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Low temperature adsorption of ^{3}He on silica aerogel surface and its influence on ^{3}He spin kinetics

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Abstract. Significant influence of the aerogel surface heterogeneity on the processes of ^{3}He nuclear magnetic relaxation at temperatures 1.5 – 4.2 K is discovered. This influence appears, for instance, in differences of the ^{3}He T_1 relaxation times for small portion of ^{3}He, adsorbed at different temperatures. Binding energy data of ^{3}He and distributions of this energy in two types of aerogel were obtained experimentally. Adsorbed ^{3}He molecules with binding energies 60 – 250 K play supreme role in processes of nuclear magnetic relaxation of gaseous and liquid ^{3}He in aerogel.

1. Introduction

Supreme role of ^{3}He layer adsorbed on an aerogel surface in processes of nuclear magnetic relaxation was studied earlier [1,2]. The spin kinetics of ^{3}He in the silica aerogel was studied above the Fermi temperature of liquid ^{3}He. The magnetic relaxation times T_1 and T_2 of adsorbed, gaseous, and liquid ^{3}He in the 95 % porosity aerogel at a temperature of 1.5 K were obtained by means of pulse nuclear magnetic resonance techniques. It was found that T_1 in all three cases is proportional to the frequency, whereas T_2 is frequency independent. It was proposed that the longitudinal relaxation proceeds due to the exchange motion in the solid adsorbed ^{3}He film. The intrinsic relaxation mechanisms in the liquid and gas phases are much weaker than the relaxation through the adsorbed surface layer. A theoretical model of relaxation in the adsorbed ^{3}He layer has been proposed. This model takes into account the filamentary structure of the aerogel.

In the present work we report experimental data on heterogeneous adsorption potential of ^{3}He in powder silica aerogel and in bulk aerogel (95%) with filamentary structure. The influence of this heterogeneous potential on magnetic relaxation of ^{3}He was also observed.

2. Experimental setup and samples

The aerogel samples were placed in glass tube (pyrex) which was tightly connected with ^{3}He gas handling system. For removing the impurities from the aerogel surface the samples were annealed at temperature 800 K during 30 min under continuous pumping.

The temperature of the NMR cell with aerogel samples has been controlled by ^{4}He vapor pumping and was measured by Allen-Bradley thermometer (10 Ohm resistance at room temperature). The pressure was measured at room temperature part of the gas handling system, using pressure gauge ILMVAC PIZA 111.

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The longitudinal magnetization relaxation times T_1 of ^{3}He was measured by the saturation-recovery method using FID signal. The spin-spin relaxation times T_2 was measured by Hahn method. The handmade pulse NMR spectrometer has been used (frequency range 3 – 50 MHz). The pulse NMR spectrometer is equipped by resistive electrical magnet with a magnetic field strength up to 1 T. Two types of aerogel were studied: silica aerogel (95%) with filamentary structure and powder aerogel (EMP-SAP (silica aerogel fine powders) EM-POWER CO. LTD (Korea)). Transmission Electron Microscopy images (TEM) of the samples are presented in figure 1 and figure 2. The particles size of powder aerogel is about 1 - 10 mkm.

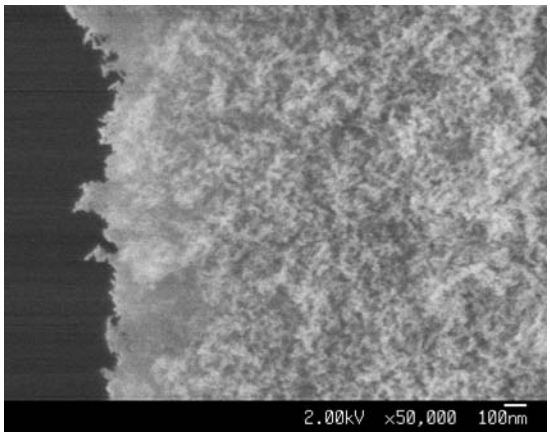

Figure 1. TEM of silica aerogel with filamentary structure

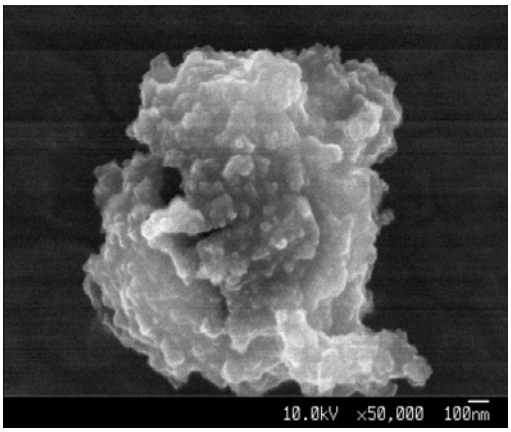

Figure 2. TEM of powder silica aerogel

3. Experiments

Adsorption isotherms of ^{3}He and ^{4}He in an aerogel are presented in figure 3. These isotherms were obtained by following method: at the temperature T = 4.2 K small portions of gaseous ^{3}He were introduced into the cell, which were further thermalized during about 30 min [3] and after that the equilibrium pressure was measured. After each step a new portion of gaseous ^{3}He was introduced. The adsorption capacity of complete layer was measured and was equal to 4 cm^3 for bulk aerogel and 12 cm^3 STP for powder aerogel.

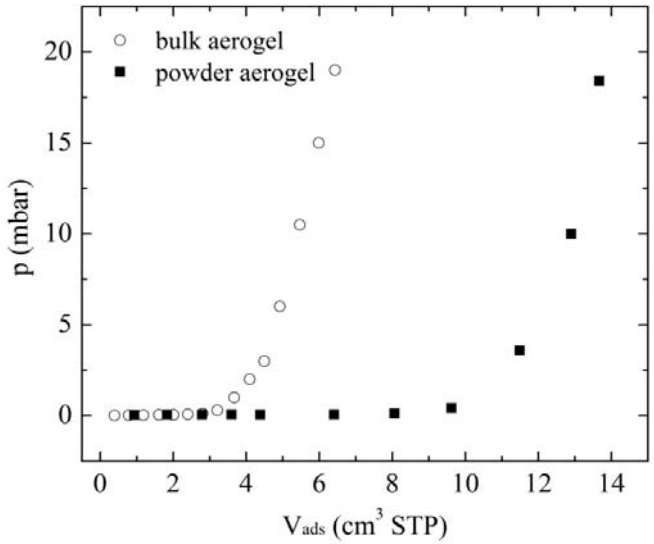

Figure 3. Adsorption isotherms of ^{3}He in bulk and powder aerogel samples at temperature 4.2 K.

In previous works [1,2] the spin kinetics of ^{3}He in the silica aerogel was studied above the Fermi temperature of liquid ^{3}He for complete adsorbed layer. In the figure 4 experimental data of ^{3}He nuclear longitudinal relaxation time T_1 for incomplete adsorbed layer (V_{ads}=0.8 cm^3 STP for bulk aerogel and 2 cm^3 STP for powder aerogel) are presented as a function of the temperature. The adsorbed layer was prepared at two different temperatures (1.5 K and 4.2 K) and as can be seen from figure 4 the behavior in these cases is completely different for both aerogel samples. It can be explained by taking into account heterogeneity of adsorption potential of ^{3}He on aerogel surface since adsorption of ^{3}He at different temperatures in these cases will fill the surface different manner.

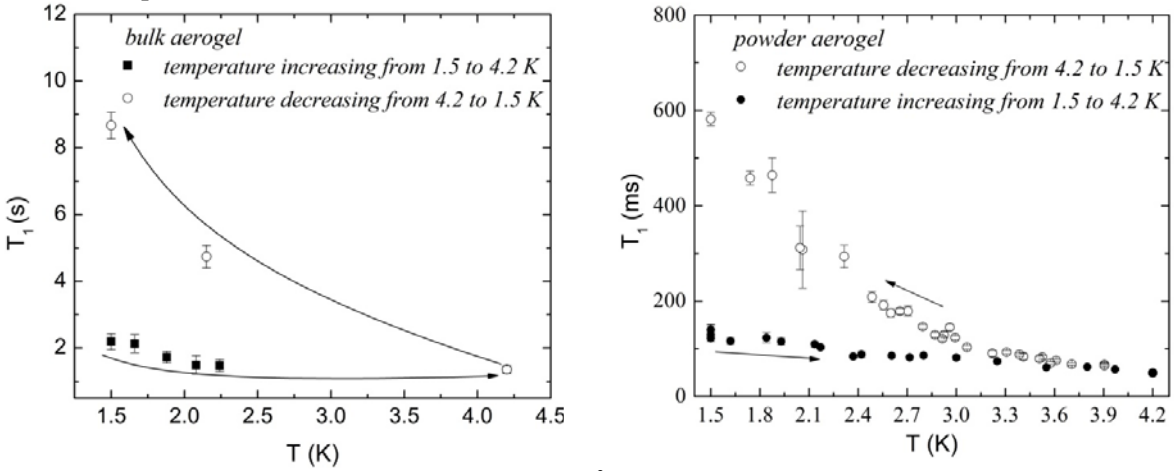

Figure 4. The temperature dependences of ^{3}He nuclear longitudinal relaxation time T_1 for incomplete adsorbed layer. The adsorbed layer was prepared at two different temperatures (1.5 K and 4.2 K).

For obtaining ^{3}He adsorption data and calculation binding energies of ^{3}He on the aerogel surface following method was used. At room temperature whole gas handling system together with NMR cell was filled by gaseous ^{3}He in amount of V(^{3}He) = 12 cm^3 STP (first experiment) and V(^{3}He) = 24 cm^3 STP (second experiment) in case of powder aerogel and 4.7 cm^3 STP in case of bulk aerogel. The whole gas handling system consists of "cold part" (capillary in cryostat and NMR cell) and "hot part" (external capillary, calibrating volume and a pressure gauge). The amount of ^{3}He was chosen, taking into account figure 2. After equilibrium pressure in whole system was achieved the cryostat begun cool down slowly (1 K/min at temperatures above 10 K and 0.05 K/min at low temperatures), using helium flow system. The cooling process leads to pressure decreasing in the system because of two processes: gas cooling in a "cold part" and adsorption on the aerogel surface. The experimental data are presented in figure 5. If we will take into account the redistribution of helium gas between "cold" and "hot" parts of the system during the experiment, the dependence of ^{3}He adsorbed amount versus the temperature could be obtained. In the figure 5 results of ^{3}He adsorption in powder aerogel in case of V(^{3}He) = 24 cm^3 STP are presented.

It can be seen that adsorption continues at lower temperatures, less than 4 K. The case of adsorption experiment with V(^{3}He) = 24 cm^3 and measurements of ^{3}He longitudinal relaxation times T_1 during ^{3}He adsorption experiment in powder aerogel in the temperature range 1.5 – 4.2 K are presented in figures 6 and 7.

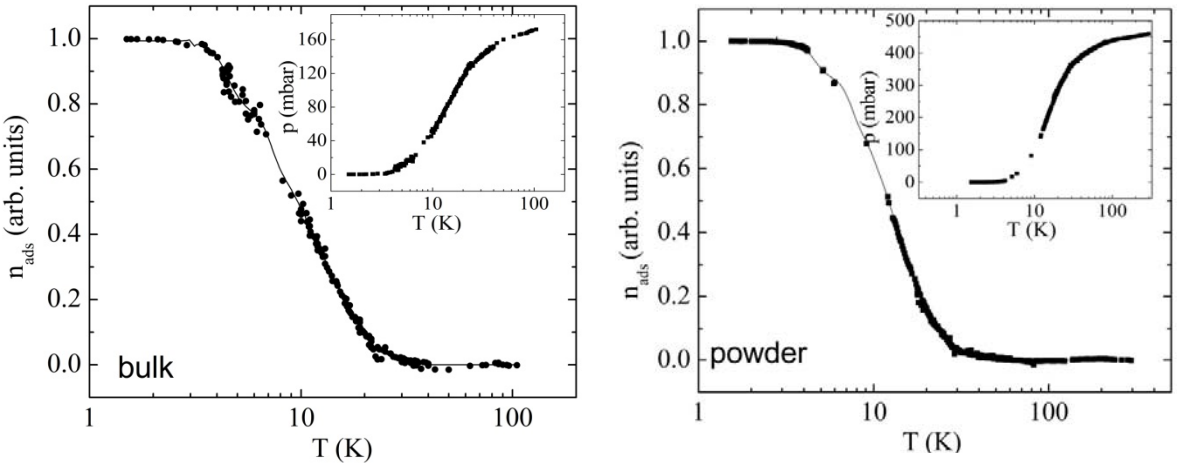

Figure 5. Temperature dependences of ^{3}He amount adsorbed in aerogel: dots – experimental data (V(^{3}He) = 4.7 cm^3 STP -experiment with bulk aerogel, V(^{3}He) = 12 cm^3 STP- experiment with powder aerogel), lines – Langmuir model (see text). On inserts shown the p(T) dependences.

From figure 7 it is clear, that additional amount of ^{3}He in the cell (case of V(^{3}He) = 24cm^3) plays the role of the "additional load" for relaxation process and does not have significant intrinsic relaxation mechanisms. The experimental data can be described, using HR model [4]:

$$T_1 = T_{1s} \cdot N_2(T) / N_1 \qquad (1)$$

where T_{1s} – ^{3}He longitudinal relaxation time in adsorbed (surface) layer. N_2 – amount of ^{3}He molecules in the NMR cell, which depends on temperature. N_1 – amount of ^{3}He molecules in adsorbed layer (V(^{3}He) = 12 cm^3 STP). The solid line in figure 7 represents calculations, taking into account equation (1) and redistribution of gaseous ^{3}He between "cold part" and "hot part" at low temperatures.

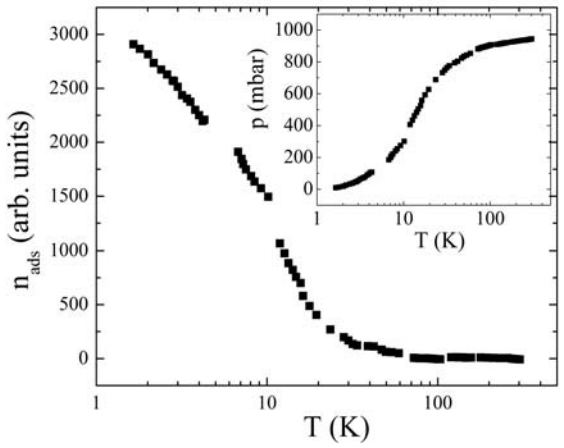

Figure 6. Temperature dependence of ^{3}He amount adsorbed in powder aerogel in case of V(^{3}He) = 24cm^3 STP.). On insert shown the p(T) dependence.

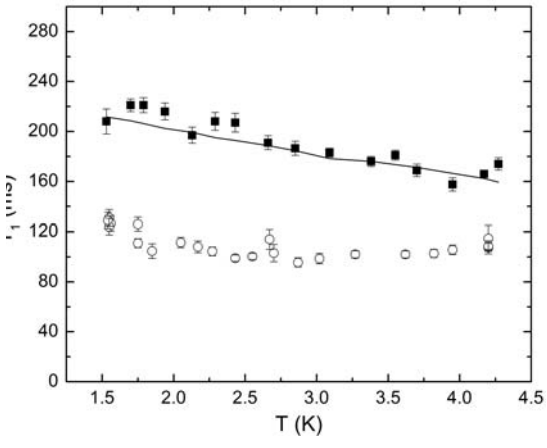

Figure 7. The temperature dependences of ^{3}He longitudinal relaxation time T_1 in powder aerogel: ○ – V(^{3}He) = 12 cm^3 STP, ■ – V(^{3}He) = 24cm^3

The binding energies for adsorption experiments (V(^{3}He) = 4.7 cm^3 STP (bulk aerogel) and V(^{3}He) = 12 cm^3 STP (powder aerogel)) below monolayer capacity can be estimated from Langmuir model [5] by eq.:

$$\theta = p/(p + p^*), \qquad (2)$$

where Θ – degree of layer filling

$$p^* = \alpha T^{5/2} / \left\langle \exp[-H^S / kT] \right\rangle, \quad (3)$$

where α – constant, T – temperature, H^S – Hamiltonian of ^{3}He interaction with an aerogel surface. In the case of low temperatures it can be rewritten as:

$$\left\langle \exp[-H^S / kT] \right\rangle \approx \exp[-\varepsilon_m / kT] \quad (4)$$

where ε_m – ^{3}He binding energy on aerogel surface.

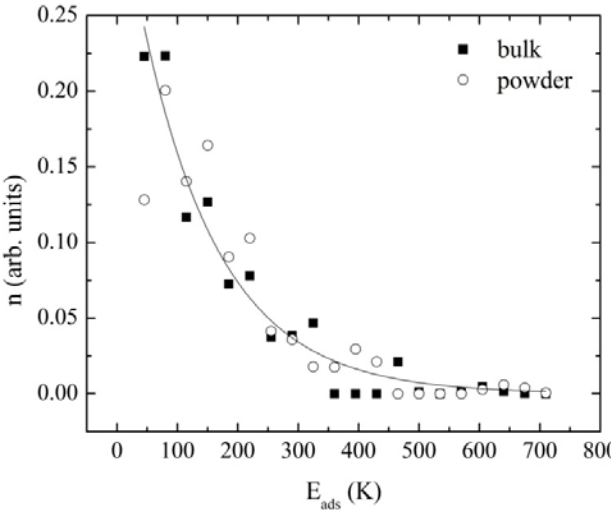

Figure 8. Binding energy distributions of ^{3}He in two types of aerogel

The experimental data on figure 5 was fitted by twenty processes like (2) with binding energy lies in range 45-700K (figure 5). As can be seen from figure 8 observed distributions are similar and can be characterized by exponential function with characteristic parameter 130±11 K.

4. Conclusions

Considering all presented experimental data, significant influence of aerogel surface heterogeneity on the processes of ^{3}He nuclear magnetic relaxation at temperatures 1.5 – 4.2 K was described. For instance, this influence appears in differences of ^{3}He T_1 nuclear relaxation times for small portion of ^{3}He, adsorbed at different temperatures. Binding energy data of ^{3}He on the surface of powder silica aerogel obtained experimentally. It was shown that the binding energy is in the range of 60 – 250 K. Adsorbed ^{3}He molecules with binding energies in range 60-250 K plays dominant role in processes of nuclear magnetic relaxation of gaseous and liquid ^{3}He in aerogel.

Acknowledgements

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References

[1] Klochkov A V, Kuzmin V V, Safiullin K R, Tagirov M S, Tayurskii D A and Mulders N 2008 *JETP Lett.* **88** 823
[2] Klochkov A, Kuzmin V, Safiullin K, Tagirov M, Yudin A and Mulders N 2009 *J.Phys.: Conf. Ser.* **150** 032043
[3] Alakshin E M, Gazizulin R R, Klochkov A V, Kuzmin V V, Mulders N, Tagirov M S and Tayurskii D A 2011 *JETP Lett.* **93** 223

[4] Hammel P C and Richardson R C 1984 *Phys. Rev. Lett.* **51** 1441
[5] Steele W A 1970 *J. Low Temp. Phys.* **3** 257