

BRIEF COMMENTS ON TEST RESULTS OF BOTH METAL AND ELASTOMER VACUUM SEALS

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**BRIEF COMMENTS ON TEST RESULTS OF BOTH
METAL AND ELASTOMER VACUUM SEALS**

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Introduction

A brief over-view of the subject of sealography was given at the recent EBIS Symposium held at Brookhaven. The results will be published elsewhere. However, results of metal vacuum seal tests, conducted at Brookhaven National Laboratory will be herein summarized. The note then summarizes results of work done by the first author, at the Stanford Linear Acceleration Center, on the extended outgassing properties of elastomers including Viton® and Buna N®.

Considerations in Selection of a Vacuum Seal

When selecting a particular seal joint design for use in ultra-high vacuum (UHV) application we must make a number of compromises. Considerations which must be entertained in selecting a joint design include the: 1) reliability; 2) material outgassing rates; 3) seal permeation rate; 4) bakeability; 5) mating surface materials; 6) ease of installation; 7) required sealing force; 8) flange sizes in typical use; 9) provision for cleaning of parts; 10) initial costs; 11) operating (i.e., replacement) costs; 12) seal availability for replacement; 13) degree of radiation "hardness"; 14) shelf life; 15) handling and storage fragility; and 16) safety. Most of these 16 variables are coupled. Each was briefly discussed in the talk, but such discussion is beyond the scope of this paper.

Metal Seals

All-metal seals are self-destructive in that they can only be reused, at most, two to three times. Common varieties of all-metal seals include the: 1) Con-Flat® (i.e., knife-edge); 2) Wheeler® (special wire seal);^[1] 3) Foil (Al, In, Cu); 4) Wire (Al, Au, Ag, Cu, In, etc.); 5) C-Rings, coated with soft metals; 6) C-Rings, either coated or uncoated, and reinforced w/spring materials; 7) C-Rings, with "diamond" edges, and spring reinforced (i.e., the Delta® Seal); and 8) "diamond" seals. Advantages and disadvantages of several seal configurations were discussed in the talk. Only the results of recent metal seal tests at Brookhaven will be reported. The seal configurations tested included:

- A. Inconel® C-Rings, coated with In,
- B. Spring reinforced, Inconel® C-Rings, jacketed with Al,
- C. Spring reinforced, Inconel® C-Rings, coated with Pb,
- D. Spring reinforced, Inconel® C-Rings, jacketed with an Al sheath on which "diamond" edges are machined (i.e., the Delta® seal),
- E. Conventional Al "diamond" seals.

Applications at the Alternating Gradient Synchrotron (AGS) require that leak tight, all-metal vacuum seals be made with flange materials including aluminum, stainless steel and enamel coated stainless steel. For the greater part, most of the metal seals in the AGS were Type-A of the above listing. However, problems were encountered from time to time with these Inconel® C-Rings spontaneously cracking. Past studies indicated that such problems resulted from H₂ embrittlement, due to some process in the fabrication of the seals. Also, In coated seals had very poor shelf life. Oxides of In would form on the sealing surfaces, even though the seals were stored in sealed bags. Rather than "study" these problems again, we faced up to the fact that the seals were marginal, at best, and launched a study program to find a suitable alternative.

Seals of the above listed configuration were tested. The test consisted of squeezing the seal between a stainless steel mandrel, and sealing plates made of the three different materials. 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. A leak detector with a sensitivity of $\sim 10^{-10}$ Torr- ℓ /sec He was used. Any indication of a leak constituted a seal failure.

Results of these tests are given in Tables 1-4. The numbers listed vertically in the columns in the first three tables represent the number of times the seal was cycled. Stress-strain measurements, and yield data were recorded. 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 and their respective indexing with the seal 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 additional data in any one column implies that the seal would not reseal on the next application of pressure. The results for the In coated C-Rings was very poor. This is because they were used "as is", on removal from the sealed bags in which they had been stored for many months. New, freshly coated C-Rings would have yielded far more reliable results. But, this is just a restatement of the initial shelf life problem.

Because of these results, the Delta[®] Seal was adopted as the standard seal for future use in the AGS. During the 1988 summer shutdown, approximately 300 Delta[®] Seals were used to replace the In coated, 21.6 cm diam. C-Rings used throughout the AGS. Problems of installation technique or inexplicable failures occurred in $\sim 3.0\%$ of these seals at the time they were installed. However, after successful installation, there have been no failures (i.e., leaks) in these seals.

Elastomer Seal Test Results

The most widely used elastomer seal material is Viton A®. However, where large quantities of seals (i.e., O-rings) are needed, cost considerations sometimes dictate use of another elastomer, such as Buna N®. If cost is no object, Polyimide has the best vacuum characteristics of all the elastomers. However, this material has some disadvantages. Peacock provides an excellent review paper on the properties and uses of elastomers.[2]

In tests at Stanford, elastomer gasket materials were exposed to high vacuum for up to three years in duration. The purpose of these tests was to determine if there were irreversible hardness changes in the materials as a consequence of this exposure. Approximately 100 specimen O-ring segments, measuring 0.51 cm ϕ x 2.5 cm long, were individually suspended on wire frames mounted within Cu pinch-off tubes. These, in turn, were welded to a large manifold pumped by a 500 L/sec sputter-ion pump. Specimens were pinched off this manifold over a three year period. The weight and hardness of each specimen was measured before and after extended immersion in high vacuum. Some samples were baked at moderate temperatures (i.e., ≈ 200 C°). Numerous control samples, not exposed to vacuum, were also tested for hardness and weight changes throughout the duration of the experiment. Results of the vacuum tests were as follows:

1) All Buna N® O-rings showed significant irreversible weight loss as a consequence of exposure to vacuum. This lost material is probably not exclusively water vapor, but rather plasticizers and polymers which would contaminate the vacuum system (e.g., plug sieve materials; poison cathodes). Irreversible weight losses in unbaked Buna-N® O-rings amounted to approximately 10 to 50 Atm./cm³ of O-ring.

2) Unbaked Viton-A® O-rings showed no significant irreversible weight loss or hardness change. Weight losses in Viton® ranged from 3 to 7 Atm./cm³ of O-ring. Hardness changes in unbaked Viton-A® were completely reversible. With baked Viton-A® O-rings, the higher

the initial Shore-A durometer hardness reading, the less the irreversible weight loss due to baking. This suggests the presence of less plasticizers and unreacted polymers in the initially harder compounds.

References

- 1) Roth, A., J. Vac. Sci. Technol. **9**, No.1, 14 (1972).
- 2) Peacock, R., N., J. Vac. Sci. Technol. **17**, No. 1, 330 (1980).
- 3) Wahl, H., et. al., CERN Tech. Note SPS/81-8, December, 1981.
- 4) The authors are indebted to Alain Poncet of CERN PS. Dr. Poncet provided the authors with the data which led to this study. He has extensive experience in UHV sealography, including the successful use of "diamond" seals throughout the CERN Proton Synchrotron.

Table 1. Sealing Force For Metal Seals of Different Configuration
When Sealing Against Two Stainless Steel Surfaces

Sealing Force in lbs./inch	In Coated C-Ring			In Coated C-Ring			In Coated C-Ring			Pb Coated w/Spring			Pb Coated w/Spring			Pb Coated w/Spring			Delta Seal			Delta Seal			Delta Seal			Al Diamond			Al Diamond			Al Diamond			Al C-Ring w/Spring			Al C-Ring w/Spring			Al C-Ring w/Spring		
Sample No.->	1	2	3	1	2	3	1	2	3	1	2	3	1	2	3	1	2	3	1	2	3	1	2	3	1	2	3	1	2	3	1	2	3	1	2	3	1	2	3						
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Table 2. Sealing Force For Metal Seals of Different Configuration
When Sealing Against An Aluminum and Stainless Steel Surface

Sealing Force in Lbs./Inch	In Coated C-Ring			In Coated C-Ring			In Coated C-Ring			Pb Coated w/Spring "C"			Pb Coated w/Spring "C"			Pb Coated w/Spring "C"			Delta Seal Ⓢ			Delta Seal Ⓢ			Delta Seal Ⓢ			Al Diamond			Al Diamond			Al Diamond			Al C-Ring w/Spring			Al C-Ring w/Spring			Al C-Ring w/Spring		
Sample No.->	1	2	3	1	2	3	1	2	3	1	2	3	1	2	3	1	2	3	1	2	3	1	2	3	1	2	3	1	2	3	1	2	3	1	2	3	1	2	3						
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Wouldn't Seal	X	X	X																																										

Table 3. Sealing Force For Metal Seals of Different Configuration
When Sealing Against A Stainless Steel & Enamel Coated Surface

Sealing Force In Lbs./Inch	In Coated C-Ring			In Coated C-Ring			In Coated C-Ring			Pb Coated w/Spring "C"			Pb Coated w/Spring "C"			Pb Coated w/Spring "C"			Delta Seal			Delta Seal			Delta Seal			Al Diamond			Al Diamond			Al Diamond			Al C-Ring w/Spring			Al C-Ring w/Spring			Al C-Ring w/Spring		
Sample No.--	1	2	3	1	2	3	1	2	3	1	2	3	1	2	3	1	2	3	1	2	3	1	2	3	1	2	3	1	2	3	1	2	3	1	2	3	1	2	3						
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Table 4. Qualitative and Quantitative Considerations
In The Selection of a Vacuum Seal Configuration.

Vacuum Seal Configuration	Sealing Force lbs./inch	Bakeable (yes, no)	Bakeout Temp. C ^o	Reuseable (yes, no)	Maximum Flange Diam. Inches	Cost (1=High)	Reliability (1=High)	Shelf Life	Fragility (1=High)
ConFlat [®] Gasket	1350 ¹	YES	625	NO	10	3	1	GOOD	5
Wheeler [®] Gasket	>1500 ²	YES	450	NO	30	1	2	GOOD	4
Indium Coated Inconel C-Ring	<400	NO	—	?	>30	2	7	POOR	4
Al Jacket C-Ring, Spring Reinforced	1000	NO	—	NO	>30	4	7	GOOD	4
Pb Coated C-Ring Spring Reinforced	400-600	?	?	?	>30	2	3	POOR	3
Al Delta [®] C-Ring Spring Reinforced	100-250	YES ³	200 ³	YES	40	4	2	GOOD	1
Diamond, Al	200-800	NO	—	NO	40	5 ⁴	3	GOOD	2