Significance of High-Temperature Vacuum Creep for Selected Refractory Alloys

Frank Ritzert
NASA Glenn Research Center

Raymond W. Buckman Memorial Symposium on Refractory Metals and Alloys
Session: Refractory Metals Characterization and Applications
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Discussion Statements

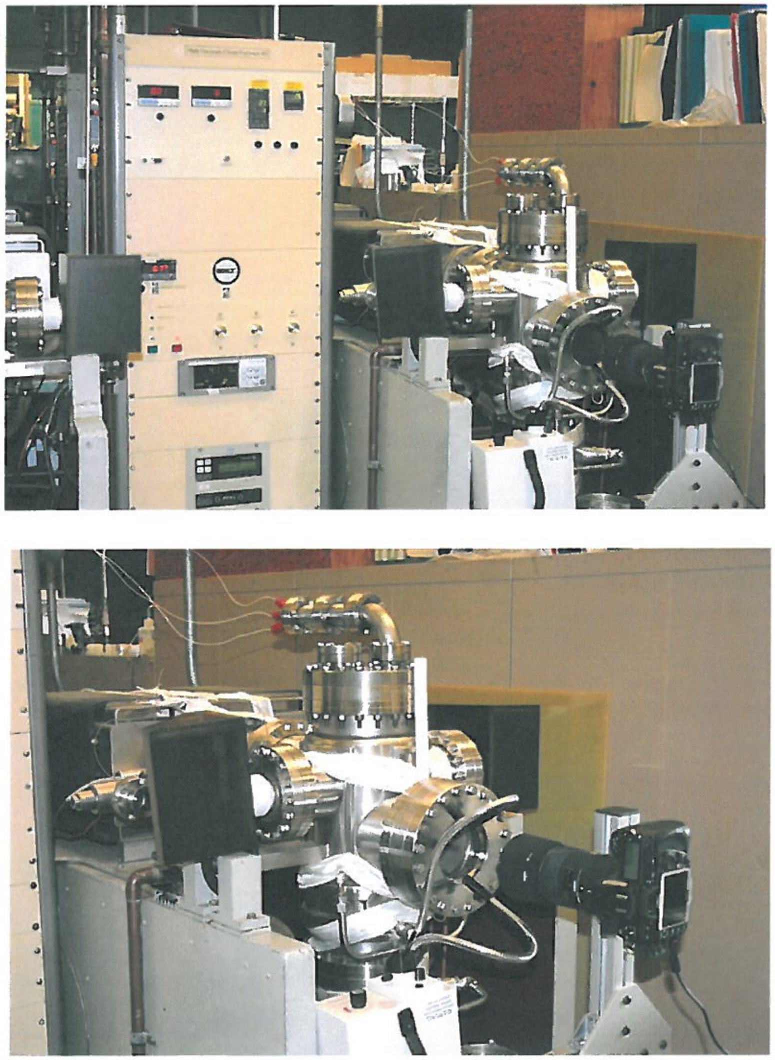
- NASA is routinely presented with projects and objectives that could benefit from the application of high-temperature, high-strength materials
- With many of these applications, creep strength is a governing material property
- Time to 1 percent creep strain is regarded as a design parameter that possesses 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>2000C
  - BCC
  - $MP_{oxide}/MP_{metal} < 1$
  - Therefore Nb, Ta, Mo, W

Recent NASA Opportunities for Refractory Alloy Application

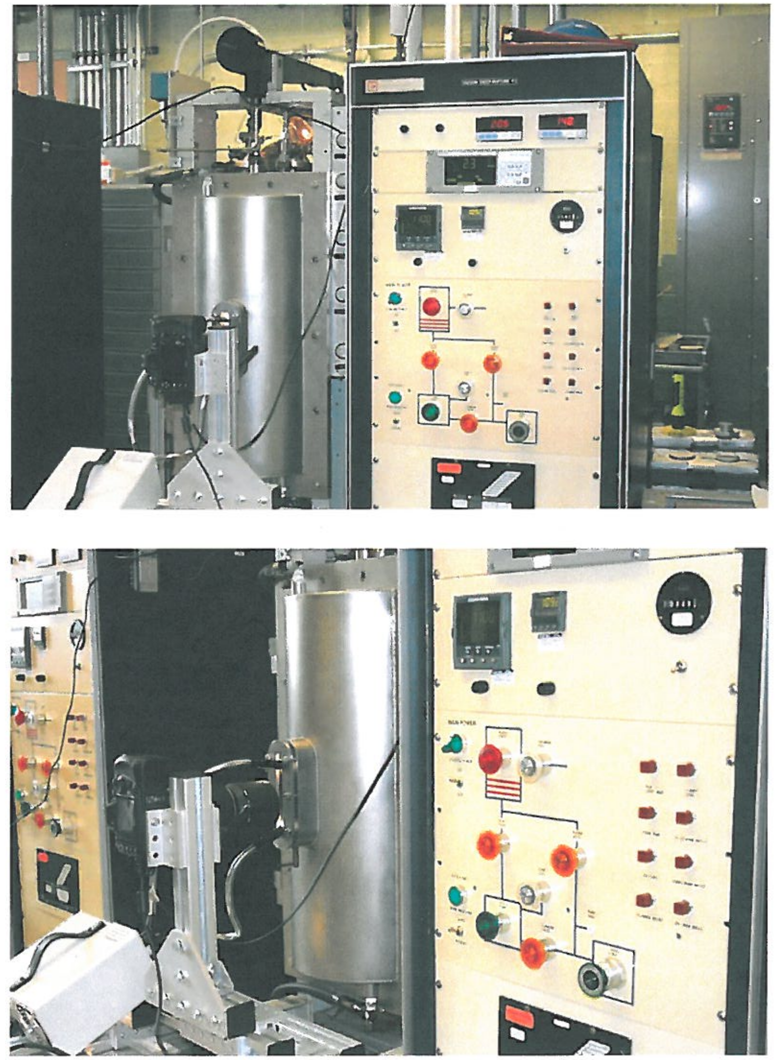
- Advanced Stirling Engine Development
- Proposed Mission to Venus

NASA Vacuum Creep Test Capability

NASA Creep Test Frames



Ultra-High Vacuum



Vacuum Creep Rupture

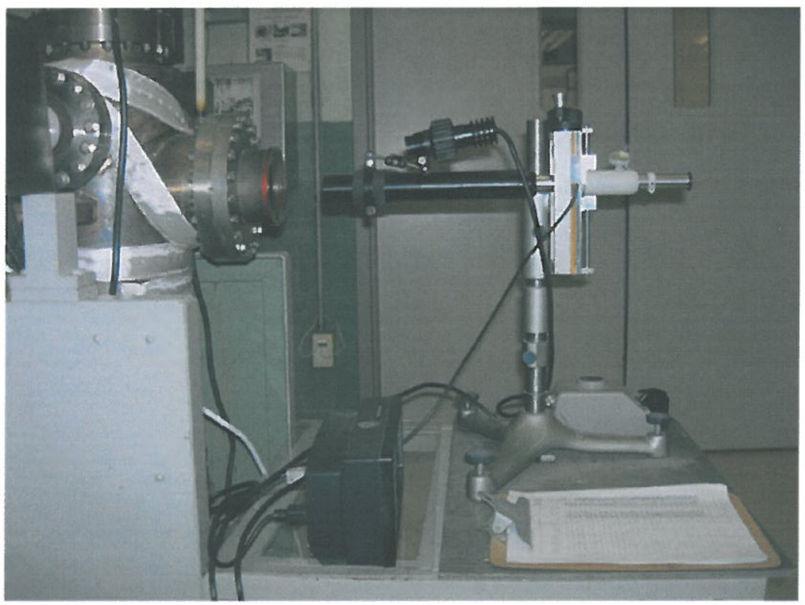
NASA Vacuum Creep Capability

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

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.



Cathetometer

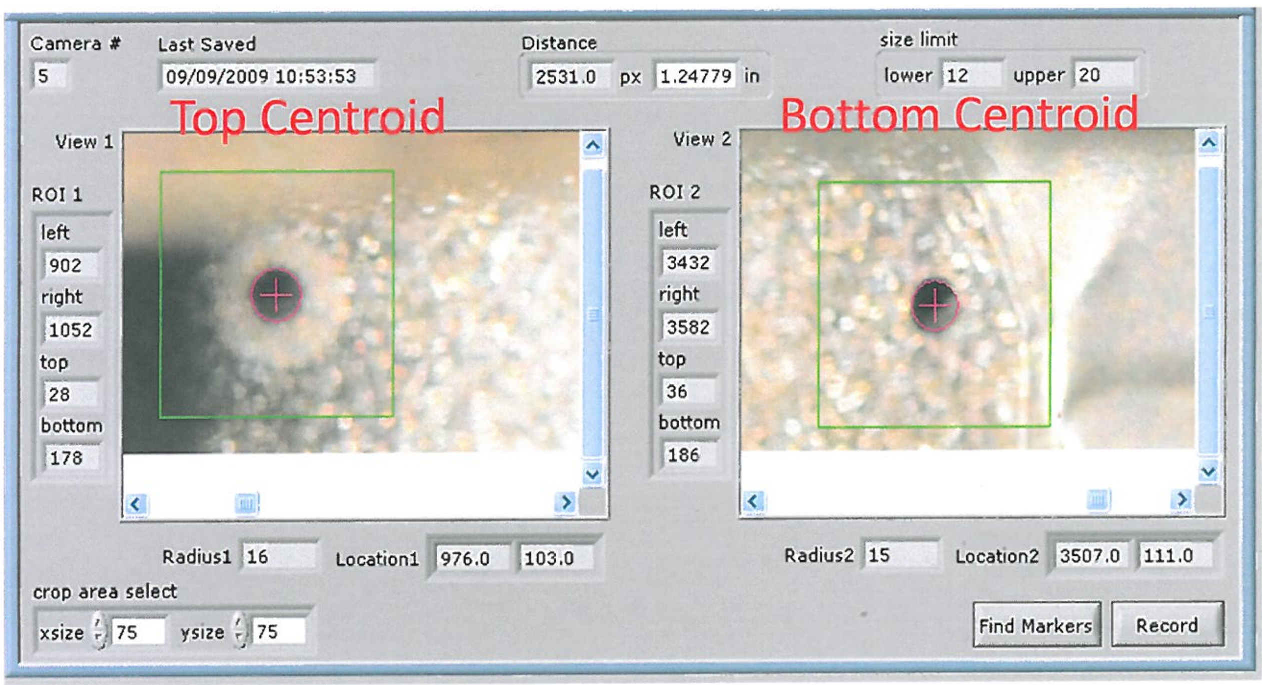
Video Extensometer

Jaster, Vickerman, Padula, Juhas; NASA Internal Communication, March 2010.

Creep Lab Video Extensometry for Vacuum Creep Testing

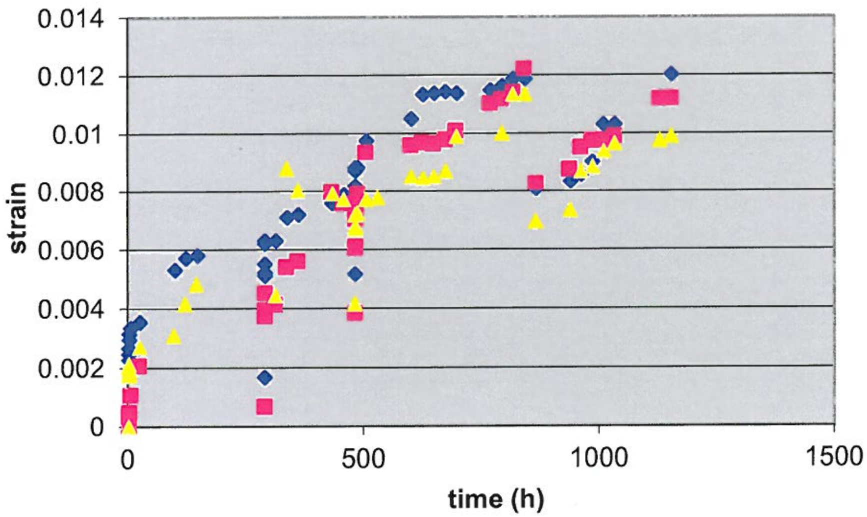
Method

- 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

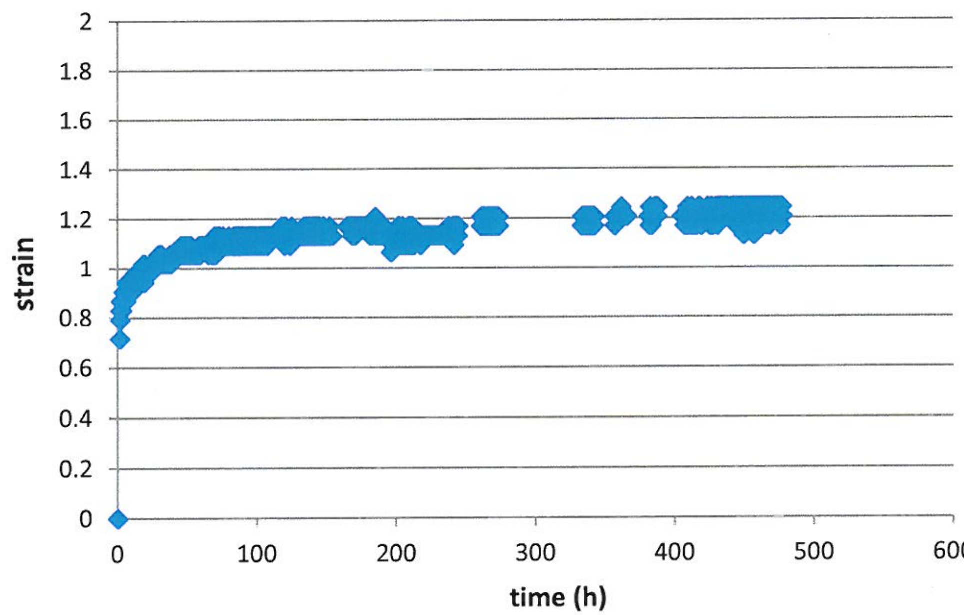


Jaster, Vickerman, Padula, Juhas; NASA Internal Communication, March 2010.

Comparison of Types of Data Possible From Both Cathetometry and Optical Extensometry



Cathetometer



Optical Extensometer

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

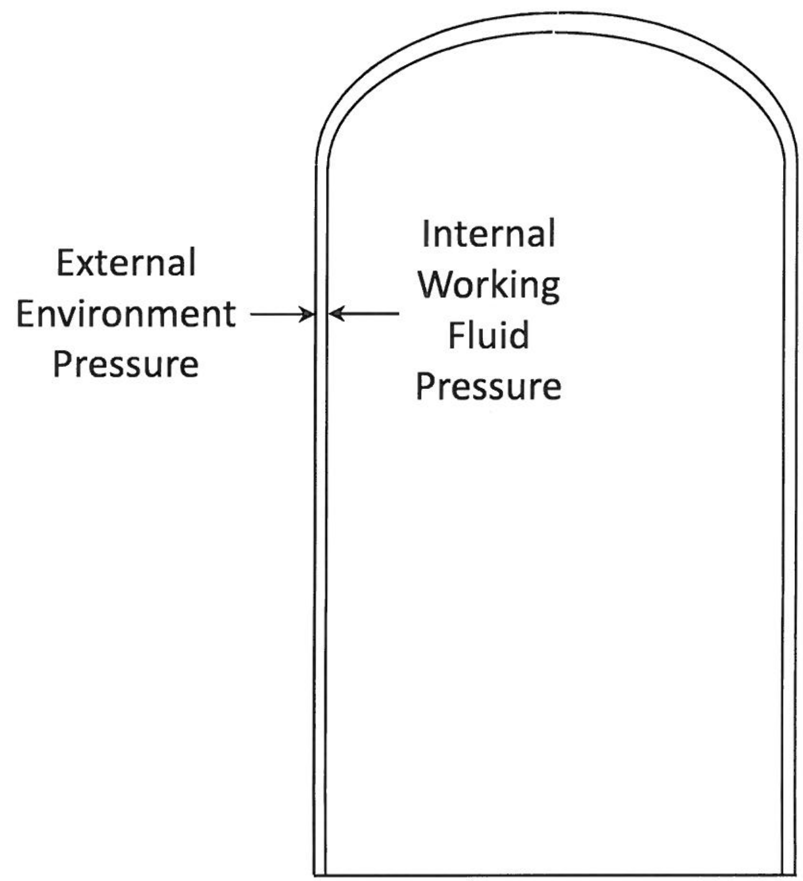
Tantalum and Rhenium Alloy Candidates

Advanced Stirling Technology

Application of Tantalum and Rhenium as a Heater Head

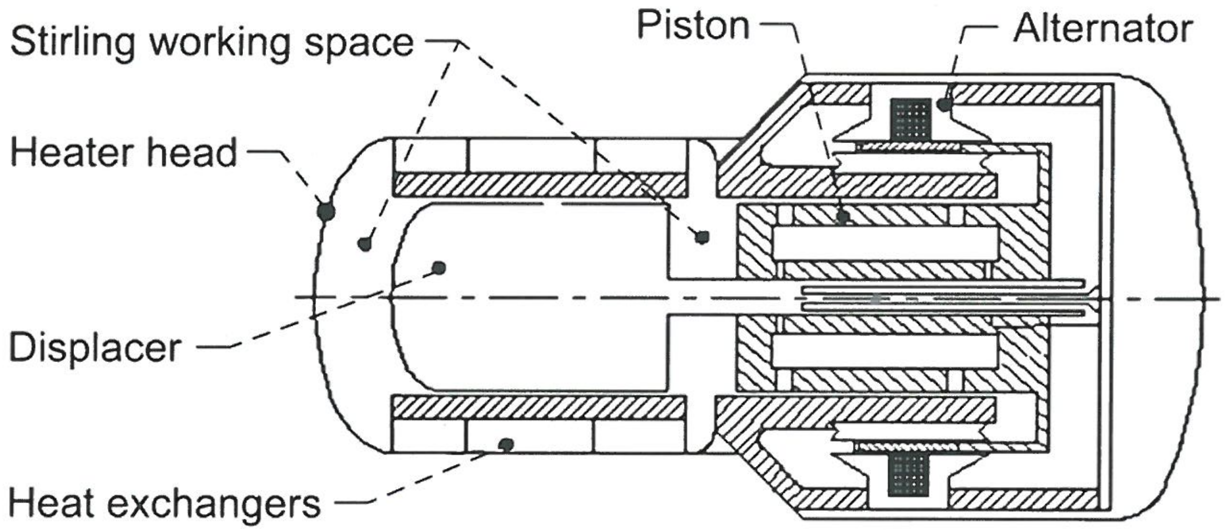
Creep Resistance of Heater Head is a Major Requirement

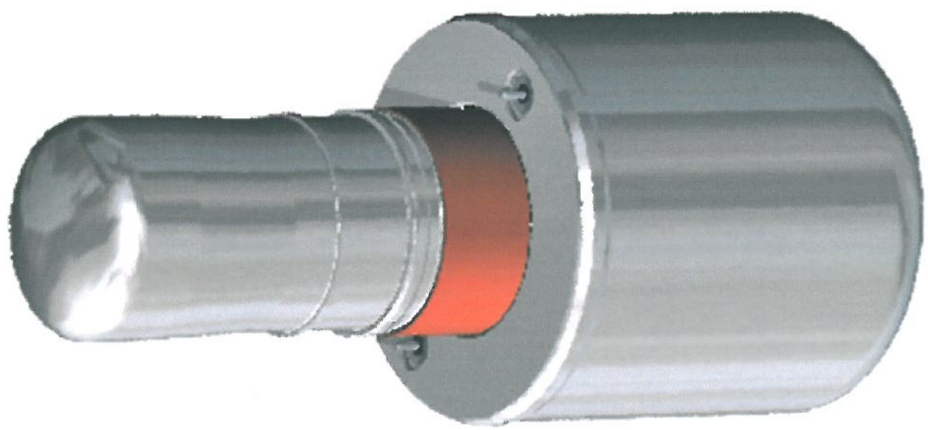
- Creep stress exerted on heater head by pressure differential across thin wall section estimated by hoop stress equation for a cylinder
  - $\sigma_{hoop} = \Delta Pr/t$
    - $\Delta P \equiv$ pressure differential across heater head wall
    - $r \equiv$ inner cylinder (heater head) radius
    - $t \equiv$ heater head wall thickness



Stirling Heater Head Identified as Most Critical Component

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

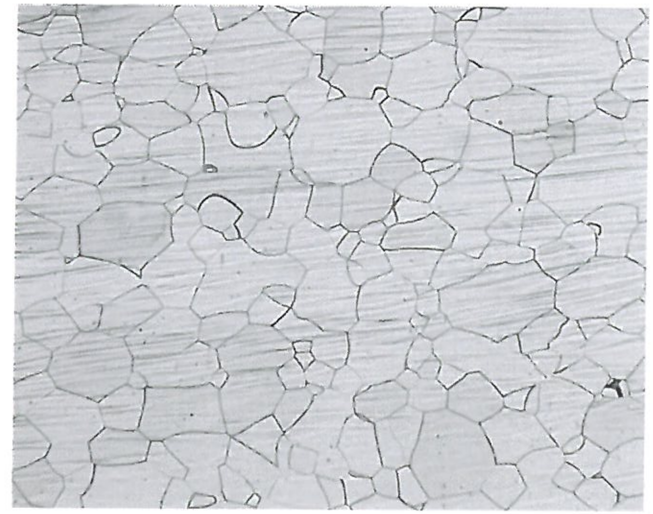




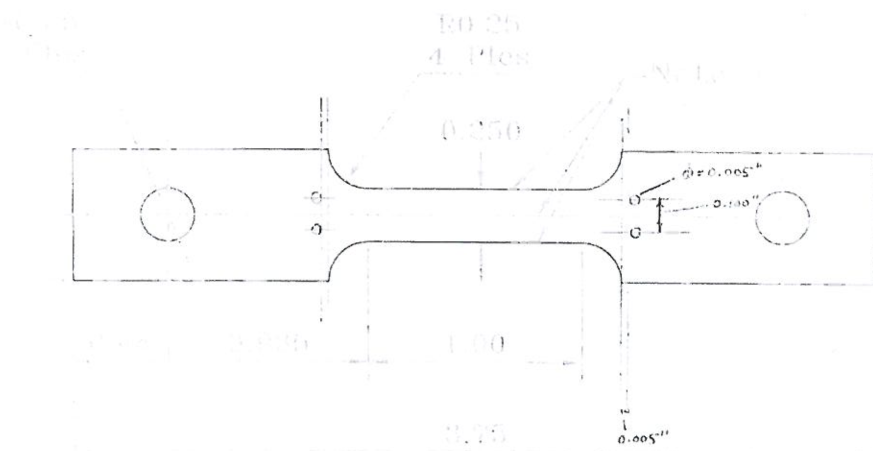
Tantalum Candidacy for Stirling Engine Heater Head

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.

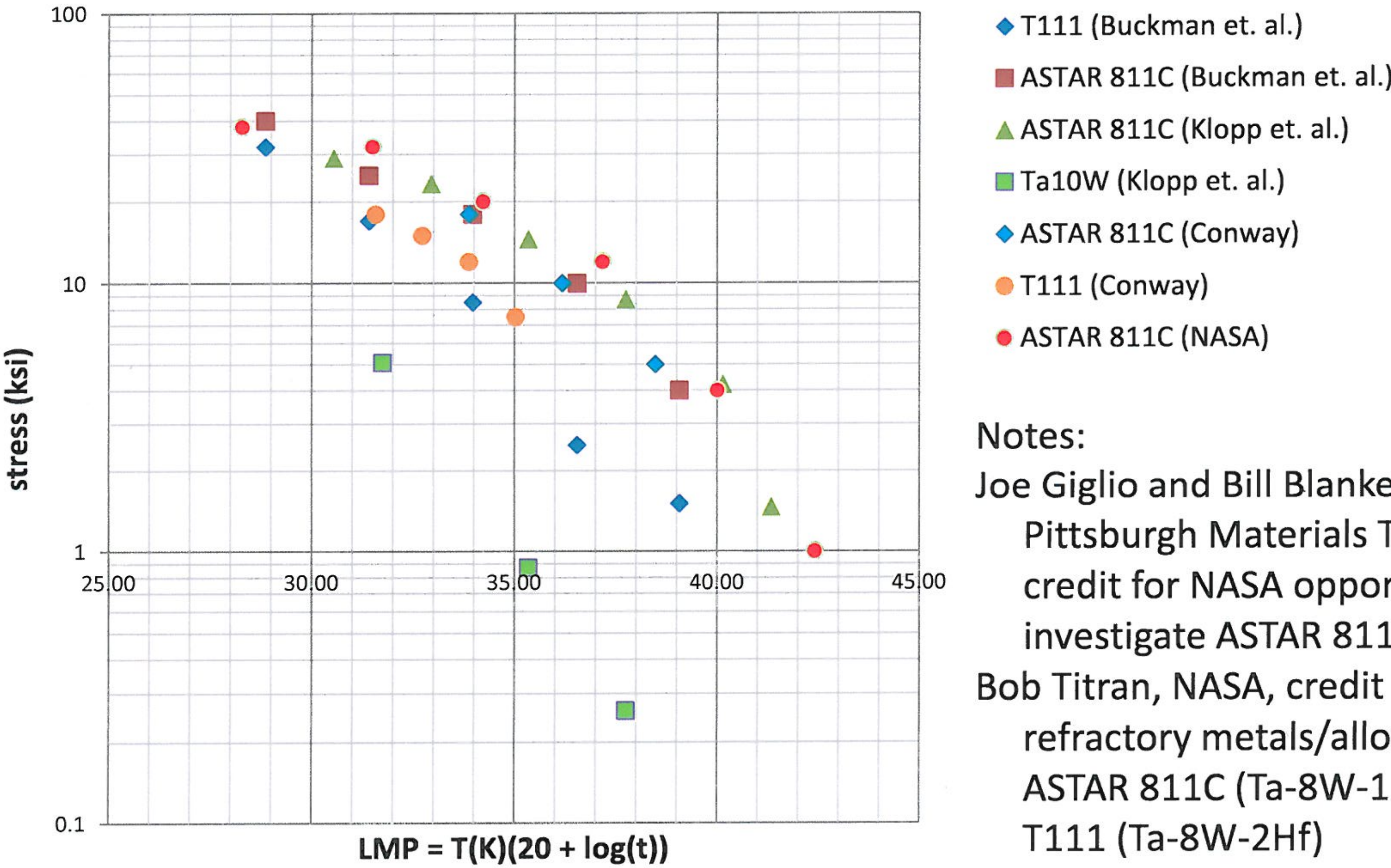


Microstructure of extruded ASTAR 811C transverse section
50X



NASA test specimen

Time to 1 Percent Creep Strain Data for Tantalum Alloys



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)

R.W. Buckman and R.R. Begley, "Development of High Strength Tantalum Base Alloys", Defense Technical Information Center, 1970.

W.D. Klopp, R.H. Titran, K.D. Sheffler, "Long-Time Creep Behavior of the Tantalum Alloy Astar 811C", NASA TP 1691, September 1980.

J.B. Conway, "Mechanical and Physical Properties of Refractory Metals and Alloys", Proceedings of Symposium on Refractory Alloy Technology for Space Nuclear Power Applications, Oak Ridge, TN, August 1983.

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

Temperature, C	Time to 1% strain, h	Stress, ksi	Steady-state creep rate, sec^{-1}
1000	169	38	1.6E-09
1100	877	32	3.1E-09
1227	660	20	2.8E-09
1350	805	12	2.8E-09
1450	1667	4	2.0E-09
1550	1892	1	9.4E-10

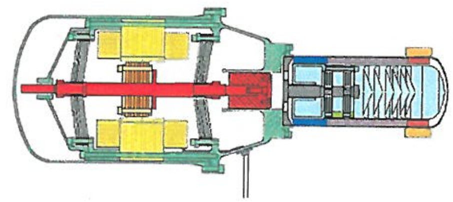
NASA SBIR SUCCESS

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



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

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

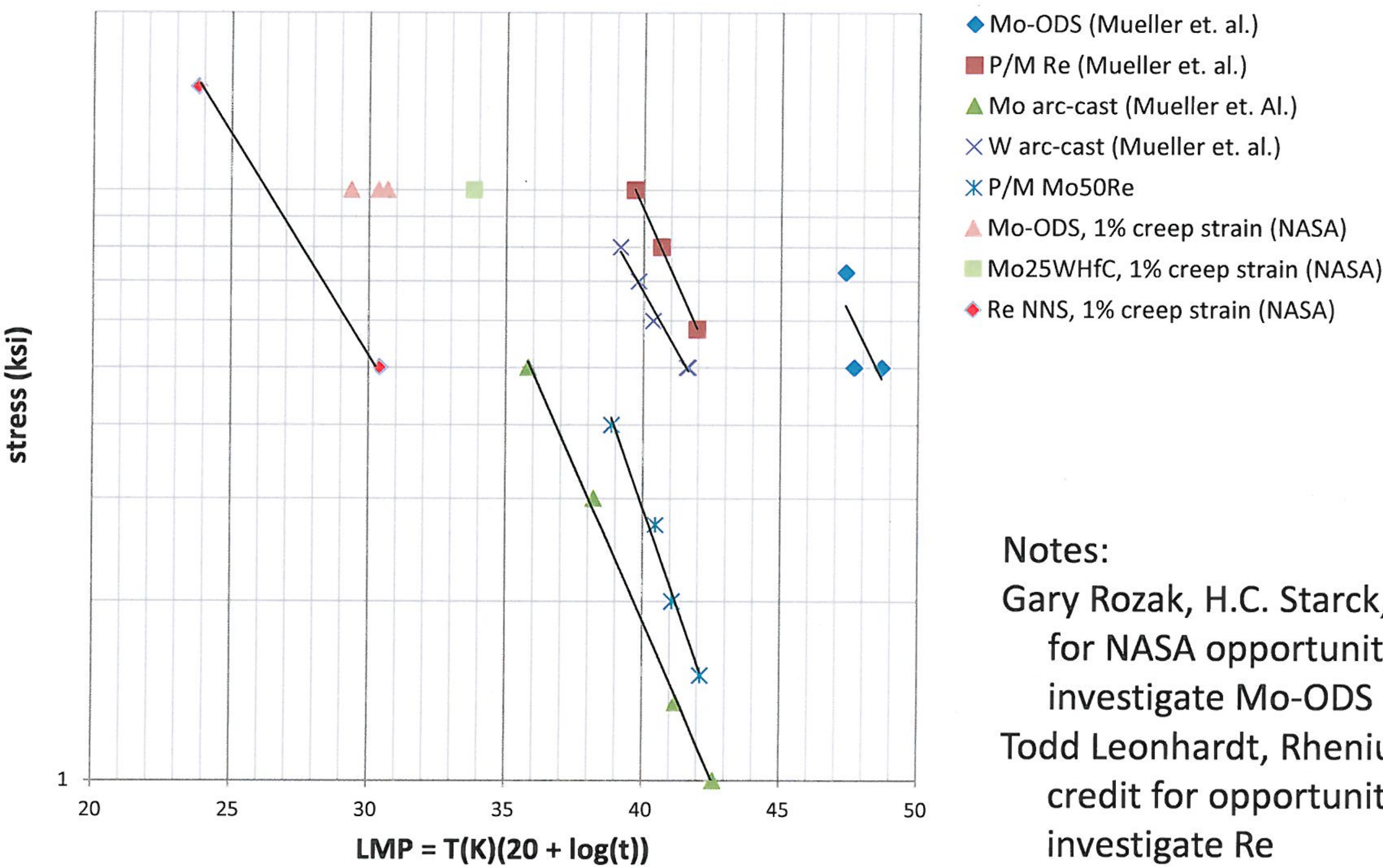
- The CIP to NNS method of manufacturing was used to produce a dome for a commercial customer.
- This method has increased the job equivalents by 2, which is directly associated with this SBIR.

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.

Source: Todd Leonhardt, Rhenium Alloys

Creep Rupture of Selected Refractory Alloys



Notes:

Gary Rozak, H.C. Starck, credit for NASA opportunity to investigate Mo-ODS

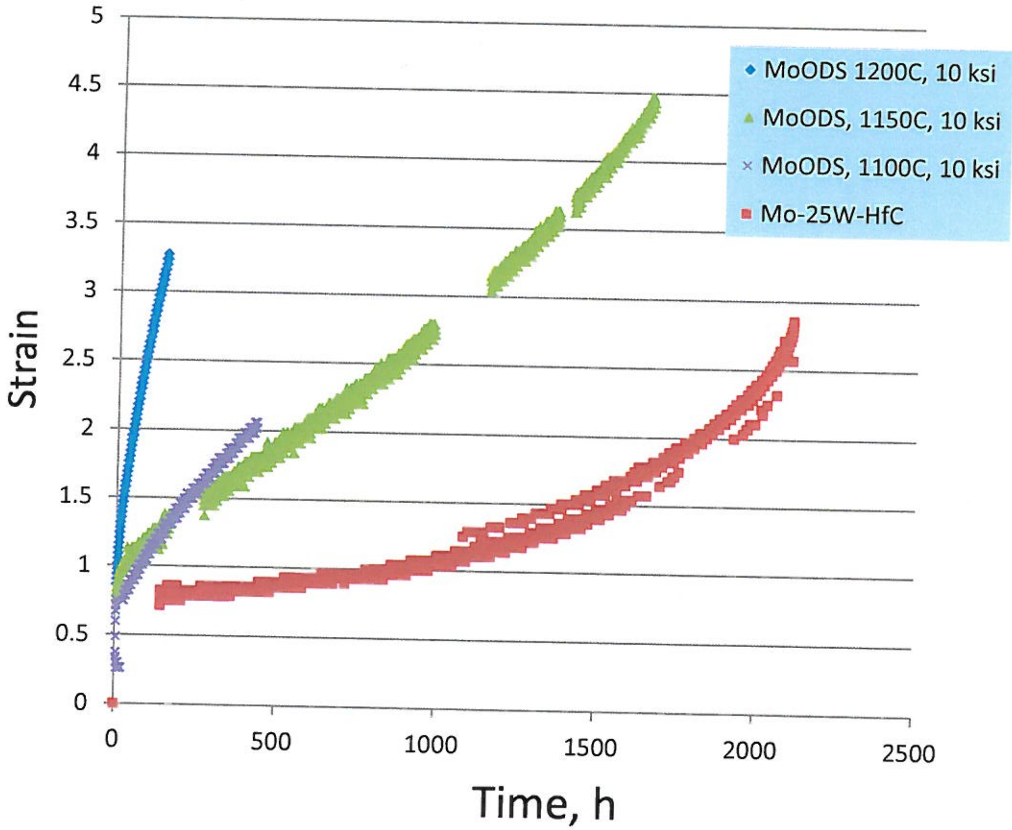
Todd Leonhardt, Rhenium Alloys, credit for opportunity to investigate Re

A.J. Mueller, R. Bianco, R.W. Buckman. "Evaluation of Oxide Dispersion Strengthened (ODS) Molybdenum and Molybdenum-Rhenium Alloys", Bettis Atomic Power Laboratory, B-T-3148.

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

ID	Temperature, C	Stress, ksi	Time to 1% creep strain, h	LMP	Steady-state creep rate, sec^{-1}
MoODS-1	1200	10	4	30.35	3.60E-08
MoODS-3	1150	10	75	31.13	4.40E-09
MoODS-4	1100	10	75.7	30.04	7.50E-10
HWM-1	1200	10	886	33.80	7.40E-10

Proposed Mission to Venus

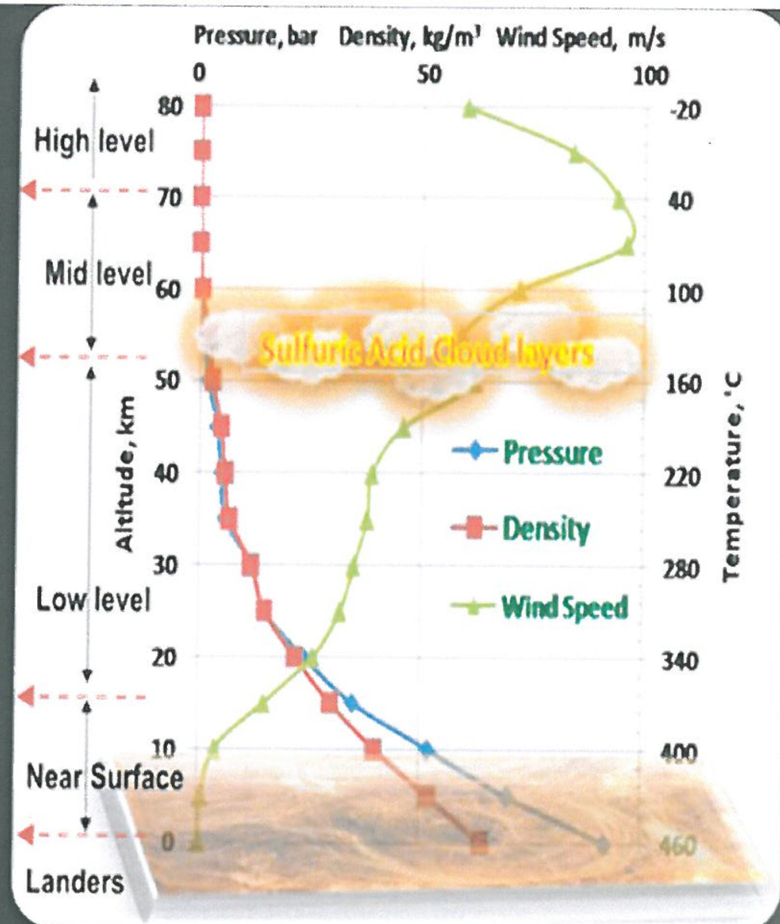
Application of Molybdenum

- High temperature
  - 1000 °F (500 °C)
- High pressure
  - 1500 psig (100 bar)
- Corrosive/Acidic
  - CO_2 (~96.5%)
  - SO_2 (130 ppm)
  - HF (5 ppb)
  - HCl (0.5 ppm)
  - NO (5.5 ppb)
  - CO (15 ppm)
  - COS (27 ppm)
  - N_2 (~3.4%)
  - H_2O (30ppm)

Acids/ Poisons (extremely corrosive): CO_2, SO_2, HF, HCl

Oxidizer: NO

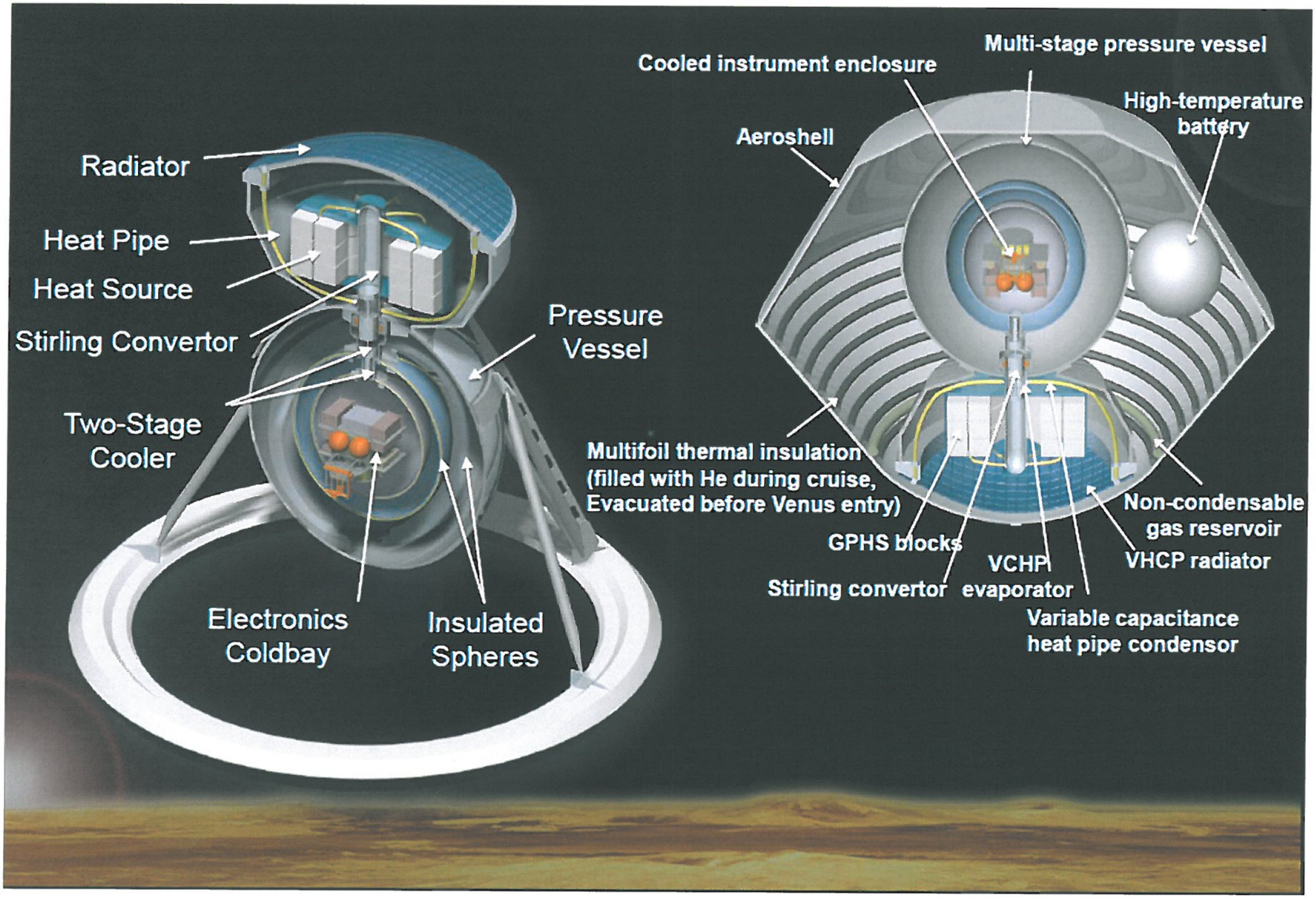
Flammable: CO, COS



Venus Atmospheric Conditions

Long-lived Venus Lander Conceptual Design: How To Keep It Cool. **R.W. Dyson, P.G. Schmitz, L.B. Penswick, G.A. Bruder.**
Denver, CO : American Institute of Aeronautics and Astronautics, 2009. 7th International Energy Conversion Engineering Conference (IECEC 2009). p. 27.

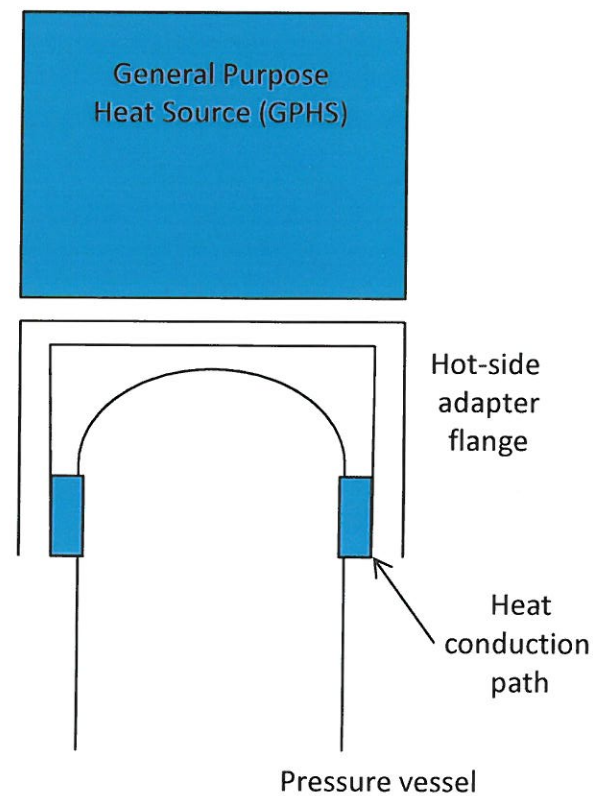
Proposed Venus Lander Design



R.W. Dyson, "Expanding Solar System Exploration with an Extreme Environment Test Chamber Stakeholder Review" NASA internal presentation, September 21, 2010.

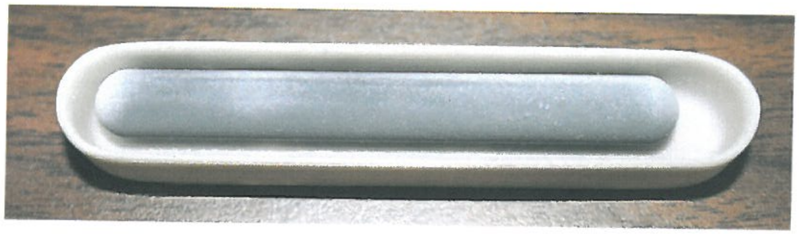
Hot-Side Adapter Flange Improvement

- 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



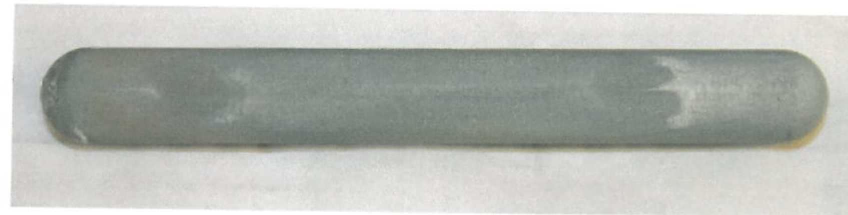
Relative Location of HSAF

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

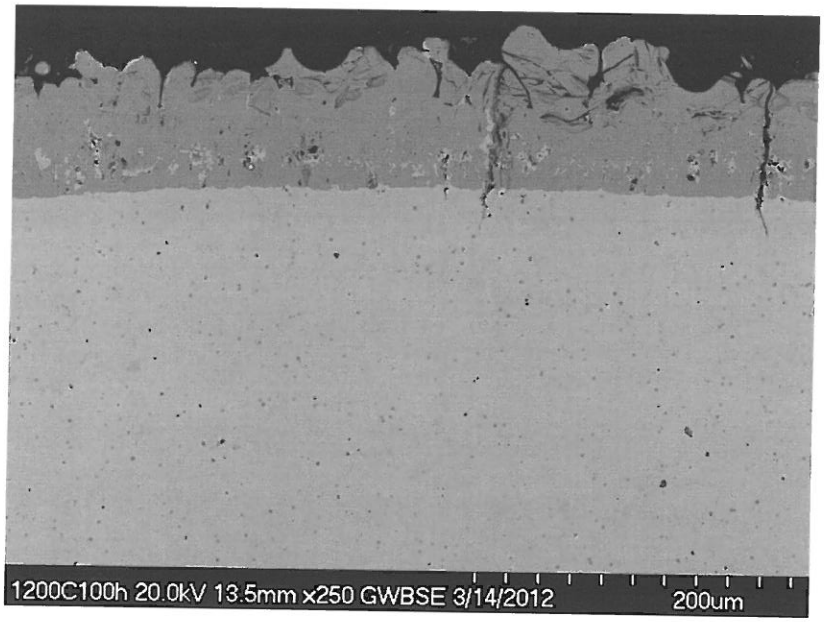


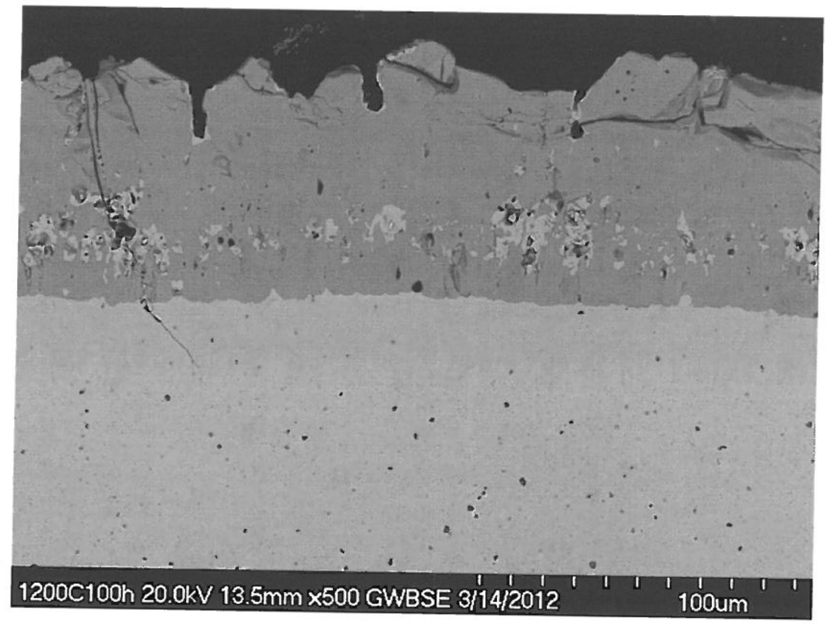
Pure Mo

- Sample exposed for 10h intervals at 1200°C in 5 ppm O_2 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