Development & Characterization of Hydrogen Reservoirs for Thyratrons and Copper Vapor Laser System

M S Tyagi, Mahesh Kumar, B L Meena, Arvind Jakhar, Alok Mishra, Hasina Khatun, and U N Pal
Electron Tubes Area, Central Electronics Engineering Research Institute, Pilani, Rajasthan India
Email: mstyagi@ceeri.ernet.in

Abstract: The hydrogen reservoir is a critical component of gas filled devices where hydrogen environment is a basic requirement. It maintains equilibrium of hydrogen pressure of the order of 30-100 Pa at a given temperature in side the gas filled devices. A class of materials, which could absorb hydrogen gas under certain temperature condition and then release the hydrogen to maintain its equilibrium pressure at the given temperature. A known family of transition elements like titanium, zirconium, lanthanum etc possesses this property. Among these, titanium is the most suitable material for its gettering properties of hydrogen. A special purpose Hydrogen reservoir has been developed for thyratron and copper vapor laser system. This paper describes the critical steps of fabrication of reservoir and its characterization. The fabrication process mainly depends on the specific size of the reservoir, gas loading process and its end use. The size of the reservoir also depends on the volume of the thyratron and copper vapor laser system in which it is to be used. The amount of gas loaded in the reservoir depends on the volume of the thyratron and temperature of the reservoir. Reservoir characteristics have been studied for the calculated amount of hydrogen gas loading. Behavior for pressure v/s heater power has been studied at different gas loadings. A study of the gas released at high temperature and gas absorbed at room temperature has also been presented in this paper.

1. Introduction
Reservoir is a critical component of all high power thyatrons. Its main function is to maintain a low hydrogen pressure of the order of 50 Pa in the face of hydrogen clean up during operation. Reservoirs are required to exhibit certain performance features. First is that it should store sufficient amount of hydrogen to last for few million pulses of thyratron operation. Second, it should be able to create the above mentioned working pressure in the thyratron volume at relatively lower temperature, somewhere between 500 – 700°C. Third, the slope of temperature vs. pressure characteristics should be as flat as possible to avoid the influence of any transient temperature on pressure. For achieving the above features of reservoir, single & double reservoir systems have been studied. This paper describes the studies conducted on hydrogen reservoirs for a 25kV/1kA thyratron that has been developed at CEERI and for copper vapor laser system for RRCAT Indore.
2. Reservoir fabrication
The constituent parts of the hydrogen reservoir are shown in figure 1. All components of the reservoir are cleaned chemically. The cleaned parts are fired in hydrogen furnace. Ni parts are fired at 800°C in dry hydrogen. Molybdenum parts are fired at 850°C in wet hydrogen. The tungsten heater coil is annealed at 1000°C in dry H₂. The insulation coating on heater coil is carried out using cataphoretic process. The heater is then baked at 100-120°C for 60-90 min in oven. This heater is then sintered in wet H₂ using RF induction furnace at 1650°C. Inner surface of reservoir sleeve is coated with alundum to a thickness of approximately 0.20-0.40 mil. The coated reservoir sleeve is baked at 100-120°C in oven for 60-90 minutes and sintered at 1650°C for 5 min. The heater coil is fixed inside the sintered reservoir sleeve. Alundum is filled in this assembly and packed to the maximum. The above sub-assembly is now sintered again at 1650°C for 5-7 minutes for making a potted heater assembly. Cleaned palladium foil (0.02mm thick and 34mm x 12mm in size) is rolled over the inner cup with the help of spot weld jig and spot-welded. 0.25g of 450µ size Ti powder which has been vacuum fired at 700°C for 20 minutes, is filled in the annular space between palladium cylinder and sleeve envelop. The potted molybdenum sleeve is put inside the sleeve envelop. It is held in position by spot welding it with a heat shield. At the other end of the reservoir the palladium foil is spot-welded with outer cup and this outer cup is spot-welded with a Ni strip. One end of the heater lead is spot-welded with this Ni strip.

![Figure1. – Hydrogen Reservoir (a) Schematic view and (b) photo of reservoir](image)

3. Reservoir for copper bromide laser
OFHC copper capsule reservoir is used in COPPER BROMIDE LASER (CBL-10) as shown in figure 2 (a) and (b). The sealed off CuBr laser tube operate with buffer gas mixture Ne+H₂. During operation there is a slow process of hydrogen loses. This lead to decreasing of power. Criteria for hydrogen deficit are a decrease of power below 6W and starting of laser emission with ring shape at low temperature (before temperature reach 410°C). This situation appears after ~200h of tube operation. The nominal power of tube can be obtained by hydrogen refreshment procedure.

The CBL 10 laser is a completely automatic source of visible laser radiation at the two visible wavelengths: the green  = 510.6 nm (~ 70% of power) and the yellow  = 578.2 nm (~ 30% of power).
3. Experimental setup

Figure 3 shows a schematic of the experimental set-up. Turbo Molecular Pump (PFEIFFER Balzers, model TCP 380) is used to create oil-free clean vacuum of the order of 10^{-5} Pa. Pumping speed of the TMP (Turbo Molecular Pump) is 300 l/s and compression ratio is 1\times10^5 for H_2. The system has two volumes V_1 (824 cc) and V_2 (367 cc). These are made of vacuum grade stainless steel T’s and I’s. The reservoir is housed in volume V_2, an ionization gauge and a capacitance gauge (Baratron, max range 1 Torr) along with their control unit are connected to volume V_1 and V_2, respectively to sense the pressure. This ionization gauge can read pressure of 10^{-9} - 1 Pa. A TC gauge is also connected to volume V_1 to sense the pressure while loading the reservoir, which can read pressure from 0.1 Pa to 200 Pa. RGA head is mounted on volume V_1. It is connected with a mass spectrometer interface unit (HIDEN MASsoft), which is connected with a personal computer (PC) to determine the partial pressure of the gases present. The working range of this unit is below 1\times10^{-3} Pa. To analyse the purity of the H_2 gas released by the reservoir in volume V_2, a Pressure Reduction System is used to operate RGA. Reservoir is heated through a regulated DC power supply (Aplab-L3210 model). The source of H_2 is a MATHESON grade gas cylinder (purity is 99.49%). Valve outlet CGA 350 is mounted on the gas cylinder and it is connected with valve 1 via isolation valve, flash arrester, and high accuracy needle valve. A leak valve is also connected with volume V_1 to fill up the required amount of H_2 in volume V_1 accurately. This valve gives maximum sealing efficiency from atmospheric pressure to 10^{-9} Pa.
4. Loading of reservoir
At the outset the whole system is baked out for a total of 36 hours (12hrs at a stretch) at a temperature of approximately 200°C using heating tapes. During bake out reservoir is also given some power (equivalent to a temp. of 200°C). Vacuum of the order of $10^{-3}$ Pa was read from the ionization gauge. Reservoir is now heated at about 800°C for about 30 minutes. RGA analysis is carried out.

Before loading the reservoir, the entire system is flushed with high purity hydrogen. During flushing no heater power is applied to the reservoir. Now, reservoir is heated at 700°C through heater leads for 15 minutes. At this time valve 1 is closed and 2 and 3 are open. This is done to get rid of contaminations present in the Ti powder. This is fixed as the loading temperature of the reservoir. After that valve 2 and 3 are closed. Volume V1 is filled with hydrogen by opening valve 1 up to a pressure of 200 Pa. This pressure is measured by TC gauge (Ionization gauge cannot be operated at this pressure). Valve 1 is closed. Reservoir temperature is adjusted to 700°C. Now valve 2 is opened to allow hydrogen gas into the volume V2 (Reservoir volume). Reservoir is allowed to absorb hydrogen for sometime. The absorption is indicated by drop in the pressure read from Baratron and TC gauges. By this measure the pressure in volume V1 and V2 decreased suddenly to a value, which is considerably, lower than the one calculated from the increase in volume (from 139 Pa (200 Pa fill) to 30 Pa). This indicates the reservoir has instantly absorbed hydrogen. After that, Reservoir power is reduced slowly from 700°C to room temperature and it is found that the reservoir absorbs almost all the 200 Pa that was introduced through volume V1, as the pressure shown by the TC gauge is about 1 Pa. The amount of hydrogen absorbed by the reservoir is $200 \times 824 \, \text{Pa} \cdot \text{cm}^3$. This we term as 200 Pa loading. For 400 Pa loading, the same loaded reservoir is kept at 700°C again. Volume V1 is filled with another 200 Pa and same procedure is followed as for 200 Pa loading. The amount of hydrogen loaded into the reservoir will now be $2 \times 200 \times 824 \, \text{Pa} \cdot \text{cm}^3$. This is 400 Pa loading of the single reservoir. Similarly the reservoir can be loaded for more gas fills of hydrogen till it gets saturated i.e. it does not absorb the hydrogen any more.

5. Characterization of the reservoir
Tests were conducted to observe various characteristics of these reservoirs. Pressure heater current characteristics was the major one. This alone decides the suitability and capability of the reservoir to be used for a particular thyatron. In this test heater current is increased in steps from zero and pressure generated in volume V2 is recorded for different gas loadings. Slope of the curve thus obtained sets one criterion for the selection or rejection of the reservoir. Long-term behavior judged from the reproducibility and stability of the pressure vs. current curve is another useful criterion to fix the usefulness of the reservoir under test.

6. Result and discussion
Figure 4 shows those heater power vs pressure characteristics of single reservoir system. We find that for higher gas loadings pressure generated by the reservoir inside volume V2 is higher. For 600 Pa loadings, reservoir generates 28 Pa at 9 watts. This shows that the reservoir is not sufficiently loaded to create the required pressure of the order of 50-70 Pa. The volume of the actual thyatron in which the reservoir will be operating is about 100 cc. It may be assumed then that, the hydrogen pressure attained in the thyatrons by this reservoir may touch 50 Pa mark. It is known that scaling is not linear. So three times reduction in the volume will not result in the increase in pressure by the same proportion. Also the curve obtained is steep at this operating point i.e. at 9 W. Any change in the temperature will influence the pressure sufficiently.
Figure 4 – Variation of pressure in volume $V_2$ with change in heater power for various gas loadings – Single reservoir case

Figure 5 presents results obtained using two reservoirs, $R_1$ and $R_2$. For different wattage (temp) to the $R_2$, temp vs. pressure characteristics have been obtained by varying heater power of the $R_1$. Figure very clearly shows the control exercised through $R_2$ on the characteristics. A flatter characteristics with higher pressure (50 Pa) created at about 15 W has been obtained for the gas loading of 1200 Pa. The results have been encouraging and it has been planned to continue with these experiments with improved experimental set-up to standardize the development and use of these reservoirs.

Figure 5 – Variation of pressure in volume $V_2$ with heater power of $R_1$ for different heater powers of $R_2$ gas loading is 1200 Pa (i.e. $6 \times 200 \times 824$ Pacc) – Double reservoir case
Figure 6. Equilibrium Pressure vs. Heater Power – OFHC Copper Capsule Reservoir Case

Acknowledgement
Authors are grateful to Dr. Chandra Shekhar, Director, CEERI and Dr. Vishnu Srivastava, H.O.D., MWT, Area for their keen interest in the work. We are also grateful to Prof. K Frank, visiting scientist, from University of Erlangen Germany, for many useful interactions.

Reference:
[1] S. Goldberg and J. Roenstein, 1961 *Advances in electronics and electron physics* Volume XIV (Academic press)
[2] G. Schaefer, M. Kristiansen, Guenther (edited), 1991 *Gas discharge closing switches* (Plenum Press, New York, USA)