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Low δ^{18}O zircons, U-Pb dating, and the age of the Qinglongshan oxygen and hydrogen isotope anomaly near Donghai in Jiangsu Province, China

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Abstract—Zircons from metamorphosed granites exposed near Qinglongshan have $\delta^{18}O_{VSMOW}$ values of −7 to 0‰ in both grain rims and cores. The concordant $^{238}U/^{206}Pb$ ages of zircon cores are 684 to 754 Ma with rims at 221 Ma. Discordant $^{238}U/^{206}Pb$ ages range from 242 to 632 Ma. Results demonstrate a Neoproterozoic age for the origin of the Qinglongshan oxygen and hydrogen isotope anomaly. The low δ^{18}O values were imprinted on the rocks by a hydrothermal system charged with meteoric water from a cold climate. Groundwater circulation was driven by heat from cooling granitic magma. The geologic age of the hydrothermal system correlates with that of the Nantuo tillite in the Sinian strata of the South China block, suggesting that Qinglongshan's cold climate may be a manifestation of Neoproterozoic "snowball Earth." *Copyright © 2002 Elsevier Science Ltd*

1. INTRODUCTION

The Qinglongshan oxygen and hydrogen isotope anomaly records an ancient hydrothermal system subducted into the upper mantle during Triassic continental collision, metamorphosed under coesite–eclogite facies conditions, and exhumed to Earth's surface (Yui et al., 1995; Zheng et al., 1996, 1998; Rumble and Yui, 1998). The anomaly is defined by δ^{18}O values as low as −7.7‰ for quartz and −10.1, −10.7, and −14.6‰ for coexisting garnet, omphacite, and rutile, respectively (Zheng et al., 1996; Rumble and Yui, 1998; Table 1). Phengites are depleted in both $^{18}O/^{16}O$ and D/H with δ^{18}O and δD values of −5.7 and −127‰, respectively (Rumble and Yui, 1998; Table 2). It may be seen that all of the minerals in the ultrahigh pressure (UHP) metamorphic assemblage are depleted in $^{18}O/^{16}O$ and D/H and show an approach to equilibrium intermineral fractionation (Yui et al., 1995; Zheng et al., 1996, 1998; Rumble and Yui, 1998). The isotopic relationships were interpreted as indicating a presubduction, pre-UHP metamorphic age for acquisition of the depleted isotopic signature. The low ^{18}O and δD values of the anomaly have attracted attention from researchers not only because they afford a baseline by which to measure crust–mantle interactions during subduction, but also because they may offer proof of the existence of an ancient cold climate.

It has been proposed that the granitic rocks of Hushan and Fangshan outcropping within the Qinglongshan area provided the heat necessary to heat groundwater, drive convection, and promote isotope exchange between meteoric water and rocks (Rumble and Yui, 1998). Dating the granites provides a test of the proposal. A finding of granite ages younger than protolith ages of wall rocks but older than the age of UHP metamorphism would support the hypothesis. We report new analyses for $^{18}O/^{16}O$ and U-Pb in zircons from metamorphosed granites indicating a late Proterozoic age for the hydrothermal system, consistent with the hypothesis of granite-powered groundwater convection.

2. GEOLOGY

The Qinglongshan hydrothermal system outcrops over a known area measuring 50 by 50 km (Rumble and Yui, 1998). Qinglongshan is part of a larger hydrothermal system that includes a 50-km outcrop area in Dabieshan (Fig. 1), the rocks of which are also depleted in $^{18}O/^{16}O$ (Baker et al., 1997; Fu et al., 1999; Zheng et al., 1999). The two areas are now separated by 500 km of left-lateral, strike-slip displacement on the post-Triassic Tanlu Fault (Fig. 1; Rumble, 1998). The hydrothermal system thus has a minimum length scale of 100 km. Qinglongshan shows the characteristic features of unmetamorphosed hydrothermal systems, including unusually low δ^{18}O and δD_{VSMOW} combined with strong local heterogeneity (Taylor, 1971). Both the Qinglongshan and Dabieshan systems are preserved within coesite–eclogite facies rocks of the Dabie-Sulu orogenic belt formed during Triassic collision and subduction of the South China block beneath North China (Fig. 1). Triassic metamorphic conditions at Qinglongshan are estimated as 700 to 890°C and pressure greater than 28 kbar (Zhang et al., 1995).

3. PREVIOUS AGE DETERMINATIONS

Published geochronology on Qinglongshan rocks demonstrates the age of coesite–eclogite metamorphism to be Triassic—for example, 226 (Sm-Nd isochron), 219 (Rb-Sr isochron; Li et al. 1994; Li, 1996), and 217 Ma (U-Pb, lower discordia intercept; Ames et al., 1996). The evidence of oxygen isotope intermineral equilibration under high temperature conditions supports a premetamorphic age for acquisition of the low δ^{18}O

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Table 1. δ^{18}O (per mil) values of minerals from metamorphosed granites of Hushan and Fangshan.

Sample	Zircon	Abraded zircon	Quartz	Feldspar	Biotite
L001-3	−5.9	−5.6			
L006-5 (>200)	−4.9				
L006-5 (<200)	−4.2				
L006-5 (120)	−5.2				
L1008	−5.9	−5.6			
95-FA-R1	−2.4	−1.9			
95-FA-R1B	−5.7	−4.7			
95-HU-R2	−0.2	+0.2			
95-HU-R3	−7.4	−7.1			
95-FA-1A[a]			−1.6	−3.0	−6.9
95-FA-1C[a]				−1.3	−6.0
95-HU-1A[a]			−2.5	−2.0	−6.1
95-HU-1B[a]			−2.4	−3.8	−6.5
95-HU-1C[a]			−2.3	−4.3	−7.7

[a] Data from Rumble and Yui (1998), table 1.

signature (Rumble and Yui, 1998). Whole-grain analysis of zircon gives an upper discordia intercept at 762 ± 28 Ma, interpreted as the protolith age of Qinglongshan eclogite (Ames et al., 1996).

4. METHOD OF STUDY

We chose to focus analytical work on metamorphosed granites for two reasons. First, the granites have been suggested as the source of heat that drove the ancient hydrothermal system (Rumble and Yui, 1998). An accurate granite age should test the hypothesis and constrain the age of associated hydrothermal activity. Second, granites typically contain sufficient zircons to make possible the application of a number of analytical techniques. We sought to measure not only U-Pb ages but also δ^{18}O values in aliquots of zircon mineral separates to verify the age of ^{18}O/^{16}O-depleted oxygen. Large unweathered samples of metamorphosed granite were readily obtained from working quarries at Fangshan and Hushan located 8 and 2 km south of Qinglongshan, respectively (Rumble and Yui, 1998; Fig. 2).

Zircons were separated from crushed samples of metamorphosed granitic rocks via conventional methods of density and magnetic separation. By definition, $\delta^{18}O_{VSMOW} = 1000 \times [(R_A/R_{VSMOW}) - 1]$, where $R_A = {}^{18}O/{}^{16}O$ for unknown A and VSMOW is Vienna standard mean ocean water (Coplen, 1995). Oxygen isotope analyses were made on size fractions of <200 to 100 mesh weighing 1.5 to 2.5 mg. Oxygen was released for analysis by heating minerals with a CO_2-infrared laser in the presence of BrF_5 (Sharp, 1990). Oxygen gas was analyzed directly in the mass spectrometer but first separated from BrF_5 cryogenically. By-products of fluorination such as NF_3 and CF_4 were removed by adsorption via molecular sieve 5A (Rumble and Hoering, 1994). The oxygen analyses were standardized to a value of 5.8 for the UWG-2 garnet standard of Valley et al. (1995). A zircon from Tete, Mozambique (USNM R12984), analyzed at both the Geophysical Laboratory and the University of Wisconsin (Valley et al., 1994), gives $\delta^{18}O_{VSMOW}$ values of 5.0 and 5.3‰ at the University of Wisconsin and 5.3, 5.0, 4.9, and 5.1‰ at the Geophysical Laboratory. Whole-grain oxygen isotope analyses include both core and rim material. Analyses of cores were performed on zircons from which rims had been removed via air abrasion.

Uranium–lead analyses of rims and cores of individual zircon grains were made with a SHRIMP ion microprobe at Stanford University. A correction for common lead has been applied (Ireland and Gibson, 1998; Fig. 3).

5. METAGRANITE SAMPLES

The metamorphosed granite samples from Hushan and Fangshan are unweathered blocks weighing 5 to 7 kg collected from

Table 2. U-Pb analyses of zircons.

Sample	U (ppm)	Th (ppm)	Th/U	^{204}Pb/^{206}Pb	^{207}Pb/^{206}Pb	^{238}U/^{206}Pb	F 206	206/238 age (Ma)
M2								
1.1	117	1	0.009	0.00580	0.0598	28.93	10.69	219.1
2.1	224	3	0.013	0.00337	0.0551	28.77	6.21	220.2
3.1	106	3	0.028	0.00701	0.0369	29.31	12.90	216.3
4.1	201	3	0.015	0.00242	0.0564	28.34	4.46	223.5
5.1	153	2	0.013	0.00375	0.0572	27.11	6.90	233.5
L001-3								
1.1	119	97	0.815	0.00163	0.0668	8.06	2.85	753.7
2.1	548	158	0.288	0.00067	0.0561	16.45	1.22	380.4
3.1	894	377	0.422	0.00052	0.0592	13.78	0.95	451.5
4.1	222	152	0.685	0.00121	0.0686	9.71	2.14	632.1
5.1	196	203	1.036	0.00069	0.0642	8.56	1.21	712.2
L10008								
1.1	387	287	0.742	0.00045	0.0641	8.94	0.79	683.9
2.1	1047	24	0.023	0.01577	0.0488	28.66	28.85	221.1
3.1	2316	268	0.116	0.03023	0.0134	26.09	54.22	242.5
4.1	146	88	0.603	0.00385	0.0707	10.6	6.83	581.2
5.1	401	100	0.249	0.02162	0.0362	18.57	38.69	338.1

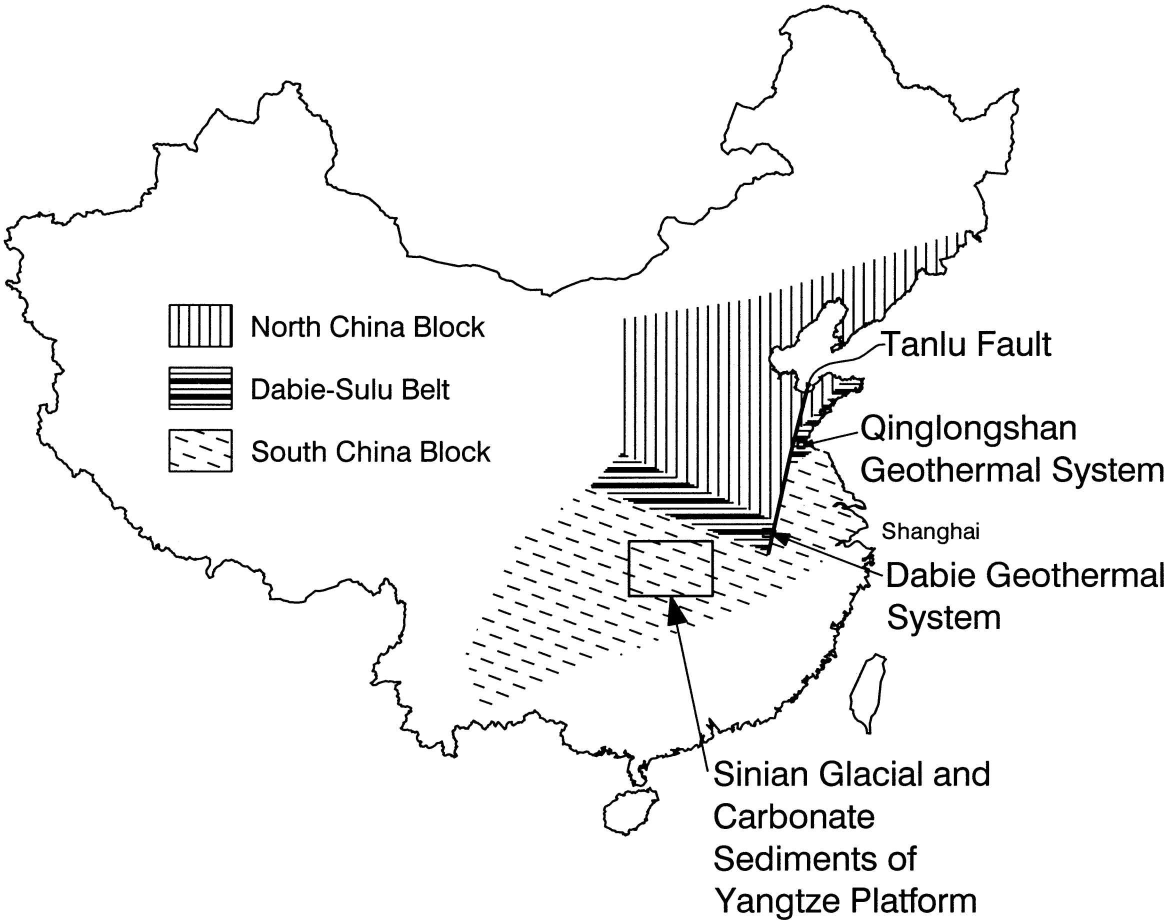

Fig. 1. Location of Qinglongshan and Dabie hydrothermal systems with coeval glacial and carbonate sediments of Yangtze platform. Also shown are Precambrian North China and South China blocks. The Dabie-Sulu orogenic belt formed when the two blocks collided in Triassic time.

active quarries. The metagranites are weakly foliated and consist of plagioclase, microcline, quartz, and biotite with minor garnet, epidote–clinozoisite, magnetite, monazite, amphibole, sphene, apatite, zircon, and allanite (Giorgis et al., unpublished data). Rare phengite inclusions in plagioclase have Si^{4+} contents of 3.4 atoms per formula unit recording the influence of UHP metamorphism (cf. Carswell et al., 2000).

Zircon crystals are 100 to 400 μm long with rounded to prismatic habit and are found as inclusions in feldspar. Zircon cores contain up to 10 vol% of inclusions of biotite, quartz, K-feldspar, magnetite, and apatite. Cathodoluminescence reveals that inclusion-rich cores lack oscillatory zoning but instead, have a chaotic, amoeboid structure of shadowy, rounded forms. Zircon rims are inclusion free and show irregular, patchy zoning under cathodoluminescence (Giorgis et al., unpublished data).

6. RESULTS

6.1. Oxygen Isotopes

Zircons from Hushan and Fangshan metagranites are heterogeneous from sample to sample, ranging from 0 to −7‰, $\delta^{18}O_{VSMOW}$ (Table 1). Within a given rock sample, zircon aliquots of different grain size show differences in δ^{18}O of up to 1‰ (Table 1, L006-5). Air-abraded zircons, presumably reflecting core vs. rim heterogeneity, are as much as 1‰ enriched in ^{18}O/^{16}O relative to whole-grain analyses (Table 1, 95-FA-R1B). The evidence of ^{18}O/^{16}O zonation in single crystals is weaker than that observed in zircons from low δ^{18}O rhyolites from Yellowstone caldera (core-to-rim gradients of 5‰; Bindeman and Valley, 2000) but it is consistent with zircon's known refractory behavior (Watson and Cherniak, 1997).

The hand-specimen-scale heterogeneity of δ^{18}O in zircon reflects heterogeneity in previously analyzed metagranites (Table 1; Fig. 2) and in wall rocks. Garnet separated from samples of Qinglongshan eclogite ranges from −1.6 to −10.3‰. Quartz from wall rock gneisses and eclogites is +2.0 to −7.7‰ (Rumble and Yui, 1998; Table 1). Isotopic heterogeneity is a characteristic feature of unmetamorphosed hydrothermal systems. Sheppard and Taylor (1974) report sericites from Butte, Montana, USA, ranging from −10 to +10‰ in δ^{18}O and −175 to −110‰ in δD. Such large isotopic heterogeneities are attributed to local differences in the intensity of alteration caused by differences in water/rock ratio, temperature, proportion of meteoric vs. magmatic water, proximity to permeable fracture

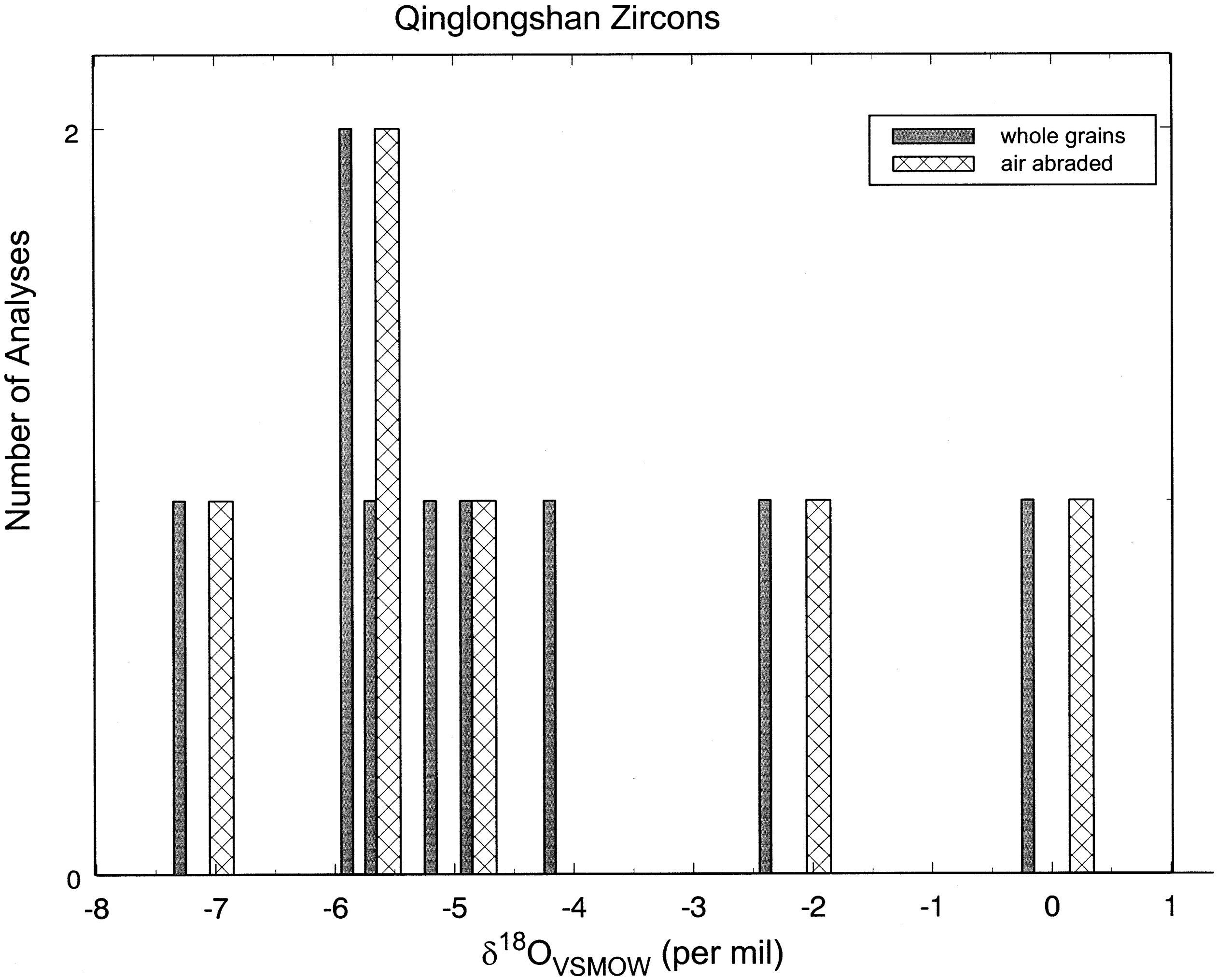

Fig. 2. Histogram of zircon $\delta^{18}O_{VSMOW}$ values. Core data were obtained by air-abrading previously analyzed whole grains.

zones, and superposition of successive hydrothermal events (Taylor, 1977).

6.2. U-Pb Isotopes

Two samples of Qinglongshan metagranites, L001-3 and L10008, give concordant zircon core ages of 684, 712, and 754 Ma and one concordant rim age of 221 Ma (Fig. 3). Zircon from a metamorphosed garnet peridotite collected at Zhimafang, 5 km west of Fangshan, has somewhat discordant ages of 216 to 233 Ma. The zircon core ages are consistent with data on late Proterozoic bimodal magmatic activity that accompanied rifting of a passive margin along the northern edge of the South China block (Ames et al., 1996). Compilation of pre-Triassic $^{238}U/^{206}Pb$ zircon ages from metagranites, gneisses, and eclogites of the Dabie-Sulu orogenic belt shows 60 of 77 analyses in the range 600 to 800 Ma (Xue et al., 1997; Hacker et al., 1998, 2000; Maruyama et al., 1998). Of special interest in the present study are whole-grain U-Pb ages of zircons from granites, eclogites, and gneisses whose upper concordia intercepts record igneous events in the interval 728 to 782 Ma (Ames et al., 1996). We conclude that Qinglongshan granites and their hydrothermal system are manifestations of a regional tectonic–magmatic event that took place during continental rifting in late Proterozoic time.

7. ORIGIN OF LOW $\delta^{18}O$ ZIRCONS

Granites intruded near Earth's surface, and their wall rocks are known to acquire oxygen and hydrogen isotope signatures of local meteoric water (Taylor, 1977). Intrusion of a hot magmatic body initiates groundwater convection by cracking wall rocks and establishing lateral temperature gradients driving convection. Heated water exchanges isotopes with wall rocks. Because flow system permeability is dominated by fractures, isotopic alteration is heterogeneous on scales of centimeters to meters. As the intrusion crystallizes and cools, the convecting groundwater system collapses on the intrusion and alters the minerals of the intrusion itself. Low $^{18}O/^{16}O$ magmas are generated in areas experiencing repeated episodes of intrusion; younger plutons assimilate and remelt the altered products

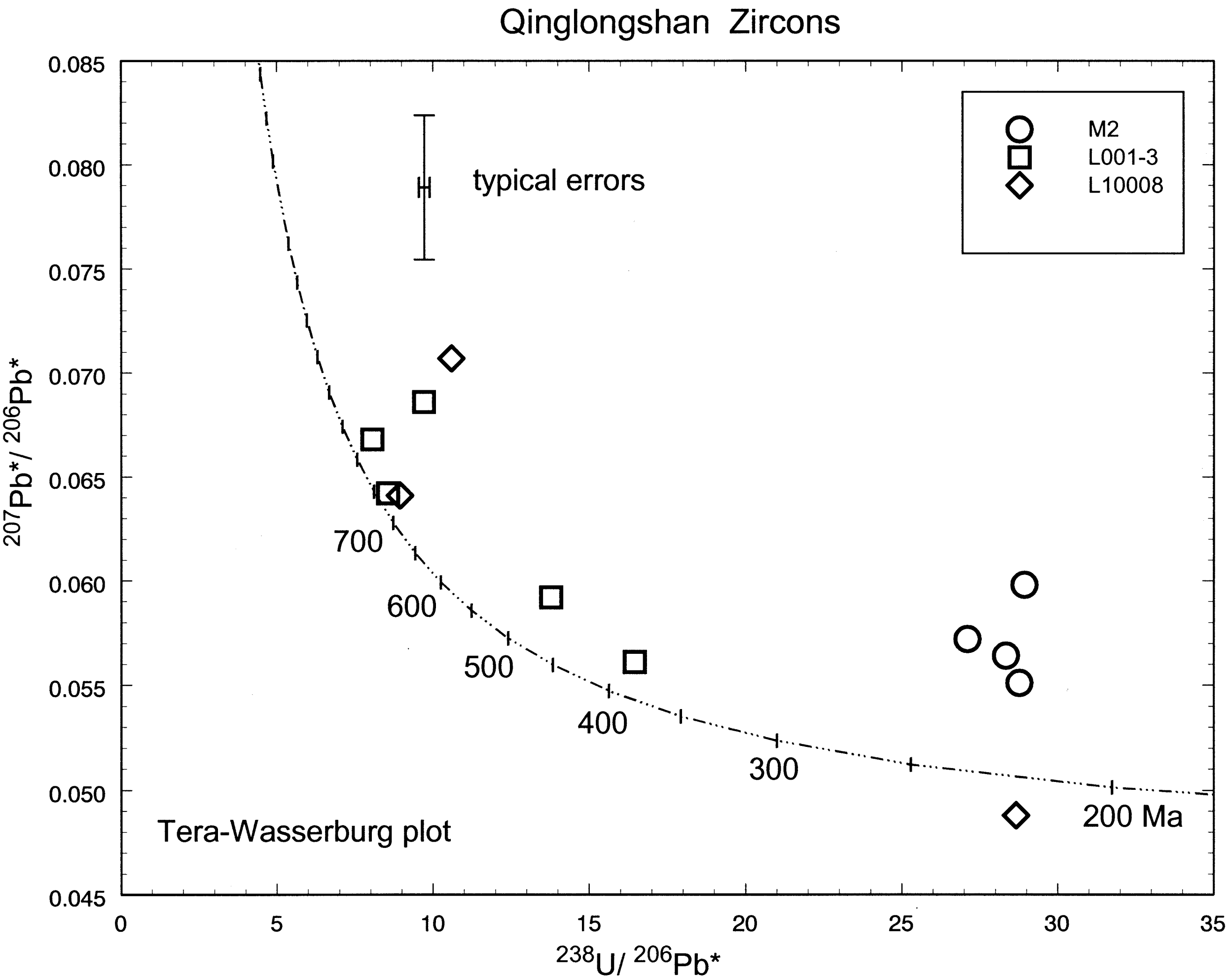

Fig. 3. Tera-Wasserburg plot of SHRIMP analyses of cores and rims of zircons from Qinglongshan metamorphosed granites. Labeled points on concordia curve give age in millions of years.

of older hydrothermal systems and thus acquire an oxygen isotope composition averaged over a range of previous hydrothermal events (Bacon et al., 1989). Rift zone tectonics as postulated for South China's continental margin during the late Proterozoic are the most favorable environment for generation of low $\delta^{18}O$ magmas (Taylor, 1977).

The zircons most depleted in $\delta^{18}O$ analyzed to date are from low $\delta^{18}O$ rhyolites erupted after the collapse of the Yellowstone caldera (Bindeman and Valley, 2000). Measured values on small-size zircons are as low as −0.4‰, but rim compositions calculated from mass balance are −2.5‰. The zircons acquired their low $^{18}O/^{16}O$ rims when a catastrophic episode of caldera collapse suddenly dropped hydrothermally altered wall rocks into a hot magma. The altered rhyolites were remelted and erupted as low $\delta^{18}O$ lavas, but zircons successfully resisted not only the earlier alteration, but also the remelting event, and they preserved core compositions of +4‰ (Bindeman and Valley, 2000). We cannot reconstruct the magmatic history of the Qinglongshan hydrothermal system in such detail. But Yellowstone offers an example of how continental rift tectonics can give rise to felsic magmas and zircons with low $\delta^{18}O$.

8. FOSSIL HYDROTHERMAL SYSTEMS AS PALEOCLIMATE PROXIES

Depletions in $\delta^{18}O$ and δD in meteoric water are controlled by a number of factors in the hydrosphere–atmosphere cycle. Temperature-dependent fractionation during evaporation and condensation of water is the principal physical mechanism determining isotopic compositions. The observed first-order controls on the isotopic composition of surface waters include latitude, altitude, distance from the littoral, and, in the case of single storms, the cumulative amounts of water that have precipitated along the storm's track (Criss, 1999). Because ancient hydrothermal systems preserve the $\delta^{18}O$ and δD values of ambient meteoric waters, their use as paleoclimate proxies has been emphasized by a number of researchers (Blattner and Williams, 1991; Nevle et al. 1994; Brandriss et al. 1995). But to their capacity to record meteoric waters must be added the important advantage that the magmatic components of such systems may be accurately dated with texture sensitive geochronologic methods. Perhaps the ultimate achievement in hydrothermal paleoclimatology to date has been the mapping of Mesozoic polar climates in South Island, New Zealand, and in

West Antarctica. At Mt. Hartkopf, on the Ruppert Coast, Antarctica, a Cretaceous quartz monzodiorite dated at 112 Ma has quartz with $-6.3‰$ $\delta^{18}O$. Basaltic fragments in a volcanic breccia from Bailey Nunatak are a world record, at $-16.2‰$ $\delta^{18}O$ (Blattner et al., 1997; cf. Gregory et al., 1989).

Applying the concept of paleoclimate proxies, it is noted that Qinglongshan has phengites from garnet–omphacite–phengite–rutile eclogites with δD as low as $-127‰$ (Rumble and Yui, 1998). Assuming this value as an approximate measure of late Proterozoic meteoric water, the corresponding value for $\delta^{18}O$ in meteoric water is $-16‰$ (Craig, 1961). Under conditions of present climate and topography in eastern China, these values correspond to a latitude greater than 50°N, far to the North of Qinglongshan's current 35°N (Sheppard, 1986; Mizota and Kusakabe, 1994). The foregoing estimate of paleolatitude is beset with untested assumptions, however. The impact of UHP metamorphism on D/H ratios in hydrous minerals is unknown. Such factors as altitude vs. latitude and distance from the littoral are unknown in detail for the Neoproterozoic of eastern China. Given that the isotope anomaly is widespread in outcrops over a length scale of 100 km, the hypothesis of a high-altitude, isolated mountain peak can be eliminated. It may also be argued that the passive margin sedimentary succession of the South China block is evidence for continental-scale, subdued, sea-level topography; thus, a high-altitude Tibetan plateau environment is ruled out. Perhaps the most prudent conclusion to draw is that the Qinglongshan oxygen and hydrogen isotope anomaly gives unambiguous evidence of an unusual climate but that its specific cause cannot be uniquely determined.

9. CORRELATION WITH STRATA OF SOUTH CHINA BLOCK

Sinian carbonate, clastic, and glacial sediments of the Yangtze platform (Fig. 1) lie nonconformably on the 819 Ma Huangling batholith and extend with some disconformities to the base of the Cambrian (540 Ma). There are two glacial horizons, the Nantuo and the Changan tillites (Wang et al., 1981). The Nantuo is younger and more extensive, outcropping discontinuously for more than 1500 km across the Yangtze platform. Analysis of zircons from volcanic tuffs immediately underlying the Nantuo gives U-Pb ages of 736 and 748 Ma, in the same range as U-Pb ages of Qinglongshan protoliths (Ma et al., 1980, 1984; Gan et al., 1993). The U-Pb ages on volcanic tuffs suggest that Nantuo tillites may be correlative with Sturtian glaciation, a glacial episode that has been proposed as a worldwide event (Hoffman et al., 1998). Despite disagreements concerning the precise correlation of Neoproterozoic glacial events (Knoll, 2000), it is clear that protoliths of Qinglongshan's orogenic belt overlap the age range of Sinian sediments. We correlate the Qinglongshan hydrothermal system with Nantuo glaciation, within the uncertainties of age determinations.

10. PALEOMAGNETIC PALEOLATITUDE OF SINIAN SEDIMENTS

The correlation of Qinglongshan protoliths with Sinian sediments has interesting implications for estimating paleolatitude. Recent paleomagnetic investigations of three outcrop areas of Sinian sediments on the Yangtze platform by different laboratories give paleolatitudes of 37°, 34°, and 38° (Zhang and Piper, 1997; Evans et al., 2000). Measured samples were collected from strata both underlying and overlying the Nantuo tillites and thus demonstrate a stable paleogeography throughout the glacial epoch. Assuming the 5° present-day north–south latitude separation between Qinglongshan and the site of the paleomagnetic measurements to be equal to the maximum Neoproterozoic separation, the active hydrothermal system may have lain as close to the equator as 29° or as distant as 43°. By comparing the paleomagnetic to the isotopic estimate of paleolatitude, it can be seen that Qinglongshan is depleted in $^{18}O/^{16}O$ and D/H, relative to present-day climate.

11. NEOPROTEROZOIC ICEHOUSE

Recognition of a worldwide association of glacial deposits with marine limestones on Neoproterozoic platforms combined with isotopic evidence of synchronicity has led to an hypothesis of global glaciation just before the Cambrian (Hoffman et al., 1998). Deposits typical of a cold climate from high latitude or high elevation are intimately interbedded with flat-lying limestones and dolostones, indicating deposition at or below sea level in a warm, low-latitude climate. The Nantuo tillites interstratified with Doushantuo dolomites on the Yangtze Platform exemplify this association. Such an association of seemingly diametrically opposed deposits is rare in the ancient geologic record and absent from modern Earth. The new U-Pb ages measured for Qinglongshan metagranites are within the same age range as that of the Sturtian glacial episode. The correlation may be viewed as providing an explanation for the low $\delta^{18}O$ and δD values—that is, that they reflect a Neoproterozoic climate radically altered in relation to present-day climate. But Qinglongshan may also be viewed in a different light: If it is accepted that the low $\delta^{18}O$ and δD values record cold climate and if the paleomagnetic estimates of a temperate paleolatitude are correct then there is evidence in hand of an unusually cold climate, consistent with if not validating the global icehouse hypothesis. Given the uncertainties and unverified assumptions discussed above, however, the evidence must be regarded skeptically. Suppose, however, that low ^{18}O and δD hydrothermal systems were found to be prevalent in the Neoproterozoic. The existence in China of a cold-climate hydrothermal system preserved against all odds despite a history of continental collision, subduction, and coesite–eclogite metamorphism suggests that there may be more such systems as yet unrecognized. Just such an example is known from the Seychelles Islands, where 748 to 755 Ma (Tucker et al., 2001) granitic rocks and their wall rocks have $\delta^{18}O$ as low as -0.3 to $+3.3‰$ (quartz) and δD as low as $-109‰$ (Taylor, 1977).

Our discovery of low $\delta^{18}O$ zircons recording a surprisingly cold climate lying at a temperate paleolatitude and dating from the same time period as that proposed for low-latitude, sea-level glaciation provides a provocative new fact to consider in the ongoing debate concerning the "snowball Earth." It is certain that one such fact neither validates nor negates the icehouse hypothesis; nevertheless, our results are consistent with it. A global study of igneous intrusions of Neoproterozoic age holds some hope that other fossil hydrothermal systems would be found. Such systems would stand as paleoclimate proxies for which U-Pb ages could be measured. The existence of paleoclimate proxies in regions remote from glacial deposits raises the possibility of reconstructing Neoproterozoic climate over a broader area and at the same time achieving a more quantitative measure of the synchronicity of known glacial deposits.

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