Contract No:

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Thermal Stability Testing of LANA.75

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Outline

LANA Introduction

Historically relevant work

- He-3 Release
- Controlled Oxidation

Thermal Stability Testing

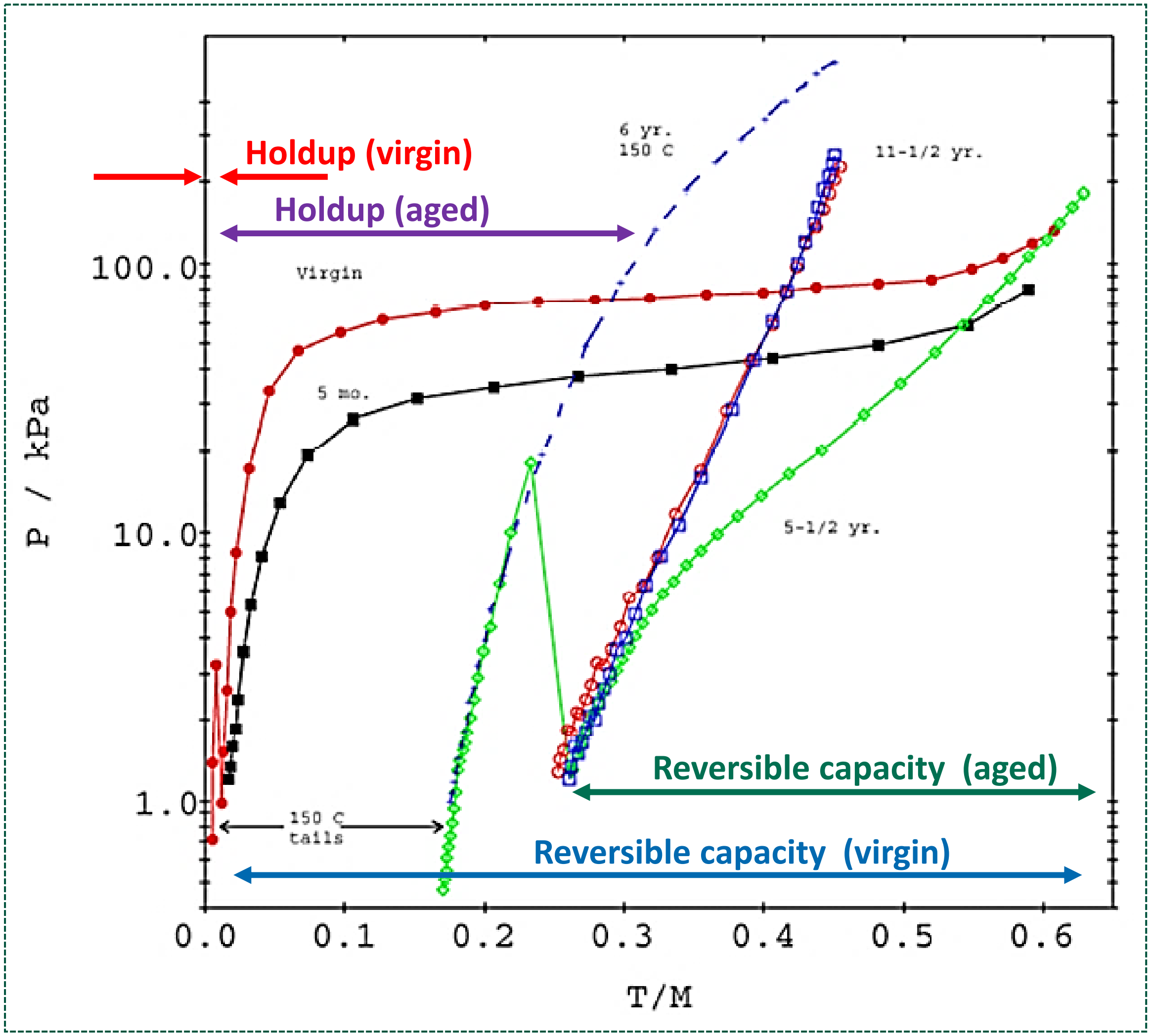
Path Forward

Acknowledgements

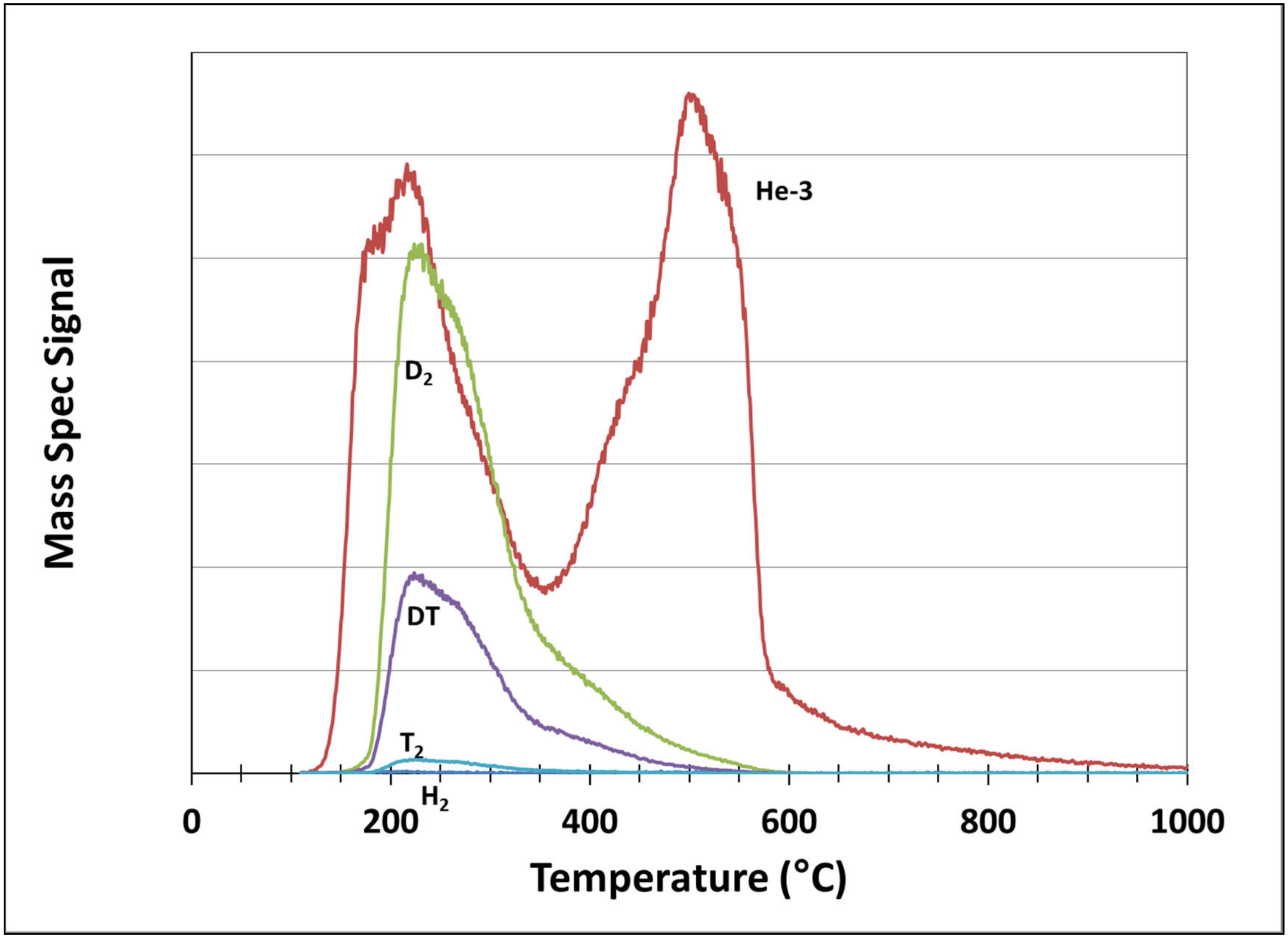
Tritium Aging of $LaAl_{4.25}Al_{0.75}$ (LANA.75)

- **LANA beds are limited lifetime components due to decay of tritium to He-3 within the metal matrix**

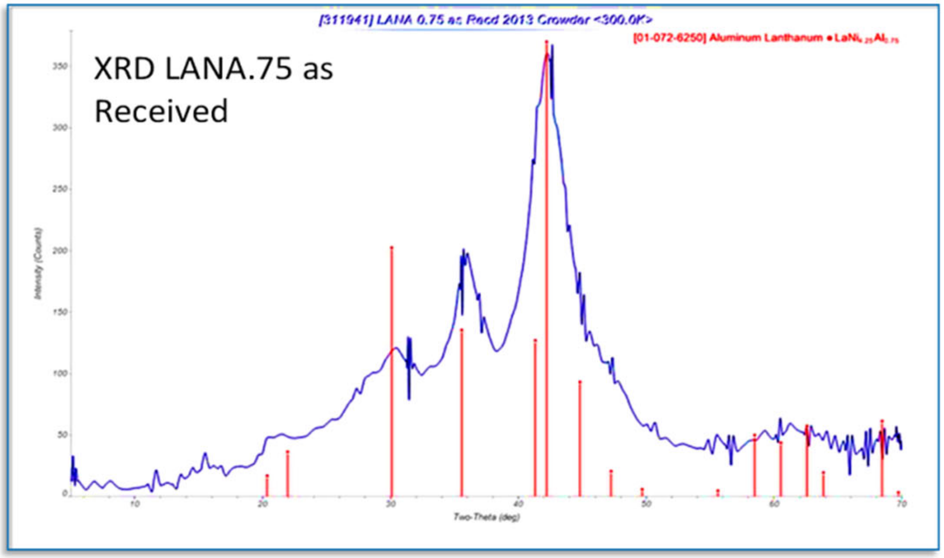
- **Tritium aging effects on isotherms**
  - Formation of “heel”
    - *Inventory hold-up*
    - *Reduced capacity*
  - Decreased plateau pressure
  - Eventual loss of plateau
  - Eventual weeping of He-3

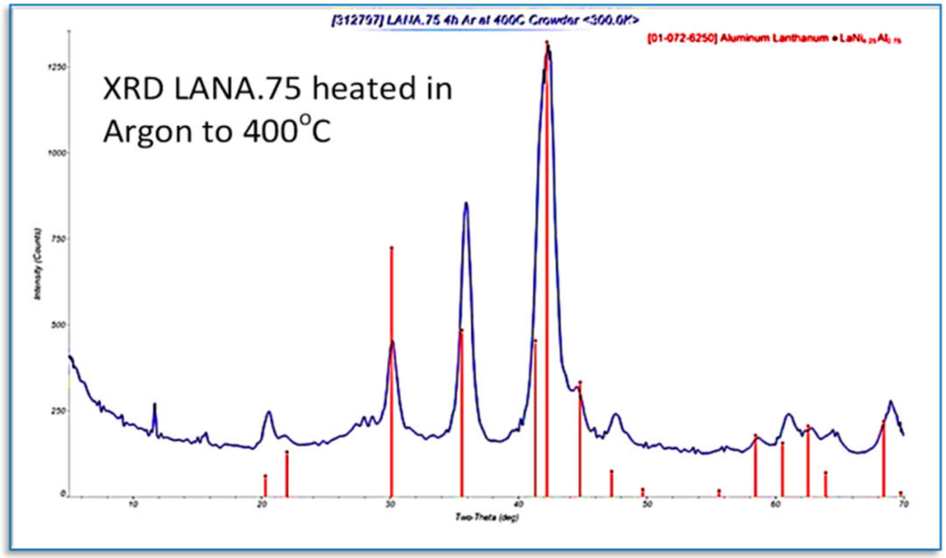


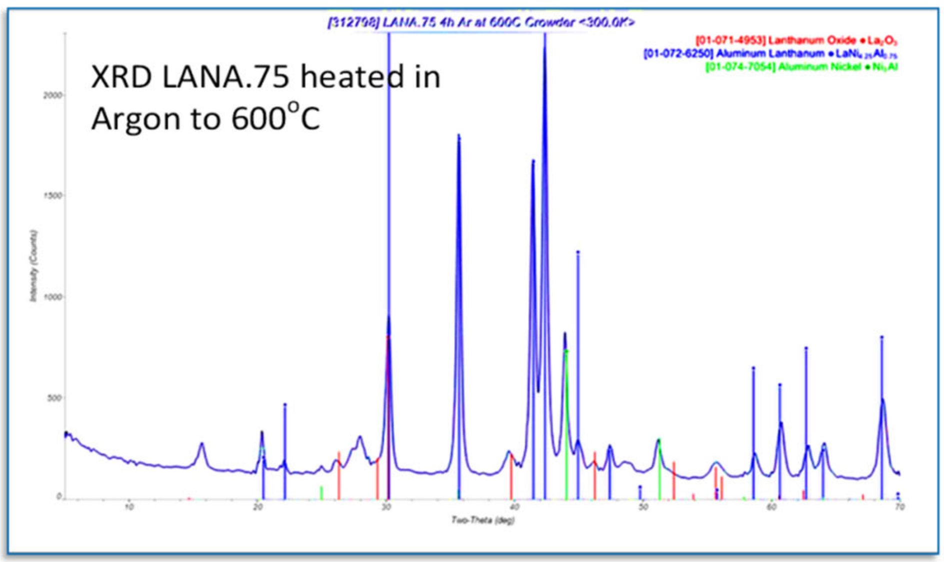
Thermal Release of He-3 from Tritium-Aged LANA

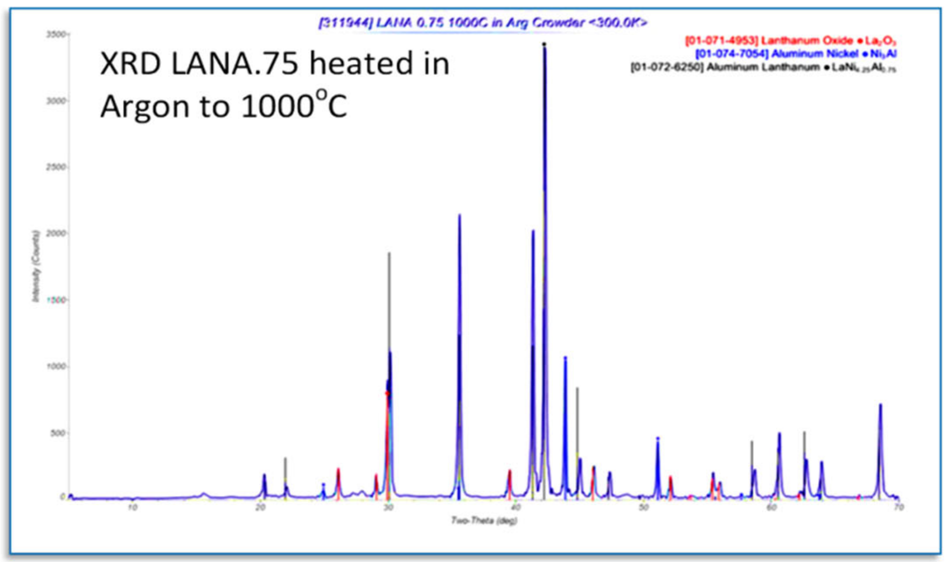


Controlled Oxidation of Tritium-Aged LANA – XRD Results of Argon Testing

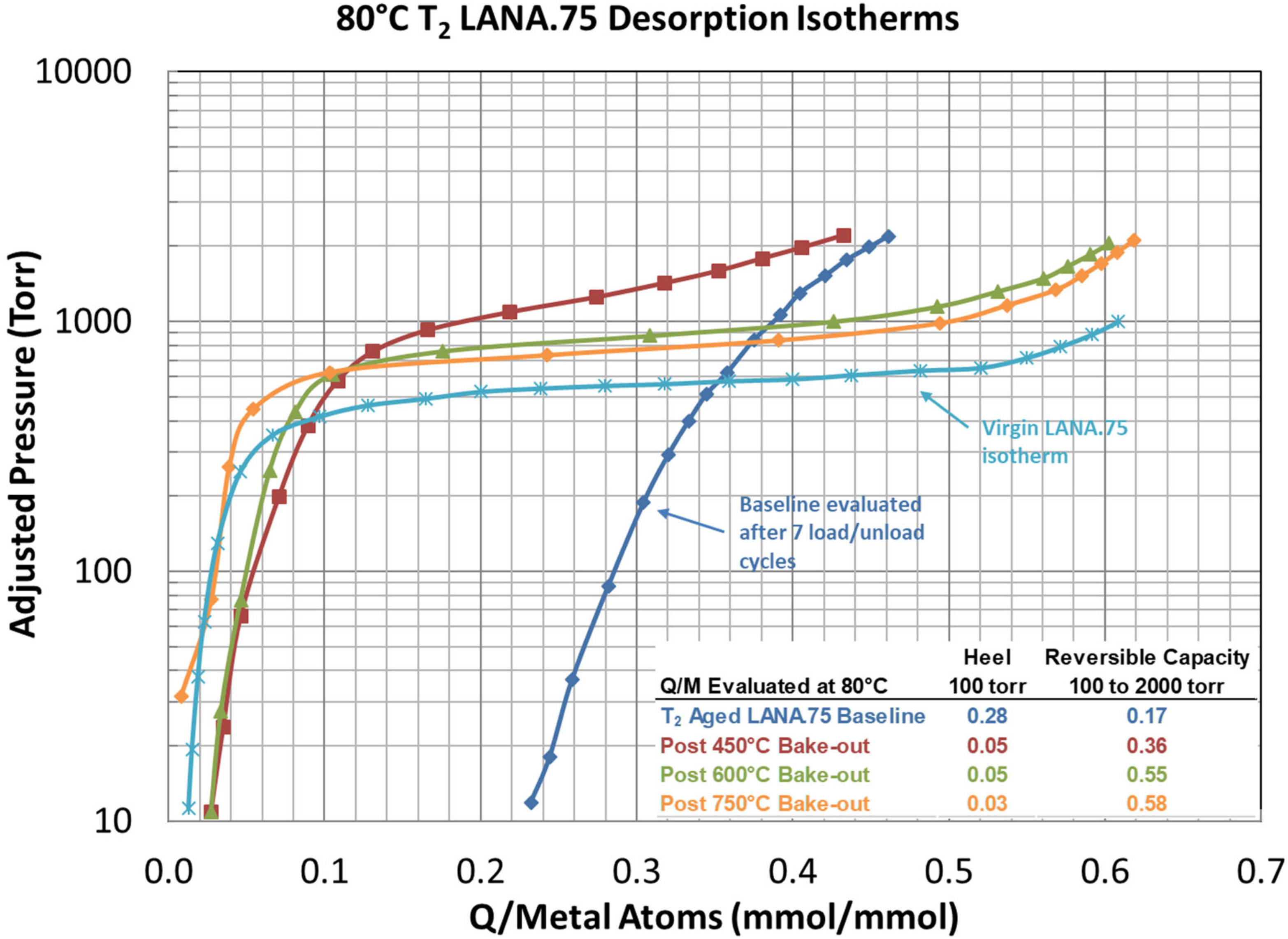








Isotherm Results



Q/M Evaluated at 80°C	Heel 100 torr	Reversible Capacity 100 to 2000 torr
T_2 Aged LANA.75 Baseline	0.28	0.17
Post 450°C Bake-out	0.05	0.36
Post 600°C Bake-out	0.05	0.55
Post 750°C Bake-out	0.03	0.58

Potential Impacts

If crystallinity is restored, isotherm performance should also be restored.

It may be possible to design LANA beds that can be regenerated in place.

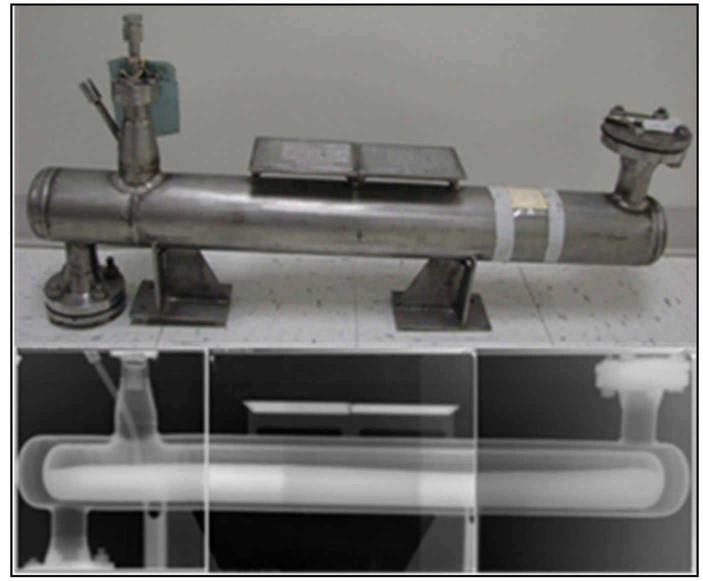
Assume:

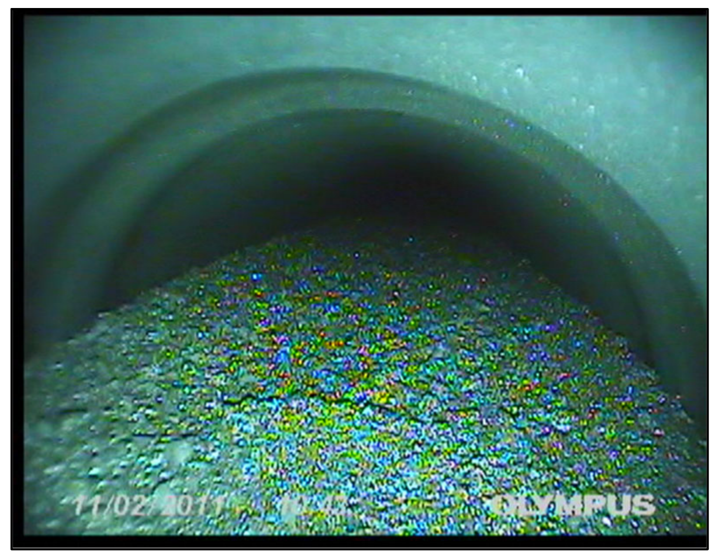
- **50-year facility life**
- **~10 years/bed life**
- **X beds in the facility**

	Beds Replaced			
Year	**No Regeneration**	**1 Regeneration**	**2 Regenerations**	**4 Regenerations**
10	X	-	-	-
20	X	X	-	-
30	X	-	X	-
40	X	X	-	-
Total New Beds	**4X**	**2X**	**X**	**0**

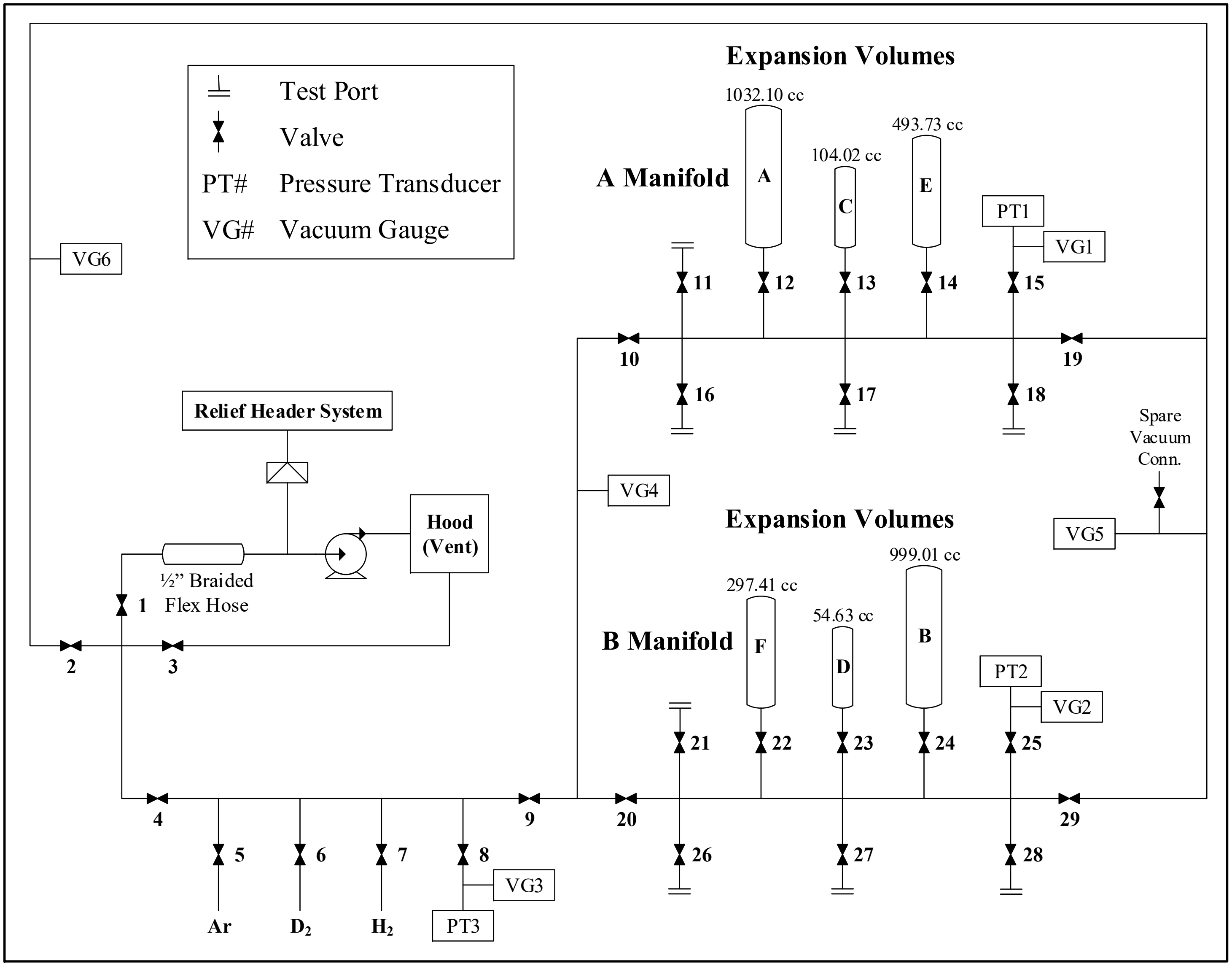
Regenerated LANA.75 Summary

- **Crystallinity restored as measured by XRD**
- **Released significant fraction of He-3 trapped in the metal**
- **Reduced/eliminated "heel" of hydrogen trapped in the metal**
- **Restored reversible capacity of the hydride**
- **Exhibited a higher plateau pressure than a "virgin" sample**
  - Differences in sample heating?
  - Loss of aluminum?





Thermal Stability Test System



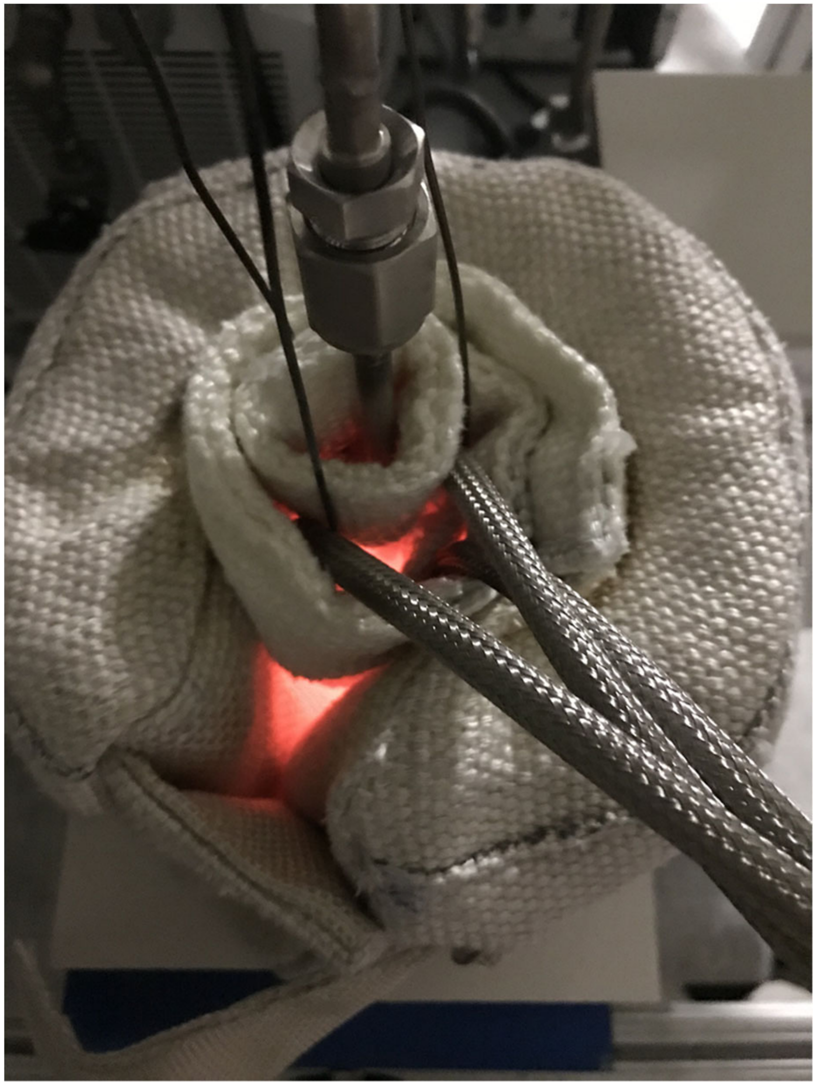
Thermal Stability Test Plan

Load sample to test cell (~5 g)

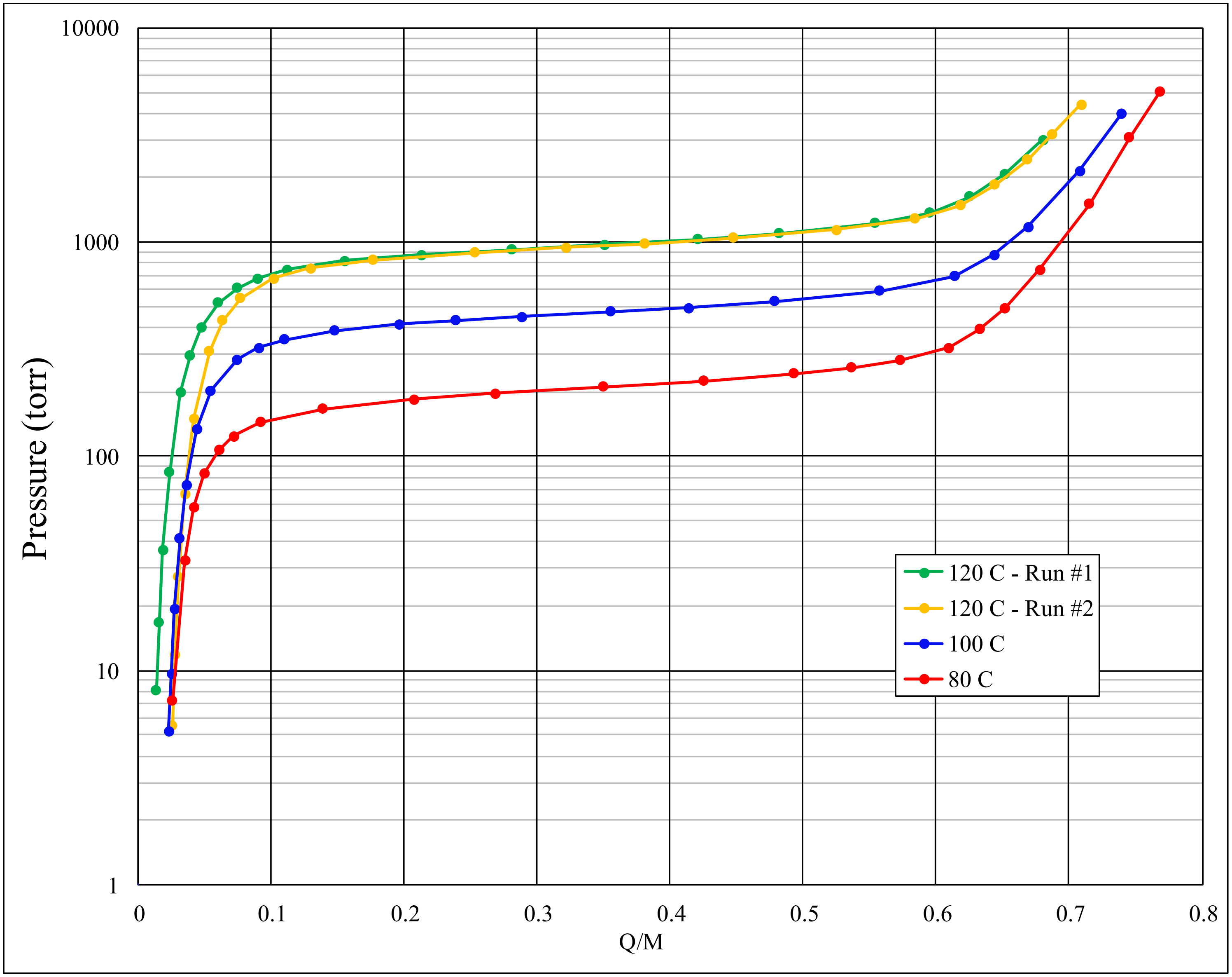
Activate hydride/perform heel exchanges on hydride in test cell at 80 °C

Test Activities

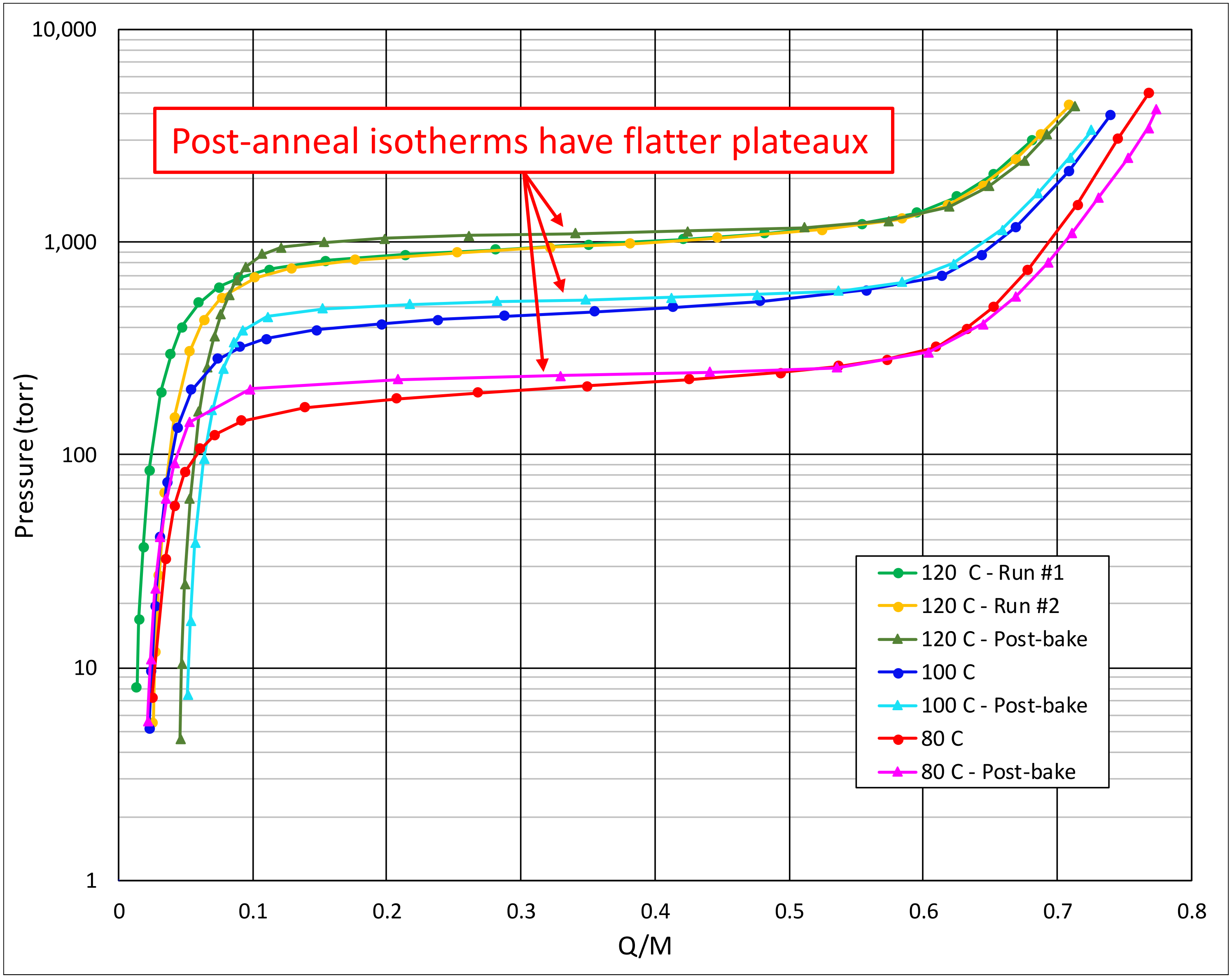
- *Collect verification isotherm at 80 °C*
- *Recover pre-anneal material for analytical testing (~1.5 g)*
- *Collect pre-anneal isotherms at 80, 100, and 120 °C*
- *Anneal under vacuum at 750°C for 200 hours*
- *Collect post-anneal isotherms at 80, 100, and 120 °C*
- *Recover post-anneal material for analytical testing – Particle Size, ICP, XRD, SEM (~1.5 g)*



Thermal Stability Testing – Pre-Anneal Isotherms

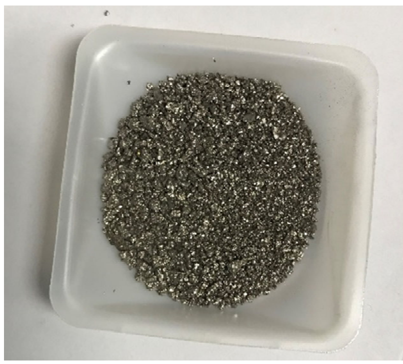


Thermal Stability Testing – Post-Anneal Isotherms



Thermal Stability Testing – Particle Size Analysis and ICP-ES Results

	MV (μm)	MA (μm)	MN (μm)	σ (μm)
Virgin Material	548.6	474.6	148.5	120.1
Pre-Anneal Material	17.98	14.45	9.04	8.17
Post-Anneal Material	21.37	18.83	13.42	6.97



Virgin Material

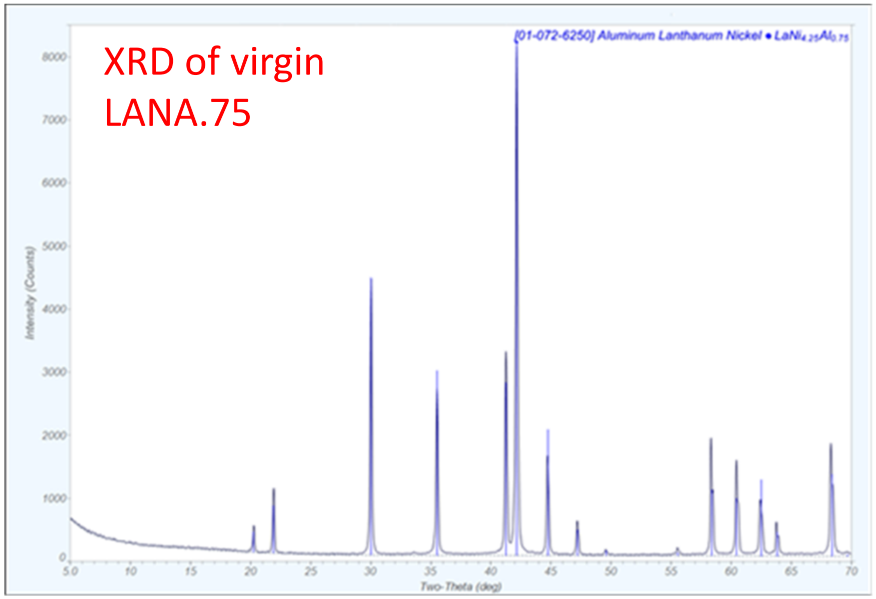
Post-Anneal Material

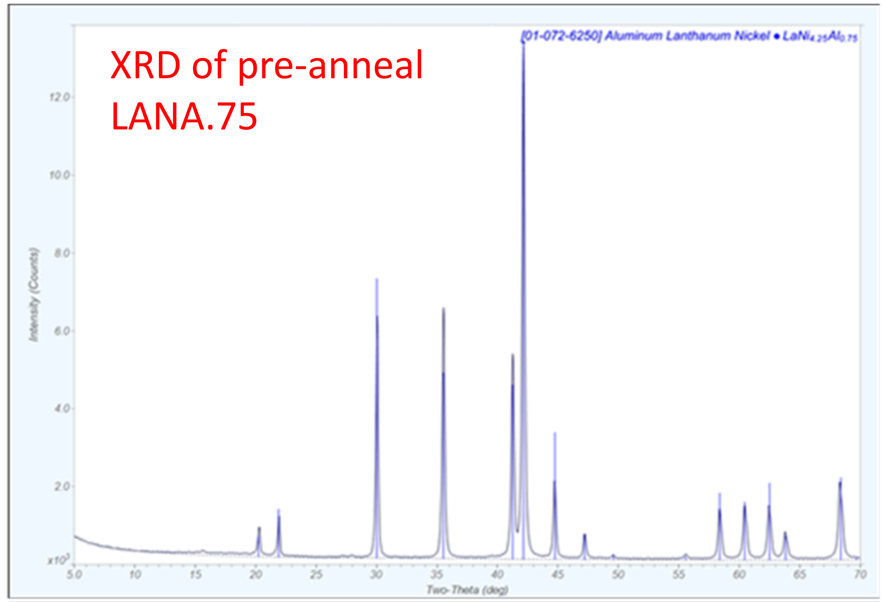
	Composition
Virgin Material	$La_{1.00}Ni_{4.21}Al_{0.77}$
Pre-anneal Material	$La_{1.00}Ni_{4.20}Al_{0.74}$
Post-anneal Material	$La_{1.00}Ni_{4.24}Al_{0.72}$

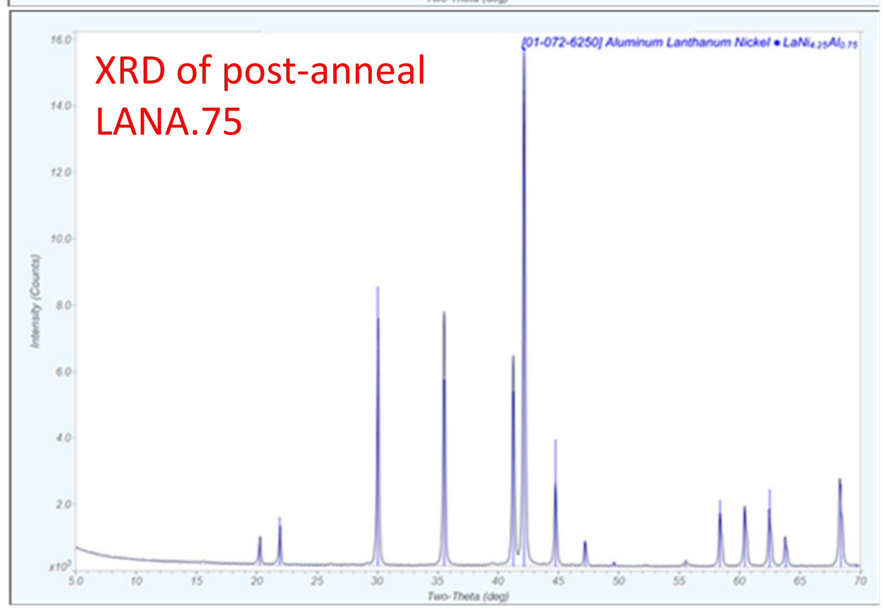
No loss of Al

* Ca and Mg also detected above instrument detection limits, but were 3-5 orders of magnitude lower than La, Ni, or Al.

Thermal Stability Testing – XRD Results

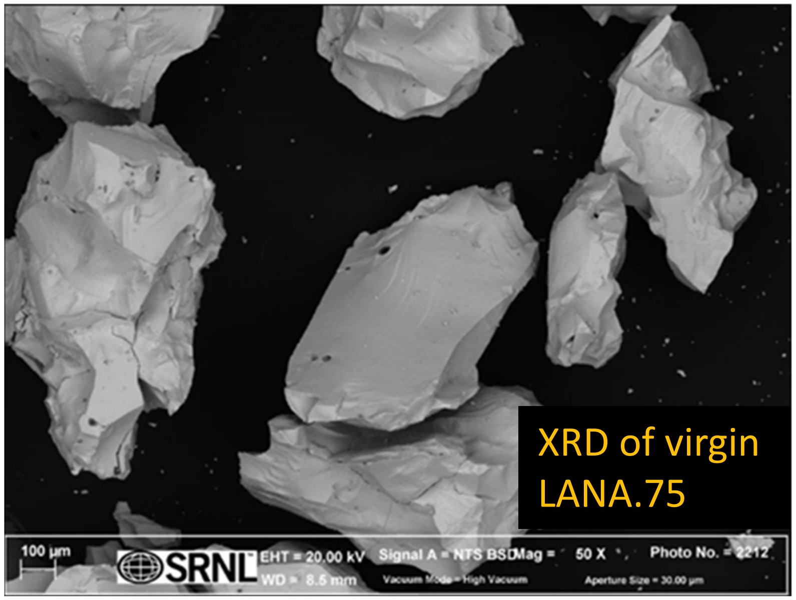


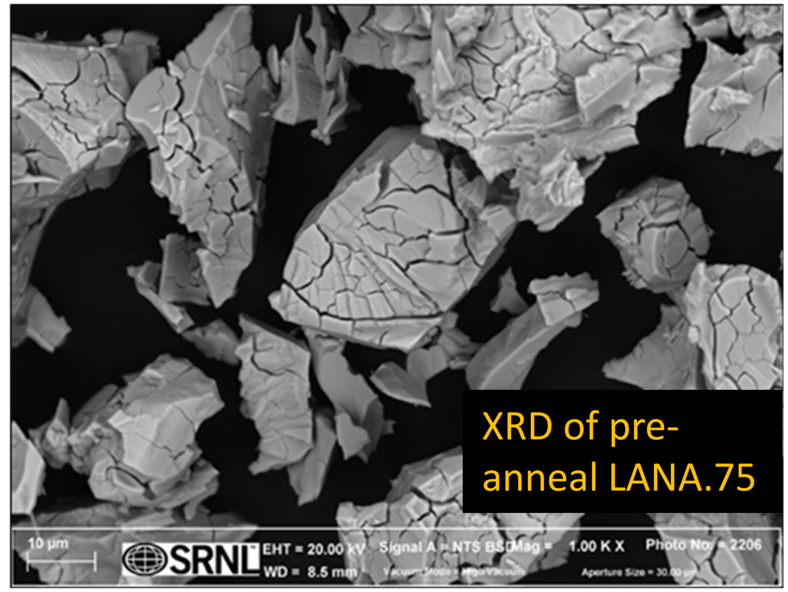


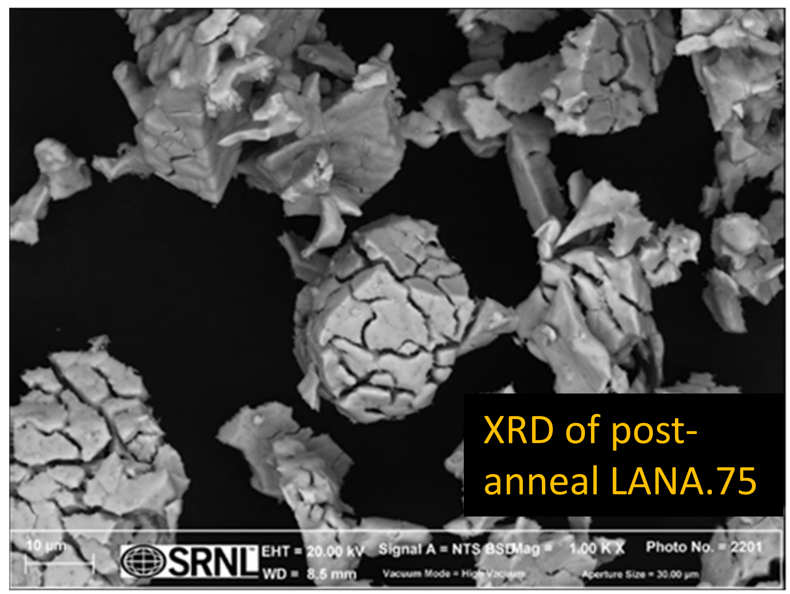


- Little to no contamination of the material
- Heating the material does not change the crystalline structure

Thermal Stability Testing – SEM Results

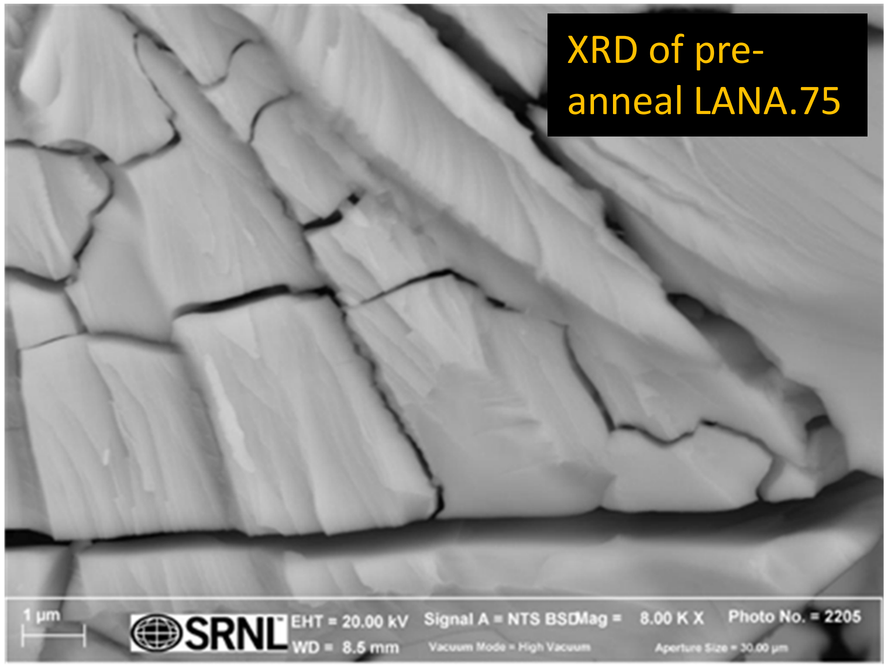


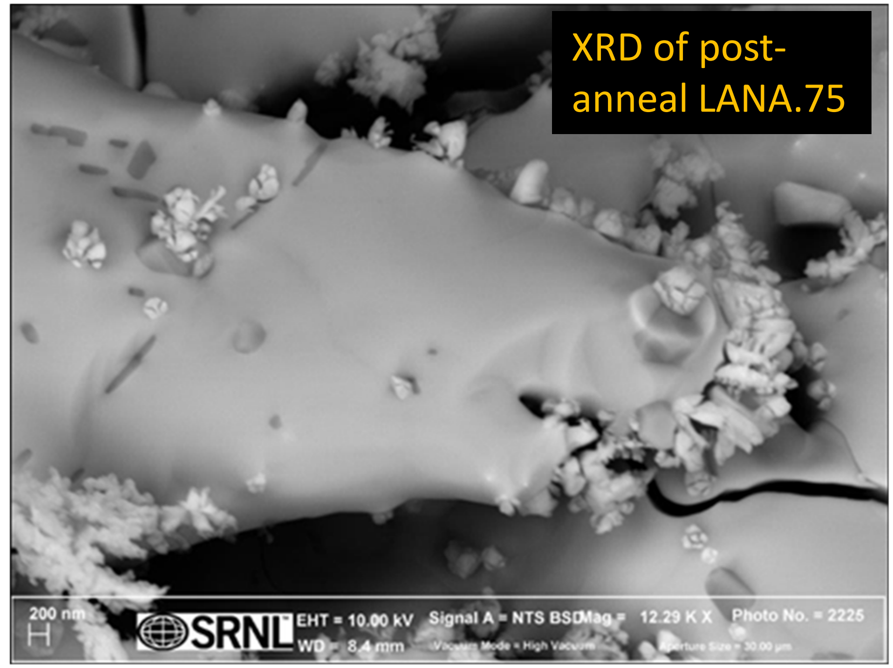




- Cracks form when virgin material is exposed to hydrogen due to decrepitation
- Unusual growths appeared on the post-anneal material

Thermal Stability Testing – Growths on Post-Anneal Material





EDS performed on all materials

- Flat surfaces show La, Ni, and Al as chemical composition
- Growths on virgin and post-anneal materials show excess oxygen
- Growths are oxides due to passivation of pre-anneal material?

Conclusions / Path Forward

Conclusions

– Pre-anneal material and post-anneal material isotherms have similar shapes, but the plateau region of the post-anneal material is flatter

– Particle size analysis showed that the pre-anneal and post-anneal material had similar sizes

– ICP analysis showed no significant changes in the elemental composition

– XRD analysis showed that there was no change in the crystalline structure due to heating

– SEM analysis showed growths on the post-anneal material

Path Forward

– Additional testing on the material with no passivation or recovery of sample prior to the annealing process

Acknowledgements

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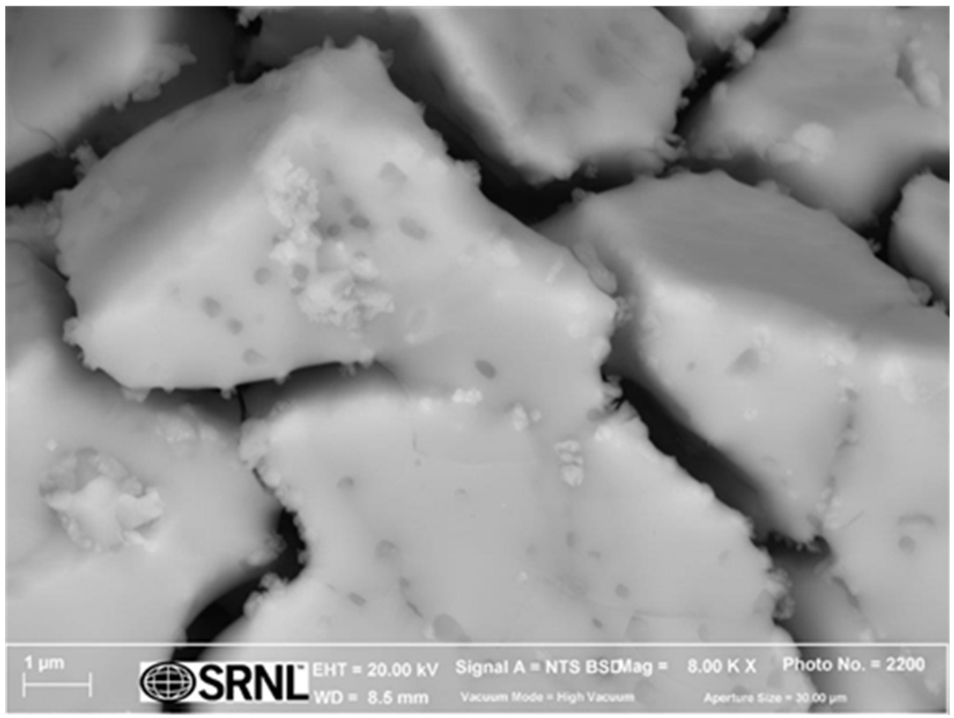

Thank you for your attention

Questions?