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Reconstructing Tonian seawater $^{87}Sr/^{86}Sr$ using calcite microspar

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ABSTRACT

The Tonian Period followed a long interval of relative stasis and led into the climatic extremes and biological radiations of multicellular life during the Cryogenian and Ediacaran Periods, respectively. However, despite its pivotal situation, it remains relatively understudied, in large part due to the lack of robust age constraints. A combination of fossil evidence, radiometric ages, and isotopic constraints reveal that carbonate strata on the North China craton were deposited between ca. 980 and ca. 920 Ma, thereby filling a gap in marine archives. Here we present $^{87}Sr/^{86}Sr$ data from selected calcite microspar cements, which filled early diagenetic “molar tooth” cracks, along with data from demonstrably well-preserved bulk carbonate samples. These new data show that seawater $^{87}Sr/^{87}Sr$ rose in stages from ~0.7052 at ca. 980 Ma to ~0.7063 by ca. 920 Ma, after which a return to low values coincided with the eruption of the Dashigou large igneous province across the North China craton. We also present a new Neoproterozoic seawater $^{87}Sr/^{86}Sr$ curve, which reveals that the general trend toward higher $^{87}Sr/^{87}Sr$ during the Tonian Period was checked repeatedly by the input of less-radiogenic strontium from a series of eruptive events, both coincident with and prior to the main breakup of Rodinia. The weathering of Tonian volcanic provinces has been linked to higher carbon burial, glaciation, and oxygenation due to the high phosphorus content of flood basalts. Here we show that the weathering of major volcanic provinces affected material fluxes and ocean chemistry much earlier than previously envisaged.

INTRODUCTION

The strontium isotopic composition of seawater is homogeneous around the globe within analytical precision (McArthur, 1994; Kuznetsov et al., 2012) and varies over time in response to the balance between two distinct sources of strontium: (1) less-radiogenic Sr that enters the oceans via Sr exchange between seawater and ocean lithosphere, and (2) isotopically variable, but generally more-radiogenic, riverine Sr derived from the weathering of differentiated continental crust (Brass, 1976; Gaillardet et al., 2014; McArthur et al., 2012). The isotopic composition of rivers can vary considerably depending on the relative contribution from older, more-radiogenic terrains versus less-radiogenic mantle-derived igneous rocks such as basalt. Strontium isotope stratigraphy (SIS) can therefore help to constrain not only the ages of sedimentary successions but also the relative influence of tectonic factors, such as seafloor spreading, emplacement of juvenile volcanic provinces, and continental weathering rates, on ocean composition (Veizer, 1989; McArthur, 1994). Although SIS is well established in Phanerozoic studies because of the abundance of mineralogically stable biogenic materials such as low-Mg calcite shells, its application to Proterozoic strata is still dependent upon variably preserved bulk carbonate rock.

Despite inherent challenges, significant progress has been made toward constructing a Neoproterozoic seawater $^{87}Sr/^{86}Sr$ curve using bulk carbonate samples (Derry et al., 1992; Shields, 1999; Halverson et al., 2007; Kuznetsov et al., 2017), and recently Cox et al. (2016) extended their compilation to 1050 Ma (see the GSA Data Repository[1] for more details). All previous studies documented a general increase in seawater $^{87}Sr/^{86}Sr$, from ~0.705–0.709, over the course of the Neoproterozoic. However, details remain speculative because most published data suffer from poor age control, such as Tonian data from Siberia and the Ural Mountains (e.g., Kuznetsov et al., 2006, 2017), and/or are difficult to correlate globally (cf. Cox et al., 2016) due to lack of biostratigraphic control and the non-uniqueness of carbon isotope trends (Melezhik et al., 2015). Nevertheless, previous studies suggest that SIS has potential for both stratigraphic correlation and environmental interpretation of Neoproterozoic events, provided that well-preserved marine carbonate samples can be placed within the improving, global stratigraphic framework.

This study improves Neoproterozoic SIS by specifically targeting demonstrably well-preserved and age-constrained examples of calcite microspar cements (CMCs), which fill early diagenetic cracks, commonly referred to as “molar tooth structure”, and other cavities. Our new data for the North China craton fill a gap in the record between ca. 980 to ca. 920 Ma toward a new Sr isotope curve for Neoproterozoic seawater.

GEOLOGICAL BACKGROUND AND AGE MODEL

The North China craton has an Archean to Paleoproterozoic basement and unmetamorphosed Mesoproterozoic to Neoproterozoic sedimentary cover that was deposited in a shallow marine environment. The Huaibei region (Jiangsu, China), the research area of the present study, is situated on the southern margin of this eastern North China craton block (Fig. 1) and contains a thick succession of largely carbonate strata that correlate with the Jinxian Group in the Dalian (Liaoning, China) area.

Detrital zircon and intrusive diabase zircon and baddeleyite U-Pb ages indicate an early Neoproterozoic age for the Huaibei and Jinxian successions (Liu et al., 2006; Gao et al., 2009; Yang et al., 2012; Wang et al., 2012). A Tonian age is also supported by age-suggestive macrofossils (Dong et al., 2008; Xiao et al., 2014), age-diagnostic acritarchs (Tang et al., 2013, 2015), and limited published carbon-isotope (Zang and Walter, 1992; Yang et al., 2001; Zheng et al., 2004; Xiao et al., 2014) and Sr-isotope (Fairchild et al., 2000; Yang et al., 2001; Xiao et al., 2014; Kuang et al., 2011) data. Dike swarms and sills, intruded along the southeastern margin of the North China craton between ca. 920 and 900 Ma, provide a minimum age for the successions and are named the Dashigou-

CDS (Chulan-Dalian-Sariwon)[[correct?]] large igneous province (LIP) (Peng et al., 2011). The similarity in intrusion ages across the North China craton (including the Korean peninsula) implies that widespread crustal extension and related magmatism occurred shortly after deposition had ceased at Jinxian and Huaibei, possibly due to pre-magmatic regional uplift after ca. 0.92 Ga (Zhang et al., 2016; Zhu et al., 2019). Recent detrital zircon (He et al., 2016**[[He et al., 2016 is not in the reference list.]]**; Wan et al., 2019**[[Wan et al., 2019 is not in the reference list.]]**) and magmatic baddeleyite ages for Jinxian (Fu et al., 2015; Wang et al., 2012) and Huaibei successions (Zhu et al., 2019) constrain the maximum depositional age of uppermost carbonate successions to ca. 920 Ma (see the Data Repository). Based on all available geochronological data, deposition of these carbonate strata ranged between ca. 980 Ma and ca. 920 Ma (see the Data Repository).

METHODS

We collected 235 carbonate samples from the Huaibei Group. In order to evaluate their suitability for Sr isotope stratigraphy, all samples underwent thorough diagenetic screening using a combination of field- and laboratory-based observations. Samples were initially vetted in the field, whereby limestone examples of early-lithified cavity-filling CMC were favored. Samples were studied petrographically before targeted analysis of microdrilled powder for their trace elemental, as well as stable carbon and oxygen, and radiogenic Sr isotopic compositions.

Stable isotopes (δ^{13}C and δ^{18}O) were analyzed at two laboratories: the Bloomsbury Environmental Isotope Facility at University College London (UCL, UK), on a ThermoFinnigan Delta PLUS XP mass spectrometer attached to a ThermoScientific

Gas Bench II device; and the State Key Isotope Laboratory for Palaeobiology and Stratigraphy, Nanjing Institute of Geology and Palaeontology, Chinese Academy of Sciences, on a Finnigan MAT-253 mass spectrometer fitted with a Kiel IV carbonate device. Both laboratories have controlled temperatures of 22 °C ± 1 °C and relative humidity of 50% ± 5%.

The use of trace element ratios for diagenetic screening has been discussed in many publications (McArthur, 1994; Kaufman and Knoll, 1995; Montañez et al., 1996; Jacobsen and Kaufman, 1999; Brand, 2004; Brand et al., 2012), although there are no agreed criteria (see Fig. DR3 in the Data Repository). For our study, no cutoff criteria have been applied, but three simple principles were applied for elemental screening: (1) low Mn/Sr mass ratio (in most cases ≤0.5); (2) high Sr concentration (in most cases ≥200 µg/g); and (3) low Mg/Ca mass ratio (in most cases <0.01). Elemental analyses were carried out at UCL, using both inductively coupled plasma (ICP)–optical emission spectrometry (Varian 720-ES) and quadrupole ICP–mass spectrometry (Varian 820-MS). For Sr isotope analyses, a sequential leaching technique based on Bailey et al. (2000) was applied before extraction of Sr using cation-exchange columns. Analyses were carried out at Royal Holloway, University of London (RHUL, UK), and also at Nanjing University (NU, China) by the lead author. Samples were leached sequentially twice in dilute acetic acid (0.13 M in RHUL; 0.05 M in NU). Standard ion chromatography was used on the second leach (20%–70% of the total carbonate sample) to concentrate Sr and eliminate Rb before analysis by thermal ionization mass spectrometry (Phoenix Isotopx at RHUL, with isotopic standard SRM 987 mean **[[^{87}Sr/^{86}Sr = ?]]**0.710240 ± 8**[[Should this have a decimal point and some number of zeroes before it, to indicate what decimal place**

this refers to?]], 2 SD [standard deviations]; and Thermo Scientific Triton at NU, with SRM 987 mean **[[^{87}Sr/^{86}Sr = ?]]**0.710244 ± 3**[[What decimal place is this?]]**, 2 SD).

RESULTS

Values of $\delta^{13}C_{carb}$ (carb—carbonate) and ^{87}Sr/^{86}Sr of Huaibei Group samples in this study are presented in Figure 1. The data show that most Huaibei $\delta^{13}C_{carb}$ values lie between ~0‰ and +5‰, averaging +2.6‰ (±1.4‰), which is similar to previously published early Tonian data from the southern Ural Mountains (Kuznetsov et al., 2006; 2017). Lowermost bulk and CMC ^{87}Sr/^{86}Sr values from best-preserved samples, based on the screening described above, define a gentle fall from ~0.7058 to ~0.7052 from the Jiayuan to the Jiudingshan Formation, followed by a return to ~0.7056, a slight dip to ~0.7055, and a final rise to ~0.7061 through the Wangshan Formation (Fig. 1). The profile described here traces the lowest value for stratigraphic levels for which systematically less-radiogenic CMC and some well-preserved bulk samples are both present, and to which the strictest screening has been applied. The curve, therefore, represents a conservative estimate for primary oscillations of the contemporaneous seawater ^{87}Sr/^{86}Sr curve. Published data from the Jinxian Group (Dalian) imply a further rise to ~0.7064 in the uppermost units there (Fairchild et al., 2000; Kuang et al., 2011), which are dated to ca. 920 Ma (Yang et al., 2012; Zhang et al., 2016).

THE NEOPROTEROZOIC STRONTIUM ISOTOPE CURVE AND DISCUSSION

Here we use the compilation of Cox et al. (2016) as a foundation for a new seawater ^{87}Sr/^{86}Sr curve. The general age models of individual successions were constructed either from basic thermal subsidence modeling where possible, or by linear interpolation between correlated ages based on the assumption of constant sedimentary rates (Cox et al., 2016). The latter is used for the Huaibei data from this study and Xiao et al. (2014) in Figure 2. The trend outlined in our study is similar to that reported for the Urals by Kuznetsov et al. (2017), which could indicate that the North China craton and Urals successions are of comparable age. This would be in agreement with the approximate ages assigned by Cox et al. (2016) to those successions. Furthermore, it suggests that the overall rise is followed by a return to less-radiogenic values of ~0.7053, documented from the Uk Formation in the southern Urals (Kuznetsov et al., 2006).

The new curve (Fig. 2B**[[Fig. 2 does not appear to have a panel B (check all call-outs in the text)]]**) confirms an overall trend toward increasing seawater $^{87}Sr/^{86}Sr$ values through the entire Neoproterozoic, punctuated by “knickpoints” or falls in the curve. The general trend indicates therefore increasing influence from weathering of radiogenic continental crust relative to hydrothermal input, punctuated by intervals of lower $^{87}Sr/^{86}Sr$ when Sr sources to the oceans became less radiogenic. The part of the curve that covers the interval of this study (ca. 980–920 Ma) shows a dip from ~0.7058 to ~0.7052 (similar to that seen also in the southern Ural Mountains), then an abrupt rise to ~0.7064 before a sharp fall to ~0.7052 by ca. 920 Ma, which approximately coincides with the eruption of the Dashigou LIP (Peng et al., 2011) that presumably increased the influx of less-radiogenic Sr via both hydrothermal input and basalt weathering. This extensional magmatism could represent early signs of Rodinia breakup, but proximity to contemporaneous arc magmatism to the east (Kee et al., 2019) implies lithospheric thinning in a craton interior, and possibly a backarc setting instead. Other falls in Tonian seawater $^{87}Sr/^{86}Sr$ were also preceded by LIP eruptions, e.g., the **[[Provide a geographic**

location for each of the following LIPs]]Baish, Guibei, Kangding, Shaba, and later Franklin events (Fig. DR3) just before the onset of Sturtian "snowball Earth".

Although the weathering of LIP basalt may lead initially to a decrease in the seawater $^{87}Sr/^{86}Sr$ value (flood basalt generally exhibits a near-mantle Sr isotope composition), the age distribution of widespread extension, represented by passive margins and the breakup of supercontinents, correlates well with increasing seawater $^{87}Sr/^{86}Sr$. In this regard, the staged breakup of the supercontinent that followed later Tonian LIP eruption events could have exposed old, more-radiogenic craton interiors to weathering at newly formed passive margins, and could have changed the climates of continental interiors, potentially enhancing erosion and therefore chemical weathering. Following the final phases of Rodinian assembly, this could explain why, following episodic steep dips of the global curve, seawater $^{87}Sr/^{86}Sr$ continued to rise toward its eventual high point of ~0.709 (Goddéris et al., 2017).

Our new updated compilation of strontium isotopes (Fig. 2B) and LIPs (see details in Fig. DR3) hints that the weathering of LIPs had a considerable influence on ocean composition well before the postulated timing of Rodinia breakup. Chemical weathering of freshly erupted mafic volcanic rock at low latitudes was likely a major source of nutrient phosphorus to the Tonian ocean (Horton, 2015; Gernon et al., 2016; Cox et al., 2016; Jenkyns, 2010; Pogge von Strandmann et al., 2013), rendered oligotrophic and ferruginous after prolonged denudation of the long-lived supercontinent Rodinia (Guilbaud et al., 2015). Nutrient input into a largely anoxic ocean would have driven carbon (and potentially also pyrite) burial at productive ocean margins, while the subsequent oxygenation could conceivably have facilitated the opportunistic radiation of large, aerobic eukaryotes reported from the North China craton (Dong et al., 2008; Tang et al., 2013, 2015). Pending further study, and consistent with reports of major carbon-isotope fluctuations in these and correlative successions (Hua and Cao, 2004; Xiao et al., 2014; Park et al., 2016; this study), we postulate an earlier, more eventful end to the "boring billion" than previously envisaged.

CONCLUSIONS

This is the first study that specifically uses carbonate components (in this case, demonstrably early and isotopically pristine, cavity-filling calcite microspar cements as well as well-preserved bulk carbonate) to reconstruct Neoproterozoic seawater $^{87}Sr/^{86}Sr$. Together with published data, we document a series of oscillations in $^{87}Sr/^{86}Sr$ that can plausibly be linked to the weathering of known volcanic provinces (Fig. 2). Although the weathering of large igneous provinces has previously been implicated in end-Tonian events coincident with supercontinent breakup, we conclude that the weathering of flood basalts exerted a considerable influence on ocean composition well before the postulated breakup of Rodinia.

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FIGURE CAPTIONS

Figure 1. Carbonate carbon and strontium isotope data for Huaibei Group in the Huaibei area (Jiangsu), North China craton. VPDB—Vienna Peedee belemnite. Data are shown alongside a stratigraphic log of the Huaibei Group, published ages for the eastern block of North China craton, and inferred correlation between the Huaibei and Jinxian Groups. **[[Define the grain-size abbreviations used at the bottom of the rock log]]** Also shown is a geological map of the eastern block of the North China craton (NCC). Data points that did not pass screening are not shown.

[[In the figure, in the column headings at top, capitalize only the first word and proper nouns in each heading, spell out “Stratigraphic”, correct the spelling of “height”, and change “maps” to “map”. Under “Published ages”, adjust the

topmost age so that the superscript “1” isn’t overprinted by the red box; change instances of “~” to “ca. ”. In the map, include a north arrow; redo the labels that look like they have been stretched diagonally (they should only be rotated, not skewed); change hyphen to en dash for “Trans–”, and make “orogen” lowercase; make instances of “belt”, “massif”, and “block” (except for the one in all caps) lowercase; capitalize “Ocean”. In the legend, capitalize (only) the first word of each label, plus proper nouns (make “block” and “belt” lowercase); add an explanation for crossbedding(?) symbol and the “SB” label shown along the rock log; the “Stromatolite bank” symbol does not appear to be shown in the figure; spell out “CMC”; reword “Unfinished formation” to make it clear what this means (and make sure that the gray-dashed symbol actually appears in the figure). At the bottom of the figure, the citation “He et al., 2016” is not in the reference list; change periods to em dashes after reference ID numbers; put reference years in parentheses instead of setting them off with commas; insert a comma after “Yang et al.”; in the “YPM” definition, change the colon to an em dash, and make the definition all lowercase with no bold letters]]

Figure 2. Isotopic evolution of Neoproterozoic seawater: proposed Neoproterozoic seawater $^{87}Sr/^{86}Sr$ curve (black line with blue halo); new compilation of global carbonate $\delta^{13}C$ (~~gray~~ circles; VPDB—Vienna Peedee belemnite); updated large igneous province (LIP) record during 1050–500 Ma (light-red bars; bar heights indicate size of LIP); and supercontinent cycle during 1050–500 Ma (Bradley, 2008) (red and green horizontal bars) **[[Explain the black vertical hatch marks shown in the supercontinent cycle]]**.

Light-blue columns in background mark three known glaciations, from old to young: Sturtian, Marinoan, and Gaskiers. The updated compilation of LIPs from 1050–500 Ma is based on Ernst et al. (2008), and the updated compilation at http://www.largeigneousprovinces.org/. Additionally, the sizes of ca. 920 Ma Dashigou LIP and Bahia-Ganila LIP (in the North China craton) were taken from Peng et al. (2011) and Chaves et al. (2018) **[[respectively?]]** (for more detail, see Fig. DR3 [see footnote 1]). For $\delta^{13}C$ data, gray circles are published data compiled by Cox et al. (2016); green circles are data from Xiao et al. (2014); red circles are from this study. For $^{87}Sr/^{86}Sr$ data, red stars are data from this study; details of all other data (diamonds) can be found in Cox et al. (2016).

[[In the figure, fix the cut-off superscripts on the left side of the figure; at the top, make “assembly” and “breakup” lowercase (make “breakup” one word); make “glaciation” lowercase.]]

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