

Non-evaporable Getter Investigation

BNL--46473

at the National Synchrotron Light Source,\*

DE91 017005

Henry J. Halama and Yaohua Guo\*\*

Brookhaven National Laboratory

Upton, New York 11973

**ABSTRACT**

We report H<sub>2</sub>, CO and CO<sub>2</sub> pumping speed measurements on a linear pump using NEG strip St 707 (Saes Getters Inc) as a function of both adsorbed gases and number of activations. To simulate the conditions of an operating storage ring a gas mixture of 50% H<sub>2</sub>, 35% CO and 15% CO<sub>2</sub> is used in all measurements. Initial measured pumping speeds of  $>450 \text{ l m}^{-1} \text{ s}^{-1}$  decrease to 200 and  $100 \text{ l m}^{-1} \text{ s}^{-1}$  for H<sub>2</sub> and CO, respectively, after 70 air exposures. A further drop to  $50 \text{ l m}^{-1} \text{ s}^{-1}$  for CO occurs at CO loading of  $1 \times 10^{-2} \text{ Torr l m}^{-1}$ . CO<sub>2</sub> and CO pumping speeds are about the same. The photon stimulated desorption (PSD) for both baked and activated strips is an order of magnitude lower than that for stainless steel.

**I. INTRODUCTION**

Nonevaporable getter (NEG) modules and strips have been used in accelerators<sup>1</sup> and transfer lines<sup>2</sup> and much information has been accumulated in publications and internal reports. In particular, Benvenuti<sup>3,4</sup> has done definitive work on the high temperature NEG type St 101. Comparable data do not exist on lower temperature type St 707 which is more suitable for storage rings, especially those using stainless chambers. In addition, several questions should be answered such as:

\*Work performed under the auspices of U.S. DOE under contract DE-AC02-76CH00016

\*\*On leave from Southeast University, Nanjing PRC.

**MASTER**

1. How many times can NEG be regenerated before its performance become unacceptable, and
2. Can NEG strips be used as clearing electrodes?

We have therefore undertaken comprehensive studies of ST 707 getter and hope that the results presented will be useful to accelerator technologists.

## II. PUMPING SPEED MEASUREMENTS

The three most important properties required by an operating storage ring are:

- a. no-beam pressure of  $\leq 1 \times 10^{-10}$  Torr
- b. good pumping speed in  $10^{-10}$  -  $10^{-9}$  Torr ranges
- c. multiple regeneration capability without unacceptable degradation of NEG equilibrium pressure and pumping speed.

Most gases evolve in storage rings during the initial stages of conditioning due to photodesorption of synchrotron radiation. The gas composition is approximately 40-50% H<sub>2</sub>, 30-40% CO, 15-25% CO<sub>2</sub> and some methane and water. During operation, as the walls of the vacuum chamber are cleaned the concentration of all gases except H<sub>2</sub> decreases. In order to simulate the operating conditions of a storage ring, a mixture of 50% H<sub>2</sub>, 35% CO and 15% CO<sub>2</sub> was used rather than individual gases, since CO and CO<sub>2</sub> inhibit the pumping speed of hydrogen<sup>4</sup>. Pure H<sub>2</sub>, CO and CO<sub>2</sub> test gases were used only for a short time to obtain their pumping speeds. Furthermore, in dedicated light sources with many beam lines, especially those proposed for VLSI chip production, the danger of the NEG being exposed to the atmosphere is much greater than that for a well controlled research facility.

### A. Experimental set-up

The schematic of the apparatus, shown in Fig. 1, consists of a gas supply which

contains the above gas mixture, pure H<sub>2</sub> and CO as test gases and Ar for calibration. All gases can be valved off and the lines are pumped out by a sorption pump, S<sub>1</sub>. The gases are introduced into chamber A via V<sub>1</sub>, where the pressure is measured by G<sub>2</sub>. An orifice, O, having a conductance of 1 l/s for N<sub>2</sub>, separates A and B in order to determine the gas flow. The main chamber, B, a 2.5m long stainless steel tube 8.8 cm in diameter, contains a 2.2m long 3cm wide St 707 NEG strip (Fig 2) equipped with a thermocouple, calibrated gauges and RGAs.

Pumping speed, S, in l m<sup>-1</sup> s<sup>-1</sup>, is computed from the differential equation of a linear pump (Fig.2):

$$V \frac{dP}{dt} = Q - SP + C \frac{d^2P}{dx^2} \quad (1)$$

In steady state  $\frac{dP}{dt} = 0$  and Equation 1 becomes

$$\frac{d^2P}{dx^2} - \frac{SP}{C} + \frac{Q}{C} = 0 \quad (2)$$

which yields the following solution:

$$S = C \left[ \frac{1}{L} \cosh^{-1} \left( \frac{P_3 - P_0}{P_4 - P_0} \right) \right]^2 \quad (3)$$

where Q and C are the outgassing rate and the conductance of chamber B, L is the length of the NEG strip, P<sub>3</sub> and P<sub>4</sub> are the pressures measured by G<sub>3</sub> and G<sub>4</sub>, respectively, and P<sub>0</sub> is the background pressure before the test gas is let in.

### B. Measurements and Discussion

During the course of our investigation we have tested 2 NEG strips which were exposed to dry nitrogen 20 times and to air of varying degrees of humidity 63 times. The results of NEG 1 are used only to corroborate the data from NEG 2. Before each measurement the entire system is baked out to 200°C for 48 hours.

At the end of each bake the NEG is regenerated at temperatures from 400° to 700°C resulting in pressures during regeneration of  $10^{-5}$  to  $10^{-3}$  Torr.

At the end of each regeneration TMP is valved off and  $V_3$ , is open. After cool-down the background pressure with  $S_3$  on reaches the  $10^{-11}$  Torr range within 2 days and hydrogen accounts for 95% of the residual gas. Valving off the triode,  $S_2$ , raises the pressure slightly due to negligible NEG methane pumping.

After reaching an equilibrium,  $H_2$ , CO and  $CO_2$  pumping speeds,  $S$ , of the unloaded NEG were measured for several flow rates,  $Q$ , between  $5 \times 10^{-5}$  and  $5 \times 10^{-4}$  Torr  $\ell$   $s^{-1}$ . Only small differences in  $S$  were recorded for the above pressure range. Using the gas mixture, we observe in Fig 3 about the same pumping speed for  $H_2$  and CO up to a significant gas loading (0.5, 0.35, and 0.15 Torr  $\ell/m$  of  $H_2$ , CO and  $CO_2$ , respectively).  $CO_2$  pumping speed, not plotted in Fig. 3, is about the same as that of CO. This is in contrast to the behavior for single gas loading where  $S_{H_2}$  remains high up to several Torr  $\ell/m$ . The same effect was observed by Hseuh<sup>5</sup> and Benvenuti<sup>4</sup> using St 101. After a few air exposures,  $S_{CO}$  decreases faster than  $S_{H_2}$  (Fig. 4). In addition, very long periods between measurements (several days) seem to increase all pumping speeds.

In Fig. 4,  $H_2$  and CO pumping speeds show a monotonic decrease with the number of exposures to air. The biggest decrease occurs during 20 exposures, after which the pumping speed levels off to still acceptable values for storage rings. In this figure, we also plot the amount of gas loading at which  $S_{CO}$  drops to 80  $\ell$   $s^{-1}$   $m^{-1}$ . Even though only the quantity of CO is indicated, each point contains  $H_2$ , CO and  $CO_2$  in a ratio of 5:3.5:1.5. In contrast to air exposures, 20 exposures to dry nitrogen produce only a small decrease in pumping speed.

Finally, we have investigated activation temperatures and found that optimum pumping speed is achieved by heating the NEG strip to 400° - 450°C for

about 30 minutes. Temperatures above 550°C produced lower speeds, possibly due to high pressures during activation. An attempt to restore high S after 40 air exposures by heating it above 600°C yielded inconclusive results for CO and significantly lower S for H<sub>2</sub>.

### III SYNCHROTRON RADIATION STUDIES

NEG devices used in operating storage ring vacuum chambers are generally shielded from both synchrotron radiation and resulting photons by slotted metal walls. In order to gain more experience with photon beams we have installed the NEG and its chamber (Fig. 2 and 3) in the U10B beamline described previously<sup>6</sup>. Briefly, a white photon beam having critical energy of 500 eV irradiates the wall of an 8.8 cm diameter chamber at 11 milliradian incidence. The normalized pressure rise,  $\Delta P/I$ , due to desorbed neutral gases is measured using calibrated BA gauges and RGAs. In this experiment the photon flux is  $4 \times 10^{14}$  photons s<sup>-1</sup> mA<sup>-1</sup>.

After a two day 200°C bake-out the chamber is exposed to direct photons. Since the NEG strip is mounted on the bottom of the chamber (Fig. 2) only reflected photons and photoelectrons can strike it. Biasing the NEG negatively produces only a negligible decrease in  $\Delta P/I$ . We therefore conclude that most desorption takes place at the stainless steel tube surface (Fig. 5).

At a dose of  $3 \times 10^{21}$  photons/m the NEG was activated and after H<sub>2</sub> and CO pumping speeds were measured, the chamber was again exposed to photons. No large initial pressure peak was observed which confirms the fact that during regeneration adsorbed gases diffuse into the bulk of the getter and do not contaminate the system. As seen in Fig. 5  $\Delta P/I$  decreased by a factor of four and kept decreasing at a steeper slope during exposure. We can conclude that at a dose of  $10^{23}$  photons/m  $\Delta P/I$  would be ten times lower with NEG pumping.

At the end of photon exposure the total of pumped gases was estimated to be 0.2, 0.015 and 0.008 Torr l/m for H<sub>2</sub>, CO and CO<sub>2</sub>, respectively. During this run H<sub>2</sub> pumping speed was almost unchanged while CO speed dropped by a factor of two. At this concentration, CO effect on H<sub>2</sub> is still negligible.

Finally, we have applied both positive and negative potential to the strip to see if it could be used as a clearing electrode. As seen in Fig. 6, applying negative bias prevents photoelectrons from reaching the NEG strip and expels photoelectrons produced in the strip by diffusely reflected photons. A very small decrease in both the current and pressure is therefore observed. When positive voltage is applied, photoelectrons produced by the incident photon beam at the wall are collected by the NEG. At 200 V, photoelectron current saturates (all photoelectrons have been collected) and reaches 2.3  $\mu$ A per mA. As the voltage is further increased, the energy of the electrons hitting the NEG strip also increases, resulting in a proportional pressure rise due to electron desorption. By 3 kV the pressure has increased almost 30 times. We note that at V = 0, partial pressures of H<sub>2</sub>, CO, CO<sub>2</sub> and CH<sub>4</sub> which were approximately 83, 10, 4 and 3 percent, respectively, changed to 40, 25, 25 and 10 percent when +4 kV were applied to the NEG strip. Fig. 7 depicts the ratios as a function of applied voltage.

After this series of experiments the NEG chamber was opened, rotated 90°, pumped down and baked to 200°C for two days. After cool-down, when the background pressure reached  $3 \times 10^{-10}$  Torr, the shutter was opened and the NEG exposed to direct synchrotron light, yielding almost an order of magnitude lower pressure than stainless steel (Fig.5). This unexpectedly large improvement could be due to:

- a. roughness and waviness of the getter which may considerably increase

the local angle of photon incidence, resulting in lower PSD<sup>7</sup>.

- b. net pumping speed when the NEG is heated above 200°C which was measured in this experiment to be 10-20 l/s.m for H<sub>2</sub> and 1 - 2 l/s.m for CO. Activating the getter decreased  $\Delta P/I$  by only a factor of two with a very shallow slope.

#### IV CONCLUSIONS

Our investigation was aimed primarily at answering the questions pertinent to using NEG strips as linear pumps in electron storage rings and, in particular, in compact rings. Since CO and CO<sub>2</sub> inhibit H<sub>2</sub> pumping speed, all measurements reported were done with gas mixtures. Based on our work we draw the following conclusions:

1. Initial H<sub>2</sub> and CO pumping speeds of St 707 getter strip are about the same, i.e.,  $> 400 \text{ l s}^{-1} \text{ m}^{-1}$ . CO speed decreases faster than H<sub>2</sub> speed with subsequent regeneration after air exposures (Fig. 3). H<sub>2</sub> and CO pumping speeds of St 101 are approximately two to three times higher.<sup>3,4</sup>
2. Exposure to dry nitrogen has only a small negative affect on NEG behavior. For any controlled opening of a storage ring vacuum system, LN<sub>2</sub> boil-off should be used.
3. Even after 70 air-exposure activation cycles, the pumping speed is still acceptable. However, regenerations have to be done more frequently (Fig.4).
4. If the NEG is brought to atmospheric pressure, activations for optimum pumping speed occurs at 400 - 450°C for thirty minutes. Temperatures below 400°C are sufficient, if NEG is routinely regenerated after its speed drops below a desired level ( $\sim 50 \text{ l s}^{-1}$

$m^{-1}$ ).

5. NEG can be exposed both to reflected and direct photons with no adverse affects. PSD from both baked and activated NEG is significantly lower than from stainless steel (Fig.5).
6. NEG strips can be used as clearing electrodes, provided that only negative voltage is applied to them, which constitutes normal operation. Positive voltage causes excessive outgassing due to photoelectron bombardment (Fig. 6).

#### V ACKNOWLEDGEMENTS

Fruitful discussions with C. Benvenuti, C. L. Forester and H.C. Hseuh are gratefully acknowledged. We thank C. Lanni for his indispensable technical assistance and data taking.

#### DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

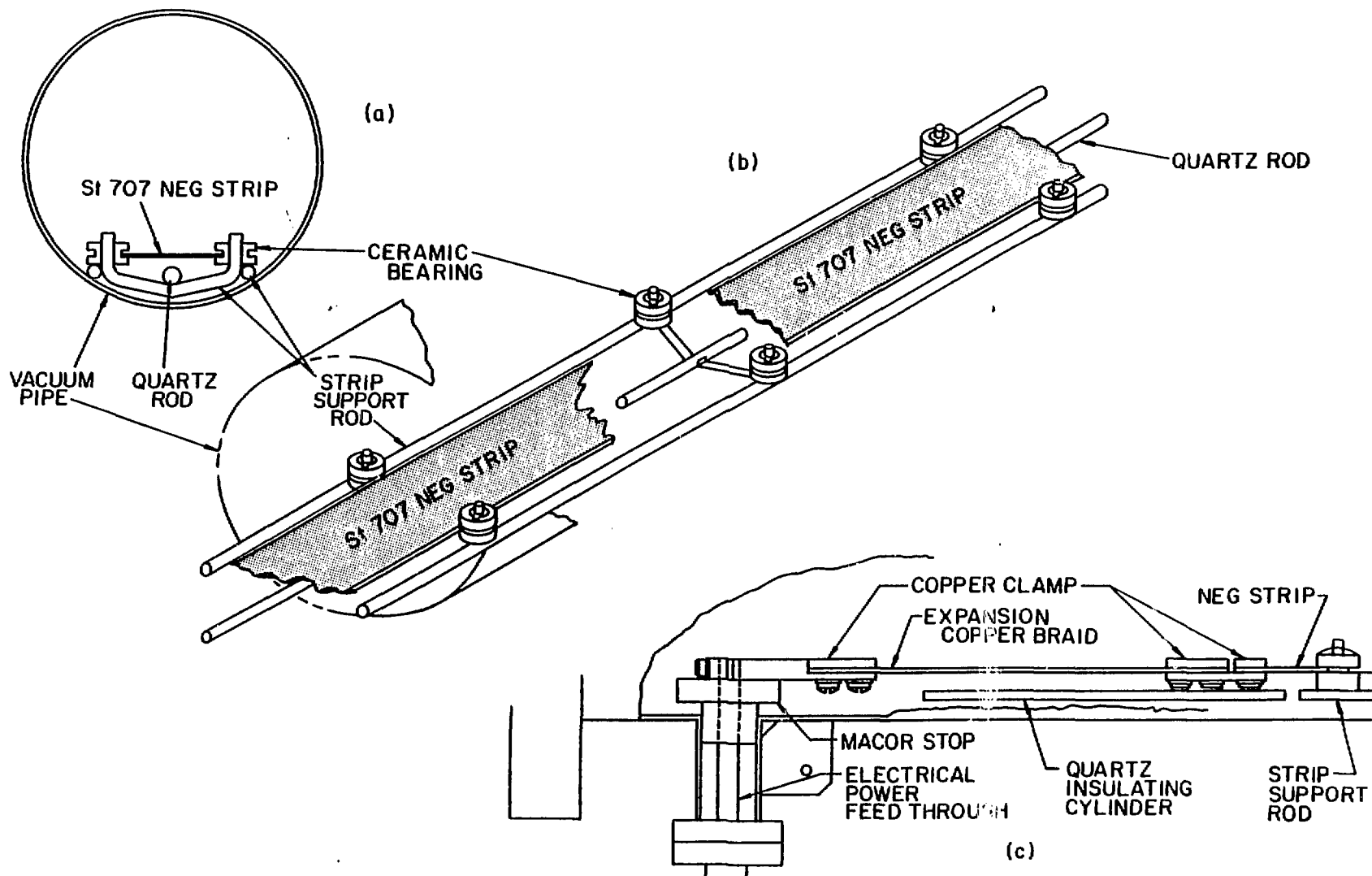
### **REFERENCES**

1. C. Benvenuti, Nucl. Instr. Methods 205, 391 (1983).
2. H. C. Hseuh et al, NS-32, No.5, 3797 (1985).
3. C. Benvenuti and F. Francia, J. Vac. Sci. Technol. A6 (4), 2528 (1988).
4. C. Benvenuti and F. Francia, CERN-LEP-VA/89-61 (1990).
5. H.C. Hseuh and C. Lanni, J. Vac. Sci. Technol. A1 (2) 1283 (1983).
6. T. Kobari and H. Halama, J. Vac. Sci. Technol. A5 (4) 2355 (1987).
7. Gröbner et al, Vacuum 33 (7) 397 (1983).

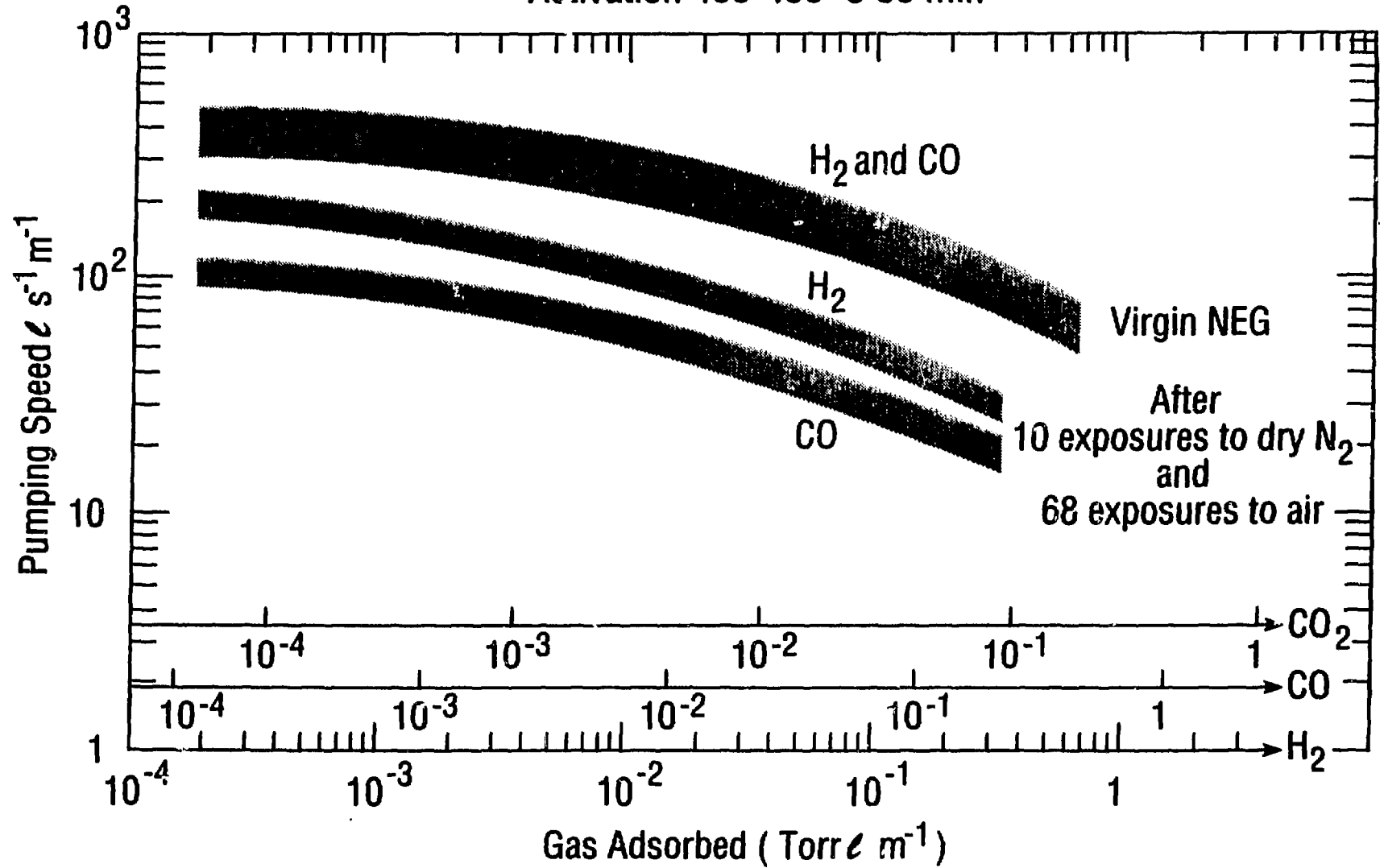
### FIGURE CAPTIONS

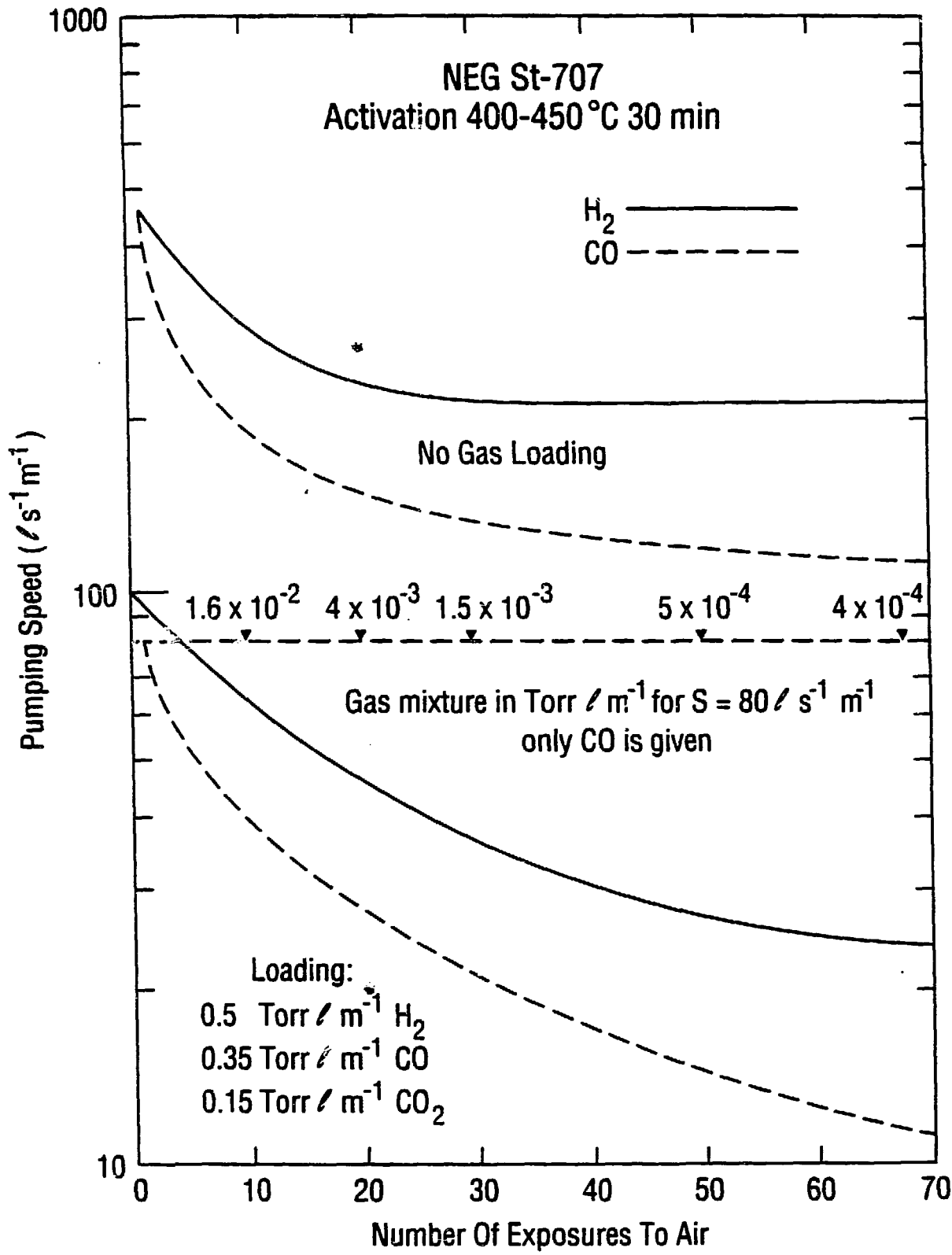
- Fig. 1 NEG experimental setup:  $G_1$  through  $G_4$  BA gauges;  $V_1$ -bleed valve;  $S_1$  sorption pump;  $S_2$ -Ion pump; O-orifice; Ft-feedthrough; Th-thermocouple.
- Fig. 2 NEG mounting in the measuring chamber, ID = 8.8 cm.
- Fig. 3  $H_2$  and CO pumping speed vs. amount of pumped  $H_2$ , CO and  $CO_2$  mixture.
- Fig. 4  $H_2$  and CO pumping speed vs. number of air exposures. Also plotted is the amount of gas mixture pumped for  $S_{CO} = 80 \text{ l.s}^{-1} \text{ m}^{-1}$ .
- Fig. 5 Pressure rise due to photons incident on a) beam tube (solid line) and b) on NEG strip (dashed line).
- Fig. 6 Pressure ratio (triangles) and NEG current (circles) vs. NEG voltage.
- Fig. 7 Partial Pressure ratios of  $CO_2$ ,  $CH_4$ , CO and  $H_2$  vs. NEG voltage.

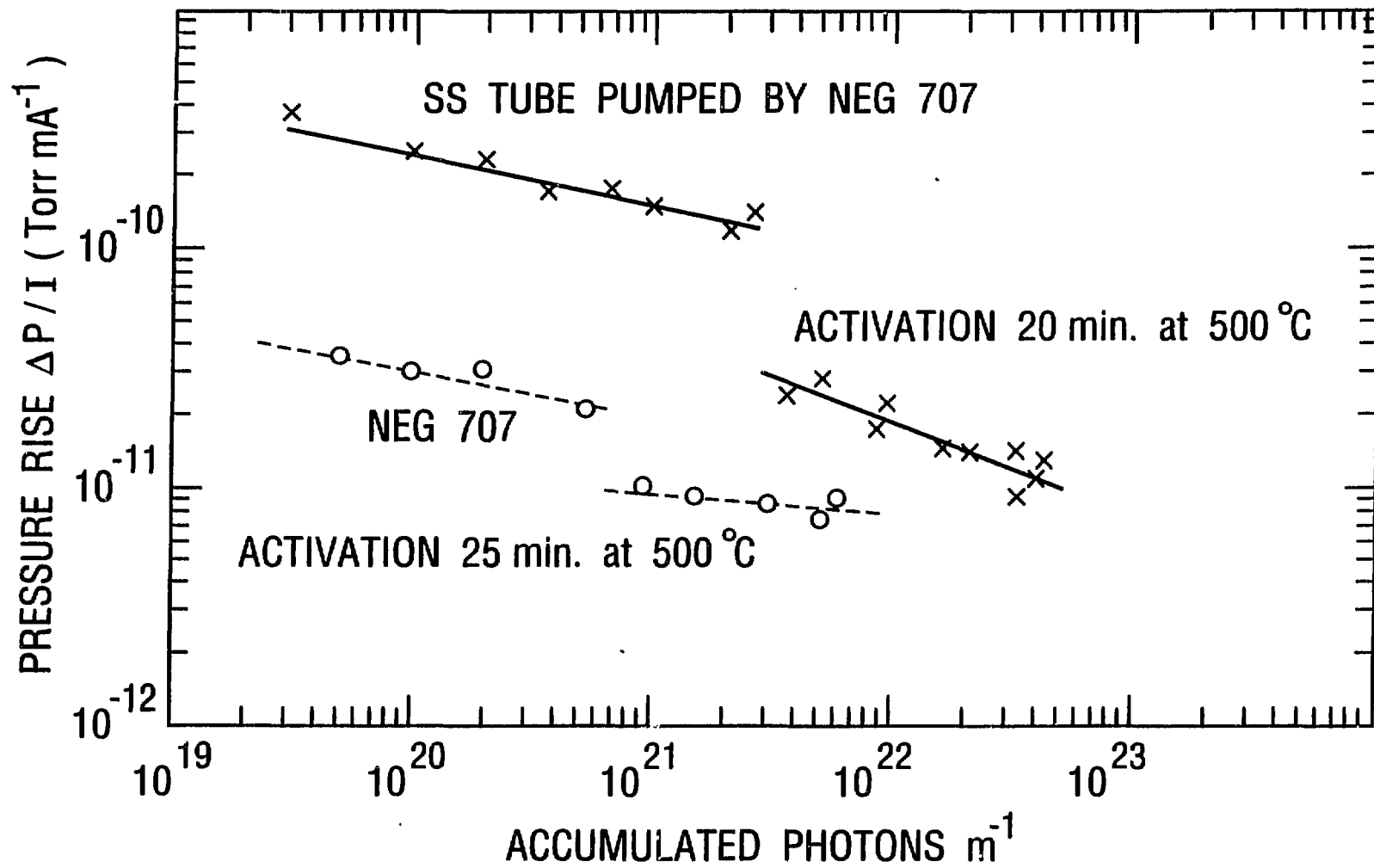


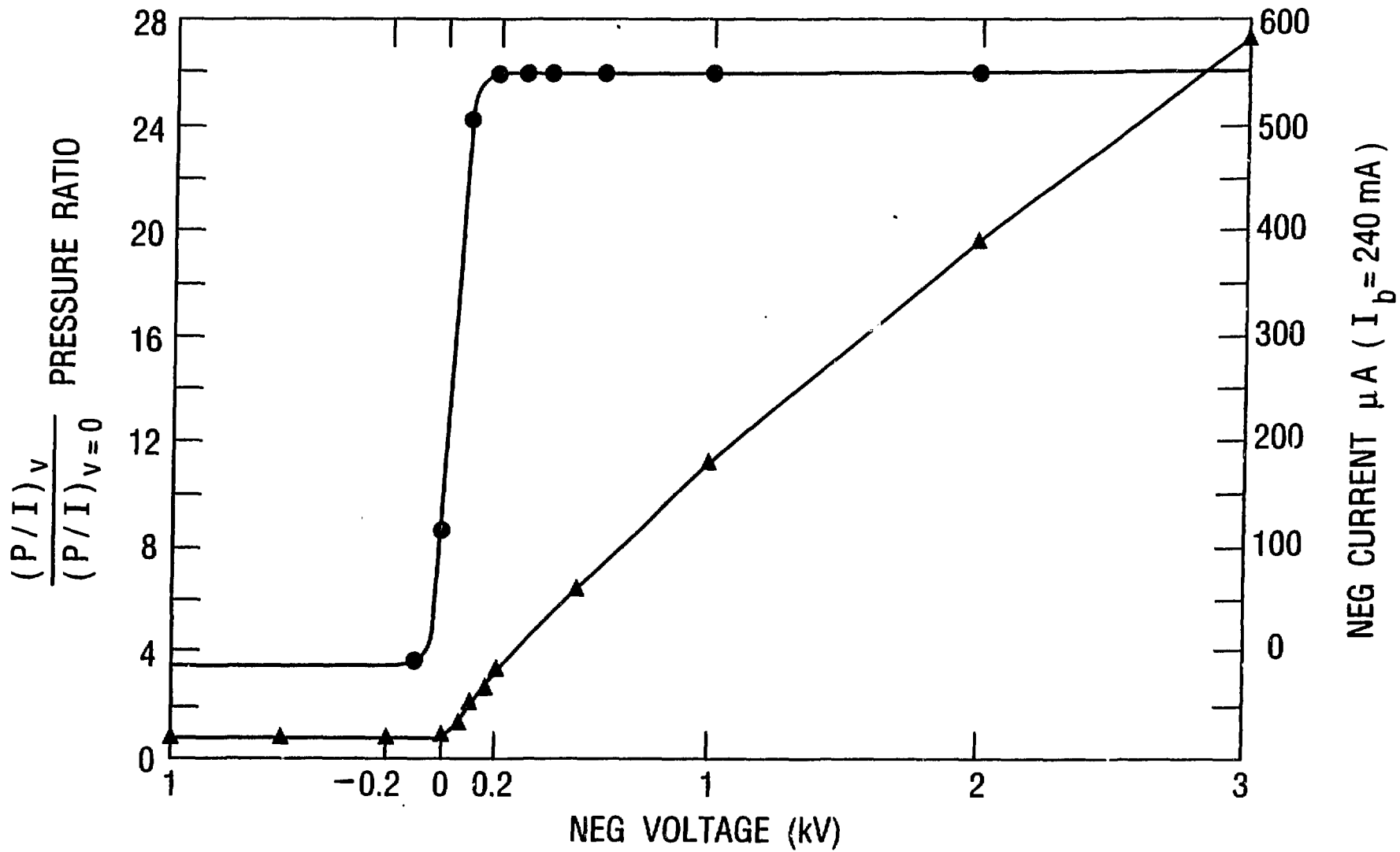


NEG St-707  
Activation 400-450 °C 30 min









Partial Pressure Ratio  $\frac{(P/I)_V}{(P/I)_{V=0}}$

