Diamantane particles in cryomilled nanocrystalline aluminium

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- Background diamantane
- Sample prep: Cryo milling, thermal exposure
- XRD, TEM: Grain growth
- APT: Diamantane (com-)position
- Influence of laser & voltage
- Conclusions

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Diamantane Molecules

- Member of the diamondoid family of hydrocarbons
- Diamond-cubic, hydrogen terminated carbon cages
- Differs from other particles normally added to Al
  - Much smaller ($<1$ nm vs. $\sim100$ nm)
  - Molecules weakly held together by H bonds that can break up during cryomilling
- Source: Chevron Molecular Diamond Technologies
Cryomilling

- Thermal stability limited -> cryo milling
- Powders milled with steel balls in liquid nitrogen
  - 8 hours at 180 rpm
  - 31:1 (ball:powder) weight ratio

- Diamantane added prior to milling
  - 1wt% is sufficient to cover the grain boundaries for a grain size of 20 nm

- Outgassing in Al canisters

- Hot Isostatic Press (HIP)
  - Pressure of 103 MPa at 693K or 793K
  - 2 hr
Thermal stability

- Commercially Pure Al (Zhou et al. 2001)
  - Above 748K, mean grain size: $d > 100$ nm
  - Contains oxides, nitrides, carbides
- CP Al + diamantane (Maung et al. 2011)
  - Substantially reduced grain growth

\[ T_m = 0.83T_m \]
Thermal stability

HIP: 103 MPa, 2h, 793 K

\[ d = 155 \text{ nm} \quad d = 75 \text{ nm} \]

Without Diamantane

0.5% Diamantane

APT: Overview of 4 material types (after HIP)

Al without addition

Al + 4 wt.%Mg

Al + 1 wt.% diamantane

Al + 4 wt.%Mg + 1 wt.% diamantane
A more detailed look at the positions

- AlN at GBs
- Diamatane at triple junction

Al + 1wt.% diamantane

30 nm
A more detailed look at the compositions

- AlN at GBs
- Diamatane at triple junction
- Oxide/Nitride around diamantane
- No excessive local magnification effect

**Proxigram - Interface 4**

<table>
<thead>
<tr>
<th>T=30K</th>
<th>Det. Rate</th>
<th>Al</th>
<th>C</th>
<th>H</th>
<th>N</th>
<th>O</th>
</tr>
</thead>
<tbody>
<tr>
<td>Laser: 50 pJ</td>
<td>0.2%</td>
<td>45</td>
<td>18</td>
<td>30</td>
<td>0.4</td>
<td>0.4</td>
</tr>
<tr>
<td></td>
<td>1%</td>
<td>60</td>
<td>16</td>
<td>22</td>
<td>0.5</td>
<td>1</td>
</tr>
<tr>
<td></td>
<td>2%</td>
<td>58</td>
<td>26</td>
<td>14</td>
<td>1</td>
<td>1.2</td>
</tr>
<tr>
<td>Voltage fraction 15 %</td>
<td>0.2%</td>
<td>34</td>
<td>20</td>
<td>38</td>
<td>4</td>
<td>2</td>
</tr>
</tbody>
</table>
Conclusions

- Diamantane in cryomilled NC Al and Mg alloys: Remarkable thermal stability.
- Consolidation of bulk NC alloys with $D < 100$ nm.
- Mg had less influence on grain growth mechanism in the presence of diamantane.

- Diamantane runs surprisingly well in APT
- Diamantane at grain boundaries and especially triple junctions
- Mg, AlN, AlO in separate clusters along the GBs
- Diamantane clusters contain Al,N,O
Thank you for your attention

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