

The significance of early Permian and early Carboniferous U-Pb zircon ages in the Bossòst and Lys-Caillaouas granitoids (Pyrenean Axial Zone)

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The Axial Zone of the Alpine Pyrenees exposes Ediacaran-Palaeozoic metasediments and orthogneisses intruded by abundant Variscan granitoids that resulted in elongated HT metamorphic domes separated by tight synforms with low-grade metasediments. Yet, the age of the granitoids within the metamorphic domes remains debated, hindering the correlation of Variscan deformation stages and the HT metamorphism between the different domes. Here, we dated two granitoids located within classical metamorphic domes using U-Pb LA-ICP-MS zircon geochronology: the Bossòst and the Lys-Caillaouas granitoids in the Garona and Lys-Caillaouas domes, respectively. The results indicate that the leucogranitic facies of both granitoids have Early Permian crystallization ages at 295 ± 2 Ma and 297 ± 2 Ma, respectively. Furthermore, we found a small population of inherited zircon with Serpukhovian (331–323 Ma) ages in the Lys-Caillaouas granitoid, reinforcing the existence of an elusive but widespread early Carboniferous magmatic event in the Pyrenees. These ages discard two previous hypotheses explaining the age disparity observed in the different domes: either that the HT-LP metamorphism is of Visean age only or that the dome formation was diachronous along the Axial Zone, ranging from Visean in the central part to late Carboniferous in the eastern Axial Zone. The early Permian age found in the leucogranitic facies of the Bossòst granitoid questions the proposed Visean age for the formation of the Garona dome and the peak HT-LP metamorphism and points to an Early Permian age as most likely in line with what is observed in the Lys-Caillaouas Massif.

KEYWORDS

granitoids, HT-LP metamorphism, metamorphic domes, Pyrenees, U-Pb age, Variscan

1 | INTRODUCTION

Isotopic dating in granitoids alongside their relationship with the host rocks are key to constraining the age of deformation stages in metamorphic terranes (Paterson & Tobisch, 1988; Paterson, Tobisch, & Vernon, 1989). In the Pyrenees mountain range, a kilometre-scale Alpine antiformal stack exposes at its core the basement, often referred to as the Pyrenean Axial Zone (Figure 1). This zone is composed by Ediacaran-Palaeozoic metasediments and orthogneisses intruded by a

large number of Variscan granitoids. Structurally, the Axial Zone is characterized by a polyphase Variscan deformation that resulted in elongated HT metamorphic domes that follow the trend of the Pyrenean belt separated by tight synforms with low-grade metasediments (e.g., Carreras & Capella, 1994). Despite the large number of tectonometamorphic studies since the 1960s, two main problems remain unsolved: how and when the metamorphic domes were formed.

The Variscan deformation in the western and southern parts of the Axial Zone is characterized by folds associated with south-directed

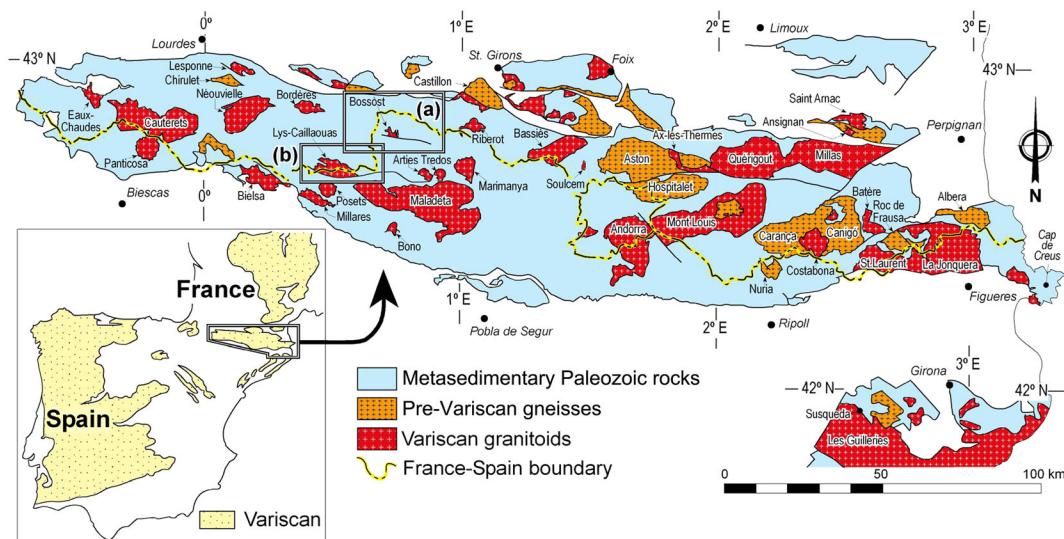


FIGURE 1 Geological map of the Axial Zone of the Pyrenees showing the location of the Bossòst (a) and Lys-Caillaouas (b) metamorphic domes. In the bottom right, the map of the Catalonian Coastal Ranges [Colour figure can be viewed at wileyonlinelibrary.com]

thrusts and a low grade or lack of metamorphism. The domes reveal the deepest levels of the Variscan crust in which median to high-grade metasediments, orthogneisses, and granitoids outcrop. Three main foliations characterize the Variscan deformation. The first two are related to folds and thrusts developed at low-grade conditions, whereas the third is related to shear zones developed under LP-HT conditions (García-Sansegundo, Poblet, Alonso, & Clariana, 2011). The Alpine deformation has minor effects in the Variscan structure and includes thrusts with a general top-to-the-south movement, producing a local change of attitude and tightening of Variscan folds (e.g., Gutiérrez-Medina, Alonso, & García-Sansegundo, 2012; Mey, 1968), and the local development of foliations in the absence of metamorphism and magmatism.

The LP-HT metamorphism is restricted to the metamorphic domes and has been attributed to the intrusion of the so-called deep-seated granitoids (e.g., Carreras & Capella, 1994). The main feature of such granitoids is the lack of well-defined narrow contact aureoles, outcropping within wide areas where staurolite, cordierite, and/or andalusite assemblages and migmatization appear (e.g., Autran, Fonteilles, & Guitard, 1970; Mezger & Passchier, 2003; Mezger & Regnier, 2016). Representative examples are the Cap de Creus pegmatites, granitoids of Canigó-Carança Massif, and the Lys-Caillaouas, Ax-les-Thermes, Soulcen, and Bossòst granitoids (Figure 1).

Among all the different tectonic models previously proposed for the origin of the domes (e.g., Carreras & Capella, 1994 and references therein), two main opposing views persist nowadays. Some authors propose that the domes, the flat-lying schistosity, and HT-LP metamorphism formed under an extensional regime, although there is disagreement about the timing of this event during the Variscan cycle (e.g., Aerden, 1994; Kriegsman, Aerden, Bakker, Denbrok, & Schutjens, 1989; Mezger & Gerdes, 2016; Mezger & Passchier, 2003; Van Den Eeckhout & Zwart, 1988; Vissers, 1992). Others propose a dextral transpressive regime during the dome formation (Carreras & Capella, 1994; Esteban et al., 2015). The authors supporting a transpressive regime usually cite as evidence the attitude of magnetic fabrics and

mineral lineation in the granites, which are arranged subhorizontal and slightly oblique to the Variscan trend in the Axial Zone. However, the oblique and subhorizontal arrangement of structures has also been related to extensional detachments in other parts of the Variscan orogen in Western Europe (e.g., Lopez-Sánchez, Marcos, Martínez, Iriondo, & Llana-Fúnez, 2015 and references therein), making this criterion ambiguous. Lastly, some authors have taken an intermediate position on this matter, stating that some domes would have been formed due to a local extension in a broad transpressive regime (Denèle et al., 2014; Mezger, 2009).

Despite these significant differences, there is also consensus on some of the features that characterize the domes. For example,

- (i) the dome-shaped structure was essentially formed during the Variscan cycle, although some domes could be accentuated by Alpine tectonics;
- (ii) the formation of flat-lying schistosities was coeval with the development of HT-LP metamorphism; and
- (iii) the development of a HT-LP metamorphism and migmatization was coeval with the emplacement of the so-called deep-seated granitoids.

Due to the aforementioned features, there is a lot of interest in dating the plutons linked with the HT-LP metamorphism. This will allow correlating the metamorphism and structures across the different metamorphic domes and ultimately help to understand their origin. Unfortunately, most of these “deep-seated” granitoids lack reliable dating because they contain many inherited zircon grains (e.g., Denèle et al., 2014). Indeed, the “deep-seated” granitoids have provided two well-differentiated isotopic ages using U-Pb zircon/monazite dating:

- (i) Late Carboniferous ages. An age of 306 ± 2 Ma (U-Pb LA-ICP-MS) found by Denèle et al. (2014) in the Ax-les-Thermes granitoid (Aston dome), and another of 300 ± 2 Ma (U-Pb SIMS)

- SHRIMP) by Esteban et al. (2015) in two facies of the Lys-Caillaouas granitoid (Lys-Caillaouas Massif).
- (ii) Early Carboniferous (Visean) ages of ca. 337–339 Ma (U-Pb LA-ICP-MS) found by Mezger and Gerdes (2016) in the Saulcen (Aston dome) and different facies of the Bossòst granitoids (Garona Dome).

The disagreement in the age of emplacement of these granitoids is odd since it is always related to similar deformation and metamorphic events along the Axial Zone (Denèle et al., 2014; Hilario, 2004; Mezger & Passchier, 2003; Mezger & Gerdes, 2016). The late Carboniferous ages agree with the well-known late- to post-orogenic magmatism observed through the entire Variscan belt and well documented in the Pyrenees (Denèle et al., 2014; Druguet, Castro, Chichorro, Francisco Pereira, & Fernandez, 2014; Evans, Gleizes, Leblanc, & Bouchez, 1998; Gleizes, Crevon, Asrat, & Barbey, 2006; Martínez et al., 2016; Maurel, Respaut, Monie, Arnaud, & Brunel, 2004; Paquette, Gleizes, Leblanc, & Bouchez, 1997; Pereira et al., 2014; Roberts, Pin, Clemens, & Paquette, 2000; Romer & Soler, 1995). Zircon and monazite grains with early Carboniferous ages have also been found in some granitoids in the Pyrenees (Denèle, 2007; Denèle et al., 2014; Mezger & Gerdes, 2016), but the significance of these ages is still controversial. For example, Denèle et al. (2014) concluded that zircon grains with early Carboniferous ages in the Ax-les-Thermes granitoid are artefacts due to mixing ages and only late Carboniferous and pre-Silurian ages have geological significance. However, the zircon age clusters found by Mezger and Gerdes (2016) in the Bossòst and Saulcen granitoids are tight and formed by many zircon grains (>10), being the probability of finding such tight clusters by chance negligible. Accordingly, we think that the mixing age hypothesis proposed by Denèle et al. (2014) cannot be generalized for the entire Axial Zone and that some zircon grains truly record an early Carboniferous magmatic event.

It should be also added that zircon and monazite grains with early Carboniferous ages (ca. 320 Ma), considered either as xenocryst (i.e., inherited crystals) or as syn-magmatic, have been found in other metamorphic domes in the adjacent Variscan massifs (e.g., Gutiérrez-Alonso, Fernández-Suárez, López-Carmona, & Gärtner, 2018; López-Moro et al., 2018; Martínez et al., 2016; Martínez, Reche, & Iriondo, 2008; Poilvet, Poujol, Pitra, Van Den Driessche, & Paquette, 2011). A particularly well-documented case is the Tormes Dome in the Iberian Massif, where the limited overprint of the widespread late Carboniferous–early Permian magmatism allowing to recognize several igneous rocks with an unequivocal early Carboniferous age (López-Moro et al., 2018). Overall, these examples support the existence of an early Carboniferous magmatic event in some areas during Variscan times. Its effect, however, seems to be obscured due to the overprinting of the widespread late Carboniferous–early Permian magmatic event.

With the current state of affairs, two interpretations are possible in relation to the timing of the formation of the metamorphic domes in the Axial Zone:

- (i) Domes formed at different stages of the Variscan cycle and cannot be correlated in time despite showing a similar sequence of tectonic and metamorphic events.

- (ii) All the domes and related metamorphism formed contemporarily, and the geological meaning of one of the two different Variscan zircon ages is misinterpreted.

To shed some light on this matter, we dated the Bossòst and the Lys-Caillaouas granitoids, both located in different domes with similar structural relations with the host rocks but, strikingly, different emplacement ages. In both, the samples were collected in facies that have not been previously dated.

2 | GEOLOGICAL SETTING

The Bossòst and Lys-Caillaouas granitoids outcrop in the central part of the Pyrenees, the Bossòst granitoid in the southern part of the Garona Dome and the Lys-Caillaouas granitoid farther south, in the Lys-Caillaouas Massif (Figures 1 and 2). Both granitoids emplaced in a Cambro-Ordovician sequence, the Jujols Series (Cavet, 1957), composed by quartzite, slates and interbedded limestones, and outcrop within areas of HT-LP metamorphism (García-Sansegundo & Alonso, 1989; García-Sansegundo, Martín-Izard, & Gavaldà, 2014; Kriegsman et al., 1989). Overlying these series, there are late Ordovician slates, sandstones, conglomerates and limestone, and Silurian bituminous slates (García-Sansegundo, Gavaldà, & Alonso, 2004). Between both domes, the Aran Valley Synclinorium exposes more than 1,000 m of slates, limestones, sandstones, and quartzites of Devonian age (García-López, García-Sansegundo, & Arbizu, 1991; García-Sansegundo, 1992; Kleinsmiede, 1960), which is locally affected by the HT-LP metamorphism (Figure 2).

2.1 | Structure and metamorphism of the host rocks

Four main Pre-Alpine deformation phases affect the metamorphic rocks of the Garona and Lys-Caillaouas metamorphic domes:

- A pre-Variscan south-verging cleavage (S_E) with no folds associated. This foliation appears solely in the Cambro-Ordovician rocks that lie under the upper Ordovician unconformity and has been linked to/with the development of extensional pre-Silurian faults in the area (Casas, 2010; García-Sansegundo et al., 2004; García-Sansegundo et al., 2011; García-Sansegundo et al., 2014).
- D1 structures consisting of north-verging E-W recumbent or inclined folds with an associated horizontal foliation (S_1). These structures are best developed in the metamorphic domes (Garona and Lys-Caillaouas massifs), being S_1 the dominant foliation, and fade out toward shallower levels in the Aran Valley Synclinorium (García-Sansegundo, 1992, 1996; García-Sansegundo et al., 2014; García-Sansegundo & Alonso, 1989; Kriegsman et al., 1989). The foliation S_1 appears as a slaty cleavage in the Upper Ordovician-Devonian sequence and as a crenulation cleavage in the places where the previous S_E foliation developed (Clariana & García-Sansegundo, 2009; García-Sansegundo et al., 2011).
- D2 structures consist of south-directed thrusts with associated E-W upright folds affecting D1 structures. The thrusts are detached on Silurian shales and the Cambro-Ordovician sequence.

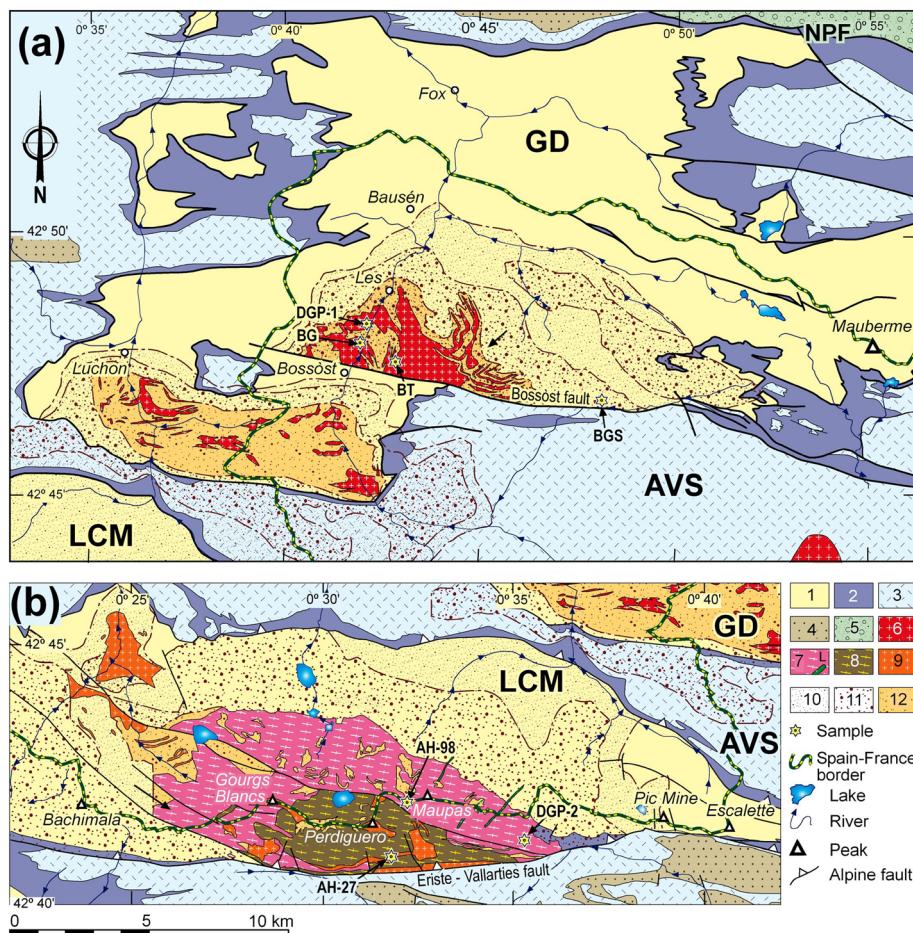


FIGURE 2 Detailed maps and sample locations. (a) Geological map of the Garona dome with the location sample DGP-1 (this study) and samples BG, BT, and BGS of Mezger and Gerdes (2016). (b) Geological map of the Lys Caillaous Massif with the location of sample DGP-2 (this study) and samples AH-27 and AH-98 of Esteban et al. (2015). NPF: North-Pyrenean Fault; GD: Garona Dome; AVS: Aran Valley Synclinorium; LCM: Lys-Caillaous Massif; 1: Cambro-Ordovician rocks; 2: Silurian shales; 3: Devonian series; 4: syn-orogenic Carboniferous series (Culm); 5: Mesozoic cover. Bossost granitoid; 6: Leucogranites and pegmatites. Lys-Caillaous granitoid; 7: porphyritic granodiorite (L, lamprophyre); 8: quartz-diorite; 9: leucogranite. Metamorphic zones, 10: Biotite zone; 11: Andalusite-Staurolite-Cordierite zone; 12 Sillimanite zone. Geological maps modified from Clin et al. (1986), García-Sansegundo (1992), García-Sansegundo and Ramírez Merino (2013), and Kriegsman et al. (1989) [Colour figure can be viewed at wileyonlinelibrary.com]

Folds with a pervasive crenulation lineation dominate below the detachment level placed in the Silurian shales, while a crenulation cleavage (S_2) dominates above this detachment in the Aran Valley Synclinorium (García-Sansegundo, 1990, 1992, 1996; Matte, 1969).

- D3 structures correspond to shear zones with variable thickness (3–5 km) developed around the deep-seated igneous bodies. The foliation (S_3) associated lies almost parallel to the S_1 , making its distinction difficult. Based on a detailed porphyroblast-matrix relation study, Mezger and Passchier (2003) correlated in a single event the development of D3 extensional shear zones, the intrusion of the Bossost granitoid, and the peak of the LP-HT metamorphism in the Garona Dome.

Two large faults with an E-W direction limit the dated granitoids to the south, the Bossost Fault in the Garona dome and the Eriste-Vallarties Fault in the Lys-Caillaous Massif (Figure 2). Both faults are interpreted to have started their tectonic history during the development of D2 structures (García-Sansegundo, 1992; Hilario,

2004; Pérez Cáceres & García-Sansegundo, 2012). However, both granitoids contain mylonites near these faults, indicating that they were also active after the granite crystallization, even during the Alpine cycle.

The emplacement of the Lys-Caillaous and Bossost granitoids are spatially related to a LP-HT metamorphism (M2) that overprints a previous greenschist- to amphibolite-facies metamorphism (M1). In both domes, the LP-HT metamorphism developed around the intrusions, where it can be recognized a prograde zoning with biotite, staurolite-andalusite-cordierite, and sillimanite-cordierite towards the contact with the granitoids (Clin, 1964; Kriegsman et al., 1989; Zwart, 1963, 1979). The granitoids never cut the LP-HT metamorphic zonation (i.e., the isograds). The absence of garnet and the abundance of cordierite are typical. In the Lys-Caillaous Massif, staurolite is absent due to a high Mg/(Fe + Mg) ratio in the host rocks (Kriegsman et al., 1989). In the Garona Dome, the growth of staurolite overprints the S_1 and S_2 foliations, while andalusite and cordierite grew during the development of D3 extensional shear zones (Mezger & Passchier, 2003).

3 | SHAPE AND PREVIOUS AGES IN THE BOSSÒST AND LYS-CAILLAOUAS GRANITOIDS

The Bossòst granitoid (Figure 2a) mainly consists of granites, leucogranites, and pegmatites arranged subparallel to the dominant S_1 foliation (Figure 2a). Small pegmatitic sills and veins outcrop bordering the main body, whereas the granite appears homogeneous in the central part. The granites are fine- to medium-grained, although locally they appear coarse-grained along with an increase in K-feldspar content (Figure 3a). The major constituents are quartz, albite, K-feldspar, muscovite, and, sometimes, biotite. Apatite, garnet, fibrolite, and zircon appear as accessory minerals. Cordierite, tourmaline, and beryl occur occasionally. Mezger and Gerdes (2016) obtained the following Laser Ablation U-Pb zircon ages: 338 ± 3 Ma ($n = 11$) in the muscovite granite, 337 ± 2 Ma ($n = 27$) in a granite sill, and 329 ± 4 Ma ($n = 4$) in the tonalitic

facies (samples BG, BGS, and BT in Figure 2a, respectively). They consider the first two as the age of crystallization.

The Lys-Caillaouas granitoid outcrops in the southern part of the Lys Caillaouas Massif, with the Eriste-Vallartés Fault cutting across the granitoid southwards (Figure 2b). The granitoid consists of three main facies (Clin, 1964; Clin et al., 1986): (i) porphyritic granodiorites that occur as lenticular bodies with oriented quartz, phyllosilicates, albite, large K-feldspars, and biotite; (ii) homogeneous coarse-grained quartz diorites with feldspar, hornblende, and biotite; and (iii) fine-grained leucogranites that occur in lenticular bodies subparallel to the dominant foliation S_1 . In contrast to the Bossòst granitoid, the Lys-Caillaouas granitoid has a laccolithic shape subparallel to the S_1 foliation, and the occurrence of large xenoliths is very common (Figure 3b). Esteban et al. (2015) obtained three different SIMS U-Pb zircon ages: 300 ± 2 Ma ($n = 17$) and 307 ± 3 Ma ($n = 11$) both in the porphyritic granodiorites facies and 299 ± 1 Ma ($n = 8$) in the quartz diorites (samples

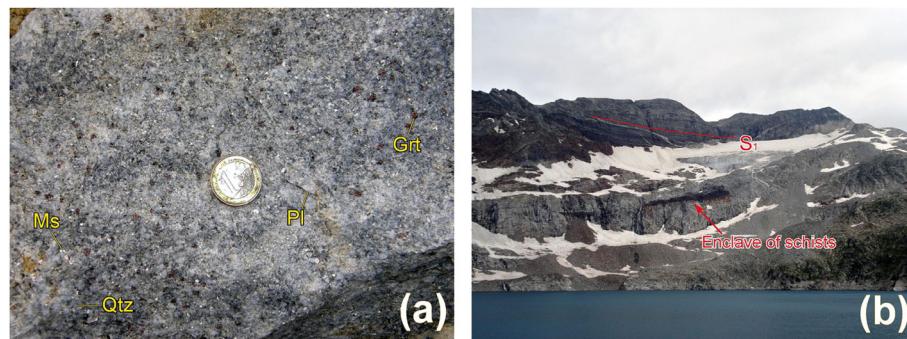


FIGURE 3 (a) The aspect of the Bossòst granitoid in the quarry south of Les. (b) The aspect of the Lys-Caillaouas granitoid. Notice the xenoliths with the S_1 cleavage. Abbreviations: Grt - garnet, Ms - muscovite, Pl - plagioclase, Qtz - quartz. Scale: coin 2.5 cm diameter [Colour figure can be viewed at [wileyonlinelibrary.com](#)]

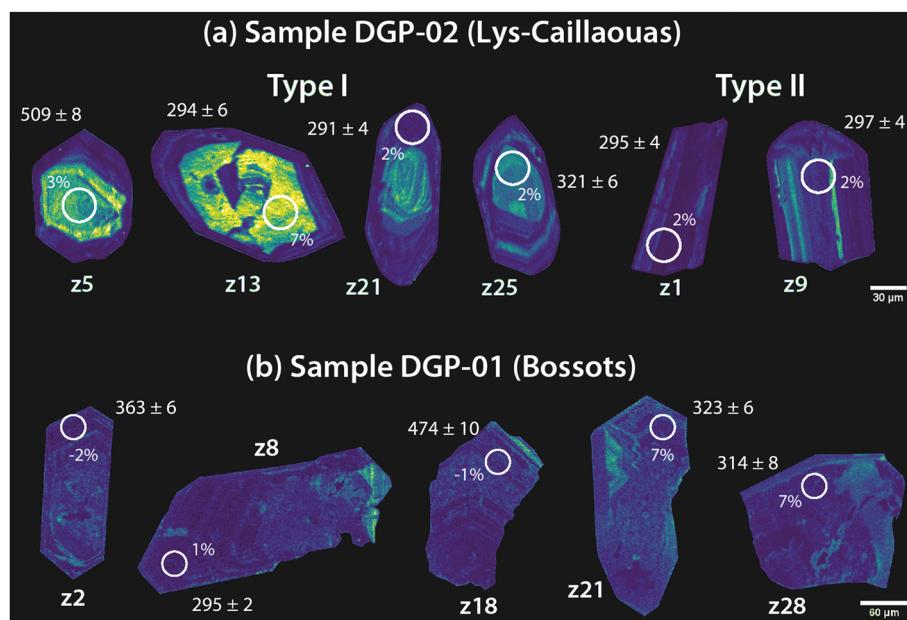


FIGURE 4 SEM-CL image of representative zircon grains from samples DGP-01 and 02. Circles represent the spot size and the location. The ages are reported in million years at the 2σ level of precision. Zircon adjacent numbers represent the spot number and the percentage of discordance, respectively [Colour figure can be viewed at [wileyonlinelibrary.com](#)]

AH-98 and AH-27 in Figure 2b, respectively). They consider the 300 ± 2 Ma age as the age of granitoid emplacement.

4 | SAMPLE AND ANALYTICAL METHODS

Sample DGP-01 (Bossòst granitoid) was collected on the western bank of the Garona River located in the south-west of the Les village, where this granitoid is quarried as an ornamental rock ($42^{\circ}48'08.4''N$, $0^{\circ}41'54.2''E$; Figure 2b). The sample is a fresh undeformed leucogranite. Sample DGP-02 (Lys-Caillaouas granitoid) was collected in the Remuñe stream, located on the western bank of the Benasque valley ($42^{\circ}41'08.3''N$, $0^{\circ}34'55.5''E$; Figure 2b). The sample corresponds to a fresh undeformed porphyritic granodiorite. In both cases, about 10 kg of rock were collected.

Zircon grains were extracted from rock samples using conventional mineral separation methods at Centro de Geociencias in Universidad Autónoma de México (UNAM). Zircon standard Plešovice (337 Ma; Sláma et al., 2008) plus unknowns were mounted in epoxy,

ground to nearly half thickness, and polished with 6 and 1 μm grit diamond suspension abrasive, later cleaned in distilled water and 1 N HCl, and gold-coated for maximum surface conductivity. CL images were acquired with a JEOL 5600 SEM at Denver University and enhanced using the ImageJ application (Schneider, Rasband, & Eliceiri, 2012).

Zircon grains from both samples were dated following the method described in Solari, Gomez-Tuena, Pablo Bernal, Perez-Arvizu, and Tanner (2010) using the laser-ablation inductively coupled plasma mass spectrometry (LA-ICP-MS) U (Th)-Pb analysis at the Isotopic Studies Laboratory in UNAM. Briefly, a 30-sec ablation was used, producing a circular sample area of about 33 μm in diameter and 25 μm in depth. For the closest possible control of the measured Pb/U ratios, U, and Th, the analysis sequence started measuring a glass standard NIST SRM 612 (~38 ppm U). This is followed by five zircon analyses of the Plešovice standard, five unknowns, and thereafter another zircon standard for each five unknowns, finishing with two analysis of zircon standard plus one of NIST SRM 612 standard (Gehrels, Valencia, & Ruiz, 2008; Jackson, Longerich, Dunning, & Fryer, 1992; Solari et al., 2010). The LA-ICP-MS data were reduced using the application UPb.

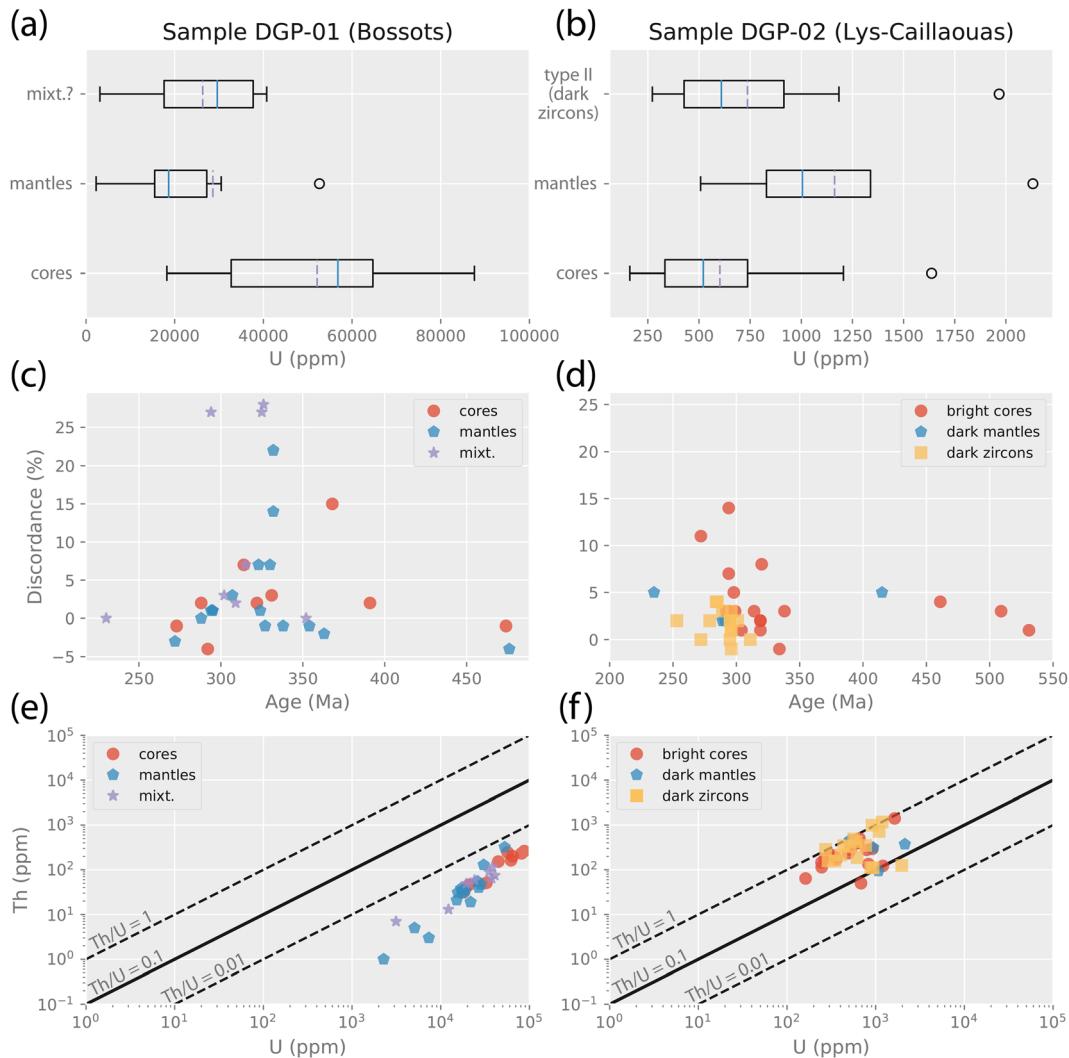


FIGURE 5 Plots showing features of the different zircon domains from the studied samples. (a and b) Box plots showing the U-content in different zircon domains. Solid and dashed lines indicate the median and the mean values, respectively. Boxes are the interquartile range (IQR), whiskers the $1.5 * IQR$ range and empty dots outliers. Outliers with extreme values were cut for clarity. (c and d) Discordance versus age plots. (e and f) Th versus U content plots [Colour figure can be viewed at wileyonlinelibrary.com]

age (Solari & Tanner, 2011) and plotted using the software Isoplot/Ex 4.15 (Ludwig, 2012). The ratios were corrected offline for common Pb, using the algebraic approach proposed in Andersen (2002). U–Pb age data for zircon were plotted on Wetherill (1956) charts along with a model common Pb line based on Stacey and Kramers (1975), and U–Pb ages calculated using Concordia and Concordia-Intercept ages.

5 | RESULTS

5.1 | Sample DGP-01: Bossòst granitoid

Cathodoluminescence (CL) images show euhedral zircon grains with double-ended prismatic shapes when unbroken (Figure 4b). Zircon grains have dark cores with a notable mottled patchy texture, occasionally showing an oscillatory zoning. The mantles surrounding the cores (e.g., z2, z8, and z28 in Figure 4b) are usually darker with an oscillatory zoning, although with some exceptions (e.g., z28 in Figure 4b). We made 33 analyses with 25 lying within the $\pm 5\%$ of discordance, 9 in the mottled cores, 15 in the mantles, and the rest are suspected of containing parts of the core and the mantle (Figure 5c, see Table 1). Zircon grains have an unusually high U-content. This is very notable in the mottled dark cores, with a median of 56,800 ppm and values up to 90,000 rather common (Figure 5a). The dark mantles have a lower uranium content, with a median of 18,630 ppm, and leaving aside two outliers (z24 and z31), values are below 30,000 ppm (Figure 5a). Both the cores and the mantles have very low Th/U values (<0.006), more typical of metamorphic zircon ($\ll 0.1$; Figure 5e). Although the dispersion of individual zircon ages is wider in the cores than in the mantles, the age range completely overlaps.

The Wetherill plot displays a complex age pattern, with a great dispersion of ages between ca. 260 and 360 Ma (Figures 5c and 6). Only one tight concordant cluster exists. The cluster, formed by three analyses performed in the zircon mantles, yields a concordia age of 295 ± 2 Ma ($n = 3$; 2-sigma: MSWD = 2.8; Figure 6). Beside this, there is a clear pattern of discordance comprising analysis from the dark mantles (Figures 5c) that yields a lower intercept age at 326 ± 3 Ma ($n = 8$; MSWD = 1.15; prob. of fit = 0.33; Figure 6).

5.2 | Sample DGP-02: Lys-Caillaouas granitoid

CL images show euhedral zircon grains with double-ended prismatic or bipyramid shapes when unbroken (Figure 4a). Based on the CL features, two types of zircon grains can be distinguished. One group, called type-I zircon, shows bright cores and dark oscillatory-zoned mantles containing both prismatic and bipyramid shapes (Figure 4a). Some of the mantles cut the growth zoning in the cores, implying dissolution and reprecipitation during the formation of the mantles (e.g., Figure 4a, z5 and z25). The other group, called type-II zircon, consists of prismatic dark zircon with no differentiated cores that rarely developed dark unzoned tips (Figure 4a). We made 40 analyses, 20 in the bright cores, 4 in the dark oscillatory-zoned mantles, and 16 in the type-II zircon (see Table 2), with 34 out of 40 analyses falling within the $\pm 5\%$ of discordance (Figure 5d). Zircon grains show moderate U-contents, mostly below 1,500 ppm. Both zircon types show similar U-contents (medians of 521 and 610 ppm, respectively).

The type-I zircon mantles show slightly higher U-content values (Figure 5b), but this could be an artefact due to a very small sample size for this zircon domain ($n = 4$). The individual ages of the different zircon domains and types overlap, but the ages of type-II zircon are mostly younger than 300 Ma, while the range of ages in the type-I zircon is wider (Figure 5d). All zircon types and domains show similar Th/U ratios typical of magmatic zircon (Figure 5f).

The Wetherill plot displays a complex age pattern with two tight concordant zircon clusters (Figure 7). The older cluster, fully formed by type-I bright cores, yields a concordia age at 320 ± 3 Ma ($n = 3$; 2-sigma; MSWD = 3.3; Figure 7). The younger cluster, fully formed by the type-II zircon, yields a Concordia age at 297 ± 2 Ma ($n = 6$; 2-sigma; MSWD = 2.8). In addition, the Concordia plot reveals two discordance patterns around the young tight cluster (Figure 7). One of them essentially consists of zircon analysis performed in the bright cores that along with zircon grains belonging to the younger cluster yields a lower intercept age at 298 ± 2 Ma ($n = 11$; MSWD = 0.83; prob. of fit = 0.60). The other is formed by a mix of type-I dark mantles and type-II zircon, yielding an upper intercept age similar to those obtained above (ca. 300 Ma). However, despite the good fit ($n = 12$; prob. of fit = 0.95), the error obtained is large due to the regression line intersecting the concordia curve at a very low angle.

6 | DISCUSSION

6.1 | Age of Bossòst and Lys-Caillaouas granitoids

Zircon grains in the Bossòst and Lys-Caillaouas granitoids have in common complex age patterns and two interpretable ages: one early Permian (293–299 Ma) and the other Serpukhovian–Bashkirian (Late Mississippian–Early Pennsylvanian; 317–329 Ma).

In the Lys-Caillaouas granitoid, type-II zircon grains show features typical of magmatic zircon and yield an age of 297 ± 2 Ma. This early Permian age represents, therefore, the age of crystallization of the Lys-Caillaouas leucogranitic facies. This age also agrees with those obtained by Esteban et al. (2015) in the basic (299 ± 1 Ma) and the porphyritic facies (300 ± 2 Ma), indicating that all the granite facies were emplaced in a very short period. Accordingly, we consider zircon with early Carboniferous ages (ca. 320 ± 2 Ma) as xenocrysts. In fact, all of them come from the analyses performed in the type-I zircon cores, and thus, these cores represent old zircon grains incorporated in the Permian magmatic event. Regarding the early Permian intercept ages, the upper intercept suggests a Pb-loss effect in some Permian zircon, while the lower intercept suggests either a core-rim mixing, a Pb-loss effect, or a combination of both in the inherited zircon grains.

The Bossòst granitoid only provides one concordant tight cluster that yields an early Permian age of 295 ± 4 Ma. This age is similar within the error to the age found in the Lys-Caillaouas granitoid and most likely represents the crystallization age of the Bossòst leucogranitic facies. This crystallization age is incompatible with the Visean ages obtained by Mezger and Gerdes (2016) in different facies of the Bossòst granitoid. The fact that we obtained this Permian age from zircon mantles with Th/U ratios typical of metamorphic zircon

TABLE 1 LA-ICP-MS U-Th-Pb data (sample DGP-01) for zircons from the Bossost granitoid

| Data point ^a | Domain | U ^b (ppm) | Th ^b (ppm) | 207Pb/ 235U ^c | | 206Pb/ 238U ^c | | 208Pb/ 232Th ^c | | 207Pb/ 235U | | 206Pb/ 238U | | 207Pb/ 232Th | | 208Pb/ 232Th | | Best age (Ma) | | Best age $\pm 1\sigma^a$ | | | | |
|-------------------------|--------------|-------------------------|--------------------------|-----------------------------|---------------|-----------------------------|---------------|------------------------------|---------------|----------------|---------------|----------------|---------------|-----------------|---------------|-----------------|---------------|------------------|---------------|-----------------------------|--------|-------|-----|---|
| | | | | $\pm 1\sigma$ | $\pm 1\sigma$ | $\pm 1\sigma$ | $\pm 1\sigma$ | Rho | $\pm 1\sigma$ | $\pm 1\sigma$ | $\pm 1\sigma$ | $\pm 1\sigma$ | $\pm 1\sigma$ | $\pm 1\sigma$ | $\pm 1\sigma$ | $\pm 1\sigma$ | $\pm 1\sigma$ | $\pm 1\sigma$ | $\pm 1\sigma$ | $\pm 1\sigma$ | | | | |
| Zircon_22 | Mantle-core? | 40,748 | 75 | 0.002 | 0.05064 | 0.00122 | 0.25318 | 0.00806 | 0.03626 | 0.00059 | 0.790 | 0.01143 | 0.00766 | 0 | 230 | 4 | 229 | 7 | 224 | 52 | 230 | 153 | 230 | 4 |
| Zircon_31 | Mantle | 154,529 | 534 | 0.003 | 0.04977 | 0.00045 | 0.29567 | 0.00543 | 0.04307 | 0.00069 | 0.870 | 0.02504 | 0.00075 | -3 | 272 | 4 | 263 | 4 | 184 | 20 | 500 | 15 | 272 | 4 |
| Zircon_36 | Core | 18,231 | 32 | 0.002 | 0.05090 | 0.00203 | 0.30398 | 0.01461 | 0.04331 | 0.00077 | 0.790 | 0.01364 | 0.01856 | -1 | 273 | 5 | 270 | 11 | 236 | 86 | 274 | 370 | 273 | 5 |
| Zircon_5 | Core | 81,598 | 233 | 0.003 | 0.05366 | 0.00070 | 0.33642 | 0.00668 | 0.04563 | 0.00068 | 0.750 | 0.19282 | 0.01099 | 2 | 288 | 4 | 294 | 5 | 357 | 27 | 3,564 | 186 | 288 | 4 |
| Zircon_7 | Mantle | 7,345 | 3 | 0.000 | 0.05226 | 0.00050 | 0.32947 | 0.00360 | 0.04572 | 0.00022 | 0.490 | 0.01435 | 0.00581 | 0 | 288 | 1 | 289 | 3 | 297 | 23 | 288 | 116 | 288 | 1 |
| Zircon_17 | Core | 87,586 | 262 | 0.003 | 0.05028 | 0.00047 | 0.31998 | 0.00511 | 0.04626 | 0.00060 | 0.810 | 0.04096 | 0.00164 | -4 | 292 | 4 | 282 | 4 | 208 | 20 | 811 | 32 | 292 | 4 |
| Zircon_37 | Mantle | 26,845 | 40 | 0.002 | 0.05276 | 0.00062 | 0.33991 | 0.00461 | 0.04672 | 0.00029 | 0.550 | 0.01465 | 0.00589 | 1 | 294 | 2 | 297 | 3 | 319 | 25 | 294 | 117 | 294 | 2 |
| Zircon_8 | Mantle | 21,704 | 19 | 0.001 | 0.05262 | 0.00054 | 0.34002 | 0.00389 | 0.04687 | 0.00022 | 0.480 | 0.01470 | 0.00545 | 1 | 295 | 1 | 297 | 3 | 312 | 24 | 295 | 109 | 295 | 1 |
| Zircon_27 | Mantle | 2,271 | 1 | 0.000 | 0.05292 | 0.00056 | 0.34107 | 0.00405 | 0.04675 | 0.00023 | 0.460 | 0.01465 | 0.002175 | 1 | 295 | 1 | 298 | 3 | 325 | 22 | 294 | 433 | 295 | 1 |
| Zircon_13 | Mantle-core? | 12,228 | 13 | 0.001 | 0.05429 | 0.00136 | 0.35900 | 0.00958 | 0.04796 | 0.00031 | 0.430 | 0.01499 | 0.02596 | 3 | 302 | 2 | 311 | 7 | 383 | 58 | 301 | 517 | 302 | 2 |
| Zircon_40 | Mantle | 28,403 | 47 | 0.002 | 0.05421 | 0.00185 | 0.36451 | 0.01550 | 0.04877 | 0.00085 | 0.690 | 0.01524 | 0.01871 | 3 | 307 | 5 | 316 | 11 | 380 | 72 | 306 | 373 | 307 | 5 |
| Zircon_35 | Mantle-core? | 37,493 | 64 | 0.002 | 0.05343 | 0.00059 | 0.36317 | 0.00457 | 0.04913 | 0.00030 | 0.480 | 0.29523 | 0.02421 | 2 | 309 | 2 | 315 | 3 | 347 | 23 | 5,229 | 378 | 309 | 2 |
| Zircon_28 | Core | 56,802 | 244 | 0.004 | 0.05757 | 0.00058 | 0.39419 | 0.00678 | 0.04984 | 0.00070 | 0.810 | 0.28845 | 0.02279 | 7 | 314 | 4 | 337 | 5 | 513 | 23 | 5,123 | 358 | 314 | 4 |
| Zircon_10 | Mantle-core? | 38,327 | 117 | 0.003 | 0.05740 | 0.00063 | 0.39643 | 0.00617 | 0.05013 | 0.00055 | 0.710 | 0.31364 | 0.01756 | 7 | 315 | 3 | 339 | 4 | 507 | 22 | 5,514 | 270 | 315 | 3 |
| Zircon_33 | Core | 62,014 | 163 | 0.003 | 0.05440 | 0.00060 | 0.38301 | 0.00547 | 0.05115 | 0.00047 | 0.640 | 0.23065 | 0.01522 | 2 | 322 | 3 | 329 | 4 | 388 | 23 | 4,195 | 250 | 322 | 3 |
| Zircon_21 | Mantle | 15,161 | 21 | 0.001 | 0.05770 | 0.00255 | 0.40869 | 0.01930 | 0.05137 | 0.00049 | 0.400 | 0.01594 | 0.03495 | 7 | 323 | 3 | 348 | 14 | 518 | 91 | 320 | 695 | 323 | 3 |
| Zircon_25 | Mantle | 26,029 | 55 | 0.002 | 0.05345 | 0.00101 | 0.37945 | 0.00832 | 0.05149 | 0.00046 | 0.570 | 0.01612 | 0.00647 | 1 | 324 | 3 | 327 | 6 | 348 | 40 | 323 | 129 | 324 | 3 |
| Zircon_12 | Mantle-core? | 19,395 | 49 | 0.003 | 0.07835 | 0.00212 | 0.55532 | 0.01664 | 0.05166 | 0.00067 | 0.430 | 1.63290 | 0.11757 | 27 | 325 | 4 | 448 | 11 | 1,156 | 50 | 19,567 | 903 | 325 | 4 |
| Zircon_3 | Mantle-core? | 35,094 | 90 | 0.003 | 0.07505 | 0.00180 | 0.55886 | 0.01746 | 0.05180 | 0.000104 | 0.640 | 1.63820 | 0.12942 | 28 | 326 | 6 | 451 | 11 | 1,070 | 45 | 19,608 | 992 | 326 | 6 |
| Zircon_24 | Mantle | 52,642 | 320 | 0.006 | 0.05255 | 0.00148 | 0.37666 | 0.01141 | 0.05199 | 0.00043 | 0.400 | 0.01631 | 0.00340 | -1 | 327 | 3 | 325 | 8 | 309 | 60 | 327 | 68 | 327 | 3 |
| Zircon_1 | Mantle | 5,064 | 5 | 0.001 | 0.05806 | 0.00306 | 0.42022 | 0.02342 | 0.05250 | 0.00049 | 0.360 | 0.01627 | 0.04957 | 7 | 330 | 3 | 356 | 17 | 532 | 121 | 326 | 986 | 330 | 3 |
| Zircon_29 | Core | 32,738 | 51 | 0.002 | 0.05488 | 0.00055 | 0.40079 | 0.00564 | 0.05271 | 0.00052 | 0.700 | 0.41858 | 0.01967 | 3 | 331 | 3 | 342 | 4 | 407 | 21 | 7,067 | 280 | 331 | 3 |
| Zircon_9 | Mantle | 17,509 | 30 | 0.002 | 0.06365 | 0.00293 | 0.46347 | 0.02277 | 0.05281 | 0.00057 | 0.410 | 0.01620 | 0.03560 | 14 | 332 | 3 | 387 | 16 | 730 | 102 | 325 | 708 | 332 | 3 |
| Zircon_38 | Mantle | 18,133 | 36 | 0.002 | 0.07098 | 0.00121 | 0.51911 | 0.01285 | 0.05285 | 0.00095 | 0.730 | 1.60760 | 0.13986 | 22 | 332 | 6 | 425 | 9 | 957 | 32 | 19,372 | 1,084 | 332 | 6 |
| Zircon_14 | Mantle | 15,595 | 31 | 0.002 | 0.05291 | 0.00047 | 0.39272 | 0.00619 | 0.05390 | 0.00070 | 0.830 | 0.04450 | 0.00142 | -1 | 338 | 4 | 336 | 5 | 325 | 19 | 880 | 27 | 338 | 4 |
| Zircon_30 | Mantle-core? | 24,104 | 59 | 0.002 | 0.05302 | 0.00080 | 0.41456 | 0.01036 | 0.05606 | 0.00112 | 0.800 | 0.24312 | 0.03647 | 0 | 352 | 7 | 352 | 7 | 330 | 32 | 4,399 | 593 | 352 | 7 |
| Zircon_26 | Mantle | 16,917 | 38 | 0.002 | 0.05305 | 0.00053 | 0.41298 | 0.00568 | 0.05646 | 0.00049 | 0.710 | 0.01769 | 0.00344 | -1 | 354 | 3 | 351 | 4 | 331 | 21 | 355 | 68 | 354 | 3 |

(Continues)

TABLE 1 (Continued)

| Data point ^a | Domain | U ^b (ppm) | Th ^b (ppm) | 207Pb/ 235U ^c ±1σ | 207Pb/ 235U ^c ±1σ | 206Pb/ 238U ^c ±1σ | Rho | 208Pb/ 232Th ^c ±1σ | % disc ^d | 206Pb/ 235U ±1σ | 207Pb/ 235U ±1σ | 206Pb/ 232Th ±1σ | 208Pb/ 232Th ±1σ | Best age (Ma) | ±1σ ^a | |
|-------------------------|--------------|-------------------------|--------------------------|------------------------------------|------------------------------------|------------------------------------|---------|-------------------------------------|---------------------|-----------------------|-----------------------|------------------------|------------------------|------------------|------------------|---|
| Zircon_2 | Mantle-core? | 19,126 | 35 | 0.002 | 0.05265 | 0.00048 | 0.42022 | 0.00565 | 0.05793 | 0.00057 | 0.740 | 0.04978 | 0.00154 | -2 | 363 | 3 |
| Zircon_19 | Core | 64,701 | 201 | 0.003 | 0.06380 | 0.00089 | 0.52952 | 0.01250 | 0.05881 | 0.00112 | 0.810 | 0.91370 | 0.06579 | 15 | 368 | 7 |
| Zircon_16 | Mantle-core? | 3,134 | 7 | 0.002 | 0.08103 | 0.00162 | 0.67641 | 0.01866 | 0.06084 | 0.00116 | 0.690 | 0.74030 | 0.33656 | 27 | 381 | 7 |
| Zircon_15 | Core | 44,610 | 153 | 0.003 | 0.05592 | 0.00067 | 0.47965 | 0.00884 | 0.06245 | 0.00087 | 0.760 | 0.27322 | 0.01639 | 2 | 391 | 5 |
| Zircon_18 | Core | 20,814 | 46 | 0.002 | 0.05568 | 0.00052 | 0.58738 | 0.00846 | 0.07630 | 0.00084 | 0.760 | 0.39989 | 0.01120 | -1 | 474 | 5 |
| Zircon_20 | Mantle | 30,488 | 129 | 0.004 | 0.05420 | 0.00054 | 0.57073 | 0.01077 | 0.07659 | 0.00123 | 0.850 | 0.17902 | 0.00859 | -4 | 476 | 7 |

^a Sample analysed July 2010 on the LEI-UNAM LA-ICP-MS.^b U and Th concentrations (ppm) are calculated relative to analyses of trace-element glass standard NIST 612.^c Isotopic ratios corrected relative to Plesovice standard zircon (Sláma et al., 2008). Common Pb corrections made using the Andersen (2002) method.^d Percentage discordance obtained using the following equation ($100 \times [(\text{age}^{207}\text{Pb}/^{235}\text{U}) - (\text{age}^{207}\text{Pb}/^{238}\text{U})]/(\text{age}^{207}\text{Pb}/^{235}\text{U})$). Positive and negative values indicate normal and inverse discordance, respectively.

(Hoskin & Black, 2000; Rubatto, 2002; Williams & Claesson, 1987) might suggest that we dated a metamorphic instead of the crystallization event. However, we discard this hypothesis based on the following reasoning:

- Despite being uncommon, low (<0.1) Th/U ratios in magmatic zircon have been reported elsewhere (e.g., Ladenberger et al., 2014; Lopez-Sanchez, Aleinikoff, Marcos, Martínez, & Llana-Fúnez, 2016; Schärer, 1984; Zeck & Whitehouse, 1999). In fact, they are rather common in peraluminous granitoids that enclose zircon with U-content above 1,000 ppm (see figure 5 in Lopez-Sánchez et al., 2016), as it occurs in the sample dated.
- Zircon cores have similar low Th/U ratios (Figure 5e). Consequently, if the Th/U ratio is used as a condition to discriminate between magmatic and metamorphic origin, all the zircon enclosed in the granite sample must be regarded as metamorphic in origin, which is unlikely.
- The Visean zircon grains found by Mezger and Gerdes (2016) are completely different from those obtained in this study. Indeed, with the exception of zircon obtained from a granite sill, their appearance and Th/U ratios are completely different from ours. Furthermore, all zircon reported by Mezger and Gerdes (2016) show lower U contents. All this suggests that the early Carboniferous and the early Permian zircon comes from different magmatic events.

Based on the above considerations, we think that the leucogranitic facies of the Bossòst granitoid crystallized at early Permian times. Accordingly, the lower intercept that yields an early Carboniferous age suggests an early Carboniferous thermal event imprinted in pre-Variscan zircon. Strictly based on the available zircon U-Pb data, the question whether the main magmatic event and the concurrent HT metamorphism in the Garona dome is early Permian or Visean remains unsettled. Our data, however, confirm the need to carry out a detailed study of the different granite facies and their structural relationships to set a reliable relationship between the HT-LP metamorphisms event and the crystallization of the different facies of the Bossòst granitoid.

When U-Pb ages from the main metamorphic domes along the Axial Zone are compared (Table 3), the Lys-Caillaous and the Canigou-Carançà domes are the only ones that present deep-seated granitoids with conclusive late Carboniferous to early Permian ages. In contrast, the Garona and Aston domes provide both early Carboniferous and late Carboniferous to early Permian zircon. In the Garona Dome, the different facies of the Bossòst granitoid provide both ages. In contrast, two different granitoids separated more than 20 km apart provide the different ages in the Aston Dome, the Soulcem (Mezger & Gerdes, 2016) and the Ax-les-Thermes (Denèle et al., 2014) granitoids, respectively. Mezger and Regnier (2016) have proposed two hypotheses to explain this age disparity across the domes: (i) the deep-seated granitoids, the HT-LP metamorphism, and related flat-lying schistosity are of Visean age only or (ii) the emplacement of deep-seated granitoids are diachronous across the Axial Zone, ranging from Visean in the central part to late Carboniferous in the eastern Axial Zone.

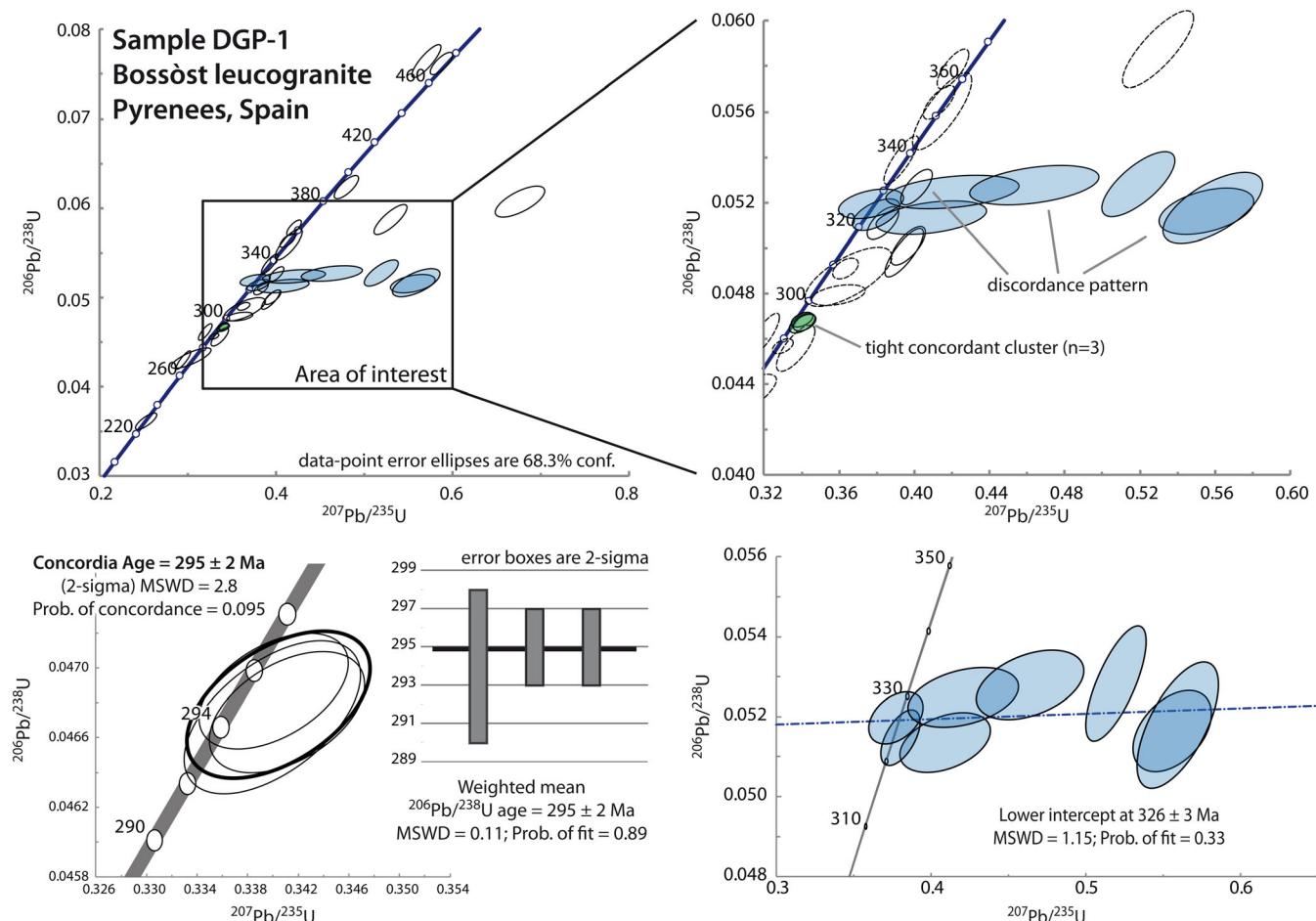


FIGURE 6 Wetherill and weighted average plots showing the different zircon dates in the sample DGP-01. Filled error ellipses are the ones used to estimate the different Concordia and intercept ages. Error ellipses and boxes are 2σ [Colour figure can be viewed at wileyonlinelibrary.com]

Our U-Pb zircon data discard both premises since the Lys-Caillaouas granitoid is located in the central part of the Axial Zone and provides a reliable U-Pb early Permian crystallization age.

6.2 | Potential sources for the early Carboniferous zircon grains in the Axial Zone

Visean-Bashkirian zircon grains appear frequently as inherited ages in most of the granitoids that outcrop within the metamorphic domes along the Pyrenees, including those studied here. Some igneous bodies have been dated as Serpukhovian (e.g., Martínez et al., 2008; Mezger & Gerdes, 2016), and migmatization in the Aston dome has been tentatively dated between 340 and 320 Ma (Mezger & Gerdes, 2016). Furthermore, Martínez et al. (2016) found zircon grains with Visean-Bashkirian ages in flysches along the adjacent Catalan Coastal Ranges 80 km south of the Pyrenean Axial Zone (Figure 1). All these data point to a widespread early Carboniferous magmatic event. Yet, the magmatic bodies that produced early Carboniferous zircon populations in the Pyrenees remain elusive. In addition, this contrasts to what is observed in the Central-Iberian zone of the Variscan Iberian Massif, where the Visean magmatic activity and the related magmatic bodies are well constrained (e.g., Gutiérrez-Alonso et al., 2018).

Biostratigraphic (Delvolv  , 1981; Delvolv   & Perret, 1989; Delvolv  , Souquet, Vachard, Perret, & Aguirre, 1993; Feist & Galtier,

1985; Sanz-L  pez, Perret, & Vachard, 2006) and geochronological (detrital zircon population) data (Mart  nez et al., 2016) from syn-orogenic sedimentation (Culm deposits) in the Pyrenees, Montagne Noire, and Catalan Coastal Ranges indicate that Variscan deformation was active at least during the 360 to 315–310 Ma period (Tournaisian–Bashkirian). Hence, a probable cause for the origin of early Carboniferous zircon in the Pyrenees is a magmatic event due to thermal relaxation, which produced small segregated igneous bodies and the local migmatization in deep zones of the crust during the Variscan collision. This is in line with what Guti  rrez-Alonso et al. (2018) recently proposed for the adjacent Iberian (Variscan) Massif. The age of migmatization inferred by Mezger and Gerdes (2016) in the Aston dome, between 340 and 320 Ma, agrees with this hypothesis. Similarly, the Bashkirian ages (ca. 319 Ma) recently found in the Louzas granite and their related migmatites in the adjacent Variscan Montagne Noire (Poujol et al., 2017) support this hypothesis. The overprint of the widespread late Carboniferous to early Permian magmatism would explain the difficulty of finding and dating early Carboniferous magma bodies *in situ* in the Axial Zone. Compared to the Iberian Massif, it appears that the outcrop of the areas of the crust affected by this event is more limited in the Axial Zone or that the early Carboniferous magmatism was significantly less voluminous, making it more difficult to recognize.

TABLE 2 LA-ICP-MS U-Th-Pb data (sample DGP-01) for zircons from the Lys-Caillaous granitoid

| Data point ^a | Domain | U^b (ppm) | Th^b (ppm) | $^{207}Pb/^{235}U^c$ $\pm 1\sigma$ | $^{206}Pb/^{238}U^c$ $\pm 1\sigma$ | Rho | $^{208}Pb/^{232}Th^c$ $\pm 1\sigma$ | % disc ^d | $^{206}Pb/^{238}U$ $\pm 1\sigma$ | $^{207}Pb/^{235}U$ $\pm 1\sigma$ | $^{207}Pb/^{206}Pb$ $\pm 1\sigma$ | $^{208}Pb/^{232}Th$ $\pm 1\sigma$ | Best age (Ma) | $\pm 1\sigma^a$ | | |
|-------------------------|--------------|----------------|-----------------|---------------------------------------|---------------------------------------|---------|--|------------------------|-------------------------------------|-------------------------------------|--------------------------------------|--------------------------------------|------------------|-----------------|-----|---|
| Zircon_36 | Dark mantle | 2,132 | 374 | 0.18 | 0.05412 | 0.00054 | 0.27709 | 0.00367 | 0.03705 | 0.00032 | 0.660 | 0.01449 | 0.00017 | 5 | 235 | 2 |
| Zircon_31 | Dark Zr | 1,967 | 125 | 0.06 | 0.05246 | 0.00063 | 0.28931 | 0.00412 | 0.04000 | 0.00027 | 0.540 | 0.01255 | 0.00009 | 2 | 253 | 2 |
| Zircon_14 | Bright core | 1,637 | 1,409 | 0.86 | 0.05900 | 0.00153 | 0.35011 | 0.01033 | 0.04304 | 0.00028 | 0.360 | 0.01332 | 0.00008 | 11 | 272 | 2 |
| Zircon_30 | Dark Zr | 1,094 | 722 | 0.66 | 0.05165 | 0.00094 | 0.30688 | 0.00669 | 0.04309 | 0.00026 | 0.460 | 0.01355 | 0.00007 | 0 | 272 | 2 |
| Zircon_17 | Dark Zr | 1,184 | 1,171 | 0.99 | 0.05310 | 0.00052 | 0.32457 | 0.00368 | 0.04422 | 0.00026 | 0.500 | 0.01361 | 0.00014 | 2 | 279 | 2 |
| Zircon_15 | Dark Zr | 514 | 329 | 0.64 | 0.05453 | 0.00071 | 0.33888 | 0.00504 | 0.04503 | 0.00032 | 0.480 | 0.01366 | 0.00019 | 4 | 284 | 2 |
| Zircon_6 | Dark Zr | 617 | 187 | 0.30 | 0.05433 | 0.00103 | 0.33803 | 0.00721 | 0.04512 | 0.00029 | 0.350 | 0.01410 | 0.00009 | 4 | 285 | 2 |
| Zircon_7 | Dark mantle | 508 | 457 | 0.90 | 0.05317 | 0.00074 | 0.33659 | 0.00515 | 0.04584 | 0.00028 | 0.420 | 0.01394 | 0.00015 | 2 | 289 | 2 |
| Zircon_24 | Dark Zr | 773 | 349 | 0.45 | 0.05389 | 0.00059 | 0.34082 | 0.00416 | 0.04579 | 0.00024 | 0.440 | 0.01428 | 0.00016 | 3 | 289 | 1 |
| Zircon_21 | Dark mantle | 937 | 313 | 0.33 | 0.05307 | 0.00071 | 0.33825 | 0.00557 | 0.04623 | 0.00028 | 0.510 | 0.01449 | 0.00008 | 2 | 291 | 2 |
| Zircon_26 | Bright core? | 317 | 213 | 0.67 | 0.05428 | 0.00103 | 0.34612 | 0.00689 | 0.04627 | 0.00027 | 0.300 | 0.01444 | 0.00019 | 3 | 292 | 2 |
| Zircon_4 | Bright core | 591 | 398 | 0.67 | 0.05420 | 0.00070 | 0.34969 | 0.00496 | 0.04671 | 0.00027 | 0.410 | 0.01423 | 0.00016 | 3 | 294 | 2 |
| Zircon_12 | Bright core | 267 | 177 | 0.66 | 0.06182 | 0.00190 | 0.39768 | 0.01352 | 0.04665 | 0.00034 | 0.310 | 0.01436 | 0.00010 | 14 | 294 | 2 |
| Zircon_13 | Bright core | 246 | 114 | 0.47 | 0.05656 | 0.00085 | 0.36563 | 0.00645 | 0.04669 | 0.00043 | 0.520 | 0.01474 | 0.00021 | 7 | 294 | 3 |
| Zircon_1 | Dark Zr | 602 | 424 | 0.70 | 0.05316 | 0.00090 | 0.34353 | 0.00622 | 0.04675 | 0.00029 | 0.350 | 0.01382 | 0.00018 | 2 | 295 | 2 |
| Zircon_32 | Dark Zr | 936 | 110 | 0.12 | 0.05289 | 0.00060 | 0.34209 | 0.00479 | 0.04691 | 0.00032 | 0.550 | 0.01471 | 0.00010 | 1 | 296 | 2 |
| Zircon_38 | Dark Zr | 435 | 346 | 0.80 | 0.05374 | 0.00064 | 0.34890 | 0.00475 | 0.04703 | 0.00030 | 0.480 | 0.01429 | 0.00016 | 3 | 296 | 2 |
| Zircon_9 | Dark Zr | 272 | 287 | 1.05 | 0.05316 | 0.00069 | 0.34704 | 0.00506 | 0.04722 | 0.00031 | 0.460 | 0.01434 | 0.00016 | 2 | 297 | 2 |
| Zircon_8 | Dark Zr | 908 | 993 | 1.09 | 0.05163 | 0.00067 | 0.33789 | 0.00482 | 0.04734 | 0.00028 | 0.420 | 0.01444 | 0.00016 | -1 | 298 | 2 |
| Zircon_10 | Bright core | 162 | 64 | 0.39 | 0.05527 | 0.00094 | 0.36156 | 0.00662 | 0.04735 | 0.00032 | 0.370 | 0.01465 | 0.00021 | 5 | 298 | 2 |
| Zircon_2 | Dark Zr | 290 | 157 | 0.54 | 0.05233 | 0.00094 | 0.34341 | 0.00662 | 0.04747 | 0.00033 | 0.360 | 0.01453 | 0.00017 | 0 | 299 | 2 |
| Zircon_16 | Dark Zr | 404 | 214 | 0.53 | 0.05280 | 0.00069 | 0.34595 | 0.00494 | 0.04740 | 0.00028 | 0.400 | 0.01434 | 0.00016 | 1 | 299 | 2 |
| Zircon_33 | Bright core | 521 | 283 | 0.54 | 0.05415 | 0.00065 | 0.35405 | 0.00477 | 0.04740 | 0.00029 | 0.450 | 0.01410 | 0.00017 | 3 | 300 | 2 |
| Zircon_34 | Dark Zr | 564 | 490 | 0.87 | 0.05367 | 0.00070 | 0.35450 | 0.00505 | 0.04785 | 0.00028 | 0.400 | 0.01415 | 0.00017 | 2 | 301 | 2 |

(Continues)

TABLE 2 (Continued)

| Data point ^a | Domain | U^b (ppm) | Th^b (ppm) | $^{207}Pb/^{235}U^c$ $\pm 1\sigma$ | $^{207}Pb/^{235}U^c$ $\pm 1\sigma$ | $^{206}Pb/^{238}U^c$ $\pm 1\sigma$ | Rho | $^{208}Pb/^{232}Th^c$ $\pm 1\sigma$ | $^{206}Pb/^{238}U^d$ $\pm 1\sigma$ | % disc ^d | $^{207}Pb/^{235}U$ $\pm 1\sigma$ | $^{207}Pb/^{232}Th$ $\pm 1\sigma$ | $^{208}Pb/^{232}Th$ $\pm 1\sigma$ | Best age (Ma) | $\pm 1\sigma^a$ | |
|-------------------------|--------------------|----------------|-----------------|---------------------------------------|---------------------------------------|---------------------------------------|---------|--|---------------------------------------|---------------------|-------------------------------------|--------------------------------------|--------------------------------------|------------------|-----------------|----|
| Zircon_39 | Bright core | 917 | 294 | 0.32 | 0.05287 | 0.00053 | 0.35210 | 0.00412 | 0.04824 | 0.00029 | 0.520 | 0.01423 | 0.00020 | 1 | 304 | 2 |
| Zircon_3 | Dark Zr | 348 | 158 | 0.45 | 0.05254 | 0.00074 | 0.35854 | 0.00607 | 0.04945 | 0.00047 | 0.550 | 0.01503 | 0.00024 | 0 | 311 | 3 |
| Zircon_23 | Bright core | 245 | 146 | 0.60 | 0.05494 | 0.00077 | 0.33791 | 0.00574 | 0.04990 | 0.00029 | 0.390 | 0.01504 | 0.00018 | 3 | 314 | 2 |
| Zircon_28 | Bright core | 492 | 236 | 0.48 | 0.05373 | 0.00064 | 0.37635 | 0.00519 | 0.05078 | 0.00035 | 0.500 | 0.01562 | 0.00020 | 2 | 319 | 2 |
| Zircon_11 | Bright core | 359 | 170 | 0.47 | 0.05805 | 0.00130 | 0.40753 | 0.01019 | 0.05092 | 0.00031 | 0.350 | 0.01579 | 0.00009 | 8 | 320 | 2 |
| Zircon_37 | Bright core | 350 | 192 | 0.55 | 0.05304 | 0.00080 | 0.37341 | 0.00625 | 0.05097 | 0.00038 | 0.430 | 0.01482 | 0.00019 | 1 | 320 | 2 |
| Zircon_25 | Bright core | 626 | 396 | 0.63 | 0.05359 | 0.00080 | 0.37897 | 0.00679 | 0.05113 | 0.00050 | 0.550 | 0.01448 | 0.00017 | 2 | 321 | 3 |
| Zircon_19 | Bright core | 683 | 50 | 0.07 | 0.05242 | 0.00071 | 0.38421 | 0.00663 | 0.05316 | 0.00047 | 0.630 | 0.01668 | 0.00019 | -1 | 334 | 3 |
| Zircon_35 | Bright core | 1,206 | 122 | 0.10 | 0.05478 | 0.00058 | 0.40695 | 0.00603 | 0.05388 | 0.00048 | 0.670 | 0.01682 | 0.00015 | 3 | 338 | 3 |
| Zircon_27 | Dark mantle | 1,074 | 94 | 0.09 | 0.05849 | 0.00060 | 0.53577 | 0.00657 | 0.06643 | 0.00038 | 0.530 | 0.02058 | 0.00013 | 5 | 415 | 2 |
| Zircon_20 | Bright core | 655 | 502 | 0.77 | 0.05887 | 0.00065 | 0.60345 | 0.00800 | 0.07417 | 0.00055 | 0.550 | 0.02518 | 0.00028 | 4 | 461 | 3 |
| Zircon_5 | Bright core | 792 | 280 | 0.35 | 0.05986 | 0.00060 | 0.67954 | 0.00896 | 0.08222 | 0.00071 | 0.650 | 0.02531 | 0.00030 | 3 | 509 | 4 |
| Zircon_29 | Bright core | 619 | 400 | 0.65 | 0.05861 | 0.00058 | 0.69521 | 0.00872 | 0.08589 | 0.00066 | 0.610 | 0.02732 | 0.00036 | 1 | 531 | 4 |
| Zircon_18 | Bright core-mantle | 289 | 171 | 0.59 | 0.06380 | 0.00083 | 0.87519 | 0.01284 | 0.09930 | 0.00068 | 0.460 | 0.03137 | 0.00044 | 4 | 610 | 4 |
| Zircon_40 | Bright core | 828 | 134 | 0.16 | 0.09248 | 0.00088 | 1.47753 | 0.01778 | 0.11587 | 0.00072 | 0.590 | 0.03412 | 0.00024 | 23 | 707 | 4 |
| Zircon_22 | Dark Zr | 880 | 112 | 0.13 | 0.18400 | 0.00149 | 7.17650 | 0.07683 | 0.28302 | 0.00198 | 0.650 | 0.04824 | 0.00063 | 25 | 1,607 | 10 |
| | | | | | | | | | | | | | | 2,134 | 10 | |
| | | | | | | | | | | | | | | 2,689 | 14 | |

^aSample analysed July 2010 on the LEP-UNAM LA-ICP-MS.^bU and Th concentrations (ppm) are calculated relative to analyses of trace-element glass standard NIST 612.^cIsotopic ratios corrected relative to Plesovice standard zircon (Sláma et al., 2008). Common Pb corrections made using the Andersen (2002) method.^dPercentage discordance obtained using the following equation ($100 \times [(age^{207}Pb/^{235}U) - (age^{207}Pb/^{238}U)] / (age^{207}Pb/^{235}U)$). Positive and negative values indicate normal and inverse discordance, respectively.

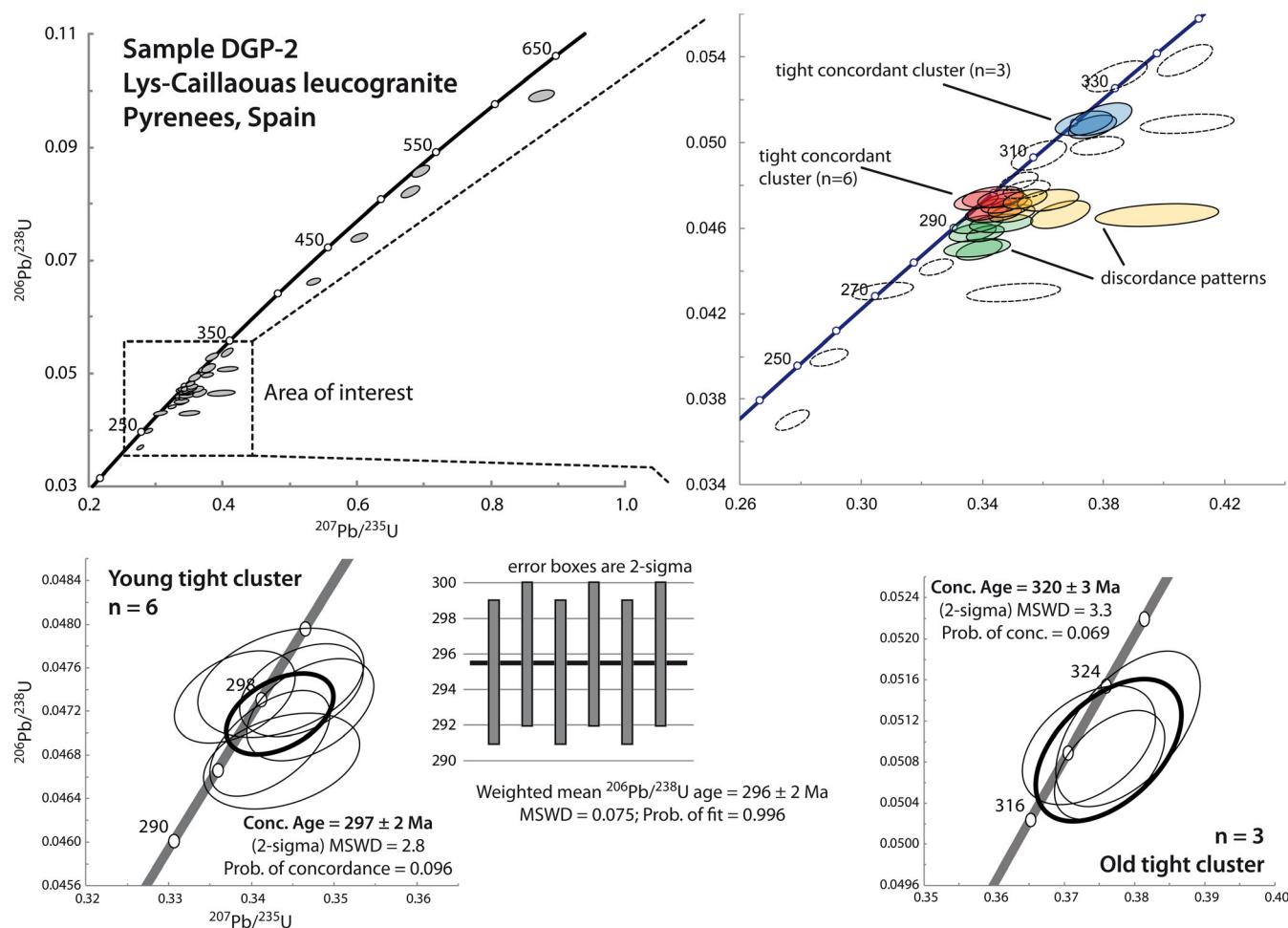


FIGURE 7 Wetherill and weighted average plots showing the different zircon dates in the sample DGP-02. Filled error ellipses are the ones used to estimate the different Concordia ages. Error ellipses and boxes are 2σ [Colour figure can be viewed at wileyonlinelibrary.com]

TABLE 3 U–Pb existing ages in the main metamorphic domes within the Axial zone

| Metamorphic dome | Early Carboniferous | | | | Late Carboniferous to early Permian | | | |
|------------------|---------------------|----------|------------------------------------|--------------------------|-------------------------------------|----------|-----------------------|-----------------------|
| | Num. | Age (Ma) | Method | Reference | Num. | Age (Ma) | Method | Reference |
| Lys-Caillaouas | 1 | 320 | Concordia zircon U–Pb ^a | This study | 3 | 297 | Concordia zircon U–Pb | This study |
| | | | | | | 299 | Concordia zircon U–Pb | Esteban et al. (2015) |
| | | | | | | 300 | Concordia zircon U–Pb | Esteban et al. (2015) |
| Garona | 2 | 338 | Concordia zircon U–Pb | Mezger and Gerdes (2016) | 1 | 295 | Concordia zircon U–Pb | This study |
| | 1 | 337 | Concordia zircon U–Pb | Mezger and Gerdes (2016) | | | | |
| | 1 | 326 | Lower intercept U–Pb | This study | | | | |
| Canigou-Carança | No found | | | | 1 | 305 | Concordia zircon U–Pb | Denèle et al. (2014) |
| Aston | 1 | 339 | Concordia zircon U–Pb | Mezger and Gerdes (2016) | 1 | 306 | Lower intercept U–Pb | Denèle et al. (2014) |
| Hospitalet | No data | | | | No data | | | |

^aFound in zircon cores and interpreted as inherited zircon grains (i.e., xenocrysts).

7 | CONCLUSIONS

Our data provide a crystallization age of 297 ± 2 Ma for the Lys-Caillaouas granitoid and, for the first time, an early Permian age of

295 ± 2 for the leucogranitic facies of the Bossòst granitoid. We also provide the first evidence of inherited zircon with early Carboniferous ages in the Lys-Caillaouas granitoid. Some discordant zircon grains in the Bossòst granitoid also suggest an early Carboniferous thermal

event recorded in pre-Variscan zircon. These data reinforce the idea that there was an elusive but widespread early Carboniferous magmatic event in the Pyrenees.

The crystallization age of Lys-Caillaous granitoid establishes an early Permian age for the HT-LP metamorphism peak in the Lys-Caillaous Dome. This discards two previous hypotheses that tried to explain the disparity of ages across different metamorphic domes in the Axial Zone: (i) a general Visean age for the HT-LP metamorphism or (ii) the diachronic formation of domes along the Axial Zone, ranging from Visean in the central Axial Zone to late Carboniferous in the eastern Axial Zone. The early Permian age found in the leucogranitic facies of the Bossòst granitoid questions the Visean age for the formation of Garona Dome and the HT-LP metamorphism, as proposed by Mezger and Gerdes (2016), and points to an Early Permian age as most likely, in line with what is observed in the Lys-Caillaous Dome.

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CONFLICTS OF INTEREST

The authors declare no conflict of interest.

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