

Measurement of outgassing rates of Kevlar and S-Glass materials used in torque tubes of High Tc Superconducting (HTS) Motors

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Abstract

Torque tubes in High Temperature Superconducting (HTS) motor transfer torque from superconducting field winding rotor to the room temperature shaft. It should have minimum heat conduction property for minimizing the load on cryo-refrigerator. Generally, these torque tubes are made with stainless steel material because of high strength, very low outgassing and low thermal contraction properties at cryogenic temperatures and vacuum conditions. With recent developments in composite materials, these torque tubes could be made of composites such as Kevlar and S-Glass, which have the required properties like high strength and low thermal conductivity at cryogenic temperatures, but with a reduced weight. Development and testing of torque tubes made of these composites for HTS motor are taken up at Bharat Heavy Electricals Limited (BHEL), Hyderabad in collaboration with Central Institute of Plastics and Engineering Technology (CIPET), Chennai and Indian Institute of Technology (IIT), Kharagpur. As these materials are subjected to vacuum, it is important to measure their outgassing rates under vacuum conditions before manufacturing prototype torque tubes. The present study focusses on the outgassing characteristics of Kevlar and S-Glass, using an Outgassing Measurement System (OMS), developed at IIT Kharagpur. The OMS facility works under vacuum environment, in which the test samples are exposed to vacuum conditions over a sufficient period of time. The outgassing measurements for the composite samples were obtained using pressure-rise technique. These studies are useful to quantify the outgassing rate of composite materials under vacuum conditions and to suggest them for manufacturing composite torque tubes used in HTS motors.

Keywords: HTS Motor, torque tube, Kevlar, S-Glass, vacuum, outgassing

1. INTRODUCTION

Every solid material contains gas particles in its bulk or on its surface, when it is produced or processed. When a solid material is put into vacuum, at least one surface of it gets exposed to the vacuum space and gas effuses from the solid material into the vacuum as a load to the vacuum pump. This phenomenon is referred to as "Outgassing". Materials when exposed to vacuum will outgas to varying degrees by physical desorption or evaporation from the surfaces. The outgassing rate of a solid is defined by American Vacuum Society as: "the instantaneous net amount of gas leaving the material per unit of time" [1]. The specific outgassing rate of a surface is expressed as outgassing rate per unit area with a unit of $\text{mbar l sec}^{-1} \text{cm}^{-2}$. Outgassing rates are particularly influenced by porosity of the sample, surface history such as exposure to moist atmosphere, baking, etc. This outgassing poses many difficulties in the design of vacuum systems for different applications. In the present study, this problem is arising from outgassing of torque-tube materials (Kevlar/S-Glass) used in vacuum cryostat of High Temperature Superconducting (HTS) motor.

In the recent years, a number of HTS motors were developed with high power density, light mass, more

compact and high efficiency [2-5]. In India, BHEL R & D, Hyderabad had already initiated research in developing a 200 kW indigenous HTS motor. A HTS motor has HTS magnets mounted on the rotor, cooled by a cryogen at 40 K and a conventional copper winding located on the air-core stator. The cryogen is supplied to the rotor via a cold medium transfer coupling system at the non-driven end. Two torque tubes (fashioned as a cylinder, illustrated in Fig. 1) connect the room temperature end shaft with the low temperature HTS magnet. The torque tube plays an important role in reducing the heat-in leak to the winding volume, supporting the weight of the rotor and transferring torque from the cold temperature end (such as the HTS rotor-magnet) to the ambient temperature end of the machine under all steady-state and transient conditions [6], [7]. With recent developments in composite materials, these torque tubes could be made of composites such as Kevlar and S-Glass, which have excellent mechanical and thermal properties at cryogenic temperatures [8]. Reduced outgassing from the torque tube in HTS motor cryostat is very important during the operation to achieve better ultimate vacuum and to reduce the heat-in-leak from ambient. Also, loss of constituents from the material due to outgassing under vacuum may also cause degradation in physical and mechanical properties. Hence there is a need to understand the outgassing characteristics of these Kevlar and S-Glass materials used in torque tubes.

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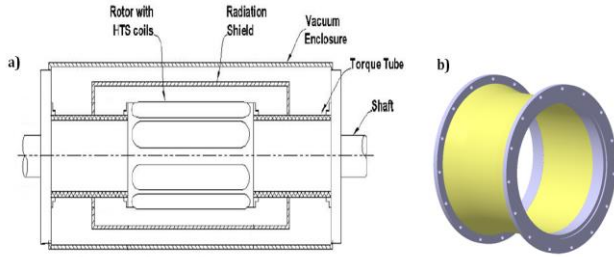


Fig. 1. (a) Schematic of HTS motor assembly, (b) 3D model of a composite torque tube.

However, studies elucidating the outgassing rates of Kevlar and S-Glass materials are not available in the literature till date, to the best of our knowledge.

An over-view on the activities of outgassing characterization using different methods is given here: In the literature, researchers have reported on the outgassing characteristics under vacuum conditions using various methods, such as the pressure-rise method, the throughput method and the weight-loss method [1]. G. L. Gregory [9] reported the outgassing rates of three gases in a vacuum chamber using pressure-rise method. Variable conductance method was employed by Berman *et al.* [10] to measure the outgassing properties of a vacuum system. The work of N. Yoshimura [11] gave insights into the measurement of outgassing rates of a solid material using differential pressure-rise method. Yang *et al.* [12] investigated the outgassing behaviour of a vacuum chamber by Twin-Gauge throughput method. An extensive review on the three common methods, namely the pressure-rise method, the throughput method and the switching between two pumping paths (SPP) for evaluating the outgassing properties of materials, is also available in the published literature [13]. In this paper, we have measured the outgassing rates of Kevlar and S-Glass material samples, which are chosen as the potential candidates for torque-tube construction of HTS motors using an in-house developed Outgassing Measurement System (OMS) by employing pressure-rise or rate of rise method. Furthermore, to analyze the outgassing gas species from the torque-tube materials under vacuum, residual gas analysis is also carried out in the present work.

2. EXPERIMENT

The outgassing behaviour of torque tube materials namely Kevlar – 49 and S-Glass – 10 samples were studied. Table 1 gives the description of the samples and their geometry. Fig. 2(a) and Fig. 2(b) give the photographs of samples of S-Glass and Kevlar placed inside the vacuum chamber.

TABLE I
COMPOSITE SAMPLES USED FOR OUTGASSING MEASUREMENT.

Material	Dimension (cm)	Surface Area (cm ²)	Shape
Kevlar 49	27 x 18 x 0.18	~ 980 cm ²	Sheet
S-Glass 10	27 x 18 x 0.18	~ 980 cm ²	Sheet

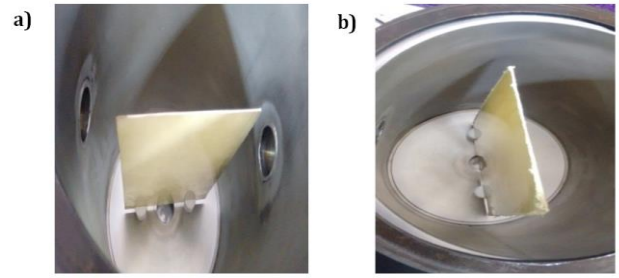


Fig. 2. (a) S-Glass sample placed inside test-chamber (b) Kevlar sample placed inside test-chamber.

2.1. Description of OMS

The schematic arrangement and the photograph of OMS experimental setup used in the present work for vacuum outgassing measurement are shown in Fig. 3 and Fig. 4 respectively. The test apparatus used in this investigation mainly comprises a stainless steel test chamber with a volume of 22 liters. The system is pumped down by a turbomolecular pump (Oerlikon-Leybold TURBOVAC 361) backed by a double stage rotary-vane pump (Oerlikon-Leybold TRIVAC D8B). Small ports on top of the chamber allow a bourdon-tube vacuum gauge (1013 – 100 mbar) and a commercially available Dual Gauge™ (Pfeiffer TPG 262: 100 – 10⁻⁷ mbar) to be mounted on the chamber for measuring the pressure. The test chamber can be isolated from the vacuum pumps by means of two isolation valves. For residual gas analysis of torque tube materials, Residual Gas Analyzer (RGA) from Pfeiffer vacuum was used. Prisma™ 80 Quadrupole Mass Spectrometer QMS - 200 having a mass range of 1 to 200 amu has been used to analyze and quantify the outgassing gas species from the torque tube materials. The RGA connected to the test chamber through a needle valve is pumped with a turbo-molecular pump resulting in a pressure of about $\leq 10^{-5}$ mbar. The needle valve allows outgassing gases to be sampled by RGA in a controlled manner. The whole experimental setup is maintained at room temperature throughout the experiment.

2.2. Measurement Procedure

Prior to the measurement, all the connections are made tight to ensure a leak-free system. Initially the blank test

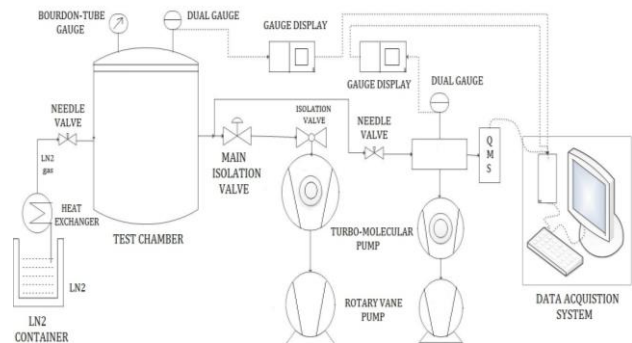


Fig. 3. Schematic of the vacuum system used for outgassing measurement of torque tube materials.

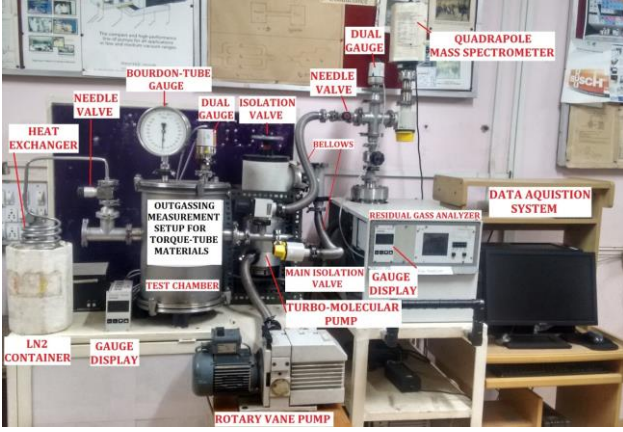


Fig. 4. Experimental setup for outgassing measurement of torque tube materials.

chamber (i.e. without the Kevlar/S-Glass samples) is pumped down by a rotary (backing) pump down to 10^{-2} mbar followed by a Turbo-molecular pump to 10^{-5} mbar. Upon attaining a steady-state value, main isolation valve was closed to isolate the test chamber from the vacuum pumps. After isolation, to measure the evolved gas quantity, the chamber's pressure-rise curve was obtained by measuring the pressure at different intervals of time (starting from $t = 0$) using the LabVIEW™ data acquisition program, interfaced with the vacuum gauges. The above experiment is repeated with the samples (without any surface treatment) of Kevlar and S-Glass placed inside the chamber. The difference in pressure-rise rates with and without sample is multiplied with the volume of the chamber (V) and divided by the surface area (A) of the sample, to get the outgassing rate (\dot{Q}) of the sample in the units of $(mbar\text{-lit}) / (sec\text{-}cm^2)$ using equation (1).

$$\dot{Q} = \frac{V}{A} \left[\left(\frac{dp}{dt} \right)_{\text{chamber with sample}} - \left(\frac{dp}{dt} \right)_{\text{empty chamber}} \right] \quad (1)$$

where $dp = P_1 - P_2$

Since the measurement is based on differential measurement, the effect of system's background leak rate is eliminated. The effect of residual moisture present in the system on measurement accuracy is decreased by baking the chamber at 200°C for 24 hours. To eliminate the memory effects due to outgassing of previous test sample placed in the chamber, the chamber was initially purged with pure nitrogen gas obtained from evaporating liquid nitrogen. Further, to establish the uniform test conditions, the chamber was baked at 200°C for 24 hours and blank measurements were performed between the sample measurements.

3. RESULTS AND DISCUSSIONS

Fig. 5 shows a comparative plot of pump-down characteristics of test chamber containing no sample, Kevlar sample and S-Glass sample. The variation of

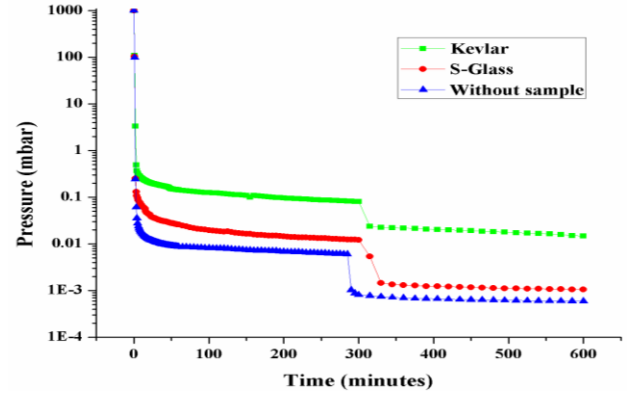


Fig. 5. Pump-down characteristics without sample and with Kevlar/S-Glass samples in the test chamber as a function of time.

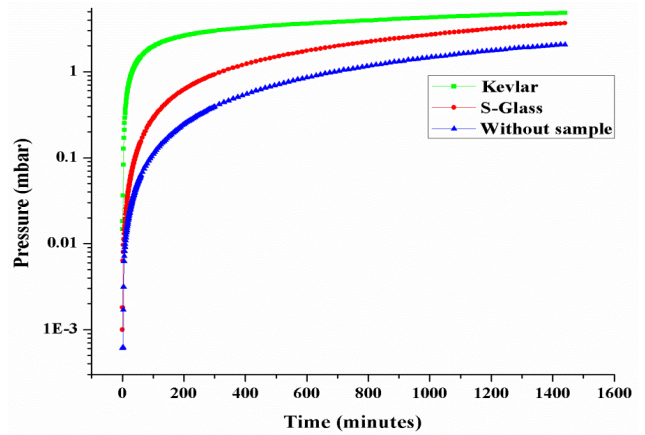


Fig. 6. Pressure-rise in the test chamber as a function of time, after isolation from the vacuum pumps, without sample and for Kevlar/ S-Glass samples.

pressure inside the test chamber under consideration with respect to time, starting from 0 to 600 min. is shown. For same time-period of pumping (15 hours), the pump-down curve corresponding to the S-Glass has less value of ultimate pressure ($8E-4$ mbar) compared to Kevlar ($9E-3$ mbar) of same area indicating that outgassing in S-Glass is lesser.

Typical pressure-rise in the chamber after isolation from the pump as a function of time is depicted in Fig. 6 for background, Kevlar and S-Glass samples. In each case, after isolating the test chamber from pumps, the pressure-rise was recorded for a period of 24 hours (1440 minutes). It is observed that for same time-period of measurement (24 hours), the pressure rise corresponding to the Kevlar was more compared to S-Glass sample indicating that the outgassing of Kevlar is more than that of S-Glass.

The specific outgassing rate of Kevlar and S-Glass samples as a function of time is shown in Fig. 7. The plot clearly shows that the outgassing is significant for both Kevlar and S-Glass samples. This also shows that material needs further improvement to reduce outgassing. However, in the actual operation of HTS motor, the torque tubes made out of these materials are cooled to low temperatures and hence are expected to outgas less. The

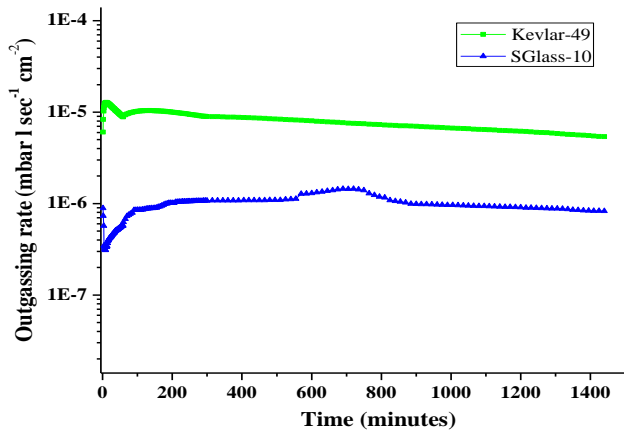


Fig. 7. Outgassing rates for Kevlar and S-Glass samples.

figure also shows that the magnitude of outgassing rates for S-Glass [order of 10^{-6} (mbar l)/(sec-cm²)] is smaller than that for Kevlar [order of 10^{-5} (mbar l)/(sec-cm²)].

Fig. 8 shows the residual gas analysis of vacuum chamber with and without torque tube materials. For this,

the concentration of various gases (i.e. hydrogen, water, nitrogen and oxygen etc.) typically released from the materials are plotted as a function of time.

By subtracting the mass spectrums of the empty chamber from the mass spectrums obtained with the torque tube materials (Kevlar and S-Glass) the following observations could be made: The concentrations of gases released from the Kevlar/S-Glass sample in the test chamber under vacuum are mostly water (H₂O), nitrogen, oxygen, Argon and CO₂. Also, it is observed that the H₂O related peaks are distinct in the chamber without sample and chamber with Kevlar/S-Glass test samples. In descending order, the concentration of H₂O was more in the chamber with Kevlar sample followed by the chamber with S-Glass sample and the chamber without test sample. The increased concentration of H₂O in the chamber with test samples of Kevlar and S-Glass is due to the desorption of H₂O from the surface during the measurement. Further, additional peaks of hydrocarbons (C_xH_y) were observed from Kevlar sample. This may be due to the evaporation of unreacted resin in the Kevlar sample.

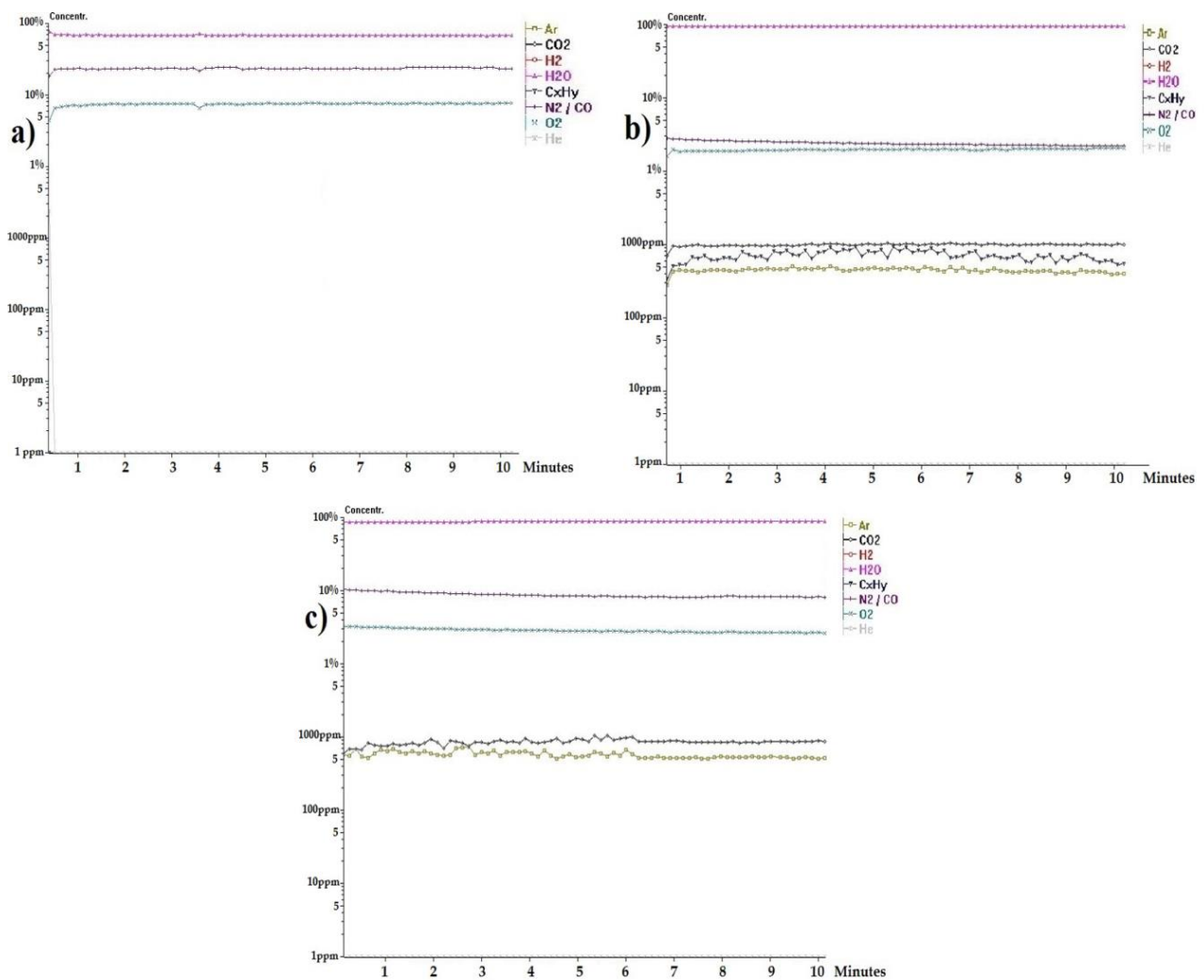


Fig. 8. RGA spectra of different gases for (a) chamber without test sample (b) chamber with Kevlar sample and (c) chamber with S-Glass sample.

4. CONCLUSIONS

An experimental setup was developed in-house for measuring the outgassing characteristics of two composite torque tube materials, namely Kevlar and S-Glass under vacuum conditions to qualify them for torque tube construction in HTS motors. Residual gas analysis was also carried out for the composite material samples to understand the nature of the chemical species during outgassing. It was found that Kevlar-49 is outgassing more than that of S-Glass-10. A signature of unreacted hydrocarbon is also observed in the mass spectrum of Kevlar sample, indicating the need for process improvement. However, it is expected that these materials outgas less at the operating cryogenic temperatures of HTS motor. Hence selection criteria of torque tube material in HTS motors should be based on several criteria such as mechanical strength and outgassing at cryogenic temperatures of these materials.

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