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A revised Ordovician age for the Miranda do Douro orthogneiss, Portugal. Zircon U-Pb ion-microprobe and LA-ICPMS dating

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ABSTRACT

The Miranda do Douro orthogneiss was believed to be the oldest magmatic rock of the Central Iberian Zone, on the base of a U-Pb discordia upper intercept of 618 ± 9 Ma. Nevertheless, new ion-microprobe and LA-ICPMS U-Pb zircon dating revealed that the crystallization age was 483 ± 3 Ma. The orthogneiss also contains a 605 ± 13 Ma zircon population that indicates that the source-rock for the Ordovician magma was Pan-African. Moreover, a few ~3.17 Ga zircon grains were also recorded. These grains are the oldest found so far in Iberia, and its occurrence would suggest the involvement of an Archean crust in the Pan-African orogeny.

KEYWORDS **U-Pb dating. Ollo de Sapo. Pre-Variscan. Central Iberian Zone. Gneiss.**

INTRODUCTION

The Central Iberian Zone is a 600 km wide band located at the axis of the Variscan Belt of Spain and Portugal (Martínez Catalán et al., 2004a). It comprises two domains, the Ollo de Sapo Domain in the north, and the Schist-Greywacke-Complex Domain in the south (Fig. 1A). The first is named after the large, mainly volcaniclastic Ollo de Sapo (Toad's Eye) Fm, mainly composed of fine-grained and coarse-grained felsic gneisses and unconformably overlain by Arenig (472-479 Ma) sedimentary rocks (Parga-Pondal et al., 1964; Díaz Montes et al., 2004; Martínez Catalán et al., 2004b). The precise age of the Ollo de Sapo Formation is not yet well-known. It has been debated at length, with estimations that range from Proterozoic to Ordovician. Ongoing zircon dating enables us to date it provisionally at ~485-495 Ma.

Spatially associated with the Ollo de Sapo gneisses there are several deformed granitoids which, except in one case, have been dated by conventional U-Pb or Rb-Sr at around ~480 Ma (Lancelot et al., 1985; Valverde-Vaquero and Dunning, 2000; Vialette et al., 1987). The only exception was the Miranda do Douro orthogneiss (MDO; Fig. 1B) that yielded a discordia with an upper intercept of 618 ± 9 Ma, considered to be the age of crystallization (Lancelot et al., 1985). Therefore, the MDO would be the oldest magmatic rock of the Central Iberian Zone, only approached by the Sisargas gneiss (590 Ma, also an upper intercept of a discordia; Allegret and Iglesias, 1987), which crops out in the allocthonous basement of the Galicia Media-Tras Os Montes Zone. Far away at the southern boundary of the Central Iberian Zone (Fig 1), the gabbros and diorites of Aljucén occur (580 ± 3 Ma, Pb-Pb stepwise evaporation, unpublished data of the authors) and Mérida (575 ± 13 Ma, U-Pb ion microprobe, Bandrés et al., 2004).

Nonetheless, it seems doubtful whether the upper intercept of Lancelot et al., (1985) discordia represents the MDO crystallization age. First, because the MDO is a deformed crustal granitoid which generally would contain inherited zircon components. Second, because the MDO stands out only in this aspect. The rest of its field, petrographic and geochemical features are totally comparable to the Ordovician granitoids of the Ollo de Sapo Domain.

To determine the time span of the magmatism in the basement is crucial for understanding the pre-Variscan evolution of Central Iberian. The question is whether the current basement of the Variscan belt of Iberia was mainly built during the Cadomian, as it is currently believed, or during the lower Ordovician. This question has also been recently posed in SW France (Delaperrière and Respaut, 1995; Roger et al., 2004; Laumonier et al., 2004) and is of primary importance to understand the pre-Upper Ordovician evolution of the west European Variscan belt. For this reason, we revisited the geochronology of the MDO using U-Pb ion microprobe and LA-ICPMS spot analysis.

GEOLOGY AND PETROGRAPHY

The Mirando do Douro Orthogneiss is located between Portugal and Spain where the Douro river makes the boundary of both countries (Fig. 1). The orthogneiss is a NW-SE elongated small body, with a maximum length of ~12 km and maximum width of ~5 km, emplaced in the Schist-Greywacke Complex (Iglesias and Ribeiro, 1981). It is a mesocratic augen-gneiss, locally slightly migmatised, which is composed of quartz, zoned plagioclase crystals, abundant biotite and K-feldspar. The augen are formed by either K-feldpar or syneussis of plagioclase. As accessories it contains apatite, zircon, monazite, rare xenotime, ilmenite and occasional Fe sulfides. Under the microscope it has a foliated almost hypidiomorphic granular texture with no cataclasis and few strained grains, thus indicating it was ductile deformed. Myrmekites are locally abundant. Its chemical composition corresponds to a slightly peraluminous granodiorite, with $SiO_2 \approx$ 65-66%, CaO ≈ 2.8-3.0%, $Na_2O \approx$ 3.1-3.2%, $K_2O \approx$ 3.3-4.0% and an alumina saturation index ≈ 1.05-1.1.

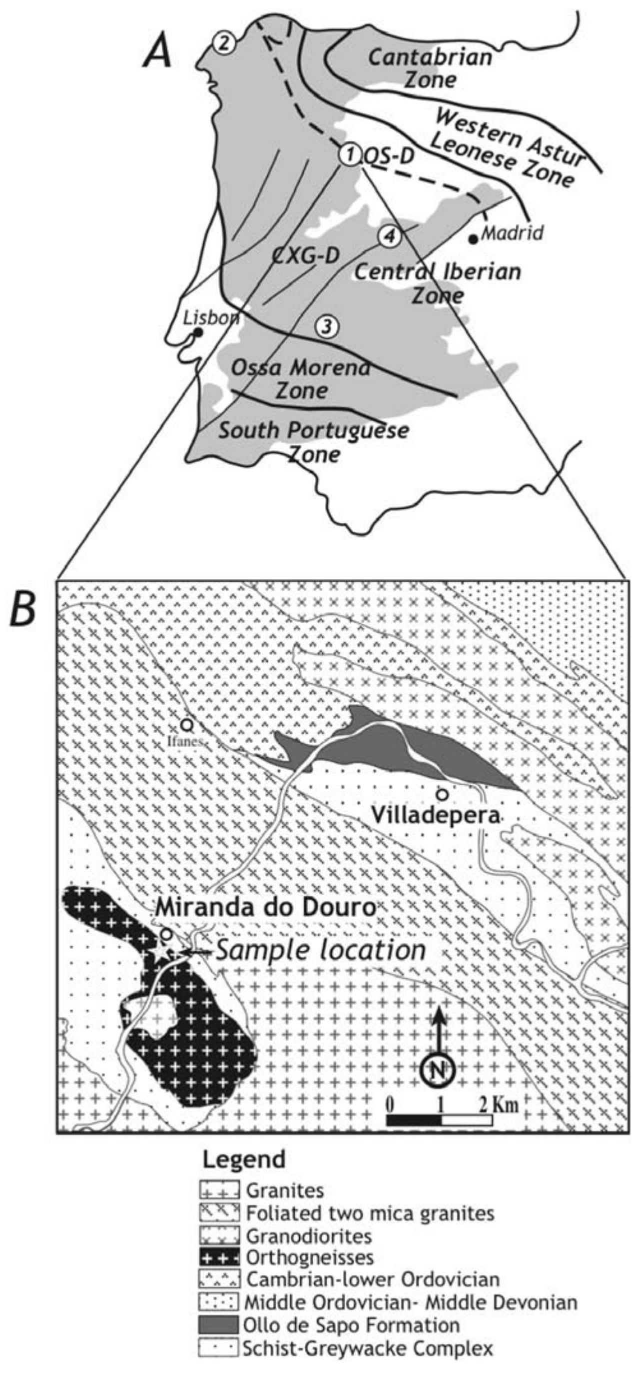

FIGURE 1 | **A) Scheme of the Iberian Massif (in grey) and position of the igneous bodies of the Central Iberian Zone previously dated as Vendien: 1: Miranda do Douro; 2: Sisargas granitic orthogneisses; 3: Aljucén and Mérida gabbro-dioritic units (3). Only the latter have been precisely dated. Miranda do Douro is Ordovician (this work). Probably Sisargas is also Ordovician (see text for further explanation). If so, the oldest granitoid in the Central Iberian Zone is the Almohalla Orthogneiss (4) dated at c.a. 543-546 Ma (Bea et al., 2003; Zeck et al., 2004). B) Geological scheme of the Miranda do Douro orthogneisses with location of samples. Modified from Iglesias and Ribeiro (1981).**

SAMPLES AND METHODS

About 15 kg of an exceptionally fresh sample of the orthogneiss outcropping near the Miranda do Douro village (UTM coordinates 29T easting 727,600 northing 4,596,900) was collected for zircon separation (Fig. 1B). Zircon was separated using conventional magnetic and heavy-liquid techniques. Once mounted and polished, zircon grains were studied by cathodoluminescence imaging under the SEM before ion-microprobe and laser ablation analyses.

Sixteen U-Th-Pb analysis were done on ten grains using a Cameca IMS-1270 ion microprobe at the Nordsim facility in Stockholm (Table 1). Analytical methods broadly follow those described by Whitehouse et al., (1999 and references therein). U/Pb and Th/Pb ratios were calibrated using the Geostandards 91500 reference zircon (1065 Ma; Wiedenbeck et al., 1995) and include a propagated error component from replicate analyses of 91500 during the analytical session. Errors on $^{207}Pb/^{206}Pb$ ratios are either the observed analytical uncertainly or the counting statistics error, whichever is highest.

Additional 30 U-Th-Pb analyses were carried out on 25 grains using a LA-ICPMS system at the University of Granada. Twelve of these analyses were useless because of excessive common-lead, probably contributed from the zircon substratum; the rest are also shown in Table 1. The LA-ICPMS system consisted of a torch-shielded quadrupole Agilent-7500 spectrometer and a 213 nanometers Nd-YAG Mercantek laser unit. Ablation was done in a He atmosphere with a 60 μm diameter laser beam and a repetition rate of 5 Hertzs. Spots were pre-ablated for 60 seconds with laser energy of 50%. Ablation was done for 90 seconds with laser energy of 75% moving the sample stage upwards 5 μm every 30 seconds. To minimize the U-Pb fractionation during ablation, the surface to be ablated was placed 0.20 mm below the laser focus. The glass NIST-610, which contains 409 ppm Pb and 460 ppm U (Pearce et al., 1997), was used as an external standard. The following isotope ratios, determined by TIMS of the University of Granada, were also used: $^{204}Pb/^{206}Pb$ = 0.06, $^{207}Pb/^{206}Pb$ = 0.9127, $^{208}Pb/^{206}Pb$ = 2.1898, $^{206}Pb/^{238}U$ = 0.2501, $^{208}Pb/^{232}Th$ = 0.5402. The coefficient of variation (100*standard deviation/average) on the 12 replicates of NIST-610 measured in the same session, was ± 2.4% for $^{206}Pb/^{238}U$ and ± 0.3% for $^{207}Pb/^{206}Pb$. The accuracy was estimated by comparing the results of analyzing the same population of very uniform grains from a diorite with the Nordsim (307 ± 3 Ma) and the LA-ICPMS (309 ± 9 Ma).

Common Pb corrections assume that most contaminant Pb is present on the surface of the analysed grains or in the resin, and has a composition that can be approximated using the Stacey and Kramers (1975) model for the present day. Table 1 presents the "207-corrected" ages which are calculated by projecting the uncorrected analysis onto concordia from the assumed common $^{207}Pb/^{206}Pb$ composition. All ages were calculated using the decay constant recommendations of Steiger and Jäger (1977).

RESULTS AND DISCUSSION

The results are shown in Table 1. Ion microprobe and LA-ICPMS data reveal the same picture (Fig. 2): two concordant or nearly-concordant populations are identified; the most abundant is located at ~480-485 Ma and the other at ~605-610 Ma (Fig. 3). There are also another younger and highly-discordant population at about ~350 Ma, and a few older discordant measurements that yield a discordia line (d1, Fig. 2) with an upper intercept at 3,167 +57/-48 Ma.

The ~480-485 Ma population is formed of a few grains which, according to their CL images, have no inherited cores (Fig 4, grains *z-10*, *z20* and *z-12*) and, more abundant, wide rims over older cores (Fig. 4, grains *z-16* and *z30*) that in both cases look magmatic. The ~605-610 Ma population comprises a few grains with uniform age (Fig. 4, grain *z-14*) or, more often, cores

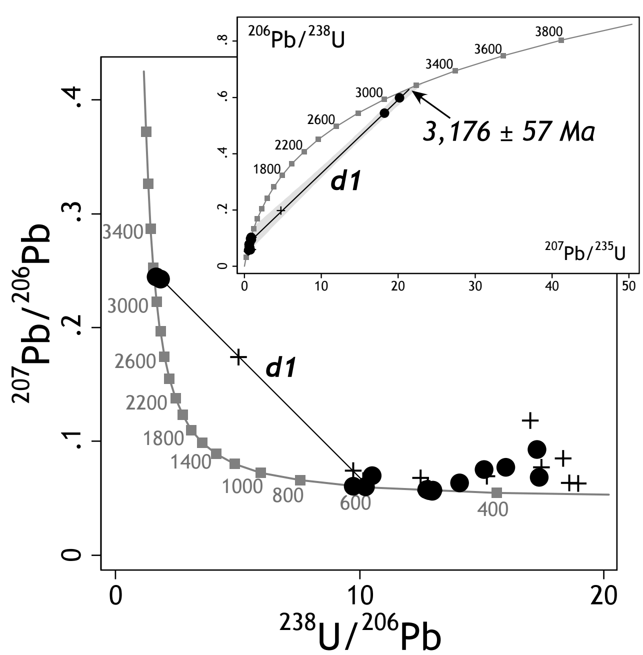

FIGURE 2 | **Tera-Wasserburg and conventional concordia-discordia plots. Dots represent ion-microprobe data. Crosses are LA-ICPMS data. See text for explanation.**

TABLE 1 | **U-Pb ion microprobe and LA-ICPMS data of zircon grains from the Miranda do Douro orthogneiss. For grains with same reference, the character "b" means rim and "c" means core.**

grain	Pb (ppm)	Th (ppm)	U (ppm)	$^{207}Pb/^{206}Pb$	$^{208}Pb/^{206}Pb$	$^{206}Pb/^{238}U$	$^{208}Pb/^{232}Th$	207 cor. age (Ma)	$^{206}Pb/^{238}U$ age (Ma)	$^{207}Pb/^{235}U$ age (Ma	$^{208}Pb/^{232}Th$ age(Ma)	$^{207}Pb/^{206}Pb$ age(Ma)
						Ion-microprobe data						
z-9c	95	68	1447	0.07744	0.01827	0.06254	0.02353	380± 5	391	519	470	1133
z-9b	23	41	332	0.07543	0.03313	0.06622	0.01719	403±5	413	532	345	1080
z-10c	25	23	293	0.05767	0.02615	0.07848	0.02530	487±6	487	492	505	517
z-10b	29	34	351	0.0566	0.02912	0.07747	0.02254	481±6	481	480	451	476
z-11	21	26	257	0.05623	0.03265	0.07708	0.02407	479±6	479	476	481	462
z-12	29	28	346	0.05666	0.02859	0.07781	0.0266	483±6	483	482	531	478
z-13b	89	44	1529	0.09305	0.00793	0.05794	0.01546	345±5	363	564	310	1489
z-13c	61	139	569	0.06985	0.07681	0.09521	0.02897	579±8	586	661	577	924
z-14	29	71	339	0.05743	0.06482	0.07689	0.02303	477±8	478	483	460	508
z-15b	39	75	636	0.06859	0.04092	0.0576	0.01934	354±5	361	442	387	887
z-15c	92	204	830	0.06003	0.07993	0.09777	0.03077	601±8	601	602	613	605
z-16b	87	828	808	0.05681	0.33410	0.07765	0.0245	482±6	482	482	489	484
z-16c	70	219	580	0.06078	0.11809	0.10259	0.03105	630±8	630	630	618	632
z-17	30	42	396	0.06372	0.03771	0.07095	0.02441	437±8	442	492	488	732
z-19c	398	77	516	0.24446	0.04459	0.59844	0.17307	2875	3024	3100	3226	3149
z-19b	94	53	128	0.24254	0.11533	0.54437	0.14674	2545	2802	3001	2768	3137
						Laser-Ablation ICPMS data						
z20	34	94	449	0.05891	0.06971	0.0777	0.02515	481±11	482	497	502	564
z21	202	2522	3182	0.08531	0.25968	0.05452	0.01729	329±8	342	503	347	1323
z22	146	894	2451	0.07731	0.12222	0.05735	0.01860	349±8	360	484	373	1129
z24	34	125	345	0.06029	0.10369	0.09813	0.02710	603±14	603	606	541	614
z25	55	141	530	0.07439	0.10053	0.10271	0.03765	619±15	630	731	747	1052
z26	56	201	563	0.06078	0.10609	0.09891	0.02847	608±14	608	613	567	632
z27	16	113	183	0.05875	0.22125	0.07842	0.02731	486±11	487	499	545	558
z28c	102	1601	645	0.06515	0.80141	0.09807	0.03065	599±14	603	642	610	779
z28b	67	1060	581	0.06042	0.63902	0.07822	0.02652	483±11	486	510	529	619
z29	171	841	2463	0.11856	0.24278	0.05885	0.04048	339±8	369	684	802	1935
z30b	46	48	643	0.05675	0.02288	0.07665	0.02340	476±11	476	477	468	482
z30c	133	101	582	0.17413	0.15312	0.19818	0.16871	1023±24	1166	1778	3151	2598
z31	28	175	407	0.06934	0.15759	0.06572	0.01976	403±10	410	495	396	909
z34	26	146	180	0.06557	0.59689	0.09865	0.07030	602±14	607	647	1373	793
z35	31	56	415	0.05694	0.04373	0.07875	0.02460	489±11	489	489	491	489
z37	42	608	384	0.06786	0.51806	0.07998	0.02533	489±12	496	567	506	864
z38b	36	397	629	.06321	0.17980	0.05276	0.01456	327±8	331	384	292	715
z38c	36	475	579	.06363	0.27547	0.05383	0.0175	334±8	338	393	351	729

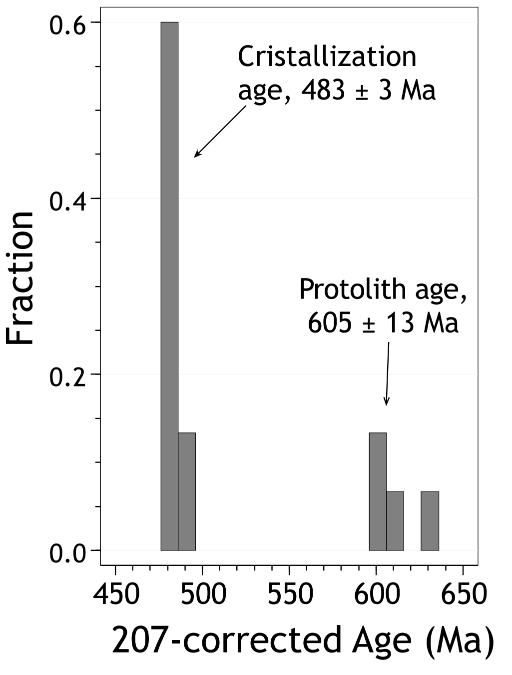

FIGURE 3 | **Distribution of nearly-concordant ($^{206}Pb/^{238}U$ age / $^{207}Pb/^{235}U$ age > 0.95) 207-corrected ages of the Miranda do Douro orthogneiss. The most abundant population, with an average of 483 ± 3 Ma represents the crystallization age (11 grains, 12 analyses). The 605 ± 13 Ma population is composed of inherited grains and represents the age of the source rock for the Ordovician magma (8 grains, 8 analyses).**

rimed by large ~480-485 Ma overgrowths (Fig. 4, grain *z-16*). They also look magmatic. Variscan ages are mostly found in narrow rims (Fig. 4, grain *z-13*), frequently narrower than the spot analysed by either the ion probe (~30 μm) or the laser (~60 μm). One of the three oldest discordant ages appears as a core rimmed by an Ordovician overgrowth (Fig. 4, grain *z30*) and the other two oldest ages are the rim and the core of a single grain (Fig. 4, grain *z-19*). The significance of these populations can be interpreted as follows:

The youngest, and always discordant, population yielded a 207-corrected age of 344 ± 14 Ma (95% conf. interval, calculated on 8 measurements made on 7 grains, Table 1), close to the peak of Variscan anatexis dated in the Peña Negra Complex (~332 Ma, Montero et al., 2004). It thus represent the Variscan imprint on the MDO, mesoscopically reflected by local migmatization.

The most abundant and highly concordant population (12 measurements on 11 grains, Table 1) yielded a precise age of 483 ± 3 Ma (95% conf. interval), which we suggest represents the crystallization age of the Miranda do Douro orthogneiss. Therefore, it is not Vendian, as proposed by Lancelot et al., (1985), but Ordovician, with the same age as the other orthogneisses spatially associated with the Ollo de Sapo.

The rest are inherited crystals. The well-defined magmatic-looking Vendian population yielded an age of 605 ±13 Ma (95% conf. interval, calculated on 8 measurements made on 8 grains, Table 1). This suggests that they were derived from the source rock of the MDO magma, which would therefore have consisted of a Pan-African igneous rock. The lack of preserved rocks of this age indicates that they have been actively recycled during the Cadomian and early-Caledonian times. The occurrence of ~600 Ma cores in zircon from early-Ordovician granites has also been found in the Variscan terrains of southern France (Laumonier et al., 2004).

The zircon grains along the discordia d1, with an upper intercept of ~3.17 Ga are, to the authors' knowledge, the oldest found so far in Iberia. Though it is difficult to determine their precise meaning, all evidence points to them representing relicts of the oldest crust reworked during the building of the pre-Variscan basement of Central Iberia.

Considering the age pattern of the MDO, it is not surprising that the analysis of zircon concentrates would produce a discordia such as the one found by Lancelot et al., (1985). In the light of single-crystal data, it seems clear

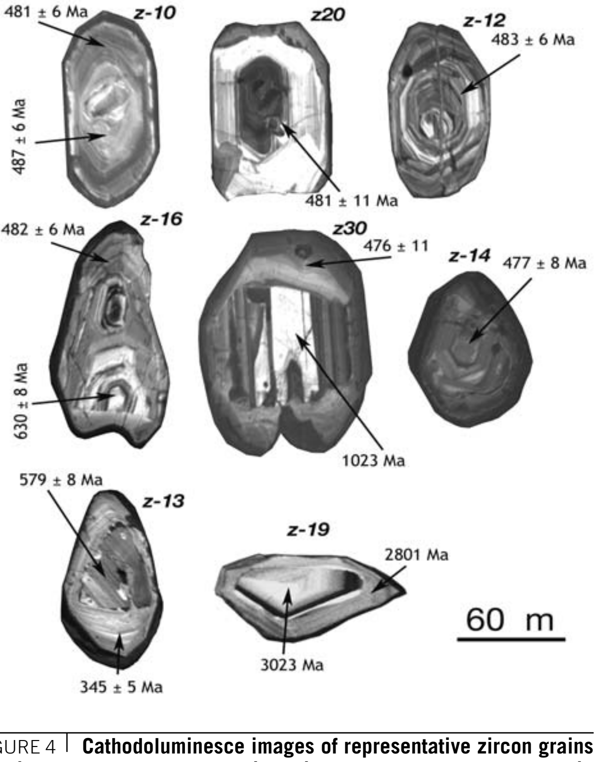

FIGURE 4 | **Cathodoluminesce images of representative zircon grains of Miranda do Douro orthogneiss. Zircon labels are the same as in Table 1. Note that ages are 207-corrected except those of the core of grain *z30* and the core and rim of grain *z-19*. These are $^{206}Pb/^{238}U$ ages and made a discordia with upper intercept at 3.17 Ga (Fig. 2).**

that its upper interception (610 Ma) actually reflects the ~605 Ma protolith-derived population instead of the crystallization age.

CONCLUSIONS

The Miranda do Douro orthogneiss is neither neo-Proterozoic nor the oldest magmatic rock of the Variscan belt of Iberia, as previously believed. It is Ordovician in age, with a crystallization age of 483 ± 3 Ma, very close to the rest of the orthogneisses spatially associated with the Ollo de Sapo Formation.

The Ordovician magma that formed the MDO was derived from a 605 ± 13 Ma igneous rock. Some zircon grains from the source were not dissolved in the magma and are currently found as a concordant relict population in the orthogneiss. This implies that a Pan-African protolith was actively involved in the Ordovician magmatism.

The Miranda do Douro orthogneiss also contains the oldest zircon grains so far found in Iberia, with a nearly concordant age of ~3.17 Ga. Further analytical works would be required to properly understand the meaning of these grains. However, we tentatively suggest that the zircon grains might record the involvement of an Archean crust in the Pan-African orogeny.

There is a Variscan imprint on all these populations at 344 ± 14 Ma, which reflects the extensive melting of the middle crust of central Iberia between ~350 Ma to ~295 Ma (Montero et al., 2004).

The U-Pb upper discordia intercept of the Sisargas orthogneiss (Allegret and Iglesias, 1987), considered the second oldest magmatic rock of Central Iberia does not seem to record the crystallization age, but the presence of inherited Pan-African zircon populations as in the MDO. Future single-crystal work will clear up this possibility. In the meantime, the oldest magmatic rock precisely dated in the middle and northern domains of the Central Iberian Zone is the Almohalla gneiss (543 ± 6 Ma, Bea et al., 2003; 546 ± 3, Zeck et al., 2004)

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