

Outgassing Rate of the Copper-plated Beam Tube for ISABELLE\*

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Summary

The ultrahigh vacuum system of the intersecting storage accelerator, ISABELLE, will consist of two interlaced rings of stainless steel beam tubes with a circumference  $2\frac{1}{2}$  miles each. To obtain a good heat conduction during bakeout and to reduce the resistive wall instability during beam operation, a 1mm thick copper coating will be electroplated to the outer surface of this 1.5mm thick beam tube. To minimize the beam loss due to beam-gas collision, the pressure inside the beam tube is required to be  $1 \times 10^{-11}$  Torr ( $N_2$  equivalent) or less. To achieve this ultrahigh vacuum, the outgassing rate of the 304LN stainless steel tubes has been reduced to  $\sim 1 \times 10^{-13}$  Torr·l/cm<sup>2</sup>·sec by vacuum firing at 950°C for one hour. However, during acid-bath electroplating of copper, significant amount of hydrogen will be reintroduced and trapped in stainless steel which will substantially increase the outgassing rate (to  $\sim 2 \times 10^{-12}$  Torr·l/cm<sup>2</sup>·sec). The outgassing characteristics of these copper-plated beam tubes are studied and discussed within the scope of diffusion and energy of activation. Methods to reduce the outgassing rate to an acceptable level ( $\sim 1 \times 10^{-13}$  Torr·l/cm<sup>2</sup>·sec) are also given.

I. Introduction

The Intersecting Storage Accelerator, ISABELLE, currently under construction at Brookhaven National Laboratory, consists of two rings having a circumference of 3.8 Km each. To provide a very clean environment for the circulating proton beam, the ultrahigh vacuum (UHV) system of ISABELLE is designed to operate at an average pressure of  $1 \times 10^{-11}$  Torr ( $N_2$  equivalent). This UHV system consists of  $\sim 1400$  8.8 cm diameter, 5.5m long stainless steel beam tubes pumped by a combination of titanium sublimation pumps ( $\sim 1000$  l/sec each) and sputter ion pumps ( $\sim 20$  l/sec each). The choice of 304 LN stainless steel as beam tube material as well as the need of copper sleeve on the outer surface of beam tubes has been described previously.

The ultimate pressure achievable inside such long tubes will depend on the pumping speeds of the ion pump and Ti sublimator, the conductance of the tubes and the outgassing rate of the beam tube material. The first two parameters are governed by the design of the other systems (superconducting magnets, cryogenics, etc.). The outgassing rate of the beam tubes should, therefore be lowered as much as possible. The outgassing rate of vacuum-fired (at 950°C with pressure  $< 1 \times 10^{-4}$  Torr for 1 hour) 304LN stainless steel beam tube is  $\sim 1 \times 10^{-13}$  Torr·l·cm<sup>-2</sup>·sec<sup>-1</sup> (Ref 3) which is acceptable.

The enormous force generated on the copper sleeve during the quench of superconducting magnets limits the method of copper coating to electroplating. Significant amount of hydrogen will be reintroduced into stainless steel during electroplating. This trapped hydrogen will later diffuse through stainless steel into UHV system and substantially increase the outgassing rate.

This paper describes the measurements of the outgassing rate of several stainless steel tubes with and without electroplated copper on the outer surface. The effects of heat treatment are also discussed within the scope of diffusion, and energy of activation.

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II. Measurements

The setup used in our study of the outgassing rate of the sample tubes is shown schematically in Fig. 1. Sample tube with one end sealed was attached through the bakeable valve to the monitoring chamber which was separated from the pumping station (with a pumping speed  $\sim 1000$  l/sec for hydrogen) by a copper gasket having an 8mm diameter orifice. A Bayard-Alpert type ionization gauge (with x-ray limit less than  $1 \times 10^{-11}$  Torr) was used to measure the pressure inside the monitoring chamber.

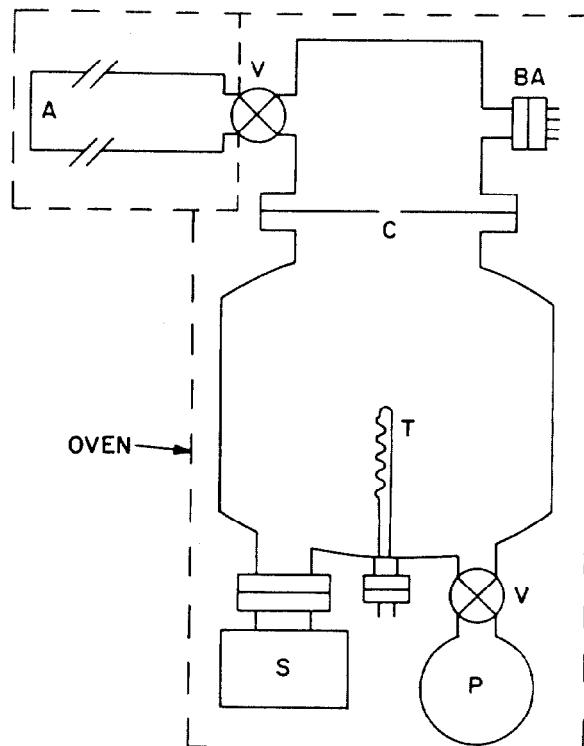


Figure 1: Experimental setup for outgassing measurements. A. - sample tube; V. - bakeable valve; BA. Bayard Alpert type ionization gauge; C. - orifice; T. - Titanium sublimator; S. - sputter ion pump; P. - Turbomolecular pump.

The calibrated conductance method<sup>3,4</sup> which measures the gas flow across the orifice was used in our study to obtain the outgassing rate  $Q$  of the sample tube.  $Q$  can be expressed as  $Q(\text{Torr}\cdot\text{l}\cdot\text{cm}^{-2}\cdot\text{sec}^{-1}) = C \times \Delta P/A$  where  $A$  is the total inner surface area of the sample tube,  $\Delta P$  is the change of the pressure inside the monitoring chamber between open and close of the bakeable valve, and  $C$  is the conductance of the orifice. A value of 20 l/sec for  $C$  was used in our calculation since  $>95\%$  of the outgassed species was found to be hydrogen<sup>5,6</sup>. The surface area  $A$  of each sample tube was  $\sim 7600$  cm<sup>2</sup> (8.8 cm O.D., 1.5 mm wall and 2.75 m long). The background pressure of the monitoring chamber with bakeable valve close was  $< 3 \times 10^{-11}$  Torr which ensured that outgassing rates down to  $1 \times 10^{-14}$  Torr·l·cm<sup>-2</sup>·sec<sup>-1</sup> were detectable.

Table I Outgassing rates Q of the sample tubes\* after sequential bakeouts

Sequential Bakeout Conditions		Outgassing Rate (Torr · l · cm <sup>-2</sup> · sec <sup>-1</sup> )						
Temp. (°C)	Duration (hr.)	A	B	C1	C2	C3	C4	D
300	24	1x10 <sup>-13</sup>	2x10 <sup>-12</sup>	3x10 <sup>-12</sup>	2x10 <sup>-12</sup>		2x10 <sup>-12</sup>	2x10 <sup>-12</sup>
300	70		2x10 <sup>-13</sup>	1x10 <sup>-12</sup>	6x10 <sup>-13</sup>	7x10 <sup>-13</sup>	1x10 <sup>-12</sup>	1x10 <sup>-12</sup>
500	24			1x10 <sup>-13</sup>	1x10 <sup>-13</sup>	2x10 <sup>-13</sup>	3x10 <sup>-14</sup>	3x10 <sup>-13</sup>

\*304 LN stainless steel tubes 3.5" O.D., 0.060" wall, 9' long with the following treatments:  
 Sample A: vacuum fired at 950°C for one hour at a pressure less than 1 x 10<sup>-4</sup> Torr.  
 Sample B: unfired  
 Samples C1-C4: vacuum fired at 950°C for one hour; electroplated with 1 mm thick copper on the outer surface  
 Sample D: unfired; electroplated as Samples C1-C4.

After chemically cleaning, some sample tubes were vacuum fired (at <1 x 10<sup>-4</sup> torr and at 950°C for 1 hr), sealed then electroplated with ~1mm thick copper, while others were either electroplated or vacuum fired. These sample tubes were then mounted on the test setup for outgassing measurements.

After the sample tubes had been baked out at either 300°C or 500°C for desired periods of time, the outgassing rates were determined using the calibrated conductance method described above. The bakeout conditions as well as the obtained outgassing rate for these sample tubes are listed in Table I. To understand the origin and the mechanism of the outgassing, measurements were also made at elevated temperatures. The results were plotted out in Figure 2 as a function of temperature.

gassing rate was observed after further bakeouts. The vacuum-fired electroplated tubes (samples C1-C4) showed outgassing rates around 2 x 10<sup>-12</sup> Torr · l · cm<sup>-2</sup> · sec<sup>-1</sup> after the initial bakeout. These were significantly higher than those of the unplated tubes (i.e. sample A). The outgassing rates of the plated tubes were reduced about one half after consecutive bakeouts at 300°C. A reduction of the outgassing rate to ~1 x 10<sup>-13</sup> Torr · l · cm<sup>-2</sup> · sec<sup>-1</sup> (the acceptable outgassing rate for ISABELLE beam tube) was achieved only after a 24 hr. bakeout at 500°C.

The outgassing rates of two unfired tubes were also studied. The electroplated tube (sample D) had outgassing rates similar to those of the vacuum fired then electroplated tubes. After the initial bakeout, the outgassing rate of the unfired tube (sample B) was consistent with that reported by Halama, however it dropped off rapidly to 2 x 10<sup>-13</sup> Torr · l · cm<sup>-2</sup> · sec<sup>-1</sup> after the second 300°C 70 hour bakeout. More measurements on other unfired and unplated tubes are in progress to verify these results.

The rate controlling step and mechanism of the outgassing from stainless steel tubes (either vacuum fired or not, and either electroplated or not) can be understood qualitatively from the heat of diffusion (ΔE) of hydrogen which is the only diffusible gas inside the bulk of the stainless steel. By plotting the outgassing rate as a function of temperature, as shown in figure 2, the heats of diffusion ΔE can be calculated from the slopes of the curves according to

$$Q = Q_0 e^{-\Delta E/RT}$$

where R is the gas constant (1.99 cal · K<sup>-1</sup>). The measured ΔE values for both the electroplated tube (sample C1) and the bare stainless steel tube (sample A) are 10 ± 1 Kcal/mole. This similarity in heat of diffusion suggests that the rate determining step in both tubes is the diffusion of hydrogen through stainless steel and the presence of the electroplated copper has little influence.

The measured ΔE values for the two unfired tubes (samples B and D) are several Kcal/mole lower than those of the vacuum fired tubes; at lower temperature (T < 160°C) the measured ΔE values for these two tubes also decrease somewhat. These variations in heat of diffusion with temperature and between vacuum fired tubes and unfired tubes can be explained qualitatively on the basis of lattice diffusion and grain boundary diffusion (phase boundary diffusion).

Barrer classified the diffusion of gases (especially hydrogen) in metal into two major categories; the lattice diffusion and the grain boundary diffusion. The activation energy required for grain boundary diffusion is less than that of lattice diffusion, therefore, at lower temperature the grain boundary diffusion process occurs much more rapidly than lattice diffusion. The vacuum firing process and high temperature bakeout anneal the stainless steel, and enhance the growth of the crystallites in the stainless steel which

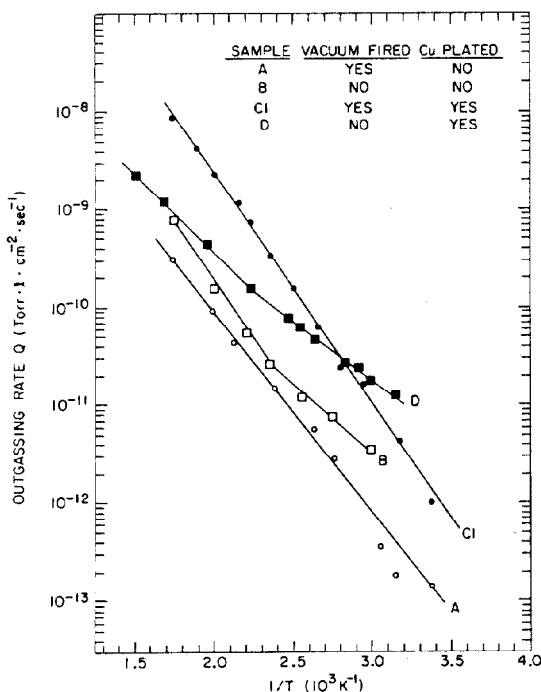


Figure 2: Outgassing rate of the stainless steel tubes as a function of the temperature.

### III. Results and Discussion

After the initial 24 hr. bakeout at 300°C, the outgassing rate of the vacuum fired tube (sample A) was ~1 x 10<sup>-13</sup> Torr · l · cm<sup>-2</sup> · sec<sup>-1</sup> which is consistent with previous studies<sup>3,4</sup>. No significant decrease in out-

lowers the amount of grain boundary diffusion. The slopes of curves in Figure 2 suggest that the lattice diffusion process dominates the vacuum fired tubes while the unfired tubes contain a mixture of lattice diffusion and grain boundary diffusion with grain boundary diffusion dominating at lower temperatures.

#### IV. Conclusion

The vacuum fired stainless steel tubes have an outgassing rate of  $\sim 1 \times 10^{-13}$  Torr·l·cm<sup>-2</sup>·sec<sup>-1</sup> which can meet the pressure requirement of the ISABELLE UHV system, therefore, can be used as the ISABELLE beam tubes. The additional hydrogen entrapped into stainless steel during the electroplating of copper raises the outgassing rate of the stainless steel tube to  $\sim 2 \times 10^{-12}$  Torr·l·cm<sup>-2</sup>·sec<sup>-1</sup>. By baking-out at higher temperature (i.e. 500°C for 24 hours), the outgassing rate could be lowered to acceptable level ( $1 \times 10^{-13}$  Torr·l·cm<sup>-2</sup>·sec<sup>-1</sup>).

The origin and mechanism of outgassing can be classified as either lattice diffusion or grain boundary diffusion or a mixture of both. The lattice diffusion process is the dominant process in vacuum fired tubes, while the diffusion process of the unfired tubes contains the mixture of the lattice and the grain boundary.

With an expected outgassing rate of  $\sim 1 \times 10^{-13}$  Torr·l·cm<sup>-2</sup>·sec<sup>-1</sup> for the ISABELLE beam tubes (after electroplating and high temperature bakeout), the achievable pressure inside the beam tubes can be estimated according to

$$P(x) = P_0 + Q \left[ \frac{L}{S} + \frac{x}{C} \left( L - \frac{x}{2} \right) \right]$$

Where  $P_0$  = the pressure at pumping station ( $\sim 5 \times 10^{-12}$  Torr)

$Q$  = outgassing rate ( $1 \times 10^{-13}$  Torr·l·cm<sup>-2</sup>·sec<sup>-1</sup>)

$L$  = one-half the distance between pumping station (275 cm)

$S$  = pumping speed at pumping station (1000 l/sec = 10 cm<sup>3</sup>/sec)

$x$  = distance from pumping station ( $x=L$  at center of beam tube)

$C$  = conductance of beam tube per unit length ( $3.1 \times 10^4$  cm<sup>3</sup>/sec)

The pressure halfway between pumping stations (higher pressure bump) will be  $P_0 + 4 \times 10^{-12}$  Torr. and the averaged pressure inside the beam tubes will be  $P_0 + 3 \times 10^{-12}$  Torr.

#### V. Acknowledgements

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