



A new cost effective composite getter for application in high-vacuum-multilayer-insulation tank



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ABSTRACT

H₂ is the main residual gas in the chamber of high-vacuum-multilayer-insulation tank (HVMIT) and is adsorbed by the expensive getter PdO. Adsorption characteristics of more cost effective getters (CuO, CuO & 5A and CuO & C) were investigated and adsorption products were analyzed by measuring the pressure decrease in a known volume as function of time using our designed experimental setup. CuO & C was more suitable to adsorb H₂ in HVMIT than the other getters investigated. H₂ adsorption amount and pumping speed were significantly larger than CuO and CuO & 5A getter systems. Working temperature could be also reduced with respect to these two getter bed, this being advantageous from the operational point of view. Adsorption isotherm was type I as accurately described by Langmuir model. H₂ sorption amount was 170.6 mL(stp)/g at equilibrium pressure not higher than 5.8×10^{-2} Pa, saturated sorption amount was 296.4 mL(stp)/g at room temperature, and sorption products were Cu and H₂O (g). When adsorption equilibrium was obtained, HVMIT was fed with liquid nitrogen. Interlayer pressure decreased sharply to 5.83×10^{-4} Pa in a stepwise shape for 10 h. This vacuum level is appropriate for ensuring good insulation level in the tank.

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1. Introduction

The lifespan of high-vacuum-multilayer-insulation tank (HVMIT) mainly depends on insulation performance [1]. In general, maintaining the interlayer pressure of HVMIT to less than 10^{-2} Pa is necessary to achieve optimum multilayer insulation. Therefore, adsorbents have been widely applied into HVMIT to maintain high vacuum degree [2]. Massive residual H₂ and a small amount of residual N₂ and O₂ are present in the chamber [3–7]. N₂ and O₂ from HVMIT leakage can be completely adsorbed by activated carbon (C) or molecular sieve 5A (5A) at cryogenic states. However, H₂ adsorption capacity of C or 5A is extremely small at and above the temperature of liquid nitrogen [1,8,9]. Consequently, H₂ from the outgassing of metal and multilayer insulation materials is the key problem. Non-evaporable getters based on zirconium have been used in ultra-high vacuum systems and electric-vacuum devices because of their remarkable adsorption performance for H₂ [10–12]. However, these getters cannot be applied into HVMIT

because of high activation temperature requirement, which is typically around 300–500 °C [13,14]. In addition, their sorption performance decreases along with activation temperature [11]. To date, transition metal oxides, such as PdO and PdO & AgO [13,15–17] dominate the H₂ getter market of HVMIT. However, these getters have a relatively high unit price and limited adsorption capacities [4]. Hence, exploring a new H₂ getter is needed.

Copper oxide (CuO) has been used as a catalyst or catalyst precursor in the study of reaction mechanisms that involve H₂ as reactant or product through X-ray diffraction (XRD), X-ray photoelectron spectroscopy (XPS), Transmission electron microscope (TEM), and thermogravimetric methods [18–23]. Only few studies have investigated the H₂ adsorption performance of CuO and CuO-doped getters. Our research showed that H₂ can be efficiently adsorbed by the more cost effective H₂ getter CuO & C. C played a significant role in the physisorption of H₂ and increased the contact time of H₂ and CuO at the same time. C also inhibited the sinter of CuO in the sorption process to some extent. H₂O from the adsorption products and the rest of the residual gases were adsorbed by 5A located at the bottom of the exterior wall in the inner tank of HVMIT. In addition, CuO cannot react with C at our

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working temperatures according to our primary tests.

In this study, we investigated the effects of 5A and C on the adsorption characteristics of inexpensive H₂ getter CuO and found that CuO & C was suitable to adsorb H₂ in the HVMIT. In addition, we obtained the adsorption isotherm and analyzed the adsorption products of CuO & C by measuring the pressure decrease in a known volume as function of time.

2. Experiments

2.1. Samples

Commercial CuO, 5A, and C were obtained from Sinopharm Chemical Reagent Shanghai Co., Ltd., Shanghai TiHo Molecular Sieve Co., Ltd., and Shanghai Xing Chang Activated Carbon Co., Ltd., respectively. CuO has an average particle size of 200 μm and a purity of 99.9%. The diameter of 5A and the particle size of C are 3–5 mm and 20 mesh (0.85 mm), respectively. The inert gaseous He and active gaseous H₂ were purchased from Wujiang Messer Industrial Gas Co., Ltd. (purity ≥ 99.999%, which satisfies the standard of GB/T7445-1999). The experimental plan is shown in Table 1, and three sets of experiments were carried out.

- (i) Adsorption characteristics of CuO, CuO & 5A, and CuO & C were obtained at different adsorption temperatures (AST: Adsorption temperature) and the same initial H₂ pressure (IHP: Initial hydrogen pressure) in Experiments 1, 2, and 3.
- (ii) H₂ was adsorbed by CuO & C at the same AST and different IHP in Experiment 4.
- (iii) Adsorption products of CuO & C were analyzed in Experiments 5 and 6.

2.2. Experimental setup

As shown in Fig. 1, the experimental setup is mainly composed of the following parts.

- (i) Vacuum system: Buffer pipe, buffer tank, and test tank were evacuated by vacuum pumps.
- (ii) Measurement and collection system: ZDF-5227 compound vacuum gauges equipped with resistance and ionization gauges were used to measure the pressure of buffer pipe, buffer tank, and test tank. The temperature was measured by Omega type T thermocouple, which extended into a chamber through an aviation plug and was collected with Keithley2700. The pressure and temperature data were recorded via a computer.
- (iii) Air supply system: He and H₂ tanks supplied high-purity gases for the experiments.
- (iv) Tank system: The volumes of the getter container, buffer pipe, and buffer tank were 0.12, 1.28 and 48.63 L, respectively. The chamber volume was 49.69 L with a residual volume of 42.94 L (chamber volume minus the volume of 5A

and multilayer insulation materials). The actual test volume was 43.06 L (residual volume plus getter container volume). All the volumes were accurately calibrated with the inert gaseous He [3].

1, 3, 5. Resistance gauges. 2, 4, 6. Ionization gauges. 7. Aviation plug. 8, 9, 10, 11, 12, 13, 14, 15, 16. High-vacuum flap valves. 17, 18. Vacuum pumps. 19. He tank. 20. H₂ tank. 21. Buffer pipe. 22. Buffer tank. 23. Test tank. 24. Multilayer insulation material. 25. Molecular sieve 5A. 26. Getter container. 27. Getter temperature controller. 28. Vacuum gauge. 29. Data acquisition instrument. 30. Computer.

2.3. Experimental procedure

The buffer pipe, buffer tank, and the chamber were first separately evacuated to less than 1×10^{-2} Pa. While evacuating, the inner tank of the test tank was heated continuously with an electrical heating rod at 160 °C to induce the outgassing of metal and multilayer insulation materials and activate 5A for 48 h. While heating, the chamber was filled with gaseous nitrogen and pumped down. This process was repeated five times so as to remove contaminants and water from the multilayer insulation materials. Afterward, H₂ was charged into the chamber. To adjust the interlayer H₂ pressure (ILHP: Interlayer hydrogen pressure) to a proper value, the buffer pipe, buffer tank, and chamber were charged with H₂ in subsequent steps. Finally, after standing the test tank for half an hour, valve 10 was opened and measurement was automated using a computer. When ILHP became constant within 2 h, sorption equilibrium was attained.

3. Results and discussion

3.1. Leakage and outgassing

The leakage and outgassing rate is an important parameter to evaluate the performance of HVMIT, which is measured on grounds of static pressurizing method and can be calculated by the formula (1).

$$lor = \frac{\Delta P \times V}{\Delta t} \quad (1)$$

where *lor* is leakage and outgassing rate, Pa·m³/s; Δ*P* is variation of interlayer pressure during Δ*t*, Pa; *V* is interlayer volume, m³, and Δ*t* is time interval, s.

Interlayer pressure showed a logarithm growth and then a slight linear growth with time (Fig. 2). To ensure the accuracy of experimental datum, the experiment of leakage and outgassing were repeated 3 times in Fig. 2A and the mean value was shown in Fig. 2B. The maximal and minimal variations of interlayer pressure are curves 2nd and 3rd which are respectively 7.68×10^{-1} and 7.08×10^{-1} Pa within 24 h. The average variation and the average leakage and outgassing rate are 7.35×10^{-1} Pa and 3.65×10^{-7} Pa·m³/s separately.

Table 1
Information of the experiments.

Experiment code	Getters in getter container	Adsorbent in chamber	Getter temperature (°C)	H ₂ pressure (Pa)
Experiment 1	CuO (5 g)	1000 g 5A	60–260	50
Experiment 2	CuO & 5A (1 g:5 g)	1000 g 5A	60–200	50
Experiment 3	CuO & C (1 g:5 g)	1000 g 5A	60–160	50
Experiment 4	CuO & C (1 g:5 g)	1000 g 5A	160	50,500,900
Experiment 5	CuO & C (1 g:5 g)		25–160	2850
Experiment 6	CuO & C (1 g:5 g)	1000 g 5A	100	2850

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