mok2-6	1	0.000049	0.057441	0.34	509	7
Mok2-6	2	0.000034	0.057543	0.60	512	13
Mok2-1	1	0.000034	0.057354	0.66	505	14
Mok2-1	2	0.000029	0.057403	0.70	507	15
Mok2-4	1*	0.000239	0.057048	1.7	493*	39
Mok2-4	2*	0.000163	0.076645	0.63	1112*	12
Mok2-4	3*	0.000352	0.103951	1.12	1696*	23
Mok2-9	1	0.000448	0.057691	0.48	518	11
Mok2-10	1	0.000252	0.057367	0.10	506	2
Mok2-10	2	0.000041	0.057429	0.14	508	3
Mok2-10	3	0.000116	0.576624	0.17	517	4
Mok2-11	1*	0.000079	0.084993	0.35	1315*	7
Mok2-9 Mok2-10 Mok2-10 Mok2-10 Mok2-11	1 1 2 3 1*	0.000448 0.000252 0.000041 0.000116 0.000079	0.057691 0.057367 0.057429 0.576624 0.084993	0.48 0.10 0.14 0.17 0.35	518 506 508 517 1315*	

* Not used for calculation of crystallization age.

²⁰⁴Pb/²⁰⁶Pb corrected for mass fractionation and common lead, see text.

SE = Standard Error. Crystallization age 2σ level = 510 ± 4 Ma.

SE = $2*\sigma/\sqrt{n}$. However, the 95 % confidence interval for the final age is given by $(X - t_{(0.025)}\sigma/\sqrt{n}, X + t_{(0.025)}\sigma/\sqrt{n})$, where X and σ are the average and the standard deviation of measured steps, n the number of steps, and $t_{(0.025)}$ is the upper (0.025) point of the t-distribution for n-1 degrees of freedom (see Johnson and Bhattacharyya, 1984).

 87 Sr/ 86 analyses were done after separation by ion-exchange resins using conventional methods. External precision (2 σ) measured in 10 replicates of the standard WS-E (Govindaraju *et al.*, 1994) was about \pm 0.003 % rel. for 87 Sr/ 86 Sr. 87 Rb/ 86 Sr ratios were measured directly by ICP-MS (Montero and Bea, 1998), with an external precision better than \pm 1.2 % rel. (2 σ).

Crystallization age of Monesterio granodiorite

Isotopic data from stepwise Pb evaporation in single-zircons from the Monesterio granodiorite are summarized in table 1.

Under the cathodoluminiscence microscope, four coreless zircons were selected. They were idiomorphic and had oscillatory zoning, so we assumed they were truly magmatic. Three of them yielded several steps with a uniform age from rim to core close to 510 Ma. The other zircon gave only one step with nearly the same value. All the steps in these four crystals (fig. 2) gave a mean age of 510 ± 4 Ma (at 95 % confidence), which we consider the best estimation of the time of crystallization.

We also analysed two crystals that had a anhedral core. One of them, the long prism Mok-2.4, yielded three steps with different ages. Step 1, representing the outer rim, yielded 493 ± 39 Ma, with high common Pb content; step 2, representing the intermediate



Fig. 2.—Mean-age and confidence interval representation of eight measurements in four different zircon grains from the granodiorite of Monesterio. Lines connect individual measurements on a single zircon grain.

zone, gave 1112 ± 12 Ma, and step 3 provided 1696 ± 23 Ma. The age of the first step is virtually identical to that of coreless zircons and is likewise supposed to represent the age of magmatic crystallization. The third step represents a minimum estimate of the core age, and the second step probably represents a mixing value. The short prism, Mok-2.11, gave one step with 1315 ± 7 Ma, obviously a mixing value between the ages of the old core and the younger rim.

We therefore conclude that the Monesterio granodiorite crystallized at circa 510 Ma and its protolith had a component with Upper Proterozoic zircons with a minimum age of 1696 Ma. These values correspond roughly to the lower and upper intercepts on concordia given by U-Pb data on zircon concentrates (Ochsner, 1993).

Age of Migmatization

In the ⁸⁷Rb/⁸⁶Sr vs. ⁸⁷Sr/⁸⁶Sr diagram (fig. 3), the four whole-rock samples and the K-feldspar and plagioclase concentrates plot on an isochron at 511 ± 40 Ma with initial ⁸⁷Sr/⁸⁶Sr = 0.70914 ± 0.00048. The goodnes-of-fit is excellent, as revealed by a MSWD value of 1.7. Data are closely spread in a narrow range of ⁸⁷Rb/⁸⁶Sr, which accounts for the high error in the age estimates. The muscovite concentrate does not plot on the same isochron but is considerably younger (table 2) so that the muscovite - whole-rock age is circa 438 Ma. This effect is probably due to the partial loss of radiogenic ⁸⁷Sr, highly incompatible in micas, either as a consequence of later thermal events (see discussion in Azor *et al.*, 1995) or simply caused by hydrothermal alteration.

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 Table 2.—Rb-Sr isotopic data of minerals and whole-rock samples from migmatite leucosomes surrounding the Monesterio granodiorite.

Ref	Rb(ppm)	Sr(ppm)	⁸⁷ Rb/ ⁸⁶ Sr	⁸⁷ Sr/ ⁸⁶ Sr
Sok6-pl.	88.5	341	0.750	0.714623
Sok6-kfsp	92.3	305	0.876	0.715382
Sok6-wr	75.4	339	0.644	0.713836
Sok6-ms	157	45,7	10.034	0.772519
Mob-1 wr	79.5	325	0.708	0.714355
Mob-2 wr	136	301	1.308	0.718613
Mob-3 wr	112	298	1.084	0.717189

Mob-1, Mob-2, Mob-3 and Sok6-wr are whole-rock samples. Sok6-pl, Sok6-kfsp, and Sok6-ms are plagioclase, K-feldspar and muscovite concentrates respectively. The muscovite concentrate has not been used for the isochron.

Discussion and conclusions

The crystallization age of Monesterio granodiorite and the migmatization event in the surrounding migmatites is the same, about 510 Ma. However, the much lower initial ⁸⁷Sr/⁸⁶Sr, the calc-alkaline chemistry, and lack of graphite precludes this granodiorite from having been derived from the same protolith, suggesting instead that granodioritic melts: (1) originated from a feldspar-rich deeper source with lower ⁸⁷Sr/⁸⁶Sr than the metapelites, and (2) were later intruded into a ductile migmatitic core, as indicated by the above-described field relationships.

Intrusive rocks with an age close to that of the Monesterio granodiorite are common in the Ossa Morena zone. The Pallares granodiorite has an identical age (507 \pm 21 Ma Sm-Nd on apatite, 495 \pm 8 Ma, lower intercept of U-Pb discordia on zircons, Schäfer, 1990) and similar low initial 87 Sr/ 86 Sr \approx 0.706 (Cueto et al., 1983). The strongly peraluminous Higuera de Llerena and Riscal orthogneisses have a Rb-Sr age of 495 ± 13 Ma but with $\frac{87}{5}$ Sr/ $\frac{86}{5}$ r ≈ 0.711 (Azor *et al.*, 1995). The plutonic complex Táliga-Barcarrota, which includes peraluminous granitoids (Táliga), and peralkaline granitoids and mafic alkaline rocks (Barcarrota), yields a K-Ar age (amphibole, biotite, whole rock) of 505 ± 10 Ma (Galindo et al., 1987). All these data suggest the existence of a significant melting event during the Late Cambrian, apparently unrelated to the Cadomian orogeny, that involved protoliths with very different geochemical and isotopic signatures.

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Fig. 3.—⁸⁷Rb/⁸⁶Sr vs. ⁸⁷Sr/⁸⁶Sr plot of leucosome whole-rock and mineral concentrate samples.

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