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FRP Dewar for measurements in high pulsed magnetic fields

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ABSTRACT

We describe a liquid helium glass-fibre reinforced plastic (G-FRP) Dewar which we designed, fabricated and tested for excitation spectrum measurements in high pulsed magnetic fields of up to 50 T. The sensitivity of high-resolution magnetic measurements carried out at low temperatures in such high fields is limited inevitably by magnetic and electric properties of the structural Dewar materials involved. Magnetic properties of various G-FRP Dewar-purpose materials are explored with a χ-meter furnished with a RF SQUID magnetometer. The Dewar materials and multilayer insulation effects contributing to the magnetic response signal are analyzed. It has also been discovered that field noise caused by the magnetization of the Dewar materials can be suppressed substantially by using special glass–epoxy technologies. The liquid helium evaporation rate is 3.8 l/day while the hold time is 27 h, the influencing factors are discussed.

1. Introduction

High magnetic fields of 25–50 T act on both the spin and the orbital motion of charged particles quantizing states and localizing free carriers on nanometer scale (see e.g. [1] for the technique of low-temperature multi-frequency electron spin resonance). This allows characterization of the ground state, the symmetry and time reversal properties of many systems. Simultaneous use of the high magnetic fields and the low temperatures provides one with a powerful tool for changing the quantum properties to generate new states of matter, to cause dimensionality cross-over in physical processes and to induce quantum phase transitions. In order to test samples in magnetic fields by different research methods, a variety of special-design Dewar vessels operating in 1.5–4.2 K temperature range has been developed [2,3] solen. However, under influence of pulsed magnetic fields of about 50 T, magnetic and electric properties of the Dewar structural materials could dominate in affecting the sensitivity of magnetic measurements. Firstly, the pulsed magnetic fields dramatically increase the magnetization of vessel materials thus contributing additionally to the sample response signals. Secondly, electric fields caused by high-rated variation of the magnetic field induce electric currents that bring about various undesirable electrical, thermal and mechanical effects.

Family of G-FRP Dewars is the most traditional and effective solution for cooling systems intended for material tests to minimize Johnson noise, thermal electromotive forces and local eddy currents. However, no remarkable systematic study of magnetic field noise effects due to magnetization of Dewar materials in high-amplitude magnetic fields has been undertaken before. The obtained signals are typically smeared out and the measurement resolution tends also to deteriorate with use of conventionally designed FRP Dewars whose vacuum space of the vessel tail is blanketed with classic multi-layer insulation and armored by vapor-cooled metal-string thermal shields. In order to create an apt low-noise Dewar for magnetic excitation spectrum measurements, one should focus on the magnetic properties of relevant materials and the design alternatives [4]. The present paper is dedicated to a detailed analysis of low-temperature magnetic properties of diverse G-FRP materials and is concentrated mainly on the question of how to minimize the effect of magnetization of the Dewar components. Particular attention was paid to the design of thermal radiation shields and multi-layer insulation blankets of Dewars, with aim to reduce eddy currents and Joule overheating [5].

2. Design and electrical properties of FRP Dewar

Fig. 1 shows the detailed sketch of the FRP Dewar with its overall sizes indicated and the scheme of a Dewar-equipped setup. The setup is intended for testing samples of various materials in pulsed magnetic fields produced by a liquid-nitrogen-cooled solenoid. The tail outer diameter is 18 mm, i.e. by 2 mm less than the conjugated inner diameter of the solenoid (see Fig. 1). The Dewar tail is designed as a combination of two coaxially aligned G-FRP tubes with

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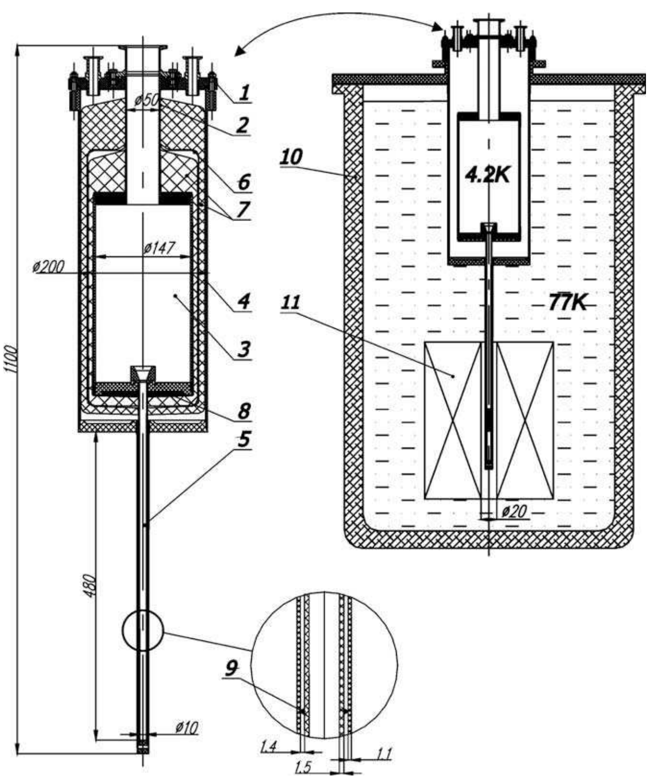

Fig. 1. Schematic drawing of a measurement setup with FRP Dewar intended for EPR investigations in high pulsed magnetic fields up to 50 T. Here is: 1 – upper flange with evacuation vacuum facility and safety valve; 2 – 160 mm-long neck; 3 – main 4.3-l liquid helium tank; 4 – outer jacket; 5 – tail for centering cooled samples (max. 10 mm in size) inside solenoid; 6 – thermal-radiation shield; 7 – multi-layer insulation blanket; 8 – cryogenic sorption pump; 9 – thin centering FRP rims; 10 – liquid nitrogen bath; 11 – LN_2-cooled solenoid with port diameter of 20 mm.

wall thicknesses of 1.5 mm and 1.1 mm, correspondingly, whereas vacuum space between them is only 1.4 mm (inset in Fig. 1). The tail length is 500 mm that guarantees fairly large distance between the main Dewar tank and the pulsed magnet. Coaxiality of these tubes along their entire length is provided by mechanical adjustment when gluing together the Dewar parts, their position being fixed by two thin centering FRP rims (9) to space the parts. It should be noted that absence of a multi-layer insulation inside vacuum spacing of the tail (Fig. 1), on the one hand, inevitably leads to an increase in heat inflow to the Dewar but, on the other hand, totally eliminates eddy-currents generated heating.

With such a design, the heat flux Q caused by irradiation from the wall with temperature $T_1 = 77$ K to the low-temperature tail part having temperature $T_2 \leqslant 4.2$ K can be estimated [6] as follows:

$$Q = \varepsilon_n \cdot \sigma_{SB} \cdot (T_1^4 - T_2^4) \cdot S_1 \tag{1}$$

where ε_n is reduced emissivity for two FRP cylindrical surfaces, $\sigma_{SB} = 5.67 \times 10^{-8}\ \mathrm{W\,m^{-2}\,K^{-4}}$ is Stefan-Boltzmann constant for black body irradiance, S_1 is 77-K surface area. Assuming $\varepsilon_n \approx 0.8$, we get $Q \approx 46$ mW.

Since the main liquid helium tank is about 0.5 m away from the solenoid, the stray magnetic field is essentially reduced as $H(z) \approx H_0/(r^2 + z^2)^{3/2}$ in this region. Here r is the solenoid radius and z is a typical distance from its center. This factor allows one to use a blanket of modified super-insulation in this part of the Dewar, along with a sorption pump to evacuate the vacuum space [7], and a thermal shield to reduce radiation heat inflow to the helium storage tank. To suppress eddy currents [8] induced by the pulsed magnetic field, the double-side aluminum (~100 nm thick) coating on the *Mylar*® surface was mastered out surgically in so a manner that the typical size of the metal pieces were 1–3 mm wide, whereby the electrical specific resistance of the sheet was estimated as $\rho > 0.1\ \Omega\,\mathrm{m}$. For the same sake, the thermal radiation shield (mounted at the Dewar neck in the 65 K area) was composed of a set of vertically laid 0.5 mm dia copper wires. All wires of the shield were electrically isolated from each other to prevent circular currents. The super-insulation blanket (compiled of 15 layers of 12 μm-thick *Mylar*® sheets interspaced equally by 8 μm-thick basalt paper sheets) were placed between radiation shield and walls of the helium tank. Vacuum spacing between the radiation shield and the outer jacket was filled with another 15 layer-blanket of the same super-insulation composition, with average winding density of 2.6 layers per millimeter [9]. It should be noted that efficiency of the radiation shield is deteriorated by gaps between the wires. Moreover, emissivity of every layer of super-insulation becomes incremented by about 20% due to damages of the reflecting aluminum film of *Mylar*® sheets. Yet these two factors, however, do not practically contribute too much to the total heat inflow rate since the outer Dewar jacket is situated in liquid nitrogen bath. Heat inflow along the neck with a heat-insulating plug was evaluated as being approximately 30% of the total heat power input. Liquid helium evaporation rate from the Dewar becomes steady in an hour after the Dewar filling and is about 3.8 l/day (~ 100 mW), thus being determined by almost 50% due to heat inflow to the tail part.

3. Magnetic properties of FRP materials

Magnetic susceptibility of G-FRP materials $\chi(T)$ was measured by an RF SQUID-magnetometer [10] within temperature interval $T = 4.2$–100 K and at magnetic fields ranging from 5 mT up to 3.5 T. The temperature determination accuracy was within ±0.25 K while that of magnetic field was ±0.5%. The χ values were calculated using the equation

$$\chi = A \cdot M_s \cdot H^{-1} \cdot m_s^{-1} \cdot \rho \tag{2}$$

where A is the SQUID-magnetometer constant in $A\,\mathrm{m}^2\,\mathrm{V}^{-1}$ (S.I. units), M_s is a signal measured in Volts which is proportional to the sample magnetic moment, H is the strength of magnetic field in $A\,\mathrm{m}^{-1}$ and m_s is the sample mass in kg, ρ is the sample material density in $\mathrm{kg\,m^{-3}}$ determined by hydrostatic weighting.

Fig. 2 represents the magnetic susceptibility $\chi(T)$ as a function of temperature in 5 mT field for three materials. The first one is winded G-FRP material of the Dewar main tank which is produced

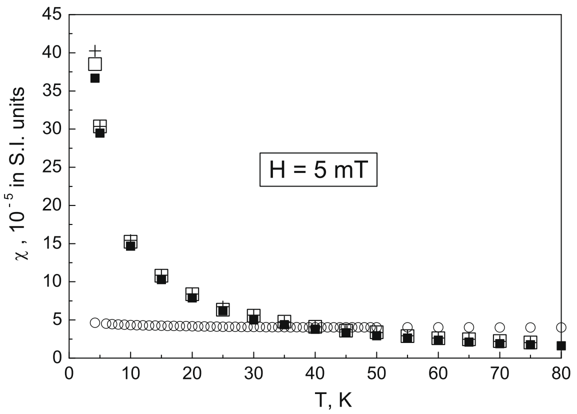

Fig. 2. Magnetic susceptibility vs. temperature for three types of FRP materials: ■ – stamped G-FRP sheets, □ and + – material of the Dewar cylindrical wall, where the magnetic field is applied in the glass-fibers plane and normal to it, respectively; ○ – composite with boron-nitride filler.

with Epikote 828 resin (similar to Soviet-GOST epoxy-diane resin ED-22), isomethyl-tetrahydrophthalic anhydride (iso-MTHPA) by HEXION^{TM} Specialty Chemicals and electric insulating glass cloth E3/1-100 (specification by Soviet GOST 19907-83). The other is a stamped G-FRP material used to fabricate all plate parts, glass textolite epoxy–phenolic (GTEP-1), analogous to G10 sheet material. The sharp increase of $\chi(T)$ below $T \leqslant 10$ K witnesses for existence of paramagnetism in these materials caused presumably by presence of ferromagnetic impurities. Third tested material is a composite based on epoxy-diane resin ED-20 (specification by Soviet GOST 10587-84) and polyamidoimide resin hardener L-20 with boron-nitride powder as a filler in a mass ratio of components 3:2:3.

The assumption about ferromagnetic impurities is confirmed by field dependence $\chi(H)$ for a sample of the first above-described material measured at 4.2 K in fields of up to 3.5 T (Fig. 3). At a preset temperature, $\chi(H)$ curve evidently goes through its maximum at a magnetic field of about 0.1 T that is typical for ferromagnetic materials. The maximum value of magnetic susceptibility of the tested G-FRPs at 4.2 K is $\chi \approx 4.5 \times 10^{-3}$ in S.I. units. Detailed studies of $\chi(T)$ of some binders and filler fibers have shown that the E-glass fibers were the main contributors to the magnetic susceptibility.

Fig. 4 exhibits the temperature dependencies $\chi(T)$ in 20 mT magnetic field for the two types of widely used fillers, glass fabric E3/1-100 and E-glass textile tape (specification by Soviet GOST 19907-83). One can see that their temperature dependencies and peak values of $\chi(T)$ are close to those obtained for the G-FRPs (see Fig. 2). This is an evidence for the principal influence of ferromagnetic impurities (e.g., Fe_2O_3) in glass-fibers on magnetism of the G-FRP materials. A slight difference between absolute values of both $\chi(T)$ and $d\chi/dT(T)$ observed for these two filler material types (Fig. 4) is presumably associated with diverse compositions of the impurities therein.

Fig. 5 demonstrates the temperature dependencies $\chi(T)$ for four compositions of epoxy impregnants (binders) and for a sample of Corning 8871 glass. As seen from the plots, the binders have weak temperature dependencies $\chi(T)$, with maximum $\chi \sim 10^{-5}$ at 4.2 K in dimensionless S.I. units, and are most probably diamagnetics. It is worth noting that such glasses as Corning 8871 (lead glass containing about 42% SiO_2 and 49% PbO with Li_2O, Na_2O, K_2O additives), have at 4.2 K value of $\chi(T)$ close to that of pure quartz fabric, and their paramagnetism can partly be compensated by diamagnetism of binder materials.

The most significant result is that, using fabric made of special sorts of glass, one can obtain G-FRP materials for Dewar tail with net magnetic susceptibility as low as $\chi(T) \approx (1,\ldots,3) \times 10^{-5}$ at $T = 4.2$ K. Additionally, these weak-magnetic materials are also capable of minimizing the spectral density of magnetic noise caused by the Dewar temperature fluctuations [11] which is proportional to $\partial\chi/\partial T$. The value of $\partial\chi/\partial T$ at low temperatures can be reduced by almost two orders of magnitude through a proper selection of the composite materials.

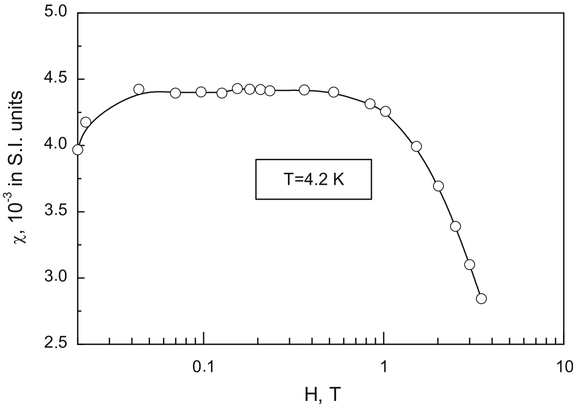

Fig. 3. Effect of magnetic susceptibility saturation within strong magnetic fields.

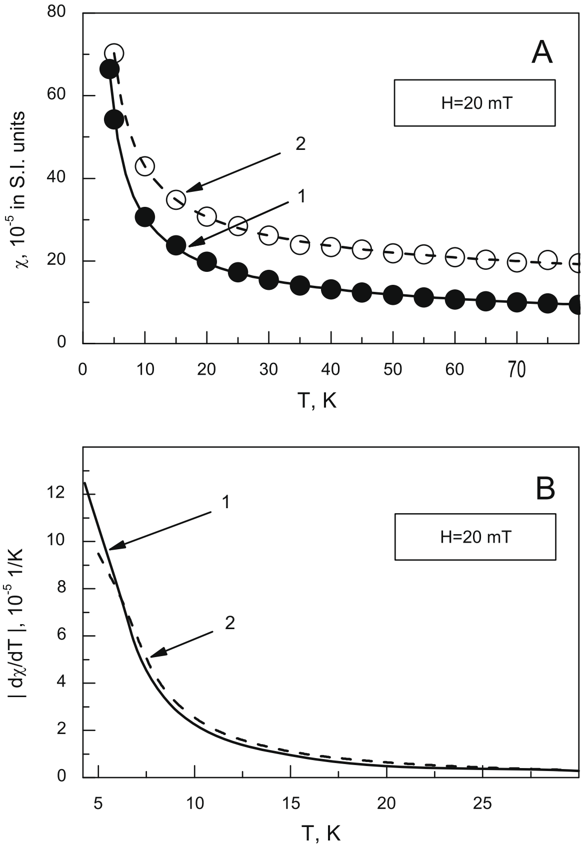

Fig. 4. A – magnetic susceptibility vs. temperature; B – derivatives $d\chi/dT$ as a functions of temperature for: 1 – glass fabric, 2 – glass textile tape.

Tests of cryogenic adsorbents have shown that magnetic susceptibility of activated charcoal at $T = 4.2$ K was almost by an order of magnitude higher than that of the synthetic graphite adsorbents. The synthetic expanded-type graphites with $\chi_{(T=4.2\ K)} \approx 5 \times 10^{-4}$ turned out to be the best among all the tested samples to evacuate the vacuum gap of Dewar. Since the sorption pump is far away

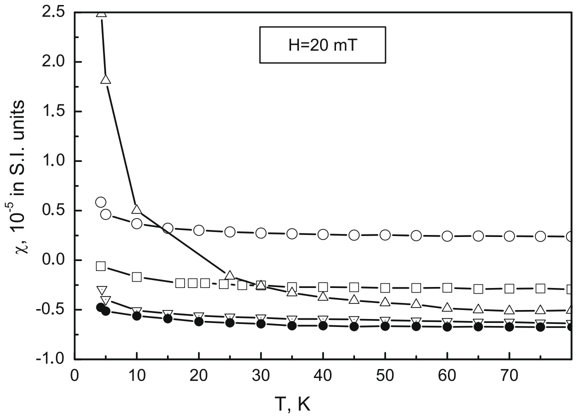

Fig. 5. Magnetic susceptibility vs. temperature for: ▽ – cryogenic adhesive based on epoxy-diane resin ED-20 and hardener L-20 prepared by special polymerization process (1 h at 120 °C, 2 h at 85 °C); □ – the same cryogenic adhesive, polymerized at room temperature; • – epoxy-diane resin ED-20 and iso-MTHPA (polymerization schedule – 1 h at 100 °C, 3 h at 120 °C, 7 h at 150 °C); ○ – Epikote 828 resin (epoxy-diane resin analogue to ED-22) and iso-MTHPA (polymerization schedule – 1 h at 100 °C, 3 h at 120 °C, 7 h at 150 °C); △ – bulk glass Corning 8871.

from the solenoid, its magnetic contribution to the signal of a sample-under-test is rather negligible.

4. Summary

The created prototype of 4.3-l liquid helium G-FRP Dewar enables one for investigating low-temperature properties of various substances in pulsed magnetic fields of up to 50 T. Operational time of the filled Dewar is more than 27 h. The inner diameter of the vessel tail is 10 mm. The liquid helium evaporation rate is mostly determined by the heat inflow to the Dewar tail (50%) and by the short length (160 mm) of the neck (30%).

The investigation of magnetic susceptibility as a function of temperature for some G-FRP materials, epoxy binders and various fillers has shown that, with optimal choice of the structural materials, the own magnetic moment $M(T) = \chi(T) \cdot H \cdot v$ of the Dewar tail (where v is a volume of its magnetized part), can be reduced by two orders of magnitude as compared to routinely used G-FRP materials. Moreover, the usage of such G-FRP materials essentially suppresses the spectral density of magnetic noise of the Dewar tail, which is caused by temperature fluctuations. We believe that special design of the thermal radiation shields and the multilayer insulation blankets used in the Dewar described in the paper will practically eliminate the field-induced eddy currents.

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