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Evidence for cavity-dwelling microbial life in 3.22 Ga tidal deposits

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Geology, 2015

SAMPLE LOCALITY

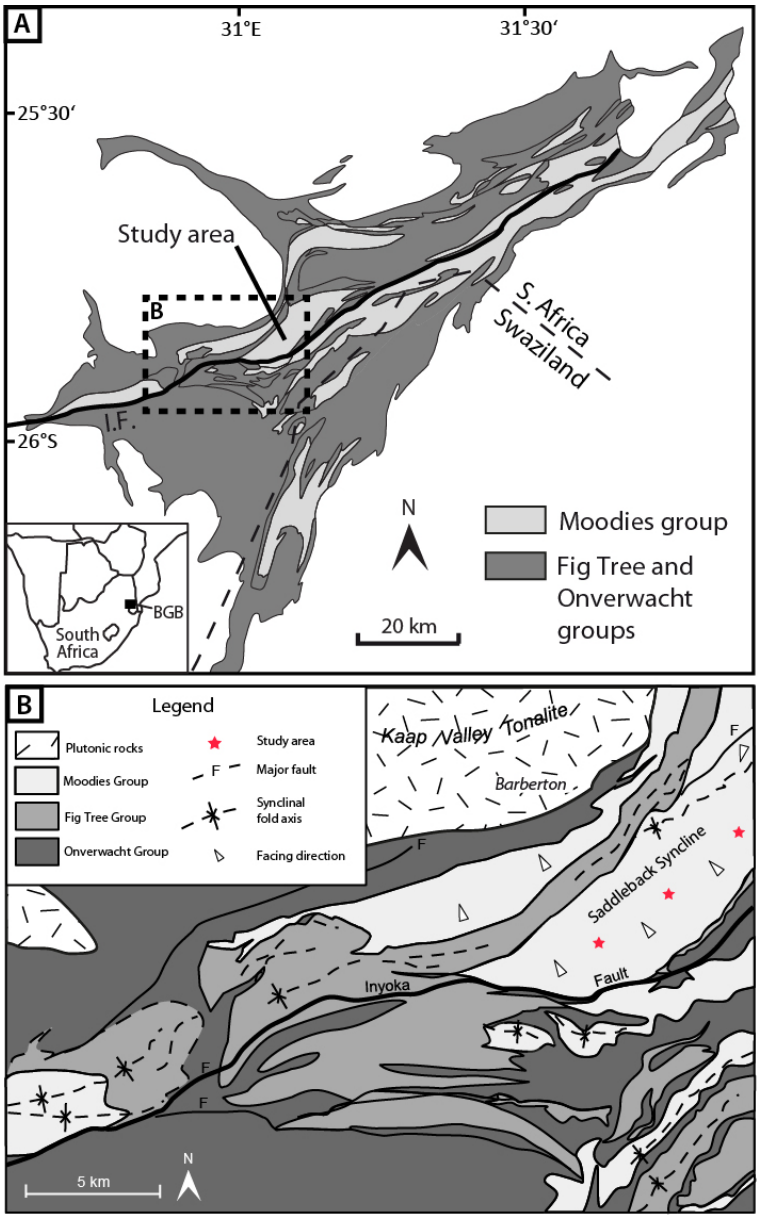

Figure DR1. Maps of the Barberton Greenstone Belt (BGB) in northeastern South Africa. A: Simplified geological map of the BGB showing the study area north of the Inyoka Fault (I.F). B: Detail of inset in A depicting the main study area in the Saddleback Syncline (modified after Heubeck et al., 2013).

BULK CARBON ANALYSIS

For bulk organic carbon analysis of the microbial mats, kerogenous material was manually separated from fresh and unweathered sandstone samples and then ground to a fine powder using an agate mill. Stable isotope analysis of organic carbon was performed using a THERMO Finnigan MAT V isotope ratio mass spectrometer, coupled with a THERMO Flash EA 1112 elemental analyzer via a THERMO Finnigan Conflo III-interface at the Museum für Naturkunde, Berlin, Germany. Approximately 100-400 µg of sample powder was put into a 10 ml exetainer. After sealing the exertainer with a septum cap the remaining air was removed by flushing with helium for five minutes at a flow of 100 ml/min. After, 30 µl of anhydrous phosphoric acid were injected through the septum into the sealed exetainer and reacted for ~1.5 h. Reference gas was CO_2 from a cylinder calibrated against the PeeDee Belemnite (PDB) standard. Repeated measurements of carbon isotope ratios ($\delta^{13}C$) relative to PDB standard had a precision better than 0.15‰.

For bulk carbon and oxygen isotope analysis of the carbonates the carbonate remnants were microdrilled from polished rock chips at the University of Potsdam and analyzed using a GasBench II linked to a MAT-253 ThermoFisher ScientificTM isotope ratio mass spectrometer at the Freie Universität Berlin, Germany. The external error of the measurements is ±0.06 % for $\delta^{18}O_{PDB}$ and ±0.04 % for $\delta^{13}C_{PDB}$; based on the reproducibility of the in-house reference material (Laaser marble).

SIMS ANALYTICAL CONDITIONS

The $^{13}C/^{12}C$ ratios were measured using a Cameca IMS 1280HR at the Institute of Earth Sciences of the University of Lausanne (Switzerland). We used a 10 kV Cs^+ primary beam, a ~0.6 nA current, resulting in a ~10 µm rastered beam size. The electron flood gun, with normal incidence, was used to compensate charges. ^{13}C and ^{12}C secondary ions, accelerated at 10 kV, were collected on multi-collection mode using a faraday cup (^{12}C) and an electron multiplier (^{13}C). A mass resolution of ~6000 MRP was obtained, to well resolve interferences on ^{13}C. The Faraday cup was calibrated at the beginning of the session. Each analysis took ~7 minutes, including pre-sputtering (60 seconds) and automated centering of secondary ions. This setting allowed an average reproducibility of ±1.6‰ (2 SD external precision) on an in house charcoal standard for a session, and internal error for each analysis of ~0.3‰ (2 SE internal precision). The standard was directly mounted on the sample holder. It cannot be excluded, that part of the variability of the measurements on the sample are partially due to polishing relief. Nevertheless, the measured values correspond excellently with bulk measurements, which show a similar range in composition.

TABLE DR1. DATA FROM CAMECA IMS 1280-HR ANALYSIS

Session	Sample	$\delta^{13}C$[a] (‰)	± 2 SD[b] (‰)	Raw $\delta^{13}C$[c] (‰)	± 2 SE[d] (‰)
1	Standard	-26.54	1.59	-92.64	0.29
1	Standard	-25.32	1.59	-92.64	0.15
1	Standard	-26.83	1.59	-91.55	0.16
1	**13-441_A1**	**-23.72**	**1.59**	**-93.16**	**0.58**
1	**13-441_A1**	**-26.18**	**1.59**	**-90.17**	**1.32**
1	**13-441_A1**	**-29.64**	**1.59**	**-92.76**	**1.20**
1	**13-441_A2**	**-30.82**	**1.59**	**-96.34**	**1.45**
1	**13-441_A2**	**-21.32**	**1.59**	**-97.65**	**1.93**
1	**13-441_A2**	**-24.60**	**1.59**	**-88.25**	**1.33**
1	Standard	-27.89	1.59	-91.66	0.14
1	Standard	-25.76	1.59	-95.08	0.14
1	Standard	-25.85	1.59	-93.07	0.15
1	Standard	-26.42	1.59	-93.28	0.16
1	Standard	-26.02	1.59	-93.95	0.14
2	Standard	-25.87	1.32	-82.01	0.14
2	Standard	-26.14	1.32	-82.64	0.14
2	Standard	-26.46	1.32	-84.08	0.13
2	Standard	-26.69	1.32	-84.66	0.13
2	Standard	-26.03	1.32	-84.36	0.13
2	Standard	-26.93	1.32	-85.62	0.13
2	**13-441_A1**	**-22.28**	**1.32**	**-81.27**	**2.93**
2	**13-441_A1**	**-26.04**	**1.32**	**-85.39**	**3.31**
2	**13-441_A1**	**-24.25**	**1.32**	**-83.95**	**1.81**
2	**13-441_A2**	**-27.81**	**1.32**	**-87.87**	**1.61**
2	**13-441_A2**	**-32.30**	**1.32**	**-92.71**	**2.03**
2	**13-441_A2**	**-29.39**	**1.32**	**-90.16**	**1.71**
2	Standard	-27.59	1.32	-88.71	0.15
2	Standard	-26.25	1.32	-87.68	0.18
2	Standard	-25.59	1.32	-87.38	0.14
2	Standard	-25.27	1.32	-87.42	0.15
2	Standard	-26.81	1.32	-89.31	0.14

[a] Carbon isotope composition of kerogen expressed in ‰ relative to the PDB standard corrected for instrumental mass fractionation and drift. Drift correction was based on the linear correlation between the isotopic value of the standard and the time of the measurement recorded by the machine.
[b] External precision (2 SD), defined as 2σ standard deviation of bracketing standard analysis.
[c] Raw carbon isotope composition of kerogen (i.e., non-corrected for instrumental mass fractionation) expressed in ‰ relative to the PDB standard.
[d] Internal precision (2 SE), defined as 2σ.

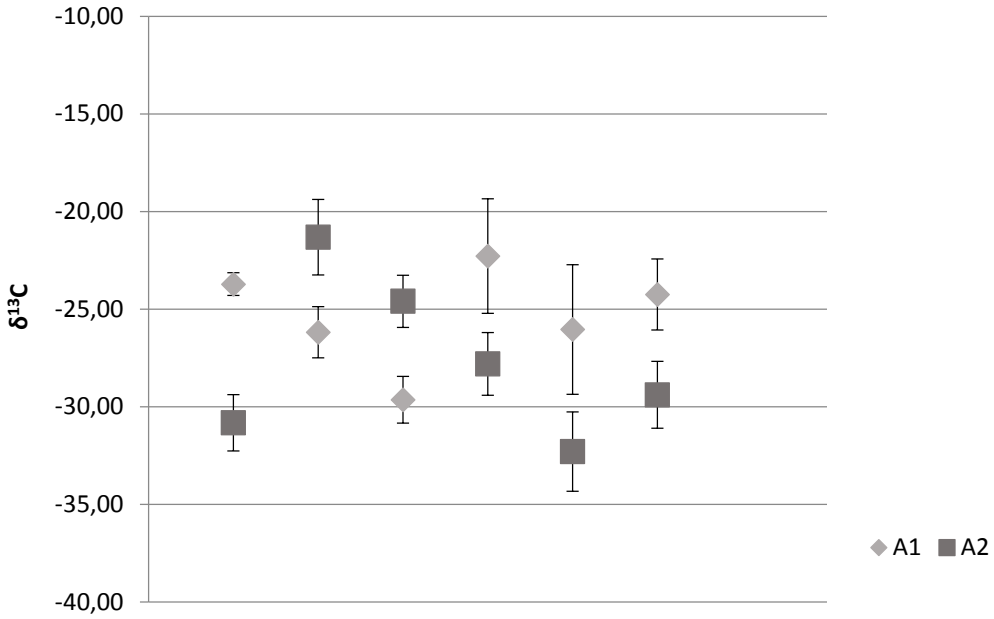

Figure DR2. All $\delta^{13}C_{PDB}$ SIMS values (n=12). Error bars show internal errors (2 SE).

RAMAN MICRO-SPECTROSCOPY

Raman intensity maps were acquired on 6 polished thin sections using a Thermo Scientific DXRxi Raman Imaging Microscope with 4 mW and an exposure time of 0.01 seconds at the Friedrich-Schiller-Universität Jena, Germany (14 scans). The maps show an increased kerogen concentration in the dark laminae (visible in petrographic thin sections), whereas secondary fractures that cross-cut the silicified cavities do not contain any traces of kerogen (Fig. DR3A and B). The Raman spectra indicate a greenschist facies metamorphic overprint (Fig. DR3C,Wacey, 2009), which is consistent with the peak metamorphic temperatures of the host rocks during the tectonic events 3.1-3.2 and 2.7 Ga ago and thereby supporting the syngenicity of the kerogenous microstructures (Heubeck and Lowe, 1994; Tice et al., 2004; Javaux et al., 2010).

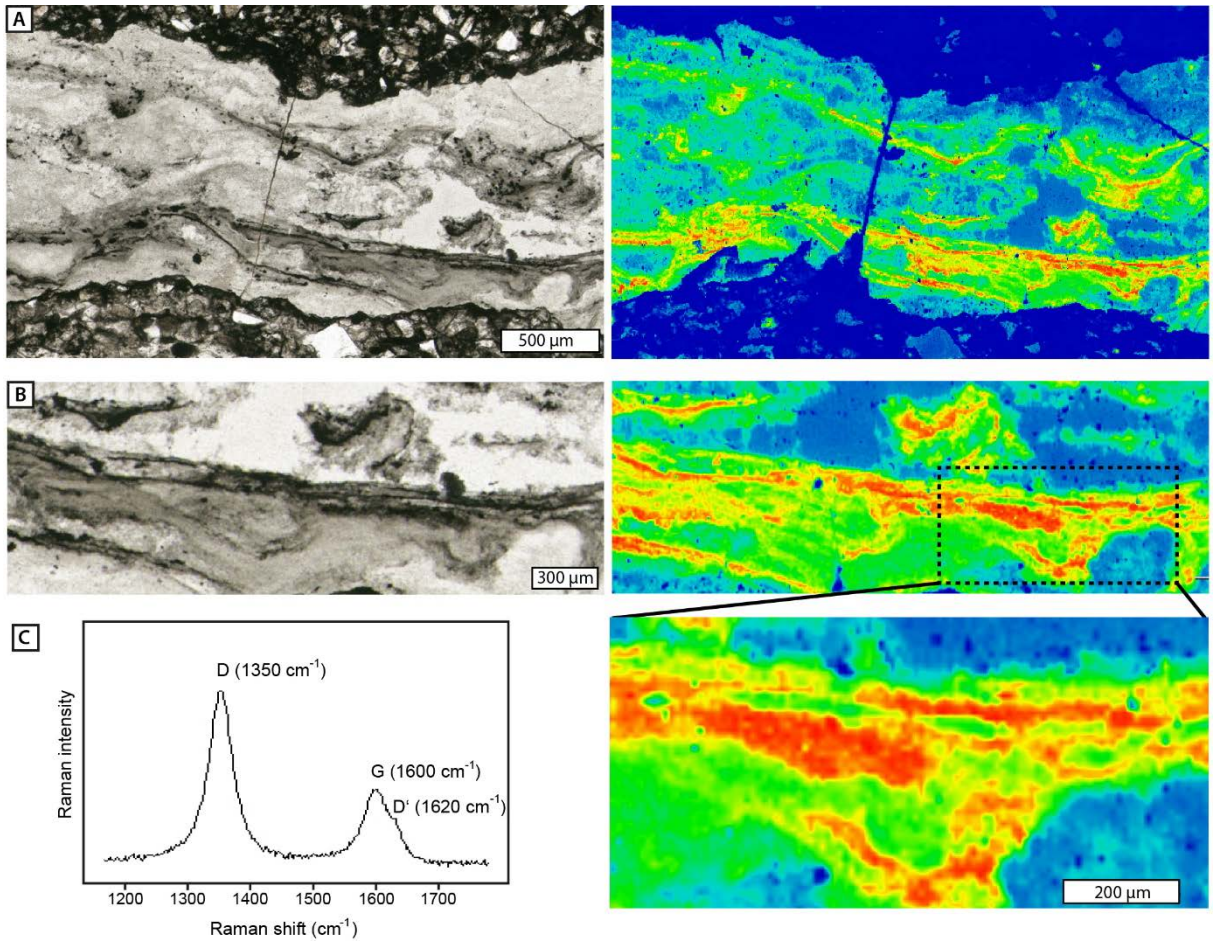

Figure DR3: Raman micro-spectroscopy. A, B: Thin section photomicrographs (left) and corresponding Raman intensity maps (right) of the silicified cavities showing the downward-oriented accretion and the kerogenous composition of the dark laminae. Red colors indicate kerogen-rich areas. C: Representative first-order Raman spectrum of the kerogen with the characteristic disordered peaks for amorphous carbon (D and D') and the graphite peak (G).

SEM ANALYSIS

For scanning electron microscopy (SEM), chert samples without weathered surfaces or major fractures were selected in order to avoid contamination following the guidelines by Westall and Folk (2003). Samples (n=10) were mechanically broken from thin section rock chips, cleaned in an ultrasonic bath and immediately dried and gold-coated. Sample analysis was performed at the Freie Universität Berlin using a ZEISS SUPRA 40 VP SEM operating with 20 kV acceleration voltage in secondary-electron (SE) or backscatter-electron (BSE) imaging mode.

SIZE DISTRIBUTION OF FILAMENTOUS MICROSTRUCTURES

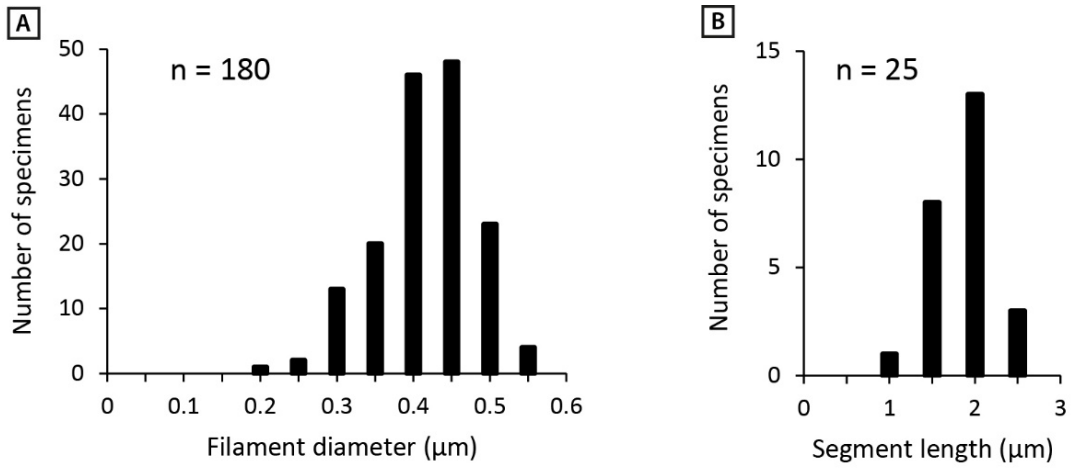

Figure DR4: Frequency distribution histograms for A: External filament diameters and B: Filament segment lengths of the preserved microfossils embedded within the cavity chert.

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