A Comparison of Outgassing Measurements for Three Vacuum Chamber Materials

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Abstract. Outgassing measurements of three UHV materials (304 Stainless Steel (SS), 316L SS and 6061T-6 aluminum) were made using the conductance and accumulation techniques as given in the AVS Recommended Practice for Outgassing Measurements [1]. Measurement results indicate good agreement between the two techniques. This study was undertaken to help determine the vacuum limitations of the photoelectron guns used at Jefferson Lab and to aid in the choice of vacuum chamber materials that will be used in the construction of future photoelectron guns. The outgassing rate measured for all of the chamber materials, regardless of technique, was about $1x10^{-12}$ Torr·l/s·cm^2.

INTRODUCTION

Jefferson Laboratory's accelerator delivers polarized electron beams with energies up to 6 GeV to three experimental halls. The polarized electrons are produced from photoemission cathodes in 100 kV electron guns. The vacuum requirements for these guns are very demanding, since the photocathode lifetime is limited by ion backbombardment from the ionization of the residual gas in the cathode-anode gap. To better understand the vacuum chamber limitations of the photoguns presently used at Jefferson Lab and in the interest of obtaining lower vacuum pressure and longer gun operating lifetime in the future, outgassing measurements were made in vacuum chambers constructed of 304 stainless steel, 316L stainless steel and 6061T-6 aluminum. Two independent techniques, called the orifice method and the rate of rise method, were used to measure the outgassing rates for each chamber. A description of the chambers, the measurement techniques and the results of the study are described in the following sections.

CHAMBERS

To compare the performance of other vacuum chamber materials to that of our standard 304 stainless steel, new chambers were constructed from 6061-T6 aluminum and 316L stainless steel. The three chambers are of similar size, with a volume about 15 liters and a surface area of approximately 4000 cm^2. Each chamber consists of a 10 inch diameter cylindrical body with two 13¼ inch diameter conflat end flanges. At one end, the chamber is separated by an all-metal valve from a bake pump that consists of a cross with a 30 l/s ion pump, a Residual Gas Analyzer (RGA) [2], and a NonEvaporable Getter (NEG) [3] pump. At the other end of the chamber, an orifice with a conductance of 0.862 l/s separates the chamber from an all metal valve, a 30 l/s ion pump and an extractor gauge. Small ports on the sides of each chamber allow an extractor gauge and a spinning rotor gauge to be mounted in the main chamber.

The 304 stainless steel chamber had previously been used in our production photogun with no chemical or mechanical preparation of the surface. The nominal roughness of this chamber (measured by means of a portable surface profilometer [4]) is 3.7 μm. This chamber had undergone 10 baking cycles before being used in these tests. The 316L stainless steel chamber was constructed for the purpose of these experiments. Prior to attaching the flanges to the chamber, it was vacuum fired at 900°C and a pressure of $5x10^{-6}$ Torr for 4 hours. The chamber was then electropolished and the flanges were welded on. The measured surface roughness for this chamber was 0.8 μm. The third chamber is constructed from 6061-T6 aluminum by Atlas Bimetal [5] using a process to join stainless steel flanges to a short aluminum stud. An aluminum weld was then used to join the flange and stud to the aluminum walls. The aluminum weld and the bimetallic seam have been robust through 4 bakes at 250°C for 30 hours each. The surface roughness for this chamber was measured at 1.3 μm.

CHAMBER PREPARATION

In order to measure the effective outgassing rates of each chamber, the stainless steel chambers were degreased with Micro [6], while the aluminum chamber underwent a chemical cleaning with the SLAC aluminum cleaning procedure [7]. Prior to assembly, all chambers were cleaned with acetone and methanol in an ultrasonic cleaner and then rinsed in hot de-ionized water. The chambers were assembled and roughed down to $1x10^{-5}$ Torr using a Drytel [8] molecular drag pump. The ion pumps were turned on and the chamber was pumped overnight and then leak checked using the RGA. A hot-air oven was assembled around the chamber, and the chamber was baked for 30 hours at a temperature of 250°C. Following the bakeout, the chamber was again leak checked with the RGA and the filaments of the extractor gauges were degased using the manufacturer's degas cycle and allowed to settle for at least 12 hours before any measurements were made.

OUTGASSING MEASUREMENTS
Orifice Method

To measure the effective outgassing rates of each chamber, two measurement techniques were used. The first method, known as the orifice or throughput method [1], requires two chambers to be separated by an orifice of known conductance (see Figure 1). The test chamber has side walls made of the material under test and contains a calibrated extractor gauge [9]. The pumping chamber on the other side of the orifice has an ion pump and another calibrated extractor gauge. Before the test begins, both valves are open and the pressure throughout the apparatus is less than $1x10^{-10}$ Torr. The main chamber ion pump is then valved off leaving only the conductance limited pumping through the orifice. Values of the pressure on both sides of the orifice, measured after allowing 10 hours for the conductance limited pumping to stabilize, are shown in Table 1.

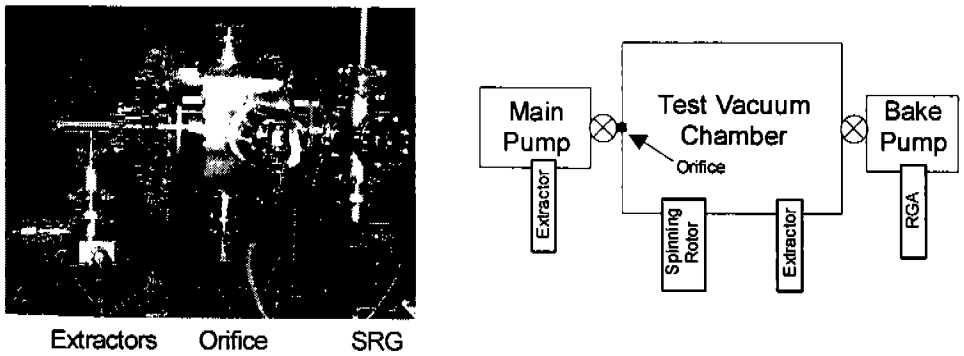

Figure 1. Orifice method experimental setup. Orifice separates the test chamber from a pumping chamber. Both sides of the chamber are pumped down, then the valve between the test chamber and the bake pump is closed leaving only conductance limited pumping through the orifice.

TABLE 1. Orifice Method Pressures

	Chamber Pressure (Torr)	Pump Pressure (Torr)
304 Stainless Steel	$1.85x10^{-9}$	$1.67x10^{-10}$
316L Stainless Steel	$2.46x10^{-9}$	$2.16x10^{-10}$
6061-T6 Aluminum	$1.96x10^{-9}$	$1.26x10^{-10}$

Table 1. Pressure on both sides of the orifice are shown here after pumping the chamber down to ultimate pressure then removing all pumping except for through the orifice. The conductance limited flow through the orifice was allowed to stabilize for 10 hours before readings were made in each case.

The outgassing rate Q can be determined using the equation $Q = \frac{C \cdot \Delta P}{A}$ with C the conductance of the orifice, ΔP is the pressure difference between the two sides of the orifice as measured by the two extractor gauges, and A the surface area of the chamber under test.

The orifice method has the advantage that it can be made in a short time. However, its relies on the calibration of two UHV vacuum gauges and the calculated conductance of the orifice, which will lead to some uncertainty in the measurements. A correction factor must be used with the extractor gauges to account for the fact that the gauge is calibrated for nitrogen and the gas in the system is over 90% hydrogen [10]. In addition, the hot cathode of the extractor gauges affect the vacuum quality, releasing gas at low pressures and pumping at higher pressures [11].

Rate of Rise Measurement

The second outgassing rate measurement is known as the rate of rise or accumulation method [1]. In this method, the chamber under test is closed off from all pumping and the pressure is tracked as a function of time. In order to minimize the disturbance of the vacuum during this measurement, a spinning rotor gauge (SRG) [12] was used, which is a direct reading gauge and unlike hot cathode gauges not affect vacuum quality. The outgassing rate, Q, can be calculated using $Q = \frac{\Delta P}{\Delta t} \cdot \frac{V}{A}$ where ΔP is the final minus the initial pressure, Δt is the time (in seconds) of the measurement, V is the volume of the chamber under test, and A is the surface area of the chamber.

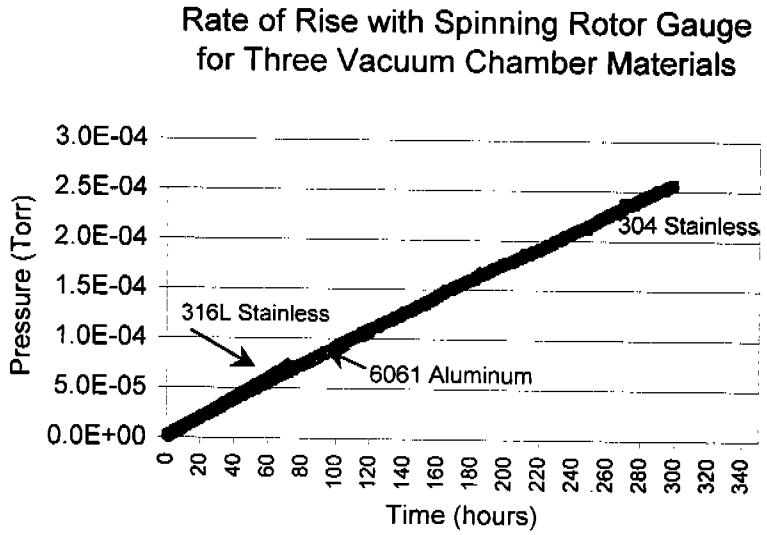

Figure 2. Pressure vs. time data for the three chamber materials is shown here. Each chamber was first baked for 30 hours at 250°C then pumped down to the chamber base pressure (~$1x10^{-10}$ Torr) before removing all pumping and tracking the rate of rise. For rate of rise outgassing measurements, only the first 70 hours of data for each chamber were considered.

This method allows an analysis of the gases accumulated throughout the measurement. At the end of the rate of rise measurement, the accumulated gas is at a pressure of ~$1x10^{-3}$ Torr. The valve can be opened to the RGA in the Bake Pump region and the species of gas which have accumulated can be determined. As

expected, hydrogen is the predominant gas species (over 90%) in this UHV system, with CO and CO_2 as less prevalent gases. Since both the SRG and the extractor gauge require a calibration factor for the species of gas, knowing the composition of the accumulated gas is quite useful.

The major drawback of the rate of rise method is the long time that is necessary for the measurement. Since room temperature fluctuations and vibrations can affect data, it is necessary to make these measurements for times of at least 30 hours and even as long as 100+ hours. In addition, the SRG cannot read pressures under $1x10^{-6}$ Torr.

RESULTS

The outgassing rates determined by both the orifice method and the rate of rise method agree quite well. Data shown in Table 2 summarizes the results of the two different methods for each chamber material.

TABLE 2. Measured Outgassing Rates

Chamber Material	Orifice Method (Torr·l/s·cm^2)	Rate of Rise Method (Torr·l/s·cm^2)
304 Stainless Steel	$0.97x10^{-12}$	$1.1x10^{-12}$ (70 hours)
316L Stainless Steel	$1.3x10^{-12}$	$1.2x10^{-12}$ (70 hours)
6061-T6 Aluminum	$1.1x10^{-12}$	$1.1x10^{-12}$ (70 hours)

CONCLUSIONS

The outgassing measurements reported here indicate that the three chamber materials have similar outgassing rates independent of measurement technique. The measured outgassing rates for each material regardless of the method was quite close to $1x10^{-12}$ Torr.l/s.cm^2. Previous measurements had indicated that aluminum might have a lower outgassing rate than stainless steel [13]. The 316L stainless is a lower carbon content material than the 304 stainless, which was also believed to lower hydrogen content in the walls and outgassing rate. However, the results of these measurements could not determine a significant difference between the outgassing rates of the three chamber materials.

Several possible explanations exist for the measured results. In this experimental setup, the test chambers had only sidewalls made of the material under test while the end flanges were the same 304 stainless in all circumstances. Thus, only half of the surface area of each chamber was the material under test, and this relatively small percentage of surface area could mask the differences in the material properties.

Secondly, the accumulated heating times and surface preparations varied for the three chambers under test. The 304 stainless chamber had been baked at least ten

times before starting the testing. Though stored in air between uses, the accumulated baking time of ~300 hours may have led to a lowered hydrogen content in the material and a lower outgassing rate. The successive bakes also have caused a distinct yellow oxide to grow on the outside of the chamber, which may play a role in limiting any possible diffusion of hydrogen through the walls of this particular chamber. The 316L stainless chamber was vacuum fired at 900°C for four hours, which was intended to lower the hydrogen content in the walls. Paul Redhead [14] has proposed that a vacuum firing step such as this can move hydrogen from deep traps to shallow traps causing an increase in outgassing rate. In addition, this vacuum firing was followed by an electropolishing step which can increase the hydrogen content in the walls and thus increase the outgassing rate. Ideally, electropolishing would occur before the vacuum firing procedure to liberate any deposited hydrogen. Both the aluminum and the 316L stainless were new chambers at the beginning of these tests, leading to a much shorter accumulated bake time than the 304 chamber. The effect of the chemical cleaning on the hydrogen content in the walls of the aluminum chamber should also be characterized.

FUTURE STUDIES

The goal of these studies is to determine the vacuum materials and preparation techniques that will optimize the operating pressure of the Jefferson Lab photoinjector in order to increase the lifetime of the photocathodes. Future work will include a comparison between different coatings on the chambers such as NEG, SiO_2 or TiN treatments. In addition, a systematic measurement will be made of the changes in outgassing rates introduced by vacuum windows and high voltage ceramics that are needed in these photoguns.

ACKNOWLEDGEMENTS

This work was supported by US DOE Contract No. DE-ACO5-84-ER40150.

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