



# Prometheus and Constellation Materials Research and Testing Overview

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# Outline

- New Technologies and New Materials meet Flight Program
- Reactor Materials Selection
- Material Radiation Resistance
  - Testing in four different radiation environments
- Superalloy Creep Testing
- Superalloy-Refractory Alloy Joining
- Superalloy, Stainless Steel, Copper Brazing
- Superalloy-Refractory Cross Contamination
- Na and NaK Compatibility
- Radiator Panel Materials Selection



# New Technology & New Materials meet Flight Program

## **Space Reactors are different than Terrestrial Reactors**

- Optimum designs are different; optimum materials are different
- Preferred core materials for non-standard designs do not have extensive property databases
- Commit to using standard, terrestrial materials or Commit to doing data generation

## **Advanced Materials and Manufacturing enable better component performance**

- New technologies, new components, new materials add risk
- Balance system requirements with each new technology/component/material risk
- Commit to risk reduction; for materials that means property data generation and testing

## **Non-Reactor Components have added radiation requirements in nuclear environment**

- Established components and materials still require scrutiny in new environments

Adopt a “Flight Qualification” Mindset Early in Materials Selection

# Materials Challenges for Nuclear Power and Propulsion

## Reactor Materials Selection

- Fuel, Core Structure, Control Surfaces, Interfaces

## Material Radiation Damage outside the Reactor Subsystem

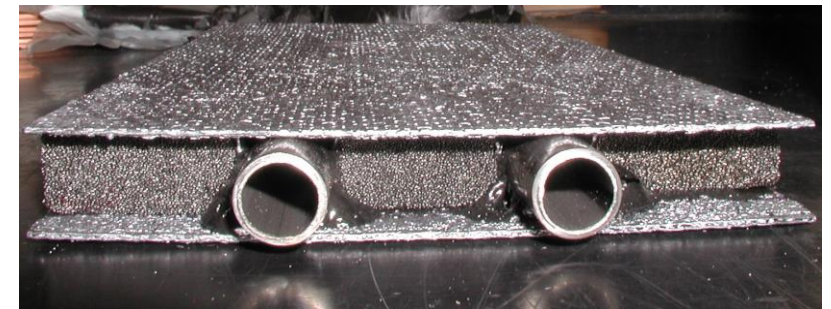
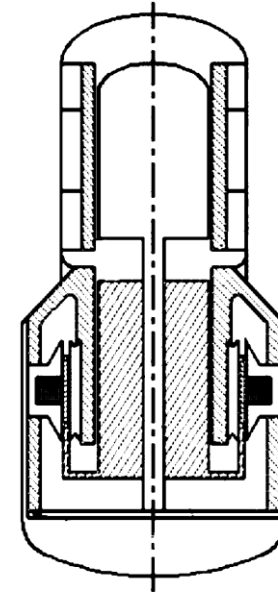
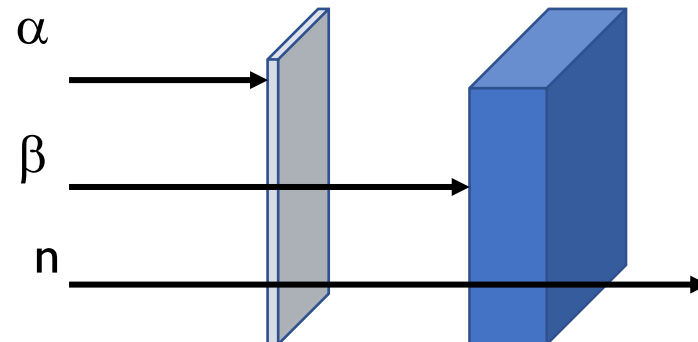
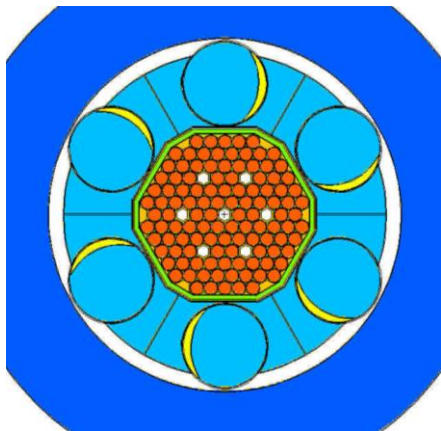
- Structure, Motor/Generator/Electronics, Organic Polymers

## Materials for High Performance Power Conversion

- Highest possible operating temperatures for decades of life

## Materials for High Performance Radiator

- Reliable heat transfer in lightest possible structure



# Reactor Materials Selection

## Reactor Core Structure Depends on Temp, Fluence, Lifetime

- Iron alloys are best characterized for long life, high total fluence, but lower temperatures
- Nickle alloys may be acceptable for lower total fluence and intermediate temperatures (Angeliu, LM-06K033, 2006)
- Higher temperature alloy or ceramics require investment

Increasing Temperature Capability →

|                         | Austenitic SS | Ni Superalloys | Nb Alloys   |
|-------------------------|---------------|----------------|-------------|
| Physical Properties     | Green         | Green          | Green       |
| Fabrication             | Green         | Green          | Yellow      |
| Joining                 | Green         | Green          | Yellow      |
| Thermal Stability       | Yellow        | Green          | Green       |
| Mechanical Properties   | Yellow        | Green          | Yellow      |
| Coolant Compatibility   | Green         | Green          | Green       |
| Fuel Compatibility      | Yellow        | Yellow         | Yellow      |
| Irradiation Performance | Green         | Yellow         | Yellow      |
| Technological Maturity  | Green         | Green          | Red Hatched |
| Reactor Heritage        | Green         | Red Hatched    | Yellow      |

# Material Radiation Resistance Introduction

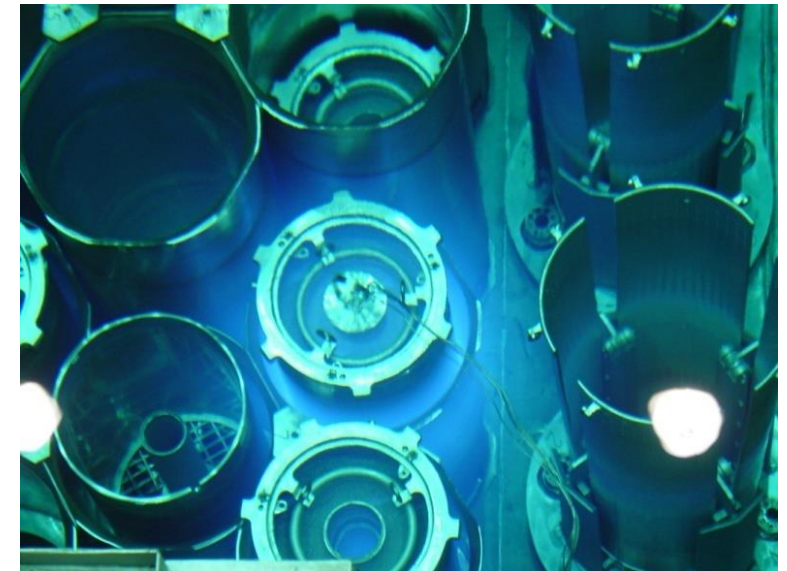
## Standards

- Radiation Damage falls within general requirements of NASA-STD-6016C Standard Materials and Processes Requirements for Spacecraft
- Methodology guidelines were enumerated in NASA SP-8053 Nuclear and Space Radiation Effects on Materials
  - Define Environment
  - Review Anticipated Radiation Response
  - Determine Performance in Predicted Environment

## Studies

- Prometheus/JIMO studies began testing of certain materials
- Constellation/FSP completed baseline radiation hardness specification and initial characterization: NASA/TM-2011-216992

## ORNL HFIR Spent Fuel Pool Testing



# JIMO Irradiation Screening

## Ohio State Research Reactor to Survey Various Coating

- Thermal control paint: Two paints, Three substrates, Two exposures up to  $2 \times 10^{15}$  n/cm<sup>2</sup>
  - No visual or emittance-measured degradation
- Solid Lubricants: Five thin films and One thick coating, Three exposures up to  $1 \times 10^{16}$  n/cm<sup>2</sup>
  - Thin coatings evaluated with bend and scratch+pull testing
  - Thick coating evaluated with pneumatic adhesion tester
  - Limited measurable degradation

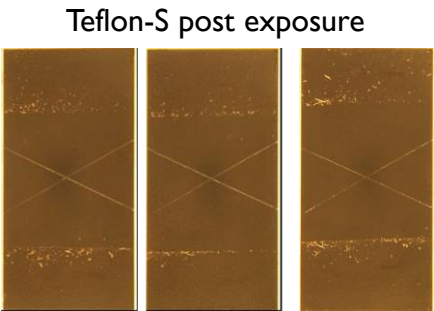
OSU Reactor



Exposure Facility



AZ93 white on C/polyimide  
Pristine,  $2 \times 10^{13}$ ,  $2 \times 10^{15}$



Teflon-S post exposure

| n/cm <sup>2</sup>    | Teflon-S | Emralon<br>333 | Xylan 1620 | Endura<br>420-10 | Endura<br>440-11 | Korolon<br>800 |
|----------------------|----------|----------------|------------|------------------|------------------|----------------|
| Pristine             | No/trace | No peeling     | No peeling | Trace peeling    | No/trace         | No/trace       |
| $1.5 \times 10^{15}$ | No/trace | No peeling     | No peeling | Trace peeling    | No peeling       | Up to 1.8 mm   |
| $3 \times 10^{15}$   | No/trace | No peeling     | No peeling | No/trace         | No peeling       | Trace peeling  |
| $1 \times 10^{16}$   | No/trace | No peeling     | No peeling | Trace peeling    | No peeling       | Up to 1.8 mm   |

# Material Radiation Resistance for Power Conversion System (PCS)

**Candidate PCS components not closely scrutinized for radiation hardness prior to JIMO/FPS**

## Categorized by:

- Metallic alloys—insensitive to ionization and excitation damage due to shared conduction electrons. Typically withstand fluences much greater than  $10^{20}$  neutrons/cm<sup>2</sup> before displacement/capture damage notably influences *structural properties*
- Magnetic alloys—insensitive to ionization and excitation damage but sensitive to magnetic order loss due to thermal spikes and displacement damage
- Polymeric materials—most sensitive, some can be affected by ionizing radiation as low as 0.1 MRad

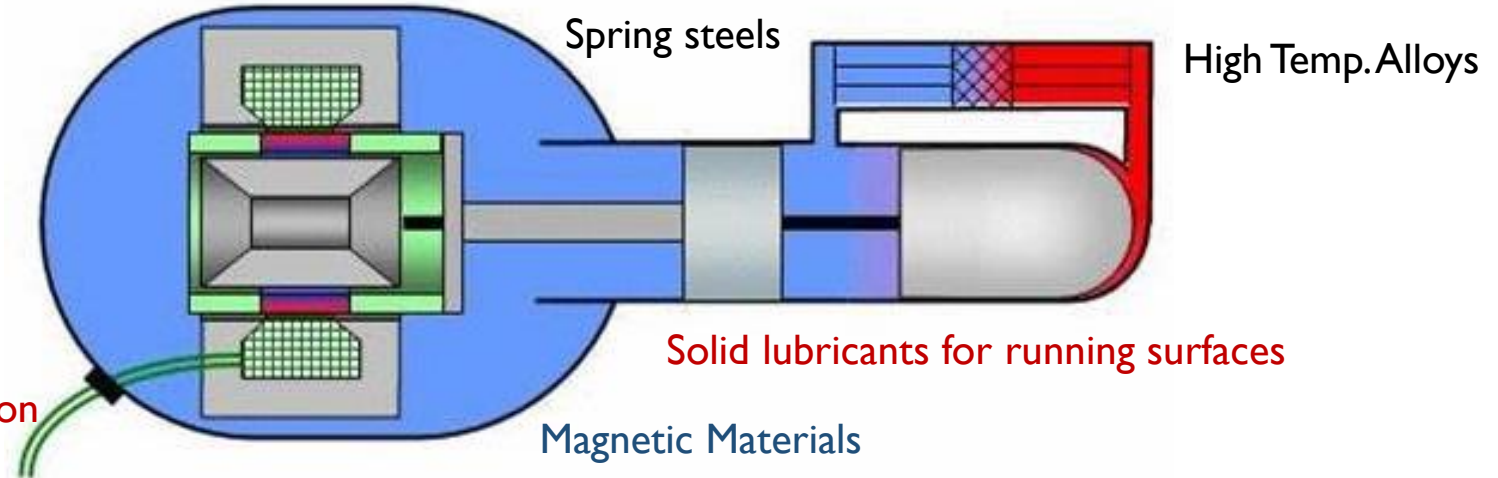
Low Temp. Alloy housing & supporting structures

### Bonding Materials:

- Permanent Magnet Bonding
- Feedthrough Bonding/Sealing
- Secondary Bonding on Fasteners
- Lamination Bonding
- Coil Potting

### Insulation Materials:

- Coil Wire Insulation
- Electrical Wire Insulation



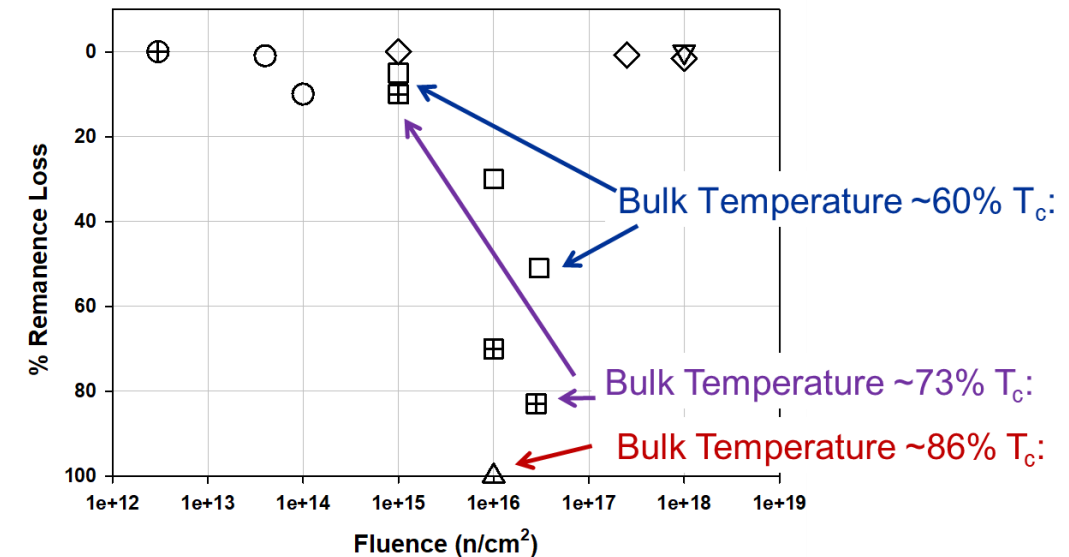
# Material Radiation Resistance—Magnetic Materials

## Magnetic Material Guidelines based on literature

- Permanent magnets should have minimal rad effects up to 50%  $T_c$  in low demagnetizing fields in space fission-relevant radiation environment
- Radiation damage is less well characterized for soft magnetic materials and is believed to be minimal at fluences below  $10^{18}$  n/cm<sup>2</sup>
- Radiation likely to accelerate temperature driven changes, based on Kähkönen damage theory
- NASA TM-2012-217752

## Rare Earth Perm Mag Rad Damage

Combined results from 7 studies



Literature suggested that baseline magnetic materials acceptable for projected FSP mission



# Material Radiation Resistance Summaries

## Polymeric Material Background

- Sensitivity to radiation effects is governed by polymer backbone chemistry, additives, temperature, and environment
- Damage through inelastic energy transfer: Ionizing energy leads to excited electron states, breaks covalent bonds, & forms free-radicals
- Chemical cross-linking and/or chain-scission dependent on temperature and radiation environment.
- Irradiation Temperature and Oxygen Presence greatly impact radiation damage.
- In general, higher temperature polymers (aromatic) have greater radiation resistance
- ***Irradiation literature dominated by room temperature exposures in air, not necessarily relevant to FPS conditions***
- ***Radiation damage typically measured by residual tensile strength, not necessarily relevant to FPS conditions***

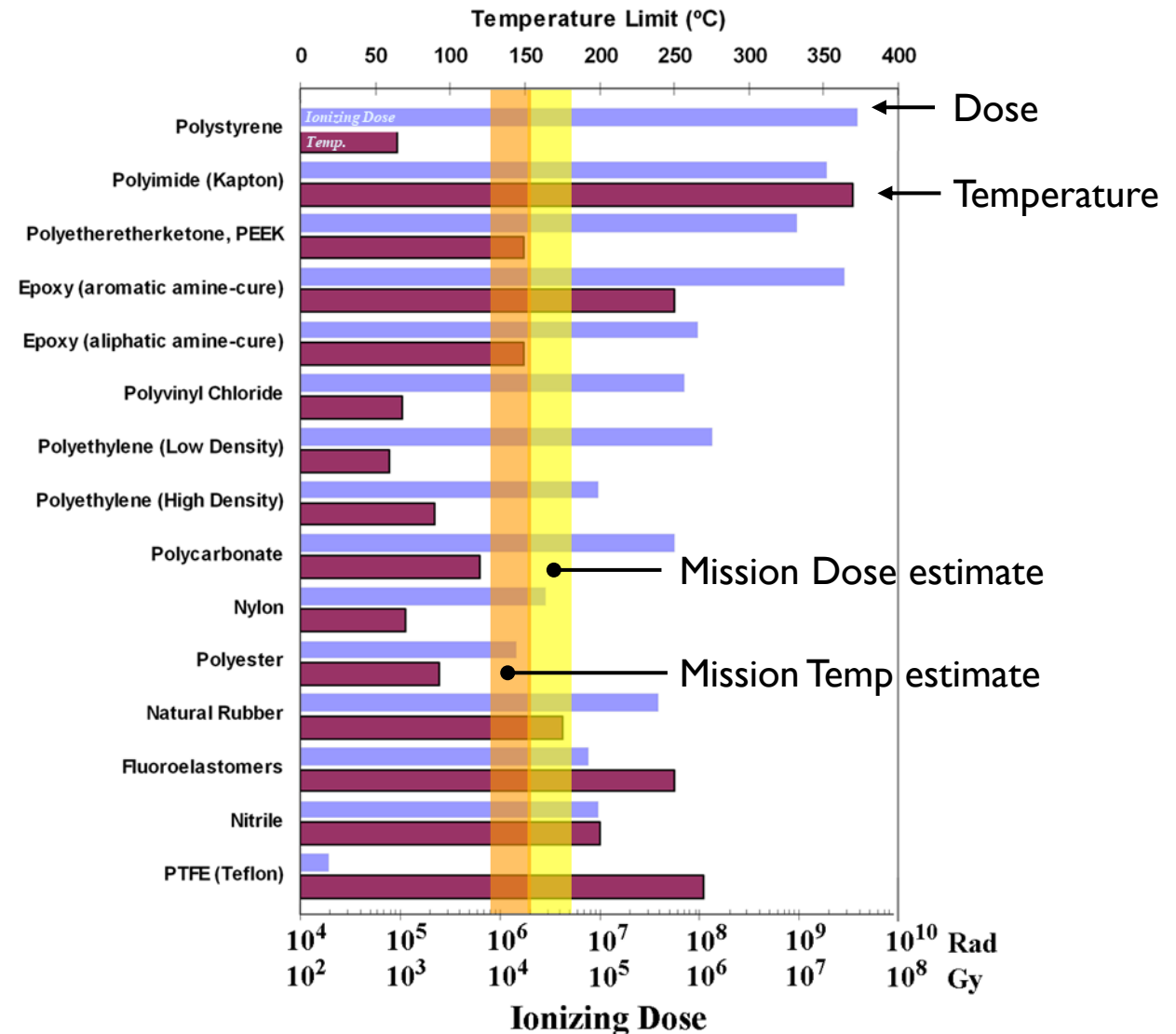
Verification Testing Recommended for baseline Fission PC System

# Material Radiation Resistance—Polymers

## Detailed Background Review

- Leonard, Keith J. et al.,  
“Radiation Sensitivity, Dose and Temperature Limitations of Polymeric Materials for Application in a Fission Surface Power Convertor”,  
in Space Technology and Applications International Forum (STAIF-2008) 25th Symposium on space Nuclear Power and Propulsion February 14th, 2008
- PTFE / Teflon stands out as a high temperature polymer with low dose limit

## Dose / Temperature Resistance





# Material Radiation Resistance—Polymer Evaluation

## Component Materials Evaluated

- Epoxy Bonding/Potting Material
  - Lap-shear Bonding Strength
- Elastomeric O-rings
  - Leak Testing, Compression Set, and Tensile Strength
- Polymer-insulated Wire
  - Nano-indentation Measured Hardness, Elastic Modulus, and Scratch-test Damage
- Heat Shrink Tube
- Solid Lubricant Coating

## Irradiation Facilities Utilized

- Oak Ridge National Lab HFIR Spent Fuel Pool
- Texas A&M Univ TRIGA Reactor
- Sandia National Lab Gamma Irradiation Facility

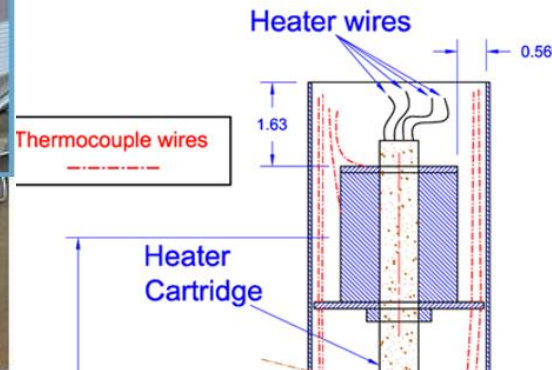
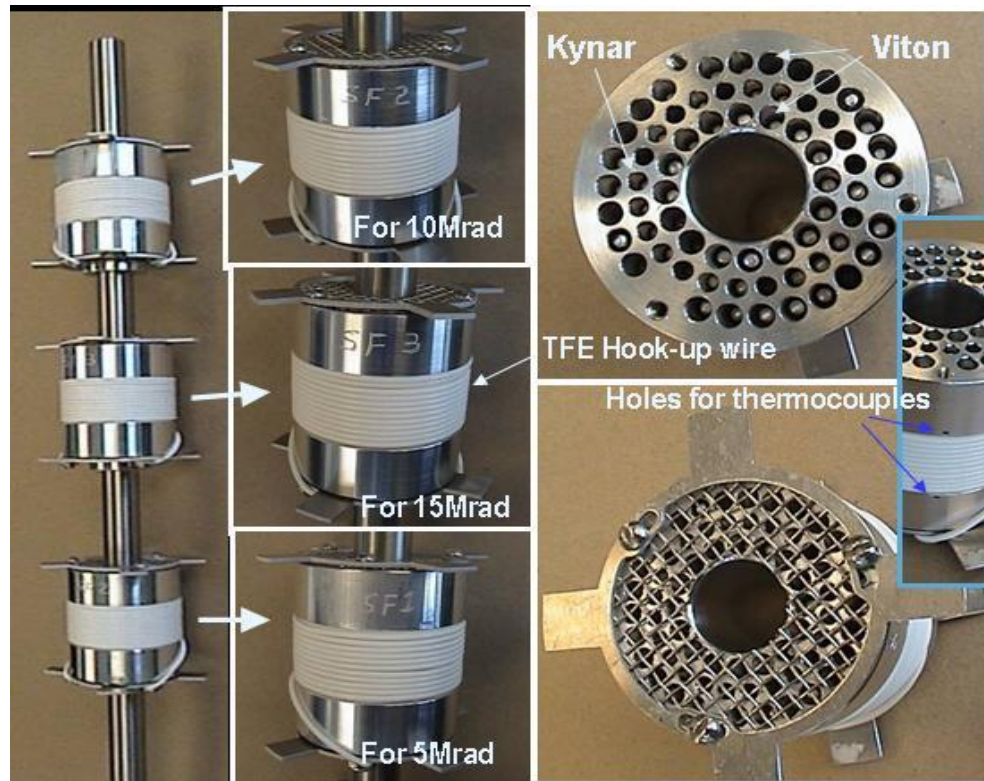
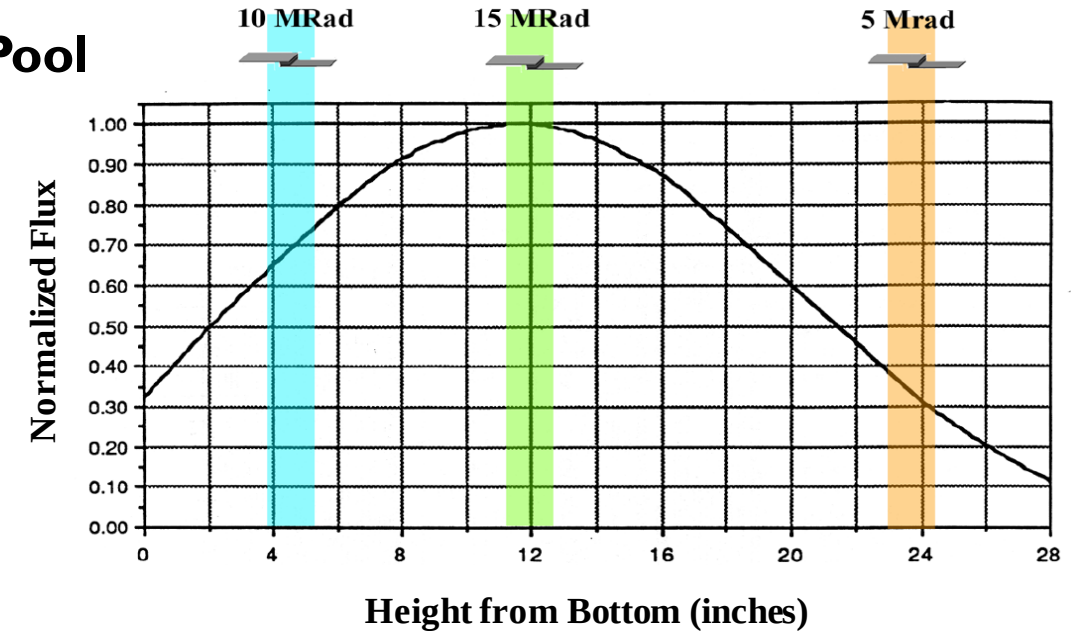
## Extensive Post-Irradiation Evaluation

- Optical and Electron Microscopy
- Typical TGA, DMA, FTIR
- Mechanical Testing

# Material Radiation Resistance—Polymer Evaluation

## ORNL High Flux Isotope Reactor (HFIR) Spent Fuel Pool

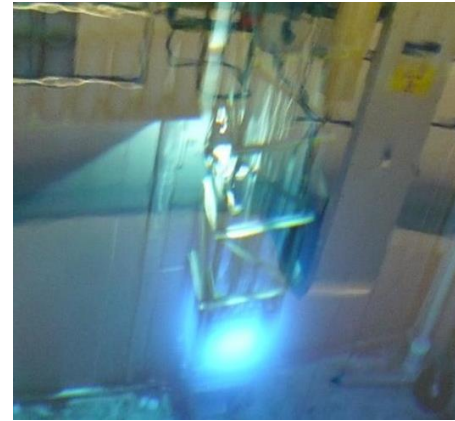
- Dose rate controlled by location
- Temp: 400, 423 K (125, 150C)
- Inert, Ar cover gas



# Material Radiation Resistance—Polymer Evaluation

## Texas A&M Univ TRIGA Reactor

- $1 \times 10^{14}$  neutrons/cm<sup>2</sup>, 1.4 Mrad
- $5 \times 10^{14}$  neutrons/cm<sup>2</sup>, 5.4 Mrad
- Temp: 400, 423 K (125, 150C)
- Inert, He cover gas



Reactor coupled to irradiation cell

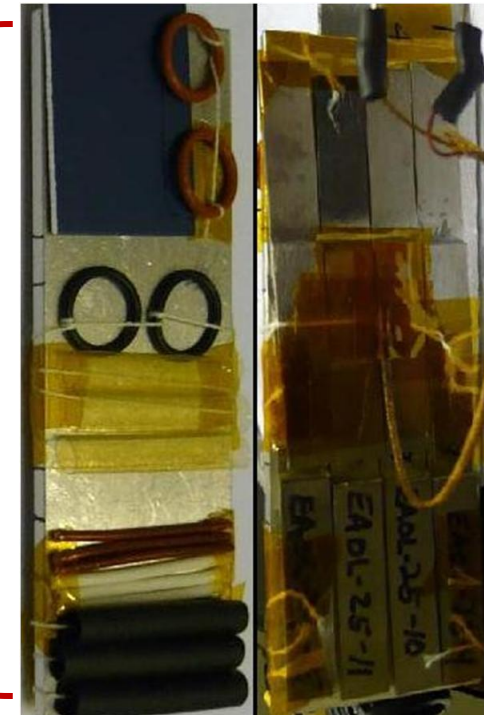
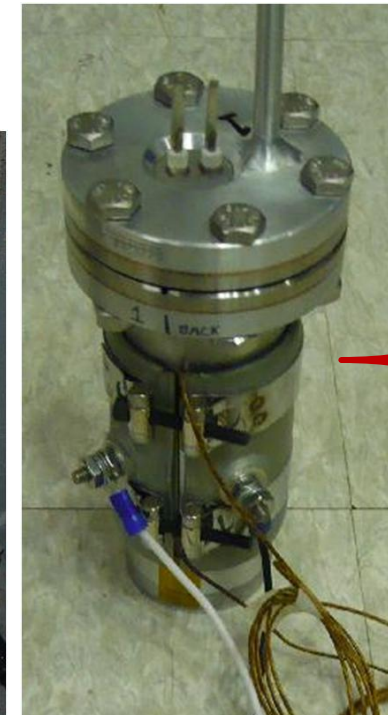
Test articles in irradiation window

Dosimetry



Mireles, Omar R., et al.: Proceedings of 8th International Energy Conversion Engineering Conference (AIAA), Paper 769917, Nashville, TN, 2010.

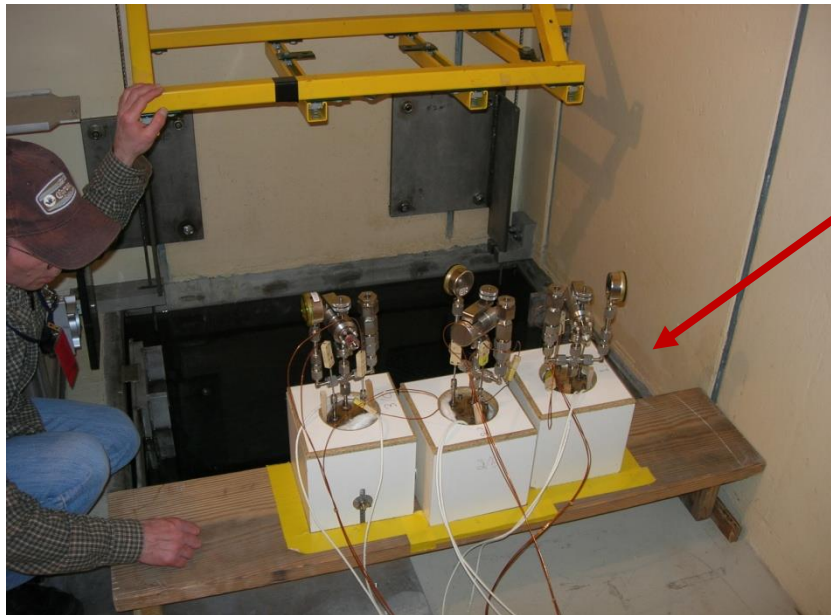
Test articles in irradiation window



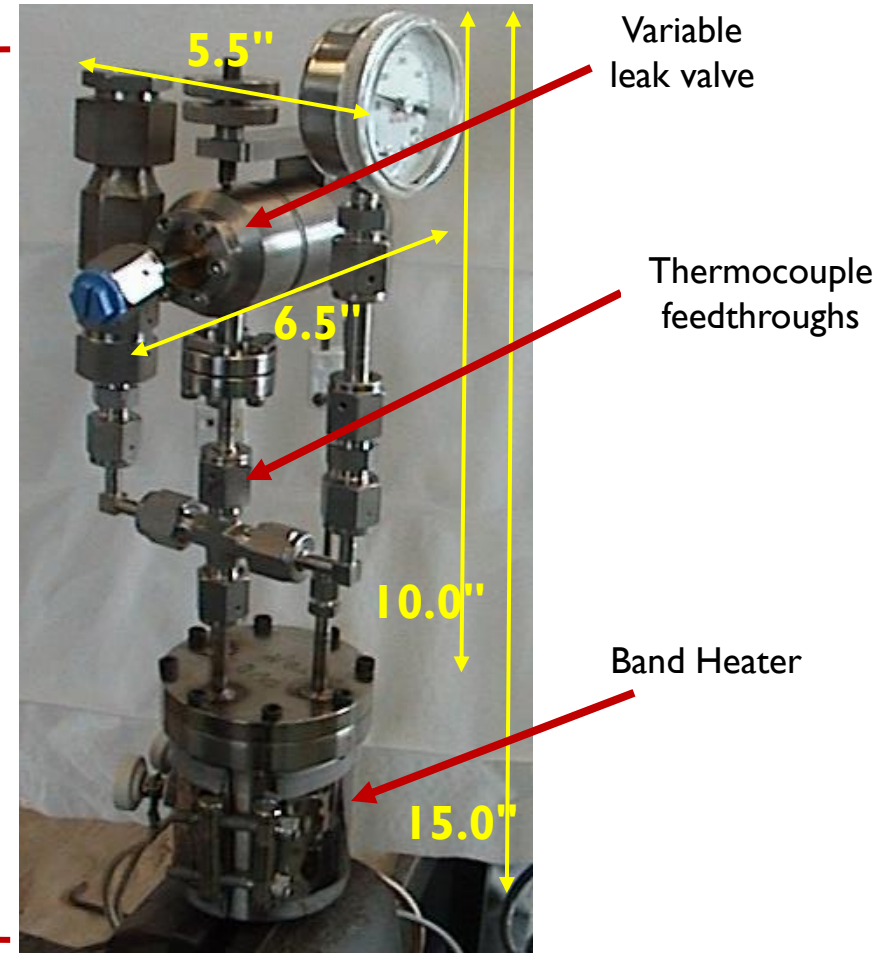
# Material Radiation Resistance—Polymer Evaluation

## SNL Gamma Irradiation Facility

- Dose Rates: 100 rad/s, 1000 rad/s
- Temp: 400, 423 K (125, 150C)
- Outgassing Measured at 500 kRad, 1 MRad, 5 MRad, 10 MRad



## Gamma In-situ Outgassing Evaluation



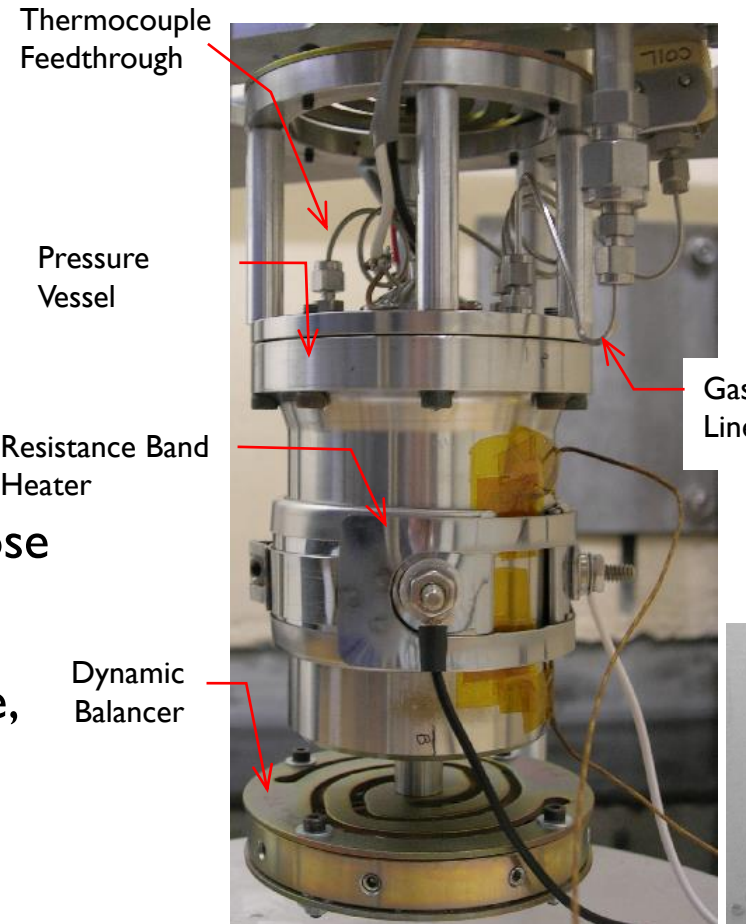
# Stirling Alternator Radiation Test Article (SARTA)

## SNL Gamma Irradiation Facility

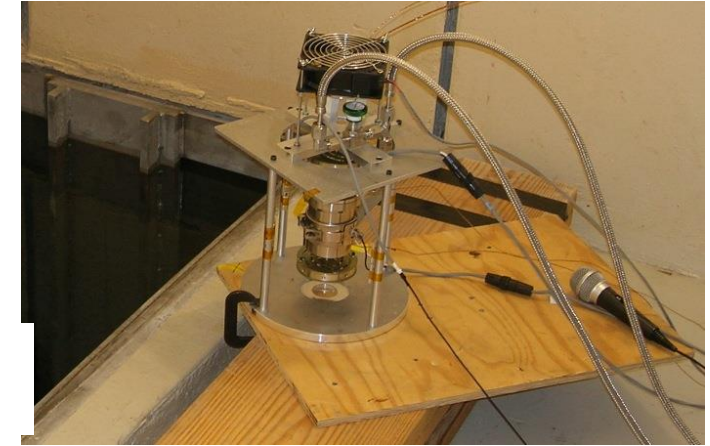
- Dose Rate: 90 rad/s, 850 rad/s
- Temp: 363K, 400K (90C, 125C)
- Electrically motored

## Performance comparison

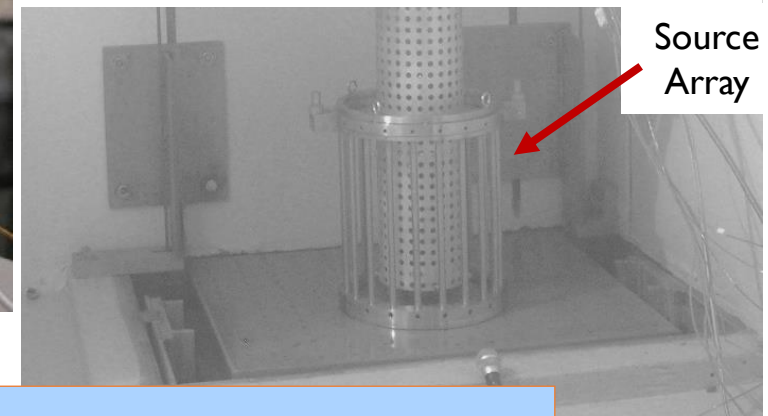
- Pre, Post irradiation & as a function of dose
- Operating conditions: P, i, V, S, Pr, T
- Electrical Integrity: Resistance, inductance, resistance-to-ground
- Waveform shape



## Low Dose Rate Configuration



## High dose rate configuration



18 irradiation steps up to ~40 Mrad

# Polymer Materials Radiation Hardness Summary

## Post-irradiation properties were typically within scatter of baseline

- O-ring exhibited post-irradiation dimensional change, but no loss in functionality based on leak testing and compression-set testing
- Two epoxy adhesives tested and experienced minimal degradation
- Kynar heat became brittle from temperature alone and irradiation

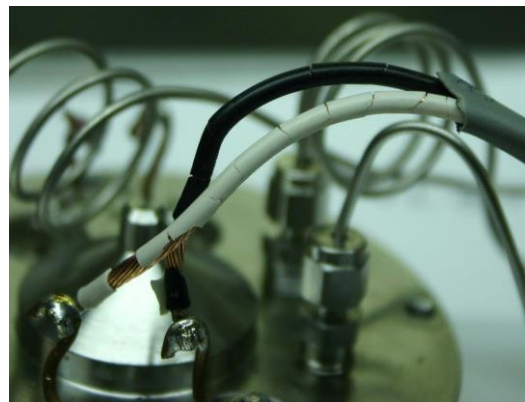
## Major exception was PTFE/Teflon coating for wires

- PTFE coating exhibited increasing damage in HFIR pool exposure
- Excessive damage after 40 MRad SARTA testing

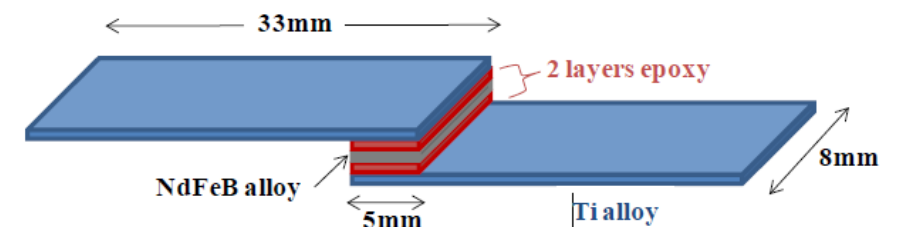
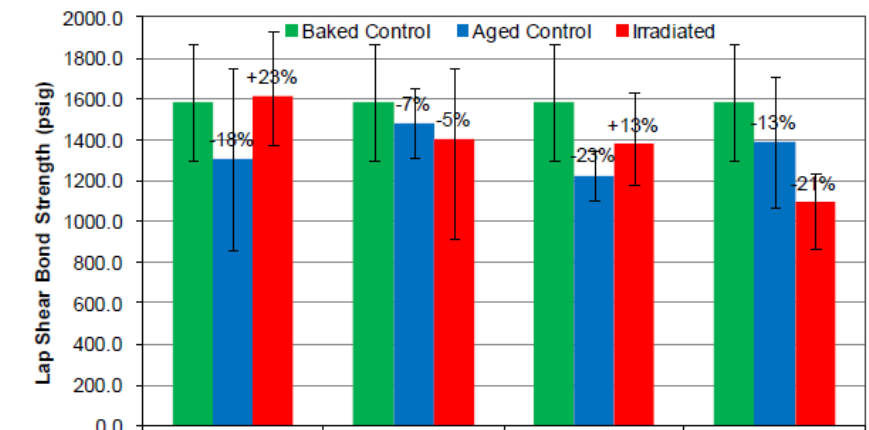
SARTA Kynar heat shrink post 40 MRad



SARTA PTFE post 40 MRad



Lap Shear for four TAMU conditions



# Superalloy Creep Testing

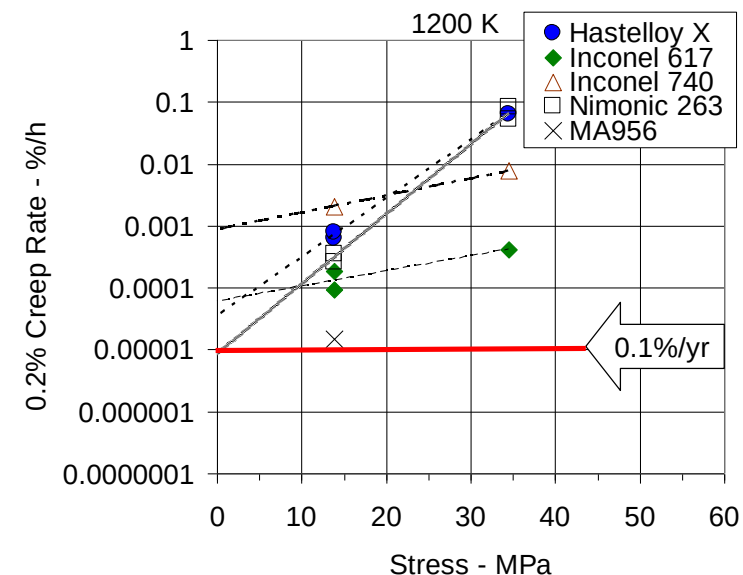
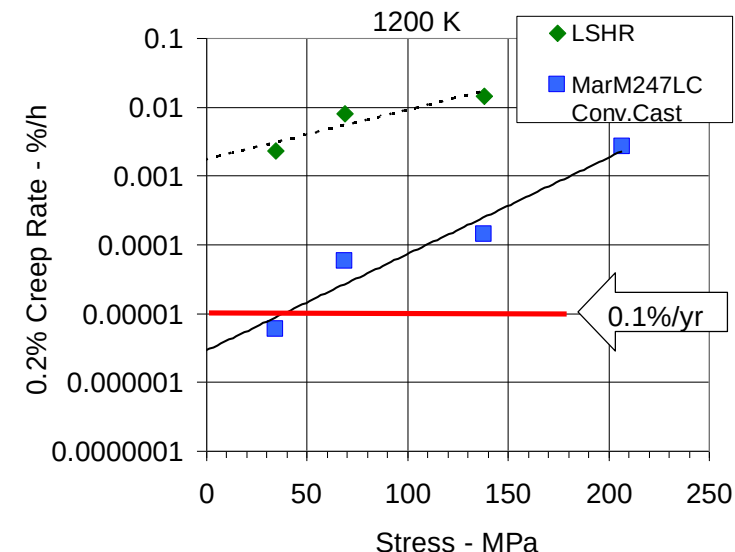
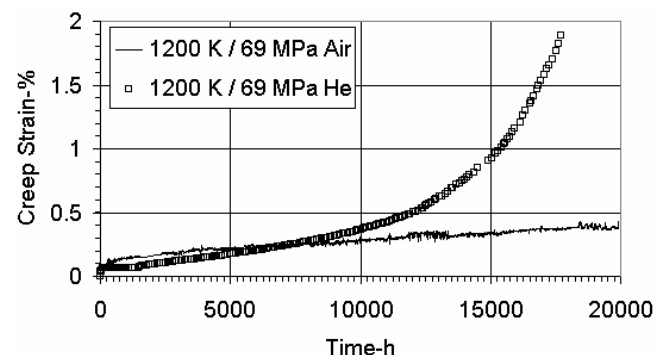
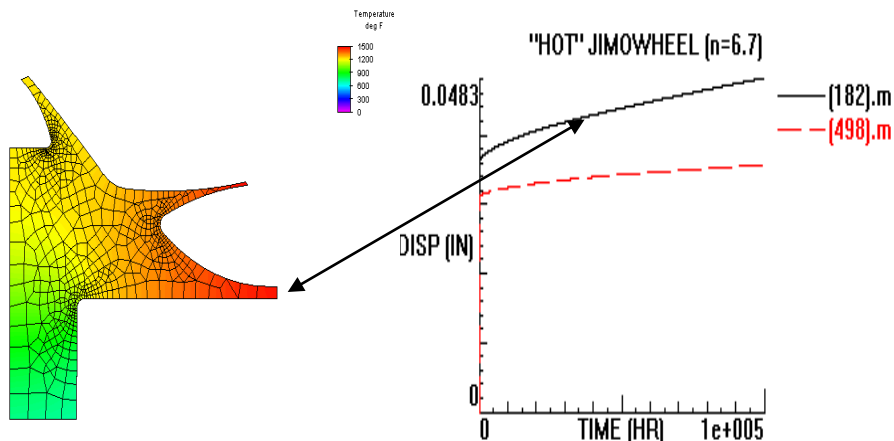


## Creep testing need for long mission lives

- Common testing is 1000 hrs which can be extrapolated to 10,000 hour lives
- Need at least 10,000 hr testing for 100,000 hr (10 yr) life

## Results

- Screened Five alloys at 1090 K and 1200 K
- MarM-247 predicted to have minimal creep in application
- Deleterious phases for some heritage alloys
- Testing in He indicates possible decrease in creep strength



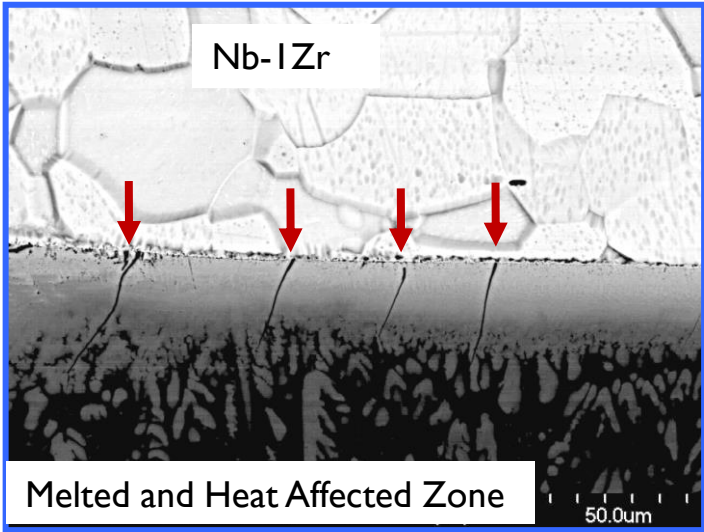
# Superalloy-Refractory Alloy Joining

## Prior work found welding problematic

- found 12  $\mu\text{m}$  limit for reaction thickness in welded heat affected zone

## Surveying alloy combinations and joining techniques

## Surveyed diffusion bonding, brazing, and long-term stability



### Superalloys

**IN617**

(55Ni-22Cr-12.5Co-**9Mo**-1Al-0.07C)

**HA230**

(59.8Ni-1.3Fe-21.6Cr-0.34Co-1.5Mo-**14W**)

### Refractory Alloys

**Mo-47Re**

**FS-85**

(60.2Nb-28Ta-11W-0.8Zr)

**Hastelloy X**

(46.3Ni-21.6Cr-18.9Fe-**8.9Mo**-1.9Co-0.085C-0.5Si)

**MarM247**

(59.9Ni-8.3Cr-10Co-**10W-3Ta**-5.5Al-1Ti-0.7Mo-1.5Hf)

**Ta**

**T111**

(90Ta-8W-2Hf)

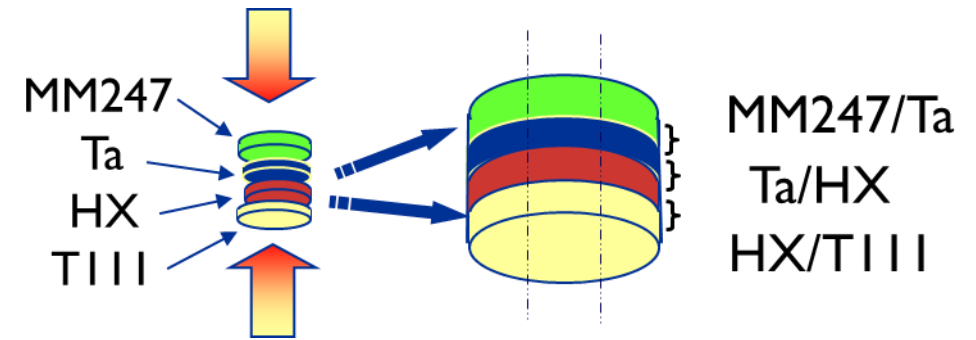
**ASTAR 811C**

(Ta-8W-1Re-0.7Hf-0.025C)

# Superalloy-Refractory Alloy Joining

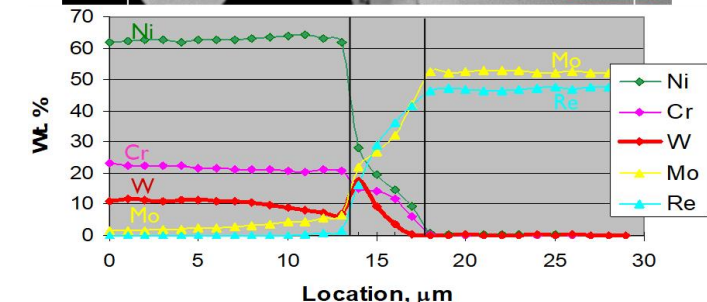
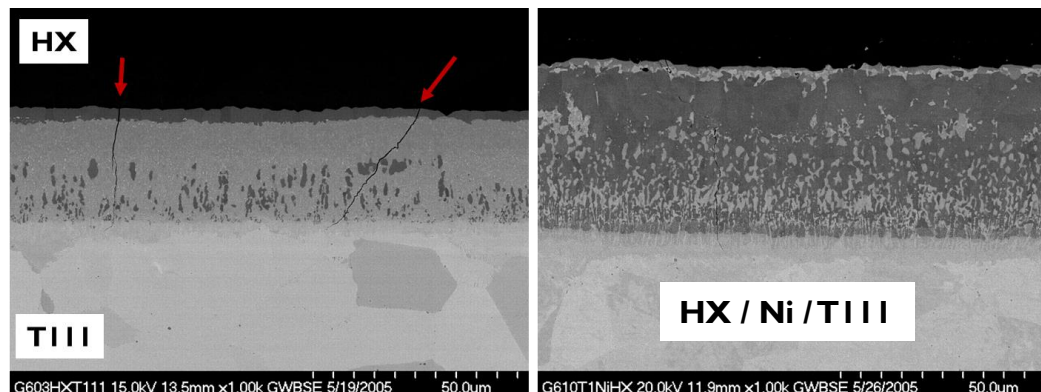
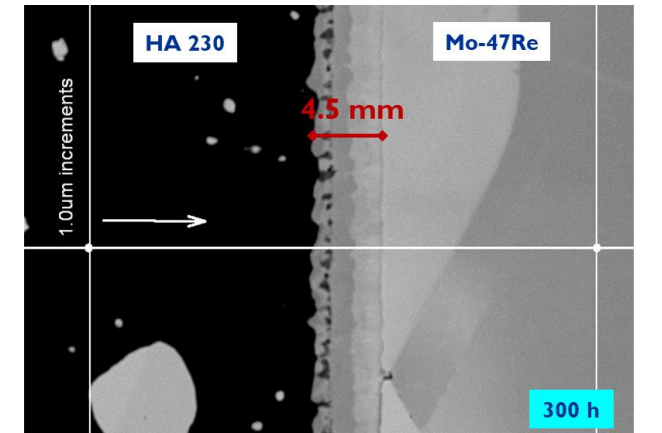
## Diffusion bonding or Hot Isostatic Pressing Equivalent

- Created bonds at 1150 and 1300 K
- With and Without Ni and Mo interlayers
- Studied interface reaction as a function of time



## Results

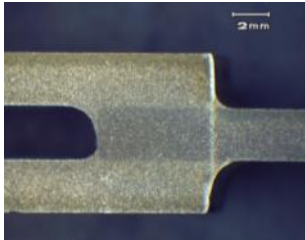
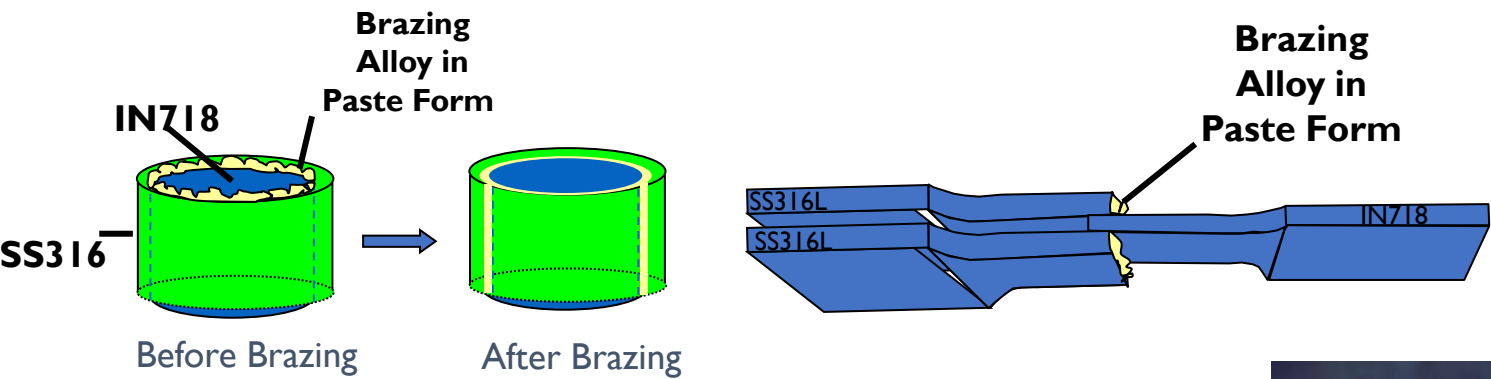
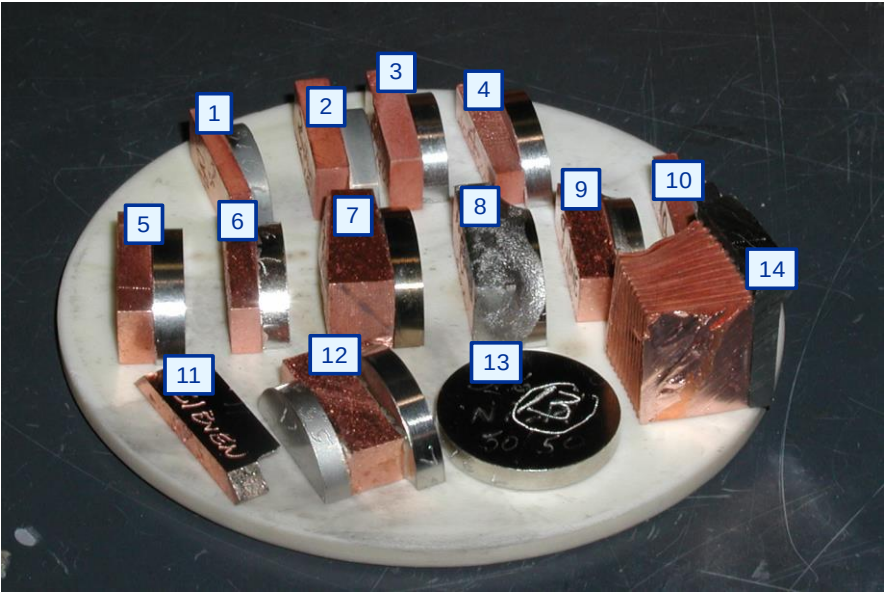
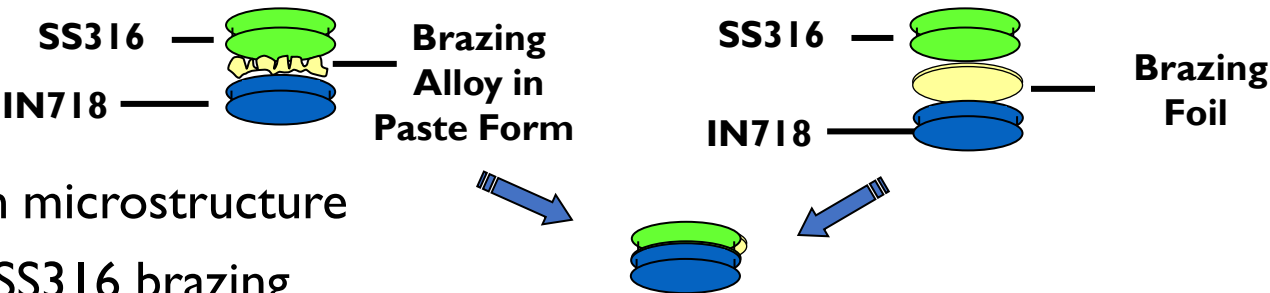
- Reaction zone width grows parabolically with time, limited or no porosity
- Mo-47Re with IN617 or HA 230 4x smaller reaction than with Ta or Nb alloys
- Lowest interdiffusion rate for HA230 / Mo47-Re couple
- Ni interlayer reduced deleterious phases and diffusion zone cracking



# Superalloy, Stainless Steel Cooper Brazing

## Brazing Studies from coupons to components

- Screened numerous Ni & Au based brazes
- Nioro (82Au, 18 Ni) had best balance of properties and processing window for SS316 to IN718
- Combined brazing with superalloy aging for optimum microstructure
- AMDRY 930 (Ni-22Mn-5Cu-7Si) selected for Cu to SS316 brazing



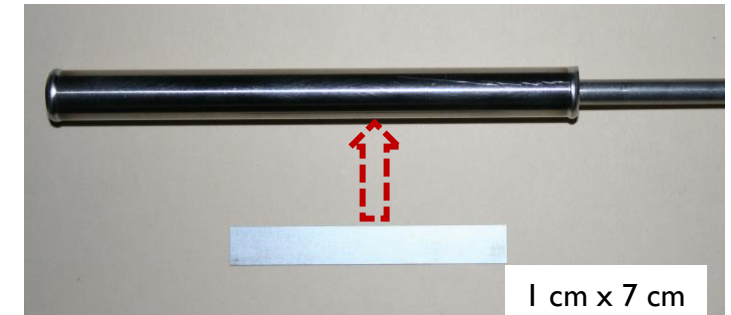
# Superalloy-Refractory Cross Contamination

## Multiple heritage studies expressed concern over refractory – superalloy contamination

- Trace elements in superalloys can be detrimental to refractory alloys, especially Nb alloys
- For example, Charlot, et al. “Corrosion of Superalloys and Refractory Metals in High Temperature Flowing Helium” 1967. Simulate proposed Advanced Test Reactor Gas Loop. Emphasized effects of multiple charges, multiple start-ups. Contaminant pick up in refractory alloys, but contaminant source not identified

## More controlled exposures attempted

- Individual Mo-47Re, ASTAR 811C (Ta-base alloy), and Nb-1Zr coupons encapsulated in Hastelloy X and high purity He
- Four conditions 1150 K and 1300 K for 500 and 1000 hours in a tube furnace under flowing argon; Only one sample per condition
- Mo-47Re had minimal C, O, N increase and insignificant change in room temperature tensile
- Nb-1Zr picked up more O than was available in HX and had significant ductility loss
- ASTAR had notable contaminant pick up and notable ductility loss



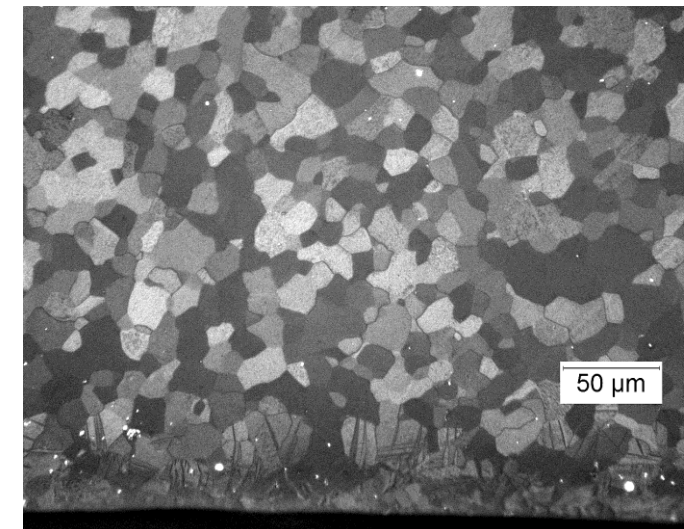
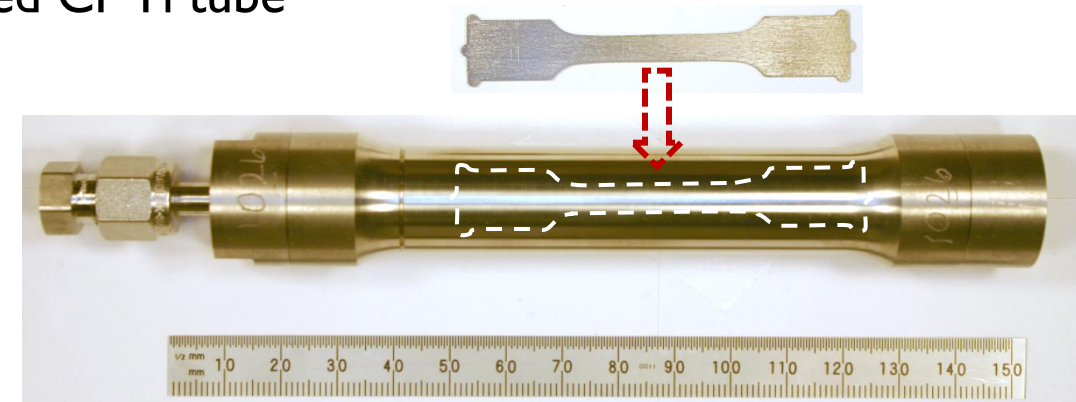
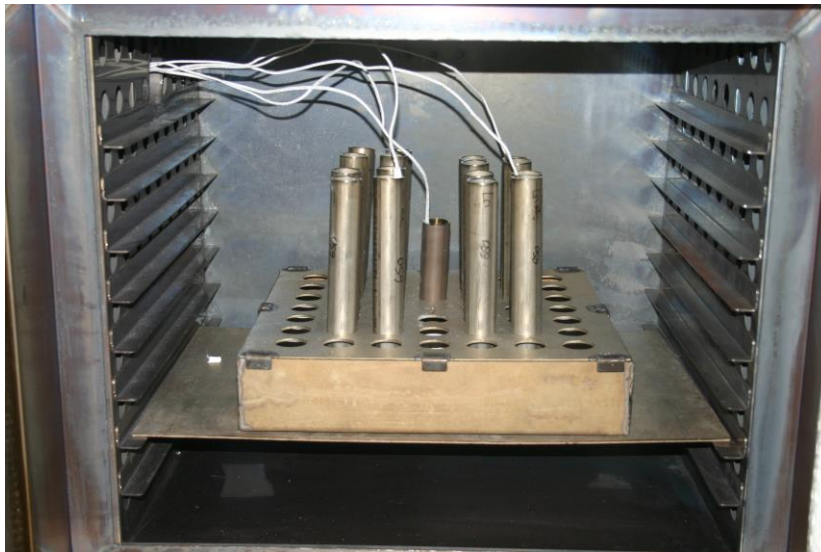
Still an open issue. Nb alloys are very sensitive to contamination.

# Na and NaK Compatibility

**Na and NaK Compatibility well established for some older alloys**

**Na/NaK/Titanium alloys compatibility not known**

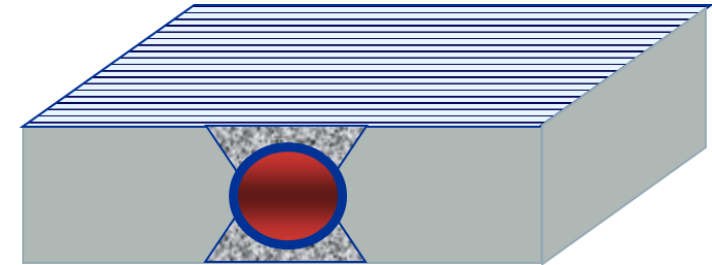
- Commercially pure Ti samples was immersed in NaK in sealed CP Ti tube
- Samples held at 550 and 650 K for up to 10,000 hrs
- Chemically, mechanically, and microstructurally evaluated
- **No significant changes**



# Radiator Panel Materials Selection

## Large Radiator Area—Mass and Performance Crucial

- Analysis showed that “saddle” region not chokepoint
- Polymer or Ceramic Matrix Composite significant mass savings
- CMC attractive for temperature capability, but poor ductility and joining are limiting factors
  - Cusil-ABA paste braze best joining to Ti
- PMC best mass, performance balance
  - Studied filler for increased through thickness conductivity



Increasing Temperature Capability →

|                | PMC    | Al Alloys   | CMC         |
|----------------|--------|-------------|-------------|
| Mass           | Green  | Yellow      | Green       |
| Database Size  | Yellow | Green       | Yellow      |
| Joining        | Yellow | Green       | Red Hatched |
| Tailorable CTE | Green  | Red Hatched | Red Hatched |
| Fabricability  | Yellow | Green       | Yellow      |
| Stiffness      | Green  | Yellow      | Green       |
| Ductility      | Yellow | Green       | Red Hatched |



# Materials Research and Testing Lessons Learned

## **Start with a Materials/Process Qualification Mindset**

- Strive for reliability rather than performance whenever possible
- Let system engineering guide decisions for enhanced performance
- “Buy” new technology/component/material judiciously and budget for necessary qualification

NASA and Other Agencies have enabling technology programs; targeted component materials research should be in those programs, not in flight programs

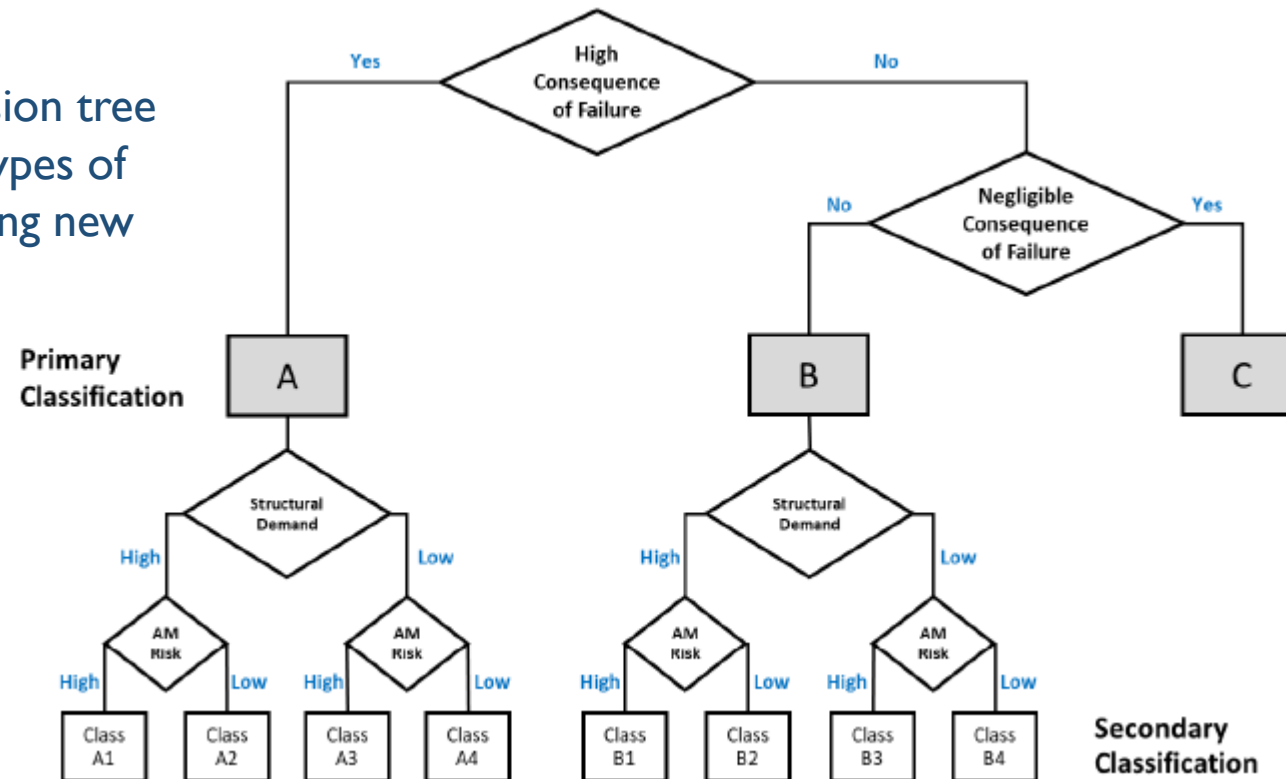
**However...**

# Qualification Guidelines Exist for New Materials and Processes

## In 2021 NASA Office of Chief Engineer approved NASA-STD-6030 ADDITIVE MANUFACTURING REQUIREMENTS FOR SPACEFLIGHT SYSTEMS

- Provides a blueprint for approving radical new processes and new alloys
- Methodology shared and favorably received by GIF-AMME-TF Workshop (International Workshop on Adv Materials & Manufacturing for Generation IV Reactors)

Consequence of Failure decision tree establishes the amount and types of test data required for qualifying new material or process





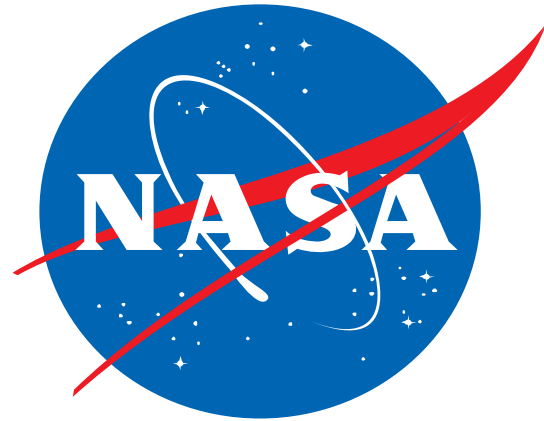
# Acknowledgements

## **These are primary contributors:**

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