Automated NanoSIMS measurements & presolar grains

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...wasting away in Austria

Ernst Zinner
…checking for roid ragers at daughter's swim meet
Automated NanoSIMS measurements & presolar grains

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Thanks to John Valley & Noriko Kita for the invitation!
How do we know they’re presolar?

Isotopes!

- A grain from a single star will likely have an isotopic composition noticeably different from this average.
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- A grain from a single star will likely have an isotopic composition noticeably different from this average.
Presolar grains...all grown up

10,000+ grains *individually* analyzed

Carbides, oxides, silicates, nitrides, etc.

IMS-f series, SHRIMP, 1280, NanoSIMS, etc.
Where do we go from here?

Better spatial resolution: \( \leq 10 \) nm

Search for rare grains
$\delta^{i}\text{Si}/^{28}\text{Si} = \frac{[i\text{Si}/^{28}\text{Si}]_{\text{measured}}}{[i\text{Si}/^{28}\text{Si}]_{\odot}} - 1 \times 1000$
Please visit: http://presolar.wustl.edu/~pgd/

>99% of all SiC, oxides, & silicates
SiC X grains: supernova dust!

\[
\begin{align*}
\delta^{29}\text{Si}/^{28}\text{Si} & \quad \delta^{30}\text{Si}/^{28}\text{Si} \\
\hline
-800 & -600 \\
-400 & -200 \\
0 & 0 \\
\end{align*}
\]
Example: SiC from Qingzhen meteorite

X grains: $\lesssim 0.25\%$

X2 grains: $\sim 1/4$ of all Qingzhen X grains

Abundance of X2 grains: 0.063\%
SiC X grains: supernova dust!

Previous X data
Qingzhen SiC
Search for rare grains

Automated, high-throughput measurements

Lots of instrument time, which is costly

Efficient grain definition & sorting algorithms
Direct Imaging

Defocus ~50-100 μm static primary beam over area to analyze

Send entire ion image through mass spectrometer

Use a CCD camera to image channel plate/fluorescent screen…or SCAPS

Raster Imaging

Raster small (~1-0.1 μm ) primary beam over an area

Synchronize secondary ions w/primary ion raster

Reconstruct original location of sputtered ions

Use electron multipliers
“...what one fool can do, another can.”
Automatic measurement technique

1) Sputter clean sample surface
2) Acquire ion images
3) Automatically define particles to measure
4) Make high mass resolution (HMR) measurements
5) Move sample stage and repeat
Integrated into Cameca software
SiC grains from Indarch meteorite

Grain size: 0.25-0.45 μm

As size goes ↓, number of Z grains goes ↑
One week of manual measurements

Indarch IH6 manual grain mode

\[ \delta^{29}\text{Si}/^{28}\text{Si (‰)} \]

- Mainstream
- A+B grains
- X grains
- Y grains
- Z grains
- X Si\textsubscript{3}N\textsubscript{4}

\[ \delta^{30}\text{Si}/^{28}\text{Si (‰)} \]
One week of automatic measurements

\[ \delta^{29}\text{Si}/^{28}\text{Si} \]

\[ \delta^{30}\text{Si}/^{28}\text{Si} \]
### Automatic vs Manual*

<table>
<thead>
<tr>
<th>Group</th>
<th>Number</th>
<th>Percentage</th>
<th>Criterion</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mainstream</td>
<td>670</td>
<td>437</td>
<td>¹²C/¹³C=10 - 100 &amp; δ²⁹Si ≈ 1.4 δ³⁰Si</td>
</tr>
<tr>
<td>AB</td>
<td>39</td>
<td>27</td>
<td>¹²C/¹³C&lt;10</td>
</tr>
<tr>
<td>X</td>
<td>9</td>
<td>8</td>
<td>¹²C/¹³C&gt;100</td>
</tr>
<tr>
<td>Y</td>
<td>40</td>
<td>35</td>
<td>δ²⁹Si or δ³⁰Si&lt;-100‰</td>
</tr>
<tr>
<td>Z</td>
<td>54</td>
<td>42</td>
<td>δ²⁹Si&lt;0 &amp; δ³⁰Si 25‰ from MS line</td>
</tr>
<tr>
<td>Unique</td>
<td>2</td>
<td>0</td>
<td>Don’t fit into well defined groups</td>
</tr>
<tr>
<td>Total</td>
<td>814</td>
<td>549</td>
<td>Instrument time: Roughly Equal (1 week)</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Man hours: <strong>Auto &lt;&lt; Manual</strong></td>
</tr>
</tbody>
</table>

* Zinner et al, GCA, 71, 4786-4813, 2007
Spinel grains from Murray meteorite

Spinel (MgAl$_2$O$_4$) rich fraction

Average grain size: 0.45 μm

As size goes ↓, number of presolar spinel goes ↑
Previous oxide/silicate data
Murray CG Zinner 2005
Murray CG auto

$^{17}$O/$^{16}$O

$^{18}$O/$^{16}$O

area-13_30_1_mg_9

OC2

WOWSA!
Individual vs Bulk

Δ^{17}O/^{16}O  Δ^{18}O/^{16}O

NanoSIMS SE  FE-SEM
Advantages

*No* operator fatigue

*Customizable*: integrated into instrument software

Can be used for other applications!
Conclusion

Absent new hardware, software is key

Automated, high-volume measurements required

User’s constant presence unnecessary

Fully integrated into Cameca instrument software
Drawbacks: Time consuming

<table>
<thead>
<tr>
<th>Activity</th>
<th>Time</th>
</tr>
</thead>
<tbody>
<tr>
<td>Presputter</td>
<td>2 min</td>
</tr>
<tr>
<td>Acquire image (400μm²)</td>
<td>5 min</td>
</tr>
<tr>
<td>+ HMR measurement</td>
<td>2 min  (x 10)</td>
</tr>
<tr>
<td><strong>Time per area</strong></td>
<td><strong>27 min</strong></td>
</tr>
<tr>
<td><strong>Number of areas</strong></td>
<td><strong>x 144 (225 x 225 μm)</strong></td>
</tr>
</tbody>
</table>

Total time: 2.7 days!