Design of a fibre reinforced plastic anticryostat for magnetorelaxometric measurements

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A new method for the characterization of magnetic nanoparticles is based on the analysis of the temperature dependence of the Néel relaxation sample signal. The presented cryostat extends the investigated temperature range from 300 K to 77 K down to the boiling point of liquid helium at 4.2 K. We designed an anticryostat to use only one liquid helium cryostat for the sample as well as for the Low-T_C SQUID. Therefore it is necessary to study the permeation process through the used fibre reinforced plastic (FRP) material and the adhesive joints between the components.

INTRODUCTION

The magnetic properties of ferrofluids are strongly influenced by the distribution density of energy barriers in these many particle systems [1]. The low signal level of the magnetisation relaxation demands the application of non-magnetic and electrical insulating materials due to prevent self-induced eddy currents within the cryostat walls. The high permeation constant of helium through any kind of synthetic material inhibits long measurement periods. It is necessary to characterise the permeation conductivity through the used FRP materials. Because of the competing influence of the heat conductance along the wall of the cryostat small wall thicknesses are useful. This results in a high pressure gradient and a high helium permeation through the wall material. This permeation process restricts the lifetime of the insulating vacuum.

EXPERIMENTAL

We created a permeation measurement set-up to characterise the gas transport due to the permeation process through composites used as wall materials. The basic arrangement of the whole measurement set-

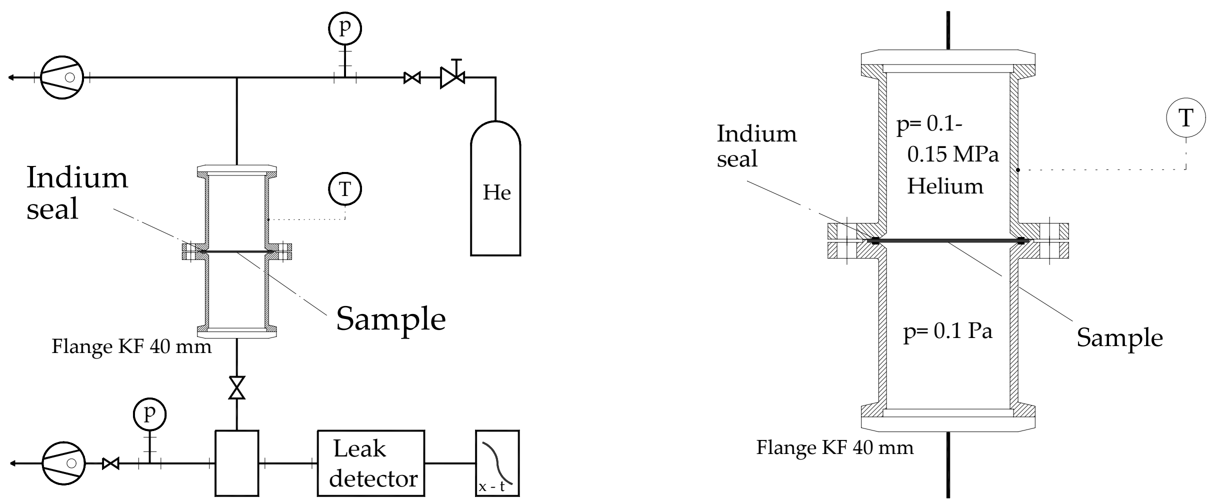

Figure 1 Permeation measurement assembly (a) and the permeation cell in detail (b)

up and the permeation cell are shown in Figure 1. We tested thin plates of various FRP materials with a diameter of 41 mm and a thickness between 1 and 1.5 mm depending on the different distributors. All permeation measurements have been made with helium. The permeation process is strongly influenced by the ratio of glass and epoxy resin, the glass composition and the reinforcement structure. In addition the effect of machining and the adhesive joint material must be taken into consideration. In order to compare these measurements we conditioned the same FRP-material with various adhesive and lacquer layers. We have used a commercial leak detector with a detection limit of $5 \cdot 10^{-11}$ $\mathrm{mbar} \cdot \mathrm{dm}^3/\mathrm{s}$ leak rate. The leak rate q_L corresponds to a p-V-current regarding to standard temperature and standard pressure (STP). The measured leak rate has to be standardised to an area of A = 1 m^2 and a pressure gradient across the wall thickness of $\Delta p / \Delta x = 1\mathrm{bar}/1\mathrm{mm}$ [2]. The temperature dependence of the permeation can be described by an Arrhenius equation [3] (see Equation 2):

$$q_{Perm} = q_L \cdot \frac{\Delta x}{A \cdot \Delta p} \tag{1}$$

$$q_{Perm} = q_{Perm,0} \cdot exp\left(-E_P/RT\right) \tag{2}$$

With q_{Perm}: permeation conductivity, E_P: activation energy of permeation, R: gas constant, T: absolute temperature. Hence it is sufficient to study the permeation process from 290 K to 310 K (see Figure 2). The cold parts of the cryostat at a wall temperature of about 4.2 K can be neglected because the permeation values approximately decrease by a factor of 10^{10} already at the liquid nitrogen temperature of 77 K [4,5].

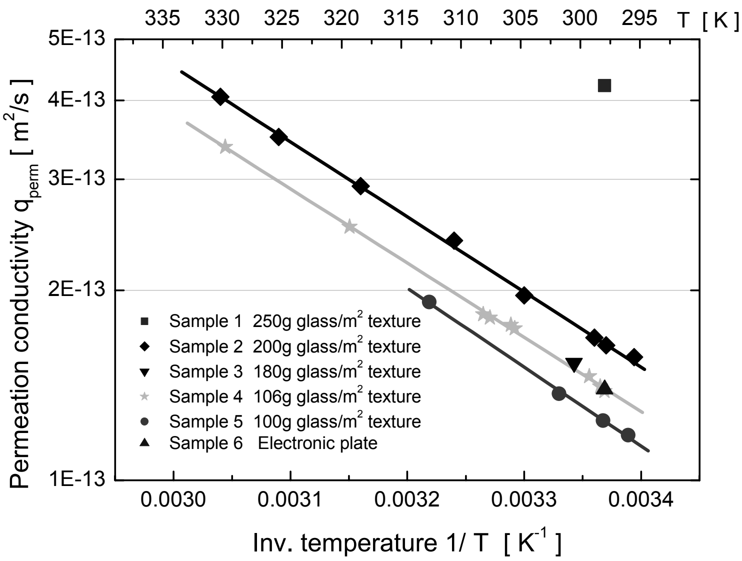

Figure 2 Temperature dependence of the helium permeation for different FRP materials

The legend in Figure 2 explains the amount of glass at every inserted texture inside the epoxy resin. The higher the amount of glass per square meter the coarse is the glass texture. Every FRP sample has consequently the same glass fibre volume inside the epoxy resin (55 %) except sample 1. This sample additionally consists of a higher glass partition of about 69 % fibre volume.

In a further step we studied the influence of the adhesive joints and of one first lacquer film coating (see Figure 3). In order to investigate the gas permeation through adhesive joints we conditioned one 200 g glass/m^2 sample with 100 holes having a diameter of 1 mm and filled them with adhesive. The calculated value of the adhesive material is shown in Figure 3. The permeation value is four times higher than the untreated FRP material. The same 200g glass/m^2 sample was used as a substrate of the deposited lacquer films. Film thicknesses below 0.05 mm have no influence on the permeation value. The result is an identical permeation amount compared to the untreated sample.

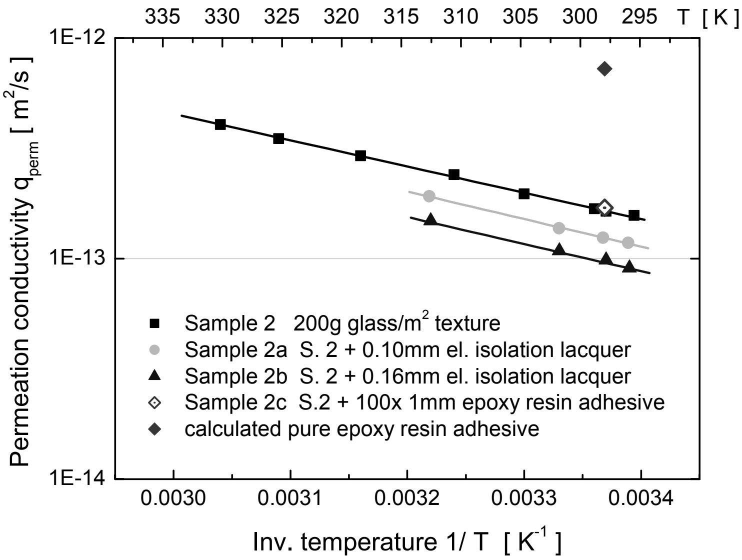

Figure 3 Modification of the helium permeation by coating methods and adhesive joints

RESULTS AND DISCUSSION

By means of the designed permeation gauging set-up we are able to investigate the influence of the surface coating and adhesive joints at different FRP materials. The thinner the glass texture the lower is the permeation value q_{Perm}. Adhesive joints have to be dimensioned five times wider along the pressure difference direction to compensate the higher permeation value. Machined surfaces and adhesive joints can be additionally coated with thin lacquer films to decrease gas permeation. That requires a deposited film thickness greater than 0.1 mm. In consideration of all these results we designed an anticryostat that extends the measuring range below 77 K down to the boiling temperature of liquid helium (see Figure 4).

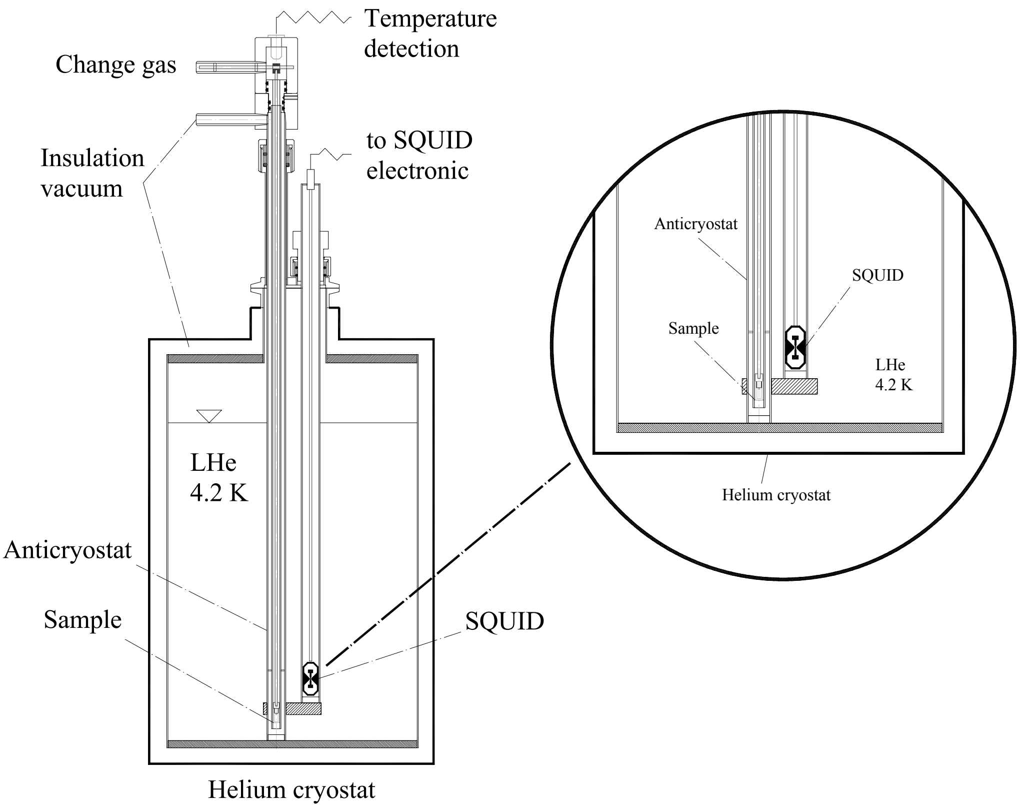

Figure 4 Relaxation measurement set-up including the anticryostat

First measurements were accomplished with a ferrofluid consisting of Maghemit (γ-Fe_2O_3)-particles whereby a temperature of 77 K confines the measurement range to a particle size above 15 nm. The maximum of these distributions moves to lower temperatures with decreasing particle size. Figure 5 shows the energy barrier density distribution of γ-Fe_2O_3 versus the temperature. The maximum at 13 K corresponds to a particle size of about 7 nm. That's why measurements below 77 K are of physical note.

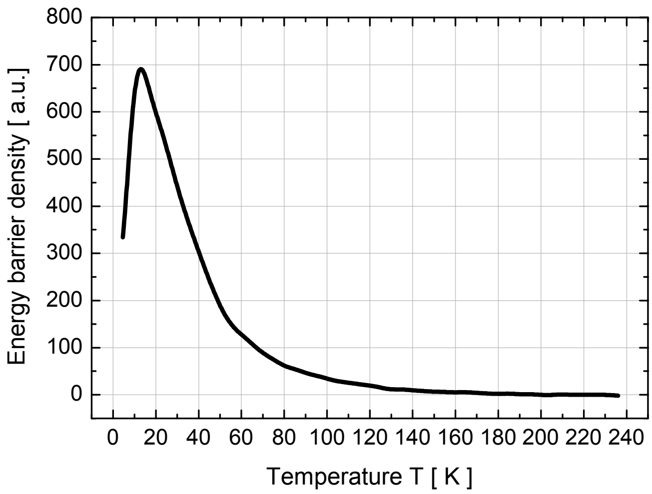

Figure 5 Energy barrier density distribution of γ-Fe_2O_3 versus temperature

CONCLUSIONS

We designed an FRP anticryostat for magnetorelaxometric measurements with a minimum distance between the SQUID-gradiometer and the sample position. The experimental set-up has been tested successfully and a first particle size distribution at about 7 nm is presented.

To improve the lifetime of FRP helium cryostats other coating materials and methods have to be tested. The mechanical properties especially the adhesive strength down to 4.2 K limits the material variety. There are eligible candidates among these, like lead borate glasses and lead borosilicate glasses. The films will be sputtered as surface layers with a thickness of more than 5 µm.

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