Oxygen isotope composition of meltwater from a Neoproterozoic glaciation in South China

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ABSTRACT

The water cycle is an integral part of Earth surface dynamics, and water's oxygen-isotope composition retains information about the forcing and response of Earth's local and global climate. Water isotope signals of the recent geological past can be directly obtained from archives such as ice cores, groundwater, or pore fluid. For the more distant past, mineral proxies have to be used. Multiple episodes of global glaciation may have occurred in the Neoproterozoic Era, of which the record of oxygen-isotope composition of glacial melt-water is sparse; the few records that are derived from carbonate minerals are prone to late-burial and metamorphic alteration, and therefore subject to alternative explanations. Here we present a case in which meltwater $\delta^{18}O$ is retrieved from barite (BaSO$_4$) and malachite (Cu$_2$CO$_3$(OH)$_2$)–associated sulfate (MAS) in a diamictite in Kaiyang, Guizhou, South China. The core of our argument is based on the lowest-ever-published sulfate $\delta^{18}O$ values found in the barite and MAS, reaching as low as –20.3‰ (Vienna standard mean ocean water, VSMOW). These data suggest that the water involved in the oxidative weathering of these chalcocite clasts had a $\delta^{18}O$ value of –34‰ ± 10‰, similar to that of polar glaciers today. Excluding the possibility of glacier meltwater alteration during the past 600 m.y. after the deposition of the diamictite, the sulfate mineral assemblage reported here provides an important constraint on the nature of the Neoproterozoic glaciation that the Kaiyang diamictite represents.

INTRODUCTION

When the oxygen- and hydrogen-isotope composition of liquid, vapor, or solid water are measured, information such as water-cycle dynamics, local meteorology, plant physioecology, temperature, and the relative humidity of the moisture source are revealed (Bowen and Wilkinson, 2002; Landais et al., 2008; Lee et al., 2007; Rozanski et al., 1993; Schrag et al., 1996). Accordingly, valuable information from water isotope composition in Earth's distant past can be obtained if suitable proxies are available.

The isotope composition of glacial water is of particular interest for studying global Quaternary paleoclimate. Due to the well-preserved ice-core record for the past several hundred thousand years, the δD and $\delta^{18}O$ of ice-core ice have become indispensable for this purpose, as have the recently employed $\delta^{17}O$ (Landais et al., 2008).

The isotope signal of glacial water in the more distant past has been sparse. Usually, extremely ^{18}O-depleted signals in rocks or minerals suggest the involvement of glacial meltwater in precipitation or in meteoric-hydrothermal alteration. The 1.9 Ga Paleoproterozoic mid-grade metamorphic terrestrial silicate rocks in the east Baltic Shield have $\delta^{18}O$ values as low as −27.3‰ (Vienna standard mean ocean water [VSMOW]; in the subsequent text, the standard of comparison is VSMOW and the abbreviation is omitted, except where we used the Peedee belemnite [PDB] or

Vienna PDB [VPDB]), probably inherited as an alteration signal from glacial meltwaters from the 2.4–2.3 Ga Paleoproterozoic glaciation to its parent rock (Bindeman and Serebryakov, 2011). Some of the calcite cements of siltstone and early calcite spars in the Nuccaleena Formation, deposited after the ca. 635 Ma Marinoan glacial meltdown in Australia, have $\delta^{18}O$ values as low as ~−25‰ (VPDB) (equivalent to ~+5‰ VSMOW), and were interpreted to indicate glacial meltwater signal (Kennedy et al., 2008). Similarly, the upper carbonate unit of the Lantian Formation in Anhui, South China, possesses extremely low $\delta^{18}O$ values, ranging from −18.6‰ to −25.6‰ (VPDB) (equivalent to ~+11.5 to +4.5‰ VSMOW), which was suggested to be deposited in meltwater of the Gaskiers glaciation, a younger Neoproterozoic glaciation (Zhao and Zheng, 2010).

Multiple glaciations may have occurred in the Cryogenian and Ediacaran Periods of the Neoproterozoic Era. Some of those glaciations may have been so severe that the Earth was in a "snowball" state with ice covering the entire Earth from pole to pole (Hoffman et al., 1998). Glacial melt-water isotope signals, if available, could provide information on the nature and dynamics of the global water cycle associated with Neoproterozoic global glaciation events. For example, modern continental or polar glacial ice has a $\delta^{18}O$ value at least as negative as −30‰ while alpine glaciers have a much less negative $\delta^{18}O$ value, with its bulk usually no more negative than −17‰ even in moderately high latitudes (Schotterer et al., 1997). Modern marine ice has a $\delta^{18}O$ value at ~+2‰ (Tison et al., 2001). Although highly valuable, the above-mentioned two studies on isotope signals of Neoproterozoic glacial meltwater have been exclusively on carbonate minerals. One problem with carbonate is that its $\delta^{18}O$ value is sensitive to both temperature and the $\delta^{18}O$ of water from which it precipitated or re-equilibrated. The 5‰ value can be achieved at 22 °C with water $\delta^{18}O$ at −24‰, or at 320 °C with water $\delta^{18}O$ at 0‰, or at some combination in between such as at 121 °C with water $\delta^{18}O$ at −10‰, a reasonable scenario during low-temperature hydrothermal alteration or deep-burial recrystallization with meteoritic water influence. Furthermore, carbonate $\delta^{18}O$ is known for its vulnerability to alteration by post-depositional processes.

Here we present a case in which the meltwater $\delta^{18}O$ value can be inferred from the $\delta^{18}O$ of sulfate derived from oxidative weathering of chalcocite clasts in a continental glacial deposit. Specifically, sulfate minerals within or in the immediate vicinity of chalcocite clasts in a Neoproterozoic diamictite in the South China Block possess the most negative $\delta^{18}O$ value of sulfate ever reported. This new proxy should prompt us to take a fresh look at continental glacial deposits.

GEOLOGICAL AND PETROGRAPHIC CONTEXT

The Yangtze Block or South China Block has one of the best-exposed Neoproterozoic stratigraphic sequences in the world (Jiang et al., 2011; Zhang et al., 2008). Most of the studies in this region have been conducted on platform and platform-margin facies, where the Nantuo diamictite and its associated cap carbonates are well exposed. In central Guizhou and central-eastern Yunnan provinces, which are close to two exposed land masses known as the Qianzhong and Kangdian paleolands, respectively, the occurrence of diamictite of the Marinoan glaciation is less clear. No typical cap carbonates have been identified in these localities. Deposits

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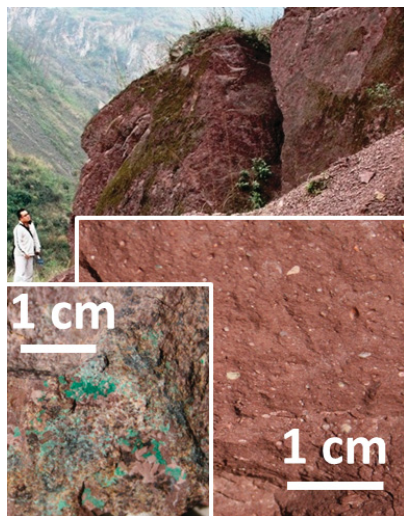†E-mail: bao@lsu.edu.

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of Marinoan and lower Doushantuo Formation equivalents are dominated by purple-colored clastic rocks. In the Kaiyang area, central Guizhou, a diamictite unit (~8 m thick) overlies red-purple thin-bedded siltstones and mudstones, and has a lower layer (~1.5 m) rich in fist-sized clasts and an upper massive layer (~6.5 m) composed of mostly sand- and silt-sized clasts and fine pebbles (<1 cm in size), but with no boulders. This diamictite unit is overlain by ~45 m of reddish medium-bedded sandstones interbedded with siltstones and shales, which are further overlain by greyish-green sandstones. The diamictite is freshly exposed and has experienced little metamorphic alteration. The sequence is of late Neoproterozoic age on the basis of stratigraphic correlation and sedimentary features. Although a radiometric age remains elusive, an earlier study (Liu et al., 1966) supports its glacial origin and tentatively correlated it with the Marinoan Nantuo diamictite found in much of South China.
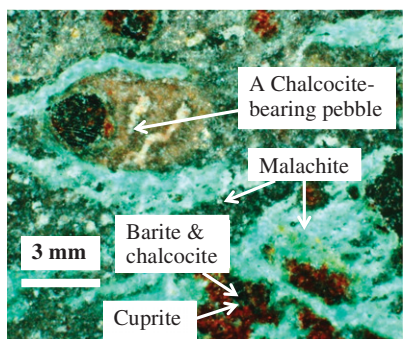
We observed a distinctly bright-green patch on a freshly exposed outcrop of the upper massive part of the Kaiyang diamictite (GPS reading: 27°08.11′N, 106°51.64′E) (Fig. 1). Petrographic (thin sections and slabs), X-ray diffraction (XRD), and scanning electron microscope (SEM) examination reveal that the bright-green mineral is malachite ($Cu_2(CO_3)(OH)_2$), occurring in the vicinity of a cluster of chalcocite clasts in the diamictite (Fig. 2). Importantly, the chalcocite clasts are "corroded" with two other mineral phases, barite and cuprite (Figs. 2 and 3), which, unlike the malachite, are not observed outside the chalcocite clasts in the bulk diamictite.
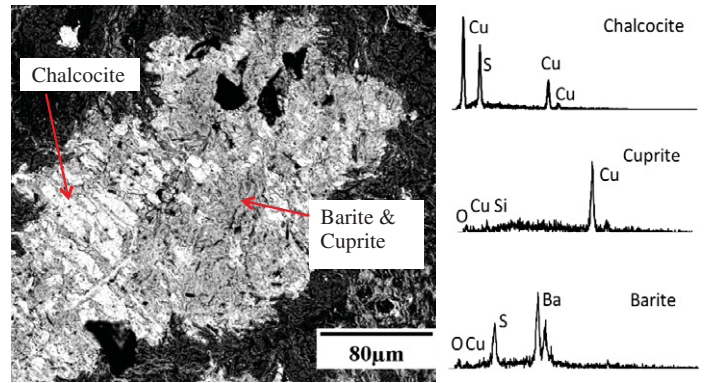
SULFATE ISOTOPE RESULTS

The method for the extraction and measurement of sulfate sulfur- and oxygen-isotope composition from barite and malachite can be found in the GSA Data Repository[1]. Both barite sulfate and malachite-associated sulfate (MAS) display arrays of $\delta^{34}S$ and $\delta^{18}O$ values among multiple samples taken

Figure 3. Backscattered scanning electron microscope image of a polished slab (left) showing a chalcocite grain (bright white) surrounded by barite and cuprite (gray). Mineral identification was assisted by readings from energy-dispersive spectrometry on individual spots (right).

within or from the vicinity of the chalcocite clasts. For MAS, the closer the samples are to the chalcocite clasts, the more negative these values are. Further away from the clasts, both the $\delta^{34}S$ and $\delta^{18}O$ become more positive and more scattered. Most remarkably, both barite sulfate and MAS possess extremely low $\delta^{18}O$ values, as low as –20.3‰. Note that the lower end of the sulfate $\delta^{34}S$ value is at ~–12.2‰, the same as that of the chalcocite, and none of the sulfate has a distinct ^{17}O anomaly (Fig. 4; Table DR1 in the Data Repository). In addition, the $\delta^{13}C$ and $\delta^{18}O$ of the malachite display a good linear array with those of the dolostone clasts in the diamictite (Fig. DR1).

DISCUSSION

Sulfate with $\delta^{18}O$ values as low as –20.3‰ is known only in modern polar areas where sulfate oxygen incorporates a portion of the extremely low $\delta^{18}O$ signal from glacial meltwater. In the last comprehensive review of published sulfate $\delta^{18}O$ data, Van Stempvoort and Krouse (1994) showed

Figure 1. Outcrop and close-up views of upper massive part of Kaiyang diamictite, central Guizhou, South China. Upper panel: Light-colored peaks in distance are overlying dolostones of late Neoproterozoic Dengying Formation (younger than ca. 555 Ma) (Zhang et al., 2005). Lower left panel: Green patches are malachite minerals in vicinity of chalcocite clasts (X-ray diffraction data not shown). Lower right panel: Sand- and silt-sized clasts in the diamictite.

Figure 4. $\delta^{18}O$ (Vienna standard mean ocean water, VSMOW) and $\delta^{34}S$ (Vienna Canyon Diablo Troilite, VCDT) of malachite-associated sulfate or barite sulfate extracted with different methods and at different distances from chalcocite clasts. Standard deviation for $\delta^{18}O$ and $\delta^{34}S$ are ±0.5‰ and ±0.2‰, respectively, smaller than the symbols in the diagram.

Figure 2. Microphotograph of a polished Kaiyang (South China) diamictite showing green malachite and reddish cuprite in vicinity of dark chalcocite clasts (reflected light). Barite is not easily identified on slab surface.
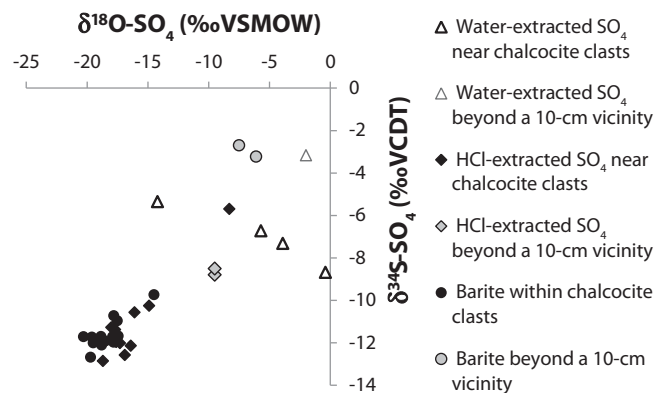
that, with no exception, all natural-occurring sulfate with $\delta^{18}O$ less than $-10‰$ are of high-latitude origin. In fact, except for the case of groundwater sulfate from glacial tills in southern Alberta, Canada, where the $\delta^{18}O$ of sulfate reaches $-19‰$ (Hendry et al., 1989), all other high-latitude cases have sulfate $\delta^{18}O$ no more negative than $-12‰$. Recent data from mine tailings from an alpine site in central Chile has sulfate $\delta^{18}O$ values as low as $-10‰$ (with an average of $-7‰$) (Dold and Spangenberg, 2005). A study in the MacKenzie River Basin, Canada ($55°N$ to $65°N$), showed that sulfate from oxidative weathering has a $\delta^{18}O$ value as negative as $-12‰$ where the river water $\delta^{18}O$ reaches as low as $-22.5‰$ (Calmels et al., 2007). Therefore, except for southern Alberta (Hendry et al., 1989), known sulfates with $\delta^{18}O$ lower than $-18‰$ are only from Antarctica (Bao and Marchant, 2006; Socki et al., 2008). Even in the southern Alberta case where the sulfate-water oxygen-isotope fractionation was believed to be minimal, the groundwater sulfate is probably derived from oxidative weathering in old glacial meltwater.

The low-$\delta^{34}S$ end-member MAS and barite sulfate have the same $\delta^{34}S$ value as that of chalcocite (Fig. 4), indicating that barite and MAS in the close vicinity of the chalcocite clasts were the product of direct oxidation of chalcocite. This is because sulfate derived from the oxidation of sulfide has the same $\delta^{34}S$ as that of its source sulfide (Seal et al., 2000). Thus, the clustered $\delta^{18}O$ and $\delta^{34}S$ values around $-20‰$ and $-12‰$, respectively, in Figure 4 belong to sulfate derived from oxidative weathering of chalcocite clasts in the Kaiyang diamictite when the diamictite was influenced by glacial meltwater.

Chalcocite (Cu_2S) clasts were apparently mixed into the Kaiyang diamictite from an old source, the Mesoproterozoic rocks where copper ore deposits are known to be widespread in nearby paleolands (Huang et al., 1996). For most geologists, a patch of green malachite on an outcrop is an indication of copper-bearing minerals in the host rock, and the malachite is most likely of recent weathering origin. We cannot rule out the possibility that part of the malachite in the Kaiyang diamictite is indeed of recent weathering origin. However, the stable oxygen and sulfur isotope data of the sulfate, especially the extremely negative $\delta^{18}O$ values and their pattern, argue for a syndepositional origin for barite within, and some malachite in the immediate vicinity of, the chalcocite clasts. Note that although geological, petrographic, and diagenetic assessment of the mineral assemblage in question is consistent with our conclusion, the conclusion does not hinge upon it. The conclusion is based solely on the stable isotope data. The sulfate $\delta^{18}O$-$\delta^{34}S$ pattern (Fig. 4) and its scatter toward positive ends are an indication of the addition of later groundwater sulfate, which could have been as late as just before consolidation of the diamictite in the Neoproterozoic or as late as recent exposures. The linear array of the $\delta^{13}C$ and $\delta^{18}O$ for malachite-bearing rocks sampled at the vicinity of the chalcocite clasts also support this conclusion (Fig. DR1). The carbonate adjacent to the chalcocite clasts is a mixture of probably the original precipitates and those of the dolostone clasts commonly seen in the Kaiyang diamictite. A likely formation model for the mineral assemblage is that upon oxidative weathering of the chalcocite, sulfate was released into pore spaces where Ba^{2+}, which is an abundant element in clastic rocks, competed with Cu^+ to form sulfate minerals. Due to the low solubility of $BaSO_4$, Cu^+ ended up either as Cu_2O (cuprite) or as copper carbonate minerals such as malachite when the pore solution later dried up. Some of the original sulfate was also trapped in the malachite as MAS.

Could the low-$\delta^{18}O$ sulfate in Kaiyang have formed at any time after the Neoproterozoic glaciation? It is possible but very unlikely. Meltwater from glacier ice, not alpine snowmelt at a low- to mid-latitude site, is required to explain the low sulfate $\delta^{18}O$ value. The Kaiyang diamictite is overlain by >1000 m of Neoproterozoic and Paleozoic sedimentary rocks, and was only recently uplifted due to the rise of the Tibetan Plateau in the late Cenozoic. Glacial meltwater in the Phanerozoic, if any, would have had to penetrate to depths to oxidize the solidified diamictite, a scenario difficult to reconcile with an oxidizing and dry condition required to

convert chalcocite sulfide to sulfate, to mobilize Cu, and to precipitate barite, cuprite, and malachite. Besides, there is no evidence of late meteoric water alteration in these sedimentary units. Finally, the probability of chalcocite oxidation by glacial meltwater after the uplift of Guizhou-Yunnan Plateau in the late Cenozoic is low because central Guizhou has not been glaciated since its uplift, due to its low-latitude position.

To estimate the $\delta^{18}O$ of Neoproterozoic meltwater that must have been responsible for the extremely ^{18}O-depleted sulfate, we have to know the fractionation factor α^{18} between sulfate and ambient water during oxidative weathering. It is known that both H_2O and O_2 contribute to the oxygen in the final sulfate, which complicates the α^{18} value. As demonstrated by a recent experimental study (Kohl and Bao, 2011), the final apportionment of the two oxygen sources in SO_4^{2-} is determined by a set of non-equilibrium, dynamic processes that are dependent on chemical conditions. This means that the α^{18} (sulfate-water) value varies widely. Using modern sulfate-groundwater paired data, especially those from western Canada (van Stempvoort and Krouse, 1994, their figure 7; Calmels et al., 2007), we estimate conservatively that the $\delta^{18}O$ difference between sulfate and water ($\Delta\delta$) is at $~14‰ \pm 10‰$. Thus, the most negative sulfate $\delta^{18}O$ value $-20.3‰$ would indicate a $\delta^{18}O$ value at $~-34‰ \pm 10‰$ for Neoproterozoic pore water in the Kaiyang diamictite. Water with this negative $\delta^{18}O$ value can only be found in polar glaciers today. At hydrothermal conditions, the $\Delta\delta$ value is likely to decrease somewhat due to higher temperatures. Given an extreme case where an oxidizing fluid is at $-10‰$ and a $\Delta\delta$ at $~5‰$, we would get sulfate $\delta^{18}O$ value at $-5‰$, still much higher than the $-20.3‰$ seen in Kaiyang. Thus a hydrothermal process with typical meteoric water could not have produced the observed $\delta^{18}O$-negative sulfate.

The lack of distinct ^{17}O depletion in the malachite or barite sulfate in the Kaiyang diamictite suggests that either the sulfate was not formed during a brief window immediately after the Marinoan meltdown at ca. 635 Ma, or the ^{17}O signal was not preserved (Bao et al., 2008). However, as it stands now the Kaiyang diamictite can be of any age between 725–551 Ma in the late Neoproterozoic as constrained by the base of the Cryogenian (Zhang et al., 2008) and the age of the overlying Dengying Formation (Condon et al., 2005).

South China was near the paleoequator at ca. 600 Ma (Macouin et al., 2004) and was located at lower than $30°N$ in paleolatitude during much of the 700–600 Ma period (Li et al., 2008). The extremely low $\delta^{18}O$ (SO_4) will have to be explained by glacier meltwater at low latitudes. While glacier ice at low latitudes does constitute a strong support for a "snowball" Earth origin of the Kaiyang diamictite, there has been no speculation or model prediction on the $\delta^{18}O$ of continental glacier ice or sea ice during or at the initial meltdown of the "snowball" Earth in the Neoproterozoic. If the Kaiyang diamictite turns out to be much younger than 600 Ma, we would have to consider the possibility of local continental glaciation because by this time the paleolatitude of South China could have been at $30°–40°N$ (Zhang et al., 2009). Adding to the issue is that the seawater $\delta^{18}O$ value at 700–600 Ma may not be the modern value of $0‰$ but could be as low as $-7‰$ (Kasting et al., 2006). There are many unknowns about the Neoproterozoic hydrosphere at this time. Our study, however, offers a unique set of mineral proxies for meltwater isotope signals and provides solid constraints on any paleoclimate model that accounts for the Neoproterozoic glaciation event.

CONCLUSION AND IMPLICATIONS

When pieces of chalcocite ore from underlying Proterozoic rocks were clasts in the Neoproterozoic Kaiyang diamictite in Guizhou, South China, oxidative weathering produced cuprite, barite, and some of the malachite within or in the immediate vicinity of the clasts. The lower end of the $\delta^{18}O$ value for barite sulfate and malachite-associated sulfate is at $-20.3‰$, indicating a $\delta^{18}O$ value of $-34‰ \pm 10‰$ for ambient water. Despite the uncertainty in its age, our sulfate isotope data indicate that the Kaiyang diamictite was deposited on land and is the record of one of the

Neoproterozoic glaciations that had almost the same meltwater $\delta^{18}O$ value as that of modern glaciers in Antarctica, which also independently supports the glacial origin of the Kaiyang diamictite. Our conclusion hinges upon the extremely negative sulfate $\delta^{18}O$ values and could be independently tested in the future using the oxygen-isotope composition of cuprite intimately mixed with barite and chalcocite.

A lack of information on the isotope composition of glacier ice in the Neoproterozoic, and in fact for almost all other geological glaciation events, is not surprising. The problem lies in the difficulty of identifying continental deposits as well as minerals of weathering origin at a time when fossils or root traces were not available. This study reports a new approach through which paleoweathering signals can be explored in diamictites or other types of continental deposits. These deposits are critical to information on Earth surface conditions that are unattainable in marine deposits.

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