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LABORATORY TECHNIQUES

Parameters Optimization of the FRP Dewar Intended for Biomagnetic Investigations

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Abstract—We represent the results of studies on noise-generating sources in glass-fibers reinforced plastic (G-FRP) Dewars as the provision units of operation environment for Superconducting Quantum Interference Detectors (SQUID). Techniques of noise suppression to a level admissible in up-date biomagnetic investigations are proposed. Novel concepts of combined thermal radiation shield and low-magnetic FRP structures reduce the level of proper noise for such Dewars to be ~5 fT/Hz$^{1/2}$. Detailed studies on penetrating of gaseous helium are undertaken for various composited materials of Dewar parts. The developed combined FRP structures for Dewar necks demonstrated that diffusion coefficient of gaseous helium can be lowered by 30–100 times in comparison with that for traditional FRP materials.

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1. INTRODUCTION

Sensitivity of the techniques that used SQUID sensors and operate in a special shielded room is deteriorated by proper noise of FRP Dewars [1–5]. One portion of spectral density $S_1(\omega)$ of this noise is related to electromagnetic fields induced by conductive elements of Dewar's thermo insulation due to heat fluctuations (known as Johnson–Nyquist thermal noise), to temperature gradients and to variations of residual fields in the shielded room. The other part $S_2(\omega)$ is determined by properties of structural materials of Dewar vessel. Magnetic susceptibility $\chi(T)$ of these materials sharply increases with lowering of temperature and its strongly nonlinear paramagnetic behavior at about $T \approx 4.2$ K leads to intensification of magnetic field variations δB, whose level is proportional to temperature fluctuations δT even in static magnetic field $H = \text{const}$, such as $S_2^{1/2}(\omega) \sim (\partial\chi/\partial T)H\delta T$.

High degree of resolution aimed for implementation of biomagnetic investigations, requires one to use DC SQUID-based detectors, and this implies that proper summarized noise of the FRP Dewar should never exceed sensitivity thereof:

$$S^{1/2}(\omega) = \sqrt{S_1(\omega) + S_2(\omega)} \approx (3\text{–}5) \times 10^{-15}\ \text{T/Hz}^{1/2}. \quad (1)$$

One of powerful sources of noise generation in biomagnetic Dewars are the layers of super insulation. Let us imagine an unlimited metal plate that has electric conductivity σ and thickness t, being at temperature T. Here, its low-frequency component of noise, in perpendicular-to-surface direction, can be represented [4] as:

$$S_B^{1/2} = \frac{\mu_0}{2}\left(\sigma t \frac{k_B T}{2\pi}\right)^{1/2} Z^{-1}, \quad (2)$$

where μ_0 is magnetic permeability of environmental space; k_B is Boltzmann constant; Z is distance to noise generating point. On assuming for electric conductivity $\sigma \approx 6 \times 10^7\ (\Omega\ \text{m})^{-1}$ of aluminum coating on Mylar sheet (being a film thickness $t \approx 100$ nm and taking into account that total blanket of Dewar's superinsulation consists of 50–80 layers), the low-frequency part of noise can be evaluated as high as $S_B^{1/2} \approx 100$ fT/Hz$^{1/2}$. As is shown in [6], such the value of spectral density of noise can be lowered to an appropriate level (1) by carving the aluminum film into a number of pieces free from any electric contact in-between. Such pieces can be described like magnetic dipoles which moments being proportional to noise current I_N and to area s_d. So, the value of their magnetic moments decreases with dipoles diameter d as $m = I_N s_d \approx (\pi/4) I_N d^2$.

Since SQUID-based detectors are typically located in a close proximity to the FRP bottom of biomagnetic Dewars, it suffices to minimize noises from underbody of the cryogenic vessel. Typically, specific outside diameter of FRP Dewars intended for multi-channel cardiograph systems makes up 100–200 mm, whereas spacing between two bottom surfaces (one cooled to

$T_1 = 4.2$ K and the other situated at $T_2 = 300$ K) is only ~5–10 mm. In so narrow vacuum gap, one can only lodge a thin package of about $n \approx 15$ layers of superinsulation with average density of 3 layers per millimeter. Due to such the package of superinsulation, power of heat inflow to the Dewar Q due to thermal irradiation through the vessel-bottom, makes up

$$Q \cong 5.7\varepsilon_{av}s_p[(T_1/100)^4 - (T_2/100)^4](1/n). \quad (3)$$

Assuming that the area of the bottom part is $s_p \approx 10^{-3}$ m^2 and value of average reduced emissivity is $\varepsilon_{av} \approx 0.05$, power of heat inflow due to (3) has to be about 70–100 mW. Therefore, in order to retain the helium evaporation rate at $v \approx 1$ l/day ($Q \approx 28$ mW), usual practice provides an application of addition thermal-radiation shield. In order to keep the undesirable noises depressed it is fabricated in a set of narrow metallic strips of copper or aluminum. But introduction of surplus amount of metal will inevitably increase the Johnson noise spectral density up to 10–12 fT/Hz$^{1/2}$ [6, 7]. Moreover, presence of metal in vicinity of a near-bottom located detecting loop of the gradiometer is a critical factor for misbalancing the sensors at high frequencies. The above-mentioned problems have been overcome in the Dewar under discussion, owing to substitution of metallic thermal shield with electronic heat conductivity η_e by a dielectric one with phonon heat-conductivity, possessing of outstanding value $\eta_{ph} \geq \eta_e$.

Usually, value of $S_2(\omega)$ for Dewars can be reduced by active electronic stabilization of helium-bath temperature, resulting in high coefficients of temperature fluctuation (δT) suppression (40–50 dB). This paper considers an alternative mechanism of reducing the spectral density of noise $S_2(\omega)$ due to decrease of value $\partial\chi(T)/\partial T$ for the Dewar bottom material.

In present-day there still remains one of FRP Dewar engineering problems—diffusion of helium gas molecules through the vessel-neck walls. Helium penetrated into vacuum cavity of the Dewar and increased the heat inflow rate [8] caused by growth of its partial pressure between layers of superinsulation. Gas diffusion coefficient K and permeability depend on properties of material and tend to increase with elevation of temperature [9], such as:

$$K = K_0\exp(-H_a/(RT)), \quad (4)$$

where K_0 is a constant for a specified material, m^2 (s Pa)$^{-1}$; H_a is energy of diffusion activation, which depends on both properties of absorbing material and on diffusing atoms, J mol^{-1}; R is universal gas constant, J (mol K)$^{-1}$; T is absolute temperature, K. In account on nature of temperature distribution inside operated Dewar from 5 to 290 K along neck length, fabrication of neck parts requires application of relevant materials with minimum heat conductivity and low gas permeability. Below, measurement results of helium penetration through FRP necks and a concept of creating the vessel-necks with minimized helium gas permeability are presented.

2. COMBINED THERMAL-RADIATION SHIELD AND DIPOLE SUPERINSULATION

Detailed study in [7] has shown that retarding the rate of liquid helium evaporation with application of copper–based thermal shield in the Dewar raises an ever problem of generating noise in bottom area. For example, a 1-mm-thick and 30-mm in diameter disk of thermal shield produces the value of spectral density of magnetic noise in a 5 mm distance greater than 30 fT/Hz$^{1/2}$, exceeding notably the limit specified by (1). Figure 1 relates to the magnetic encephalography-purpose Dewar and is to illustrate measuring signals of noise spectral density on frequency for two magnetic field components $B_Z(\omega)$, $B_X(\omega)$, whereby axis Z coincides with vertical axis of the Dewar vessel. Measuring point is at 15-mm distance from the thermal shield. In this construction thermal shield is made from inter-insulated 10-mm-wide copper strips. With such the concept of design, noise level at about 1 Hz frequency makes up 50–100 fT/Hz$^{1/2}$.

Paper [10] reports that with purpose to suppress Johnson noise in biomagnetic Dewars, authors chose to substitute metallic thermal radiation shield by dielectric one characterized by remarkable phonon-provided heat conductivity. In the range of thermal shield working temperatures ($T \approx 70$–80 K), high heat conductivities η_{ph} = 200–600 W (m K)$^{-1}$ are demonstrated by high-purity synthetic products, such as single-crystal and polycrystalline aluminum oxide. Figure 2 represents a concept of combined-type thermal shield of a magnetic encephalography Dewar, whose structure involves a set of ~0.5-mm plates of polycrystalline alumina. Individual plates are bonded together by an epoxy adhesive compound with a high thermal conductivity, whereas heat-conducting metal parts are fixed to this construction along its perimeter. Structural design of the high-purity synthetic single-crystal alumina-based thermal-shield, η_{ph} = 1500 W (m K)$^{-1}$, is shown in Fig. 3. In this structure, the critical space between outer vessel-surface situated at 300 K and the interior dielectric thermal shield is filled with nine layers of superinsulation [8, 11], including Mylar sheets bearing a bilateral 100-nm-thick coating of aluminum film. Space between the vessel-wall cooled to $T = 4.2$ K and the thermal shield is packed with six layers of the same sort of the superinsulation. Aluminum coating on every Mylar sheet on both surfaces has been carved intently to obtain a multiple pieces of maximum size d

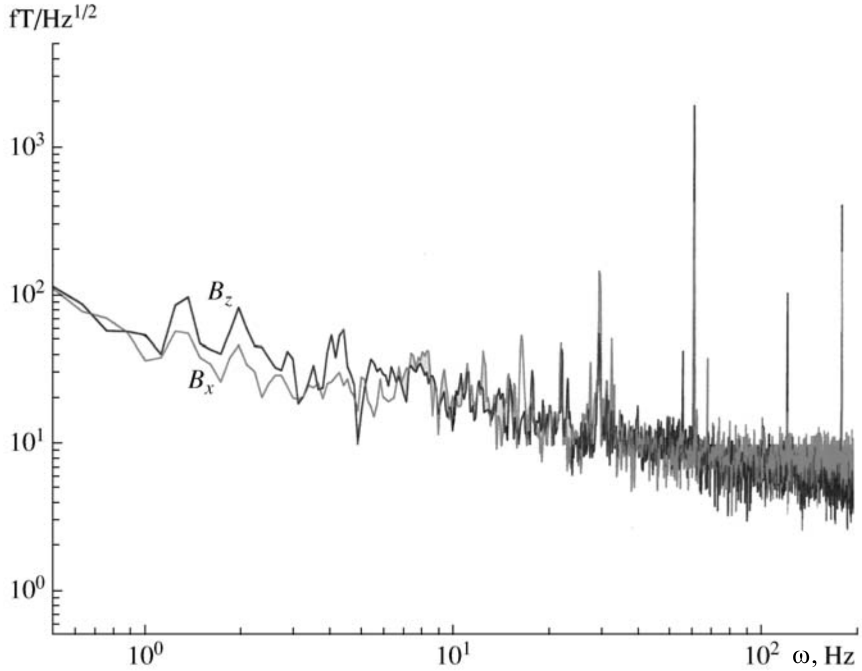

Fig. 1. Dependencies of noise spectral density on frequency for two magnetic field components $B_Z(\omega)$, $B_X(\omega)$ in a 100-channel FRP biomagnetic-purpose Dewar with metallic thermal shield. Measurements are made in a shielded room by DC SQUID detectors. At frequencies around 1 Hz, level of components' noise in vicinity of SQUIDs location makes up 50 fT/Hz$^{1/2}$.

≈ 1–3-mm. Since magnetic noise in faraway areas of the piece ($Z >> d$) is reduced in conformity with dipole as

$$S^{1/2}(\omega) \sim I_N d^2/(d^2 + Z^2)^{3/2} \approx I_N(d^2/Z^3),$$

performance of all fifteen layers of the superinsulation is in a good compliance with equation (1). It is evident that provision of pieces-dipoles of metal film is a key to reducing the superinsulation reflectivity. Due to results of measurements represented in Fig. 4 it's worth while noting that carving the aluminum film into pieces of $d \approx 3$ mm leads to ~5% decrease of the total reflectivity, whereas $d \approx 1$ mm ensures only 1–2% hereof [6]. Technologically, pieces-dipoles were carved out by method of electrical scribing of aluminum film on both sides of the substrate, so that edges of resultant aluminum strips on either side would not overlap their opposites and been in a regular shift. Thus elaborated structure of combined-type thermal shield with segment of phonon-based heat-conductivity placed at the bottom area, enables one to suppress Johnson noise to values specified by (1), with only small amount of heat-inflows tolerated to be let in through the Dewar bottom area, due to (3).

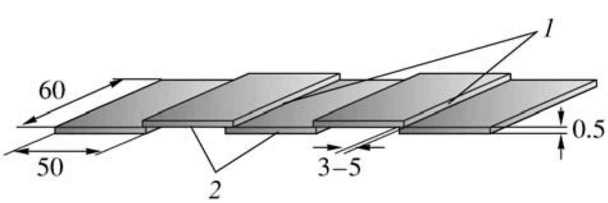

Fig. 2. Schematic view of a Dewar thermal shield with using of polycrystalline aluminum oxide plates (Al_2O_3). *1*—gluing joints, *2*—plates of polycrystalline alumina. All dimensions are in millimeters.

As stated before, value of noise component $S_2^{1/2}(\omega)$ can be suppressed by reducing the amplitude of variation $\{\partial\chi(T)/\partial T\}_{T = 4.2\,K}$. Our detailed studies have demonstrated [16] that in weak magnetic fields ($B <$ 5 mT), magnetic susceptibility of commercial G-FRP materials rests at the level of $\chi_{T = 4.2\,K} \approx 2.5 \times 10^{-4}$ and value of $\{\partial\chi(T)/\partial T\}_{T = 4.2\,K} \approx (20–25) \times 10^{-5}$ (K)$^{-1}$ (Fig. 5). For special-purpose structural composition within impregnate based on pure epoxy-dian resin typed ED-20 (Specification by GOST 10587–84) and filler of low-magnetic glass fibers typed Corning-8871 material (in volumetric ratio of polymer bond and filler as fifty-fifty), can reduce magnetic susceptibility to $\chi_{T = 4.2\,K} \approx 10^{-6}$ (dimensionless S.I. units). It is worth mentioning that in weak magnetic fields, low paramagnetic features of such glass fibers can, to some extent, be compensated for by diamagnetic momentum of a bond. Let's note that value of $\{\partial\chi(T)/\partial T\}_{T = 4.2\,K}$ for the above-mentioned special composite material, is by 25 times lower than that of common-used FRP materials. Utilization of such the "well-compensated" material at fabrication of Dewar

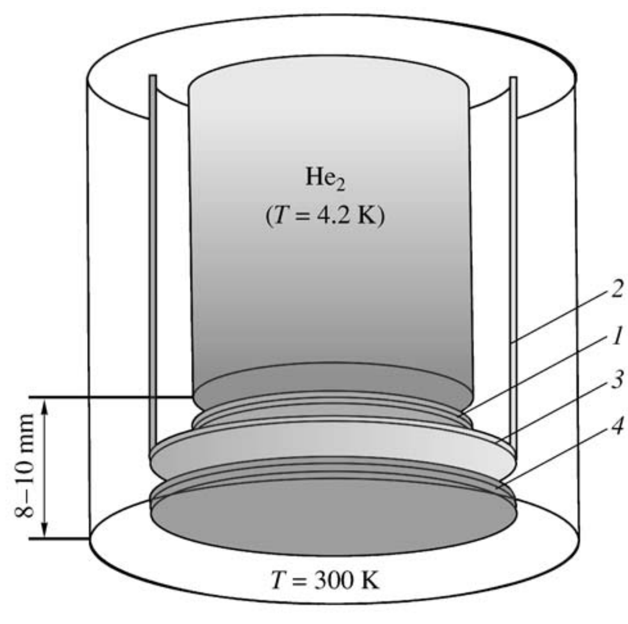

Fig. 3. Structural design of a thermal radiation shield involving parts produced with high-purity synthetic single-crystal alumina. *1, 4*—layers of superinsulation, *2*—cylinder of single-crystal alumina, *3*—disk of single-crystal alumina.

underbody segment is promising to reduce spectral densities of noise inflicted by temperature fluctuations. Moreover, application of materials with $\chi_{T=4.2\text{ K}} \approx 10^{-6}$ enables one to substantially improve symmetry of SQUID-based gradiometer sensor ($\partial B_z/\partial z$). Similar values of the integral susceptibility at $T = 4.2$ K and low amplitudes $\{\partial\chi(T)/\partial T\}_{T=4.2\text{ K}}$ are also typical for FRP compositions involving quartz filaments.

3. HELIUM DIFFUSION THROUGH FRP MATERIALS AND STRUCTURAL DESIGN OF DEWAR NECK

Within $T = 4.2$ to 300 K temperature range, integral heat conductivity of G-FRP materials $\eta(T)$ is 13 times lower than that of stainless steel. Moreover, cylindrical thin FRP shells have so strength characteristics that a Dewar neck of 150–200 mm inner diameter suffices enough with only $\delta \approx 1.5$-mm thickness of the wall. But due to critical permeability of such type shells for gaseous helium [12], neck wall thickness must usually be increased, up to 3–4 mm. If the neck part is of large diameter, this requirement will lead to undesirable increase of heat inflows to the Dewar. As a result, almost 50% of liquid helium evaporation rate is specified by heat inflow with thermal conductivity of the neck [13].

Figure 6 represents results of measurements for gaseous helium diffusion through different composite FRP materials. Parameter of gas permeability of composite materials has been evaluated by standard mass-spectrometry method [15]. As Fig. 6 has it, initial stage of the process is characterized by its nonlinear behav-

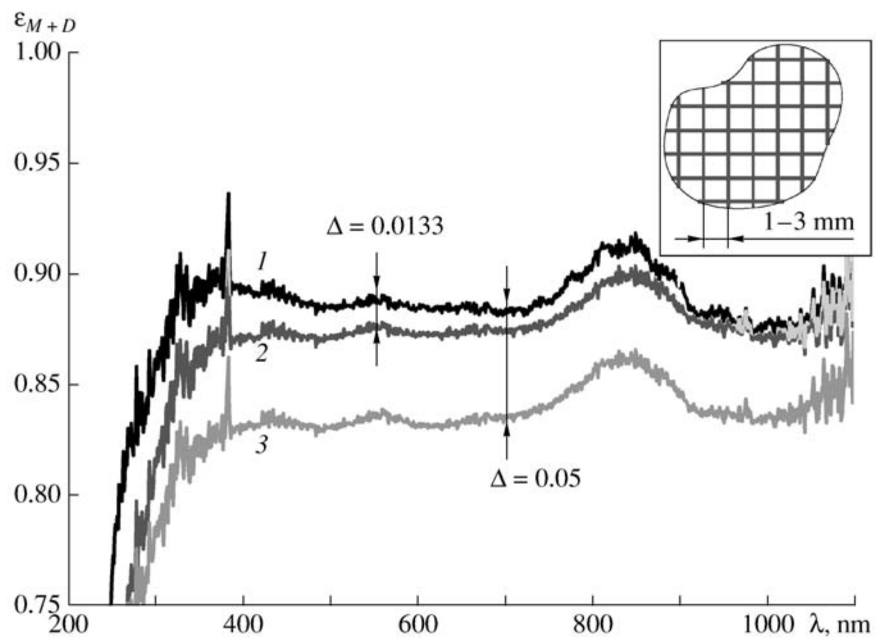

Fig. 4. Dependency of total specular and diffusive reflection on parameter of wavelength. Test-samples of aluminized Mylar film are represented as following: *1*—with the whole aluminum film remaining intact, *2*—aluminum film was carved to a set of individual 3-mm pieces, *3*—aluminum film was carved to a set of pieces of 1-mm in size. The box demonstrates a pattern of aluminum film carved in pieces.

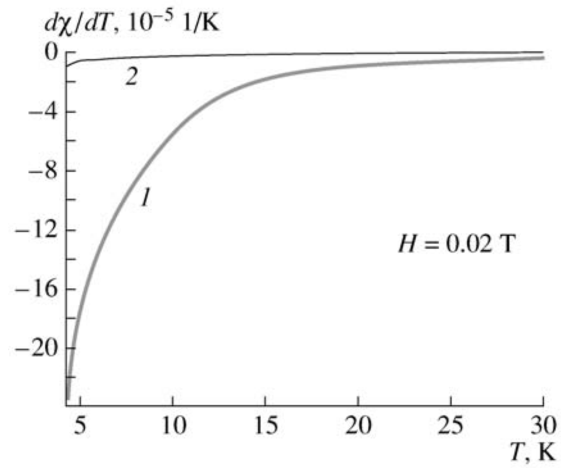

Fig. 5. Temperature dependencies of a derivative of magnetic susceptibility $\partial\chi(T)/\partial T$ for: *1*—traditional G-FRP material; *2*—"well-compensated" composite material based on ED-20 epoxy resin and low magnetized Corning-8871 glass fillers, in 0.02 T magnetic field.

ior. After definite time, the pressure in vacuum-processed volume at one side of test-sample starts to uniformly rise, thus denoting an onset of the stationary process. Time parameter of stable rise pressure setting is used in determination for coefficient of gas permeability. Thus implemented studies performed with a method of comparison, considering rates of helium inflow through cylinder-shaped test samples made of various composite materials. End faces of test samples were encapsulated, whereby inner volume was connected to TI1-14-type helium leak detector, and the whole test sample was placed into a test chamber under permanent helium gas pressure of 1.2 atm.

Definite sorts of glass (for example, such leaded glass typed Corning-8871, or F-3, F-4, F-5-type heavy flints) that contain PbO in their compositions possess low thermal conductivity $\eta_{T=300\text{ K}} \approx 0.75$ W (m K)$^{-1}$, along with exceptionally low permeability for gaseous helium, whereas diffusion coefficient is only $K \approx 4 \times 10^{-15}$ m^3 m (m^2 s Pa)$^{-1}$ [14]. These properties can be assumed for at elaboration of composite neck parts for FRP Dewars. Figure 7 represents a cross-section view of a composite neck wall, which enables one to sufficiently reduce gaseous helium diffusion into vacuum cavity of the Dewar without extra heat inflows. In this case the fabrication process of a composite neck part can be described such as: when 80% of FRP material is wrapped on a cylindrical mandrel then two layers of glass foil strips (~30-μm thick and w = 5–20-mm wide) are enclosed in wall material. In order to overlap gaps between adjacent strips second layer is applied with a half-width shift aside relatively to the first layer. With these techniques, path of diffusion track for

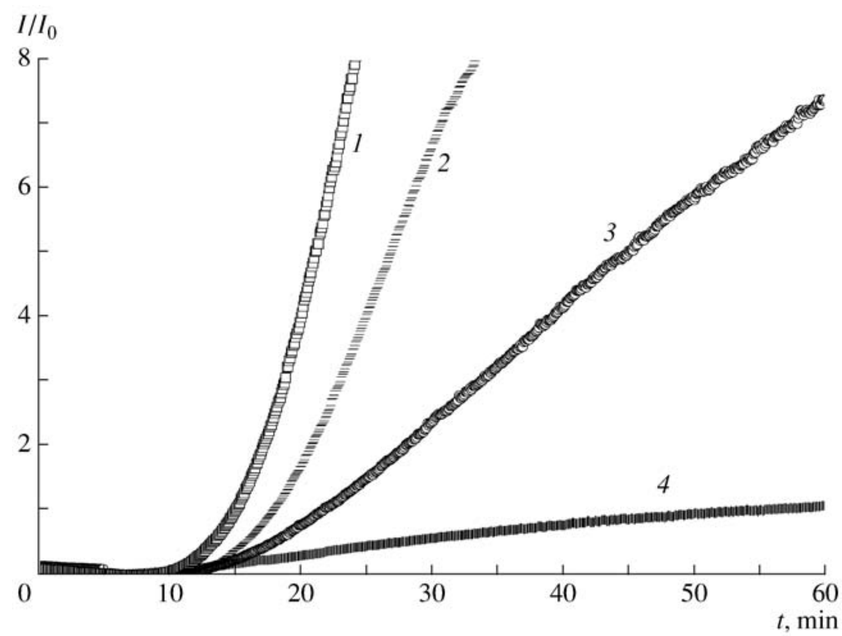

Fig. 6. Series of amount-vs.-time dependencies for effect of helium penetration through Dewar neck, with test samples made of FRP in compliance to: *1*—standard technology of FRP wrapping on; *2*—wrapping on after vacuum pre-impregnation of glass-fabric material; *3*—creating the composite material, complementing with one layer of 7-mm wide and 40-μm thick tape of F-4-type leaded glass foil; *4*—creating the composite material, complementing with one layer of 45-mm-wide and 100-μm-thick stainless steel foil tape. Indicated in relative units along the ordinate axis, there are represented reduced values of gaseous helium flow.

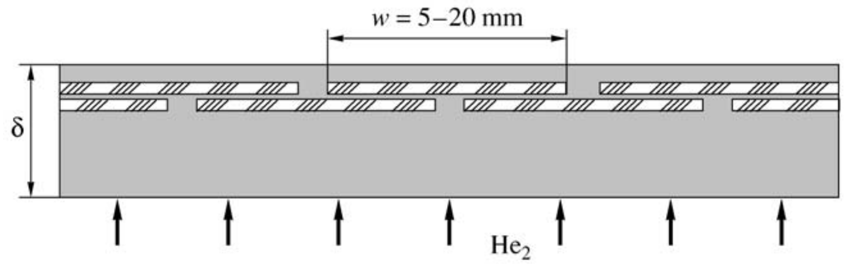

Fig. 7. Cross-sectional view of a composite 2-mm thick neck-wall with two layers of glass foil tape involved with purpose to retard the process of helium diffusion. Arrows indicate areas with partial pressure of gaseous helium. Rate of helium diffusion through the composite FRP wall is notably reduced by increase of effective length of diffusion path.

helium atoms is additionally increased, proportionally to glass strips-width. Series of dependencies, represented in Fig. 6, demonstrate a considerable reduction of helium diffusion, if at least one layer of metal or glass foil would be applied into the neck-part composite structure.

Measurements with a gas chromatograph due to methodology [15] have proven a decrease of helium diffusion coefficient from 3.2×10^{-9} m^3 m (m^2 s Pa)$^{-1}$ for *2* test-sample down to 5.7×10^{-11} m^3 m (m^2 s Pa)$^{-1}$ for *3* test-sample, see Fig. 6. These measurements were assisted by a Crystallux-4000M chromatograph designed by RPC Meta-chrom LTD (Russia) that possesses metrological sensitivity for hydrogen atoms at 2 ppm.

4. CONCLUSIONS

Spectral density of proper noise of biomagnetic FRP Dewars is reduced to 5 fT/Hz$^{1/2}$ owing to substitution of metallic thermal radiation shields by combined ones, with application of high-purity synthetic single-crystal alumina which possesses at 70 K considerable phonon provided heat conductivity. Here, the major contribution to noise generated near Dewar-bottom is produced by Johnson noise from a film of aluminum deposited onto a Mylar substrate. With purpose to minimize this drawback, whole aluminum coatings are carved into a number of electrically inter-insulated pieces being typically 1—3-mm in size.

Low frequency fluctuations of magnetic fields related to variations of plastic material temperature are effectively suppressed due to application of a novel G-FRP composition involving bond based on ED-20-type epoxy-dian resin and low magnetized glass fibers.

Decision to create the Dewar neck from the combined FRP composite material involving glass tape ensured a feasibility to lower the gaseous helium diffusivity by 50 times, and simultaneously to reduce the neck-wall thickness to an acceptable value of 2.0 mm.

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