APS Storage Ring Vacuum Chamber Section 1 Evaluation

R. Benaroya and B. Roop
Advanced Photon Source
Argonne National Laboratory
Argonne, IL 60439

Abstract

The vacuum characteristics of the APS storage ring vacuum chamber prototype, Section One (S1), is presented. The base pressure achieved was 4 x 10^{-11}, the welds contained no virtual or real leaks, the NeG strip mounting design and activation procedures have been determined, and S1 was found contaminated with hydrocarbons.

Introduction

This is an evaluation of the characteristics of the first APS Storage Ring prototype, section one chamber with NeG strips, tested in January 1990.

Experimental Set–up

A drawing of S1 is shown in Fig. 1. The chamber was held by an angle iron frame. A 360–ℓ/sec turbomolecular pump, separated from the chamber by an all–metal angle valve, and two 30–ℓ/sec ion pumps were mounted to the chamber. A quadrupole mass spectrometer type residual gas analyzer (RGA) was attached using a 2–3/4" cross. A nude ion gauge was mounted through a 5" extender on a 2–3/4" flange. Thermocouples were taped to the outside chamber wall and heating tapes were wrapped around the chamber with foil layered over them. A pair of 120–inch–long St 707 type NeG strips were mounted on stainless steel strips, electrically isolated by ceramic insulators. They were inserted into the chamber NeG slots and connected, internally, in series. The gettering material consisted of 70% Zr + 24.6% V + 5.4% Fe. It was coated, with a nominal thickness of 0.07 mm and a width of 27 mm, on each side of a 30–mm–wide Constantan (55% Cu + 45% Ni) substrate. A J–type thermocouple was welded on the bare Constantan, at the edge of the NeG strip, before insertion into the chamber.

Discussion

Baking

The base pressure of S1, after baking, as measured on a nude ion gauge was 4 x 10^{-11} Torr, with active NeG strips and ion pumps operating. Since leaks occurred during baking and the NeG strip mounting had to be modified, the chamber underwent several bake cycles. For the final bakeout, the chamber was baked at 150° C until the pressure stabilized approximately at 4 x 10^{-9} Torr after 36 hours. A general rule of thumb is that the pressure of a vacuum system will be two orders of magnitude lower after the chamber is cooled. Kinetic processes determine how many gas molecules are removed from a vacuum system and are a function of time and temperature. If a system is baked at a higher temperature, less time is required to reach an equilibrium pressure. Alternately, a lower temperature bakeout will require a longer time to reach the equivalent pressure. The kinetics of each system should be determined independently by allowing the pressure to stabilize at the bakeout temperature before cooling is initiated.

Initially, the lowest apparent pressure obtained was 1 x 10^{-10} Torr. However, the temperature of the extender on which the ion gauge was mounted was measured by a thermocouple to be 63 °C while the rest of S1 was at 23 °C. After the extender was cooled with a fan to 39 °C, the pressure reading was 4 x 10^{-11} Torr. The higher pressure reading of 1 x 10^{-10} Torr was the result of both electron and radiative heating from the filament of the ion gauge. A higher local pressure was created and the ion gauge recorded a value which was not indicative of the true chamber base pressure. Two methods to eliminate this situation are possible. The area surrounding the gauge should be cooled or area surrounding the ion gauge should be enlarged

Leak Checking

The welds contained no virtual or real leaks. This was concluded after the system had been leak tested using an external leak detector through the turbomolecular pump. In the future, the RGA will be used to determine whether there is a possibility of a leak and to leak test *in situ*. The leak detector will be attached only in cases when problems arise in leak detection due to reasons of poor conductance to the RGA. Generally, if there is a leak, the intensity ratio of mass 28 to 32 will be 3:1 or 4:1. That ratio can be modified if, for example, oxygen is pumped by the walls of the chamber or the system has been extensively outgassed. In that case, the N_2 molecule (mass 28) to N atom (mass 14) ratio of 12:1 can indicate a leak. Care must be taken to include the methane cracking fragment at mass 14 in the total mass 14 signal intensity. Additionally, if argon has not been introduced into the system, then there should be no signal at mass 40.

Conflat Seal Problems

Leaks occurred at the flanges both before and after baking. Only Ulvac flanges, lubricated aluminum bolts, aluminum washers and unannealed aluminum gaskets were used. Leaks occurred between aluminum–to–aluminum flanges and between aluminum–to–stainless steel flanges. After detection of a leak, the bolts were retorqued. The leaks which appeared after baking were attributed to the expansion of the aluminum bolts during the 150 °C bake. Eventually heat cycling no longer caused leaks to occur. One reason for this might have been the fact that the flanges had been tightened enough so they were flange to flange. Another reason for this apparent cure could have been due to the annealing of the gaskets during bakeouts. The tightening sequence which distributes the forces gradually and evenly must be used when torquing.

NeG Operation

The current required to raise the NeG strip temperature to 450 °C with a 150 °C chamber was approximately 40 amps. A new NeG strip was conditioned at 450 °C for 40 minutes. The pressure increased from 1 x 10^{-9} to 1 x 10^{-5} Torr. The gases desorbed were mainly hydrogen, carbon monoxide, water, and methane. The NeG strip mounting had to be modified after the top and bottom strips shorted against each other and deposited material onto the chamber wall. This result was attributed to the sagging of the top strip and the upward expansion of the bottom strip as they were heated during the conditioning step. This condition was eliminated by ensuring that the NeG strip was taut after insertion into the chamber and by staggering the top and bottom support clips. In this manner, the expansion of each strip during the heat cycle would not be in the same spatial area.

The residual gas analysis indicated that the gases common to an ultrahigh vacuum (UHV) system were present. These gases are H_2 (m/e = 2,1), CO (m/e = 28), H_2O (m/e = 18,17), CO_2 (m/e = 44) and CH_4 (m/e = 16,15) and perhaps O atoms (m/e = 16). CH_4 is generally assigned, based on its cracking pattern, at m/e = 15. The ratio of mass 16 to mass 15 should be 10:8. The RGA shows the ratio to be closer to 6:1. This means that the m/e = 16 intensity cannot be assigned solely to CH_4 especially since the cracking pattern for H_2O is normal.

Generally, the H_2O peak at m/e = 18 should be less intense than the CO peak at m/e = 28. That intensity ratio (18<28) indicates that a major portion of the water has been removed from the system and therefore the optimal pressure has been obtained. However, the H_2O peak in S1 always remained above the CO intensity. The most common reason for this symptom is the presence of a cold spot. However, it is difficult to believe an aluminum chamber would have a cold spot since aluminum is so conductive. Additionally, the temperature of the bakeout was monitored by a number of thermocouples dispersed along the chamber indicating that no cold spots were present. A higher local pressure due to the radiative and electron heating, analogous to the problem with the ion gauge might be the cause of the higher than expected H_2O intensity. Both the ion gauge and the RGA have an open grid design.

There was an abnormally large carbon peak at m/e = 12 (see Fig. 2) and the RGA indicated that oil and/or hydrocarbon contamination was present (Fig. 3). An ultrahigh vacuum system should be hydrocarbon free. No hydrocarbon peaks should be detected at any gain level in the RGA. The carbon peak was present at the outset, i.e., before the NeG shorted. The pattern of peaks after m/e = 44 is a signature for oil contamination where the cluster of peaks are at an average interval of 14 mass units (CH_2). Several baking cycles did not completely eliminate the oil although the intensity of the contaminant peaks did decrease.

The source of the extremely large carbon peak at m/e = 12 could result from a reaction at the tungsten filament with the hydrocarbons that are present to form tungsten carbide which then cracks to produce the carbon signal. Its source from graphite is unlikely since graphite is not volatile. It is energetically unfavorable for a hydrocarbon to be so efficiently stripped of its hydrogens that the parent peak cannot be identified. In other words, most of the intensity at m/e = 12 is not a cracking fragment unless the instrument has been improperly tuned.

If the RGA is not tuned properly, the intensity and resolution of the peaks will not be correct. If this is a problem with the RGA mounted to S1, the intensity of the carbon and water peaks could be artificially high. One quick method to test this hypothesis is to check a cracking pattern by introducing argon which has peaks at m/e = 40 and 20. The best method is to tune with a gas mixture of helium, neon, and xenon; since they are inert and therefore do not stick to the chamber walls. As a result, the partial pressure of these gases is stable as the tuning process continues.

It is highly probable that the carbon peak at m/e = 12 along with the fluorine peak at m/e = 19, and the two isotopes of chlorine at m/e = 35 and 37 are the result of electron induced desorption (EID) from the chamber walls and/or the ionizer region of the RGA. In the case of

EID, none of these peaks would be apparent if the area around the RGA ionizer region were enlarged. Since the cross that the RGA is mounted on is stainless steel, these peaks would not be indicative of aluminum EID products.

Conclusions:

- The ion gauge should be placed in a larger OD extender.
- The area surrounding the RGA ionizer should also be enlarged.
- The base pressure was 4×10^{-11} Torr with the ion pumps operating along with the NeG strips.
- The welds were leak tight.
- There were no virtual leaks.
- The NeG strip mounting design has been developed.
- The proper sequence for torquing bolts should be used.
- The chamber should be baked until the pressure stabilizes at the higher temperature before cooling is initiated.
- In production chambers, the use of extra strength stainless steel bolts (300 series) and a through bolt flange geometry, making flange to flange registering[1] and overtorquing possible, eliminated the leak problems during heat cycling.
- The use of nonlubricated bolts eliminated hydrocarbon contamination of the chambers.

Acknowledgments:

The assistance of R. Nielsen, R. Prien, and E. Wallace in setting up and running the test is gratefully recognized and very much appreciated.

References:

[1] J. D. Gonczy, et al., “UHV Seal Studies for the Advanced Photon Source Storage Ring Vacuum System,” Proceedings of the 1991 IEEE Particle Accelerator Conference, pp. 2304–6, 1991.

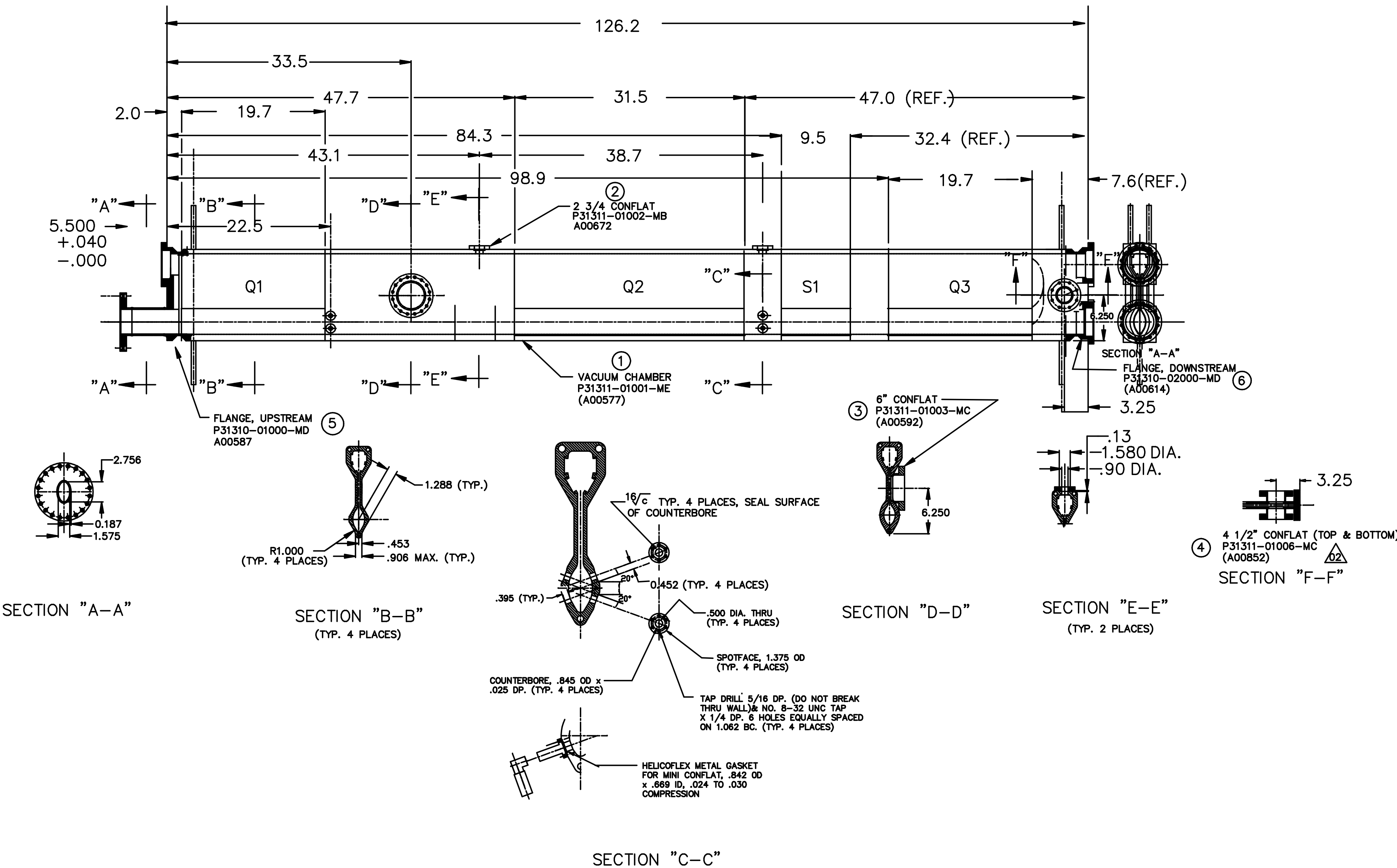

Fig. 1. Drawing of Section 1

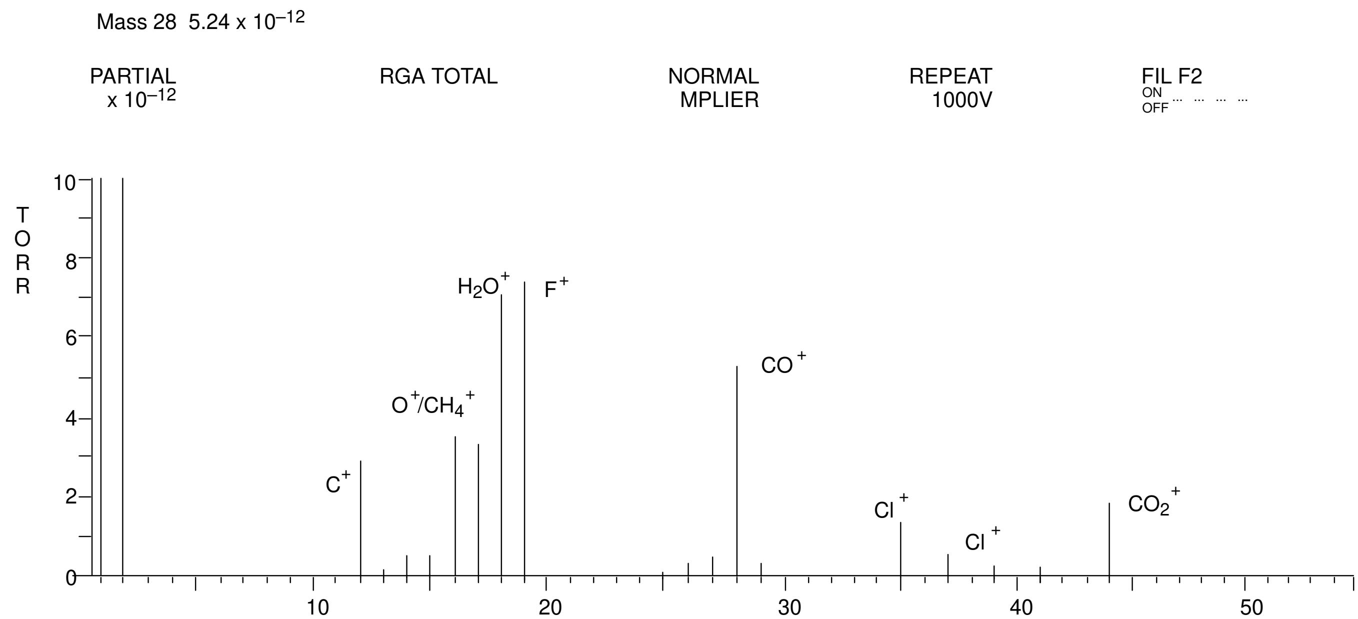

Fig. 2. Residual Gas Analysis of S1 after the final bakeout. The base pressure was 4 x 10^{-11} Torr measured on a nude ion gauge. The turbomolecular and ion pumps were on and NeG strips were activated.

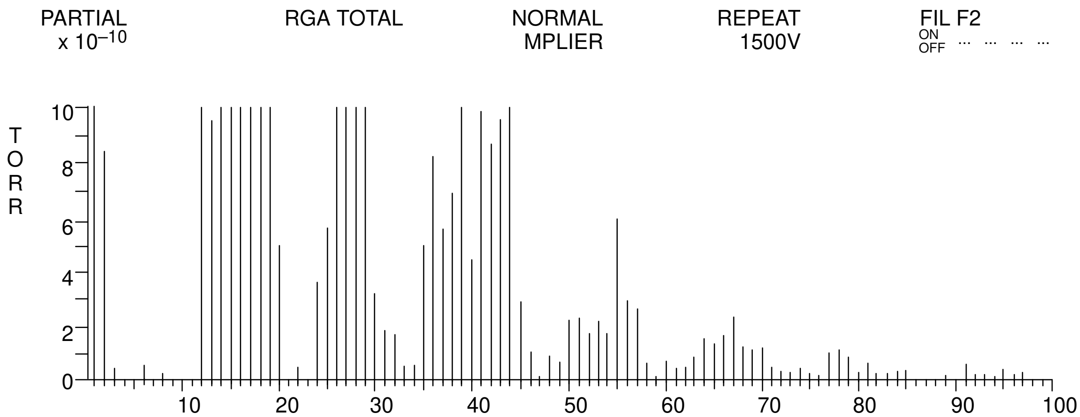

Fig. 3. Residual Gas Analysis of S1 at a higher gain than in Fig. 2. the cluster of peaks about m/e=50 indicates hydrocarbon contamination.