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RESEARCH ARTICLE

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In situ U–Pb geochronology of Pre-Salt carbonates reveals links between diagenesis and regional tectonics

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Abstract

Identification of processes driving carbonate diagenesis requires an accurate chronology, particularly along volcanic margins where complex mineral parageneses develop. The Cretaceous "Pre-Salt" carbonates formed in the volcanic proto-South Atlantic and are now extensive hydrocarbon reservoirs offshore Brazil and Angola. Primary and diagenetic textures strongly affect reservoir properties, but timings of deposition (c. Barremian–early Aptian) and diagenesis are poorly constrained. This study provides the first direct age measurements from offshore Pre-Salt carbonates using in situ U–Pb geochronology. Depositional calcites from the Santos Basin (Brazil) do not preserve depositional ages, instead yielding an Albian recrystallization age of 106.9 ± 4.3 Ma. Dolomite rhombs and bridges have Palaeocene ages of 59.2 ± 5.3 Ma, and 58.0 ± 4.6 Ma, respectively. Calcite recrystallization was linked to the onset of oceanic spreading in the South Atlantic. Contrary to existing models, dolomites formed much later, during a time of enhanced tectono-magmatism in southeastern Brazil.

1 | INTRODUCTION

Interpreting the timing of carbonate diagenesis is critical to understanding the processes that drive the evolution of geochemical signatures, rock/water interactions and ultimately, geological resources. In volcanic rifted margins, this can be complicated by multiple spatio-temporally variable hydrothermal pulses during basin evolution. Authigenic carbonates are valuable paleoenvironmental indicators in these settings, but interpretations of depositional and diagenetic fabrics, and the corresponding geochemical signals, are often poorly constrained. In situ U–Pb dating, however, can provide necessary constraints (Roberts et al., 2020; Rochelle-Bates et al., 2021), and here this technique is applied to "Pre-Salt" carbonates to reveal tectono-magmatic controls on diagenesis.

The 'Pre-Salt' carbonates are a distinctive succession of Lower Cretaceous lacustrine lithologies, which formed in the Central South Atlantic during the late stages of rifting and the breakup of Gondwana (Abrahao & Warme, 1990; Karner & Gambôa, 2007; Saller et al., 2016; Schröder et al., 2016). These carbonates now host volumetrically-significant offshore hydrocarbon reservoirs in the Barra Velha Fm. of the Santos Basin, Brazil and the informally termed "Microbialite" of the Kwanza Basin, Angola (Figure 1).

The carbonates precipitated from highly alkaline lakes, with water chemistries influenced by volcanic catchment areas (Wright & Barnett, 2015). Resultant lithologies are notably distinctive and widespread, and comprise dominantly abiogenic and autochthonous crystal shrub limestones, Mg-clays with spherulitic calcite, and laminites (summarized in Wright & Barnett, 2015; Farias et al., 2019). The depositional vs. diagenetic origins of phases and their geochemical signals are not always clear, and there is limited understanding of the nature of diagenetic overprinting (Tosca & Wright, 2018). Scarce continental biota and radioisotopic Ar-Ar (volcanic) ages result in a poorly constrained bio- and chronostratigraphy: depositional ages have been traditionally regarded as approximately mid-Aptian (e.g., Chaboureau et al., 2013; Sabato Ceraldi & Green, 2017), but more recent biostratigraphic and chemostratigraphic studies suggest a late Barremian–early Aptian age of c. 127 Ma (Cazier et al., 2014; summarized in Tedeschi et al., 2017).

Diagenesis exerted an important control on reservoir properties but was complex with multiple phases of calcite, dolomite and silica cement precipitation, as well as dissolution events (Herlinger et al., 2017; Hosa et al., 2020; Lima & De Ros, 2019). Hydrothermal processes also created significant exploration risks (e.g., thermal cracking of organics, reservoir flushing; Loma et al., 2018). To date, the timing of diagenetic events in these Pre-Salt carbonates, and thus the responsible processes, have neither been isolated nor accurately quantified. Here, Laser Ablation Inductively Coupled Plasma Mass Spectrometry (LA–ICP–MS) U–Pb geochronology is used to directly measure the ages of primary and diagenetic phases in the Brazilian Pre-Salt carbonates, providing the first direct age constraints that allow inference as to regional tectonic controls.

Statement of significance

This study provides the first direct age measurements from economically significant South Atlantic "Pre-Salt" carbonates. Results from the offshore Santos Basin (Brazil) show that apparently well-preserved lacustrine fabrics were in fact recrystallized at the onset of oceanic spreading. Thus, geochemical data from similar phases should be interpreted with caution. It is inferred that this diagenetic event was widespread in the distal proto-South Atlantic, affecting the large area over which Pre-salt carbonate reservoirs occur. Contrary to existing models, dolomites precipitated much (c. 50 Myr) later, during an interval of intensified tectonomagmatic activity in Southeastern Brazil. These data demonstrate that widespread, notably complex, diagenesis was driven by far-field tectonomagmatic processes, and provide a new framework for interpreting mineral paragenesis in the South Atlantic Pre-Salt. Results therefore highlight important, margin-scale considerations for subsurface systems in the South Atlantic and volcanic rifted margins elsewhere.

2 | GEOLOGICAL SETTING

As rifting migrated northwards along the Central segment of the South Atlantic, the incipient grabens and their sedimentary fill transitioned to regionally extensive sag basins during the Barremian and Aptian. Lacustrine carbonate sedimentation in these sag basins was followed by the precipitation of thick Aptian evaporites during the onset of a major marine incursion (Sabato Ceraldi & Green, 2017).

The transition from rift to post-rift is poorly constrained, partly because the late stages of rifting occurred during the Cretaceous magnetic quiet zone (McElhinny & McFadden, 1998). Oceanic spreading between Brazil and Angola initiated in the early Albian (c. 110 Ma; Guiraud & Maurin, 1992; Davison, 1999; Torsvik et al., 2009; Chaboureau et al., 2013; Kukla et al., 2018), and it was closely followed by a major phase of later Albian salt translation in the subsurface, due to differential post-rift thermal subsidence (Duval et al., 1992; Heine et al., 2013; Jackson et al., 2005).

3 | MATERIALS AND METHODS

Three polished samples were analysed that contain representative examples of characteristic Pre-Salt depositional and diagenetic fabrics and carbonate mineral phases from a single Barra Velha Fm. core from the distal Santos Basin, offshore Brazil. The analysed phases include primary calcite shrubs (BV1-2), and primary calcite spherulites with diagenetic phases of dolomite bridges and rhombs within an Mg-clay matrix (BV3). Petrography was established using plane-polarized light and cathodoluminescence microscopy, then identified phases were analysed by LA-ICP-MS U–Pb geochronology following the method of Roberts et al. (2017). A static spot size of 80 μm was used to analyse inferred primary calcite (shrubs and spherulites); and 45 or 50 μm to sample diagenetic dolomite phases. All quoted ages are lower intercept $^{206}Pb/^{238}U$ dates from Tera-Wasserburg plots, at 2σ, with propagated systematic uncertainties. Regressions are unanchored as the spread in data permits the accurate assessment of the upper intercept (the initial $^{207}Pb/^{206}Pb$), and goodness of fit is evaluated using the mean square of weighted deviates (MSWD). Clear outliers are likely a consequence of analysing either inclusions, different phases or mixed phases, and these data are rejected from age regressions. Standard sample bracketing uses NIST 614 glass for normalization of $^{207}Pb/^{206}Pb$ ratios (Woodhead & Hergt, 2007), and a carbonate reference material (WC-1) for $^{206}Pb/^{238}U$ ratios (Roberts et al., 2017). An additional carbonate with existing isotope dilution radioisotopic data (Duff Brown Tank; Hill et al., 2016) was used to provide an extra control on accuracy and precision. In each analytical session, the resulting ages of Duff Brown Tank were within the uncertainty of the isotope dilution age provided by Hill et al. (2016) (full datasets, detailed methodologies and statistical criteria are provided in Data S1 and S2).

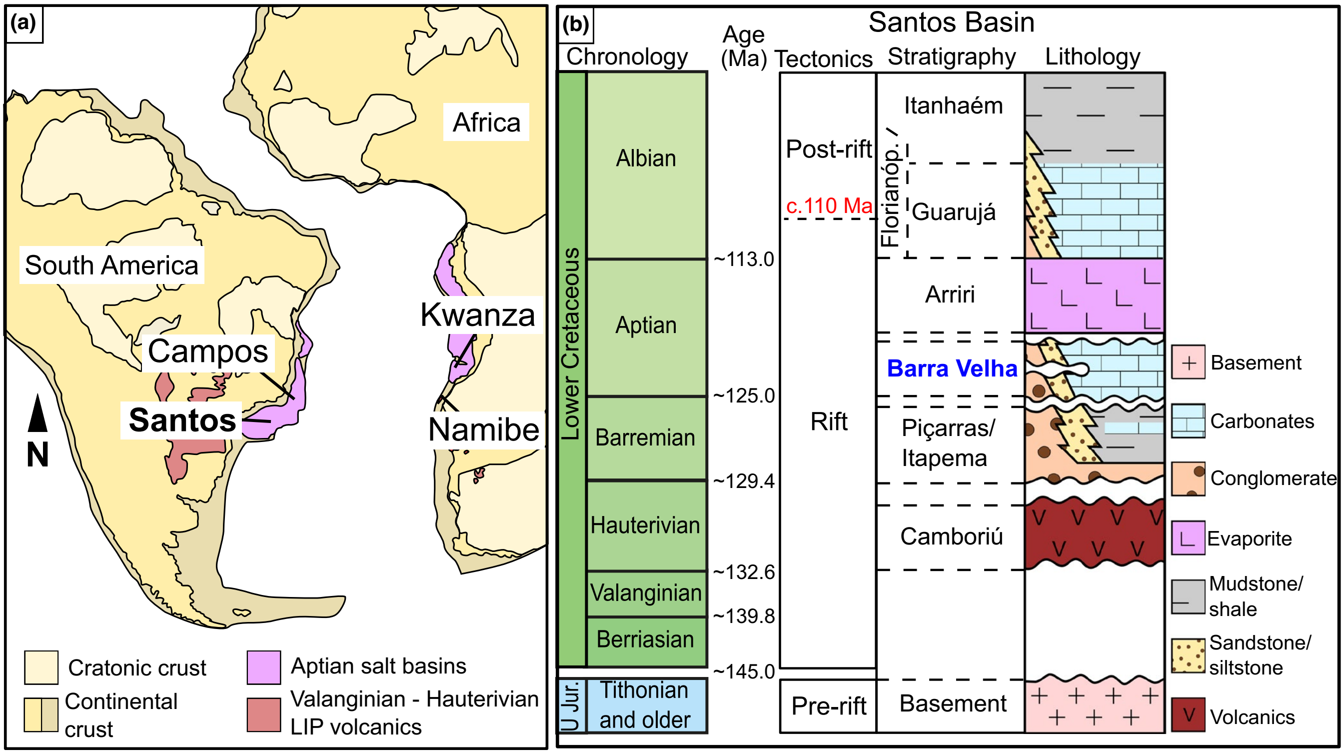

FIGURE 1 (a) Campanian plate reconstruction with conjugate Brazilian and Angolan basin locations after Torsvik et al. (2009). (b) Generalized stratigraphy for the Santos Basin (Brazil; after Moreira et al., 2007; Sabato Ceraldi & Green, 2017). Numerical ages are from Cohen et al. (2013; Updated)

4 | RESULTS

4.1 | Carbonate phases

Crystal-shrubs and spherulites are primary precipitates, probably originally of Mg calcite but now recrystallized to calcite (Saller et al., 2016). Internally, these fabrics are composed of radial-fibrous calcite crystal aggregates, with differences in the external geometry caused by different growth habits (e.g., Terra et al., 2010). Spherulites are symmetrical and typically less than 2 mm in diameter (Figure 2a). Shrubs are asymmetric, forming dense and bifurcating growths a few millimetres in length (Figure 2b).

Shrubby and spherulitic fibrous calcites both display cloudy (inclusion-rich) and clear (inclusion-poor) areas under plane-polarized light, and corresponding patchy dull to bright luminescence under CL, suggesting partial recrystallization (Figure 2a and b). Calcite spherulites are often surrounded by Mg-clays (e.g., stevensite), which contain authigenic dolomite bridges (sensu Wright & Barnett, 2015) and zoned rhombs (Figure 2C).

4.2 | Geochronological results

All U–Pb data for the calcite crystal-shrubs display high levels of scatter and generally high $^{207}Pb/^{206}Pb$ ratios (Figure 3). Successful

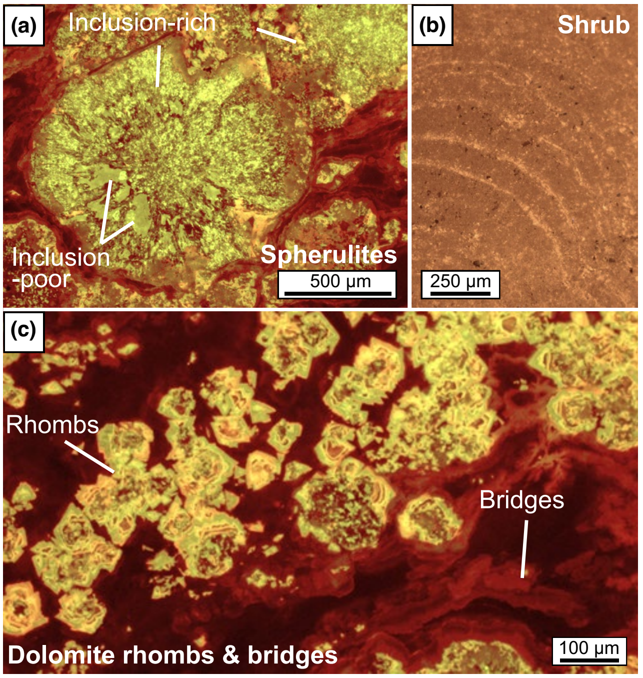

FIGURE 2 Cathodoluminescence images of dated phases from the Barra Velha Fm., Santos Basin, Brazil. (a) Calcite spherulites in mg-clay matrix. (b) Luminescence revealing growth banding in calcite shrub. (c) Dolomite bridges and rhombs floating in mg-clay matrix. Credit: Aleksandra Hosa.

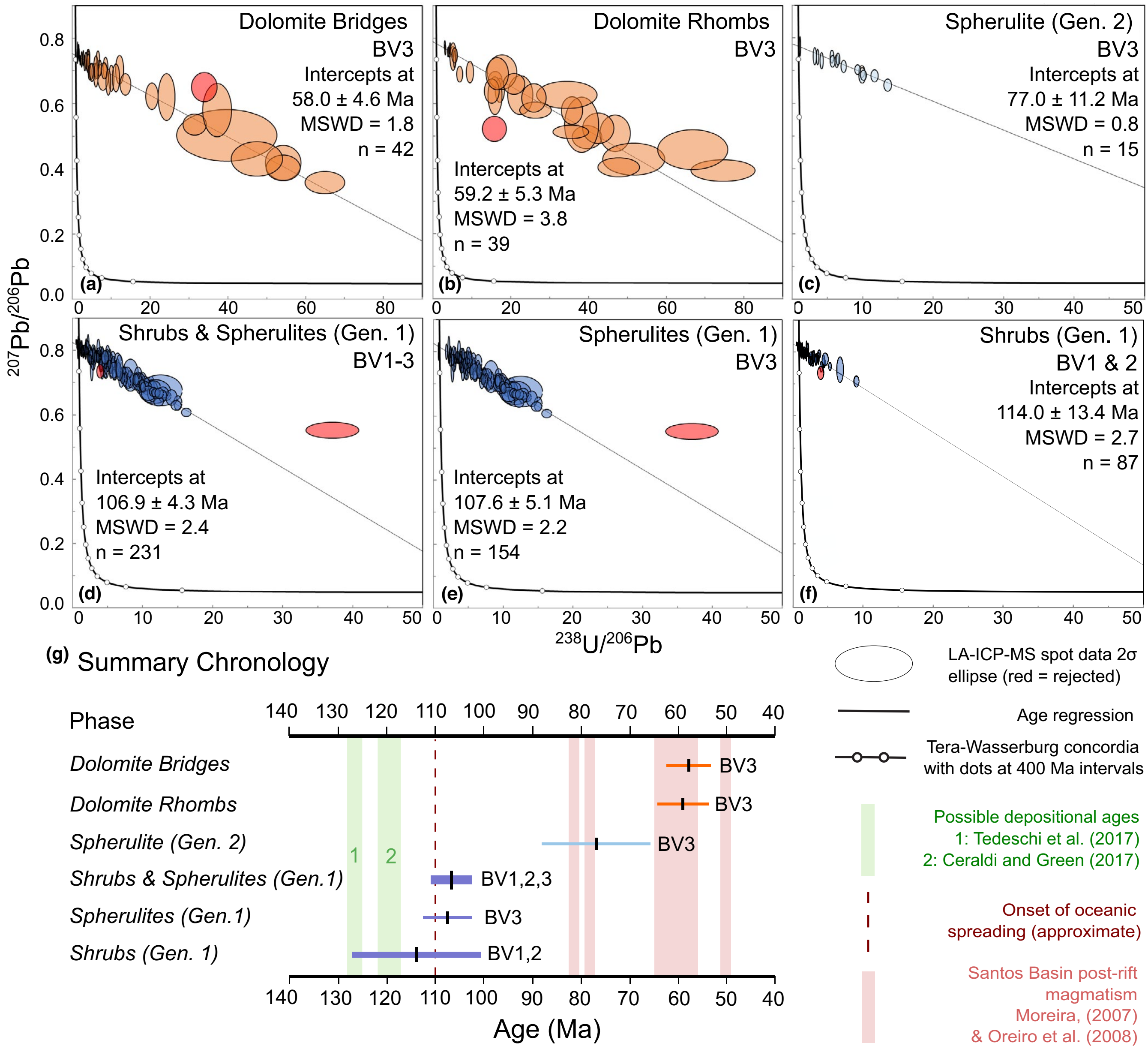

FIGURE 3 (a–f) Tera-Wasserburg concordia plots showing pooled $^{238}U/^{206}Pb$ versus $^{207}Pb/^{206}Pb$ data for carbonate phases from the studied Barra Velha Fm. Core. (g) Summary chronology shows age splits for the phases identified. Split thickness is scaled according to the number of samples that were pooled and sample numbers are listed next to the corresponding results. Quoted age uncertainty includes propagated systematic uncertainties. Datapoint error ellipses are 2σ. MSWD, mean squared weighted deviates

analyses of the shrub calcites in samples BV1 and 2 yielded ages of 107.0 ± 27.2 and 113.0 ± 15.3 Ma, respectively (Data S1, Figure S1). Spherulitic calcites in sample BV3 yield an age of 107.6 ± 5.1 Ma (Figure 3e), with a separate, notably younger spherulitic structure at 77.0 ± 11.2 Ma (Generation 2, Figure 3C). The pooled older primary calcite population (Generation 1) gives ages for all shrubs (114.0 ± 13.4 Ma; Figure 3f) and all calcite (106.9 ± 4.3 Ma; Figure 3D) (pooling criteria are given in Data S1, full datasets in Data S2).

Dolomite bridge and rhomb analyses (BV3) give ages of 58.0 ± 4.6 Ma (Figure 3a) and 59.2 ± 5.3 Ma (Figure 3b), respectively.

5 | DISCUSSION

5.1 | Diagenesis of fibrous calcite

Pooled primary calcite data for shrubs and spherulites yield an Albian (106.9 ± 4.3 Ma) age in the Santos basin. This is younger than the presumed Barremian–early Aptian depositional age of c. 127 Ma (Tedeschi et al., 2017). We interpret the calculated first-generation calcite ages to reflect diagenetic resetting of the U–Pb system, that is, recrystallization, during the Albian (Generation 1, Figure 3g). The degree of scatter in the data corresponds with the heterogeneity and degree of recrystallization inferred from petrographic observations, with shrubs displaying slightly more scatter overall. However, petrographic evidence of recrystallization does not directly correspond to differences in calculated age. Ages derived from areas of apparently very well-preserved, inclusion-rich fibrous calcite (Figures 2a, b and 3) are also diagenetic. This result demonstrates that care should be taken when interpreting "primary" geochemical signatures in these lithologies. Incompatible ages derived from some spherulites (see below) and locally high $^{207}Pb/^{206}Pb$ in some shrubs (see Data S1) suggest that recrystallization was heterogeneous, and higher levels of scatter in certain sample areas may be due to mixing between different age domains. Minor differences in recrystallization between the shrubs and spherulites could be caused by a variety of factors, including sedimentary geometries and micro-fracturing (e.g., Herlinger et al., 2017; Hosa et al., 2020).

The inferred recrystallization event coincides with the onset or early stages of oceanic spreading in the Central segment of the South Atlantic (Kukla et al., 2018). Both margins underwent a period of hyperextension with possible mantle exhumation during the late stages of rifting, followed by magmatic overprinting (Péron-Pinvidic et al., 2017). This magmatic pulse in the outer domains (sensu Peron-Pinvidic et al., 2013) facilitated final lithospheric breakup and occurs in both magma-rich and -poor settings (Bronner et al., 2011; Cannat et al., 2009; Jagoutz et al., 2007). Associated fluid-flow events and/or a spike in heat-flow during the onset–early stages of the post-rift could be responsible for the Albian (Generation 1, Figure 3) recrystallization ages recorded in this study. Enhanced thermal gradients would help to facilitate fluid circulation, possibly with exploitation of large detachment faults (e.g., Incerpi et al., 2020). Following the initiation of true oceanic spreading in the early Albian, rapid thermal subsidence (Duval et al., 1992; Jackson et al., 2005) may also have been a major driver of diagenesis within distal Pre-Salt successions. Rapid subsidence can lead to the generation and expulsion of overpressured basinal fluids, perhaps facilitated by fault and fracture reactivation as stress regimes reorganized (Frazer et al., 2014; Sibson, 1992). These processes would be most pronounced close to the incipient spreading centre and/or along deep-seated detachment faults. Indeed, radioisotopic ages indicate widespread Albian fluid flow and diagenesis in the distal Pre-salt. Hydrothermal (90–170°C) activity was dated in the Campos Basin with adularia $^{40}Ar/^{39}Ar$ ages of 107.0 ± 1.0–104.3 ± 0.8 Ma (Tritlla et al., 2018), while basalt alteration $^{40}Ar/^{39}Ar$ ages of 114.5 ± 2.9 to 106.9 ± 1.6 Ma were used to constrain a hydrothermal (c. 140°C) pulse in the Kwanza Basin (Loma et al., 2018).

We also detect a separate calcite phase that is interpreted as a younger, relatively minor phase of recrystallisation (Generation 2, Figure 3g) at 77 ± 11.2 Ma, although it cannot be discriminated petrographically. This 77 Ma age overlaps with well documented Late Cretaceous igneous activity and associated structural reactivation that occurred in both Brazil and Angola (e.g. Fiordalisi et al., 2021; Guedes et al., 2005; Jerram et al., 2019; Moreira et al., 2007; Rochelle-Bates et al., 2021). It is, therefore, possible that fluid flow or elevated temperatures related to this event caused localized further recrystallization of the samples studied here.

5.2 | Dolomite rhombs and bridges

Dolomite phases yield identical late Palaeocene–Eocene ages, 59.2 ± 5.3 and 58.0 ± 4.6 Ma for rhombs and bridges, respectively. These dolomites provide the most robust age data, partly due to a large spread in the recorded U and Pb isotopic compositions (Figure 3a,b). The higher level of scatter in the rhomb data may be due to minor contamination from the Mg clay matrix, or due to slightly different age or compositional domains within the rhombs themselves.

Results indicate that dolomite precipitation occurred via late diagenetic processes, almost certainly involving stevensite destabilization due to enhanced thermal energy and/or reorganization of basinal fluids (e.g., Tosca & Wright, 2018). This may have been supplied via deeper burial and/or by a well-documented period of intensified tectonic and magmatic activity during the Palaeocene–Eocene in southeastern Brazil (e.g., Oreiro et al., 2008). Bitumen-associated vein dolomite of Palaeocene–Eocene age are reported (Rochelle-Bates et al., 2021) from the Pre-Salt of the onshore Namibe Basin (Figure 1), formed during a similar magmatic/tectonic event on the conjugate African margin. This event may have contributed to outliers in the Brazil calcite data that were not used for age regressions, but it had little impact overall, reflecting the relative stability of the already recrystallized calcite phases.

Dolomite rhombs in other offshore Pre-Salt cores have been regarded as either a product of early Mg-silicate diagenesis (Farias et al., 2019; Herlinger et al., 2017; Lima & De Ros, 2019) or primary (syn-depositional) lake precipitates (e.g., Saller et al., 2016). Results of this study suggest that dolomite precipitation occurred, at least locally, over a much longer diagenetic time window.

6 | SUMMARY

Diagenesis of the Central South Atlantic Pre-Salt was complex and multi-phase, and our data suggest that this was driven by regional tectonic and igneous processes. By employing the recently developed in-situ U–Pb LA-ICP-MS technique, we produced the first direct age measurements for sub-surface Pre-Salt carbonate phases and identified hitherto unknown, far-field processes that drove diagenesis.

Analyses of shrubby and spherulitic fibrous calcites from the Santos Basin do not yield depositional (c. early Aptian) ages. Instead, the calculated ages point to Albian diagenetic resetting of the calcite U–Pb system via recrystallization. The ages of recrystallization coincide with the onset–early stages of oceanic spreading in the Central South Atlantic, and an associated heat spike and/or associated fluid flow in the distal sag basins may have triggered diagenetic resetting.

This major event may have been followed by minor recrystallization in the Late Cretaceous, which also coincides with a well-known magmato-tectonic event.

By contrast, authigenic dolomite rhombs and bridges have a far younger Palaeocene (Selandian–Thanetian) age. This coincides with a period of enhanced tectonism and magmatism in southeastern Brazil, which may have driven Mg-clay destabilization and hence dolomite precipitation.

We expect that the tectono-magmatic events identified here had wide-reaching, albeit spatially variable influences on carbonate diagenesis in the distal Central South Atlantic. This is further suggested by limited, but complimentary radioisotopic data from elsewhere on the Brazilian and Angolan margins (Loma et al., 2018; Tritlla et al., 2018). Our results, therefore, highlight important, margin-scale considerations for sub-surface systems in the South Atlantic and volcanic margins elsewhere.

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DATA AVAILABILITY STATEMENT

All data used in the paper are given as Supplementary Data in the Data S1 and S2.

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