NMR Studies of ^{3}He Retention and Release in Metal Tritides - A Review

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Outline

I. Basics of NMR as related to ^{3}He and T (^{3}H) – how to tell between trapping in solid phases versus gas bubbles

II. Metal Tritides – Detailed Examples:

- LiT – the ionic salt with self-decomposition
- UT_3 – strongly paramagnetic metal phase
- PdT_x – temperature dependences of NMR relaxation times used to characterize ^{3}He in bubbles

III. Examples of ^{3}He NMR for other Tritides

IV. Summary & Conclusions

Basics of Nuclear Magnetic Resonance (NMR)

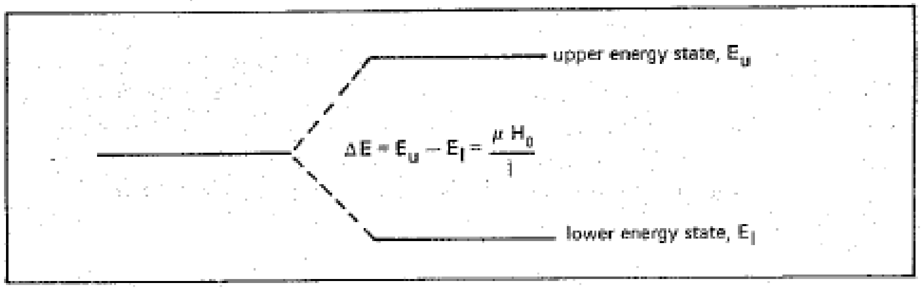


Fig. 1. Diagram illustrating the splitting of the nuclear magnetic energy level in the presence of an external magnetic field, H_0.

$\nu_I = \gamma_I H_0/2\pi$ (SHARP SIGNAL)

SPIN	I	γ_I	SENSITIVITY
H	1/2	26,751	1.000
T	1/2	28,534	1.067
^{3}He	1/2	20,378	0.762

PARAMETERS AVAILABLE FROM NMR PULSE TECHNIQUES

- INITIAL SIGNAL AMPLITUDES (SPIN CONCENTRATIONS TO WITHIN ± 5-20% DEPENDING ON SPECIES)
- T_2^* (LINESHAPE DECAY TIME)
- T_{2m} (SPIN-SPIN RELAXATION TIME)
- T_1 (SPIN-LATTICE RELAXATION TIME)

FOR I = ½ NUCLEI (I.E., H, T, ^{3}He) MAGNETIC DIPOLE INTERACTIONS DOMINATE RIGID-LATTICE PARAMETER T_{2L}

For I = ½ nuclei, $T_{2L} = T_{2d}$, which can be calculated using crystal structure and locations of both resonant and non-resonant spins

GENERAL BEHAVIOR OF NMR RELAXATION TIMES

A. IMMOBILE ATOMS ON LATTICE SITES

$$T_{2d} \simeq T_2^* \simeq T_{2m} \ll T_1$$

B. BULK GAS

$$T_{2d} \ll T_2^* < T_{2m} \simeq T_1$$

C. MOBILE ATOMS IN SOLID <u>OR</u> MICROSCOPIC GAS BUBBLES

$$T_{2d} < T_2^* < T_{2m} < T_1$$

ORDERS-OF-MAGNITUDE DIFFERENCES AMONG A., B., AND C. DEPENDING UPON ATOM DIFFUSION CONSTANT

IMMOBILE LIMIT $< \sim 10^{-12}$ cm^2/s $<$ MOBILE LIMIT

Practical Comments on NMR Characterization of ^{3}He Retention

- ^{3}He NMR is non-destructive – allows chronological studies during tritium aging
- Quantitative measurements of ^{3}He content in "solid" with precision of 5 – 20%
- Minimum NMR detection limit: ~$1x10^{20}$ ^{3}He atoms @ room temperature
- Measurement of ^{3}He relaxation times : >$2x10^{20}$ ^{3}He atoms @ room temperature (hence, no values reported before at least 3-6 months of tritium decay)
- ^{3}He T_1 and T_{2m} recoveries are nearly always highly non-exponential – hence, generally use 1/e values to define "average" relaxation times.
- Most ^{3}He NMR measurements have been done at room temperature (Exceptions of low temperatures include UT_3, PdT_x, and TiT_x [Weaver, et al.])
- ^{3}He NMR Experiments in MT_x performed at few locations (Open Literature):
    - ➢LANL (~1960 very limited)
    - ➢LLNL (~1965 → ~1970s)
    - ➢Sandia – Albuquerque (1970s)
    - ➢Sandia – Livermore (late 1980s)
    - ➢Mound Laboratory (1972 → ~1991)

Metal Tritides Studied Using NMR @ Mound Laboratory

Metal Tritide	Host Crystal Structure	Typical NMR Samples		T (^{1}H) Relaxation Times (ms)			Special Comments, Etc.
		Mass (grams)	Initial T-content (10^{20} atoms)	T_2*	T_{2m}	T_1	
LiT	FCC (NaCl)	0.28	169	0.011/0.45	-/2.6	~2000/380	Ionic salt, rigid lattice, self-radiolysis giving ~ 9% T_2 gas in bubbles & Li metal
$TiT_{1.88}$	FCC (CaF_2)	~1.0	~200	0.011	-	231	Metallic, non-stoichiometric, rigid lattice, tetrahedral T-sites
$VT_{0.50}$	Monoclinic	2.72	155	0.065	0.13	22.6	Metallic, non-stoichiometric, octahedral T-sites, mobile
$VT_{0.75}$	BCC	1.79	151	0.039	0.78	22.8	Metallic, non-stoichiometric, tetrahedral T-sites, very mobile
$Mg_2NiT_{3.96}$	Monoclinic	0.68	135	0.017	-	510	Semiconducting, NiH_4^- ions, stoichiometric, no T_2 gas seen up to 1285 days @ 300 K
$PdT_{0.65}$	FCC (NaCl)	2.42	87	0.114[a]	1.4[a]	76[a]	Metallic, ductile, very mobile tritium
$ZrNiT_{2.93}$	Orthorhombic	1.84	204	0.007/0.036	-/0.06	235	Metallic, non-stoichiometric, both rigid & mobile tritium in two sites
$BaT_{1.93}$	Orthorhombic	1.61	131	0.021	-	3380	Ionic, stoichiometric, no T_2 gas seen up to 858 days @ 300 K
$UT_{3.0}$	FCC (β-W)	~3.0	~220	0.009	-	~4	Metallic, rigid lattice, paramagnetic [ferromagnetic T_c @ 183 K]

[a]Measured on a $PdT_{0.10}$ sample aged for 8-years

Isotopic Li(H,D,T) Undergoes Extensive Swelling and Helium Outgassing During Irradiation

$$T \longrightarrow {}^{3}He^{+} + \beta^{-} + \underline{\nu}_e + 18.6\ keV \quad (t_{1/2} = 12.32\ y)$$

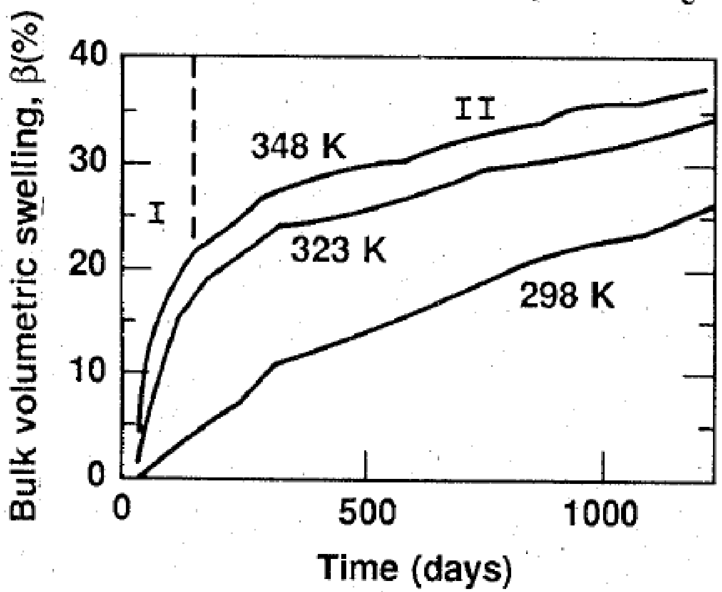
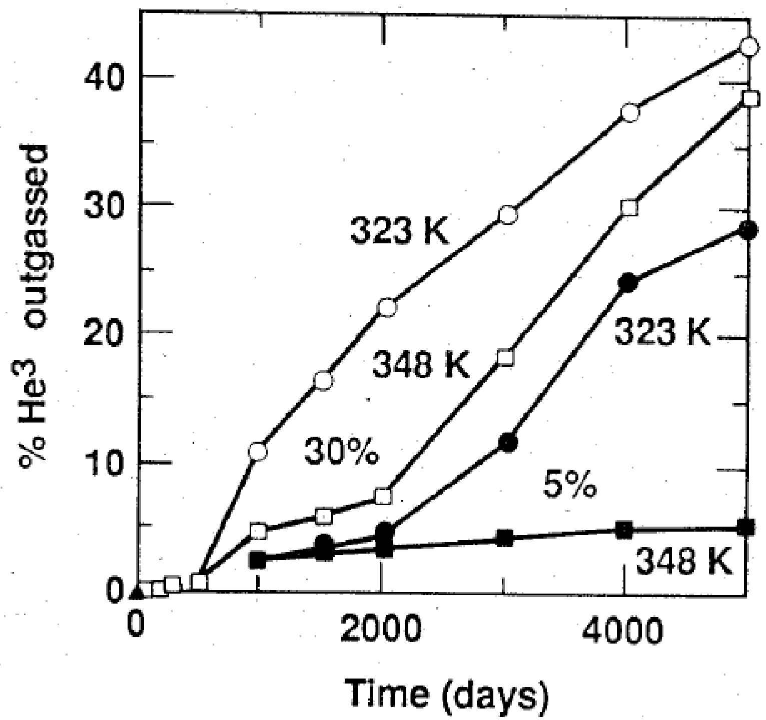


Fig. 1. Examples of bulk swelling in Li(D, T). The samples are #77, 79 and 99 with initial tritium concentrations of 45%. There are two distinct regions at 323 and 348 K: I – early fast growth and II – slow linear growth.

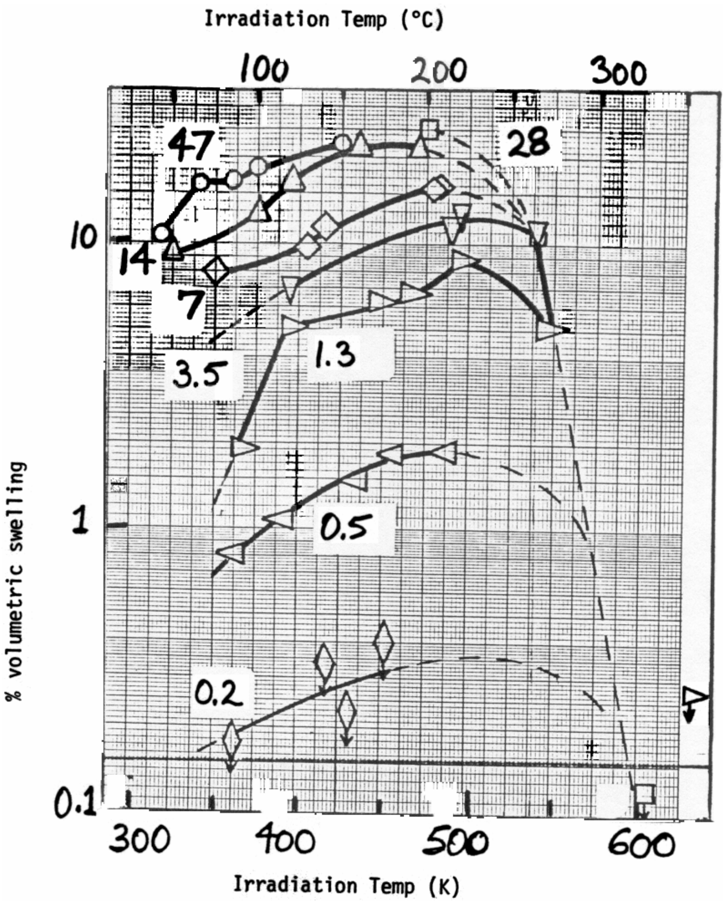


Percent of the created ³He that outgassed from four Li(D, T) samples.

Data summarized by Souers, et al. (1988)
Intense studies at LANL, LLNL, Mound Lab (1953 -- 1980s)

γ-Ray Irradiation of LiH Produced Similar Swelling & Bubbles Filled with H_2 gas along with Li metal (Radiolysis)

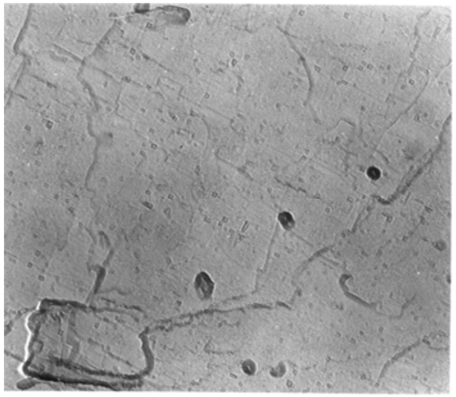
Peak Swelling between 400 K - 500K



Total Dose in Grads

P.C. Souers, et al., J. Phys. Chem. Solids 31 (1970) 1461

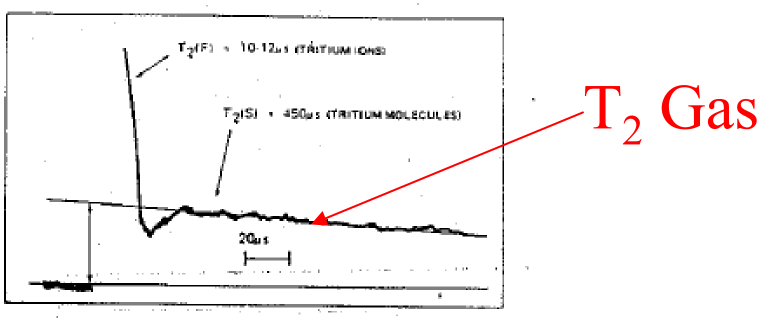
SEM images of cubic bubbles



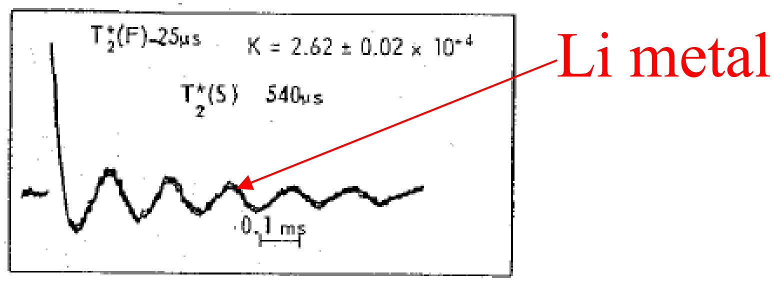
1.3 Grads @ 393 K: 5.1 % Swelling and 7.5 nm average length

1.3 Grads @ 493 K: 8.7% Swelling and 57 nm average length

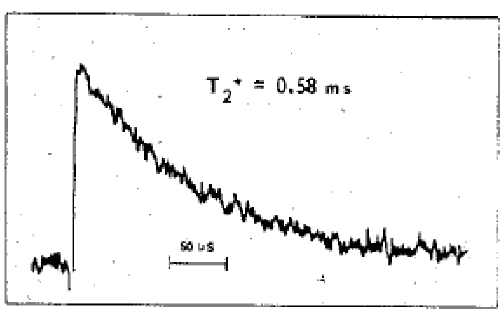
LiT Radiation Damage Characterized by Pulsed Nuclear Magnetic Resonance Methods @ Mound Lab



TRITIUM NMR SIGNAL



LITHIUM NMR SIGNAL



^{3}He NMR SIGNAL

^{3}He RELAXATION TIMES IN GAS BUBBLES

$$T_1^{-1} = T_{1B}^{-1} + T_{1W}^{-1}$$

$$T_{2M}^{-1} = T_{2B}^{-1} + \left(\frac{\lambda}{R}\right) T_{2W}^{-1}$$

WHERE

$$T_{1B} \approx T_{2B} > 10^3 \text{ SEC}$$

$$T_{1W} = \frac{4R}{3\bar{v}\, t_s} T_{1S}$$

$$T_{2S} < T_{1S}$$

HENCE, WALL RELAXATION FOR ^{3}He GIVES

$$T_1 \approx C_1 R$$

$$T_2 \approx C_2 R^2$$

$$C_2 < C_1$$

- Identified $(H,D,T)_2$ & Li metal
- Concentration from spin counts

"T" NMR Results for T_2 Bubbles in LiT from Self-Radiolysis

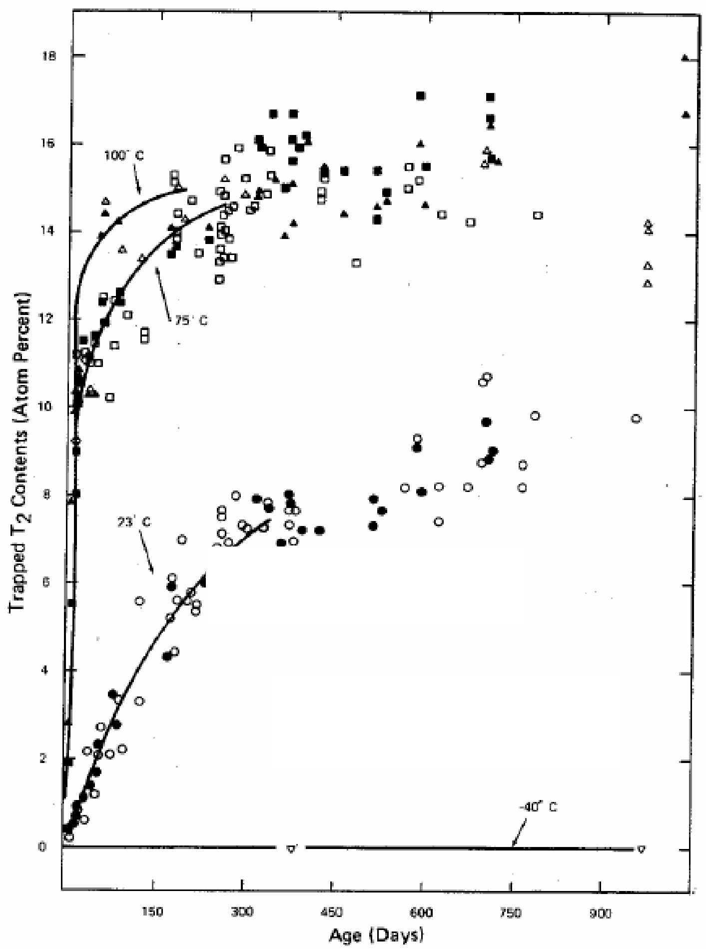


FIGURE 3 - Trapped T_2 contents in LiT samples aged at -40°C, 23°C, 75°C, and 100°C.

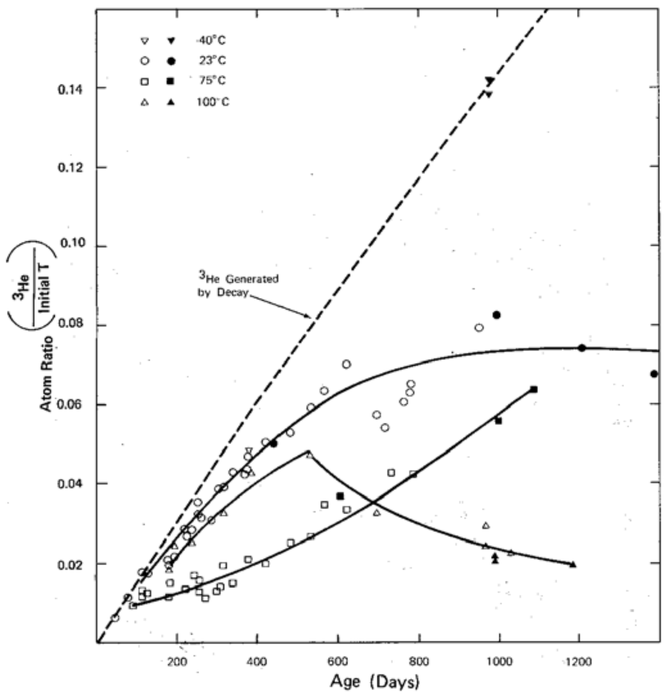
TRAPPED T_2 CONTENTS, TRITIUM RELAXATION TIMES, AND CALCULATED MEAN BUBBLE LENGTHS

Storage Temperature (°C)	Maximum Trapped T_2 Contents (a/o)	Average Relaxation Times T_1 (ms)	Average Relaxation Times T_{2m} (ms)	T_{2w} [a] (ms)	$\bar{A}_o$ [b] (nm)
23	9.5 ± 0.6	380 ± 70	2.6 ± 1.2	2.6	≤5
75	14.9 ± 1.0	290 ± 20	18 ± 6	20	32.5
100	14.8 ± 1.0	280 ± 20	29 ± 8	32	40
200	12.0 ± 0.7	140	65	120	60

[a] $\frac{1}{T_{2m}} = \frac{1}{T_1} + \frac{1}{T_{2w}}$

[b] Bubble edge length from correlation for γ-irradiated LiH by Souers, et. al., J. Phys. Chem. Solids 31, 1461 (1970).

^{3}He NMR Contents & Relaxation Time for Aged LiT



Helium Contents in LiT from NMR ^{3}He Spin Counts at Room Temperature and Resonance Frequency of 45.7 MHz

Age (days)	Decay	Atom Ratio of ^{3}He/T			
		-40°C	23°C	75°C	100°C
177	0.027	-	0.021	0.015	0.019
373	0.056	0.048	0.045	0.021	0.043
528	0.078	-	0.059	0.027	0.048
763	0.111	-	0.063	0.043	0.032
990	0.142	0.140[a]	0.082[a]	0.056[a]	0.021[a]
1190	0.168	-	0.075[a]	0.064[a]	0.020[a]

[a]From gas analyses during thermal decomposition.

^{3}He Nuclear Relaxation Times in Aged LiT from NMR Measurements at Room Temperature and 45.7 MHz

Age (days)	Spin-spin relaxation times (T_2)-ms				Spin-lattice relaxation times (T_1)-s			
	-40°C	23°C	75°C	100°C	-40°C	23°C	75°C	100°C
184	-	15	-	225	-	6.5	-	15.9
380	0.9	27	100	430	7.2	7.8	-	-
540	-	35	170	700	-	9.4	10.6	24.3
735	-	125	210	375	-	10.0	-	15.9
815	-	168	320	-	-	11.3	16.3	-
968	1.9	290	-	300	7.2	-	-	10.0

Summary of Li(H, D, T) Behavior Derived from NMR

- Swelling due to radiolytic decomposition: Molecular hydrogen & lithium metal
- T_2 and ^{3}He are trapped in microscopic gas bubbles (d < 100 nm)
- Bubble size increase with temperature (larger) and radiation dose/age
- Helium outgassing is mainly due to bursting bubbles
- Li metal formation lags formation of gas bubbles at all temperatures [Not covered in this presentation]
- Complex synergistic relation between swelling & bubble formation

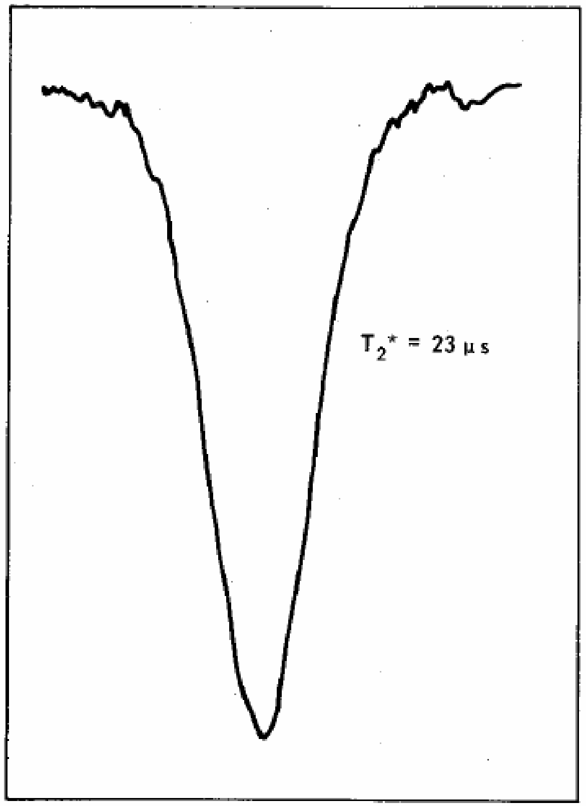
More information & references can be found:

1. P. C. Souers, et al., J. Nuclear Materials 154 (1988) 308-317.

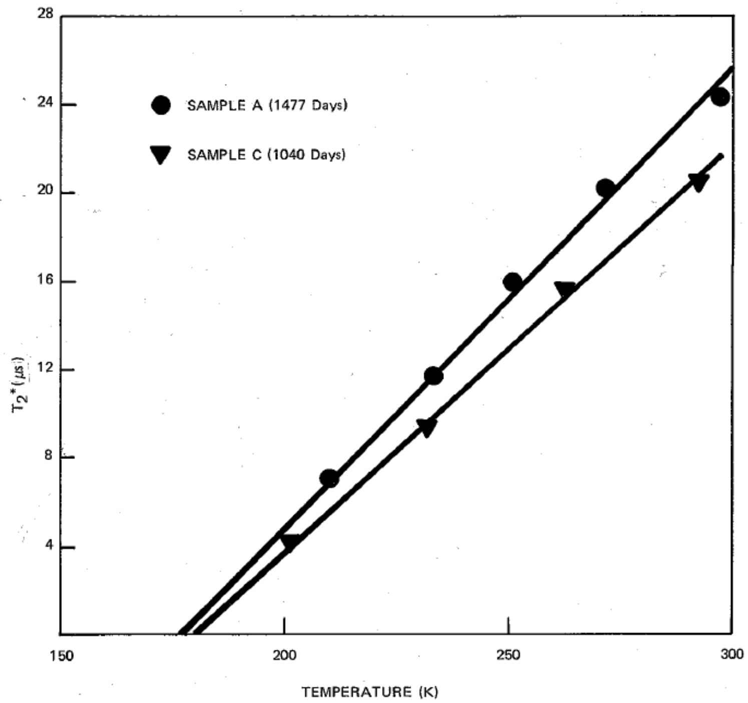
2. R. Bowman, et al., J. Nuclear Materials 154 (1988) 318-331.

Uranium Tritide (UT_3) is a Strongly Paramagnetic Metallic Phase – Widely Used for Storage Systems

[Ferromagnetic Ordering Temperature is ~183 K]

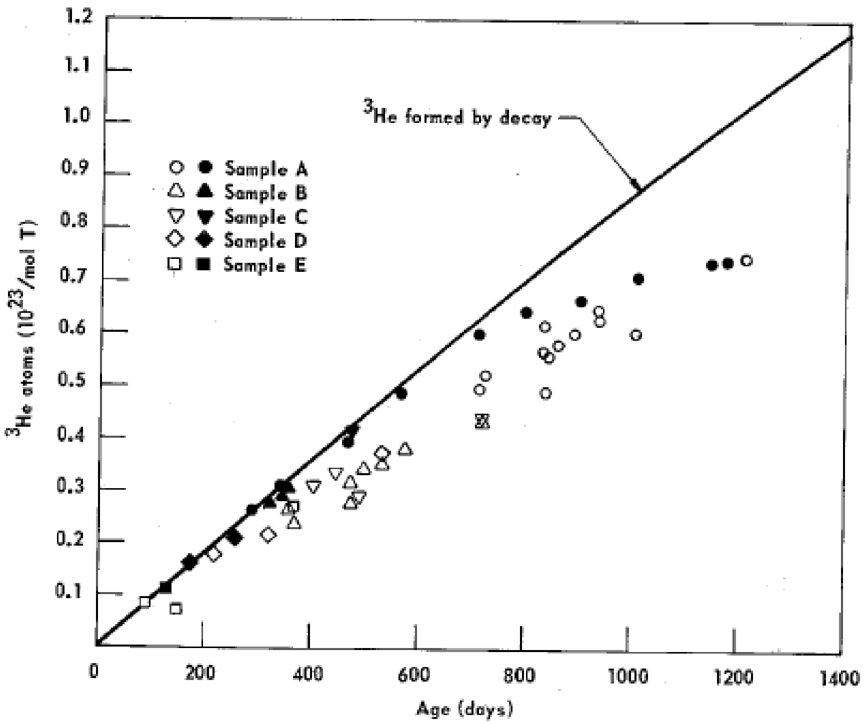


^3He spin-echo @ 20 MHz from UT_3 aged for 2 years

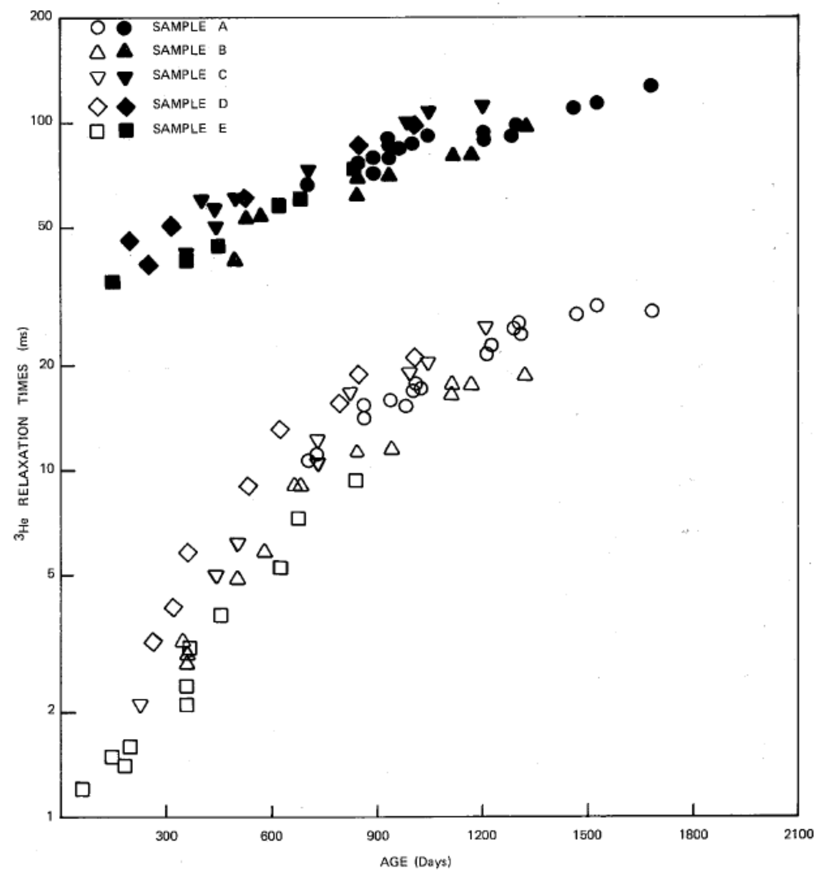


^3He T_2^* values @ 20 MHz for UT_3 decrease at lower temperatures from increasing paramagnetism approaching critical point

Age Dependence of ^3He NMR for UT_3 with Aging



^3He contents from volumetric measurements (closed symbols) and NMR spin counts (open symbols) for UT_3 samples stored and measured at room temperature.



^3He Relaxation times T_1 (closed symbols) and T_{2m} (open symbols) for UT_3 samples stored and measured at room temperature.

Bowman & Attalla, Phys. Rev. B **16** (1977) 1828.

^{3}He NMR for 8-year aged PdT_x

SOME PROPERTIES OF AGED PdT_x SAMPLE

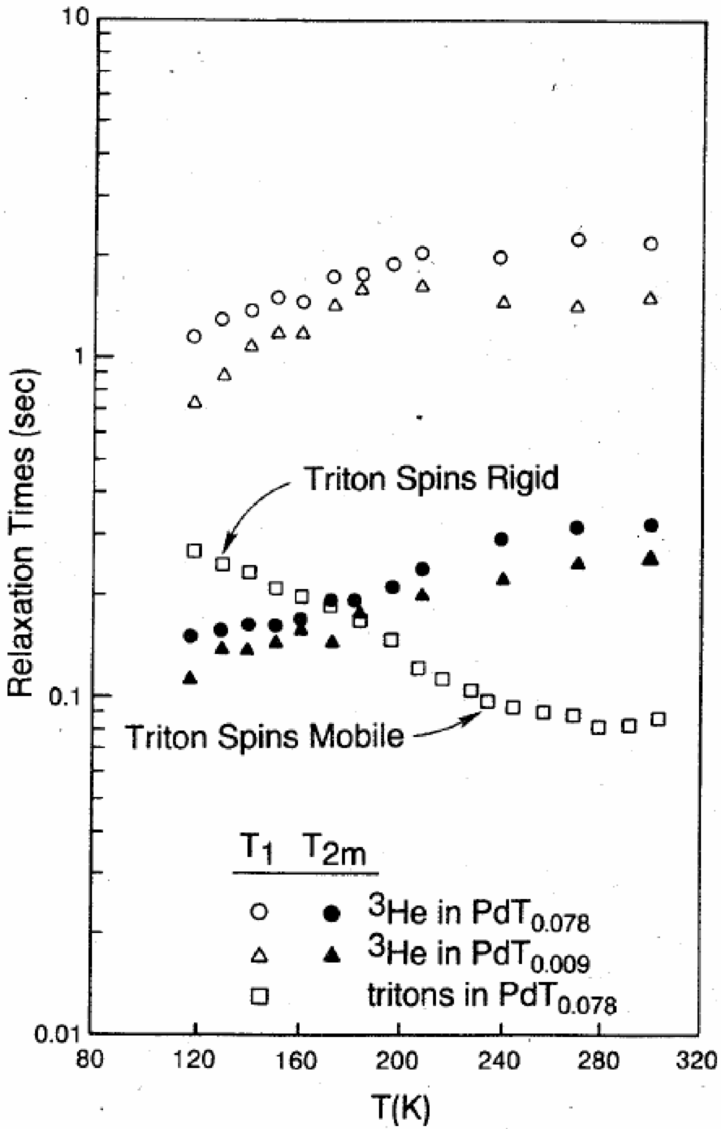
$PdT_{0.65}$ prepared about 6-74 and stored under T_2 gas until 2-81 when most tritium was removed [kept under argon or vacuum since 2-81].

NMR sample consisted of PdT_x powder [-100 mesh to +400 mesh] mixed with Al_2O_3. PVT-MS analysis indicated composition: $PdT_{0.10}$

Helium content of aged PdT_x:

NMR-spin count	13 x 10^{20} ^{3}He spins/gm
PVT-MS	16 x 10^{20} ^{3}He atoms/gm

Bowman, et al. Phys. Rev. B **37** (1988) 9447.



^{3}He relaxation times T_1 (open symbols) and T_{2m} (closed symbols) vs temperature in tritide-free aged PdT_x for $x=0.078$ (circles) and $x=0.009$ (triangles). Also shown is the triton relaxation time T_1 for $x=0.078$ (squares). All data were obtained at 45.7 MHz.

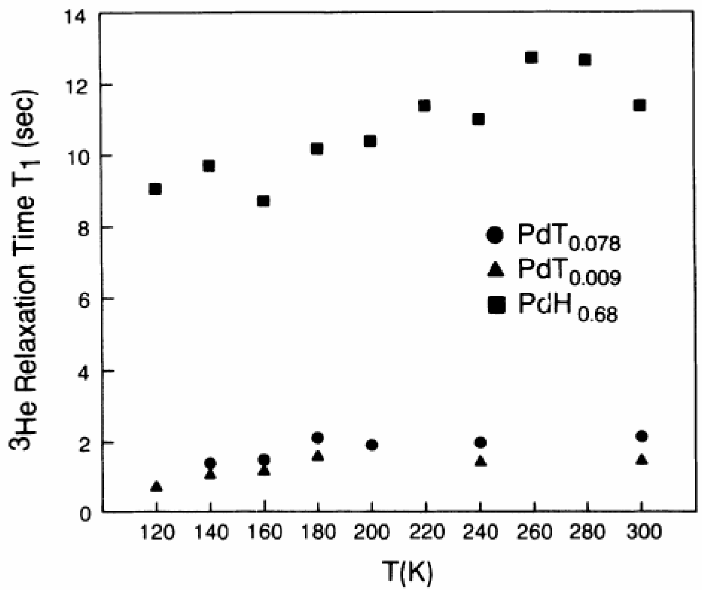


FIG. 2. ^{3}He relaxation time T_1 vs temperature, at 45.7 MHz, in tritide-free aged PdT_x for $x = 0.078$ (circles), $x = 0.009$ (triangles), and in $Pd(H,T)_{0.68}$ (squares).

Bowman, et al. Phys. Rev. B **37** (1988) 9447.

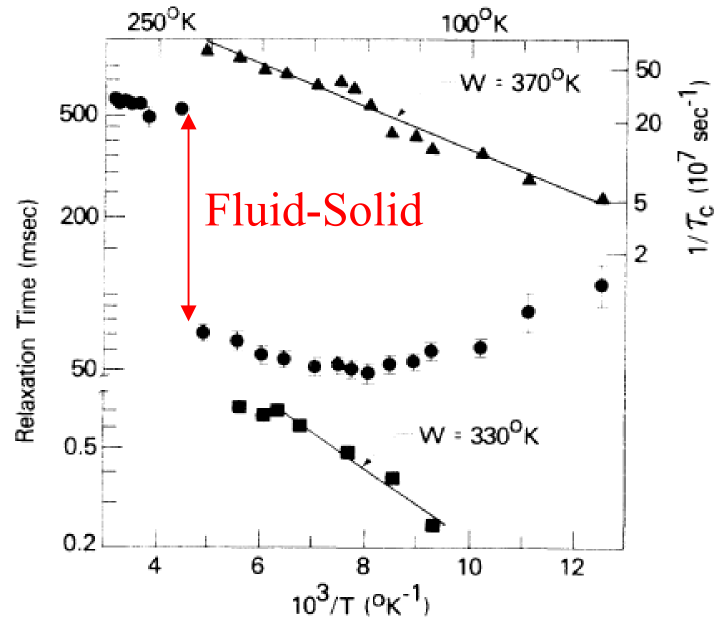


FIG. 1. Temperature dependence in 1-yr-old $Pd\,^3H_{0.62}$ of ^{3}He T_1 (filled circles) and T_2 (filled squares) relaxation times at 25 MHz (left-hand scale); and of ^{3}He jump frequency τ_c^{-1} (filled triangles) obtained from BPP analysis of T_1 (right-hand scale). The T_1 data set includes 99%-confidence-interval error bars. Note that the left-hand scale is broken.

Abell & Attalla, PRL **59** (1987) 995

TABLE III. Summary of ^{3}He NMR relaxation times at 300 K and 45.7 MHz.

Sample	Phase	T_2^* (msec)	T_{2m} (msec)	T_1 (msec)	T_{2m}/T_1
1	α	0.081	365	2120	0.172
2	α	0.077	178	2190	0.081
5	β		1680	11 350	0.148

Analysis of Low Temperature ^3He T_1 Data in Bubbles of 8-Yr PdT_x

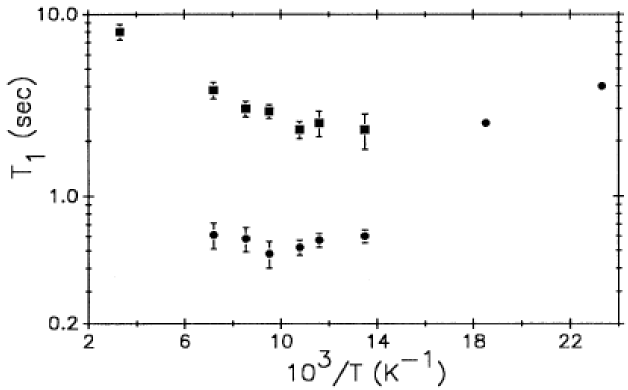


FIG. 1. Temperature dependence of spin-lattice relaxation parameter (T_1) for 8-yr-old PdT_x. Filled squares are for the fluid phase. Filled circles are for the solid phase. The error bars are estimates.

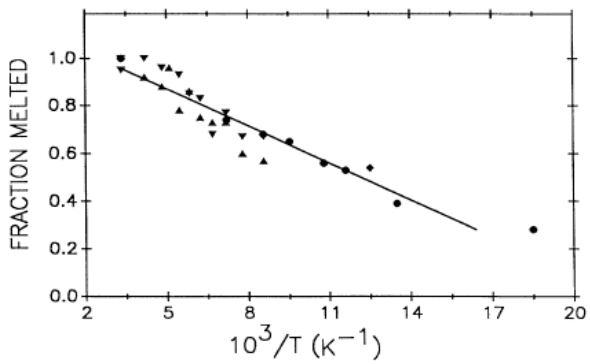


FIG. 2. Fluid phase fraction as a function of temperature from analysis of T_1 data assuming independent exponential relaxation for the two phases. Filled circles are from the present study ($\omega_0/2\pi = 142.8$ MHz), and the remaining symbols refer to analysis of T_1 data taken at 45.7 MHz on different 8-yr-old PdT_x samples (Ref. 7). The solid line is a linear fit to the data.

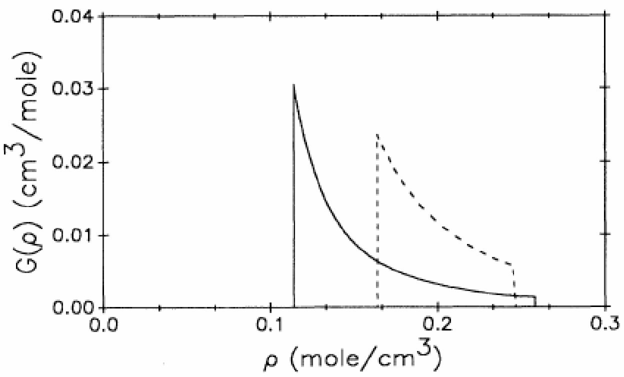


FIG. 3. The solid curve shows the density distribution for ^3He in 8-yr-old PdT_x derived from the fit to the data shown in Fig. 2, using the bulk helium EOS. The dashed curve shows the density distribution obtained for 2-yr-old PdT_x (Ref. 9).

Properties of ^3He Bubbles

Pressure = 6-11 GPa [60-110 kbar]

Mean Density = 0.20 mol/cm^3 (2 yr)

= 0.15 mol/cm^3 (8 yr)

Abell & Cowgill, Phys. Rev. B **44** (1991) 4178.

NMR of Solid ^3He Phase in 8-Year Old PdT_x

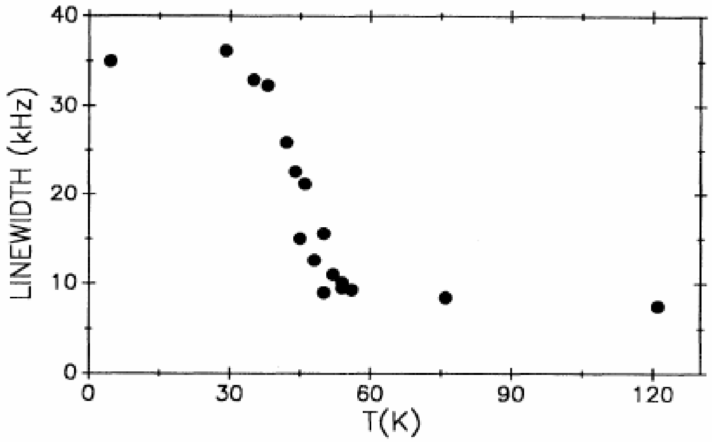


FIG. 4. Spectral linewidth of ^3He in 8-yr-old PdT_x as a function of temperature ($\omega_0/2\pi = 142.8$ MHz).

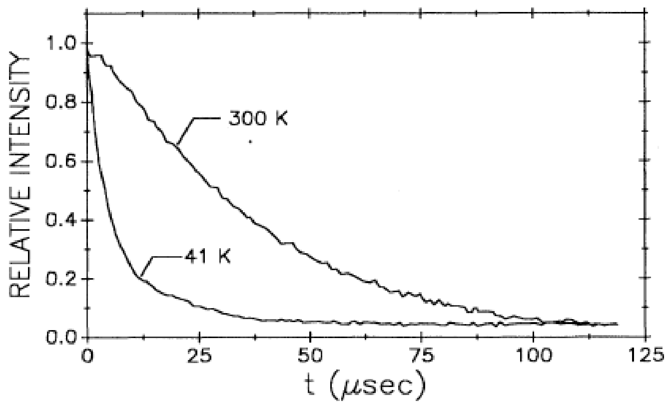


FIG. 5. ^3He FID at 300 and 41 K in 8-yr-old PdT_x ($\omega_0/2\pi = 142.8$ MHz). The 41 K FID is consistent with coexisting rigid-lattice (the rapidly decaying component) and motionally narrowed (the slowly decaying component) ^3He spins.

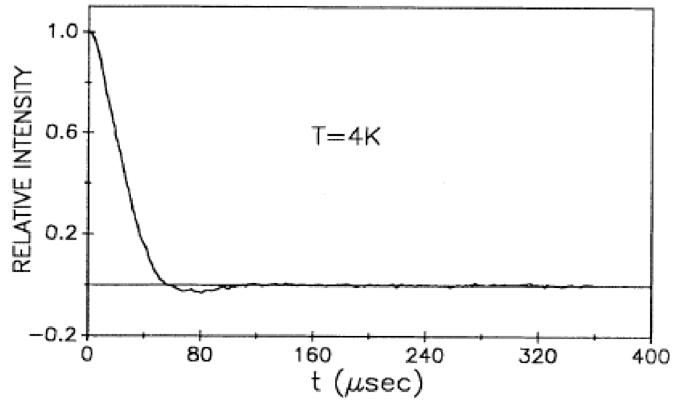
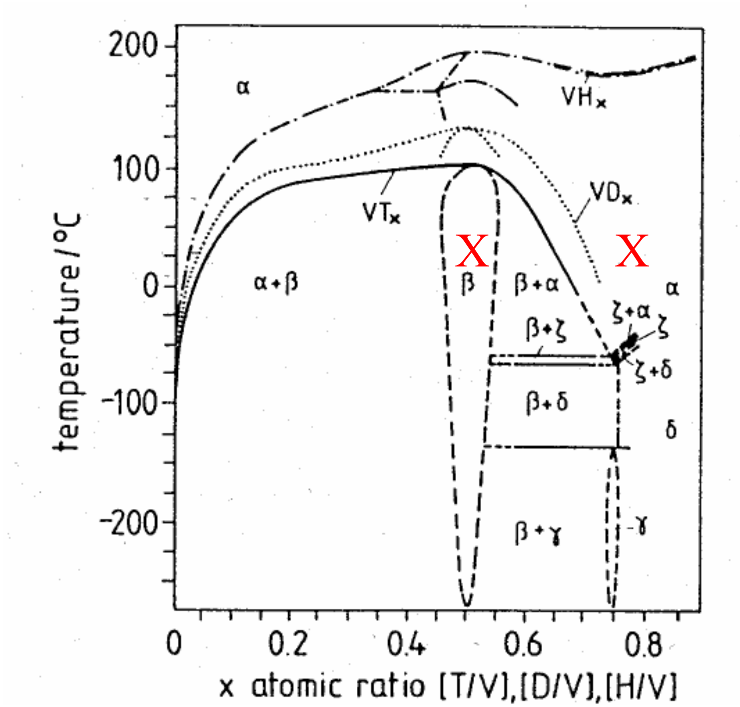


FIG. 6. Solid-echo FID for ^3He in 8-yr-old PdT_x at 4 K ($\omega_0/2\pi = 142.8$ MHz). The solid line represents the base line for the acquisition.

Abell & Cowgill, Phys. Rev. B **44** (1991) 4178.

^{3}He Retention in the VT_x Phases Via NMR



V-T Phase Diagram compared to V-H and V-D Systems (Lasser & Schober - 1987)

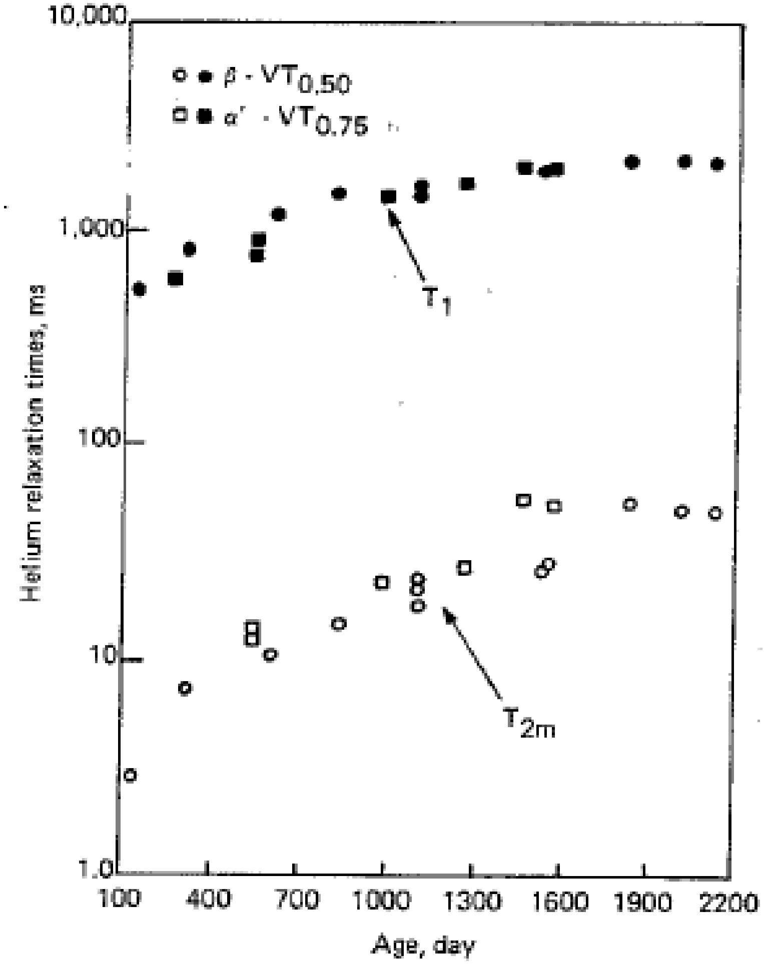


Fig. 3. Room temperature ^{3}He relaxation times T_1 (closed symbols) and T_{2m} (open symbols) for the VT_x samples.

BCC α-Phase: T = Tetrahedral sites

Monoclinic (BCT) β-Phase: T = Octahedral sites

Bowman, et al. Fusion Tech. **8** (1985) 2366.

^{3}He Relaxation Times Show Same Trends (i.e., Increasing Bubble Sizes from the Increasing T_{2m}/T_1 Ratios) for TiT_x and Mg_2NiT_x During Aging

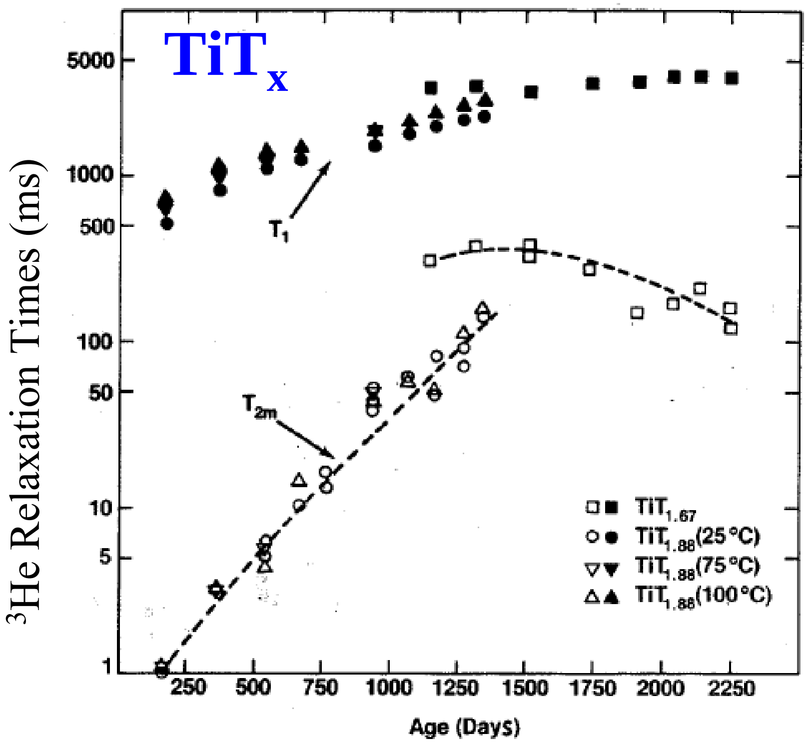


FIGURE 5 - Effect of age and storage temperature on helium-3 relaxation times in TiT_x.

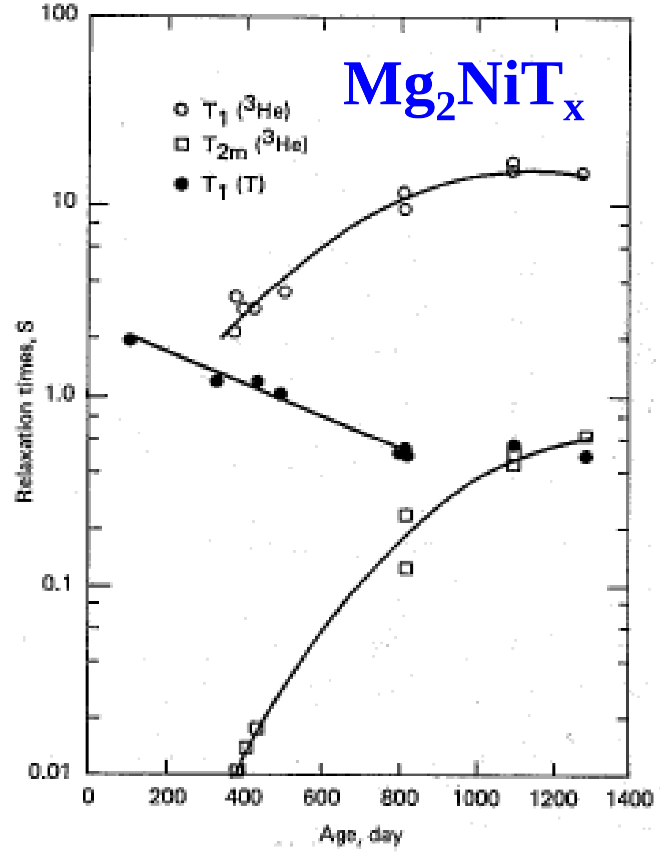


Fig. 4 - Age dependent behavior of triton T_1 and ^3He T_1 and T_{2m} relaxation times for Mg_2NiT_x samples. These parameters were measured at room temperature where the resonance frequency was 45.7 MHz.

Note: ^{3}He NMR suggests little effect on bubble properties from storage temperatures between 25 – 100 °C

Representative ^{3}He Relaxation Times for Several Metal Tritides Measured at 45.7 MHz and Room Temperature

Metal Tritide	Age (Days)	Atom Ratio for ^{3}He/T from NMR	^{3}He Relaxation Times (ms)			T_{2m}/T_1 Ratio
			T_2*	T_{2m}	T_1	
LiT	184	0.021	-	15	6,500	0.002
LiT	815	0.063 @ 763 days	-	168	11,300	0.015
$TiT_{1.88}$	168	-	0.049	0.9	500	0.002
$TiT_{1.88}$	1346	-	0.093	135	2,240	0.060
$VT_{0.50}$	140	-	0.061	2.9	530	0.005
$VT_{0.50}$	1825	0.18 (85%) @ 1548 days	0.076	54	2,240	0.024
$VT_{0.75}$	~600	0.11(79%) @ 989 days	-	~15	~900	0.017
$VT_{0.75}$	~1600	0.18 (82%) @ 1567 days	-	~70	~2,500	0.028
$Mg_2NiT_{3.96}$	403	0.09 (75%) @ 818 days	0.175	13.8	2,800	0.005
$Mg_2NiT_{3.96}$	1281	-	0.330	615	14,600	0.042
$PdT_{0.65}$	~2400	~0.41	0.081	365	2,120	0.172
$ZrNiT_{2.93}$	1050	0.101 (67%)	158	~160	~9,000	0.018
$ZrNiT_{2.93}$	1516	-	194	~250	~10,700	0.023
$BaT_{1.93}$	391	0.007 (11%)	-	-	-	-
$UT_{3.0}$	150	-	0.020	1.1	35	0.031
$UT_{3.0}$	1108	-	0.024	32.3	93	0.35

Conclusions of ^{3}He Distributions in Tritides from NMR Studies

- All tritides exhibit very similar behavior – host properties are mostly secondary, except for determining T_1 values.

- ^{3}He relaxation times at room temperature are always "Case C" from initial detection (i.e., ^{3}He atoms are only in gas bubbles)

    - Corollary: Little or no ^{3}He trapped in host lattice sites

- T_{2m}/T_1 ratio << 1 [Becoming larger with time – attributed to growth of the bubbles by factors of ~3-10]

- At long times (i.e., ~1000+ days) T_{2m}/T_1 ratios become nearly constant – attributed to reaching maximum size range where bubbles burst & tritide goes into "accelerated release" phase – this behavior varies with Tritide and its stoichiometry, etc.

- Non-exponential recoveries imply wide distribution of ^{3}He densities & pressures in bubbles that change with aging

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