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METAL AND ELASTOMER SEAL TESTS FOR ACCELERATOR APPLICATIONS*

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ABSTRACT

The vacuum system of the Alternating Gradient Synchrotron (AGS) at Brookhaven National Laboratory has more than a thousand metal vacuum seals. Also, numerous elastomer seals are used throughout the AGS to seal large beam component chambers. An accelerator upgrade program is being implemented to reduce the AGS operating pressure by x100 and improve the reliability of the vacuum system. This paper describes work in progress on metal and elastomer vacuum seals to help meet these two objectives. Tests are reported on the sealing properties of a variety of metal seals used on different sealing surfaces. Results are also given on reversible sorption properties of certain elastomers.

INTRODUCTION

The vacuum system of the Alternating Gradient Synchrotron (AGS) at Brookhaven National Laboratory has more than a thousand metal vacuum seals. Also, elastomers are used to seal numerous large beam component chambers such as ion profile monitors, wire and magnetic septa, tune meters and chambers containing ferrite magnets. An accelerator upgrade program is being implemented to reduce the AGS operating pressure by x100 and improve the reliability of the vacuum system. Elimination of the elastomers and use of more reliable metal seals is required to meet these objectives.

Two years ago, most of the metal vacuum seals used throughout the AGS were Indium coated, Inconel® X750 C-rings. These metal seals proved to be unreliable for reasons including spontaneous cracking of the Inconel C-ring and poor shelf life. The cracking phenomenon, usually completely severing a minor diameter, might happen while stored in the shipping container. In instances it has occurred to C-rings installed in the AGS, with catastrophic consequences. Inconel C-rings are usually age-hardened for added strength. They are then In coated. It is believed that this coating process results in hydrogen embrittlement which, in turn, causes the spontaneous cracking.

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The second problem is the deleterious oxidation of the In coating; this shelf-life problem can occur after storage for a year or more. It has not been verified that installed seals have problems because of this oxide formation. However, installed seals, which were once vacuum tight, from time to time inexplicably develop leaks.

Seals with this oxide layer, probably InO, will leak even under ideal test conditions. Because of this problem, technicians developed the procedure of toroidally wrapping all new and reused C-rings with three layers of In foil. This was labor intensive and caused virtual leaks on installation; however, it was the only means whereby reliable vacuum seals could be achieved.

The spontaneous cracking problem has been "solved" on several occasions by better process control. But, the vendors tend to forget the recipe. The problem of InO formation could be solved by better packaging or even storage under vacuum. However, these problems prompted us to seek alternative seal configurations.

A seal test program was initiated to identify more suitable metal seals. It was also hoped this work would also lead to the identification of metal replacement seals for the many large-diameter elastomer seals used in the AGS. Neglecting reliability problems due to radiation effects on elastomers and beam loss considerations, the presence of elastomers within the AGS results in the loss of experimental beam time. For example, accelerator sector pumpdown times are often extended by x2 due to the reversible gas sorption mechanisms of these elastomers. To both educate and motivate the staff, verification tests were conducted on outgassing rates of elastomers. These data were augmented with the results of elastomer weight-loss studies conducted at the Stanford Linear Accelerator Center (SLAC) several years ago. These results are also given.

SEALING BOUNDARY CONDITIONS

The seal boundary conditions were established by the present hardware in the AGS. Seals had to be suitable for use in existing bolted flanges with O-ring type grooves. Also, they had to be suitable for sealing Marmon-type flanges, ranging from 180 - 330 mm ϕ, and secured with four-segment, $A\ell$ clamps. In this latter application, seals are mounted between the flanges with a removable retainer, rather than being retained in O-ring grooves. Seals had to be capable of sealing against flat stainless steel, aluminum and porcelain enamel coated surfaces, all having nominal surface finishes of $\approx 1.0\ \mu m$. Some of the existing flanges have scratched surfaces. Over 500 enamel coated flanges are used in the AGS. These coated flanges are used to electrically isolate flange pairs. The sealing force for clamped, flange pairs was set at ≤ 1400 N/cm; this was set by the design of new clamps to be installed with the successful seal candidate. "Tapping" of clamps to achieve uniform sealing was prohibited, as the distinction between "tapping" and "pounding" is subjective. Prior to installing clamps, they are first sprayed with a dry lubricant.

Criteria weighed when selecting vacuum seal designs include the following: safety; reliability; bakeability; seal permeation rate; material outgassing rates; mating surface materials; mating surface finish; ease of installation (e.g., technique involved); required sealing force; applicable flange sizes; provision for cleaning of parts; initial costs; operating costs (i.e., replacement and reusability); seal availability for replacement; degree of radiation "hardness"; handling fragility, and shelf life.

METAL SEALS

Varieties of all-metal seals include the: ConFlat-type (i.e., knife-edge, coined gaskets, or stepped);[1] Wheeler● (special wire seal);[2] Foil (Aℓ, In, Cu);[3] Wire (Aℓ, Au, Ag, Cu, In, etc.);[3] C-rings, coated with soft metals such as In or Pb, and which may or may not be reinforced with a spring; C-rings of Inconel, jacketed with a ductal metal such as Aℓ, Cu and spring reinforced;[4] C-rings, with "diamond" edges, and spring reinforced (i.e., the Helicoflex Delta● Seal; Fig. 1)[5] and "diamond" seals (Fig. 2).[5]

Wheeler sufficiently characterized the first two varieties of seals for them to be dismissed as viable candidates, for reasons already noted.[1,2] Also, installing large diameter ConFlat● and Wheeler seals is time-consuming. To minimize exposure of personnel to residual radiation in the AGS, extensive use of these seals is avoided. The third and forth varieties of seals listed above were dismissed for sealing force, reliability, cost or safety reasons. Varieties of the remaining candidates were tested.

METAL SEAL TESTS AND RESULTS

Tests consisted of squeezing the seal between a 300 series stainless steel mandrel, and sealing plates of either aluminum, stainless steel or enamel coated stainless steel. The mandrel was positioned in a precision hydraulic ram. The joints were leak checked by pumping between the two plates through a hole in the bottom of the mandrel. Any indication of a leak constituted a seal failure. Test seals had major diameters of ≈ 150 mm, with minor dimensions of ≈ 5.0 mm ϕ, excepting the "diamond" seal. A leak detector with an MDS of $\leq 2\times10^{-10}$ Torr-ℓ/sec He was used. A seal test was arbitrarily terminated if seal deflection exceeded ≈ 1.0 mm during loading, or seal loading exceeded ≈ 1850 N/cm.

Results of these tests are given in Tables I-III. All data are reported. The numbers listed vertically in each column of the tables represent the sequence of cycle-loading of one seal. Load-strain data for each type of seal were recorded and are given in Fig. 3. Each time a successful seal was achieved, seal deflection was measured. Then, the hydraulic ram was "released", and the seal height again measured. The plates were not parted between measurements. That is, the indexing between the sealing plates and seal was not changed between each measurement. Because of this, one may not interpret the existence of successive data in any one column as indicating that the seal is reusable. However, the absence of a consecutive number in any one column implies that the seal would not reseal on the next application of pressure, within the prescribed limits.

Results with the In coated C-rings was very poor. The plastic bags within which these had been stored were not adequately sealed. There was visible oxidation and "finger prints" on some the surfaces. Freshly coated In C-rings probably would have yielded better results (e.g., 350 - 700 N/cm sealing force). However, test results merely emphasize the serious shelf-life problem.

The diamond seal and the Pb coated, spring reinforced C-rings proved promising when sealing against metal surfaces. However, results were poor when sealing against enamel coated surfaces. It is possible that changing the included angle of the diamond sealing edge might have improved the performance of this seal. However, the temptation of "drifting" into a seal development program was avoided. Data on the Pb coated, spring reinforced C-rings should prove useful in calculating probable C-ring stress, as a function of required seal loading, for applications of unreinforced Pb coated C-rings.

Results with the Aℓ jacketed, spring reinforced C-rings were not at all promising. Disparities in our data from that reported in the literature[4] probably stem from differences in surface finishes and our use of a lower seal loading test limit.[6]

The Helicoflex Delta® seal ("Delta", hereafter) appeared the most promising. Only two Cu jacketed Delta seals were tested (i.e., Table I). The remaining had Aℓ jackets. Tests were subsequently conducted to determine if the Delta seals could be reused. A magnet chamber, with a 240 mm ϕ, stainless steel Marmon flange on one end and enamel coated flange on the other, was used for this purpose. A Delta seal and blank Aℓ flange was installed on one end of the chamber. A like seal and stainless steel flange with pumpout was installed on the opposite end. The test included: 1) installing the Delta seals and torquing the flange clamps to provide the equivalent sealing force of 960 N/cm; 2) leak checking the seals; 3) interchanging the seals; 4) torquing the clamps to the equivalent of 1400 N/cm sealing force; and 6) leak checking the seals. This test was repeated with four pairs of seals.

No seals leaked on the first installation, and only one seal leaked on the second use. This suggested that even with dramatic changes in sealing surface topography and materials, there is >85% probability of the seal being reusable a second time. These test conditions were realistic in the context of actual seal applications in the AGS.

ELASTOMER SEAL TESTS

Weight loss and hardness change tests were conducted on three varieties of Viton-A® and five compounds of Buna-N®. Outgassing rate measurements were conducted on Viton-A and Buna-N compounds and, for comparison purposes, on a sample of polyimide. Our interest in polyimide stems from its use as an electrical insulator in many of the beam components. Besides having good tolerance of radiation,[7] after baking, its outgassing is less than Viton.[8] Peacock provided an excellent review paper of salient publications dealing with vacuum applications of elastomers.[9]

In tests at SLAC elastomer gasket materials were exposed to high vacuum for up to three years.[10] With one exception, all compound lots were purchased from the Parker Seal Company. The exception was the material referred to as "Spare ?" material. This material came as a stock of spare O-ring seals provided by the vendor at the time of delivery of over 240 klystron waveguide pumpout valves.[11] The purpose of these tests was to determine if there were irreversible weight and hardness changes in certain elastomers as a consequence of vacuum exposure.

Ninety-five specimen O-ring segments, measuring 5.1 mm $\phi \times 25$ mm long, were individually suspended on wire frames mounted within Cu pinch-off tubes (see Fig. 4). These, in turn, were welded to a large manifold pumped by a 500 ℓ/sec sputter-ion pump (see Fig. 5). Specimens were pinched off this manifold over a three-year period. The weight and Shore-A hardness of each specimen was measured before and after extended immersion in high vacuum. Some Viton-A samples were baked in the pinch-off tubes for 24 hours at ≈ 200 C°. A comparable number of control samples (i.e., >100), not exposed to vacuum, were also tested for hardness and weight changes throughout the duration of the experiment.

O-RING HARDNESS TEST RESULTS

The average prevacuum hardness of each sample is given in Table IV. Though hardness increases due to vacuum exposure in all specimens were "statistically significant", functionally these changes were of little consequence. The harder the Viton O-ring, the less evident the change in hardness as a consequence of prolonged vacuum exposure. The maximum increase in hardness of unbaked Viton, after two years vacuum exposure, was two points. However, both the control and test samples of unbaked Viton became softer by about four Shore-A points as a consequence of prolonged air exposure. There were irreversible changes in hardness of the baked Viton O-rings which amounted to at most + 2.5 points.

No correlation existed between the initial Shore-A hardness of the Buna-N samples and increases in hardness as a consequence of vacuum exposure. Also, the magnitude of the hardness increase due to vacuum exposure was statistically significant in the case of only one sample type (i.e., +6 points; compound N-532-8), though this was completely reversible. There was no correlation between hardness changes and weight loss with the Buna-N materials.

WEIGHT LOSS RESULTS

The samples exposed to vacuum were weighed ≤4h, 120h and ≥6000h after removal from the pinched-off capsules. Average differences of pre-vacuum exposure weights and post-vacuum weights, as a function of air exposure, are given in Table IV for each of these materials. The control samples yielded surprising weight change results (i.e., the far right column). That is, there were real weight increases in the Viton-A samples and weight decreases in some of the Buna-N samples as a consequence of prolonged air exposure. The population of each control sample lot numbered from 10 to 32. Therefore, there is confidence in these data.

Weight increases in the Viton control samples obscures interpretation of weight recovery data of both baked and unbaked Viton samples. For example, even after ≥6000h air exposure, the baked samples had not yet returned to their original weights. This is not surprising, as one expects plasticizers and unreacted polymers to irreversibly evolve when baking Viton.[12] However, the irreversible weight loss of these samples probably exceeds that indicated because of the tendency of Viton-A, exclusively exposed to air, to increase in weight with time. Data for the baked samples established that irreversible weight loses of the baked Viton samples were inversely related to the initial Shore-A hardness of the test samples.

Using a known, fixed-conductance method, outgassing rates were measured for unbaked Viton-A and Buna-N samples (equivalent to compounds N-219-7 and 77-545, respectively). A polyimide valve gasket was also tested for comparison purposes. These data are given in Fig. 6. Integrating the outgassing data for the Viton and Buna-N for total weight loss after one year exposure to vacuum, weight loss of the Viton sample is ~ 2.2 mg/g and weight loss of the Buna-N is ~ 6.3 mg/g. The Viton outgassing data are in remarkable agreement with weight loss data of Table IV. The Buna-N outgassing data were low by x2.5 from the weight loss data of Table IV. This in part may stem from Blears effects[13] in the measurement apparatus and the known hydrocarbon outgassing of Buna-N.[14,15]

These polymer outgassing data were used to calculate additional beam component pumping which would be required throughout the AGS in order to meet the pressure objectives. These results are reported elsewhere.[16]

COURSES OF ACTION AND CONCLUSIONS

Because of the above test results, the Delta seal was adopted as the standard seal for future use in the AGS. During the 1988 summer shutdown, approximately 200, 216 mm ϕ, In coated C-rings and 100 comparably sized "diamond" seals were replaced with new clamps and Delta seals. Installation problems or inexplicable leaks occurred in ~ 3.0% of these seals at the time of installation in the AGS. However, after successful installation, there were no Delta seal failures in the following nine months of operation. There were two failures of remaining In coated C-rings in the AGS during this period.

Hardness changes in elastomers due to vacuum exposure is functionally of no consequence. However, O-ring outgassing dominates in an unbaked system (e.g., the AGS). The nature of accelerator technology requires that portions of the AGS be vented from time to time. Reversible weight changes in elastomers due to such venting would require that beam component pumping be augmented with high-speed pumps (e.g., cryopumps). One speculates that initial capital investment and maintenance costs associated with augmentation of pumping would far outweigh recurring costs associated with the use of large-diameter metal seals. Also, there is some evidence these seals are reusable. Because of these findings, future vacuum seal work is being directed toward evaluation of the Delta seal as a candidate for replacing <u>all</u> of the large-diameter elastomer seals in the AGS. Also work is being done with Delta seals with irregularly shaped major dimensions (e.g., elliptical shaped seals).

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Viton® is a registered trade name of E.I. du Pont Memours and Company.
Inconel® is a registered trade name of the International Nickel Company.
ConFlat® and Wheeler® are registered trade names of Varian Associates.
Helicoflex® Delta is a registered trade name of the L.C.L. Cefilac Entancheite and patented by CEA Pierrelate (French Atomic Energy Commission).

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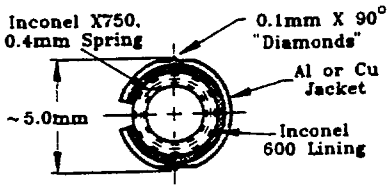

Fig. 1. Approximate Minor Dimensions of Helicoflex Delta® Seal used in the AGS.

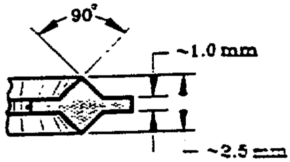

Fig. 2. Minor Dimensions of the 1100 Aluminum "Diamond Seals used in the Seal Test Program.

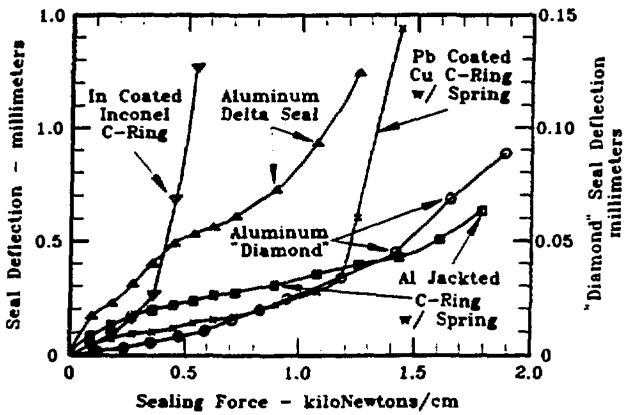

Fig. 3. Seal Deflection as a Function of Sealing Force. (Right Scale for "Diamonds"; Left Scale all Other Seals)

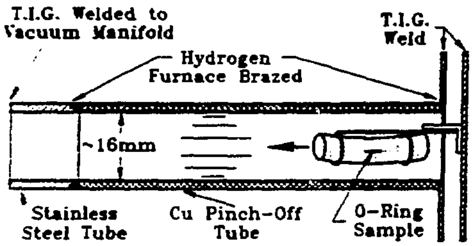

Fig. 4. Copper Pinch-off Assembly Used to House O-Ring Test Sample for Extended Vacuum Exposure.

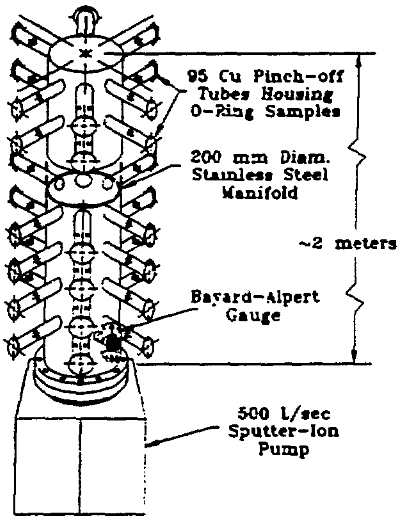

Fig. 5. Apparatus Used to Evaluate the Long Term Effects of Elastomer Exposure to Vacuum.

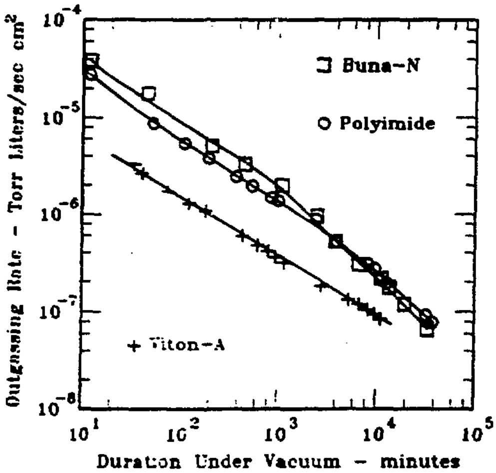

Fig. 6. Outgassing Rate of Three Popular Elastomers as a Function of Time in Vacuum.

Table 1. Sealing Force Required for a Variety of Metals Seals to Achieve Vacuum Tight Seals Against Stainless Steel Surfaces.

Seal Force Newtons per cm	In Coated C-Ring			Pb Coated C-ring w/ Spring			Helicoflex Delta® w/ Al Jacket			Aluminum Diamond Seal			C-Ring w/ Spring & Al Jacket			Delta® w/ Cu Jacket	
Sample No.->	1	2	3	1	2	3	1	2	3	1	2	3	1	2	3	1	2
177< 263																	
263< 350								1	1								
350< 438		Seal Loading Sequence					1	2				1					1
438< 525							2	3				2				1	2
525< 613							3	4		1		3				2	3
613< 700							4					4				3	4
700< 788	1,2			1,2				5				5					5
788< 876										2	1,2						
876< 963				3				6	2		3	6					6
963<1051																	
1051<1138				4	1				3								
1138<1226												7					
1226<1313				5	2			7	4								
1313<1401				6													
1401<1489					3			8				8					
1489<1576																	
1576<1664										3		9		1			
1664<1749																	
>1749														2	1		
No Seal		X	X			X							X				

Table II. Sealing Force Required for a Variety of Metal Seals to Achieve Vacuum Tight Seals Against Stainless Steel and Aluminum Surfaces.

Seal Force Newtons per cm	In Coated C-Ring			Pb Coated C-ring w/ Spring			Helicoflex Delta® w/ Al Jacket			Aluminum Diamond Seal			C-Ring w/ Spring & Al Jacket		
Sample No.->	1	2	3	1	2	3	1	2	3	1	2	3	1	2	3
177 < 263								1							
263 < 350							1	2	1						
350 < 438							2	3	2						
438 < 525									3		1				
525 < 613							3		4						
613 < 700															
700 < 786				1		1,2	4		5		2				
788 < 876	Seal Loading Sequence														
676 < 963				2	1,2	3		4		1	3				
963 < 1051															
1051 < 1138				3	3	4					4				
1138 < 1226										2		1			
1226 < 1313				4	4	5	5	5							
1313 < 1401															
1401 < 1489				5	5	6		6			5	2			
1489 < 1576															
1576 < 1664											6				
1664 < 1749															
> 1749										3					
No Seal	X	X	X										X	X	X

Table III. Sealing Force Required for a Variety of Metal Seals to Achieve Vacuum Tight Seals Against Stainless Steel and Enamel Coated Surfaces.

Seal Force Newtons per cm	In Coated C-Ring			Pb Coated C-ring w/ Spring			Helicoflex Delta® w/ Al Jacket			Aluminum Diamond Seal			C-Ring w/ Spring & Al Jacket		
Sample No.->	1	2	3	1	2	3	1	2	3	1	2	3	1	2	3
177 < 263															
263 < 350									1						
350 < 438							1	1	2						
438 < 525								2							
525 < 613									3						
613 < 700							2	3	4						
700 < 788															
788 < 876										1					
876 < 963				1			3		5						
963 <1051															
1051 <1138	Seal			2			4	4							
1138 <1226	Loading									2					
1226 <1313	Sequence						5	5	6						
1313 <1401															
1401 <1489				3			6				1				
1489 <1576															
1576 <1664														1	
1664 <1749															
>1749											2			2	
No Seal	X	X	X		X	X						X	X		X

Table IV. Summary of Weight Loss of Elastomers Stemming From Extended Exposure to High Vacuum

Baked & Unbaked O-Ring Compound		Initial Shore-A Hardness	Vacuum Weight Loss vs Hours Subsequent Air Exposure—Δmg/g			Control Sample Weight Gain Δmg/g
			4h	120h	>6000h	
Viton-A	SPARE ?	46	7.3	—	3.3	—
Viton-A	77-545	66	4.1	—	1.6	—
Viton-A	V-377-9	82	6.1	—	0.7	—
Viton-A	SPARE ?	46	2.2	1.1	—	—
Viton-A	77-545	66	1.9	1.0	—	1.3
Viton-A	V-377-9	82	1.8	1.0	—	2.0
Buna-N	N-299-5	45	48.1	—	43.1	−0.3
Buna-N	N-525-6	54	14.7	—	10.0	0.5
Buna-N	N-219-7	64	15.8	—	11.6	0.8
Buna-N	N-532-8	76	16.7	—	11.7	−0.9
Buna-N	N-552-9	88	14.6	—	10.0	−0.8