Outgassing Rate Measurement of Copper Plated Stainless Steel

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Abstract: Stainless steel (SS) and Copper are very suitable structural metals for fabrication of UHV vessels. INDUS-1 is an electron storage ring, with 450 MeV electron energy. A new Radio Frequency (RF) cavity was fabricated for INDUS-1, with copper plating on SS. The main factor deciding the vacuum compatibility of the surface is the specific outgassing rate at room temperature as well as at the operating temperature. Surface properties are extremely important in this case. i.e., surface finish should be maintained without any blisters and micro cracks, after baking. In order to evaluate these factors, one sample was prepared (SS pipe plated with copper) and tested with simulated conditions in UHV environment. This paper gives the details of the experiments and the results on specific outgassing rate of the copper plated SS sample in different conditions, namely, at room temperature and after baking at different temperatures.

1. Introduction:
INDUS-1 is a 450 MeV electron storage ring dedicated for its application as a synchrotron radiation source. RF cavity is a very important, special and sensitive electrical component of the storage ring. To improve the total performance, including the vacuum requirements, a new RF cavity was designed and fabricated. The new RF cavity was made of SS with Copper plating inside. The main factor deciding the vacuum compatibility of any vacuum component is the specific outgassing rate, at room temperature and specifically at the operating temperature. The surface properties also play an important role in the functioning of RF Cavity.

To analyze the vacuum properties of the plated surface, one sample piece was made of the same SS material and was plated with Copper. This sample was then subjected to the following tests to study the vacuum compatibility.

2. Experimental details
2.1.Experimental set-up
The experiment was conducted in a system, based on AVS standard [1]. The method is based on the throughput method, using an orifice of known conductance.

The set-up consists of two identical, cylindrical chambers made of 304 L SS with internal diameter, D. (Figure1). The height of each chamber is 3D/2. The chambers are separated by an orifice with known conductance, and are provided with Bayard Alpert gauges (BAG) for pressure measurement. The lower chamber is provided with another port for connecting it to a Turbomolecular pump (TMP)
through an all-metal right angle valve. This TMP is used for roughing and pumping during baking of the system. A Sputter Ion Pump (SIP) is used to pump system in UHV range.

Outgassing rate has a strong dependence on the temperature. So the experimental chamber was provided with a thermocouple for the temperature measurement. This was used to evaluate the sample temperature.

2.2. Specifications

| Specification   | Details |
|-----------------|---------|
| D               | 14.8 cm |
| C               | 9 l/s   |
| Chamber area    | 1360 sq.cm |
| Sample          | 304L SS pipe, electroplated with copper, 0.1 mm thick |
| Area exposed    | ~1400 sq.cm (Diameter 11 cm, height 20 cm) |
| Total area      | 2760 sq.cm |
| BAG             | VG make |
| TMP             | Varian make, 250 l/s |
| SIP             | RRCAT make 270 l/s |
| Valve           | VG make |
| Flanges         | Conflat type |
| Seals           | OFHC Copper gaskets |
| Thermocouple    | Chromel-Alumel K type |

2.3. Calculations

This experiment uses the throughput method with known conductance, for the outgassing rate calculation. The throughput of the gas passing through an orifice at any instant of time is given by:

\[ Q_{\text{total}} \text{ (mbar-l/s)} = C \times (P_1 - P_2) \]

Where \( C \) is the conductance of the orifice (l/s) separating pressure \( P_1 \) and \( P_2 \) (mbar).
Specific outgassing rate can be calculated as:

\[
Q_{\text{specific}} \text{ (mbar-l/s- sq.cm)} = \frac{Q_{\text{total}}}{\text{area exposed to vacuum (sq.cm)}}
\]

If the specific outgassing rate of the sample is expected to be comparable with that of the chamber material (SS 304L in this case), area of the chamber should be added while calculating the specific outgassing rate of the sample. So, the area considered was 2760 sq.cm.

2.4. Sample details

The sample (SS 304 pipe) was ultrasonically cleaned in trichloroethylene for 30 minutes to remove oil, grease and dust particles.

The following cleaning sequence was adopted before electro-deposition to ensure good adhesion as SS forms passive oxide film:

- Alkali cleaning for 30 minutes at 55 to 60 °C for removing remaining oil and grease.
- Immersion in 20% Nitric acid – 1.5 % Hydrofluoric acid for 5 minutes at room temperature.
- Anodic cleaning in 20 % Sulfuric acid for 1 minute at a current density of 5 A/ square decimeter.
- Cathodic cleaning in 20 % Sulfuric acid for 1 minute at a current density of 5 A/ square decimeter.
- Nickel strike in sulphate bath at a current density of 3 A/ square decimeter for 15 minutes.
- Copper deposition from low acid bath consisting of: Copper sulphate 180 - 200 gm/litre, Sulfuric acid 38-40 ml/litre and chloride 30 – 40 ppm.
  At room temperature and at current density 2 A / square decimeter.

Electro deposition was carried out using a pulsed power supply with provision for periodic reversal. The deposition time was 20 seconds (forward cycle) followed by a reversal of 4 seconds (negative cycle) at a current density of 2 A / square decimeter. Thickness of copper deposited was 100 microns. Plating time was 6.5 hours.

2.5. Calculation of the pumping scheme of RF Cavity, the end-use of this experiment.

The pumping scheme of the RF Cavity can be explained as:

Total pumping provided on RF Cavity:

- SIPS: 2 no (Effective nominal pumping speed of two SIPS, on the cavity is ~200 l/s)
- Titanium Sublimation Pumps (TSP) (~1000 l/s): 2no (But TSPs are not being considered in calculating the ultimate base pressure of the RF Cavity, since they are used at intervals to manage the pressure rise during the operation).
- Vacuum requirement of RF Cavity: ~1*10^-9 mbar, without beam and RF power.
- So, the total outgassing rate, \(Q_{\text{total, tolerable}}\), from the ‘plated’ surface: ~2 *10^-7 mbar l/s.
- The inner area of the RF cavity, which has been plated with copper is ~ 4*10^4 sq.cm.
- Tolerable specific outgassing rate \(Q_{\text{specific}}\) of the Copper plated SS surface, can be calculated as: \(Q_{\text{specific}} = 2 *10^{-7} \text{ mbar l/s/} 4 \times 10^4 \text{ sq.cm} = 5 \times 10^{-12} \text{ mbarl/s-sq.cm.}\)

3. Experiments conducted

At first the outgassing rate of the chamber without sample was calculated. For this purpose, the set-up was assembled, and baked after confirming the leak-tightness. Baking was carried out at 300 °C, for 6 hours using flexible heaters. (Since the chamber was undergoing repeated baking-s, baking duration
was restricted to 6 hours). Pumping was done through TMP, during baking. Temperature of the chamber was noted down, at intervals. The SIP was conditioned, during the cooling cycle at ~150 °C, and was kept on continuously, when it attained the full capacity of pumping. TMP was isolated from the system, at this stage. Gauges were also degassed in hot condition. After confirming the stable readings of the gauges, P1 and P2 were noted down at definite intervals, for ~48 hours. Outgassing rates were calculated for each set of readings. Specific outgassing rate (~1*10^{-12} mbar-l/s-sq.cm) was calculated using the last set of readings.

The system was vented using liquid Nitrogen boil-off and the sample was inserted into the chamber. The thermocouple was kept in close contact with the sample. The system was assembled back, and subjected to leak detection. After confirming the leak-tightness, the system was baked.

The plating quality with respect to several factors like finish, adherence to the base material etc. had to be monitored and strictly maintained from the functional point of view of the final product, i.e., RF Cavity. So, it was decided to go for a lower temperature bake-out (sample at 120 °C), followed by the higher temperature bake-out (sample at 180-200 °C).

The baking was done in two stages. At first, with sample at 120 °C (chamber at 200 °C) for 24 hrs. The pressure readings were noted for 48 hrs. Then it was baked again with sample at 180-200 °C (Chamber at 250-280 °C) for 48 hrs. The temperature / pressure readings were noted down for 48 hrs. The same experimental procedures (as those with the empty chamber) of baking, conditioning of the pump & gauges and pumping were followed for these baking procedures also.

4. Results and Conclusions:
One graph (Graph-1) was plotted with temperature of the sample and temperature of the chamber against the time, during baking. Another one (Graph-2) was plotted with specific outgassing rate vs. temperature, during the cooling cycle, followed by continuous pumping at room temperature, for a duration of 48 hrs (after baking at, 120 °C and 200 °C).

A third graph (Graph-3) was plotted with the pumping down details of the final product, RF Cavity. RF Cavity was independently baked at a temperature range of 150°C to 200°C for 24 hrs. After 24 hrs of baking, and continuous pumping for ~24 hrs, ~2.5 x 10^{-8} mbar was achieved.
4.1. Observations

- The $Q_{\text{specific}}$ of the sample, at room temperature was $4.8 \times 10^{-11}$ mbar l/s-sq.cm, after the first baking cycle, sample at 120 °C, followed by pumping for 48 hrs.
- The $Q_{\text{specific}}$ of the sample, at room temperature was $5.6 \times 10^{-12}$ mbar l/s-sq.cm, after the second baking cycle, sample at 180-200 °C, followed by pumping for 48 hrs.
- The $Q_{\text{specific}}$ of the sample at ~120 °C was $5.6 \times 10^{-10}$ mbar l/s-sq.cm at the end of first baking.
- The $Q_{\text{specific}}$ of the sample at ~120 °C was $5 \times 10^{-11}$ mbar l/s-sq.cm, during the cooling down cycle after the second baking.
- The temperature of the chamber was ~100 °C higher than the temperature of the sample, throughout the baking cycle. After ~80 minutes of switching off of the baking, both the temperatures were same. Afterwards temperature of the chamber was falling down faster than the sample. After ~5 hrs of switching off of the baking, the temperature of the chamber was lagging behind that of the sample by 40 °C.
- The graph-3 above shows the vacuum behaviour of the RF cavity during cooling down cycle, after the baking. It was observed that the expected vacuum ($1 \times 10^{-9}$ mbar) was not achieved after 24 hours of pumping after baking. This result can be explained as, due to the shorter baking schedule (24 hrs only, in place of 48 hrs for the sample), shorter pumping time (24 hrs in place of 48 hrs for the sample), other complicated structures inside the RF Cavity and the trapped volume near the lip welding of the RF Cavity.

4.2. Conclusion

- The $Q_{\text{specific}}$ of the Copper plated SS was reduced by ~1 decade, when the baking temperature was increased from 120 °C to 200 °C. This reduction was observed both at 120 °C and room temperature. The $Q_{\text{specific}}$, after baking at 200 °C, $5.6 \times 10^{-12}$ mbar l/s-sq.cm at room temperature and $3 \times 10^{-11}$ mbar l/s-sq.cm at 50 °C, (the maximum operating temperature of RF cavity) are so nominal that the vacuum requirements of the RF cavity can be met easily.
- The plating quality was analyzed after the baking processes and was found satisfactory.
- The chamber/sample temperature graph will be useful to approximate the temperature of the Copper plated inner side of the RF Cavity, by measuring the outer temperature, during the baking/operation of RF Cavity.
- The vacuum requirement of the new RF Cavity of Indus-1 can be met, by extending the baking duration, prolonged pumping and proper conditioning, during the operation. It is confirmed that the Copper plating in the inner side of the Cavity will not affect the vacuum performance adversely.

5. Reference

[1] Redhead P A 2002, J. Vac. Sci. Technol. A 20(5) 1667-75

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