Significance of High-Temperature Vacuum Creep for Selected Refractory Alloys

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Discussion Statements

• NASA is routinely presented with projects and objectives that could benefit from the application of high-temperature, high-strength materials
• With many of these applications, creep strength is a governing material property
• Time to 1 percent creep strain is regarded as a design parameter that possesses a built-in factor of safety
• Significant amount of data in the literature, much of which was generated for the SP-100 program in the 70’s, 80’s and 90s
• Buckman definition for refractory metal:
  – $T_m > 2000^\circ C$
  – BCC
  – $M_{\text{oxide}} / M_{\text{metal}} < 1$
  – Therefore Nb, Ta, Mo, W
Recent NASA Opportunities for Refractory Alloy Application

- Advanced Stirling Engine Development
- Proposed Mission to Venus
NASA Vacuum Creep Test Capability
NASA Creep Test Frames

Ultra-High Vacuum

Vacuum Creep Rupture
<table>
<thead>
<tr>
<th><strong>Ultra-High Vacuum</strong></th>
<th><strong>Vacuum Creep Rupture</strong></th>
</tr>
</thead>
<tbody>
<tr>
<td>6 test frames</td>
<td>5 test frames</td>
</tr>
<tr>
<td>1650°C maximum temperature capability tungsten mesh heaters</td>
<td>1650°C maximum temperature capability tantalum heaters</td>
</tr>
<tr>
<td>10E-10 Torr vacuum capability</td>
<td>10E-7 Torr vacuum capability</td>
</tr>
<tr>
<td>Pan limit: 120 pounds</td>
<td>Video extensometry</td>
</tr>
<tr>
<td>Video extensometry</td>
<td>Computer program data acquisition</td>
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<tr>
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<td>Eurotherm temperature controllers</td>
</tr>
<tr>
<td>Eurotherm temperature controllers</td>
<td>Programmable logic controllers (PLC)</td>
</tr>
<tr>
<td>Exterior water cooled chamber</td>
<td>quality and safety</td>
</tr>
<tr>
<td>Ion pump (500 liters/sec capability)</td>
<td>automation (pumpdown)</td>
</tr>
<tr>
<td>electro pneumatic isolating gate valve</td>
<td>development of new logic schemes</td>
</tr>
<tr>
<td>Chamber bakeout</td>
<td>New mechanical pumps and turbopumps</td>
</tr>
<tr>
<td>custom fitted jacket</td>
<td>significant cleanliness improvement</td>
</tr>
<tr>
<td>Tantalum thermal radiation shields</td>
<td>eliminates backstreaming of oil</td>
</tr>
<tr>
<td>New temperature and vacuum controls</td>
<td>New temperature and vacuum controls</td>
</tr>
</tbody>
</table>
NASA-Developed Optical Extensometry
Creep Lab Video Extensometry for Vacuum Creep Testing

- Video Extensometer provides higher accuracy, automation, and better performance over the legacy cathetometer system.
- Hardware Cost = $3,000 per frame.

Creep Lab Video Extensometry for Vacuum Creep Testing

Method

• Image region of interest is searched using one of three Measurement options to find each fiduciaries’ centroid.
  (fit circle, centroid, manual pick)
• 4,200 pixel vertical resolution with sub-pixel interpolation
• Provides twice the accuracy over the legacy system
  • Resolution estimated at .06% strain on a 2 inch gauge
• Fully automated reducing human error
• Sampling rate = 1 image per 3 seconds
• New cameras can provide higher resolution and offer 24+ fps

Comparison of Types of Data Possible From Both Cathetometry and Optical Extensometry
Several Issues Important to Keep in Mind When Reviewing Creep Data

• The following topics/characteristics are known/reported to have real effects on material performance:
  – Where the material was made
  – Batch-to-batch variation
  – Processing method
  – Heat treatment
  – Average grain diameter
Stirling Engine Heater Head Development

Tantalum and Rhenium Alloy Candidates
Advanced Stirling Technology
Application of Tantalum and Rhenium as a Heater Head

Creep Resistance of Heater Head is a Major Requirement

- Creep stress exerted on heater head by pressure differential across thin wall section estimated by hoop stress equation for a cylinder
  - \( \sigma_{\text{hoop}} = \frac{\Delta P r}{t} \)
  - \( \Delta P \equiv \) pressure differential across heater head wall
  - \( r \equiv \) inner cylinder (heater head) radius
  - \( t \equiv \) heater head wall thickness
Stirling Heater Head Identified as Most Critical Component

Heater head must withstand high stresses to high temperatures in extreme environment

Stirling working space

Piston

Alternator

Heater head

Displacer

Heat exchangers

Piston, Alternator

Stirling working space

Heater head

Displacer

Heat exchangers
Tantalum Candidacy for Stirling Engine Heater Head

ASTAR 811C is a precipitation strengthened alloy (Ta-8W-1Re-0.7Hf-0.025C)

Extruded ASTAR 811C bar
Joseph Giglio
Bill Blankenship
Pittsburgh Materials Technology, Inc.

Microstructure of extruded ASTAR 811C transverse section 50X

NASA test specimen
Time to 1 Percent Creep Strain Data for Tantalum Alloys

![Graph showing time to 1 percent creep strain data for tantalum alloys.](image)

- **T111** (Buckman et. al.)
- **ASTAR 811C** (Buckman et. al.)
- **ASTAR 811C** (Klopp et. al.)
- **Ta10W** (Klopp et. al.)
- **ASTAR 811C** (Conway)
- **T111** (Conway)
- **ASTAR 811C** (NASA)

Notes:
Joe Giglio and Bill Blankenship, Pittsburgh Materials Technology, credit for NASA opportunity to investigate ASTAR 811C
Bob Titran, NASA, credit for mentoring in refractory metals/alloys
**ASTAR 811C** (Ta-8W-1Re-0.7Hf-0.025C)
**T111** (Ta-8W-2Hf)

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## Selected Information from NASA In-house Extruded ASTAR 811C Tests

<table>
<thead>
<tr>
<th>Temperature, C</th>
<th>Time to 1% strain, h</th>
<th>Stress, ksi</th>
<th>Steady-state creep rate, sec(^{-1})</th>
</tr>
</thead>
<tbody>
<tr>
<td>1000</td>
<td>169</td>
<td>38</td>
<td>1.6E-09</td>
</tr>
<tr>
<td>1100</td>
<td>877</td>
<td>32</td>
<td>3.1E-09</td>
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<tr>
<td>1227</td>
<td>660</td>
<td>20</td>
<td>2.8E-09</td>
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<tr>
<td>1350</td>
<td>805</td>
<td>12</td>
<td>2.8E-09</td>
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<tr>
<td>1450</td>
<td>1667</td>
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<tr>
<td>1550</td>
<td>1892</td>
<td>1</td>
<td>9.4E-10</td>
</tr>
</tbody>
</table>
CIP to NNS Rhenium for Stirling Engine Components

Rhenium Alloys, Inc.
Elyria, OH

INNOVATION
Cold Isostatic Pressing (CIP) of Rhenium Powder to a Near Net Shaped Pressure Vessel

ACCOMPLISHMENTS

- Expanded the technology to a CIP manufacturing method to produce near net shaped (NNS) rhenium and rhenium containing parts.
- The CIP to NNS process produced a rhenium part with a sintered density of greater than 98% of theoretical. After hot isostatic pressing without canning, the part obtained a density greater than 99%.
- The CIP to NNS process reduced the amount of rhenium powder used by 70%. This process could reduce the manufacturing time by 30% and the machining time by 50% for high-temperature Stirling engine application.

COMMERCIALIZATION

- The CIP to NNS method of manufacturing was used to produce a dome for a commercial customer.
- This method has increased the job equivalents by 2, which is directly associated with this SBIR.

GOVERNMENT/SCIENCE APPLICATIONS

- NASA requires rhenium for many space applications such as solar thermal propulsion and Stirling engine application.
- Various DoD agencies require lower cost production methods for several rhenium applications such as tactical missile components and other high-temperature or thermally cycled parts.

Components made from NNS process.
1. Kaiser Marquardt chamber, 2. TRW chamber, 3. Dome made for commercial customer

Source: Todd Leonhardt, Rhenium Alloys
Creep Rupture of Selected Refractory Alloys

Proposed Mission to Venus

Molybdenum Alloy Candidates
In-House Mo-Base Alloy Information from Creep Tests

- 10 ksi is a recurring stress of interest for several NASA application
- Steady state creep rate is useful for comparing performance under different parameters
- Carbide dispersion is more effective than oxide dispersion for creep resistance in these materials

<table>
<thead>
<tr>
<th>ID</th>
<th>Temperature, C</th>
<th>Stress, ksi</th>
<th>Time to 1% creep strain, h</th>
<th>LMP</th>
<th>Steady-state creep rate, sec⁻¹</th>
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<tbody>
<tr>
<td>MoODS-1</td>
<td>1200</td>
<td>10</td>
<td>4</td>
<td>30.35</td>
<td>3.60E-08</td>
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<tr>
<td>MoODS-3</td>
<td>1150</td>
<td>10</td>
<td>75</td>
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<td>MoODS-4</td>
<td>1100</td>
<td>10</td>
<td>75.7</td>
<td>30.04</td>
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<tr>
<td>HWM-1</td>
<td>1200</td>
<td>10</td>
<td>886</td>
<td>33.80</td>
<td>7.40E-10</td>
</tr>
</tbody>
</table>
Proposed Mission to Venus
Application of Molybdenum

- **High temperature**
  - 1000 °F (500 °C)

- **High pressure**
  - 1500 psig (100 bar)

- **Corrosive/Acidic**
  - CO₂ (~96.5%)
  - SO₂ (130 ppm)
  - HF (5 ppb)
  - HCl (0.5 ppm)
  - NO (5.5 ppb)
  - CO (15 ppm)
  - COS (27 ppm)
  - N₂ (~3.4%)
  - H₂O (30ppm)

Venus Atmospheric Conditions

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Proposed Venus Lander Design
Hot-Side Adapter Flange Improvement

• Hot-side adapter flange (HSAF) role is to shield the heater head from the GPHS. HSAF must have high thermal conductivity
  - Nickel-base superalloys have been historically chosen for HSAF application due to an attractive balance between strength and conductivity at high temperatures
  - A need for a higher strength, higher conductivity alloy for 1200 °C and above has been identified
  - Refractory alloys (e.g. molybdenum-base) are candidate materials

• Refractory alloys are highly prone to oxidation so a coating needs to be applied for protection
  - Silicide-base coatings are the state-of-the-art for refractory alloy protection
  - However, brittle silicide layers are formed

• ODS Mo and other materials could offer further improvements and increased efficiencies
  - Further protection against oxidation through crack paths could possibly be accomplished through a “Type A” sodium silicate (glass) top coat
Plansee SIBOR® Coating Offers Protection of Mo

Mo-TZM
(Mo-0.5 wt.%Ti-0.08 wt.%Zr-0.02 wt.%C)

- Sample exposed for 100 h at 1200 °C in 5 ppm oxygen-argon environment
- Mo sample did not catastrophically oxidize

Pure Mo

- Sample exposed for 10h intervals at 1200°C in 5 ppm O₂ argon environment
- Mass increases slightly
- Protection observed as well as little implication from cyclic oxidation after 4 cycles

- Steve McCrossan and Thom Coughlin, Plansee, credit for NASA opportunity to molybdenum alloys through SIBOR® environmental durability coating
SIBOR® Promotes Protection Through Silicide Layers

- Cross section of SIBOR® protected Mo-TZM
- Protection from catastrophic oxidation
- Cracks that form from expansion differences are self-healing
  - Maintaining protection from environment
- Excellent protection of molybdenum alloy substrate at 1200C for short times
Summary

• Refractory alloys can be applied to challenging applications that require high strengths to high temperatures
  – Excellent coatings have been developed to mitigate environmental durability issues
• Creep behavior is a key material property for many potential refractory alloy applications
• Creep performance/behavior is dependent on many factors that can influence the microstructure
• NASA GRC possesses state-of-the-art test equipment and data measurement/acquisition to assess material viability for space applications