NMR Signature of One-Dimensional Behavior of ^{3}He in Nanopores

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We have performed thermodynamic and NMR relaxation time measurements of ^{3}He adsorbed in the pores of the mesoporous molecular sieve MCM-41 at temperatures down to 1.7 K and at a range of frequencies up to 240 kHz. The MCM-41 substrate comprises a uniform array of quasi-1D straight pores with a diameter of 2.3 nm. We preplated the pores with a monolayer of ^{4}He to achieve an effective diameter of 1.6 nm at low temperatures. We made NMR measurements as a function of line density and frequency to investigate the spin dynamics and the effect of dimensionality. We observed $T_1 \propto \omega^{1/2}$, which is characteristic of one-dimensional diffusion. At these temperatures this arises from a classical size effect in the narrow pores. Our results demonstrate the possibility to study the spin dynamics of a 1D Tomonaga-Luttinger liquid at lower temperatures, where the ^{3}He liquid will constitute a quantum 1D system.

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One-dimensional (1D) quantum systems have proven an extremely fertile field for the development of theoretical tools to understand the influence of strong correlations in quantum matter. This includes predictions of unique behavior arising from dimensionality. A striking example is the breakdown of Fermi liquid theory, the standard model of interacting fermions, in one dimension, due to the nonperturbative influence of interactions [1–4]. A variety of physical realizations of one-dimensional systems have been identified including spin chains [5], quasi-1D organic superconductors [6], ultracold trapped atoms [7], edge states in a two-dimensional gas [8], tunneling between long ballistic wires in GaAs/AlGaAs heterostructures [9], and the observation of spin-charge separation [10].

Another approach has been the study of the isotopes of helium, both ^{4}He (a boson) and ^{3}He (a fermion), confined to nanotubes. Here most of the work has used mesoporous molecular sieves, consisting of arrays of channels of well defined diameter. There have been extensive studies of the influence of dimensionality on the superfluidity of ^{4}He [11–17]. The motivation for our experiment was the prospect of using ^{3}He confined in such nanotubes as a model system to study fermionic Tomonaga-Luttinger liquid (TLL) behavior [2]. The study of bulk ^{3}He played a central role in the development of Landau Fermi-liquid theory [18]. Also the investigation of ^{3}He films has contributed to understanding the influence of two dimensionality on strong correlations [19,20]. In both cases the ability to continuously vary the density has been of crucial importance. This advantage is coupled to intrinsic purity, the absence of a lattice, and the fact that the spin ($S = 1/2$) of the fermions themselves, arising from the ^{3}He nuclear spin, can be directly accessed by nuclear magnetic resonance (NMR).

In this spirit, ^{3}He confined in nanotubes offers the prospect of a uniform one-dimensional system of interacting fermions with tuneable line density. This is arguably the simplest system in which to seek to experimentally demonstrate the absence of single particle excitations (Landau quasiparticles), their replacement by collective excitations, and the fractionalization (spin-mass separation) central to Tomonaga-Luttinger liquids. Furthermore the dominance of known short-range van der Waals interactions lends itself to *ab initio* theory (see recent quantum Monte Carlo simulations of ^{4}He TLL in nanopores, as a function of pore diameter [21]).

Heat capacity measurements of ^{3}He adsorbed in the ^{4}He preplated pores of the mesoporous substrate FSM-16 provide evidence of a crossover from a 2D gas to a 1D quantum state at low temperatures [14,22,23]. In this Letter we report the first NMR study of the spin-dynamics of ^{3}He adsorbed on a similar substrate, MCM-41 [24]. The plating of the nanotubes by ^{4}He and the ^{3}He line density were adjusted to meet the stringent conditions required for the ultimate creation of the 1D quantum system. Our NMR technique allows the convenient measurement of the dependence of the spin-lattice relaxation time T_1 on the angular Larmor frequency ω. In this work at temperatures above the crossover we observe $T_1 \propto \omega^{1/2}$, characteristic of 1D diffusion, which in this regime arises from a classical size effect in the narrow pores. This illustrates the power of NMR to ultimately provide insight into the spin dynamics of the putative TLL.

In order to clarify the conditions necessary for the study of quantum degenerate 1D ^{3}He we first consider the case of noninteracting particles. The Fermi energy E_{F1D} of a 1D noninteracting gas of spin-$\frac{1}{2}$ particles of mass m is given by $k_B T_{\mathrm{F1D}} = (\hbar^2 \pi^2 n_L^2 / 8m)$, where n_L is the 1D line density. To realize a quasi-1D ^{3}He quantum system we require $E_{\mathrm{F1D}} < \Delta$, where Δ is the energy difference between the ground state and the first excited azimuthal state for ^{3}He in the pores [22,23]. Also the temperature T of the system must be much less than $\Delta / k_B - T_{\mathrm{F1D}}$. Matsushita *et al.*

have calculated the eigenstates of bare, noninteracting ^{3}He atoms on ^{4}He coated pores, assuming the ^{3}He cannot penetrate into the coating [22,23]. Their results provide a value of $\Delta \sim 800$ mK for an effective pore diameter of 1.6 nm. An ideal gas Fermi temperature $T_{\mathrm{F1D}} \approx 100$ mK requires a line density $n_L = 7 \times 10^8\ \mathrm{m}^{-1}$, equivalent to loading the pores with 0.01 monolayers.

This presents challenges for the detection of NMR signals, which we meet by exploiting the sensitivity of a broadband pulsed NMR dc SQUID (Superconducting Quantum Interference Device) spectrometer [25–27]. This setup allows us to study conveniently and directly the NMR relaxation times as a function of Larmor frequency $\omega = \gamma B$, where γ is the gyromagnetic ratio and B is the applied magnetic field, rather than conventional NMR relaxometry techniques relying on field cycling [28].

The mesoporous substrate MCM-41, consists of a uniform array of straight hexagonal mesopores, in the form of a powder with a pore length equal to the grain size ~300 nm. The sample used in this work had a nominal pore diameter of 2.3 nm. The effective pore diameter for ^{3}He was reduced by preplating with ^{4}He, in order to coat the inner surface of the pores with a monolayer of solid ^{4}He, to an effective diameter of 1.6 nm. This nonmagnetic layer is also crucial to replace any surface layer of solid ^{3}He. This eliminates the large background signal that would arise from the surface ^{3}He layer and spin-relaxation mechanisms that arise from exchange between ^{3}He within the pore and in the surface layer.

The MCM-41 was baked at 500 °C in vacuum for 24 hours before being transferred to the Stycast sample chamber in a helium atmosphere. It was characterized by series of ^{4}He vapor pressure isotherms measured between 1.6 and 10 K [26], using an *in situ* capacitive pressure gauge. The two-dimensional isothermal compressibility κ_{T} was determined from the vapor pressure P as $\kappa_{\mathrm{T}} = (1/k_{\mathrm{B}}Tn^2) \times [\partial n/\partial \ln P]_{\mathrm{T}}$, where n is the loading of the pores expressed as an equivalent areal density, or coverage. κ_{T} is found to exhibit two minima, the first (at $n = n_1$) corresponding to promotion to states in the pore outside the surface layer and the second (at n_f) to full pores. The ratio of the two minima ($n_f/n_1 = 1.65$) is consistent with a pore diameter of 2.3 nm, and in good agreement with measurements carried out on FSM-16 of a similar pore diameter [29]. Thus we estimate $n_1 = 10\ \mathrm{atoms\,nm}^{-2}$, as measured in that work.

Heat-capacity peaks attributed to the dimensional crossover to a 1D state have been observed for low density ^{3}He with n_4 in the range $1.05n_1$ to $1.47n_1$ [14,22,23]. Therefore in our experiments ^{4}He was added up to $n_4 = 1.12n_1$, then NMR measurements were performed with ^{3}He doses of $n_3 = 0.01n_1$ and $0.02n_1$, with subsequent additions of ^{4}He up to $n = n_3 + n_4 = 1.84n_1$. Figure 1(a) shows isotherms of the spin-lattice relaxation time T_1 and spin-spin relaxation time T_2, here measured as a function of ^{4}He dose with fixed amount of ^{3}He, $0.02n_1$. The relaxation times indicate minima close to "full pores." which we associated with the compressibility minimum, and expect to imply a local maximum in the correlation time of the motion. Exploration of this feature at lower temperatures will be the subject of future work. Figure 1(b) shows κ_{T} deduced from vapor pressure isotherms taken simultaneously with the NMR measurements, which is consistent with that measured earlier [26].

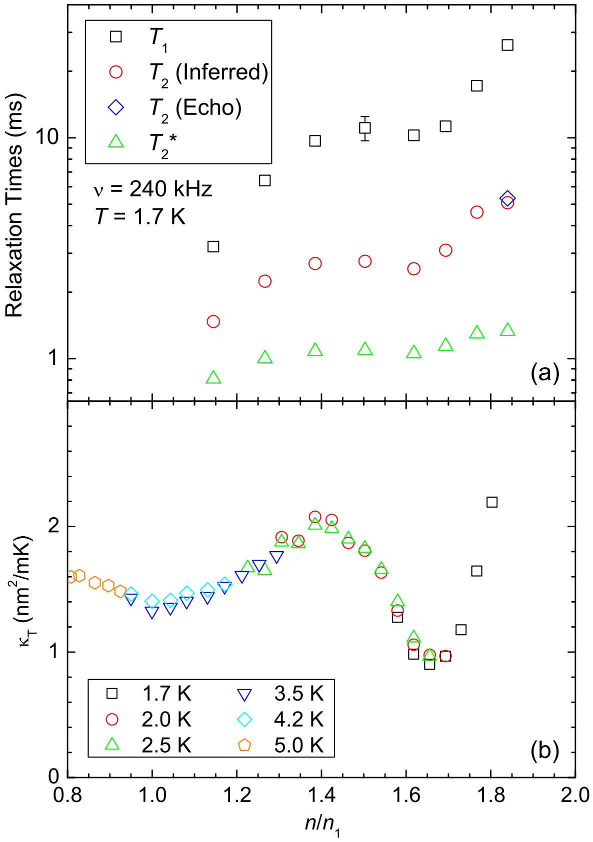

FIG. 1 (color online). NMR and thermodynamic properties of ^{3}He-^{4}He mixtures in nanopores as a function of total helium loading n. Promotion to states in the pore outside the surface layer occurs at $n = n_1$. (a) T_1 and T_2 for $n_3 = 0.02n_1$ measured at $T = 1.7$ K and a Larmor frequency of 240 kHz. For most of the coverage range $1/T_2$ was inferred from $1/T_2^*$ by subtracting an estimated magnet contribution (open circles). This approach was validated by a spin echo measurement at a high coverage (open diamond). (b) Isothermal compressibility deduced from vapor pressure measurements.

We first measured the relaxation times T_1 and T_2 for $n_3 = 0.01n_1$ on pores preplated with $n_4 = 1.12n_1$. We measured T_1 over the frequency range 80 to 240 kHz at 1.7 K. This is shown in Fig. 2 for different total helium coverages. We observed $T_1 \propto \omega^{1/2}$, characteristic of 1D diffusion of spins as discussed in detail below. The relaxation times were found to be independent of coverage on doubling the ^{3}He coverage to $n_3 = 0.02n_1$. This suggests that relaxation is governed by single-particle processes, as the spins relax

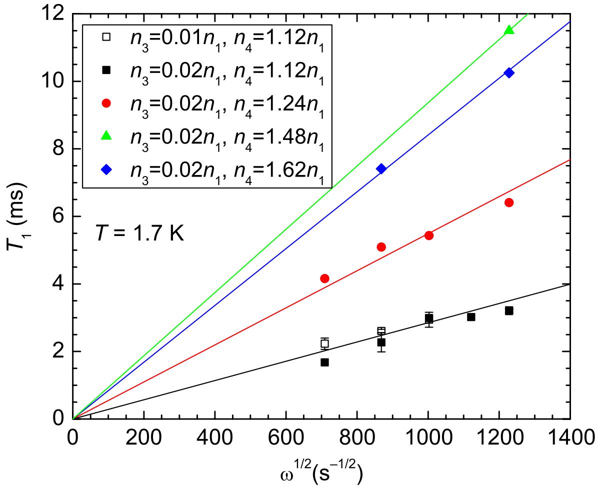

FIG. 2 (color online). Frequency dependence of T_1 in the mixture films at $T = 1.7$ K for various helium coverages. The $T_1 \propto \omega^{1/2}$ dependence (solid lines), characteristic of 1D diffusion of spins, is preserved as n_4 is increased. A fit to all T_1 data for $n_4 = 1.12n_1$ to the expression $T_1 = A\omega^{\alpha}$ gives $\alpha = 0.453 \pm 0.08$.

through motion in the local magnetic fields arising from impurities in the substrate, rather than through the intrinsic mechanism of ^{3}He nuclear dipole-dipole interactions.

This picture of impurity relaxation is consistent with evidence from our earlier measurements [26] on the T_1 relaxation of a surface ^{3}He film, as we now discuss. In that case, the temperature dependence of the relaxation times showed a minimum in T_1 of 0.55 ms at ~2.9 K for a coverage of $n_3 = 0.88n_1$. According to motional averaging theory [30] a minimum in T_1 is observed when the correlation time for local field fluctuations τ_c is the inverse of the angular Larmor frequency $2\pi\nu$. The value of T_1 at the minimum is given approximately by $(T_1)_{\min} \approx 2\pi\nu/M_2$, where M_2 is the second moment of the absorption line. For a randomly oriented line of spins separated by d, the dipole-dipole M_2 is $0.91(\mu_0/4\pi)^2(\hbar^2\gamma^4/d^6)$; for a plane of spins it is $2.9(\mu_0/4\pi)^2(\hbar^2\gamma^4/d^6)$; and for a cylindrical tube it will be somewhere between. These all give a value less than 10^8 s^{-2} for ^{3}He at any realistic density. By contrast we observed $M_2 = 2.2 \times 10^9$ s^{-2}, indicating a different source of relaxing magnetic fields. The observed M_2 would arise from paramagnetic impurities with a mean spacing of order 2 nm.

On subsequent increases in ^{4}He coverage the $T_1 \propto \omega^{1/2}$ dependence is preserved, as shown in Fig. 2. The conventional expression for $1/T_1$ is [30]

$$\frac{1}{T_1} = J(\omega) + 4J(2\omega) \tag{1}$$

when the relaxation occurs through motional averaging of internuclear dipole fields. Here $J(\omega)$ is the Fourier transform of $G(t)$, the autocorrelation function of the local field $b(t)$, the fluctuating local magnetic field seen by a representative nuclear spin as it moves around. Thus

$$G(t) = \gamma^2\langle b(0)b(t)\rangle, \tag{2}$$

where the angle brackets denote an ensemble average. At $t = 0$ it follows that $G(0) = \gamma^2\langle b^2\rangle = M_2$, and $G(t) \to 0$ as $t \to \infty$. While the short-time behavior of $G(t)$ is dependent on the detailed dynamics of the spins, the long-time behavior is determined purely by the macroscopic hydrodynamic behavior. And very generally, in this limit, $G(t) \sim t^{-d/2}$ for diffusion in d dimensions [31]. As a consequence of the Fourier transform relation between $J(\omega)$ and $G(t)$ it follows that the low frequency T_1 is determined by the hydrodynamics of the motion. This allows us to take a simple model for the correlation function in one dimension: $G(t) = M_2$ for $t < \tau_c$ and $G(t) = M_2\sqrt{\tau_c/t}$ for $t > \tau_c$. Here τ_c, the correlation time, is essentially the time for a spin to travel a distance over which its local field is correlated.

In the low frequency limit ($\omega\tau_c \ll 1$) we obtain from Eq. (1)

$$\frac{1}{T_1} = (1 + 2\sqrt{2})\sqrt{2\pi}\frac{M_2\tau_c^{1/2}}{\omega^{1/2}}. \tag{3}$$

A similar analysis in two dimensions gives T_1 approximately proportional to frequency [32], and in three dimensions it varies as $A + B\omega^{1/2}$.

However in our system relaxation occurs as a single-particle process: the relaxing fields are not the dipole fields of other ^{3}He nuclei. In that case the double-frequency term in Eq. (1) is absent and the corresponding expression for T_1 is

$$\frac{1}{T_1} = \sqrt{2\pi}\frac{M_2\tau_c^{1/2}}{\omega^{1/2}}; \tag{4}$$

this is similar to Eq. (3), but with a different numerical prefactor.

We emphasize that the emergent $\omega^{1/2}$ dependence of the low frequency T_1 follows solely from the hydrodynamics of 1D diffusion; it is independent of the details of the relaxation process [33]. In the present measurements this arises from classical one-dimensional diffusion, similar to the 1D diffusion of water in nanotubes previously observed by NMR relaxometry [34].

Fitting the data in Fig. 2 to Eq. (4) we obtain values for $M_2\tau_c^{1/2}$ as a function of coverage. At $n_4 = 1.12n_1$ we obtain $M_2\tau_c^{1/2} = 1.5 \times 10^5$ s$^{-3/2}$ at 1.7 K. Equation (4) is valid in the low frequency limit ($\omega\tau_c \ll 1$), and given that our highest Larmor frequency is 240 kHz this implies $\tau_c \ll 6.63 \times 10^{-7}$ s, and at this coverage $M_2 \gg 1.9 \times 10^8$ s^{-2}. This is consistent with relaxation by magnetic impurities as observed in the pure ^{3}He experiments [26].

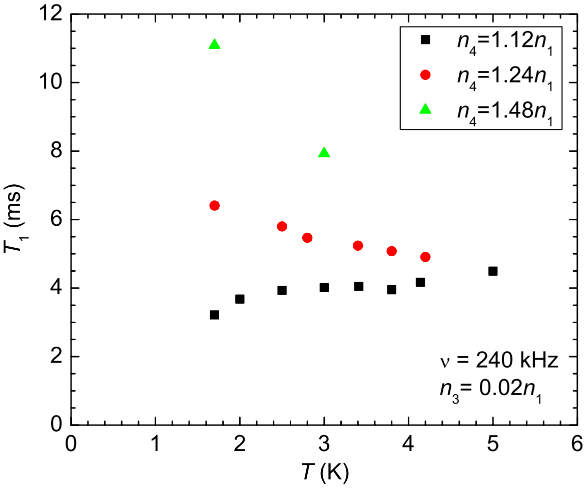

FIG. 3 (color online). Temperature dependence of T_1 for $n_3 = 0.02n_1$ and $\nu = 240$ kHz for various ^{4}He coverages.

As the ^{4}He coverage is increased the observed relaxation times increase implying a reduction in $M_2\tau_c^{1/2}$. The temperature dependence of T_1 at various ^{4}He coverages is shown in Fig. 3 at a frequency of 240 kHz. At the higher ^{4}He coverages T_1 increases with decreasing temperature. Interpretation of the temperature and coverage dependence of T_1 requires a knowledge of both the location of the ^{3}He in the film and the dynamics of the spins.

We can account for the observed behavior in terms of intermixing of the two isotopes. We model the helium in the pores as a two-component system: those atoms sitting in the localized surface layer and those occupying the quantum states within the pore. The proportion of ^{3}He spins in the surface layer depends on both temperature and ^{4}He coverage. The observation of a single relaxation time suggests rapid interchange of spins between these two components. We expect that the T_1 relaxation time will be shorter in the surface layer than in the mobile pore states. If we assume that this is the dominant relaxation mechanism then the effective T_1 is simply given by $1/T_1^e = f/T_1^s$, where f is the fraction of ^{3}He spins in the surface layer and T_1^s is the surface layer relaxation time. Both increasing ^{4}He coverage and decreasing temperature reduce f leading to an increase in the effective T_1. Of course T_1^s itself may have a dependence on these quantities, which complicates the analysis.

At low millikelvin temperatures, deep in the quantum degenerate regime, the smaller surface binding energy of ^{3}He arising from its higher zero point energy, will lead to preferential adsorption of ^{4}He in the surface layer. It should therefore be possible, by careful tuning of the ^{4}He coverage, to fill the surface layer with ^{4}He, leaving a mobile system of ^{3}He in the azimuthal and radial ground states, hence creating a one-dimensional system of tuneable line density. Measurements in this temperature range will allow a study of the relaxation times without the complication of interlayer exchange and an investigation of the ^{3}He dynamics in the 1D quantum state.

Our work demonstrates the potential of SQUID NMR on ^{3}He in nanotubes to investigate strongly correlated fermions in one dimension. We have resolved high quality NMR signals from the relatively low spin density samples, necessary to enter the quantum one-dimensional ground state. This opens the way to low temperature measurements of the ^{3}He nuclear magnetic susceptibility; the ratio of this quantity to the expected linear in T coefficient of the heat capacity determines the Wilson ratio, which in turn directly yields the TLL parameter [35,36]. This quantity can be studied as a function of ^{3}He line density. For comparison the Wilson ratio in bulk and 2D ^{3}He, fixed by the Landau parameter F_0^a, is close to 4 and only weakly density dependent, interpreted as evidence for the almost-localized fermion model [19,37,38].

$T_1(\omega)$, in the limit $\omega\tau_c < 1$, will determine the TLL diffusion coefficient independent of the details of the relaxation mechanism. This is of interest in the light of controversy over the the nature of spin diffusion in the $S = 1/2$ Heisenberg spin chain, a spin TLL [39–41]. Although we have established that intrinsic ^{3}He nuclear dipole-dipole spin-lattice relaxation is currently masked by the present level of magnetic impurities in the nanotube wall, the potential of relating the observable ^{3}He nuclear spin relaxation and nonlinear spin dynamics to the characteristic spin correlation functions of the TLL [42] requires further investigation. We anticipate that the effective exchange interaction between ^{3}He neighbors, which arises from particle permutations, will be a strong function of pore diameter and hence the structure of the wave function transverse to the pore axis. This will control the transition between a spin coherent and spin incoherent TLL [43].

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