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Conventional and ion-microprobe U-Pb dating of detrital zircons of the Tentudía Group (Serie Negra, SW Spain): implications for zircon systematics, stratigraphy, tectonics and the Precambrian/Cambrian boundary

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Abstract. Conventional multi-grain and ion-microprobe dating of detrital zircons from a very low grade metagraywacke of the Tentudía Group (upper part of the Serie Negra, Ossa-Morena Zone, SW Spain) reveals an uppermost Vendian age for the deposition of the metagraywacke. The youngest detrital zircon grain provides a maximum depositional age of about 565 Ma. Thus, these data contradict earlier Middle to Upper Riphean (ca. 1350–850 Ma) estimates on the age of the Tentudía Group and favour a Precambrian/Cambrian boundary falling into the range of 540 to 530 Ma. The presence of about 20% of Pan-African detrital zircons ranging from about 700 to 550 Ma indicates the derivation from Gondwana. From the upper intercept ages of the fan-shaped data field defined by conventionally determined zircon fractions, it can be deduced that 2.1 Ga old zircons as well as Archean zircons existed in the provenance(s) of the Serie Negra sediments. This mixing of crustal components of different ages is in line with the Nd crustal residence age of 1.9 Ga. The latter value, as well as other model ages of the Iberian Massif, indicates unusually high amounts of ancient crust to be present in the strata. This is different to other (meta)sediments of the European Hercynides and suggests that the Iberian strata of uppermost Precambrian age may contain the detritus of more internal, older parts of Gondwana than other European strata of comparable ages. Geochemical data on the analysed sample and further metagraywackes of the Tentudía Group argue for a deposition in an arc environment. Such a scenario would conform with the syn- to post-orogenic shallow marine deposition of the studied sediments. Furthermore, an upper time limit for the pre-Lower Cambrian deformational history, including two phases of regional deformation, is given by the maximum age of deposition, implying a very short time interval for deposition and deformation of the Tentudía Group. Concerning the U-Pb systematics of detrital zircon fractions, it is probable that numerous, previously published conventional multi-grain zircon data on (meta)sedimentary rocks of the European Hercynides readily can be explained by the presence of up to 20% of Pan-African detrital zircons and later Phanerozoic lead loss during metamorphic transformation of the sedimentary protoliths. Moreover, this implies that such metasediments originated from post-Pan-African sedimentary precursors.

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Introduction

Although results of conventional multi-grain U-Pb analyses of zircon suites of detrital unmetamorphosed to very low grade metamorphosed sediments have been commonly available since the early 1970s (e.g. Michot and Deutsch 1970; Grauert et al. 1974; Gebauer and Grünenfelder 1977) their interpretation is still complex due to the often observed non-linear scatter of data points. Most of these investigations which included multi-grain as well as single-crystal dating dealt with the determination of provenance ages (e.g. Michard-Vitrac et al. 1977; Gaudette et al. 1981; Gariépy et al. 1984). Only little attention could be given to relatively younger components within the detrital zircon population, as post-crystallization modification of the U-Pb system and/or contribution of younger zircons to the detritus hardly could be distinguished using conventional multi-grain U-Pb dating techniques (Gebauer and Grünenfelder 1977; Hoegen et al. 1990). The use of the sensitive high resolution ion-microprobe technique (e.g. Compston et al. 1984), however, made it possible to get a better estimate for, or even to clearly define primary magmatic crystallization ages of detrital zircons. Based on ion-microprobe work on detrital zircons from Central Europe, a vast range of Pan-African to Early Archean primary ages for individual zircon grains has been detected (Gebauer et al. 1989) or at least can be inferred (Kröner et al. 1988). Thus, Gebauer et al. (1989) and Gebauer and Williams (1990) established the existence of four Precambrian megacycles of crustal growth within the European Hercynides: 2.7–2.5 Ga, 2.2–1.8 Ga, 1.2–0.9 Ga and 0.7–0.55 Ga with only a few pre-2.7 Ga outliers extending up to about 3.8 Ga.

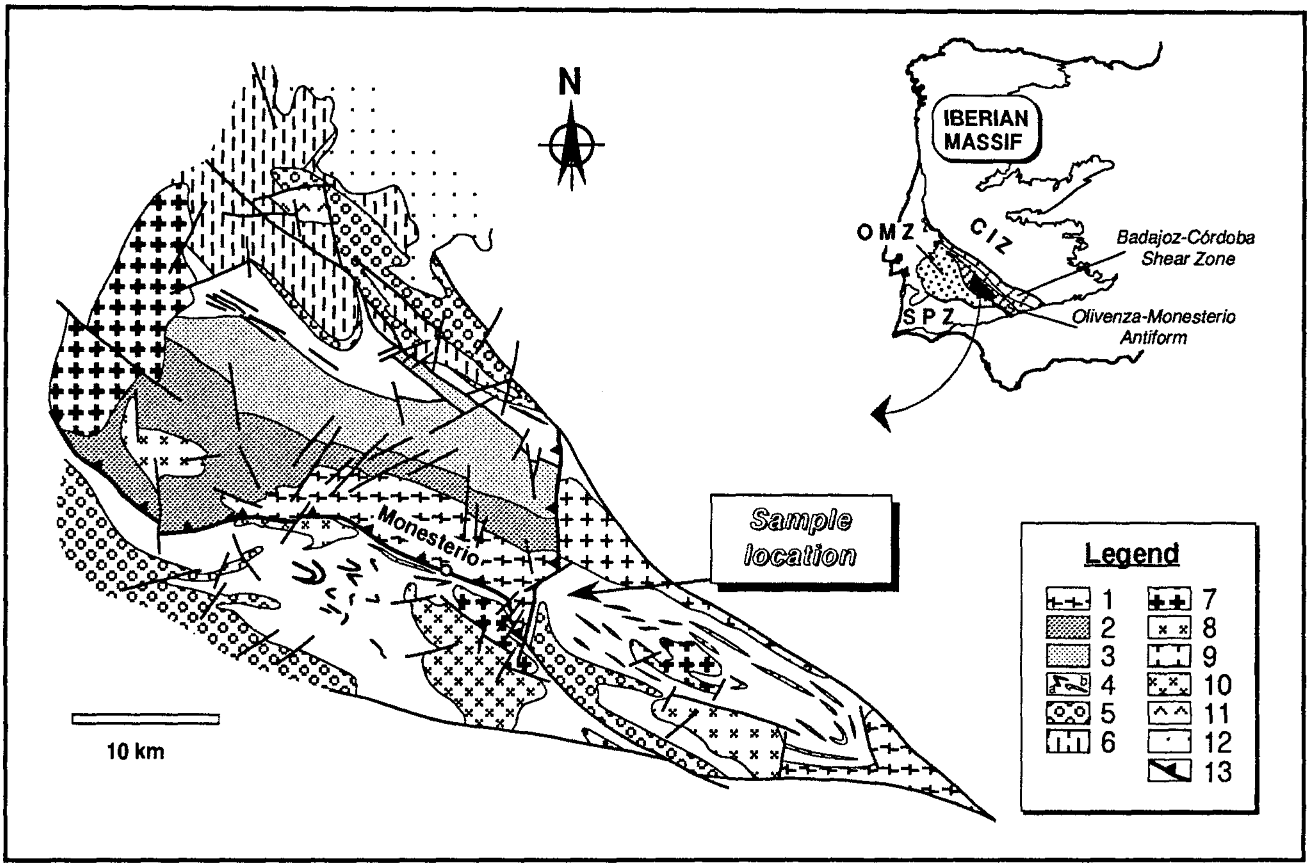

Fig. 1. Geological sketch map of the central Olivenza-Monesterio antiform (according to Eguiluz and Abalos 1992). On the right, the location of this area within the Ossa-Morena Zone and the Iberian Massif is shown (*CIZ*, Central Iberian-; *OMZ*, Ossa-Morena-; *SPZ*, South Portuguese Zone). Legend: *1-4*, Serie Negra: *1*, Monesterio migmatitic core (migmatites and granodiorites); *2* and *3*, medium and low grade areas, respectively (Montemolín Schists and Amphibolites); *4*, low grade areas (Tentudía Group) with intercalated, *a*, black quartzite and, *b*, metabasite layers; *5*, volcano-sedimentary, calc-alkaline Malcocinado Formation (northern part) and Bodonal-Cala Complex (southern part); *6*, Lower Cambrian; *7*, younger calc-alkaline granites; *8*, subvolcanic granites; *9*, older calc-alkaline granites (Pallares granodiorite); *10*, alkaline granites; *11*, mafic-ultramafic rocks; *12*, Tertiary; *13*, Monesterio thrust

From the largest of the numerous and usually strongly disrupted crystalline massifs of the European Hercynides, the existence of extensive Precambrian sedimentary basins was reported (e.g. Quesada 1990). Into one of these basins, located in the Ossa-Morena Zone (SW Spain; Fig. 1), the strata of the Tentudía Group were deposited. This succession is considered to be part of the pre-Pan-African (Cadomian; 700–550 Ma) series sensu lato within the Iberian Massif (Quesada 1990). Based on stratigraphic relationships (Arriola et al. 1984; Eguiluz 1987; Quesada 1990) and on scarce (and uncertain) acritarchs (Quesada et al. 1990) it has been considered to be Middle to Upper Riphean (ca. 1350–850 Ma) in age.

In this contribution we present conventional multi-grain analyses of morphologically characterized zircon types as well as ion-microprobe data for detrital zircons from a very low grade metagraywacke of the Tentudía Group. The principal objectives of this study are as follows: (1) to constrain the effect(s) of possible admixtures of young zircon components versus pre- and/or post-depositional lead loss from the relatively older zircon components; (2) to get detailed information on the provenance ages and especially on the age of deposition of the Tentudía Group; (3) to obtain a reliable reference point for the regional stratigraphy; (4) to further constrain similarities and/or differences in the geological evolution of Iberia when compared to studies of other parts of the European Hercynides which also use isotope-geochemical and geochemical data.

Geological outline

The Iberian Massif (Fig. 1) traditionally has been subdivided into various geological units differing in stratigraphic, structural, metamorphic and/or magmatic signature(s) (e.g. Lotze 1945). The extensive Precambrian sedimentary strata have been generally less metamorphosed during Pan-African (Cadomian), Caledonian and/or Hercynian regional metamorphism than similar sequences of Central Europe.

The Ossa-Morena Zone (OMZ, Fig. 1) comprises several tectonometamorphic units. Stratigraphic sequences range from Precambrian to Carboniferous but vary between and within distinct geological areas. Due to the mosaic of tectonically separated stripes and rhomb-shaped blocks, it is very difficult to outline the complex tectonometamorphic evolution of the OMZ. Besides the two high grade areas–the Monesterio migmatitic core and the Badajoz-Córdoba Shear Zone (Fig. 1)–the OMZ has been affected by very low to low grade metamorphism. According to various authors (e.g.

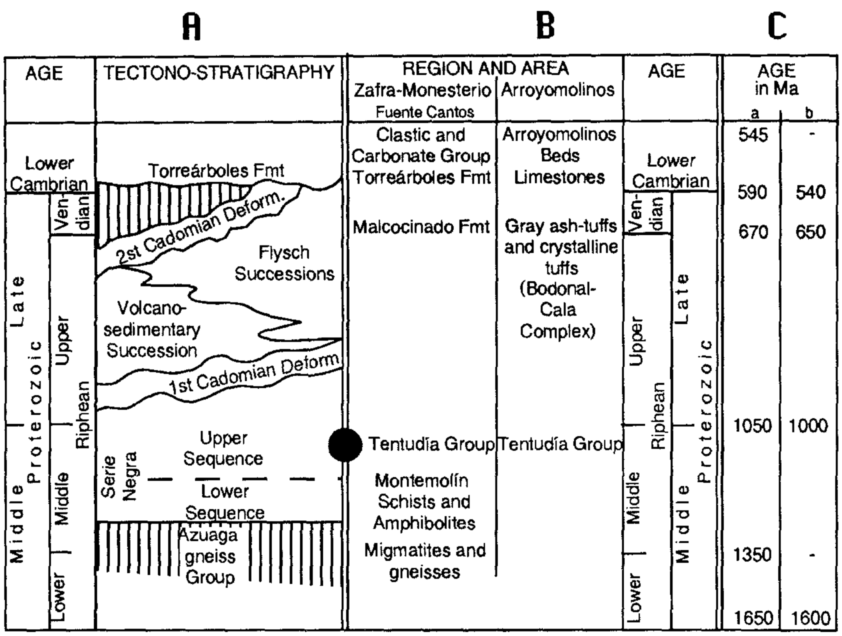

Fig. 2. Schematic tectono-stratigraphy and series sensu lato of two subunits of the central Ossa-Morena Zone: **A** according to Quesada 1990 **B** according to Arriola et al. 1984. The region Zafra-Monesterio (Arroyomolinos) is situated north (south) of the Monesterio thrust (see Fig. 1). *Circle* marks the Tentudía Group from which the sample was taken. **C** ages given in Ma refer to *a*, the "Geological time table" compiled by Haq and Eysinga (1987) and *b*, Odin and Odin (1990)

Chacón et al. 1983; Garrote et al. 1983; Herranz et al. 1986; Quesada 1986; Eguiluz 1987; Eguiluz and Abalos 1992) two Precambrian metamorphic events seem to be traceable which have not affected the lowermost Cambrian. Recent structural studies suggest the existence of two Precambrian (Pan-African) deformational events in addition to the Phanerozoic phases of deformation (Eguiluz and Ramón-Lluch 1983; Eguiluz et al. 1983; Eguiluz and Abalos 1992).

The Precambrian strata of the OMZ (Fig. 2) include at least four main successions from bottom to top: the Azuaga gneiss Group, the Montemolín Schists and Amphibolites, the Tentudía Group and the Malcocinado Formation. The latter two are of interest for this study: The Tentudía Group: the stratigraphically underlying series appear to pass gradually into this group of (1) an alternating monotonous shale and graywacke suite with some intercalations of black quartzites and carbonates; (2) various types of volcano-sedimentary rocks, (meta)tuffs and greenschists (amphibolites according to Eguiluz 1987) of variable thicknesses. For stratigraphic reasons, this sequence is supposed to be of Middle to Upper Riphean age (ca. 1350–850 Ma; Arriola et al. 1984; Eguiluz 1987; Quesada 1990).

The Malcocinado Formation: an angular unconformity is assumed to define the boundary to this calc-alkaline formation, supposed to be of Upper Riphean to Vendian age (Arriola et al. 1984; Eguiluz 1987; Sánchez-Carretero et al. 1989). The latter authors interpret this succession as volcanic products of an active continental margin related to a SW directed subduction. The complex sequence consists of andesitic tuffs, ash tuffs, limestones, conglomerates and serpentinite bodies. Based on geochemical data and lithostratigraphic affinities, its formation is genetically linked to the Bodonal-Cala Complex (Figs. 1, 2) of the Arroyomolinos area south of the Monesterio thrust (Hernández-Enrile 1971; Sánchez-Carretero et al. 1989).

The Precambrian sequences are overlain unconformably by the Lower Paleozoic Torreárboles Formation: an erosive discontinuity between the Precambrian and the Paleozoic successions as well as evidence of a Lower Cambrian fauna are reported by Liñán et al. (1984).

The metagraywacke sample

For the present study, a fine-grained, very low grade metagraywacke was sampled outside the contact-metamorphic aureole of the Pallares granodiorite (Fig. 1) and close to the basal rocks of the Bodonal-Cala Complex. According to the interpretation of Eguiluz et al. (1983) the analysed metagraywacke belongs to the uppermost part of the 500–3000 m thick Tentudía sequence.

The sampled dark grey rock is composed of layers of mainly graywacke and, subordinated, shale on the mm-scale. A slaty cleavage (D1) is crenulated by two later phases (D2 and D3) and therefore, in some cases no evidence for the original layering has been preserved. The deformations D1 and D2 are supposed to be Late Precambrian in age whereas D3 (to D5) is thought to be of Hercynian age (Eguiluz and Abalos 1992).

Mineralogically, the sample consists of fragments of quartz showing undulose extinction, feldspar and rock particles. The matrix makes up around 30% of the total rock volume and is composed of chlorite, white mica, quartz and feldspar whereas opaques, graphite and zircon occur as accessory minerals.

Analytical techniques

Zircons were separated using a Wilfley table, a Carpco magnetic drum separator, a Frantz isodynamic separator and heavy liquids (methylene-iodide and Clerici-solution). Ultimate selection of zircons was carried out by handpicking according to colour and morphology. The dissolution of zircons and the extraction of Pb and U followed the methods described by Krogh (1973); the overall analytical Pb blank was 60 pg.

All isotopic measurements were performed on a Finnigan MAT 261 equipped with 9 fixed Faraday cups and a secondary electron multiplier. Elements were loaded on Re-filaments and measured as metal species. Pb was run with silica-gel and H_3PO_4 on single, U was run with HNO_3 on double filaments. Based on NBS standard runs, correction factors for mass-dependent isotopic discrimination per atomic mass unit (AMU) for U = 0.031 ± 0.025% (2σ standard deviation) and for Pb = 0.092 ± 0.035% (2σ standard deviation) were determined. The overall reproducibility was tested with a laboratory internal zircon standard ($n = 4$) and is better than 0.7% for the Pb/U and 0.3% for the $^{207}Pb/^{206}Pb$ ratios (2σ standard deviation). Blank corrections using the measured unspiked blank Pb composition as well as an initial common lead correction according to Stacey and Kramers (1975) were applied. Concentrations of U and Pb were determined using a mixed $^{205}Pb/^{235}U$ spike. The 2σ standard deviations for the individual grain fractions were calculated following the error propagation routine reported by Roddick (1987). Decay constants conform to the values given by Steiger and Jäger (1977).

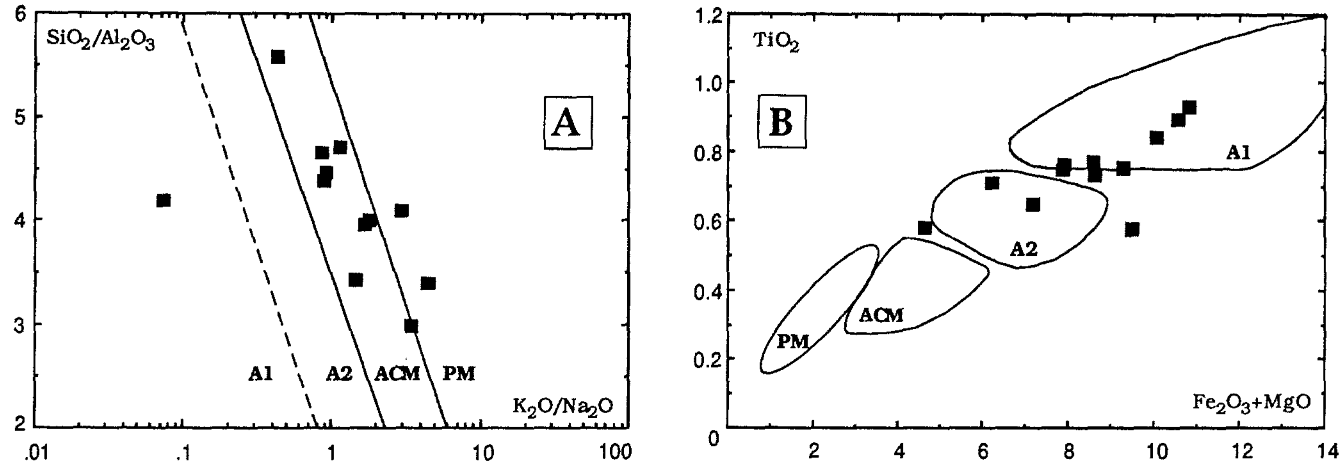

Fig. 3 A, B. Tectonic discriminant plots based on major element composition for metagraywackes from the Tentudía Group: **A** SiO_2/Al_2O_3 versus K_2O/Na_2O (Roser and Korsch 1986); **B** TiO_2 versus Fe_2O_{3total} + MgO (Bhatia 1983). For discussion, see text. (*PM*, passive margin; *ACM*, active continental margin; *A2*, continental island arc; *A1*, oceanic island arc)

Details concerning the analytical techniques used for Rb-Sr and Sm-Nd are given by Nägler et al. (1992). For a full description of the ion-microprobe technique we refer to Compston et al. (1984, 1986). All U-Pb ages are referenced to a $^{206}Pb/^{238}U$ value of 0.0928 (equivalent to 572 Ma) for the zircon standard SL13. Major and trace element composition were analysed by X-ray fluorescence spectrometry at the EMPA in Dübendorf and the 'Institut für Mineralogie und Petrographie' in Zürich.

Isotope-geochemical and geochemical results

The Sm-Nd and Rb-Sr whole-rock isotopic data for the metagraywacke are given in Table 1. The Nd crustal residence age of 1.90 Ga is within the range of mean values obtained for the Iberian crust (Ossa-Morena Zone 1.82 Ga (n = 13), Central Iberian Zone 1.92 Ga (n = 21) and Cantabrian Zone 1.96 Ga (n = 15); Nägler 1990). However, according to Nägler (1990), the mean age of the pre-Pan-African Iberian crust is in the order of 2.0 to 2.3 Ga which corresponds to an $\varepsilon_{Nd(T=550\ Ma)}$ of about $-$ 10. To meet the observed $\varepsilon_{Nd(T=550\ Ma)}$ of $-$ 6.7 of the Tentudía metagraywacke, an addition of juvenile Pan-African crust of 20% or more – depending on the assumed Nd composition of the mantle source – is necessary. Similarly, Nägler (1990) deduced an addition of about 30% of juvenile Pan-African crust to Precambrian syn-orogenic sediments of the Central Iberian Zone. These values conform with estimates based on the observed zircon systematics as well as crustal growth rates derived from zircons of different (meta)sediments of the European Hercynides (Gebauer and Williams 1990).

The very low initial $^{87}Sr/^{86}Sr$ isotopic composition of 0.69933 at 550 Ma clearly indicates that the whole-rock system must have been disturbed after deposition, i.e. probably during the Hercynian phases of metamorphism and deformation (D3–D5).

Tectonic discriminant diagrams (Bhatia 1983; Roser and Korsch 1986) based on major element compositions (Table 2 and including data from Eguiluz 1987) suggest deposition at an active continental margin (Fig. 3a) or in an arc environment (Fig. 3b). Due to the metamorphic overprint(s) of the samples, Na and K may well have been mobile. Furthermore, increasing amounts of fine-grained members, especially illite, may also influence the K_2O/Na_2O ratios towards higher values. Therefore, it seems that samples plotting within or near the passive margin field of Fig. 3a do not reflect their original tectonic setting. More indicative for the geotectonic setting of the Tentudía Group might thus be the correlation of less mobile elements, e.g. TiO_2 versus Fe_2O_{3total} + MgO. In

Table 1. Sm-Nd and Rb-Sr isotopic data of a very low grade metagraywacke (PAL-2), Tentudía Group

Sample	Sm (ppm)	Nd (ppm)	$^{147}Sm/^{144}Nd$	$^{143}Nd/^{144}Nd$ [a, b]	$\varepsilon_{Nd(T=550)}$ [c]	T_{DM} (in Ma)[d]
Total rock	4.63	22.7	0.1232	0.512031 ± 8	− 6.7	1901
Sample	Rb (ppm)	Sr (ppm)	$^{87}Rb/^{86}Sr$	$^{87}Sr/^{86}Sr$ [a]	initial$_{(T=550)}$ $^{87}Sr/^{86}Sr$	
Total rock	74.9	72.6	2.99	0.722764 ± 47	0.699326	

[a] Uncertainties (2σ standard error) refer to the last significant digits of the corresponding ratios
[b] Normalized to $^{146}Nd/^{144}Nd = 0.7219$
[c] $\varepsilon_{Nd(T)}$ values denote derivation from CHUR in parts per 10^4 at the time T (De Paolo and Wasserburg 1976). $^{143}Nd/^{144}Nd_{Chur\ today} = 0.512638$, $^{147}Sm/^{144}Nd_{Chur\ today} = 0.1967$ (Jacobsen and Wasserburg 1980)
[d] Depleted mantle model ages (T_{DM}) are calculated according to a linear depleted mantle evolution model (Goldstein et al. 1984)

Table 2. Major element data of a very low grade metagraywacke (PAL-2), Tentudía Group

Major elements (weight percent)		
SiO_2	64.48	58.97–72.21
TiO_2	0.58	0.58–0.93
Al_2O_3	14.62	12.95–19.73
Fe_2O_3 [b]	6.93	3.27–7.40
MnO	0.06	0.02–0.08
MgO	2.56	1.39–3.56
CaO	0.52	0.04–0.40
Na_2O	2.51	0.77–5.96
K_2O	2.31	0.44–3.78
P_2O_5	0.09	0.08–0.21
Cr_2O_3	0.03	–
NiO	0.00	–
LOI	4.19	–
Total	98.88	–

[a] Range of XRF-analyses of 11 metagraywackes, Tentudía Group from Eguiluz (1987)
[b] Measured as Fe_{total}
LOI, loss on ignition at 1000 °C

this diagram the samples plot roughly within both arc fields (A1 and A2 in Fig. 3b). An arc setting is consistent with previous interpretations of Eguiluz (1987) that this group represents a sedimentary sequence which was formed within a proximal shallow water environment and of Quesada (1990) that the Tentudía sediments are synorogenic depositions into a foreland or back-arc basin.

Conventional multi-grain U-Pb zircon dating

The typically detrital zircon population consists of mechanically rounded crystals with pitted surfaces. Usually the grains are not translucent and range from colourless via light-brown to reddish whereby zircons of brownish colour are most common. The light-brown grains show some of the best preserved prism faces and their mean length/width ratio is around 1.8–2.0. The reddish zircons are mechanically more rounded which is indicated by their lower length/width ratio of about 1.3–1.5.

Nine grain-size fractions classified according to colour and habit were analysed (Table 3, Fig. 4): two well rounded colourless zircon fractions, a "total" fraction containing all colours and habits, three fractions (150 μm to < 53 μm) consisting of reddish zircons and three fractions (150 μm to < 53 μm) of light-brown, sometimes slightly subhedral zircons.

The detrital zircon fractions do not form a linear array in a conventional Concordia diagram. The data points of the well rounded zircons (indices 1, 2 in Fig. 4) yield high $^{207}Pb/^{235}U$ and $^{206}Pb/^{238}U$ apparent ages, followed by reddish, rounded zircons (4, 5, 6) with U contents of 382–616 ppm and by light-brown zircons (7, 8, 9) with lower U contents (186–479 ppm). The least rounded zircons reveal the lowest $^{207}Pb/^{235}U$ and $^{206}Pb/^{238}U$ apparent ages. All data points plot in a fan-shaped field bounded by two discordia lines of 550 Ma to 2.1 Ga and 550 Ma to 2.5 Ga, respectively.

Similar fan-shaped data fields within a Concordia diagram were described by Gebauer and Grünenfelder (1977) for zircons from metasediments of Central Europe. According to these authors, the individual detrital zircon populations typically show an apparent lead loss of about 80–95% during Phanerozoic metamorphism(s). However, when compared to these data from the literature, zircon fractions 1, 2 and 4 of the Tentudía sample (Fig. 4) show far higher $^{207}Pb/^{235}U$ and $^{206}Pb/^{238}U$ apparent ages. This implies that at least these three fractions did not suffer a comparably strong, apparent lead loss due to Phanerozoic metamorphism. The fraction "75–58 μm colourless" shows a relatively high U content and is one of the smaller size fractions, i.e. it has a relatively large surface to volume ratio. Thus, when compared with the other grain-size fractions this fraction is one of the most suitable candidates for a post-magmatic lead loss. However, from the position of the data points in the Concordia plot (index 2, Fig. 4) this fraction is by far the least discordant and may have lost at most 17% of radiogenic lead e.g. 420 Ma ago, the age of a medium- to high-grade

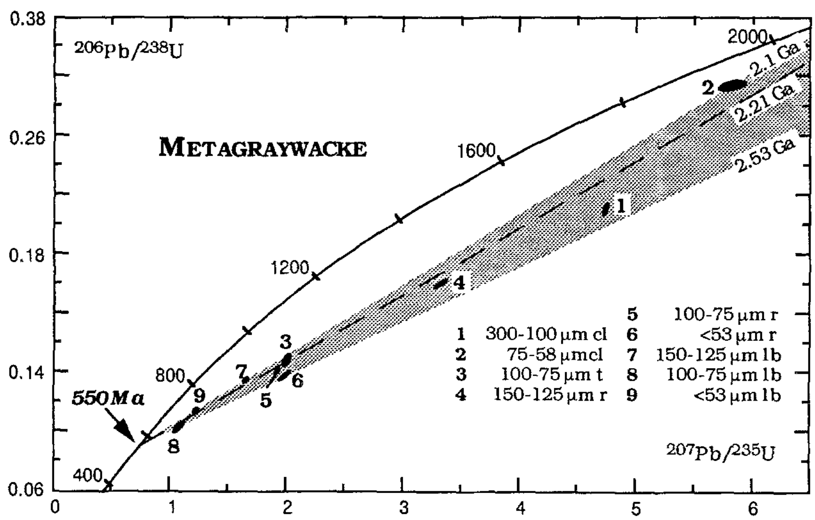

Fig. 4. Concordia diagram with conventional multi-grain U-Pb data obtained on size fractions of different detrital zircon populations extracted from a very low grade metagraywacke (PAL-2), Tentudia Group. The data points plot within a typical fan-shaped field known from other pre-Caledonian metasediments of the European Hercynides. The *dashed line* represents a reference line from 550 Ma through the analysed total fraction (index 3). *Error ellipses* correspond to the 95% confidence level. (*cl*, colourless; *r*, reddish; *lb*, light-brown; *t*, total fraction)

Table 3. Conventional multi-grain U-Pb isotopic data of detrital zircons of a very low grade metagraywacke (PAL-2), Tentudía Group

No.	Fraction (μm)	Weight (mg)	Concentration (ppm) U	Pb*	Pb^{co}	$^{206}Pb/^{204}Pb$ [a]	Isotopic ratios[b,c] $^{206}Pb^*/^{238}U$	$^{207}Pb^*/^{235}U$	$^{207}Pb^*/^{206}Pb^*$
1	300-100, cl	0.41	200	55.3	18.2	168	0.2496 ± 38	4.748 ± 12	0.13799 ± 246
2	75-58, cl	0.34	554	201.2	35.4	306	0.3331 ± 26	5.825 ± 89	0.12682 ± 145
3	100-75, t	1.25	393	61.0	0.8	3979	0.1472 ± 28	2.016 ± 28	0.09931 ± 137
4	150-125, r	0.69	382	81.1	2.6	1621	0.1992 ± 21	3.329 ± 39	0.12120 ± 29
5	100-75, r	1.73	455	67.3	0.7	4983	0.1395 ± 9	1.929 ± 13	0.10027 ± 13
6	< 53, r	1.02	616	88.2	1.1	4201	0.1368 ± 23	1.998 ± 35	0.10588 ± 17
7	150-125, lb	0.80	186	26.7	0.7	2068	0.1338 ± 7	1.663 ± 11	0.09017 ± 32
8	100-75, lb	1.29	479	53.3	1.8	1580	0.1023 ± 27	1.086 ± 32	0.07698 ± 70
9	< 53, lb	0.90	394	48.1	1.0	2444	0.1135 ± 3	1.229 ± 4	0.07852 ± 10

cl, colourless; r, reddish; lb, light-brown zircons; t, total zircon fraction; *, radiogenic Pb; co, Common Pb
[a] Measured $^{206}Pb/^{204}Pb$ corrected for mass discrimination and spike contribution
[b] Corrected for blank and common lead
[c] Standard deviation (2σ level) refers to the last significant digits of the corresponding ratios and reflects the calculated analytical error according to Roddick (1987)
[d] Correlation coefficients of $^{206}Pb/^{238}U$ versus $^{207}Pb/^{235}U$

Table 3 (continued)

Apparent ages (Ma)[c] $^{206}Pb^*/^{238}U$	$^{207}Pb^*/^{235}U$	$^{207}Pb^*/^{206}Pb^*$	Correlation coefficient [d]
1436 ± 19	1776 ± 21	2202 ± 31	0.723
1854 ± 13	1950 ± 13	2054 ± 20	0.685
886 ± 16	1121 ± 9	1611 ± 26	0.693
1171 ± 12	1488 ± 9	1974 ± 4	0.982
842 ± 5	1091 ± 4	1629 ± 3	0.980
827 ± 13	1115 ± 12	1730 ± 3	0.996
810 ± 4	995 ± 4	1429 ± 7	0.863
628 ± 16	747 ± 16	1121 ± 18	0.953
693 ± 2	814 ± 2	1160 ± 3	0.927

metamorphism in the northern adjacent Badajoz-Córdoba Shear Zone (Schäfer 1990; Schäfer et al. 1991). More probably, however, it lost much less lead and the weak degree of discordance of this fraction is due rather to the admixture of younger zircons. Therefore, the data pattern in Fig. 4, reflecting differences in colour and habit as well as U and Pb concentrations in the zircons, rather indicates the existence of concordant and variably discordant zircons of different ages derived from different geological sources. This deduction is also indicated by ion-microprobe data on this rock and on other (meta)sediments of the European Hercynides (Gebauer et al. 1989).

From the position of data points within the Concordia diagram (Fig. 4) it is likely that some zircons are derived from source rocks with average ages ranging between 2.1 and 2.5 Ga, i.e. Middle and Lower Proterozoic continental crust. The position of the data points of the coarsest, well rounded fraction (index 1) in particular suggests that the existence of Archean rocks in the respective provenances is probable. The present positions of the data points probably result from mixing of Archean and Proterozoic–including Pan-African–zircons. A relatively high contribution of zircons crystallized during the Pan-African orogeny is especially likely in fractions 8 and 9 of Fig. 4.

Assuming the distribution of data points to be the result of mixing of zircons of different ages and variable discordancy rather than resulting from lead loss during metamorphism, the lower 'age' (550 Ma) of the fan-shaped field is likely to reflect a maximum age for the deposition of the metagraywacke.

Ion-microprobe U-Pb zircon dating

To test the probable existence of Pan-African zircons, seven selected grains of size fraction "100-75 μm light-brown" (index 8 in Fig. 4) have been analysed by ion-microprobe using the SHRIMP at the ANU in Canberra. The U-Th-Pb data are given in Table 4.

The results of seven spots obtained on seven different grains show a range of Th (71–465 ppm) and U (140–345 ppm) concentrations and Th/U ratios (2.38–0.32). Compared to the corresponding bulk fraction (479 ppm U), the lower contents observed on the analysed spots of the seven single grains are probably due to the

Table 4. Ion-microprobe isotopic data of single zircons (grain-size fraction "100–75 μm light-brown") of a very low grade metagraywacke (PAL-2), Tentudía Group

Grain	Concentration (ppm) U	Th	Pb*	Th/U	$\frac{^{206}Pb}{^{204}Pb}$ [a]	Isotopic ratios[b] $\frac{^{208}Pb^*}{^{206}Pb^*}$	$\frac{^{208}Pb^*}{^{232}Th}$	$\frac{^{206}Pb^*}{^{238}U}$	$\frac{^{207}Pb^*}{^{235}U}$	$\frac{^{207}Pb^*}{^{206}Pb^*}$	Apparent ages (Ma)[b] $\frac{^{208}Pb^*}{^{232}Th}$	$\frac{^{206}Pb^*}{^{238}U}$	$\frac{^{207}Pb^*}{^{235}U}$	$\frac{^{207}Pb^*}{^{206}Pb^*}$
43	195	465	30	2.38	419	0.7107 ± 96	0.0287 ± 9	0.0979 ± 27	1.005 ± 97	0.0745 ± 66	571 ± 17	602 ± 16	706 ± 49	1054 ± 180
44	345	110	31	0.32	1274	0.0916 ± 33	0.0263 ± 12	0.0914 ± 25	0.758 ± 29	0.0602 ± 15	525 ± 24	564 ± 15	573 ± 17	610 ± 53
45	287	201	37	0.70	1611	0.2011 ± 39	0.0328 ± 11	0.1151 ± 31	1.026 ± 44	0.0646 ± 19	651 ± 22	702 ± 18	717 ± 22	762 ± 62
47	160	124	18	0.80	718	0.2373 ± 57	0.0301 ± 11	0.0990 ± 28	0.810 ± 47	0.0594 ± 28	599 ± 22	608 ± 16	603 ± 26	581 ± 104
50	140	71	15	0.50	667	0.1543 ± 48	0.0321 ± 14	0.1053 ± 29	0.846 ± 43	0.0583 ± 23	639 ± 27	645 ± 17	623 ± 23	542 ± 85
51	219	129	24	0.59	282	0.1677 ± 61	0.0288 ± 13	0.1024 ± 28	0.906 ± 50	0.0642 ± 28	575 ± 26	628 ± 16	655 ± 26	747 ± 93
54	226	275	28	1.22	1828	0.3836 ± 59	0.0315 ± 10	0.0995 ± 27	0.744 ± 51	0.0543 ± 33	627 ± 20	611 ± 16	565 ± 29	382 ± 135

* Radiogenic Pb
[a] Measured $^{206}Pb/^{204}Pb$
[b] Standard deviation (1σ level) refers to the last significant digits of the corresponding ratios

Table 4 (continued)

fact that the relatively larger grains were chosen for ion-microprobe dating and indicate that the bulk fraction includes smaller-sized zircons with higher U concentrations. This is supported by the next largest size fraction, "150–125 μm light-brown" (Table 3), yielding an average U content of only 186 ppm and thus falling below the mean U content of the seven analysed spots.

To test for possible Pb loss and differential movement of Th and U, radiogenic $^{208}Pb/^{206}Pb$ ratios were plotted versus $^{232}Th/^{238}U$ ratios (Fig. 5). When compared to the relatively small time interval covered by the analysed zircons, the very large half-lives of ^{232}Th and ^{238}U make this plot insensitive to age. Within their 2σ errors all points but one fall on a regression line, indicating that there has been no fractionation of Th and U and no differential movement of radiogenic ^{208}Pb and ^{206}Pb. The datum point 54 lies very slightly above this line. The corresponding grain reveals the highest $^{206}Pb/^{204}Pb$ ratio; there is no significant increase in the U content relative to the other zircons (Table 4) and its $^{206}Pb/^{238}U$ age coincides with those of the main group. Thus, again there are no indications for Pb loss and the slight offset of grain 54 is probably due to post-crystallization Th/U fractionation. Generally, absence of lead loss is also typically observed if spots are located in magmatic domains which are undisturbed in their pattern of growth zoning (Gebauer, unpublished).

Five out of seven zircon spot analyses are concordant within their 1σ error (Fig. 6). One of the remaining two spots (grain 54) is also concordant within the 2σ errors. It seems that grain 43 includes an inherited lead component as the $^{207}Pb/^{206}Pb$ age of 1054 ± 180 Ma (Table 4) is outside the 1σ range observed for all other grains. In fact, the position of the 20–30 μm spot as given on the cathodo-luminescence picture of grain 43 (Fig. 7) suggests that parts of an inner core and parts of the outer rim were analysed simultaneously.

The obtained ages are interpreted to reflect primary magmatic ages of the youngest mother rocks in the source region of the Tentudía sediments. This interpretation is based on the fact that all ages were obtained on spots

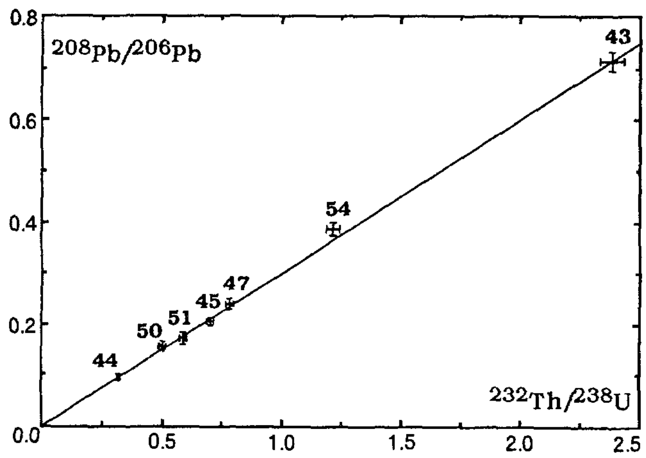

Fig. 5. Radiogenic $^{208}Pb/^{206}Pb$ versus $^{232}Th/^{238}U$ plot for single spot analyses of detrital zircons from fraction "100–75μm light-brown" of a very low grade metagraywacke (PAL-2), Tentudía Group. The fit of data implies that the grains have seen no differential movements of Th and U or of ^{208}Pb and ^{206}Pb

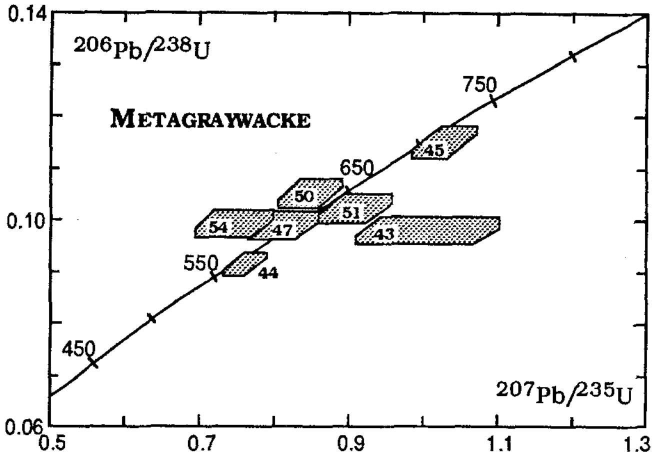

Fig. 6. $^{206}Pb/^{238}U$ versus $^{207}Pb/^{235}U$ plot for single spot analyses of detrital zircons from fraction "100–75 μm light-brown" (index 8 in Fig. 4) of a very low grade metagraywacke (PAL-2), Tentudía Group. *Boxes* correspond to the 1σ error

which were, according to their cathodo-luminescence pattern, located within the comagmatic, multiply and euhedrally zoned and therefore concordant part of the individual zircon crystal.

An onset of the Pan-African magmatic cycle may be represented by the oldest observed age of ~700 Ma ($^{206}Pb/^{238}U$, grain 45), followed by a major orogenic phase around 645 to 600 Ma. Five $^{206}Pb/^{238}U$ ages of single spots are within this range which is also known by conventional multi-grain U-Pb zircon data to be a phase of igneous activity in this region (Schäfer 1990). The youngest primary U-Pb age at ~565 Ma ($^{206}Pb/^{238}U$ age, grain 44) is probably related to magma formation at the end of the Pan-African cycle.

Similarly, all seven $^{208}Pb/^{232}Th$ ages (~650–525 Ma; Table 4) also fall into the range of typical Pan-African ages. Thus, the Th-Pb ages serve as a good mutual control and support the obtained $^{206}Pb/^{238}U$ ages. Furthermore, as all data fall into the Pan-African age range, there can be hardly any doubt that these youngest analysed zircons are derived from Gondwana.

Discussion

Mixing of crustal components

Gebauer and Williams (1990) proposed that around 50% of the continental crust present at the Precambrian/Cambrian border existed already at the end of the Archean (2.5 Ga), whereby around 40% of the continental crust was formed in the time span of 2.7 Ga to 2.5 Ga. This might explain that for conventional multi-grain zircon dating of metasedimentary rocks upper intercept ages older than 2.7 Ga are unlikely and ages clustering around 2.0 Ga are frequent within the European Hercynides. Obviously, the presence of Proterozoic zircons around 2.0 Ga, 1.0 Ga and to a lesser degree at 0.6 Ga (Gebauer and Williams 1990) are responsible for upper intercept

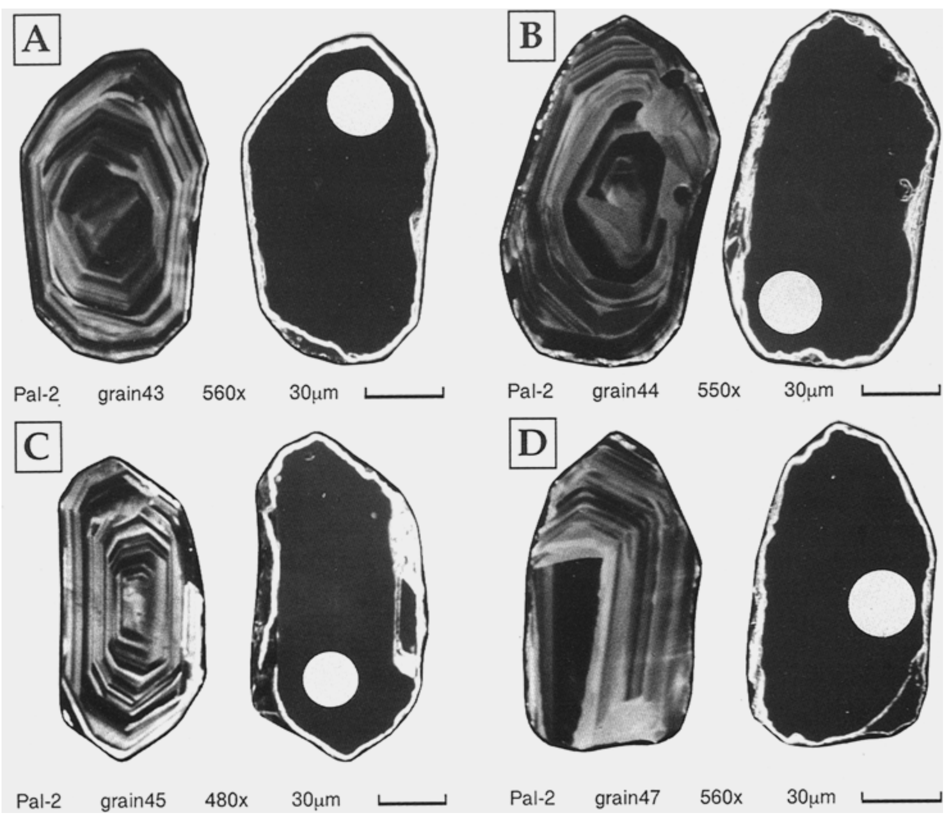

Fig. 7A–D. Cathodo-luminescence, *left*, and secondary electron, *right*, pictures of zircons from fraction "100–75 μm light-brown" of a very low grade metagraywacke (PAL-2), Tentudía Group: **A** grain 43; **B** grain 44; **C** grain 45; **D** grain 47. *White circles* indicate the location of around 20–30 μm big single spots. Note that spots were set near to the rim of the oscillatory, magmatically zoned zircons to obtain crystallization ages without analysing inherited cores or core-like structures ages that mask the presence of large amounts of mainly Late Archean zircons. Furthermore, failure to observe and/or regard the youngest zircon components yield(s) too high average ages for the whole zircon population. In our example, these youngest components are reflected by the lower intercept ages (multi-grain method) and by the primary magmatic zircon ages using the ion-microprobe technique.

Mainly for the reasons given above, Nd crustal residence ages are on the average about 500 Ma younger than the corresponding conventionally determined upper U-Pb intercept ages of zircons from the same rock type (e.g. Kröner et al. 1988; Liew and Hofmann 1988; Schäfer 1990; Peucat et al. 1990). Based on ion-microprobe work, Gebauer and Williams (1990) deduced four Precambrian megacycles of crustal growth within the European Hercynides. During the period of 3.8 to 2.7 Ga about 10% of continental crust accumulated, followed by approximately 40% at 2.55 Ga, 15% at 2.0 Ga, 17% at 1.0 Ga and 18% at 0.6 Ga (Gebauer, unpublished). Taking these ages and considering a mixing of exactly these proportions to form a sediment at around 550 Ma, the respective theoretical Nd crustal residence ages would be around 1.9 Ga, perfectly in line with the observed one. In the case of the Tentudia metagraywacke, the average upper intercept age of the fan-shaped zircon data field is at about 2.3 Ga, i.e. about 400 Ma older than its Nd crustal residence age.

Provenance and depositional age of the metagraywacke

In the case of the studied metagraywacke the isotope-geochemical data as well as the U-Pb zircon data probably reflect mixing of variably old Archean with Proterozoic crustal components. Although the systematics of these data are in good agreement with those of conventional multi-grain and ion-microprobe dating of detrital zircons of the Moldanubian Zone and the Montagne Noire (Gebauer et al. 1989) it can be suggested – from detrital zircons as well as from Nd crustal residence ages (Nägler 1990; Schäfer 1990) – that the quantity of Archean crust involved is higher than for other parts of the European Hercynides. This might be due to the presence of detritus from more internal fragments closer to the nucleus of the supercontinent Gondwana.

At the time of deposition of the Tentudía Group, the mixing process(es) involved relatively recent material formed during the Pan-African orogeny, as demonstrated by the obtained primary magmatic Pan-African zircon ages. The assumption – based on conventional multi-grain U-Pb dating techniques – that the lower 'age' of the fan-shaped data field of around 550 Ma reflects a maximum age for deposition was confirmed by the ion-microprobe data. The obtained 564 ± 30 Ma primary age ($^{206}Pb/^{238}U$) of grain 44, or its maximum $^{208}Pb/^{232}Th$ age of 525 ± 48 Ma (2σ errors), provides approximate minimum ages for Late Pan-African magmatic activities. Further, as this grain is itself mechanically rounded and therefore was involved in a transport cycle, the maximum age for deposition of this grain must still be younger than 564 ± 30 or 525 ± 48 Ma.

Stratigraphy and tectonics

The Late Pan-African, i.e. Vendian maximum depositional age of 564 ± 30 Ma (or 525 ± 48 Ma) for the studied very low grade metagraywacke of the Tentudía Group requires a drastic revision of the previously assumed pre-Pan-African, Middle to Upper Riphean depositional age (ca. 1350–850 Ma according to the "Geological time table" compiled by Haq and Eysinga (1987)).

South of the Monesterio thrust (Fig. 1), the Tentudía Group appears to be overlain unconformably by the Bodonal-Cala Complex. Contrasting interpretations concerning the nature of the contact between these series are given: Ruiz López et al. (1985) assumed a tectonic contact between the Tentudía Group and the Bodonal-Cala Complex running parallel to the regional structures, whereas Eguiluz et al. (1983) and Eguiluz and Abalos (1992) have documented an angular/cartographic discontinuity between the two series which they interpreted as the consequence of a stratigraphic unconformity. According to Quesada et al. (1990), the Tentudía Group is affected by two Precambrian deformational events in contrast to the apparently overlying Bodonal-Cala Complex which shows only one stage of Precambrian deformation. Thus, assuming stratigraphic contacts between the two series, the first regional deformation must be post-564 ± 30 Ma (or 525 ± 48 Ma), the maximum depositional age for the upper part of the Tentudía Group as derived in this study. On the other hand, it must be older than the deposition of the calc-alkaline volcano-sedimentary Bodonal-Cala Complex which generally is thought to be uppermost Precambrian. The second Precambrian deformational event must have followed immediately on the first one before the onset of Lower Cambrian deposition. These two Precambrian phases of deformation and metamorphism have not been detected within the Cambrian strata. It follows that there was a very short time interval between deposition and deformation of both the Tentudia Group and the overlying Bodonal-Cala Complex and the Precambrian/Cambrian boundary.

The Precambrian/Cambrian boundary

At present, the age of the Precambrian/Cambrian boundary is rather uncertain. Whereas Harland et al. (1982) and Palmer (1983) estimated it to be at 570 Ma, Haq and Eysinga (1987) proposed an age of 590 Ma even for the base of the Cambrian. Odin (1986) and Odin and Odin (1990), however, favoured the Precambrian/Cambrian boundary to be at 530 ± 10 and 540 Ma, respectively, in line with several U-Pb zircon ages on rocks related to the problem of this boundary (Ducrot and Lancelot 1977; Compston et al. in Cowie and Johnston 1985; Dunning in Benus 1987; Gebauer et al. 1989; Compston et al. 1990).

From the Ossa-Morena Zone, Liñán and Mergl (1982) and Liñán et al. (1984) reported the occurrence of Lower Cambrian "zero level" trilobites, brachiopods and archaeocyathans in the Pedroche formation and numerous trace fossil taxa and burrows of presumed Early Cambrian age in the Torreárboles Formation. Thus, they assumed the Precambrian/Cambrian boundary to be at the base of the

Torreárboles Formation which is in turn interpreted to overlie unconformably the Malcocinado Formation and/or the Tentudía Group. Within the southern part, the Bodonal-Cala Complex is overlain unconformably by the Arroyomolinos succession (Eguiluz 1987) with fossiliferous Lower Cambrian. Therefore, the 564 ± 30 Ma (or 525 ± 48 Ma) maximum age of deposition is in line with the younger estimates for the Precambrian/Cambrian boundary.

Conclusions

1. Detrital zircons in metasedimentary rocks as well as crustal residence ages document a complex and long history of the basement of Iberia. The continental crust is clearly part of Gondwana which is implied by the existence of Pan-African rocks and their frequent erosion products.
2. Zircons extracted from a metagraywacke (Tentudía Group, Serie Negra) are derived from source rocks with apparent average ages ranging between 2.1 and 2.5 Ga. Thus, the existence of Archean rocks is also probable in the respective provenances. Pan-African detritus is present in the sample as suggested by conventional multi-grain zircon dating and as clearly demonstrated by ion-microprobe analyses on zircons which have formed within the time interval of around 700 to 550 Ma.
3. Numerous previously published conventional multi-grain zircon data on (meta)sedimentary rocks of the European Hercynides readily can be explained by the presence of up to 20% of Pan-African detrital zircons. This adds to the hypothesis that the continental crust of the European Hercynides consists of Gondwana fragments.
4. The amount of juvenile mantle-derived Pan-African crustal components is estimated to be in the range of around 20% or more. Thus, the quantity of Proterozoic and Archean continental crust is relatively high which conforms with results from U-Pb dating. Most likely an arc environment is the geotectonic scenario in which mixing took place, in line with the geological record.
5. Results from both U-Pb dating techniques (conventional multi-grain and ion-microprobe) indicate a maximum depositional age of less than 564 ± 30 Ma (or 525 ± 48 Ma). These results confirm that the Tentudía Group belongs to the Precambrian strata of the Iberian Massif but is much younger than previously thought. Furthermore, this yields a lower limit for Pan-African deformational and metamorphic phases and favours an age of 540 to 530 Ma for the base of the Cambrian.

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