Carbon and Oxygen Isotope Analyses in Biocarbonates by SIMS

Foraminifera

3D image of polished otolith

Otolith

Nacre

5 µm

Confocal Laser Fluorescent Microscopy Image of a Nautilus shell cross section

Reinhard Kozdon
Ian J. Orland, Noriko T. Kita, John W. Valley

$\delta^{18}O$ and $\delta^{13}C$ in biocarbonates by SIMS

Carbonate samples analyzed at WiscSIMS:

- foraminiferal shells
- speleothems
- nautilus shells
- mollusk shells
- fish otoliths
- corals

Samples analyzed as WiscSIMS
Identification of diagenetic calcite in foraminiferal shells – reassessment of paleorecords

Identification of diagenetic calcite in foraminiferal shells – reassessment of paleorecords

Kozdon et al. 2011

Magnitude of the Carbon Isotope Excursion (CIE) in the marine record

Early Eocene paleogeography (~55 Ma ago)

Modified after R. Blakey (http://cgeosystems.com/globalx2.html)

ODP Site 865
CIE magnitude ~ 2.5%

ODP Site 690
CIE magnitude ~ 4.0%

Magnitude of the Carbon Isotope Excursion (CIE) in the marine record

Kozdon et al., in prep.


whole shells

δ^{13}C measurements by SIMS in alteration-resistant domains

modified after McInerney and Wing, 2011

Kozdon et al., in prep.
Intrashell $\delta^{18}$O variability in foraminiferal shells

Kozdon et al., 2009

Measurements of **daily growth increments** in foraminiferal shells

Vetter et al. 2013
Wild-caught Nautilus macromphalus from New Caledonia
Nautilus shell

confocal laser fluorescence microscopy

Linzmeier et al., in prep.

Nautilus shell

One half centimeter (~20 days of growth) of Nautilus shell

Linzmeier et al., in prep.

Otoliths

Derek Hogan, UW-Madison

Otoliths (bluegill)

Weidel et al. 2007
Corals

S. erubescens

F. Andrus, R. Cobb. University of Alabama

Secondary carbonate precipitation in pore spaces
Mount preparation
Epoxy mount for ion microprobe analysis of foraminiferal shells

Double stick tape
• at least 1 inch width

Washer
• outer Ø 25.4 mm (1 inch)
• inner Ø 10 mm

Aluminum plate

Double stick tape

Transfer the shape of the washer with a marker to the plate

Place samples within the inner circle (10 mm diameter)
Example: Mounting of foraminiferal shells. Specimens are placed with the preferred orientation on the double stick tape (inner 10 mm of the washer marking). UWC-3 calcite standard (2 or 3 grains) are centered
Mold (Teflon)
• inner Ø 25.4 mm (1 inch)

Casting
approx 6 cm³ epoxy

After grinding/polishing, the epoxy plug is cut to a thickness of less than 5 mm

Sealant

Sample casting
Zircon grain mount – Evaluation of the polishing relief

~20 micron relief

ZYGO white light profilometer

Oxygen isotope ratio

KIM-5

Zircon Standard

~20 micron relief

Zircon Standard (KIM5) Relief Test

Oxygen isotope ratio reproducibility (%)

Kita et al. 2009

Average Relief (μm)

Test-1

Test-2

Test-3

No relief

Example: Evaluation of the polishing relief of an otolith

Surface Map

Reflected Light Image

Surface Profile

3D-Model

Preselection of suitable domains for SIMS
Example: Foraminiferal shells

- imaging of **uncoated** samples by SEM in environmental mode using the backscattered electron detector (BSE) has shown to be a useful approach to locate growth bands and cavities that are filled by epoxy and/or organic material.
- some of this features may not be clearly visible after coating. **Only non-porous areas can be safely analyzed with high precision and accuracy.**

Chamber wall of *N. pachyderma* (North Atlantic core top). BSE detector, sample mount uncoated.

Approx. size of a SIMS analysis pit for δ¹⁸O

Analysis pits must be placed in nonporous domains. Data from pits overlapping epoxy or organic material may be compromised.

Example of a traverse for δ¹⁸O with pit placements in suitable domains

Example: Coral
Example: Coral

This domain cannot be analyzed (porosity)
Example: Planning of $\delta^{13}C$ measurements in a foraminiferal shell. The required spot size for $\delta^{13}C$ averages several growth bands.
Example: Cultured Foraminifera

Planned traverses for δ¹³C measurements

Vetter et al., in prep
The SIMS beam spot is typically slightly elongated in x-direction. Thus, traverses with high spatial resolution should be analyzed in y-direction (pits should not overlap!).
Example: Planning of $\delta^{18}O$ measurements in mollusk nacre

Tahitian black pearl oyster – cross section

VLM 5x, Polarized light $\theta=105^\circ$

Olson et al. 2012

UWC-3 STD
SIMS Analysis
Generation of secondary ions by sputtering

- Single atoms and clusters are ejected (collision cascade)
- A small fraction is ionized.
The data for both $^{16}$O and $^{18}$O in a single spot are subdivided into a series of 20 cycles (for typical 10 µm-spot analytical conditions), and the internal precision is based on the SE of the 20 comparisons. The $^{18}$O/$^{16}$O ratio in carbonate can vary significantly with depth during a single spot analysis, leading to a high internal error. However, this depth effect is reproducible from spot to spot and it is common to obtain external precision that is significantly better than would be predicted from the internal precision.
### SIMS Data Table

<table>
<thead>
<tr>
<th>File</th>
<th>Comment</th>
<th>d18O[SMOW]</th>
<th>IMF</th>
<th>d18O_m</th>
<th>d18O-2SE</th>
<th>16O(E9 cps)</th>
<th>IP(nA)</th>
<th>Yield (E6cps/nA)</th>
<th>Yield (% of bracketing STD)</th>
<th>date</th>
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<td>UWC-3, Cs = 154</td>
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Bracket: average and 2 SD

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Bracket: average and 2 SD

Average $\delta^{18}$O[measured] of the eight bracketing STD analyses ± 2SD of the bracketing STD
Spot-to-spot precision of ±0.3‰ (2SD) in $\delta^{18}$O with 10 µm beam-spot

$^{16}$O$^-$ [cps] = $\sim 2 \times 10^9$

$^{18}$O$^-$ [cps] = $\sim 4 \times 10^6$  (FC-amplifier background: 1000 cps)
A consistent primary beam intensity is essential for carbonate analyses.
Data Processing
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<td></td>
<td></td>
<td>1/25/20</td>
</tr>
<tr>
<td><a href="mailto:20130125@90.asc">20130125@90.asc</a></td>
<td>UWC-3</td>
<td>2.976</td>
<td>0.415</td>
<td>2.149</td>
<td>0.773</td>
<td>2.780</td>
<td></td>
<td></td>
<td></td>
<td>1/25/20</td>
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</table>

- **High internal 2SE (internal precision):** pit crosscutting epoxy and/or secondary phase
- **Low ¹⁶O count rate, low yield:** sample porosity, pit crosscutting epoxy
Data plotting outside the red dashed lines (yield <97% or >103% of the yield of the bracketing standards) were excluded. These pits likely contain a higher percentage of organics or are irregular (sample porosity, cavities, cracks, secondary phases).

Data from pits with a yield of <97% or >103% of the yield in the bracketing standards were excluded. The remaining data show no correlation between yield and measured $\delta^{18}$O. The $\delta^{18}$O values are corrected for instrumental drift, but not converted to the SMOW or PDB scale.
SEM Imaging of ALL Analysis Pits
Examples of ‘regular’ and ‘irregular’ ion microprobe pits in zircon following δ¹⁸O analysis

(a) ‘Regular’ pit, showing slight asymmetry due to inclination of primary beam

(b) ‘Irregular’ pit with through-going cracks, visible in the crater walls and floor

(c) ‘Irregular’ pit with a circular ‘cavity’ at the left side (defined by arrows). The analysis hit a mineral inclusion. Preferential sputtering of the inclusion is thought to have caused this feature. Pits are approximately 2-3 µm in depth.

It is tempting to accept data from ‘irregular’ pits. Such features often have no measurable affect on isotope ratio, however non-systematic and sometimes large shifts in measured δ¹⁸O (up to +12‰ reported by Cavosie et al., 2005) demonstrate the importance to describe and evaluate ‘irregular’ pits.
Evaluation of SIMS analysis pits by SEM: Otolith
Evaluation of SIMS analysis pits by SEM: UWC-3 carbonate STD

cavities and/or inclusions data from these pits should not be used
Evaluation of SIMS analysis pits by SEM: Nautilus shell (prismatic nacre)

Regular and irregular pits in the prismatic aragonite shell

Linzmeier et al., in prep.
Potential Impact of Organics
High $^{16}\text{OH}/^{16}\text{O}$ ratios indicate the presence of H, presumably organic material, in the analysis pit. The yield in the nonporous standard (UWC-3, coarse crystalline calcite) is about 10% higher than in the porous nautilus shell. Highest yields in the nautilus shell are observed in domains with a high $^{16}\text{OH}/^{16}\text{O}$ signal (or a high proportion of organics, respectively).
Biocarbonates containing significant amount of organics (e.g. otoliths, bivalves, mollusks) should be roasted or chemically treated to remove organics. However, this may cause disintegration of the sample, therefore, “practice”- samples should be used to evaluate the best procedure.

Point logger: Targeting of domains
Sample view by the built-in CCD camera

Fragment of a foraminiferal shell
CCD-camera, blue light LED (λ=450 nm)
Point-Logger

- a high-magnification SEM image of the sample can be referenced to the sample stage
- domains can be targeted by selecting the point of interest on the image.
During analysis, the sample can be seen by an optical microscope/CCD-camera. This representative image demonstrates the aiming-process (primary beam hits at blue marker). Typically this system is used for sample navigation and aiming.

For some types of samples, the resolution of the optical microscope (left image) is insufficient for precise aiming. We can upload SEM images (field of view ~500 µm) and align them to the sample stage. Subsequently, targets can be selected using the more detailed/higher resolution SEM image.

Point Logger

Using SEM images for precise aiming

Vetter et al. 2013, GCA
doi: http://dx.doi.org/10.1016/j.gca.2012.12.046
Example: Overlapping traverses for \textit{in situ} $\delta^{18}$O in a foraminiferal chamber wall with 3 µm beam-spot sizes. The Point Logger was used for aiming.
Thank you!