LANA.75 Hydride Bed Life Extension – Background, Test Plan, and Status

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LaNi\textsubscript{4.25}Al\textsubscript{0.75} or LANA.75 has been used by the Savannah River Site Tritium Facilities for years to safely store hydrogen isotopes

- Higher molar density at moderate pressures = less glovebox space (approximately 1000x under 1 atmosphere and ambient temperature)
- Can be used as a pump by varying temperature - fewer moving parts
- Can deliver He-3 free gas – fewer unit operations

**Bed designs have evolved over the years**

- Initial designs relied on hot and cold nitrogen for thermal swings and bed jacket gas flow for in bed accountability (IBA)
- Later beds incorporated electric heaters, Al foam for heat transfer, and a U tube for IBA
- Future beds will be shorter and thicker
Tritium Aging of LANA

- LANA materials are limited lifetime components due to Decay of tritium to He-3 within the metal matrix
- Tritium aging effects on isotherms
  - Formation of “heel"
    - Inventory Holdup
    - Reduced Capacity
  - Decreased plateau pressure
  - Eventual loss of plateau
  - Eventual weeping of He-3
Bed Replacement

- Procure new beds and hydride material
- Verify new hydride performance
- Activate new beds to remove oxides
- Perform a series of isotope exchanges to remove as much tritium as practical from old beds
- Backfill old beds with inert gas
- Enter Open Glovebox Maintenance to replace beds
- Perform IBA calibration on new beds

But... there are disposal issues–

- 2010 Aoki Memorandum
  - Implies He-3 > 2 liters are to be saved
  - Retired beds contain >> 2 liters He-3
- Waste Acceptance Criteria
  - No potential for pressure generation >1.5 atm
  - No potential for explosive mixtures with water
Testing of He-3 Release from Tritium-Aged LANA at Elevated Temperatures

- Legacy LANA sample was isotope exchanged to < 1 Ci T₂/g
- Sample was passivated with air prior to recovery from legacy test cell
- Sample was transferred from SRTE to SRNL for TGA/MS testing under argon
- TGA temperature calibrated using melt points of several metals
- TGA balance calibration verified using weights
- MS calibrated using surrogates fed into the TGA purge stream
  - He-4 for He-3
  - D₂ for all hydrogen isotopes
Testing of He-3 Release - Results
Controlled Oxidation Testing of Tritium-Aged LANA at Elevated Temperatures

- Objective was to develop one step processing to recover gas and passivate the LANA particulate
- TGA/MS testing performed using dilute oxygen/balance argon stream
- TGA/MS testing repeated in argon only for comparison
- Involved heating a sample to temperature, holding for an hour, then proceeding to a maximum of 1000 °C
- Based on previous testing, XRD, TEM, and SEM testing was performed on samples held at selected temperatures
- While dilute oxygen provided a negligible advantage over pure argon, XRD results were enlightening
Controlled Oxidation Testing – XRD Results of Argon Testing

XRD LANA.75 as Received

XRD LANA.75 heated in Argon to 400°C

XRD LANA.75 heated in Argon to 600°C

XRD LANA.75 heated in Argon to 1000°C
The “Ah-Ha” Moment

If crystallinity is restored, isotherm performance should also be restored. It may be possible to design regenerable LANA hydride beds with several advantages over existing designs:

- Reduce or eliminate the need for bed change outs
- Reduce tritium process holdup
- Allow in situ He-3 recovery

= Significant cost savings to SRTE!
LANA Restoration Test Plan Development

Hydride bed temperature limits are based on materials of construction

- 316L filter media 482 °C
- Aluminum heat transfer foam ~650 °C
- Inconel filter media 815 °C
- Copper heat transfer foam >1050 °C

Test Plan

- Recover/split tritium-aged LANA sample
- Transfer ~4g to new high-temperature test cell for testing on manifold
- Send ~1g to SRNL for TGA/XRD testing at same temperatures as manifold testing
- Reactivate hydride/perform heel exchanges on hydride in test cell
- Collect baseline isotherm at 120 °C
- Heat under vacuum to 450 °C
- Collect isotherm at 120 °C
- Heat under vacuum to 600 °C
- Collect isotherm at 120 °C
- Heat under vacuum to 750 °C
- If isotherm performance is restored, reload sample with tritium to track aging effects
High Temperature Test Cell Design

Pressure protection

- Cell must be able to withstand loading at ambient temperature to the manifold rupture disc pressure (219 psia), isolating the test cell, and heating the test cell to 800 °C
- Cell cannot deliver more than 670 psia to the manifold

Thermal Concerns

- Operating: No portion of the cell (including filter gasket, valves, tubing) can exceed the design temperature under any circumstances
- Industrial Hygiene: Outer surfaces must be less than 60 °C
High Temperature Test Cell Final Product

- Zone 1 - Inconel cell and tubing to VCR weld gland 800 °C and 600 psig
- Zone 2 – VCR weld gland to VCR fitting 450 °C and 600 psig
- Zone 3 – Above VCR fitting 300 °C and 600 psig
Sample Selection

LANA75-D2

• 4.99 g sample
• 1987 - loaded with T₂, isotherm, reloaded with T₂
• 1995 – head space sampled, isotherm, cycled 21x, isotherm, loaded with D₂, cycled 5x, isotherm x2, D₂ exchanged 4x, T₂ exchanged 3x, isotherm, reloaded with T₂
• 2000 – head space sampled
• 2002 – head space sampled
• 2005 – multiple D₂ exchanges, sample passivated for recovery
## Current Status and Future Plans

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<th>Location</th>
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<tr>
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<td>Reactivate sample/ perform heel exchange</td>
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<td>Bake out at 450 °C</td>
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Questions?
Fragen?
Preguntas?