Reprocessing of Lithium Orthosilicate Pebbles

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Objective

Reprocessing of OSi pebbles by remelting

Remelting of pebbles with a decreased Li content to simulate chemical composition after $^6\text{Li}$ burn-up

Assumption for DEMO end of life burn-ups:
- 50 \% $^6\text{Li}$ enrichment will be used
- 15 \% $^6\text{Li}$ burn-up will occur

Changes in the chemical composition:
- $\text{SiO}_2$ surplus: 2.5 wt\% → 6.4 wt\%
- $\text{Li}_2\text{SiO}_3$: 10 mol\% → 24 mol\%
Fabrication Campaign Dec. 05/Jan. 06

3 batches of reference material

4 batches of ‘burn-up‘ material with a lower Li content

3 batches of remelted material (‘burn-up‘ material + LiOH)

1 batch ‘burn-up‘ material

Characterisation was carried out in the initial state and after conditioning at 970°C for 1 week ➔ Quality control of 14 samples
### Chemical Analysis

**Batch**

<table>
<thead>
<tr>
<th></th>
<th>reference</th>
<th>remelted</th>
<th>‘burn-up‘</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>SiO₂ surplus (a)</strong> / wt%</td>
<td>2.5</td>
<td>2.4</td>
<td>6.3</td>
</tr>
<tr>
<td><strong>initial:</strong> Li₆Si₂O₇ / mol%</td>
<td>11</td>
<td>10</td>
<td>31</td>
</tr>
<tr>
<td><strong>cond.:</strong> Li₂SiO₃ / mol%</td>
<td>10</td>
<td>9</td>
<td>23</td>
</tr>
<tr>
<td><strong>Impurities (b)</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>C / ppm</td>
<td>610 ... 650</td>
<td>510 ... 640</td>
<td>770</td>
</tr>
<tr>
<td>Al / ppm</td>
<td>17 ... 26</td>
<td>68, 37, 23</td>
<td>17</td>
</tr>
<tr>
<td>Co / ppm</td>
<td>&lt; 1</td>
<td>&lt; 1</td>
<td>&lt; 1</td>
</tr>
<tr>
<td>Pt / ppm</td>
<td>71 ... 83</td>
<td>93 ... 121</td>
<td>55</td>
</tr>
</tbody>
</table>

(a) calc. from analysis by Schott; (b) FZK-IMF I

60 ppm Al + 4 ppm Co

→ 50-100 years wait time for hands-on recycling

(Fischer & Tsige-Tamirat, J. Nucl. Mater. 2002)
Microstructure (surface)

- **Reference**: OSi 06/1-1...1-3
- **Remelted**: OSi 06/3-1...3-3
- **‘Burn-up’**: OSi 06/2-1

*For each category, there are images of particle sizes.*

- **Diameter** $d_{50} \approx 300 \, \mu m$
Microstructure (cross section)

Phase Diagram Li$_2$O – SiO$_2$

![Image of phase diagram and micrographs](image_URL)
Microstructure (surface) cond.

- **Reference**: OSi 06/1-1...1-3 c
- **Remelted**: OSi 06/3-1...3-3 c
- **‘Burn-up’**: OSi 06/2-1 c
Microstructure (cross section) **cond.**

**Reference**
- OSi 06/1-1...1-3 c

**Remelted**
- OSi 06/3-1...3-3 c

**Burn-up**
- OSi 06/2-1 c
### Physical Properties

<table>
<thead>
<tr>
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<th>‘burn-up‘</th>
</tr>
</thead>
<tbody>
<tr>
<td>OSi 06/1-1...1-3</td>
<td>cond.</td>
<td>cond.</td>
<td>cond.</td>
</tr>
<tr>
<td><strong>He-pycnometry</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>closed porosity / %</td>
<td>0.6</td>
<td>0.4 ... 0.6</td>
<td>0.5 ... 0.8</td>
</tr>
<tr>
<td>OSi 06/3-1...3-3</td>
<td>cond.</td>
<td></td>
<td></td>
</tr>
<tr>
<td>density / %</td>
<td>95 ... 96</td>
<td>95</td>
<td>94 ... 95</td>
</tr>
<tr>
<td>open porosity / %</td>
<td>3 ... 4</td>
<td>3</td>
<td>3 ... 4</td>
</tr>
<tr>
<td>Specific surface area / m²/g</td>
<td>0.2</td>
<td>0.1</td>
<td>0.1</td>
</tr>
<tr>
<td>Crush load / N</td>
<td>6 ... 7</td>
<td>5 ... 7</td>
<td>6 ... 8</td>
</tr>
</tbody>
</table>

**TD (OSi) = 2.40 g/cm³**

[2.42 g/cm³]
Conclusions

Reprocessing of OSi by remelting of chemically simulated ‘burn-up‘ pebbles

- no detectable differences in properties or microstructure compared to reference material
- variation of properties from batch to batch is larger than due to remelting

Pebbles with a higher surplus of SiO₂ (‘burn-up‘) exhibit
- a fine-grained microstructure
- a lower crush load in the initial state
- but this cannot be transferred to irradiated material!
Conclusions

Outlook

Reprocessing of irradiated OSi pebbles by remelting

No wet chemical reprocessing to recycle $^6$Li
But no removal of activated elements

Further decrease of impurities by using ultra pure raw materials
→ Reduction of wait-time for reprocessing

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