

## RESEARCH ARTICLE

# Dating Two Successive Rifts of the Equatorial Atlantic From the Sediments of the Buteur Ridge, Demerara Plateau (French Guiana)

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## ABSTRACT

The permanent oceanic connection between the Jurassic Central Atlantic and the Cretaceous South Atlantic was only established with the late opening of the Equatorial Atlantic, but the timing of this event remained unclear due to a lack of geological dating. In 2023, the DIADEM oceanographic cruise conducted sampling of the Buteur Ridge, offshore French Guiana, by dredging and during a manned deep submersible (*Nautilie*) dive. The Buteur Ridge belongs to the eastern rifted margin of the Demerara Plateau, formed during the Lower Cretaceous Equatorial Atlantic rift. It is a 7 km-long and 6 km-wide tilted block, located at a depth of 3750 m, where sedimentary records of the Equatorial Atlantic are outcropping, making it an ideal location for investigating the timing of this rift.

We integrate petrological observations, biostratigraphy, fission-track analyses of detrital apatites and zircons, and LA-ICP-MS U–Pb dating of authigenic apatites to reconstruct the sedimentary, diagenetic and thermal history of the sediments sampled on the Buteur Ridge.

Our results constrain the tectono-sedimentary evolution of the divergent margin of the Demerara Plateau, revealing a complex rifting history of the Equatorial Atlantic involving two distinct rifts. The onset of the first rift occurred between 130 and 125 Ma (Hauterivian), which is earlier than the previously proposed Aptian age. A second rifting then occurred during the Cenomanian, likely during the kinematic reorganisation between Africa and South America in the Equatorial Atlantic. This later rifting is evidenced on the Buteur Ridge by terrigenous sedimentation followed by telodiagenesis during basin inversion, likely related to normal fault reactivation and ridge uplift. We interpret the crystallisation of authigenic apatites, dated  $93 \pm 12$  Ma, as a record of the subsequent onset of marine transgression at the end of this second rifting, likely not occurring after the Late Cenomanian (circa 93 Ma).

## 1 | Introduction

The opening of the Equatorial Atlantic is a consequence of an E–W divergence between Africa and South America that began during the Early Cretaceous and culminated in the final separation of these continents (Campan 1995; Mascle et al. 1986;

Moulin et al. 2010; Pindell and Dewey 1982). In this process, a connection was formed between the Central Atlantic domain, which opened since the Late Sinemurian (199.5–192.9 Ma; Labails et al. 2010; Sahabi et al. 2004) to the north, and the South Atlantic domain to the south (Campan 1995; Heine et al. 2013; Moulin et al. 2010). The timing and evolution of the Equatorial

## Highlights

- Sandstones were sampled on the Cretaceous divergent margin of the Demerara Plateau (Buteur Ridge)
- Sedimentary and thermal history evidences two-rifting stage of the Equatorial Atlantic
- The onset of the first rift stage of the Equatorial Atlantic rift occurred during the Hauterivian
- The second rift stage began during the Cenomanian and likely ended during the Late Cenomanian

Atlantic oblique rift remain insufficiently constrained due to the limited availability of outcrops and samples documenting this event. The onset of this rifting is hypothesised to have occurred during the Barremian-Aptian (125.8–113.2 Ma; e.g., Heine et al. 2013; Moulin et al. 2010), with seafloor spreading proposed to have occurred from the Late Aptian (Basile et al. 2005) to the Early Cenomanian (Eagles 2007). Since Campan's (1995) study, a kinematic reorganisation, due to a change in the position of the rotation pole between Africa and South America, is inferred after the rifting based on the geometry of the oceanic fracture zones (Heine et al. 2013; Lesourd-Laux et al. 2025; Moulin et al. 2010). Indeed, the first opening phase, which was restricted to the northern part of the Equatorial Atlantic (North of St Paul fracture zone), is associated with the WNW-ESE-trending fracture zones in the Demerara region (Lesourd-Laux et al. 2025). The second opening phase corresponds to the W-E-trending fracture zones (Campan 1995; Heine et al. 2013; Lesourd-Laux et al. 2025; Moulin et al. 2010). However, as this kinematic reorganisation occurred during the magnetic Cretaceous quiet zone, its age is not constrained by data, and estimations range from 104 Ma (Campan 1995), 106 Ma (Moulin et al. 2010; Lesourd-Laux et al. 2025) to 110 Ma (Heine et al. 2013).

During the Equatorial Atlantic opening, the Demerara and Guinea plateaus (Figure 1a) represent its northernmost part, at the junction with the Central Atlantic. These plateaus acted as the last land bridge between the two continents (Loncke et al. 2022), and represent a critical region for understanding the opening of the Equatorial Atlantic Ocean (Basile et al. 2005, 2022; Benkheilil et al. 1995; Gouyet 1988; Graindorge et al. 2022; Loparev et al. 2021; Olyphant et al. 2017; Reuber et al. 2016).

The Demerara Plateau, located between 1000 and 3000 m depths, is a bathymetric extension, 400 km offshore, of the continental margin of French Guiana and Suriname (Figure 1b). It is a transform marginal plateau resulting from a two-rift history (Basile et al. 2013; Gouyet et al. 1994; Graindorge et al. 2022; Loncke et al. 2020; Loparev et al. 2021; Mercier De Lépinay et al. 2016; Reuber et al. 2016). The western divergent magmatic margin of the Demerara Plateau was part of the Central Atlantic rifting (Basile et al. 2013, 2020; Graindorge et al. 2022; Loncke et al. 2020; Loparev et al. 2021; Mercier De Lépinay et al. 2016; Pindell 1985; Reuber et al. 2016) (Figure 1b). The Equatorial Atlantic rift formed the dextral transform margin to the north and the divergent margin to the east of the Demerara Plateau (Basile et al. 2005, 2013, 2022; Campan 1995; Gouyet et al. 1994; Greenroyd et al. 2008; Loncke et al. 2022; Mercier de Lépinay et al., 2016; Museur et al. 2021; Nemčok et al. 2015) (Figure 1b).

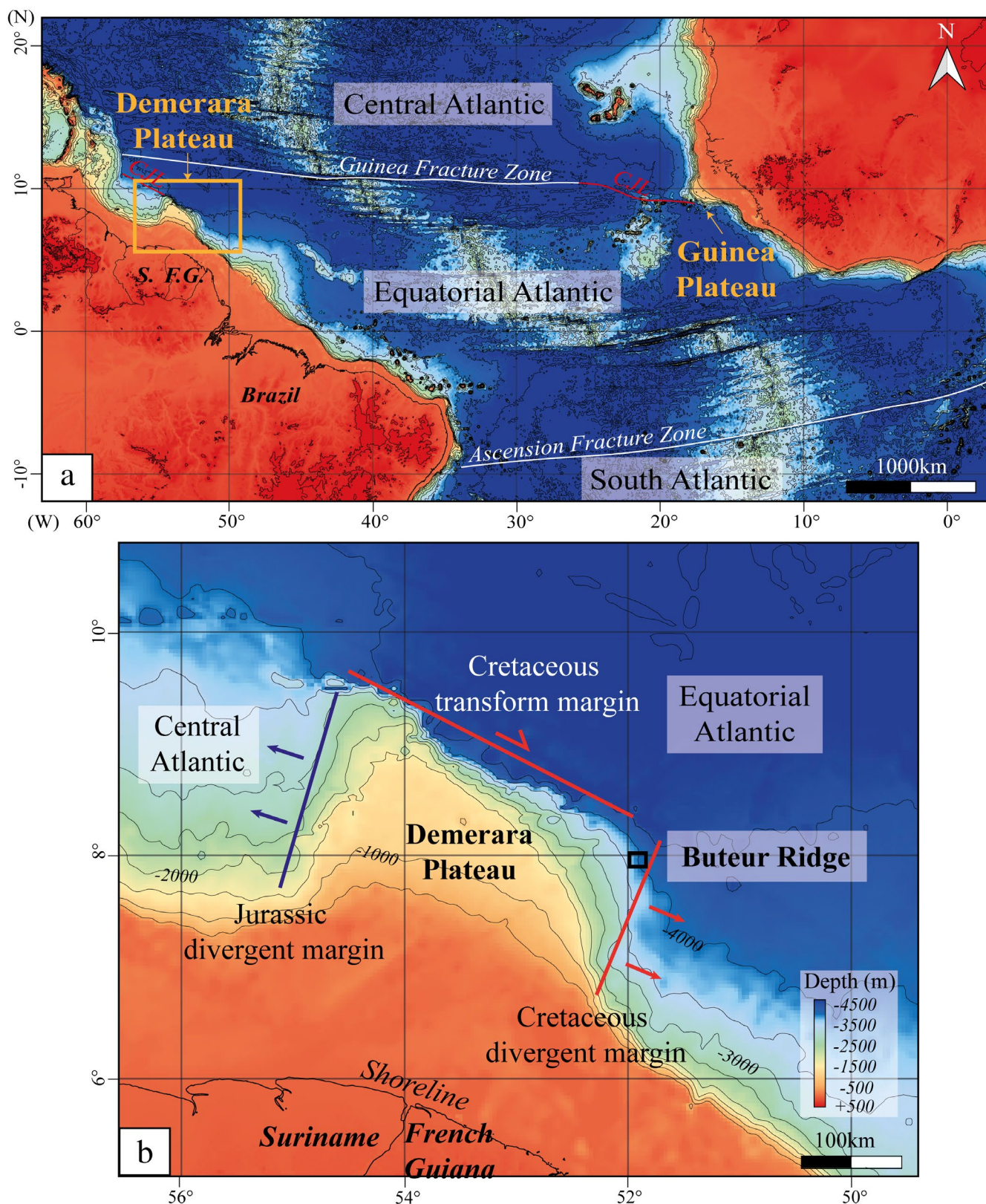
The stratigraphic record on the Demerara Plateau (e.g., Casson et al. 2021) can therefore be divided into syn-Jurassic-rift sequences, mainly lava flows observed on seismic lines as Seaward Dipping Reflectors (Reuber et al. 2016), post-Jurassic-rift Upper Jurassic to Lower Cretaceous carbonate platform sediments, syn-Cretaceous-rift (Barremian-Aptian) sequences and post-Cretaceous-rift sequences including a sub-aerial Late Albian regional unconformity.

The structure of the eastern divergent margin has been studied previously using bathymetric surveys, seismic reflection and seismic refraction data from the GUYAPLAC (Le Suave and Beuzart 2003), IGUANES (Loncke 2013) and MARGATS (Graindorge and Klingelhoefer 2016) campaigns, revealing westward-tilted blocks associated with the Lower Cretaceous (Aptian) rift (Basile et al. 2013; Loncke et al. 2022; Sapin et al. 2016). These campaigns also identify a 3750 m-deep outcropping acoustic ridge, named the Buteur Ridge, which is bounded on its western side by a normal fault (Basile et al. 2022; Coudun et al. 2025; Girault et al. 2023; Graindorge et al. 2022; Museur et al. 2021) and where a single azoic sandstone sample was dredged from the eastern slope during the DRADEM cruise (2016) (Basile 2016). This sample was included in a source-to-sink study by Girault et al. (2023). However, the model presented in Girault et al.'s study suffers from the scarcity of sufficient data and the inability to stratigraphically locate the collected samples, thus preventing a detailed reconstruction of the tectono-sedimentary history of the margin.

As part of the DIADEM oceanographic research campaign (Basile and Loncke 2023), sedimentary series from the Buteur Ridge were sampled by dredging and *Nautilie* dive in January 2023 aboard the French oceanographic vessel *Pourquoi Pas?*. Coudun et al. (2025) proposed a stratigraphic and tectonic framework for the section covered by the *Nautilie* dive (cf. following Section 2). Our paper presents the petrological, geochronological, and thermochronological analyses of the Buteur Ridge samples collected during the DIADEM campaign. Combined biostratigraphic and fission-track analyses enable the precise determination of depositional ages within a stratigraphic framework, thereby facilitating the identification of tectonic events associated with the margin formation and improving our understanding of the Equatorial Atlantic rifting.

## 2 | Study Site and Material

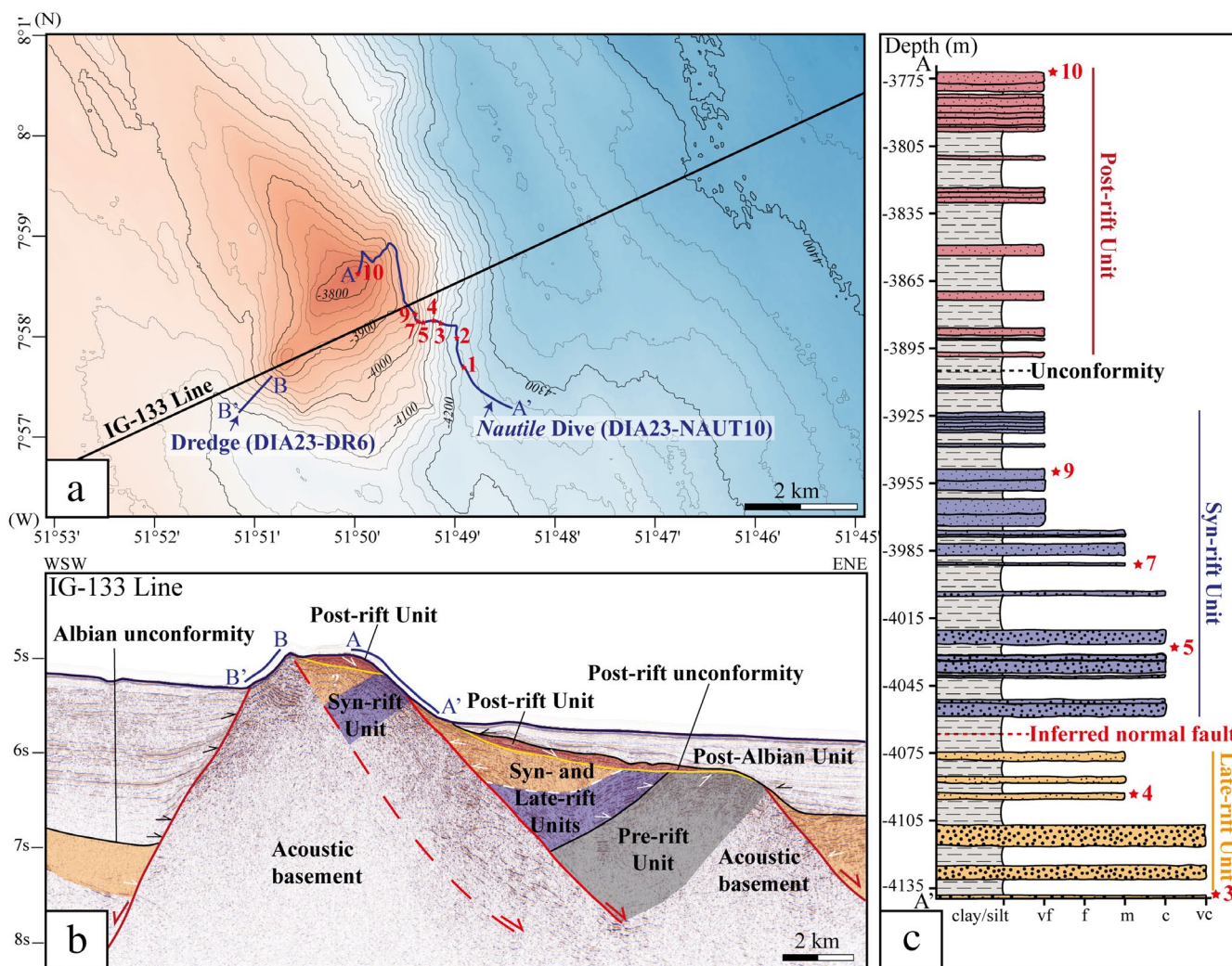
The Buteur Ridge is a 7 km—long and 6 km—wide relief, located at the junction of the northern and eastern margins of the Demerara Plateau (Figure 1b). It was first identified as an NNW-SSE-oriented acoustic basement ridge, rising to a depth of 3750 m, based on vertical and wide-angle seismic data acquired during the GUYAPLAC (Le Suave and Beuzart 2003), IGUANES (Loncke 2013) and MARGATS (Graindorge and Klingelhoefer 2016) campaigns. The Buteur Ridge is bounded to the west by a normal fault (Basile et al. 2022; Girault et al. 2023; Graindorge et al. 2022; Museur et al. 2021). During the DRADEM campaign (Basile 2016), the DRA-F1 dredge was deployed on the southeastern slope of the Buteur Ridge, at a depth of 4200–4000 m and sampled a coarse azoic quartz-cemented sandstone. This sample was thought to have



**FIGURE 1** | (a) Topographic map (background from Etopo1) of the Equatorial Atlantic and location of the Demerara and Guinea plateaus. Isobaths every 500 m. S., Suriname; F.G., French Guiana; CJL, Cretaceous Jurassic Line. (b) Topographic map (background from Etopo1) of the Demerara Plateau and location of the Buteur Ridge. Isobaths every 500 m.

originated from submarine screes (Girault et al. 2023). The crystallisation ages of detrital zircons from this sample, determined by LA-ICP-MS U–Pb dating, showed a primary mode at

600–700 Ma formed during the Brasiliano–Pan-African orogeny, whereas the zircons fission-track cooling ages were determined to be  $106 \pm 8$  Ma and  $221 \pm 12$  Ma (Girault et al. 2023).



**FIGURE 2** | (a) Topographic map of the Buteur Ridge (background from the IGUANES, DRADEM and MARGATS cruises: Loncke 2013; Graindorge and Klingelhoefer 2016; Basile 2016). Isobaths every 25m. Line AA' indicates the location of the Nautille dive path. Line BB' indicates the location of the dredge path. The red stars indicate the location of the Nautille samples. (b) Stratigraphic interpretation of seismic line IG-133 from IGUANES cruise (Loncke 2013) (adapted from Coudun et al. 2025). Vertical exaggeration: 4.1. Normal faults are indicated by the red lines. Lines along the Buteur Ridge flanks on seismic line indicate the projected Nautille dive and dredge paths. (c) Stratigraphic log of the eastern slope of the Buteur Ridge, along Nautille dive path (A'-A) (adapted from Coudun et al. 2025). The red stars indicate the sample locations. vf, very fine sand; f, fine sand; m, medium sand; c, coarse sand; vc, very coarse sand.

The source-to-sink model proposed by Girault et al. (2023) suggests that the sediments observed on the Buteur Ridge result from the erosion of the Precambrian basement of the French Guiana margin and the eastern Demerara Plateau during the early stages of the Equatorial Atlantic rifting. The eroded material, consisting of sediments derived from Brasiliano-Pan-African orogenic rocks, was therefore channelled through the Cassipore Graben towards the Demerara Plateau (Girault et al. 2023).

During the DIADEM campaign (Basile and Loncke 2023), the dredge DIA23-DR6 was deployed on the southwestern slope of the Buteur Ridge at depths between 4100 and 3920 m (Figure 2a), recovering a total of 87 siliciclastic samples. In addition, a Nautille submersible dive, DIA23-NAUT10, was conducted along the eastern slope of the Buteur Ridge, between depths of 4256 and 3761 m (Figure 2a). This dive collected eight stratigraphically in-place sandstone samples and

provided 6 h of continuous video, used by photogrammetry to build 3D models of the outcrops (Coudun et al. 2025). More details regarding the sample locations are provided in Table S1.

Based on structural measurements, Coudun et al. (2025) inferred an east-dipping normal fault along the eastern slope of the Buteur Ridge, defining a tilted block with three outcropping structural units (Figure 2b). At the bottom, the first unit (samples DIA23-NAUT10-03 and -04) is located on the hanging wall of the fault, dips to the east, and is interpreted as part of a syn-rift sequence (Figure 2b,c). Upslope, the second unit (samples DIA23-NAUT10-05, -07 and -09), which dips west, is also interpreted as a syn-rift unit on the footwall of the fault (Figure 2b,c). The dip difference between the two units is interpreted as due to fault drag. In these units, structural measurements suggest a northward progradation during sedimentation.

At the top of the Buteur Ridge, the third unit dips to the east and is interpreted as a post-rift unit separated from the syn-rift units by an erosional unconformity (Figure 2b). This unconformity truncates the syn-rift units and is interpreted to be slightly older than the Late Albian unconformity (Coudun et al. 2025). Sample DIA23-NAUT10-10 belongs to this third unit (Figure 2c). Samples DIA23-NAUT10-01 and -02, collected from massive rockfalls at the base of the eastern slope, may have originated from the third (post-rift) unit. It is noteworthy that the present-day morphology of the Buteur Ridge postdates the deposition of the post-rift unit, indicating late uplift supposed to be associated with the reactivation of the normal faults (Coudun et al. 2025).

## 3 | Methods

### 3.1 | Microfacies Analysis

Covered and uncovered 30- $\mu$ m thick thin sections of the eight samples from the DIA23-NAUT10 dive (Table S2) were prepared: at the Geo-Ocean Laboratory, Brest University, France for the uncovered thin sections and at the EPOC Laboratory, Bordeaux University, France for the covered thin sections. Sixteen samples (Table S2) representing most of the macroscopic facies found in dredge DIA23-DR6, were also selected for thin sections. Ten covered thin sections were prepared at the EPOC Laboratory and six uncovered thin sections were made at the Laboratorio Petrografico, Quart, Italy. All thin sections were systematically analysed at 25 $\times$ , 50 $\times$ , 100 $\times$  and 500 $\times$  magnifications under a petrographic polarised light microscope (Olympus BH2).

Additional analyses were carried out using a Vega3 Tescan Scanning Electron Microscope (SEM) and its Energy Dispersive X-ray Spectroscopy (EDS) probe at the Institute of Earth Sciences (ISTerre), Grenoble Alpes University, France on six samples from the *Nautilite* dive and on six dredge samples (Table S2).

In addition, the bulk mineralogical composition of the dive samples and eight samples from the dredge were evaluated by X-Ray Diffraction (XRD) analysis using a Bruker D8 X-ray diffractometer at ISTerre (Tables S2–S4). Following identification of the mineral phases, sample mineralogy was assessed quantitatively with the Rietveld program BGMN and the Profex user-interface (Doebelin and Kleeberg 2015). Clay minerals were grouped by families (kaolinite, micas, chlorites and swelling 2:1). To characterise the clay mineralogy further, the <2 $\mu$ m fraction was separated by centrifugation for five dredge samples (Tables S2 and S5), and oriented slides were prepared by pipetting a clay slurry on a glass slide. XRD data were recorded under air-dried conditions at 75% relative humidity (RH) in order to maximise hydration homogeneity of the expandable interlayers, and at 10% RH following ethylene glycol solvation. Swelling 2:1 minerals were identified further by fitting profiles of XRD patterns using both discrete phases and interstratifications containing expandable layers (e.g., chlorite/expandable, corrensite and/or mica/expandable).

Three pelitic dredge samples and one sandstone sample from the *Nautilite* dive were used for palynology, micro- and nannofossil biostratigraphic analysis (Table S2).

## 3.2 | Apatite and Zircon Fission-Track Analysis

Section I and Section II of the [Supporting Information](#) provide details regarding the fundamentals of apatite and zircon fission-track analysis and their interpretation.

### 3.2.1 | Sample Preparation

Coarse sandstone samples from the dive and dredge in sufficient quantity were selected for crushing and separation of apatites and zircons (Table S2). Samples were prepared at the ISTerre Geo-ThermoChronology (GTC) platform. Samples were crushed in a jaw crusher and disc pulveriser to break up the rocks and release the grains. Grains were sieved to a 200- to 80- $\mu$ m fraction. The heavy minerals, apatite (mineral density of 3.1–3.3 g/cm<sup>3</sup>) and zircon (mineral density of 4.5–4.6 g/cm<sup>3</sup>), were concentrated using a separation table, and separated using standard magnetic and heavy liquid separation techniques (Kohn et al. 2019). Three samples (one from the *Nautilite* dive, two from the dredge) yielded both a sufficient number of zircons and apatites (100 grains or more, as defined by Vermeesch 2004) for fission-track analysis (Table S2). Two other *Nautilite* samples contained a sufficient number of zircons for fission-track analysis, but no apatite (Table S2).

### 3.2.2 | Cooling Age Calculation

ZFT and AFT analyses were carried out at the ISTerre Geo-ThermoChronology Fission-Track Laboratory. The objective was to analyse up to 100 grains per sample in order to approximate the representative cooling age spectrum of a detrital rock sample (Vermeesch 2004). For each sample, 800 detrital zircon aliquots were manually picked and mounted in two different Teflon sheets (400 aliquots for each). As the samples were poorer in apatite, three mounts were prepared containing respectively 70 apatites for DIA23-DR6-16, 100 apatites for DIA23-DR6-18 and 400 apatites for DIA23-NAUT10-07. The apatite grains were manually picked and mounted in epoxy resin. Both zircon and apatite mounts were then polished to expose the inner surface. For detrital zircons, a multi-mount approach is recommended to reveal countable fission-tracks across the whole grain age spectrum (e.g., Naeser et al. 1987; Bernet et al. 2004). Therefore, in order to follow this approach, the two mounts of each sample were not exposed to the same chemical attack times. The first mounts were etched between 18 and 22 h and second were etched between 23 and 27 h in a NaOH-KOH melt at 228°C in a laboratory oven. The apatite surfaces were etched at 21°C for 20 s in a 5.5 mol HNO<sub>3</sub> solution as described by Donelick (2005). Once the fission-tracks were etched, we used the External Detector Method (EDM) (Fleischer et al. 1964; Naeser and Dodge 1969) and we covered the Teflon and epoxy resin mounts with low-U muscovite sheets. The samples were irradiated at Oregon State University together with IRMM541 (50 ppm U) dosimeter glasses and Fish Canyon Tuff age standards (Naeser et al. 1981) for zircons and IRMM540R (15 ppm U) dosimeter glasses and Durango (McDowell et al. 2005) and Fish Canyon Tuff age standards for apatites. After irradiation, the mica detectors were etched

in 48% hydrofluoric acid for 18 min at 20°C to reveal induced fission-tracks.

Samples were analysed and fission-tracks were counted at 1250× magnification using an Olympus BH2 optical microscope for zircons, an Olympus BX51 optical microscope, for apatites, and the FTStage 4.04 system (Dumitru 1993). The Zeta factor calibration (Hurford and Green 1983) was employed to determine the individual fission-track ages. Cooling ages were calculated following Galbraith and Green (1990) and Galbraith and Laslett (1993), using the automatic mixture model in RadialPlotter version 9.5 software (Vermeesch 2009). Tables S6–S8 provide the zircon and apatite track count data.

### 3.3 | In Situ LA-ICP-MS U–Pb Apatite Dating

Sample DIA23-DR6-18-12 contains authigenic apatites within the rock porosity. Prior to the analyses, the observation of the thin section was carried out using optical microscopy and scanning electron microscopy (BSE, CL) to reveal structural and chemical microstructures (crystal morphology, growth textures and zoning), providing suitable target areas for laser ablation.

U–Pb analyses were conducted in a single analytical session at the ISTerre Micro-Analytical Platform (IMAP), with a LA-ICP-MS setup comprising a RESOLUTION SE (Applied Spectra) 193 nm ArF excimer laser (Atlex-LR—ATL Lasertechnik) equipped with a Laurin Technic S155 sample cell and coupled to an Agilent 8900 mass spectrometer operated in single-quadrupole mode. Studied apatites and Reference Materials (RM) were ablated using a static spot ablation mode set to 24 µm diameter, 6 Hz repetition rate, and a fluence of ~1.5 J/cm<sup>2</sup>. More details regarding the LA-ICP-MS setup (e.g., pre-ablation cleaning, gas flows, ICP-MS tuning, raw ICP-MS acquisition) are provided in Table S9.

NISTSRM612 glass (Jochum et al. 2011) and three reference apatites (Madagascar apatite MAD2, 474.25 ± 0.41 Ma, ID-TIMS, Thomson et al. 2012; Morocco apatite MRC-1, 153.4 ± 0.4 Ma, ID-TIMS, Apen et al. 2021; and Brazil apatite BRZ-1, 2078 ± 12 Ma, ID-TIMS, Apen et al. 2021) were analysed alongside the studied material with a standard-bracketing approach, which allows monitoring and correcting for any temporal variation of the instrumental response, especially the relative sensitivity of lead and uranium.

In details, NISTSRM612 was used as the external calibrant to correct for <sup>207</sup>Pb/<sup>206</sup>Pb relative sensitivity (mass bias) and monitor U–Pb drift, while MAD2 apatite was used to correct for uranium and lead relative sensitivity, affected mainly by downhole, matrix-specific, fractionation. The two fold calibration scheme involved two steps of processing, respectively: (1) raw acquisitions were first processed using the Iolite-4 software package and the ‘U–Pb geochronology’ data reduction scheme (Paton et al. 2011) without any common lead correction, then (2) an in-house Excel sheet is used to perform the matrix-match U–Pb calibration (e.g., Roberts et al. 2017). MAD2 apatite was used as the primary reference material.

MRC-1 apatite and BRZ-1 apatite were used as secondary/validation materials. Determination of dates are performed using a weighted bivariate regression (model 1 of IsoplotR, see also Vermeesch 2018 and York et al. 2004) in the Tera-Wasserburg semi-total lead diagram, negating the necessity of common-lead correction. The reported date uncertainty follows the recommended workflow of Horstwood et al. (2016) and includes an internal uncertainty as given by IsoplotR, and a systematic uncertainty, that includes the long-term variance estimated by the laboratory with validation materials ( $\epsilon' = 0.5\%$ , 2s). The results of the data analysis of the standards and the sample are reported in Table S10.

## 4 | Results

### 4.1 | Microfacies

Five microfacies (Table 1) were determined by analysing the granulometry, grading, mineralogical assemblage, compaction and cementation of the rocks.

#### 4.1.1 | Facies 1: Pelites

Most of the DIA23-DR6 samples present a pelitic facies (Table 1). Macroscopically, many samples contain plant impressions or fragments (Figure 3a). Some show slight bioturbation with burrows indicating surface reworking while others display evidence of faulting such as slickenside striations. Microscopically, this facies is characterised by centimetric beds of very fine sands and coarse silts (10–150 µm in size) alternating with beds of very fine silts and clays (Figure 3b). Bedding is predominantly flat and parallel, although some sections show slightly undulating erosion surfaces (Figure 3b). Detrital grains are mostly angular with a moderate degree of sphericity. They consist of quartz (~40 wt.%), muscovite, plagioclase and potassium feldspars (~4 wt.%) (Figure 3c; Table S4). The samples are rich in clays (> 50 wt.%), including chlorite, illite, kaolinite, chlorite-expandable (corrensite) and mica-expandable ordered interstratifications (Tables S4 and S5).

Cementation is absent and compaction is minimal, as indicated by the presence of slightly compressed muscovite grains (Figure 3c). Within the porous and clay-rich areas, euhedral pyrite and manganese oxides crystallised after grain deposition (Figure 3d,e).

#### 4.1.2 | Facies 2: Poorly-Cemented Sandstone With Clay Matrix

This facies is represented by a single sample from the *Nautile* dive (DIA23-NAUT10-07) and several samples from the dredge (Table 1). It is characterised by well-sorted very fine to fine sand grains (50–300 µm in size), with the exception of sample DIA23-NAUT10-07 which is poorly sorted (grain sizes between 100 and 1200 µm). Bedded samples show flat-parallel bedding with muscovite grains aligned along the bedding planes. The mineralogical assemblage is dominated by angular quartz

**TABLE 1** | Microfacies of the detrital sedimentary rocks of the Buteur Ridge.

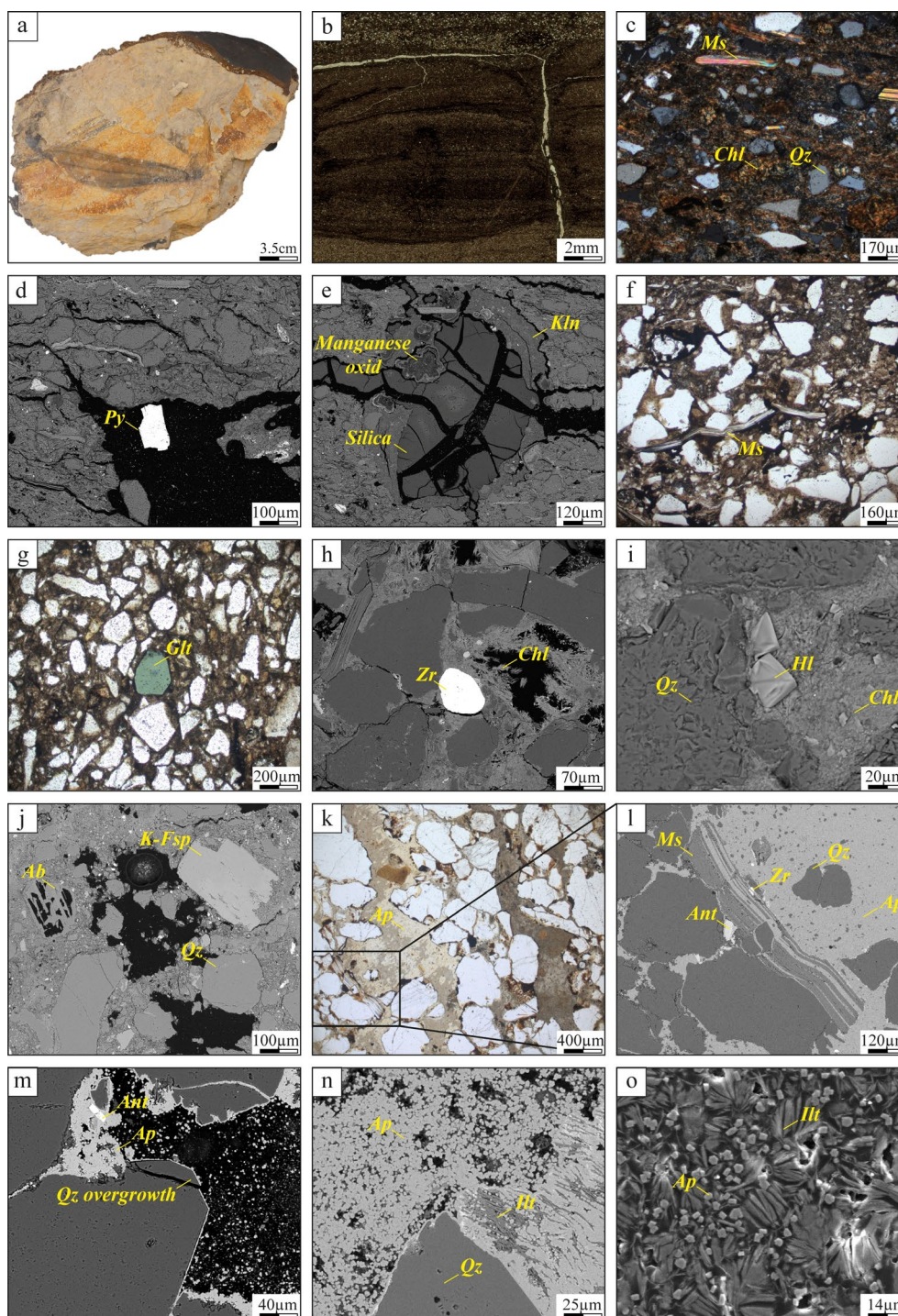
| Code     | Lithology     | Grain component                                                                                                                                                                                                                                                            | Matrix                                                                      | Cement             | Alteration                     | Compaction                                                            | Sedimentary structures                                                    | Depositional environment | Samples and thin sections                                                                                                     |
|----------|---------------|----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-----------------------------------------------------------------------------|--------------------|--------------------------------|-----------------------------------------------------------------------|---------------------------------------------------------------------------|--------------------------|-------------------------------------------------------------------------------------------------------------------------------|
| Facies 1 | Pelites       | <ul style="list-style-type: none"><li>Quartz (40%), medium sphericity and angular</li><li>Plagioclase and Potassium feldspars (4%)</li><li>Muscovites</li><li>Accessory minerals: Pyrite</li><li>Manganese oxides</li></ul>                                                | Illite<br>Chlorite<br>Kaolinite<br>Corrensite<br>Interlayer mica-expansible | —                  | —                              | Lightly deformed muscovites                                           | Flat and parallel bedding<br>Erosional surfaces<br>Bioturbation<br>Plants | Flooding plain           | <b>DIA23-DR6:</b> 02*; 03; 04*; 05; 06 (29 samples); 07; 08*; 09*; 10; 11*; 12; 13; 14* (25 samples); 21 (3 samples); 22*; 23 |
| Facies 2 | Quartzwacke   | <ul style="list-style-type: none"><li>Quartz (50%–80%), medium sphericity and angular</li><li>Plagioclase and Potassium feldspars (2%–10%)</li><li>Muscovites</li><li>Accessory minerals: Zircon<br/>Apatites<br/>Glauconite<br/>Halite<br/>Anatase<br/>Titanite</li></ul> | Illite<br>Chlorite<br>Kaolinite<br>Corrensite<br>Interlayer mica-expansible | Quartz overgrowths | Slight alteration of feldspars | Tangential contacts<br>Lightly deformed muscovites                    | Flat and parallel bedding                                                 | Flooding plain           | <b>DIA23-NAUT10:</b> 07<br><b>DIA23-DR6:</b> 15*; 16*; 17*; 18–01* to 11; 24                                                  |
| Facies 3 | Quartzarenite | <ul style="list-style-type: none"><li>Polyquartz (78%), medium sphericity and sub-angular</li><li>Plagioclase feldspars (2%)</li><li>Muscovites</li><li>Apatites (10%)</li><li>Accessory minerals: Zircon<br/>Manganese oxides<br/>Goethite<br/>Anatase</li></ul>          | Illite                                                                      | Quartz overgrowths | Slight alteration of feldspars | Pressure-solution<br>Fan fractures<br>Deformed muscovites (open book) | —                                                                         | Flooding plain           | DIA23-DR6-18-12*                                                                                                              |

(Continues)

**TABLE 1** | (Continued)

| Code     | Lithology                             | Grain component                                                                                                                                                                                                                                                                   | Matrix | Cement                                       | Alteration                                | Compaction                                                            | Sedimentary structures    | Depositional environment | Samples and thin sections                                      |
|----------|---------------------------------------|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|--------|----------------------------------------------|-------------------------------------------|-----------------------------------------------------------------------|---------------------------|--------------------------|----------------------------------------------------------------|
| Facies 4 | Quartzarenite and micaceous sandstone | <ul style="list-style-type: none"> <li>Quartz (60%–80%), high sphericity and sub-rounded</li> <li>Muscovites</li> <li>Feldspars (1%)</li> <li>Ankerite (2%)</li> </ul> <p>Accessory minerals:<br/>Zircons<br/>Calcite<br/>Halite<br/>Anatase</p>                                  | —      | Quartz overgrowths<br>Iron oxide cementation | Alteration of feldspars: Illite/Kaolinite | Pressure-solution<br>Fan fractures<br>Deformed muscovites (open book) | —                         | Fluvial                  | <b>DIA23-NAUT10:</b><br>01*; 02*; 10*<br><b>DIA23-DR6:</b> 19* |
| Facies 5 | Quartzarenite                         | <ul style="list-style-type: none"> <li>Undulatory quartz (90%), high sphericity and sub-rounded</li> <li>Plagioclase and Potassium feldspars (1%)</li> <li>Muscovites (2%)</li> </ul> <p>Accessory minerals:<br/>Zircons<br/>Anatase<br/>Ilmenite<br/>Chromite<br/>Glauconite</p> | —      | Quartz overgrowths<br>Quartz blocking cement | Alteration of feldspars: Illite/Kaolinite | Pressure-solution<br>Fan fractures<br>Deformed muscovites (open book) | Flat and parallel bedding | Fluvial                  | <b>DIA23-NAUT10:</b><br>03*; 04*; 05*; 09*                     |

*Note:* Terminology from Boggs Jr (2009). Black stars indicate thin sections. DIA23-DR6, dredge name followed by the sample number; DIA23-NAUT10, submersible-dive name followed by the sample number.



**FIGURE 3** | Microfacies 1 (a–e), 2 (f–j) and 3 (k–o) of the detrital sedimentary rocks of the Buteur Ridge. (a) Pelite fragment showing dark plant imprint (DIA23-DR6-21). (b) Flat and parallel bedded pelite with desiccation cracks (DIA23-DR6-08), transmitted light. (c) Mineralogical assemblage of pelites with oriented muscovites (DIA23-DR6-11), polarised-analysed light. (d) Pyrite crystallisation in porosity (DIA23-DR6-14-22), observed with SEM under backscattered electron beam. (e) Silica crystallisation in secondary porosity after manganese oxide precipitation and kaolinite deposition (DIA23-DR6-14-22), backscattered electron beam. (f) Lightly deformed muscovites (DIA23-DR6-15), transmitted light. (g) Grains of glauconite and quartz in clay matrix (DIA23-DR6-18-01), transmitted light. (h) Quartz, muscovite and zircon in rosaceous chlorite matrix (DIA23-DR6-15), backscattered electron beam. (i) Halite crystallisation prior to chlorite deposition (DIA23-DR6-16), backscattered electron beam. (j) Potassium feldspar and albite grains altered (DIA23-NAUT10-07), backscattered electron beam. (k) Apatites filling the secondary porosity (light, left) (DIA23-DR6-18-12), transmitted light. (l–n) Apatite crystallisations are posterior to the deposition of grains and the quartz overgrowths along which they form fringes (DIA23-DR6-18-12), backscattered electron beam. (o) Automorphous apatites posterior to illite (DIA23-DR6-18-12), secondary electron beam. Abbreviations for minerals according Whitney and Evans (2010). Ab, Albite; Ant, Anatase; Ap, Apatite; Chl, Chlorite; Glt, Glauconite; Hl, Halite; Ill, Illite; K-Fsp, Potassic feldspar; Kln, Kaolinite; Ms, Muscovite; Py, Pyrite; Qz, Quartz; Zr, Zircon.

with a moderate degree of sphericity (~50–80 wt.%) (Figure 3f) and contains potassic feldspars and plagioclases (~2–10 wt.%) (Tables S3 and S4), muscovites, detrital glauconite grains (only present in sample DIA23-DR6-18-01; Figure 3g) and few zircons (Figure 3h). Quartz grains commonly contain numerous zircon inclusions. The clay matrix is significant in these samples (~17–50 wt.%) and consists of illite and chlorite in sample DIA23-NAUT10-07 (Table S3) and of a combination of illite, chlorite, kaolinite, chlorite-expandable (corrensite) and mica-expandable interstratifications in the others (Tables S4 and S5). Sample DIA23-DR6-16 contains halite crystals concentrated in one bed and deposited prior to the clay matrix (Figure 3i). Heavy minerals crystals (anatase, titanite, etc.) are also observed in the clay matrix.

Low compaction is evidenced by tangential grain contacts and some deformation of muscovite grains (Figure 3f). Very thin rounded quartz overgrowths, indicative of a cementation phase, are visible in all samples.

#### 4.1.3 | Facies 3: Sandstone With Authigenic Apatite Crystallisations

This facies is described for a single sample from the dredge (DIA23-DR6-18-12, Table 1). This sample is a poorly sorted coarse sandstone (200–2000  $\mu\text{m}$  in size) composed mainly of angular and moderately spherical quartz (~78 wt.%) (Figure 3k), mainly polyquartz, with altered feldspars plagioclases (~2 wt.%) (Table S3) and muscovites (Figure 3l). The quartz grains contain numerous inclusions. The intergranular porosity is first partially filled by illite-rich clay assemblages and second, in localised areas, by apatites (Figure 3m–o).

Diagenesis leads to the formation of quartz overgrowths (Figure 3m). The sample has undergone severe compaction as evidenced by fractured quartz and muscovite grains and pressure-solution features (sutured grain contacts). This seems to indicate that the sandstone has undergone an advanced stage of diagenesis. Authigenic apatites (1–5  $\mu\text{m}$  in size) (~10 wt.%) (Table S3), with a hexagonal or pseudo-hexagonal structure (Figure 3o), crystallised after the formation of the quartz overgrowths (Figure 3m) and appear to be contemporaneous with the precipitation of manganese oxides. Apatites crystallised both in the intergranular porosity, between quartz grains, but they are also concentrated in specific areas, forming beds in which the primary material appears to have dissolved, generating secondary porosity (Figure 3k,l).

#### 4.1.4 | Facies 4: Sandstone Cemented by Iron Oxides

This facies includes three samples from the dive and one sample from the dredge (Table 1). Macroscopically, the samples are reddish-orange in colour (Figure 4a). The dive samples are composed of very fine to coarse-grained (60–700  $\mu\text{m}$  in size) moderately sorted grains, whereas the dredge sample is composed of coarse to very coarse well-sorted grains (500–1000  $\mu\text{m}$  in size) (Figure 4b,c). The mineralogical assemblage is dominated by blunt and spherical quartz (~80 wt.%) (Tables S3 and S4), with a

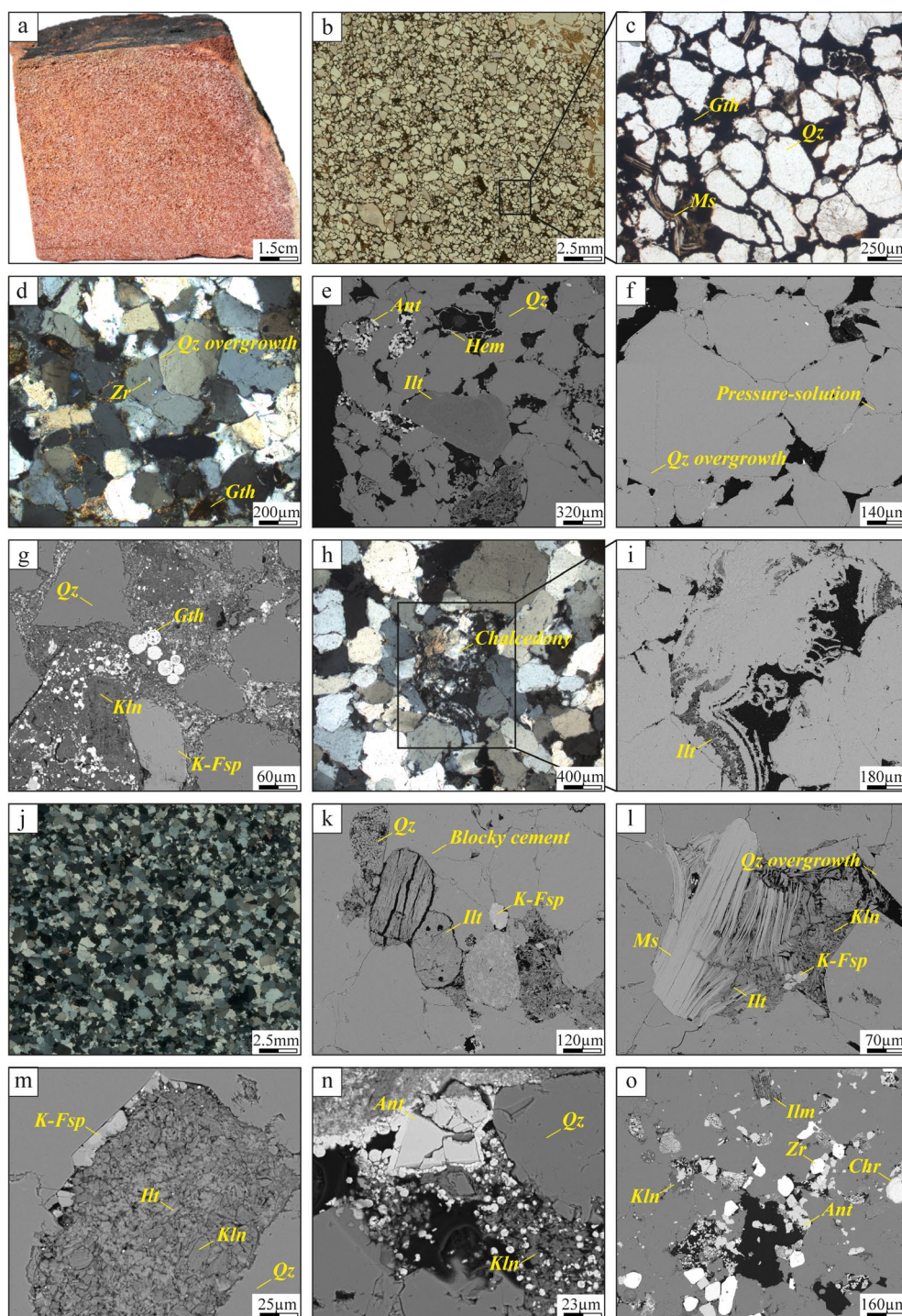
few grains of muscovite and a few zircons. The quartz content of sample DIA23-NAUT10-10 is lower (~60 wt.%) (Table S3), with a higher proportion of muscovites. The quartz grains contain numerous inclusions (Figure 4d). The clay matrix (~8–25 wt.%) consists of illite and kaolinite in dive samples and of a combination of illite and swelling 2:1 in the dredge sample (Tables S3 and S4).

The first early phase of cementation corresponds to quartz overgrowths (Figure 4d,f). Strong compaction is evidenced by the highly compressed muscovites (with an open book appearance) (Figure 4c), pressure-solution contacts with irregular surfaces (Figure 4f) and fan-shaped microfractures in quartz. These observations are consistent with an advanced stage of diagenesis. This is followed by an oxide cementation phase, predominantly goethite, except in DIA23-NAUT10-01, where haematite is more abundant (Table S3). Iron oxides are deposited as coating cement (for samples DIA23-NAUT10-01 and -02) (Figure 4e), and in sample DIA23-NAUT10-10, they are deposited as framboids (Figure 4g). At a more advanced stage (sample DIA23-DR6-19), the iron oxide cement completely binds the grains by filling the porosity (Figure 4c). Alteration of feldspars (Figure 4e) led to the formation of illite which filled the remaining porosity (Figure 4g) after quartz overgrowths. Feldspars dissolution follows iron cementation (Figure 4e) and is sometimes associated with anatase crystallisation (Figure 4e). Authigenic K-feldspars are deposited after this stage.

#### 4.1.5 | Facies 5: Well-Cemented Sandstone With Quartz Cement

Four *Nautila* dive samples are characterised by this facies (Table 1). The sandstone grains vary in size: DIA23-NAUT10-03 is a well-sorted coarse sandstone (500–1200  $\mu\text{m}$  in grain size); DIA23-NAUT10-04 is a well-sorted medium sandstone (100–600  $\mu\text{m}$  in grain size); DIA23-NAUT10-05 is a coarse bedded sandstone (100–1700  $\mu\text{m}$  in grain size) and DIA23-NAUT10-09 is a fine bedded sandstone (50–250  $\mu\text{m}$  in grain size). Samples are dominated by quartz (~90 wt.%) (Table S3), with a preponderance of spherical, blunt undulatory monoquartz (Figure 4h,j), and few muscovites. Clay matrix (~5–15 wt.%) consists of illite, kaolinite and swelling 2:1 (Table S3). Muscovite grains show alignment in the bedded samples. Sample DIA23-NAUT10-09 shows associations between fine quartz grains, large zircons, anatase, ilmenite, and chromite, highlighting the preferential deposition of heavy minerals in fine-grained beds (placer deposit formation) (Figure 4o).

A first cementation phase leads to quartz overgrowths (Figure 4l). This phase is followed by a late, incomplete phase of quartz blocking cement (Figure 4k). Samples have undergone severe compaction, as evidenced by the pressure-solution patterns in the quartz and the puncturing—or even tearing—of the muscovite fibres (Figure 4l). Feldspar alteration induced illite deposition in the ghost structures (Figure 4k) and after the compaction phase. DIA23-NAUT10-03 sample contains K-feldspar crystallisation contemporary with the clay deposits (Figure 4m). These observations are consistent with an advanced stage of burial diagenesis.



**FIGURE 4** | Legend on next page.

#### 4.1.6 | Sedimentary Petrology Synthesis

All the Buteur Ridge's samples belong to the same siliciclastic sequence, as defined by the described facies, which share many similarities, with variability mainly in grain sizes. The samples present a diagenetic phase characterised by moderate to strong compaction, quartz overgrowths and illitisation. Three facies show evidence of a second diagenetic phase, characterised by either oxide or quartz cementation, and for one sample by authigenic apatite crystallisation.

#### 4.2 | Biostratigraphy

Biostratigraphic analyses (palynomorphs and microfossils) were performed on three samples, two pelites from the dredge (DIA23-DR6-21 and -22) and a sandstone from the *Nautile* dive (DIA23-NAUT10-01). Both DIA23-DR6-21 and -22 contain the palynomorphs *Dicellaeropsis etruscus* suggesting a Tithonian to Hauterivian age. DIA23-DR6-22 also contains the palynomorphs *Subtilisphaera* spp. indicating Valanginian to Hauterivian age. Additionally, DIA23-DR6-21 contains small

**FIGURE 4** | Microfacies 4 (a–g) and 5 (h–o) of the detrital sedimentary rocks of the Buteur Ridge. (a) Sample of red sandstone (DIA23-NAUT10-01). (b) Coarse-grained sandstone with iron oxide cementation (DIA23-DR6-19), transmitted light. (c) Goethite cement binding all grains (DIA23-DR6-11), transmitted light. (d) Multiple zircon inclusions in primary quartz, with edges defined by quartz overgrowth (DIA23-NAUT10-02), polarised-analysed light. (e) Porosity induced by dissolution of minerals (probably feldspars) after precipitation of iron oxide cement and before crystallisation of anatase and ilmenite; Feldspar illitisation (DIA23-NAUT10-01), backscattered electron beam. (f) Quartz overgrowths and sutured contacts (DIA23-NAUT10-01), backscattered electron beam. (g) Goethite framboids associated with clay phases (kaolinite) (DIA23-NAUT10-10), backscattered electron beam. (h–i) Chalcedony filling porosity in well-cemented and highly compacted sandstone (DIA23-NAUT10-03), polarised-analysed light and backscattered electron beam. (j) Flat-bedded and well-sorted sandstone (DIA23-NAUT10-05), polarised-analysed light. (k) Feldspars dissolved and replaced by clays (DIA23-NAUT10-04), backscattered electron beam. (l) Highly deformed muscovite (open book) (DIA23-NAUT10-04), backscattered electron beam. (m) Fringe deposition of K-feldspars along a quartz grain, the porosity is induced by mineral dissolution and subsequently filled by illite and kaolinite (DIA23-NAUT10-04), backscattered electron beam. (n) Anatase crystallisation contemporaneous with kaolinite deposition (DIA23-NAUT10-03), backscattered electron beam. (o) Layer of heavy minerals (zircon, anatase, chromite, ilmenite) (placer-like) and feldspar dissolution replaced by iron and titanium mixtures (DIA23-NAUT10-09), backscattered electron beam. Abbreviations for minerals according Whitney and Evans (2010). Ant, Anatase; Chr, Chromite; Gth, Goethite; Hem, Haematite; Ilm, Ilmenite; Ill, Illite; K-Fsp, Potassic feldspar; Kln, Kaolinite; Ms, Muscovite; Qz, Quartz; Zr, Zircon.

abraded *Epistomina* microfossils (probably *Epistomina cf. tenuicostata*) in fine sands, suggesting a similar Tithonian to Hauterivian age. Assuming that these two samples belong to the same sedimentary unit, we interpret these results as indicating a Valanginian–Hauterivian age for the sedimentation.

Sample DIA23-NAUT10-01 contains a palynomorphic specimen of *Psilatropites* spp., suggesting a Cenomanian to Cenozoic age range.

Additionally, rare palynomorphs *Cribrerodinium* spp., *Systematophora* spp. (DIA23-DR6-21), *Tenua* spp. and *Subtilisphaera* spp. (DIA23-DR6-22) indicate marine influence, and the prominence of miospores in both samples suggests a proximal marine setting.

### 4.3 | AFT and ZFT Cooling Ages

Apparent apatite single grain cooling ages range from 73.6 to 186.3 Ma for DIA23-NAUT10-07; from 69.2 to 216.3 Ma for DIA23-DR6-16; and from 96.3 to 298.3 Ma for DIA23-DR6-18 (Table S7). The central cooling ages of the detrital apatites are given in Table 2 with their  $2\sigma$  error. The apatite cooling ages of samples DIA23-NAUT10-07 and DIA23-DR6-16 pass the  $\chi^2$  homogeneity test (0.70 and 0.38, respectively) and present dispersions of 5.9% and 0%, respectively (Table 2, Figures 5 and 6). It therefore seems relatively certain that the apatite population of each sample represents a single common age at  $122 \pm 8.3$  Ma ( $n = 30$ ) for DIA23-NAUT10-07 and at  $124 \pm 8$  Ma ( $n = 27$ ) for DIA23-DR6-16 (Figures 5 and 6). The apatite cooling ages of sample DIA23-DR6-18 fail the  $\chi^2$  homogeneity test (0.00) but the age dispersion is 15% (Table 2, Figure 5). It therefore seems likely that the observed grain-age distribution may contain two age groups at  $130 \pm 12$  and  $182 \pm 14$  Ma ( $n = 30$ ) (Figure 5).

In addition, for apatite crystals we measured the size of fission-track etch pits oriented parallel to the c-axis, referred to as Dpar (Table S8). Sample DIA23-NAUT10-07 has Dpar values ranging from 1.73 to 4.43  $\mu\text{m}$ , with only one apatite having a Dpar value of less than 1.75  $\mu\text{m}$ . Sample DIA23-DR6-16 has Dpar values between 1.24 and 2.24  $\mu\text{m}$ , with 15 apatites (out of 27) with Dpar

values below 1.75  $\mu\text{m}$ . Finally, sample DIA23-DR6-18 has Dpar values between 1.52 and 2.29  $\mu\text{m}$ , with six apatites (out of 30) having Dpar values below 1.75  $\mu\text{m}$ . Using the variations in Dpar values as a first-order proxy of chemical composition, it appears that the apatites in samples DIA23-NAUT10-07 and DIA23-DR6-18 are probably more chlorine-rich, whereas the apatites in sample DIA23-DR6-16 appear to be a mixture of chlorine and fluorine apatites. Additional chemical analyses are needed to confirm this hypothesis. Lastly, there were not enough horizontally confined track lengths that could be measured in the apatites to provide any indication of track shortening caused by partial annealing.

Apparent zircon single grain cooling ages range from 49.5 to 853.5 Ma for DIA23-NAUT10-01; from 88.1 to 956.9 Ma for DIA23-NAUT10-03; from 63.1 to 653.1 Ma for DIA23-NAUT10-07; from 68.4 to 828.9 Ma for DIA23-DR6-16 and from 89.9 to 662.4 Ma for DIA23-DR6-18 (Table S6). The central cooling ages of the detrital zircons are given in Table 2 with their  $2\sigma$  error. The zircon cooling ages fail the  $\chi^2$  homogeneity test in each sample and exhibit age dispersions ranging from 25% to 49% (Table 2, Figures 5 and 6). Two peaks were identified, for each sample, respectively at  $121 \pm 13$  Ma (25.5%) and  $316 \pm 24$  Ma (74.5%) (DIA23-NAUT10-01,  $n = 100$ ; Figure 6); at  $140 \pm 15$  Ma (45%) and  $251 \pm 30$  Ma (55%) (DIA23-NAUT10-03,  $n = 103$ ; Figure 6); at  $114 \pm 11$  Ma (33.1%) and  $254 \pm 22$  Ma (66.9%) (DIA23-NAUT10-07,  $n = 107$ , Figure 6); at  $134 \pm 17$  Ma (45%) and  $256 \pm 37$  Ma (55%) (DIA23-DR6-16,  $n = 101$ ; Figure 5) and at  $153 \pm 35$  Ma (32%) and  $252 \pm 41$  Ma (68%) (DIA23-DR6-18,  $n = 109$ ; Figure 5).

### 4.4 | Authigenic Apatite Crystallisation Ages

We conducted LA-ICP-MS U–Pb isotope analyses (Table S10) on the authigenic apatite crystallisations filling both the intergranular porosity and the secondary porosity. The isochron obtained from the analyses of the authigenic apatite gives a lower intercept age of  $93 \pm 12$  Ma ( $n = 46$ , MSWD = 0.95) in the Tera–Wasserburg diagram (all uncertainties reported are at 2s, MSWD = 0.95), with an initial  $^{207}\text{Pb}/^{206}\text{Pb}$  abundance ratio of  $0.852 \pm 0.016$  (Figure 7a, laser spots are reported in Figure 7b, Figure S1).

TABLE 2 | Results of apatite and zircon fission-track analysis.

| Mineral | Sample (DIA23-) | Number of grains | ps (10 <sup>5</sup> cm <sup>-2</sup> ) | Ns   | pi (10 <sup>5</sup> cm <sup>-2</sup> ) | Ni   | pd (10 <sup>5</sup> cm <sup>-2</sup> ) | Nd   | P (χ <sup>2</sup> ) | Dispersion (%) | Central age (Ma) | ±2σ (Ma) | U (ppm) | ±2σ (ppm) |
|---------|-----------------|------------------|----------------------------------------|------|----------------------------------------|------|----------------------------------------|------|---------------------|----------------|------------------|----------|---------|-----------|
| Zircon  | NAUT10-01       | 100              | 74.9                                   | 9661 | 5.67                                   | 731  | 3.27                                   | 4085 | 0.0                 | 49             | 226              | 34       | 86.7    | 6.8       |
| Zircon  | NAUT10-03       | 103              | 89.8                                   | 7559 | 8.93                                   | 752  | 3.29                                   | 4082 | 0.0                 | 27             | 186              | 24       | 136     | 11        |
| Zircon  | NAUT10-07       | 107              | 87.3                                   | 7848 | 8.79                                   | 790  | 3.31                                   | 4080 | 0.0                 | 39             | 183              | 24       | 133     | 10        |
| Zircon  | DR6-16          | 101              | 73.0                                   | 6733 | 7.06                                   | 651  | 3.18                                   | 4096 | 0.0                 | 32             | 183              | 24       | 111     | 9         |
| Zircon  | DR6-18          | 109              | 88.8                                   | 8942 | 7.58                                   | 763  | 3.19                                   | 4093 | 0.01                | 25             | 210              | 26       | 119     | 9         |
| Apatite | NAUT10-07       | 30               | 5.73                                   | 988  | 10.0                                   | 1725 | 15.4                                   | 6006 | 0.7                 | 5.9            | 122.2            | 17       | 10      | 1         |
| Apatite | DR6-16          | 27               | 16.6                                   | 1508 | 27.5                                   | 2503 | 15.9                                   | 5805 | 0.38                | 0              | 124              | 16       | 28      | 1         |
| Apatite | DR6-18          | 30               | 14.3                                   | 1900 | 18.5                                   | 2465 | 15.0                                   | 5855 | 0.0                 | 15             | 164              | 22       | 18      | 1         |

Note: Fission-tracks were counted dry at 1250× magnification using an Olympus BX51 microscope for apatites, an Olympus BH2 microscope for zircons and the FT-Stage 4.04 system (Dumitru 1993). Apatite fission-track ages were calculated using a zeta value of 279.20 ± 15.21 (C. Coudun) and the IRMM540R uranium glass standard (15 ppm U). Zircon fission-track ages were calculated using a zeta value of 113.94 ± 4.47 (C. Coudun) and the IRMM541 uranium glass standard (50 ppm U).

Abbreviations: pd, density of induced fission-tracks in the dosimeter uranium glass standard; pi, density of induced fission-tracks; ps, density of spontaneous fission-tracks; Nd, number of spontaneous fission-tracks; Ns, number of induced fission-tracks in the mica detector; Ni, number of induced fission-tracks in the zircon glass standard; Nt, number of induced fission-tracks in the zircon glass standard.

## 5 | Discussion

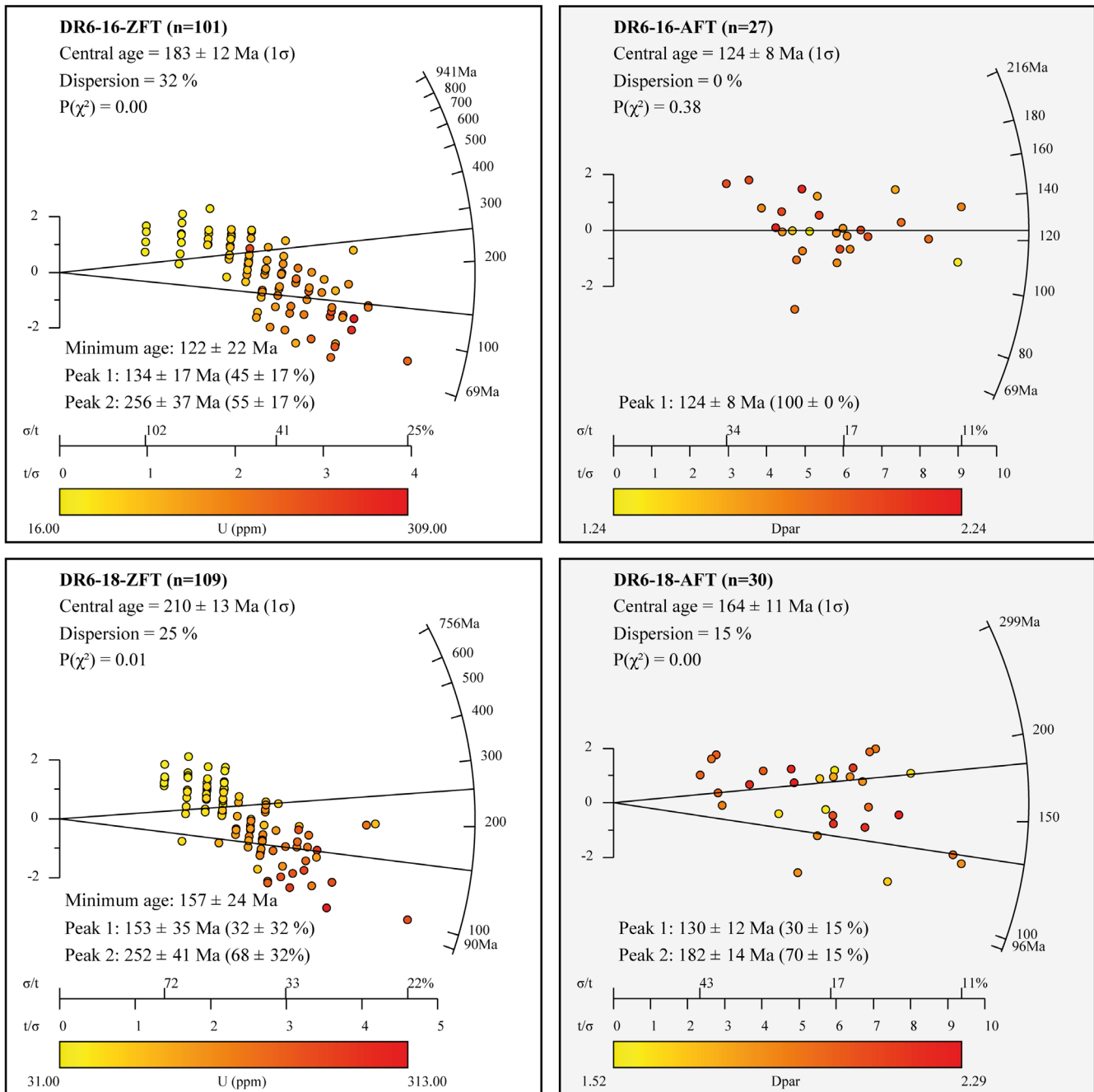
### 5.1 | Sedimentation and Diagenesis

The sedimentary facies observed on the Buteur Ridge show relatively little variability. As these facies belong to the same siliciclastic sequence, they appear to be associated with the same initial depositional environment. The microbiological assemblages of the samples suggest the vicinity of a marine environment, but the siliciclastic sedimentary facies barren of marine macrofossils likely correspond to a flooding plain, probably close to the shore. A flooding plain environment also allows the occurrence of rare evaporitic deposits, as indicated by the presence of halite crystals. Based on the palynology of the thinner sediments (DIA23-DR6-21 and DIA23-DR6-22), the depositional age is assigned to Valanginian-Hauterivian (137.05 ± 0.2–125.77 Ma from V. 2024/12 of the International Commission of Stratigraphy; Figure 8). Although the exact stratigraphic location of these dredged dated samples is not known, both the seismic lines and the petrological similarities with the samples from the log of the *Nautilite* dive suggest that they belong to the syn-rift unit.

We observed different stages of diagenesis in sediments. The first diagenetic phase involves moderate to strong grain compaction, alteration of feldspars that are dissolved or replaced by illite and crystallisation of thin quartz overgrowths that become thicker in some samples, filling partially the intergranular porosity. Only the clay-rich facies appear to be under-compacted, which is consistent with their lithology, as their high initial water content, low permeability and plastic mechanical behaviour hinder efficient fluid expulsion during burial. Illitisation is a process that can occur at temperatures as low as 50°C (Bjorkum and Gjelsvik 1988). The crystallisation of quartz overgrowths suggests more likely burial diagenesis at temperatures higher than 90°C (e.g., Chen et al. 2024; Walderhaug 1994). It is not possible to state if chlorite and corrensite are part of the sedimentary clays or were formed by diagenesis. If they form during diagenesis, it indicates temperatures exceeding 100°C–150°C (e.g., Hillier 1993; Matýsek and Jirásek 2023; Son et al. 2001).

In some samples, iron oxide cementation post-dating the quartz overgrowths indicates a second diagenetic phase. This suggests that surface weathering released iron oxide during telodiagenesis and basin inversion, after the initial burial diagenesis phase (e.g., Bello et al. 2023; Parnell 2002; Worden and Burley 2009). One of these samples (DIA23-NAUT10-10) comes from the post-rift unit at the top of the Buteur Ridge, two from the massive rock fall at the bottom of the ridge (DIA23-NAUT10-01 and DIA23-NAUT10-02), but were interpreted to belong to the same post-rift unit, and one from the dredge (DIA23-DR6-19). Among them, sample DIA23-NAUT10-01 has been dated Cenomanian or younger in age. It indicates that at the Buteur Ridge location, sedimentation was still siliciclastic at the beginning of the Late Cretaceous.

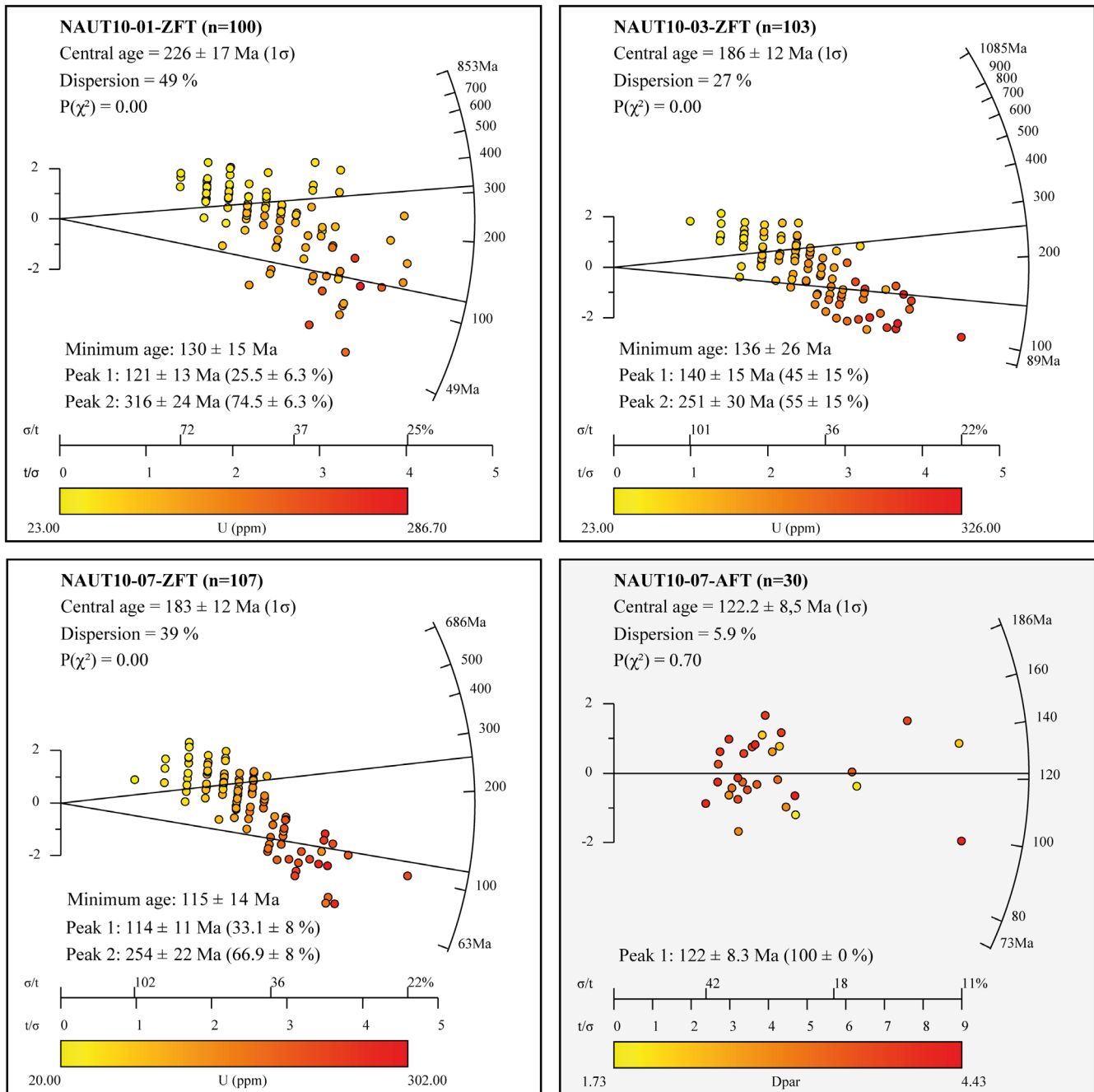
As for these post-rift sediments, we also hypothesise that sample DIA23-DR6-18-12 may have undergone a telodiagenetic phase, characterised by the formation of secondary porosity following the burial diagenetic phase. Indeed, apatite crystallisation within the secondary porosity appears to have occurred after quartz overgrowths and illite deposition in the primary



**FIGURE 5** | Radial plots of detrital zircon (left, white background) and apatite (right, grey background) fission-track cooling ages from DIA23-DR6-16 and DIA23-DR6-18 (western slope of the Buteur Ridge). The ZFT data show a clear trend of younger cooling ages in grains with higher U-content which hints at, at least, partial annealing of highly radiation-damaged grains (Bernet 2024).

porosity. According to this hypothesis, the secondary porosity would have been generated by the dissolution of the primary material, possibly in contact with meteoric waters when the sample was resurfaced. As apatite requires phosphate to crystallise, we suggest that the dissolved primary material was rich in phosphate, providing phosphate ions during an early stage of diagenesis after telodiagenesis. This stage involves the circulation of supersaturated water, possibly brackish, through sandstone pores (Bossennec et al. 2021; Bouch et al. 2002; Dar et al. 2017). Consequently, the crystallisation of authigenic apatites could have occurred contemporaneously with the influx of marine water following the telodiagenesis phase.

In summary, we identify two episodes of siliciclastic sedimentation associated with a flooding plain environment on the Buteur Ridge: a Valanginian-Hauterivian sedimentation interpreted as syn-rift from the seismic lines, and a Cenomanian or more recent post-rift sedimentation (Figure 8). The syn-rift unit is slightly older than the previously proposed Aptian age (Basile et al. 2013; Loncke et al. 2022; Sapin et al. 2016). The post-rift unit is younger than the Late Aptian–Early Albian post-rift unconformity (Basile et al. 2005, 2013; Casson et al. 2021; Sapin et al. 2016; Fonseca et al. 2024), suggesting a significant hiatus at the top of the Buteur Ridge. Both units experienced burial diagenesis after the sedimentation, the post-rift unit recording



**FIGURE 6** | Radial plots of detrital zircon (top and bottom left, white background) and apatite (bottom right, grey background) fission-track cooling ages from DIA23-NAUT10-01, DIA23-NAUT10-03 and DIA23-NAUT10-07 (eastern slope of the Buteur Ridge). The ZFT data show a clear trend of younger cooling ages in grains with higher U-content which hints at, at least, partial annealing of highly radiation-damaged grains (Bernet 2024).

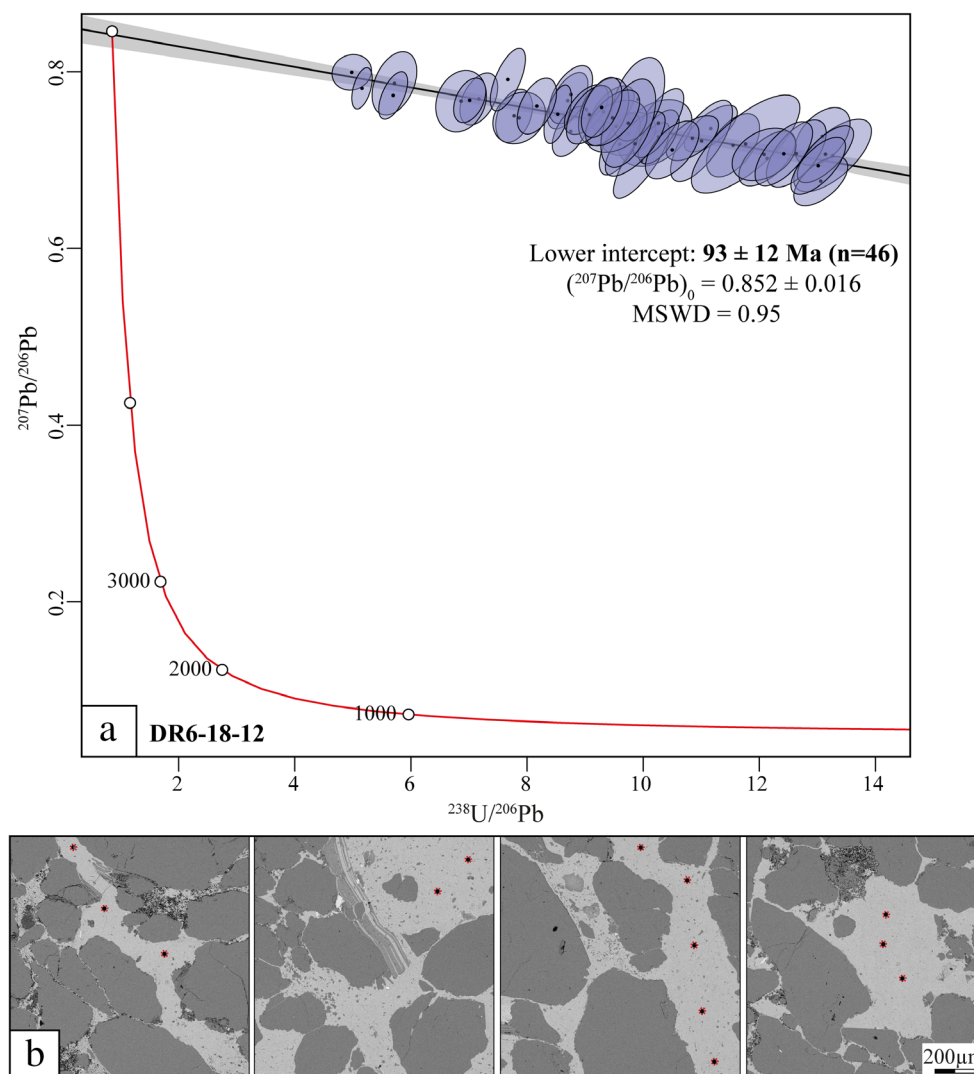
additionally a telodiagenetic phase indicating uplift and outcropping at or above sea level.

## 5.2 | Thermal History

### 5.2.1 | Fission-Tracks Partial or Total Annealing

The presence of a single cooling age population in the detrital apatites of samples DIA23-NAUT10-07 and DIA23-DR6-16, while the sample DIA23-DR6-18 from the same unit exhibits two distinct cooling age populations, suggests that total

annealing may have occurred in samples DIA23-NAUT10-07 and DIA23-DR6-16. In all samples, the zircon cooling ages from each sample form also two distinct populations. Assuming that the detrital apatites and zircons came from the same erosional source, it indicates that the apatite fission-tracks of samples DIA23-NAUT10-07 and DIA23-DR6-16 were fully annealed, while the zircon fission-tracks were not. We propose that these samples underwent a heating event that raised temperatures above the upper limit of the apatite PAZ (i.e., above  $120^\circ\text{C}$ ). Consequently, the cooling ages for these two samples ( $122 \pm 8.3$  and  $124 \pm 8$  Ma, Figure 8) could correspond to the cooling of the apatites, assuming that it occurred rapidly.



**FIGURE 7** | (a) Tera-Wasserburg diagram of the LA-ICP-MS U–Pb analysis on authigenic apatite crystallisations of the DIA23-DR6-18-12 sandstone sample, not anchored. Age includes uncertainties in 2s (including long-term excess variance). MSWD–Mean Square Weighted Deviation. (b) Examples of laser spot locations within apatite crystallisation zones, backscattered electron beam (see Figure S1 for the locations of all laser spots made on the thin section of sample DIA23-DR6-18-12).

Furthermore, as these samples belong to the syn-rift unit interpreted to have been deposited during the Valanginian-Hauterivian (137–125Ma), the minimum zircon cooling age and the first peak in sample DIA23-NAUT10-07 (125–103Ma) appear to be either contemporaneous with or younger than the syn-rift deposition, supporting the partial annealing hypothesis (Figure 8). Similarly, the minimum zircon cooling ages of other syn-rift samples overlap the depositional age (DIA23-NAUT10-03: 155–125Ma; DIA23-DR6-16: 151–117Ma; DIA23-DR6-18: 188–118Ma, Figure 8). For these samples partial annealing is also possible, but at lower temperatures than for sample DIA23-NAUT10-07. These results suggest that the detrital zircons were heated to at least 180°C, the upper limit of the PAZ, at least for sample DIA23-NAUT10-07.

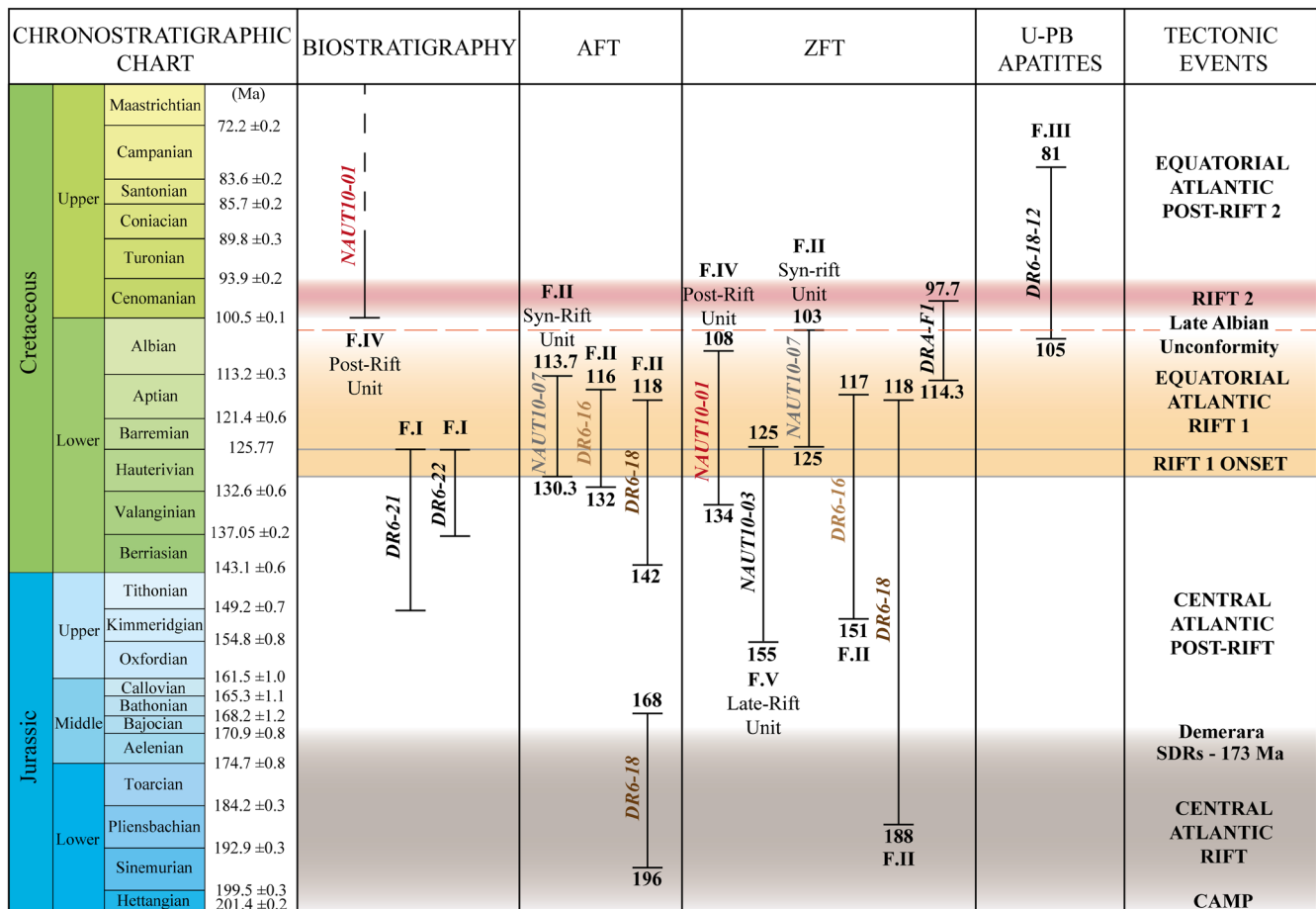
In contrast, the presence of distinct cooling age populations in the detrital apatites of sample DIA23-DR6-18 suggests that these apatites did not undergo a complete reset. However, this does not exclude the possibility of a partial reset because the cooling age of the youngest apatite peak (142–118Ma) is similar to the depositional age (Figure 8). For this to occur, the apatites would have

had to be heated to temperatures within the PAZ, that is, between 60°C and 120°C. Based on this observation, it seems unlikely that the zircons in sample DIA23-DR6-18 have undergone a partial reset. Therefore, the cooling ages observed for these zircons (188–118Ma) are more likely to represent source erosion ages (Figure 8).

Given the evidence for partial annealing, the question arises as to whether the first ZFT peak age (114.3–97.7Ma) reported by Girault et al. (2023) for the Buteur Ridge dredge sample has also undergone partial annealing. If so, this younger age can no longer be interpreted as being derived solely from source erosion. However, in the absence of apatite fission-tracks for this sample it is difficult to determine whether the zircons are partially annealed or not.

### 5.2.2 | Apatites and Zircons Cooling Before the Cretaceous Rifting

The oldest cooling ages of the zircons allow us to estimate the original provenance of the detrital material found on the Buteur



**FIGURE 8** | Compilation of chronostratigraphic results from sedimentary rocks of the Buteur Ridge, and interpretation of the associated tectonic events. Vertical bars represent the time interval associated with each sample and dating method (biostratigraphy, apatite fission-tracks, zircon fission-tracks and U–Pb on apatites). The maximum and minimum bounds of the absolute ages are indicated at the top and bottom of the bars, respectively. For the ZFT results, only the youngest age population is shown. The interpretation of tectonic events follows the second scenario presented in Figure 9. F, Facies. International chronostratigraphic chart based on Cohen et al. (2024).

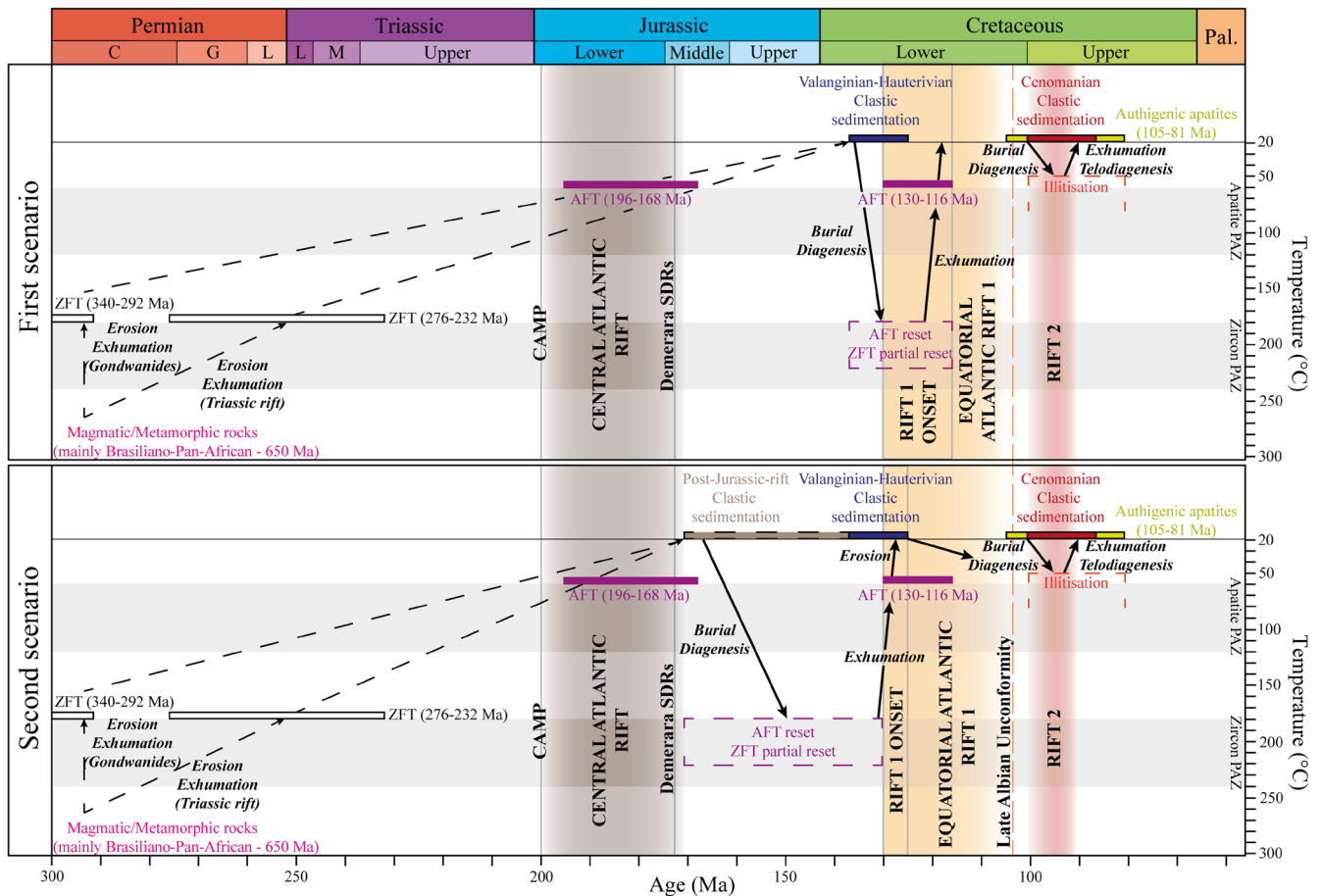
Ridge. Indeed, the crystallisation ages of the zircons found in the Buteur Ridge sample from the DRA-F1 dredge (DRADEM; Basile 2016) indicate that the detrital material is predominantly derived from the 650 million-years-old Brasiliano-Pan-African orogen (Girault et al. 2023). Similar Brasiliano-Pan-African crystallisation ages were also identified in zircons from samples at other locations on the Demerara Plateau (Girault et al. 2023). We consequently propose that at least part of the detrital material found in the DIA23-DR6 dredge and the DIA23-NAUT10 dive is also derived from the same orogen. Detrital zircons from the DRA-F1 dredge record an initial cooling phase during the Upper Triassic ( $221 \pm 12$  Ma), interpreted as a response to exhumation during the early Triassic phase of Central Atlantic rifting (Girault et al. 2023; Hames et al. 2000; Withjack et al. 2013). Similarly, detrital zircons from samples DIA23-DR6-16, DIA23-DR6-18, DIA23-NAUT10-03 and DIA23-NAUT10-07 show an initial cooling phase that appears to be between Mid-Permian and Upper Triassic ( $256 \pm 37$ ,  $252 \pm 41$ ,  $251 \pm 30$  and  $254 \pm 22$  Ma, respectively). We suggest that this cooling phase is comparable to that observed in the study by Girault et al. (2023) and can be similarly interpreted as exhumation during a Triassic tectonic phase, before the onset of the Central Atlantic Magmatic Province (CAMP) and the subsequent Central Atlantic rift (Leleu et al. 2016). In contrast, sample DIA23-NAUT10-01 shows

a slightly older first cooling phase between 340 and 292 Ma (Mid-Mississippian to Early Permian). It can be related to the Gondwanides orogeny, which occurred on the western margin of Gondwana during the Carboniferous (Dias et al. 2017), as also suggested by Girault et al. (2023) for other samples of the Demerara Plateau.

The oldest apatite cooling age from sample DIA23-DR6-18 is the interval 196–168 Ma (Figure 8), and can likely be related to the cooling of the basement associated with the Jurassic rifting of the Central Atlantic, prior to or coeval with the SDRs of the Demerara Plateau.

### 5.2.3 | Apatites and Zircons Cooling Related to the Cretaceous Rifting

As mentioned above, the sediments of the Buteur Ridge were deposited after the Jurassic rifting of the Central Atlantic. Furthermore, Musser et al. (2021) provide evidence that Seaward-Dipping Reflector (SDR) units are visible on seismic reflection data west of the Buteur Ridge, and the seismic refraction data support the interpretation that the Buteur Ridge lies on the magmatic basement of the Jurassic margin rather than on the



**FIGURE 9** | Two alternative time–temperature evolution scenarios for the sediments sampled on the Buteur Ridge, and corresponding interpretation of the tectonic evolution of the divergent margin during the Equatorial Atlantic rift. The two hypotheses differ by the stratigraphic position where detrital apatite and zircon fission-tracks were fully or partially reset. First scenario: Continuous exhumation followed by Lower Cretaceous sedimentation, then rapid burial and subsequent exhumation. This allows for the resetting of apatite and zircon fission-tracks but only over a limited time period. Second scenario: Exhumation followed by Jurassic sedimentation and burial with the resetting of apatite and zircon fission-tracks, prior to renewed exhumation through erosion and Lower Cretaceous clastic sedimentation. See text for details.

craton. Consequently, the sediments of the Buteur Ridge belong to the post-Jurassic-rift units.

The younger fission-track populations of both apatites and zircons are remarkably consistent and point out a Lower Cretaceous cooling (Figure 8). These ages can be related to the Equatorial Atlantic rifting. A comparable cooling event, dated between 140 and 90 Ma, was previously documented by Derycke et al. (2021) on rocks outcropping on the French Guiana margin and was similarly attributed to the Equatorial Atlantic rifting. The vertical displacements associated with the rifting induce exhumation of the footwall of normal faults, but also regional uplift and erosion of rift shoulders (Withjack et al. 2002). The best ages for this cooling are provided by the two samples where the apatites belong to a single population, after the fission-tracks were reset. This 130–116 Ma cooling age is coeval or post-dates the stratigraphic age (137–125 Ma) of the sedimentary unit where they were sampled (Figure 8).

When AFT indicates a reset of the fission-tracks, with a cooling age younger than the sedimentation, it points to in situ heating and cooling in the sediment. In this interpretation, apatites and zircons are deposited in the sediment as are the other

clasts, and the sediment has to be buried to reach a temperature higher than the apatite PAZ, here up to the zircon PAZ (180°C) (first scenario in Figure 9). Then exhumation has to return the apatites to lower temperatures than the apatite PAZ (<60°C) (Figure 9), that can be done rapidly in a rift tectonic setting. However, burial occurred in this interpretation in a late post-rift setting, some 30 million years after the end of the Jurassic rifting (Figure 9), when fast subsidence is not expected nor observed on the Demerara Plateau (e.g., Casson et al. 2021). For example, Olyphant et al. (2017) computed a maximum subsidence of 70 m/Myr before the Cretaceous rifting. As both burial and exhumation have to occur in the same time interval defined by the beginning of sedimentation (137–125 Ma) and the end of the cooling (130–116 Ma), it leaves a maximum of 21 million years for both burial down to 180°C and exhumation up to 60°C (Figure 9). The cooling from 180°C to 60°C can occur in 3 million years assuming a 40°C/km geothermal gradient and a km/Myr exhumation rate. In contrast, the burial has to increase the temperature by 160°C in maximum the remaining 18 million years, approximately 10°C/Myr. Of course, the heating rate depends on the geothermal gradient. A normal 35°C/km gradient implies a minimum mean subsidence rate of 350 m/Myr, unlikely as it is five times faster than the maximum observed.

But a 70 m/Myr subsidence should imply a geothermal gradient higher than 200°C/km, which is definitely not supported by the observations, as no hydrothermal fracturation and/or deposits have been observed in any sample nor in the field.

Let us consider an alternative hypothesis, based on the contemporaneity of AFT cooling ages and sedimentation in the 125–130 Ma interval. In this hypothesis, the apatites and zircons were first sedimented in the post-Jurassic-rift sequences (second scenario in Figure 9). As the end of Jurassic rifting is Middle Jurassic in age (post 173 Ma, Basile et al. 2020), it leaves the whole post-rift evolution (some 40 million years) to bury the sediment, reset the apatite fission- tracks and reach the zircon PAZ (Figure 9). It implies a subsidence rate of circa 130 m/Myr for a thermal gradient of 35°C/km, which seems reasonable, if not underestimated, for a post-rift magmatic margin (Pindell and Heyn 2022). This can be corroborated by the observed thickness of the post-Jurassic-rift on the western Demerara Plateau, which exceeds 5 km (Casson et al. 2021). Then, during the Cretaceous rift, this post-Jurassic-rift sedimentary sequence can be uplifted in the rift shoulders, exhumed along normal faults, and eroded to feed the syn-Cretaceous rift sequences (Figure 9). Rounded foraminifera and quartz overgrowths can result from such erosion of older sediments. Consequently, the reset apatites and partially reset zircons were re-sedimented in the syn-rift sediments, having recorded the cooling linked to exhumation in the rift. In this hypothesis, exhumation, erosion and sedimentation occurred during the rifting, dating the Cretaceous rift at 130–125 Ma (Hauterivian) (Figures 8 and 9). These sediments were subsequently buried and submitted to diagenesis, but with temperatures not exceeding the apatite PAZ. In this hypothesis, the sampled sediments would therefore correspond to the syn-rift sediments of the Equatorial Atlantic rift, as independently proposed from seismic lines and structural interpretations.

Furthermore, following this second option, since the sediments deposited in the Buteur Ridge basin are derived from the erosion of the post-Jurassic-rift basin, it becomes easier to understand why not all samples have the same annealing response. In fact, the sediments contributing to the formation of the syn-rift unit can be derived from the erosion of different stratigraphic layers, which have not been subjected to the same burial and therefore heating conditions. This scenario explains why, within the same sedimentary unit, we can find apatites that have undergone full annealing (e.g., DIA23-NAUT10-07 and DIA23-DR6-16), alongside apatites that have not undergone full annealing and probably only experienced partial annealing (e.g., DIA23-DR6-18) (Figure 8). However, in this scenario, the syn-rift sediments could not have been exposed to temperatures above 60°C after their deposition (Figure 9). Therefore, the formation of quartz overgrowths, which typically requires temperatures exceeding 90°C, is more difficult to envisage after the unit was deposited. Yet, samples from the syn-rift unit show quartz overgrowths. One possible explanation is that, at least, a part of these overgrowths was inherited, as suggested by the rounded overgrowths observed in particular in Facies 2 (Sanderson 1983). The proportion of inherited overgrowths is generally difficult to determine (Haile et al. 2021), and it may be possible that some of the angular, newly formed overgrowths may have resulted from the dissolution of inherited quartz overgrowths during compaction associated with burial at temperatures below 60°C. Another

possible explanation is that the apatite entered the PAZ down to 90°C. In this case, the younger cooling ages were rejuvenated, but not the older ones. Consequently, it does not modify the 130–125 Ma interval of overlapping sedimentation and apatite cooling.

Finally, the above two hypotheses differ by the interpretation of the sampled sediments, as post-Jurassic-rift with in situ heating for the first one, or as syn-Cretaceous-rift with previous thermal reset for the second one (Figure 9). The first hypothesis dates the rifting from AFT alone in the 130–116 Ma interval, but this hypothesis faces an unlikely fast burial. The second hypothesis implies that the onset of the Equatorial Atlantic rift occurred in the 130–125 Ma interval, being Hauterivian in age, from both AFT and sedimentation ages. We favour this second hypothesis which fits the observations for the whole set of samples and presents reasonable subsidence rates, geothermal gradients and exhumation rates. This Hauterivian age is slightly older than Barremian, as previously proposed by Basile et al. (2005) and Brunet et al. (1988) but consistent with the rifting ages proposed by Soares J nior et al. (2011) for the Marajo and Foz de Amazonas basins further south.

#### 5.2.4 | A Second Rifting of the Equatorial Atlantic

The continental break-up resulting from the Equatorial Atlantic rift has been proposed to have occurred during either the Late Aptian (117–113 Ma) (Basile et al. 2005), the Early Albian (113–109 Ma) (Fonseca et al. 2024) or the Late Albian (104–100 Ma) (Gouyet et al. 1994; Sapin et al. 2016; Ye et al. 2019). Consequently, the Late Albian unconformity (overlapped by transgressive sediments dated at c. 103 Ma) marking the transition from detrital and shallow water to pelagic sedimentation is generally considered to follow continental break-up for the Demerara Plateau. However, samples belonging to the post-Equatorial rift unit of the Buteur Ridge have facies (Facies 4) suggesting a distal continental environment characterised by moderately sorted detrital sedimentation. Their biostratigraphical age is Cenomanian or younger (Figure 8). This indicates that the post-rift subsidence is younger for the Buteur Ridge than for the Demerara Plateau, with the transition from detrital to pelagic sedimentation occurring during or after the Cenomanian instead of the Late Albian.

On the other hand, as previously shown by Coudun et al. (2025), the present relief of the Buteur Ridge is the result of reactivation of the normal fault, allowing syn-rift units to outcrop on the eastern flank and clastic post-rift sediments at the top of the ridge. As the reactivation of the normal fault occurred after the deposition of the post-rift unit and its subsequent diagenesis, it was caused by a post-rift tectonic event. This post-rift tectonic event may explain the difference in orientation between the Buteur Ridge normal faults (NNW–SSE; Coudun et al. 2025) and the continent-ocean boundary (NNE–SSW; Basile et al. 2013) on the eastern divergent margin of the Demerara Plateau. Both Campan (1995) and the recent kinematic model of Lesourd-Laux et al. (2025) proposed a polyphased rifting model for the Equatorial Atlantic. According to their models, a first rift was followed by a second rifting resulting from kinematic reorganisation, that led to the final opening of the Equatorial Atlantic. From interpolations of oceanic accretion rates, Campan (1995)

proposed that this second rifting started during the Albian, but no geological dates nor magnetic anomalies supported this age. Within this framework, we propose that the sediments deposited in the Cenomanian, previously described as post-Cretaceous-rift, are in fact post-first-Cretaceous-rift sediments, but pre-second-Cretaceous-rift sediments (Figure 9). We therefore propose that the uplift recorded on the Buteur Ridge is due to the onset of a second rifting of the Equatorial Atlantic that postdates the deposition of these Cenomanian sediments (Figure 8). It thus appears that the telodiagenesis highlighted by the second diagenetic phase in the post-first-Cretaceous-rift phase was likely linked to the second rifting of the Equatorial Atlantic (Figures 8 and 9). Consequently, the crystallisation of authigenic apatites, dated at 105–81 Ma and previously associated with the telodiagenetic phase, would then date the onset of marine transgression and the end of the second rifting of the Equatorial Atlantic (Figures 8 and 9). This should bracket the end of the second rift stage between the Cenomanian and 81 Ma, but likely Cenomanian in age, given the fact that it was the time when oceanic circulation between South and Central Atlantic was established (Dummann et al. 2023). There is a distance of 160 km from the Buteur Ridge to Chron 34, dated at 83 Ma (Cande and Kent 1995). If the final break-up of the Equatorial Atlantic is dated to Late Cenomanian (c. 93 Ma), this would imply an accretion rate of about 3 cm/year for the Equatorial Atlantic.

Finally, it is noteworthy that this second rift, associated with the kinematic reorganisation, affected the distal continental margin of the Demerara Plateau, where the Buteur Ridge is located, but has not been clearly identified on the proximal margin, which was affected by the first rift. The only possible exception may be an Albian-Cenomanian unconformity evidenced by Casson et al. (2021).

## 6 | Synthesis and Conclusion

In summary, this study provides valuable insights into the depositional environment, diagenetic evolution, and thermal history of the sediments sampled at the Buteur Ridge, thereby enhancing our understanding of the geological processes that shaped the divergent margin of the Demerara Plateau during the Cretaceous. By integrating thermochronological data with biostratigraphic calibration, our results offer robust evidence for a complex rifting history, marked by two distinct rifts of the Equatorial Atlantic.

For the first rift, we propose two scenarios. The first, although better aligned with the diagenetic observations, requires both high subsidence and exhumation rates during the Early Cretaceous, which appear unrealistic given the tectono-sedimentary context. Therefore, we favour a second scenario which, despite involving an additional diagenetic event, reconciles the data with more plausible sedimentation and exhumation rates. This preferred second scenario constrains the onset of rifting in the Equatorial Atlantic between 130 and 125 Ma (Hauterivian), slightly older than the previously proposed Aptian age.

Furthermore, the deposition of terrigenous detrital sediments on the Buteur Ridge during the Cenomanian indicates that

sedimentation was not yet pelagic at that time. This finding suggests that the end of the Equatorial Atlantic rifting, previously dated no later than the regional Albian unconformity, may actually be younger. In addition, the Cenomanian sediments record a phase of telodiagenesis, seemingly linked to the reactivation of normal faults and the subsequent uplift of the Buteur Ridge after sediment deposition. The telodiagenetic phase and the subsequent marine transgression, recorded by the crystallisation of authigenic apatites dated to the Late Cretaceous, could therefore correspond to the end of the second rifting of the Equatorial Atlantic. We suggest that this second rift occurred during the Cenomanian, affecting the distal continental margin of the Demerara Plateau, and may be coeval with the kinematic reorganisation of the Equatorial Atlantic.

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## Conflicts of Interest

The authors declare no conflicts of interest.

## Data Availability Statement

The data that support the findings of this study are available in the [Supporting Information](#) of this article.

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### Supporting Information

Additional supporting information can be found online in the Supporting Information section. **Data S1:** bre70076-sup-0001-Supinfo.docx.