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EVALUATION OF Zr-V-Fe GETTER PUMP FOR UHV SYSTEM*

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ABSTRACT

The nonevaporable getter pump St707 (Zr-V-Fe) from SAES Getters, Inc. may be activated and operated at lower temperatures. However, its application to a UHV system has not been reported yet. This paper reports the characteristics of the St707 getter strip in a UHV system as a linearly distributed pump. The getter is activated at $\sim 400^\circ\text{C}$ for 30 min. The pumping behavior of the getter strip for H_2 and CO is studied. The initial pumping speeds for H_2 and CO are 300 $\text{l/s}\cdot\text{m}$ and 190 $\text{l/s}\cdot\text{m}$, respectively; and decrease slowly with increasing gas load to ~ 90 $\text{l/s}\cdot\text{m}$ for H_2 at ~ 50 $\text{T}\cdot\text{l/m}$. Hydrogen equilibrium pressure at higher temperature does follow the Sieverts law and is one decade higher than that stated by SAES Getter, Inc. The application of St707 getter strip to the UHV system of ISABELLE is also discussed.

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1. INTRODUCTION

The application of nonevaporable getter (NEG) as pumps for fusion vacuum vessels and beam vacuum of particle accelerators has been reported before.¹⁻⁵ The majority of these studies were based on the well known SAES St101 NEG (86Zr/16Al). The drawbacks of St101 NEG are the required temperatures for activation (700-900°C) and operation (200-400°C), which increase the thermal stress as well as the outgassing rate of the vacuum system. The new lower temperature NEG St707⁴ (70 Zr/24.6 V/5.4 Fe) has the advantages of activation at $\geq 500^\circ\text{C}$ and operation at room temperature. However, the ultrahigh vacuum (UHV) compatibility of the St707 NEG has not been demonstrated elsewhere.

The initiative behind this work is the need for a linearly distributed pumping system for injection and beam colliding areas of ISABELLE.⁶ The major residue gases of a baked UHV system such as ISABELLE's beam vacuum are H_2 (<90%)⁷ and CO, therefore the NEG St707 strip which pumps H_2 and active gases is ideal for this purpose providing the pumping speeds are high enough and the equilibrium pressure is low enough.

In this study, we have measured the pumping behavior of the St707 strip in a vacuum chamber identical to the beam tube of ISABELLE. The pumping speeds for H_2 and CO are measured at room temperature by the conductance method. The H_2 equilibrium pressures at different gas loads and at different temperatures are also studied. Some thermodynamic quantities are derived from the adsorption isotherm and isostere. The expected pressure of a conductance limited UHV system such as ISABELLE's beam vacuum using St707 strip as pump is demonstrated.

II. EXPERIMENTAL SETUP

The experimental setup is shown schematically in Fig. 1. The all metal vacuum chamber with Conflat-flanged ports for instrumentations was constructed by utilization of one of ISABELLE's beam tubes. To ensure proper current conduction the getter strip is electron-beam welded (in vacuum) to copper blocks, which are attached to feedthroughs. Macor insulators similar to those in ref. 2 are used to isolate the getter strip from the chamber wall. The 3 cm wide constantan strip has St707 getter coated on both sides and contains ~ 15 gm getter alloy per meter. Three thermocouples are spot-welded on the edge of the strip to monitor the temperature. The four Bayard-Alpert gauges (BAG) are Varian custom-made with 0.002" collector and $\sim 5 \times 10^{-12}$ Torr x-ray limit. The IE 510 extractor gauge from Leybold-Heraeus has been calibrated against PTB⁸ standard, and has an x-ray limit $\sim 1 \times 10^{-12}$ Torr and sensitivity for N_2 ~ 7.5 Torr⁻¹. The quadrupole mass spectrometer (MS) used for partial pressure measurement is Leybold Heraeus Q200 with sensitivity down to the 10^{-13} Torr range. Balzer turbomolecular pumps (TMP) are used for roughing the main chamber during bakeout and activation; and for lowering the background pressure of the gas-in chamber. The 20 l/s sputter ion pump (SIP) is used to pump nonactive gases such as Ar and CH_4 . The main vacuum chamber except MS is bakeable to $\sim 350^\circ C$. The gas-in chamber and the MS are baked to $\sim 150^\circ C$. The 0.050" orifice between two chambers has a conductance of 0.26 l/s for H_2 and 0.07 l/s for CO. Pressure inside the gas-in chamber as well as the throughput fed into the main chamber is controlled by a Granville-Phillips servo driven valve and turbomolecular pump.

III. MEASUREMENTS AND RESULTS

The main residual gases in a baked UHV system is H_2 (>90%)⁷ and CO, hence, we have concentrated our study of the getter pumping characteristics on these two gases. First, we studied the pump down curve and equilibrium pressure without getter, with getter and with activated getter. To avoid excessive thermoexpansion of NEG and overheating of vacuum chamber, activation at 390°C x 25 min. was chosen. The pumping speeds for H_2 and CO at different gas loads were measured then. Finally, the H_2 pressure at higher temperatures and different gas loads were measured, and the adsorption isotherms and heat of adsorption were derived.

The experimental accuracy is at best $\sqrt{10}\%$ for the pumping speed and equilibrium pressure measurements, which are mainly due to systematic errors. The need for partial pressure correction during CO pumping and during activation also increased the uncertainty.

A. Pumpdown curves and Ultimate Pressure

The merit of NEG in a conductance limited vacuum chamber is best illustrated in Fig. 2, here the pump down curves with SIP, with SIP + NEG and with SIP + activated NEG are compared. Time zero is the end of each 300°C x 40 hr. bakeout. The pressure is that measured by BAG #1 and is slightly higher than those by BAGs #2 and #3. The SIP was turned on and the bakeable valve to the TMP was valved off when chamber pressure was the same as that of the TMP. The pump down curve of SIP + NEG is lower than that of SIP alone, indicating that NEG is partially activated by the 300°C x 40 hr. bakeout. However, this partial activation generates a pumping speed only comparable with that of the SIP. After activation at 390°C for 25 min. (the zigzag portion of curve C), the pump down curve and ultimate pressure are more than one decade lower than those

without activation. The pump down curves after several successive bakeouts and activations are almost identical. The equilibrium pressure of 2×10^{-11} Torr is maintained in the vacuum chamber 6 months after bakeout and activation. The major residual gases as shown in Fig. 3(b) are H_2 (94), CH_4 (1.3), H_2O (1.4), CO (1.6), and CO_2 (1.7). The major residual gases with the SIP alone (Fig. 3(a)) are H_2 (97), CH_4 (.5), CO (2), CO_2 (.2), which indicates enhanced pumping of CO by the NEG. The average CH_4 pressure should be lower since the MS is further away from the SIP.

B. Pumping Speed of NEG for H_2 and CO

The pumping speed measurement was carried out by the standard conductance method. The H_2 or CO gas was fed from a bottle through the servo-driven valve into the gas-in chamber. By maintaining a pressure between 10^{-8} Torr and 10^{-1} Torr at the gas-in chamber, a throughput of 10^{-9} - 10^{-2} T·l/s was fed through the .05" orifice into the main vacuum chamber and pumped away by the NEG. The pumping speeds were calculated using the standard Q/P method. During gas pumping, up to a 50% difference in pressure readings was observed between BAG #1 and BAG #2. The averages of those two were used as P. Little difference was found between BAG #1 and BAG #3 indicating negligible pumping of H_2 and CO by the SIP. At the same gas loads, the H_2 pumping speeds remain the same ($\pm 10\%$) at different throughputs up to 10^{-2} T·l/s·m, and the CO pumping speeds decrease with increasing throughput. The measured pumping speeds at a throughput of 5×10^{-6} T·l/s·m for H_2 and CO after various activation conditions and under different gas loads are plotted in Fig. 4. An increase of over 50% in H_2 pumping speed was observed after activation at $480^\circ C \times 10$ min., indicating the gettering efficiency is not completely restored after a $390^\circ C \times 30$ min. activation. The CO pumping speed is $\sim 60\%$ of H_2 pumping speed at a small CO gas load and de-

creases rapidly with increasing CO gas load. The pumping speed of H_2 also decreases rapidly with increasing CO gas load and reached almost zero at $Q_{CO} = .7$ T·ℓ/m. Assuming one monolayer of CO coverage at this gas load, this translates into a surface roughness factor of ~ 70 for St707 which is less than the observed 100 for St101.⁵ The lesser surface roughness might be the cause of smaller pumping speed and capacity comparing with those of St101. The compositions of residual gases during H_2 and CO pumping are shown in Fig. 3(c) and 3(d), respectively. As expected, H_2 or CO becomes major component.

C. Equilibrium Pressure at Higher T and Heat of Adsorption

The hydrogen pressure at different temperatures and gas loads will give information on the nature of hydrogen adsorption such as the heat of adsorption and the adequate activation process. The measurements were carried out at 100°C-500°C and with H_2 gas loads of 10^{-2} - 10^2 T·ℓ/m. After the desired quantity of H_2 gas was pumped by the NEG, the SIP was turned off and the getter strip was heated up to the chosen temperature. The residual gas compositions (with H_2 gas load 1 T·ℓ/m) at different temperatures are shown in Fig. 5. Partial pressure of CO remains the same while that of H_2 increases by $\sim 10^6$ times when heated from 25°C to 400°C. The adsorption isotherms with P plotted against Q at constant temperatures are shown in Fig. 6. The change in slope at an H_2 coverage ~ 1 T·ℓ/m indicates a change in adsorption mechanism. Using the surface roughness factor 70 (estimated in pumping speed measurements), 1 T·ℓ/m corresponds to about one monolayer of H_2 . The distinction between monolayer and multilayer adsorption disappears at higher getter temperatures.

The adsorption isosteres with P plotted against $1000/T$ at gas loads higher than monolayer are shown in Fig. 7. The heat of adsorption (enthalpy change) derived from the Clausius-Clayperon expression is -28.5 ± 0.7 Kcal/mole

which is in good agreement with -28.6 Kcal/mole obtained in ref. 4. This exothermic heat value is larger than the -16 Kcal/mole value for St101.⁹ However, the large negative entropy change⁴ has more than compensated the enthalpy change and makes possible the activation of St707 at temperature lower than that of St101. The obtained values for parameters A and B in Sievert's law of $\log P$ (Torr) = A + 2log q (Torr·l/gm) - B/T (°K) are A = 6.5 ± .8 and B = 6,200 ± 150. The higher value of A indicates the equilibrium pressure at higher T will be one order higher than that predicted in the SAES manual (A = 5.26, B = 6250).

IV. CONCLUSION

The experimental evidence obtained from this work has proved the applicability of the St707 getter pump in a UHV system. The getter has been activated at 400°C x 30 min. and operated at room temperature. The pumping speeds of St707 are 200 l/s·m for H₂ and 180 l/s·m for CO at a H₂ gas load of 1 T·l/m. Ultimate pressure of 2 x 10⁻¹¹ Torr was maintained after repetitive activations, several months of operation and an H₂ gas load of 1 T·l/m.

To incorporate the St707 NEG strip into accelerators or storage rings, such as ISABELLE's injection and beam colliding areas, the following scenario can be expected. Assuming a total outgassing rate of 1 x 10⁻¹¹ T·l/sec per linear cm, the peak pressure between two lumped pumps 10 meters apart will be about 5 x 10⁻¹¹ Torr;¹⁰ while the pressure due to outgassing using St707 NEG as a linearly distributed pump (assuming a linear pumping speed of 2 l/s·cm) will be 5 x 10⁻¹² Torr. Adding the 2 x 10⁻¹¹ equilibrium pressure, the achievable pressure of ≤2.5 x 10⁻¹¹ Torr represents a two-fold improvement over lumped pumps. The gas load of 1 T·l/m represents several years' operation before activation is necessary.

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FIGURE CAPTIONS

- Fig. 1. Experimental setup for getter study. The heating wire and insulation for bakeout are not shown.
- Fig. 2. Pump down curves after the end of $300^{\circ}\text{C} \times 40 \text{ hr.}$ bakeout. Open and filled triangles represent two separate bakeouts. The pressure during activation was $\sim 10^{-5}$ Torr.
- Fig. 3. Mass spectra of the residual gases (a) after bakeout with 20 l/s SIP along; (b) with activated NEG and 20 l/s SIP; (c) during H_2 pumping; (d) during CO pumping.
- Fig. 4. Pumping speeds of NEG St707 for H_2 and CO after various activation conditions and under different gas loads.
- Fig. 5. Mass spectra of the residual gases at different getter temperatures. The hydrogen gas load of NEG is 1 T·l/m.
- Fig. 6. The adsorption isotherms of hydrogen by St707 getter. The change in slope at 1 T·l/m corresponds to monolayer of hydrogen.
- Fig. 7. The adsorption isosteres of hydrogen at gas loads higher than monolayer. The heat of adsorption derived from Clausius-Clayperon expression is 28.5 Kcal/mole.

Chamber Dimension: 8.8 cm O.D. 3 m long 304LN S.S.

Getter Dimension: 3 cm wide 2.8 m long Zr-V-Fe (SI 707) Getter on constantan strip

Figure 1.

Figure 2.

Figure 3.

Figure 4.

Figure 5.

Figure 6.

Figure 7.