Understanding the Local Structure-Property Relationships of Solders in Terrestrial vs. Microgravity Environments Using Electron Microscopy and Nano-mechanical Testing

Siddhartha (Sid) Pathak
Assistant Professor
Chemical and Materials Engineering
University of Nevada, Reno, NV 89557
https://wolfweb.unr.edu/homepage/spathak/
Solders in Terrestrial vs. Microgravity Environments

- In-Space Soldering Investigation (ISSI) experiments performed aboard the International Space Station (ISS) – 2003-2005

- The ISSI data has demonstrated that a lack of buoyancy forces in microgravity can internally trap the flux created during soldering at interfaces, such as repair joints.

- Hypothesis: such internal porosity can be detrimental to the desired strength of the joint, as well as its thermal and electrical conductivity.

- Results will be instrumental in enhancing our fundamental understanding of the effects of surface tension driven convection phenomena during solidification processing operations such as brazing, soldering, and welding.

- Furthermore, the microgravity experiments represent a lowest gravity boundary condition. As such, these results could also be useful in predicting solidification behavior on other lower gravity environments (e.g. moon or Mars).

Photograph of solder drop created in gravity hanging from a silver-coated strand of copper wire

Photograph of solder drop created on the ISS in microgravity with an equilibrium "football" shape.


Mechanical Testing Tools at the Micro-to-Nano length scales

- The features of interest (porosity, dendrites etc.) in the solders have very small length scales (µm to mm)
- This requires specialized nano-mechanical tools for testing and characterization.
Mechanical Testing Tools at the Micro-to-Nano length scales

- The features of interest (porosity, dendrites etc.) in the solders have very small length scales (μm to mm)
- This requires specialized nano-mechanical tools for testing and characterization.

- **Specimen/Test Preparation**
  - Simple: a) Tensile
  - Laborious: b) Compression, c) Bending, d) Indentation

- **Data Analysis**
  - SEM
  - EBSD

- **Terrestrial Gravity**
  - Pore in micro-gravity
Investigating local mechanical response at the micro- and nano-scales: *In-situ* SEM straining capabilities at UNR

<table>
<thead>
<tr>
<th>Load Cases</th>
<th>Samples</th>
<th>Properties</th>
</tr>
</thead>
<tbody>
<tr>
<td>Micro-Compression</td>
<td>Nanowires, particles MEMS/NEMS, milled</td>
<td>$E, \sigma_y, \sigma_f, n$</td>
</tr>
<tr>
<td></td>
<td>substrates, thin films, multilayers</td>
<td>$E, K_c$</td>
</tr>
<tr>
<td>Micro 3-pt</td>
<td>Nanowires, MEMS/NEMS, milled, machined</td>
<td>$E, \sigma_y, K_c$</td>
</tr>
<tr>
<td>bend</td>
<td>structures</td>
<td></td>
</tr>
<tr>
<td>Micro-Bending</td>
<td>Nanowires, MEMS/NEMS, milled, machined</td>
<td>$E, \sigma_y, \sigma_f, n, K_c$</td>
</tr>
<tr>
<td></td>
<td>structures</td>
<td></td>
</tr>
<tr>
<td>Micro-Tension</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

[Image of Micro-Compression, Micro 3-pt bend, Cantilever Micro-Bending, Micro-Tension]
Investigating local mechanical response at the micro- and nano-scales: *In-situ* SEM straining capabilities at UNR

**Load Cases**

- Micro-Compression
- Cantilever
- Micro-Bending
- Micro-Tension

**Samples**

- Nanowires, particles
- MEMS/NEMS, milled structures
- Substrates, thin films, multilayers

**Properties**

- $E$, $\sigma$, $\epsilon$, $n$
- $K_c$
- $\sigma_y$, $\sigma_f$, $h$

**Graphs**

- He on W
- Damage, dpa vs. Depth, nm

---

Idealized primary zone of indentation:

\[ \frac{3\pi}{4} a = 2.4a \]
UNR: Nano-mechanical Testing Facilities

Ex-Situ (in air) indenters

Nanoindenter® XP
LANL Laboratory Education Equipment Gift (LEEG) Program 2017

Hysitron TI 900 TriboIndenter

In-Situ indenters

Hysitron PI 85 SEM PicoIndenter

Alemnis Indenter system
DOE FY 2018 Scientific Infrastructure Support for Consolidated Innovative Nuclear Research

FEI Scios™
Dualbeam FIB/SEM
NSF MRI grant #1726897.
Ex-situ (in air) Indentation across interfaces

- Grain 1 (soft)
- Grain 2 (hard)

**Indentation Stress, GPa**

- **E_{eff} = 215 GPa**
- **E_{eff} = 203 GPa**
- **E_{eff} = 174 GPa**

**Indentation Strain**

- **Y_{ind}**

**Distance from boundary, µm**

- **13.5 µm indenter**

**Fe-3%Si steel**

**In-situ indentation - deformation of carbon nanotubes**

Before

At max load

After unload

Micro-pillar compression: Deformation of carbon nanotube pillars

Sequence of buckle propagation

As grown

~5% strain

First buckle

70% strain

90% strain

Last buckle

Pathak, Needleman, Hart, Greer. ACS Nano 2013. Local Relative Density Modulates Failure and Strength in Vertically Aligned Carbon Nanotubes
Micro 3-point bending
Crack propagation in nanolaminates

Al-TiN 50 nm - 150 nm

Al-TiN 5 nm - 5 nm
Correlating SEM frames to mechanical data shows stable crack growth under load

Al-TiN (150nm-50nm)

(load graphs and images)

Al-TiN (5nm-5nm)

(load graphs and images)
Micro-tensile testing: Cu/Nb multilayers
Compression testing of glass pillars at different strain rates

- Sample and tip heaters capable of 800°C (1000°C currently under development)
- Ability to perform cryo temperature tests at -150°C (under development)

Microcompression strain rate jump tests on nanocrystalline and single crystalline Ni

Acknowledgment: Alemnis and EMPA, Switzerland
Summary of capabilities

- Spherical Indentation Stress-Strain
- *In-situ* Indentation

- *In-situ* SEM compression
- *In-situ* SEM 3-point bending
- *In-situ* SEM micro-tensile

- *In-situ* high-strain rate testing at elevated temperatures