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Quantification of Tritium "Heels" and Isotope Exchange Mechanisms in La-Ni-Al Tritides

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DOE Contract No. DE-AC09-89SR18035

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WSRC-TR-92-368

July 27, 1992

**QUANTIFICATION OF TRITIUM "HEELS" AND ISOTOPE EXCHANGE
MECHANISMS IN La-Ni-Al TRITIDES (U)**

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Classification: Unclassified

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INTRODUCTION

For the last several years, processes utilizing metal hydride technology have been successfully used in the SRS Tritium Facilities.^{1,2} The 233-H Replacement Tritium Facility, which is currently in start-up phase and which will be on-line in 1993, utilizes this technology extensively. In addition, hydride technology is used for loading and purification operations in the current Tritium Production operations in buildings 232-H and 234-H. Metal hydrides are used in the Tritium Facilities for product pumping and storage, recovery storage and pumping, isotope separation, compression, and purification of hydrogen isotopes (Flow through Beds). The use of metal hydrides is also being investigated for use in the Plug Flow Reverser (PFR) for an advanced TCAP isotope separation system. The use of metal hydride technology has many advantages over conventional technology including (1) safe, low pressure, high density storage of tritium and (2) low maintenance pumps and compressors in which the only mechanical working parts are valves.

Tritium aging and compatibility studies have been underway for several years in the Materials Test Facility to determine the effects of tritium on these new materials.³⁻⁹ The La-Ni-Al intermetallic materials used at SRS are based on LaNi_5 in which aluminum has been substituted for nickel to produce a compound having a general formula $\text{LaNi}_{5-x}\text{Al}_x$, where $x = 0-1$ (LANA.x). Aluminum substitution improves the stability of the compound and lowers the equilibrium isotherm pressure, allowing the formulation of alloys which exhibit a range of pressures over a given temperature range. LANA materials used for tritium processing retain helium-3 which is born in the metal lattice through radiolytic decay of tritium. The insoluble helium-3 strains the metal lattice and changes the thermodynamics of the metal-hydrogen system, leading to (1) decreases in the isotherm plateau pressure, (2) increases in the isotherm plateau slope, and (3) decreases in the reversible hydriding capacity of the materials. The latter includes the retention of some hydrogen isotope which cannot be readily removed by vacuum desorption, referred to as the tritium "heel." For continued operations, it is necessary to heat tritium aged beds to a higher temperature to achieve a constant desired operating pressure.

This study seeks to examine the phenomenon of the tritium heel in La-Ni-Al tritides. An attempt to quantify the formation will be made as well as looking at methods for recovering the bound tritium from LANA materials.

SUMMARY

Tritium aging studies have shown that $\text{LaNi}_{5-x}\text{Al}_x$, where $x = 0-1$, (LANA_x) metal tritides lose capacity with time due to the formation of irreversible bound tritium or "heel." By definition, the tritium heel cannot be removed by simple desorption under normal processing conditions. The formation and gradual increase of the heel with exposure time decreases the storage or pumping capacity of a $\text{LaNi}_{5-x}\text{Al}_x$ bed, until its performance is no longer acceptable and it must be replaced. Although the tritium heel cannot be removed by simple heating and desorption, which is the normal processing operation for removal of gas from a metal tritide bed, the tritium contained in the heel can be successfully removed to very low levels by exchange with deuterium.

BACKGROUND

Metal hydride based tritium storage and handling systems have been of interest to the DOE Complex for many years. One of the remarkable properties of many metal tritides is the retention of decay helium-3 in the metal lattice. This property varies depending on the material. For instance, in uranium tritide helium-3 is released from the solid at the generation rate within one year.¹⁰ On the other hand, palladium tritide retains the vast majority of the decay helium for 12-18 years before significant helium release occurs. LANA tritides also retain helium-3. The onset of helium release from LANA tritides has not yet been determined, however, data from the MTF indicates this is greater than five years. The retention of decay helium-3 is useful in tritium processing applications because it provides a source of high purity, helium-free tritium. The retention of helium-3 in the metal lattice also has disadvantages. As helium-3 accumulates in the lattice from decay, this insoluble component stresses the lattice. The stress is great enough to introduce short range order and to change the lattice constants. The results of this also vary from one compound to another. Palladium tritide undergoes a reduction in the equilibrium desorption isotherm pressures and a change in slope of a portion of the isotherm plateau region. LANA 0.75 and LANA 0.30 intermetallics undergo a number of changes including a change in the equilibrium desorption isotherm pressures, a change in the isotherm plateau slopes, and a reduction in the reversible absorption-desorption capacity. The latter effect is the result of the formation of tightly bound tritium which is often referred to as the tritium heel. This tritium cannot be easily removed under the normal processing conditions (150-200°C and full vacuum). In addition to the loss of production capacity, the formation of the heel also poses potential problems for the decommissioning and disposal of LANA tritium beds.

EXPERIMENTAL PROGRAM

Most of the data presented here is based on small metal tritide test samples containing approximately 4-5 grams of metal and 0.12-0.18 grams of tritium. After loading with tritium, tritium effects on the samples were characterized by determining the equilibrium desorption isotherms. Equilibrium isotherms relate the equilibrium pressure above the metal hydride to the composition of the solid at a given temperature.³ Desorption isotherms are determined by (1) loading a sample with the desired hydrogen isotope and (2) measuring the equilibrium pressure as measured aliquots of the gas are slowly withdrawn from the sample. The sample temperature is held constant during the experiment. Data relating the desorption equilibrium pressure to the composition of the solid, normally expressed in the ratio of hydrogen isotope atoms to metal atoms in the alloy (e.g. H/M, D/M, or T/M), are obtained from this experiment (see examples in Figures 1 and 2).

In this study, desorption isotherms were determined under various test conditions such as dormant aging and frequent absorption-desorption cycling. Data is also included for a large-scale LANA 0.75 tritium storage bed (Storage Bed A) which was used on the experimental manifold for 72 months as a source of high purity tritium. This bed is representative of production storage beds such as those used in RTF and room 281, and was very frequently used for storage, pumping, and compression of tritium during this period. The following is a list of samples used for this analysis and a brief description of the test histories in the Materials Test Facility.

LANA3T4B: Test sample of $\text{LaNi}_{4.7}\text{Al}_{0.3}$ (LANA 0.30, heat T-1112A-2).

Six tritium isotherms were determined.

LANA75T2: Test sample of $\text{LaNi}_{4.25}\text{Al}_{0.75}$ (LANA 0.75, heat T-1158).

Loaded 3/87. Three tritium isotherms determined in 6/87, 9/87 and 7/89. Sample exchanged with deuterium during 8/89.¹¹

LANA75T5: Test sample of $\text{LaNi}_{4.25}\text{Al}_{0.75}$ (LANA 0.75, heat T-1158).

Loaded 5/87. Five tritium isotherms determined during 5/87, 10/87, 11/87, 6/91, and 3/92

LANA75T7: Test sample of $\text{LaNi}_{4.25}\text{Al}_{0.75}$ (LANA 0.75, heat T-1158).

Loaded 7/87. Five tritium isotherms determined during 7/87, 9/87, 6/88, 7/88, and 10/92. Thermal absorption-desorption cycling conducted between isotherms #1 and #2 and between isotherms #3 and #4. The sample was exchange with deuterium during 11/89.

LANA75T8: Test sample of $\text{LaNi}_{4.25}\text{Al}_{0.75}$ (LANA 0.75, heat T-1158).

Loaded 3/90. One tritium isotherm determined during 3/90. This is the only virgin tritium isotherm (i.e. no tritium aging) of LANA 0.75.

21-Month Bed: Large scale bed 1000 grams of $\text{LaNi}_{4.7}\text{Al}_{0.3}$ (LANA 0.30). This bed was used in tritium production for 21 months before being taken out of service. During this 21-month period, the bed was in dormant storage for 15 months. The bed was desorbed at 180°C and the tritium heel was exchanged with deuterium. The bed was then opened and the contents removed for materials characterization.³⁻⁷

72-Month Bed: Contains 500 grams of $\text{LaNi}_{4.25}\text{Al}_{0.75}$ (LANA 0.75, heat T-1158). Loaded with tritium in 7/85. Heavily used as primary storage bed on the Experimental Tritium Manifold in room 101, 232-H, until its decommissioning by vacuum desorption and deuterium exchange during 8/91.¹³

The tritium heels for the exchanged samples were determined from deuterium exchange data. Analysis of the exchanged 21-Month Bed material at Mound Laboratories using heat ramp mass spectrometry and gas collection techniques showed that the residual tritium accounted for 1.3-1.7% of the total hydrogen isotopes. More exchange cycles were determined for the 72-Month Bed and the LANA75T2 sample. The heel determinations for these samples were based on P-C-T measurements on the manifold.

RESULTS

Tritium Heel Formation in La-Ni-Al Tritides

Quantification of the tritium heel formation measured from isotherms of various LANA 0.75 test samples is shown below in Table 1. Figures 1 and 2 showed the effects of aging on the equilibrium desorption isotherms.^{8,9} At tritium exposure times of less than up to about 444 days, all of the tritium absorbed on the samples can be removed at 150°C. After 861 days, there is a significant fraction of tritium that cannot be removed through routine thermal desorption. The data from Table 1 is presented graphically in Figure 3.

Table 1. Tritium Heel Formation in LANA 0.75 Test Beds as a Function of Cumulative Exposure Time

Sample	Exposure Time (Days)	Tritium Heel (T/M)
LANA75T8	0	0.00
LANA75T1	99	0.00
LANA75T7	100	0.00
LANA75T7	141	0.00
LANA75T5	149	0.00
LANA75T7	407	0.00
LANA75T7	444	0.00
LANA75T2	861	0.062
LANA75T7	904	0.070
LANA75T5	1273	0.151
LANA75T5	1423	0.120
LANA75T5	1793	0.146

Table 2 shows the tritium heel formation in other samples, including production-scale beds, as a function of cumulative tritium exposure time. These samples consisted of LANA 0.30 and LANA 0.75 samples.

Table 2. Tritium Heel Formation as a Function of Cumulative Exposure Time for La-Ni-Al Materials.

Sample	Material	Exposure Time (Days)	Tritium Heel (T/M)
21-Month Bed [†]	LANA 0.30	630	0.086
72-Month Bed [†]	LANA 0.75	2160	0.058
LANA3T4B	LANA 0.30	94	0.000
LANA3T4B	LANA 0.30	647	0.020
LANA3T4B*	LANA 0.30	797	0.005

[†]These are production-scale beds.

*Twenty absorption-desorption cycles were conducted on this sample before this isotherm.

Removal of Tritium Trapped in the Heel by Exchange with Deuterium

The deep-trapped or bound tritium heel in aged La-Ni-Al samples was effectively removed to very low levels by exchange with the lighter isotope deuterium. Deuterium exchange was used to remove the tritium heels from the 21-Month Bed³⁻⁵, 72-Month Bed¹³, and the LANA75T2 sample^{11,12}. A description of each experiment follows:

21-Month Bed: This production-scale bed was desorbed by heating under vacuum at 180°C for 1.5 hours. The final pressure was less than 2 psia. The tritium heel was then exchanged with deuterium. The bed was allowed to cool and loaded with deuterium to approximately D/M = 0.3. The bed was then heated to 200°C and the gas was desorbed. The composition of the desorbed gas was analyzed by mass spectrometry. Four exchange cycles were completed for this bed. Table 3 lists the exchange data for this bed.

Table 3. Deuterium Exchange Data for the 21-Month Bed

Exchange Cycle	D ₂ loaded (moles)	D ₂ desorbed (moles)	T ₂ desorbed (moles)	T/M
0				0.08612
1	1.845	1.208	0.429	0.02565
2	1.952	1.808	0.129	0.00747
3	1.918	1.846	0.032	0.00029
4	1.943	2.124	0.012	0.00012

The bed was then opened and the hydride powder was removed. Materials analysis of the powder at Mound Laboratories showed a total residual tritium content of 0.00090 moles for the tritide material in this bed.

72-Month Bed: This production-scale bed was removed from service and desorbed at 165°C. The residual tritium was removed by a deuterium exchange procedure using a total loading of D/M = 0.16 (in addition to the heel) followed by two desorptions into calibrated volumes at a bed temperature of 150°C. Ten exchange cycles were completed on this sample. A final tritium content determination by materials analysis of the powder has not been completed, but it is believed to be very low level in tritium based on the results of the 21-Month bed. This data is presented below in Table 4.

Table 4. Deuterium Exchange Data for the 72-Month Bed

Exchange Cycle	D ₂ loaded (moles)	D ₂ desorbed (moles)	T ₂ desorbed (moles)	T/M
0				0.05820
1	0.606	0.270	0.07532	0.03768
2	0.532	0.441	0.06082	0.02111
3	0.565	0.527	0.03663	0.01114
4	0.599	0.541	0.01879	0.00602
5	0.601	0.502	0.00892	0.00359
6	0.578	0.683	0.00668	0.00177
7	0.578	0.602	0.00316	0.00091
8	0.610	0.614	0.00224	0.00030
9	0.554	0.575	0.00072	0.00010
10	0.618	0.513	0.00037	0.00000

There was a question as to whether the tritium removed after several cycles was due largely to off-gassing of the vessel and associated hardware. To address this, the bed was allowed to lay dormant after nine exchange cycles. A period of eight months elapsed between the ninth and tenth exchange cycles. The tritium removal data for the tenth cycle falls in line with that expected from the previous cycles, suggesting that tritium offgassing from the hardware was negligible by the ninth exchange.

It had been believed that absorption-desorption cycling during the exchange process would improve the exchange efficiency. An experiment to evaluate this theory was included in the exchange experiment for the 72-Month Bed. The Bed was loaded with deuterium as stated above. The gas was desorbed to calibrated volumes at 150°C and a mass spectrometry sample was taken. The bed was then cooled (<-20°C) and heated (150°C) four times to absorb and desorb gas unto the hydride material. After three cooling and heating cycles, another mass spectrometry sample was prepared. Analysis of these samples taken before and after the thermal cycling give an indication of the effectiveness of this procedure. This procedure was used for the first five exchange cycles. Data for this study is presented below in Table 5. The thermal cycling was discontinued after five exchange cycles because there was no further improvement.

Table 5. Effectiveness of Heating-Cooling Cycles during Deuterium Exchange.

Exchange Cycle	% Tritium in Desorbed Gas	
	1st Desorption	4th Desorption
1	25.59	27.97
2	13.30	13.90
3	7.13	7.00
4	3.47	3.53
5	1.79	1.78

LANA75T2 Sample: The tritium heel from this small test sample (4.94 g of LANA 0.75) was exchanged with deuterium by loading to approximately $D/M = 0.22$ and heating the bed to 150°C to desorb the gas to calibrated volumes. Nine exchange cycles were conducted on this sample. This data is presented below in Table 6.

Table 6. Deuterium Exchange Data for the LANA75T2 Sample

Exchange Cycle	D ₂ loaded (mmoles)	D ₂ desorbed (mmoles)	T ₂ desorbed (mmoles)	T/M
0				0.06200
1	12.28	6.670	1.273	0.02687
2	8.092	7.293	0.531	0.01222
3	7.462	7.524	0.239	0.00562
4	7.844	7.890	0.110	0.00259
5	7.919	8.074	0.050	0.00120
6	7.912	8.129	0.025	0.00051
7	7.799	8.050	0.011	0.00021
8	7.888	8.143	0.006	0.00005
9	7.969	8.194	0.003	0.00000

Figure 4 is a plot of the deuterium exchange data (Tables 3, 4, and 6) for the three exchange experiments. This plot lists the tritium composition of the bed (T/M) after each exchange cycle. Figure 5 plots the tritium content of the bed material in curies per kilogram (Ci/kg) of metal hydride powder for the same experiment. Figure 6 shows this data (Ci/kg) in a semilogarithmic plot.

DISCUSSION

Heel Formation in LANA Tritides

Equilibrium tritium desorption isotherms for LANA 0.75 samples after several different aging times are presented in Figures 1 and 2.^{8,9} The effects of tritium aging on the isotherms results in a (1) lowering of plateau pressure, (2) an increase in plateau slope, and (3) a reduction in the reversible capacity of the hydride. In samples aged up to 444 days, all of the tritium in the heel could be removed under vacuum at 150°C. The presence of tritium that cannot be removed under these conditions has been defined as the heel. Actually, during the initial aging period, changes occur in the thermodynamics of the tritide which are manifested in changes in the equilibrium desorption isotherms. For virgin LANA 0.75 tritides, all of the tritium can be removed by desorption at 80°C, which is the standard temperature used for isotherm determinations on this compound in MTF laboratories. As the material ages, the isotherm plateau pressure gradually decreases and the isotherm slope increases, until it is no longer possible to remove all of the tritium from the material even at 150°C. Figure 3 shows the growth of the tritium heel for LANA 0.75 tritides.

In comparing the two materials, the LANA 0.75 samples showed a faster heel growth than LANA 0.30. This is believed to be associated with the intrinsically higher tritium equilibrium pressure in LANA 0.30. Thus for LANA 0.30, it is possible to remove larger quantities of tritium by desorption at 150°C than can be removed from LANA 0.75.

Effects of Absorption-Desorption Cycling on Tritium Heel Formation

It has been shown previously that absorption-desorption cycling of LANA metal tritide samples reduces the tritium heel and raises equilibrium desorption isotherm pressures (also see figures 1 and 2).^{3,8,9,12} This phenomenon can be clearly seen in the analyses of the "21-Month Bed" and the "72-Month Bed." The 72-Month Bed (LANA 0.75) served as the primary tritium storage and supply bed for the Experimental Tritium Manifold located in room 101, 232-H, during its entire six-year service life. It was often used daily. The bed loading never exceeded $T/M = 0.5$ and the average tritium inventory during the service life is estimated to be $T/M = 0.3-0.4$. The total cumulative tritium heel after 72 months for this bed was $T/M = 0.058$. On the other hand, the 21-Month Bed (LANA 0.30) was held in dormant storage for 15 of the 21 months of its service life. The average tritium inventory in the bed during the service life was estimated to be $T/M = 0.058$. The heel for this material was $T/M = 0.086$ when taken out of service.

It has also been observed in the LANA75T5 sample that the tritium heel after 1423 days, $T/M = 0.119$, was smaller than the heel after 1273 days, $T/M = 0.151$.⁸ It is believed that the adsorption-desorption cycle associated with the isotherm determination at 1273 days caused the reduction of the tritium heel. Previous to the 1423 day isotherm, the LANA75T5 sample had been in dormant storage for 1135 days. The reduction in the size of the tritium heel in this sample was also accompanied by an increase in the equilibrium desorption isotherm plateau pressure, a phenomenon which was also observed previously.⁸

The reduction in the size of the tritium heel due to cycling can also be seen in the LANA3T4B sample. After 649 days of exposure the sample had a tritium heel of $T/M = 0.020$. The sample was subjected to twenty absorption-desorption cycles and placed into dormant storage. After 797 days of exposure the desorption isotherm showed that the heel had diminished to $T/M = 0.0053$. This is apparently due to the effects of cycling.

Heating of a LANA tritide sample has also been shown to improve the equilibrium desorption isotherm pressure and reduce the tritium heel. An aged sample heated to $>300^{\circ}\text{C}$ under vacuum showed an increase in the isotherm plateau pressure and a reduced the heel.¹¹ Although these conditions are not within normal processing parameters, it suggests a method which may be used to extend LANA bed life.

Removal of Tritium Heel by Deuterium Exchange

Although the tritium heel could not be removed by heating the bed under vacuum, it was easily removed by exchange with the lighter isotope deuterium. During absorption and desorption of deuterium on these tritium-aged materials, the mobility of hydrogen in the lattice does not appear to be hindered by the presence of the very stable sites which constitute the heel. In fact, exchange studies with the LANA75T2 sample had shown that the removal of tritium is nearly identical with that expected simply by exchange of a virgin material given the known separation factor for the D/T system.^{11,12} This indicates that hydrogen is very mobile within the solid hydride, despite the presence of a number of sites of varying energy levels. The isotope exchange process appears to be thermodynamically controlled. Gas transfer to and from the solid (i.e. absorption and desorption) appears to be heat-transfer limited, and not kinetically limited.

An analysis was made to determine an optimum loading of deuterium for each exchange cycle. This is defined as $\Delta (D,T)/M$ which is the amount of deuterium absorbed minus the amount of deuterium and tritium removed in each exchange cycle. Deuterium exchange for the LANA75T2 sample was performed using a loading of $\Delta D/M = 0.22$

(approximately 33% of the bed capacity). The 72-Month Bed was exchanged using a total loading of $\Delta D/M = 0.15$ (approximately 20% of bed capacity). A loading of $\Delta D/M = 0.30$ (approximately 40% of the bed capacity) was used for the 21-Month Bed. Data for the three exchange experiments is presented in Figure 7. The data has a good deal of scatter, and the decontamination factor, which is fractional reduction in tritium inventory of the bed after each cycle, increases approximately linearly with an increase in the amount of gas used in the exchange.

One might question how well the small test beds, such as those in the MTF Tritium Exposure Program, can predict the behavior of large-scale beds. The data has a good deal of scatter, and within experimental error, the LANA75T2 test bed data shows good agreement with the other two exchange experiments.

Effectiveness of Thermal Absorption-Desorption Cycling during Deuterium Exchange

The use of absorption-desorption cycling of an aged LANA tritide bed had been proposed during the deuterium exchange process.¹² Once the bed was loaded with pure deuterium, a series of thermally-induced absorption-desorption cycles (to an expansion tank) was thought to improve the exchange efficiency (i.e. more of the tritium would be removed per exchange cycle). During first five deuterium exchange cycles of the 72-Month Bed, the gas analysis from the initial desorption was compared with the analysis after the fourth desorption. A slightly better exchange efficiency was observed for the first two deuterium exchange cycles. After the second cycle, the thermal cycling made no difference in the exchange efficiency. Thus the use of thermal cycling during deuterium exchange does not improve the process. This data is presented graphically in Figure 8.

CONCLUSIONS

1. The formation of tritium heels in LANA 0.30 and 0.75 tritides has been quantified. The size of the heel is very dependent upon storage and processing conditions.
2. Frequent absorption-desorption cycling of the tritide beds mitigates the formation of the tritium heel and can reduce the size of a heel once it is formed.
3. The higher pressure material LANA 0.30 showed slower heel formation than the storage material LANA 0.75, due largely to the higher intrinsic equilibrium pressure. This allows more of the tritium to be removed at the maximum processing temperature.

Also, in plant application, LANA 0.30 beds are used as compressors. Except during compressor operation, these beds will not be loaded with tritium and aging of these beds will be very slow.

4. Tritium heel removal by deuterium exchange was demonstrated and quantified. The use of absorption-desorption cycles during an exchange cycle does not improve the exchange efficiency. Residual tritium can be removed to very low levels using this procedure. For a tritide bed scheduled for removal from the process, a final tritium level can be estimated based on the number of deuterium exchange cycles.

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Figure 1. Equilibrium Desorption Isotherms at 80°C of Tritium Aged LANA 0.75.

Figure 2. Isotherm Plots of Tritium Aged LANA 0.75. The initial points in the aged plots were determined at 80°C. When the equilibrium pressure reached 10-20 torr in the isotherm, the samples were heated to 150-160°C, and a second set of isotherm points were determined. The lower points on the 150-160°C data indicates the tritium "heel".

Figure 3. The Growth of the Tritium Heel in LANA 0.75 Samples. In samples with up to 444 days of tritium exposure, all the tritium can be removed by vacuum desorption at 150°C.

Figure 4. Deuterium Exchange Data showing residual tritium content in atom ratios vs. number of exchange cycles.

Figure 5. Deuterium Exchange Data showing residual tritium content in curies of tritium per kilogram of solid powder.

Figure 6. Semilogarithmic Plot of Deuterium Exchange Data showing residual tritium content in curies of tritium per kilogram of solid powder.

Figure 7. The effects of varying deuterium loadings during the deuterium exchange experiment.

Figure 8. The Effect of Absorption-Desorption Cycling during Deuterium Exchange.

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