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REPLACEABLE Be AND Al WINDOWS FOR X-RAY BEAM LINES

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ABSTRACT

Construction details and results of on-going performance tests are presented for two ultra-high vacuum compatible, free-convection cooled window assemblies in which the window disk is easily replaceable. In the Be window assembly, an 0.25 mm thick Be disk, 38 mm in diameter is sealed into a stainless steel adapter flange using 0.5 mm diameter pure Al wire. The assembly is leak tight to better than 1 x 10^{-10} atm cm^3/sec before and after multiple bakeouts to 250° C. However, an unexplained temperature-dependent He leak, with a maximum magnitude in the low 10^{-10} atm cm^3/sec range at 250° C, appears during bakeout. Possible causes for this leak are discussed. In the Al window assembly, a 25 μm thick Al disc, 60 mm in diameter acts as the sealing medium as well as the window material over a 10 mm by 50 mm opening. This assembly remains leak tight to better than 1 x 10^{-10} atm cm^3/sec throughout multiple bakeouts to 150° C and has been successfully tested in white and monochromatic beam lines at the NSLS x-ray ring.

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Introduction

X-ray beam lines at synchrotron radiation facilities often use windows to separate various portions of the beam line; e.g. the main beam line from the storage ring vacuum, a mirror chamber operated at ultrahigh vacuum (UHV) from a monochromator operated at rough vacuum, etc. In some instances, the window requires special cooling facilities, but in a large number of situations no specific cooling, or at most free-convection cooling, provided by approximately one atmospheric pressure of air, helium, or some other gases on one side of the window, is sufficient. For these later situations, a window design which is simple, reliable, and inexpensive to fabricate would be quite useful. Full UHV compatibility and provisions for quick and easy window replacement are also desirable features. Here we present construction details and results of performance tests for two window systems which in many ways meet these requirements. One is a Be window which uses an Al wire seal, while the second is an Al window in which the window itself acts as the sealing medium.

A body of related work is found in the literature. A number of authors report Al-foil-sealed systems [1-3]. Of particular note is a design suitable for flanges which range in size from 40 mm to over 500 mm in diameter reported by Batzer and Ryan in 1963 [3]. Using stainless steel flanges, the seal can withstand thermal cycling from -196°C to 450°C, due to a design which stores significant strain energy in the tightened flanges and uses this energy to maintain the sealing forces.

Significant research on Al-wire gasket seals was conducted by groups at Edwards High Vacuum Ltd. in the late '50's and early 60's [4, 5]. They found that an Al gasket clamped between flat stainless steel flanges could produce a good UHV seal for use up to at least 400° C. However, if baked above 250° C, the Al began to adhere to the flanges and considerable force could be required to separate them. Above 370° C, the Al could not be peeled from the flanges, and if baked for long periods of time at 400° C to 450° C a brittle intermetallic compound formed.

A number of Be window assemblies have been reported in which the window element is replaceable [6-11]. Parsons describes a seal [8] which makes use of a standard pair of 34 mm diameter CONFLAT flanges and a two-piece Al assembly which substitutes for the standard Cu gasket. The Be window is sandwiched between the two halves of the Al gasket, which cold-weld together when the assembly is tightened in the flanges. This system is bakeable to 275° C. Hartman [11] recently reported experience at CHESS with several different Be window sealing techniques, including a rather complicated design in which a Be foil is sealed against a base flange using a triangular cross section Pb gasket pressed against both the window and base flange from the atmospheric pressure side of the assembly.

In general, to form a reliable metal-to-metal seal, two conditions are very important:

1. The surface finish of the sealing areas must be 0.8 μm or better.
2. Large sealing forces must be available and these forces should not release during bakeout.

In addition, because the thermal expansion coefficient of the sealing medium is rarely matched to the rest of the system, the seal should be as thin as possible, to minimize the elastic deformation that must be provided during thermal cycling.

In light of these requirements, we have adopted the basic flange geometry illustrated in Fig. 1. It consists of a male and a female flange and some metal sealing medium, which can be located in either of two places. When the sealing medium is located on the outer diameter of the male flange, we call the seal an "outside seal" (see Fig. 1). When the sealing medium is located inside the female flange, we call the seal an "inside seal". (The outside seal and inside seal are sometimes denoted in the literature as "corner seal" and "step seal" respectively.) When a metal wire is used for the sealing medium, the actual seal is made in the corner of the mating flanges. The metal is squeezed between the flat surfaces of the mating flanges and extruded into the clearance between the two diameters. In either outside or inside seal, a precise fit should be maintained between the male and female flanges, with the clearance kept to a minimum. The interpenetration of the two flanges is designed so that the outer rings with bolt holes will never come surface-to-surface [see Fig. 1]. This permits unlimited compression of the sealing medium and allows storage of strain energy in the system through elastic deformation of the flange outer ring with the flange bolts. In this way, force can be maintained on the sealing medium during thermal cycling. While the outside seal has been known in vacuum applications since the 1930's [12], the inside seal is the appropriate variant for window systems, because an additional element can be inserted between the sealing medium and the male flange (see Fig. 1).

The remainder of the paper is organized as follows: Construction details for the two window systems are first presented. Next, a description of performance tests and their results is given, followed by a short discussion, summary and conclusion.

Construction Details

The Al window assembly is illustrated in Figs. 2 and 3 and consists of three major pieces, an adapter flange which permits the window to be mounted on a standard 114 mm dia. CONFLAT flange, the Al window disk, and a retainer ring. The adapter flange and retainer ring are made of 304 stainless steel. The mating surfaces are machined to a finish of 0.8 μm, with a radial clearance of 20 μm to 60 μm. The edges of the oval opening in the adapter flange are carefully rounded on the side where the window mounts. Usually, the adapter flange side of the window is evacuated while the retainer ring side is at atmospheric pressure, therefore, any sharp edges remaining on the oval opening could puncture the window during initial evacuation.

The Al window itself is a simple foil disk, 60 mm in diameter, cut out by hand. For the version illustrated in Figs. 2 and 3, we usually use a 25.4 μm thick foil, type 1100 - H18 [13].

Assembly is as follows: The adapter flange is cleaned for UHV use. The surface of the retainer ring which presses against the Al window and the window disk itself are wiped with methanol. The window disk is dropped into the adapter flange counter bore and secured with the retainer ring. Eighteen high strength 8-32 stainless steel socket head bolts are used to secure the retainer ring (the same type of bolts usually used with 34 mm diameter CONFLAT flanges). A high temperature lubricant [14] is applied to the bolts prior to assembly, which prevents seizure after bakeout and also permits higher sealing forces to be applied for a given bolt torque. The bolts are hand tightened sequentially around and around the retainer ring, with each bolt rotated only a small amount on each pass, until all bolts are tight.

The Be window assembly is illustrated in Figs. 4 and 5. It is similar to the Al window assembly, and most of the details mentioned above apply here as well. The window disks used are high purity (99.8%) Be [15] 38 mm in diameter and 0.25 mm thick. The major difference is the sealing media. A high purity (99.999%) annealed Al wire [16] 0.5 mm in diameter is formed into a gasket ring and inserted between the window disk and the adapter flange (see Fig. 5, DETAIL "A"). The gasket ring is formed by simply wrapping a length of Al wire around an appropriate cylindrical form and twisting the ends together. The twist is rotated from the outside of the loop into the inside, and the excess cut off (see Fig. 4).

Assembly is also similar to the Al window case. The Al wire is wiped with methanol before being formed into the gasket. The gasket is dropped into the counter-bore of the adapter flange and the cylindrical form used to make the gasket is now used to flatten the twisted ends to the height of the rest of the gasket. (Otherwise, the twist can apply enough excess force to the window to crack it when the retainer ring is bolted down.) The window is now dropped in place and secured with the retainer ring using twelve normal (not high strength) 6-32 stainless steel socket head bolts. The bolts are tightened sequentially around and around, only a little at a time, until all bolts are tight. For the assembly illustrated, the 0.5 mm Al wire was typically flattened to 0.2 mm.

Performance Tests and Results

One of the most demanding requirements on a window assembly is that it remain leak tight throughout multiple bakeout cycles, without re-tightening bolts. Both assemblies were so tested. Because the Al window assembly uses a thin window (25.4 μm), it was also tested to withstand a differential pressure of 1 atm, applied rapidly, after multiple bakeouts.

The bakeout test of the Al window was set up as follows: The window assembly was mounted to a test fixture on a He mass spectrometer leak detector (Alcatel Model ASM-51). A fiberglass heating tape was secured around the circumference of the adapter flange and a chromel-alumel thermocouple monitored the temperature of the flange, a few mm beyond the outer lip of the retainer ring. A He flow of about 20 cm^3/min was supplied to the window region and confined using layers of Al foil.

The leak detector was calibrated just before the window and its fixture were mounted, and again, at the end of the run. In a run, the window was first leak-checked by hand, then enclosed in Al foil (together with the He inlet) and heated at a rate of 4°C per minute to 150° C. The temperature was held constant for one hour, then the assembly was allowed to cool to room temperature, which required several hours. During the heating and cooling cycle, the leak rate and temperature were continuously monitored on a strip chart recorder. Once the window had cooled, the Al foil covering was removed and the window leak checked again by hand.

Four runs were made on an Al window assembly and no leaks were ever detected, to a sensitivity of better than 1×10^{-10} atm cm^3/sec. It should be noted that a maximum temperature of 150°C was chosen for the test because the tensile strength loss in 1100-H18 Al becomes very large above about 150°C [17]. The holding time of one hour at the maximum temperature provides enough time for almost all of the strength loss to occur [18].

A rupture test was performed on the same Al window assembly after completion of the four bakeout runs above. The window was vented to atmospheric pressure and then evacuated on the leak detector as quickly as possible. Greater than 90% of one atm was applied in several seconds. This test was performed five times, followed by a leak check. The window remained leak tight.

Two Al window assemblies with 25 μm foils have been installed in NSLS X-Ray beam lines, both just downstream of 0.25 mm thick Be windows. One receives only monochromatic radiation after a double-crystal monochromator while the other receives 2 mrad of white radiation (filtered by the Be window just upstream). The window in white light has only been in place for about one month of running time while the second for three months. No problems, vacuum or other, have occurred.

The Be window assembly was bakeout tested using a procedure very similar to that outlined above. The window was heated to 250°C at a rate of 4°C per minute, and held for one hour at this temperature before it was cooled to room temperature. A maximum temperature of 250° C was chosen because this temperature is employed for bakeout of stainless steel components in the NSLS storage rings [19] and after baking to 250°C, the Al wire should still easily peel off the adapter flange [4].

One Be window assembly was tested for 5 heating-cooling cycles and a second assembly was tested for two cycles. The results are somewhat complicated. At room temperature, no leak was ever detected, however, as the temperature was increased toward 250°C, the detector registered an increase in the leak rate. At 250° C, a steady leak rate of between 1.3 and 3.3 x 10^{-10} atm cm^3/sec was observed; the value varied from run to run, but generally increased on subsequent runs of the same assembly. We know that the readings were real, because if the He flow was stopped, the signal decreased to zero, and if the flow was increased above its nominal value, the signal increased. As the assembly was cooled, the leak signal decreased back to zero.

To isolate the source of the leak, a special assembly was made up with a 4.8 mm thick 304 stainless steel disk in place of the usual Be window. At 250°C, the maximum leak rate observed was 0.5×10^{-10} atm cm^3/sec.

A special bakeout run was made with a Be window assembly which had already been put through five bakeout cycles. Once the window assembly was heated to 250°C, the He flow was stopped and the trapped volume over the window purged with an N_2 gas flow. The He flow was then re-started, with a flow rate of about 5 times the nominal 20 cm^3/min, and the response of the leak detector was recorded as the leak rate increased from zero to its steady state. Roughly 180 seconds were required to reach 90% of the steady state value. This is longer than the time required to completely displace the N_2 from the trapped volume (about 60 sec) or the basic response time of the detector and chart recorder when operated in the 10^{-10} atm cm^3/sec range (about 6 sec for 10% to 90% response; measured using a Varian 951-5106 leak valve and He source in a separate test). A plot of the leak rate dependence vs temperature indicated a rapid response, not the $T^{1/2}$ dependence expected for a "real" leak, i.e. a small hole or tube through which the gas flows.

Discussion

In this section, we will consider some possible causes for the observed temperature-dependent He leak in the Be window assemblies. One set of possibilities supposes that the leak is directly related to the Be window disk, while a second set supposes that the disk is not directly related.

Simple permeation of Be by He is not one possibility. Numerically, the permeation rate is the product of the solid solubility and the diffusion coefficient [20], but for inert gases in metals the solid solubility is "immeasurably small" [20,21]. Nevertheless, foreign particle inclusions [22], grain boundary corrosion [23], or grain boundary cracking due to deformation near the Al gasket, might cause the kind of leak dependence observed. At room temperature, and with Be windows considerably thinner (10 μm vs 0.25 mm in our case), He leakage through window assemblies in Si(Li) and Ge detectors has been observed [24], while no leak is apparent with exposure to air. Informal discussions with one detector manufacturer, however, brought out that two kinds of Be window assemblies are commonly used, one in which the window disk is cemented in place using an epoxy, and a second in which a proprietary soft metal gasket is used in a replaceable assembly. The He leakage is much lower in the soft-metal-sealed assemblies, which suggests that the epoxy may provide a high permeability path for some of the observed He leakage cases.

If the Be disk is not directly the cause of the observed He leak in our assemblies, some other aspect of the unit must be to blame. One major possibility is that through differential thermal expansion of the components, an adequate sealing force is not maintained during thermal cycling. This may be especially important near the twisted joint in the Al gasket, where either the additional material present or construction of the joint itself may lead to a leak at elevated temperature. Holden and co-workers reported problems with twisted-wire gaskets in their early investigations of Al wire gaskets seals [25] and discarded that design for a butt-welded joint, however, the symptoms of their problems do not fit the present case completely.

Suffice it to say that additional work is necessary if the origin of this temperature-dependent leak rate is to be satisfactorily explained.

Summary and Conclusion

We have presented construction details and results of performance tests for two free-convection cooled, UHV-compatible window assemblies for use in x-ray beam lines. The Al window assembly, with its 25 μm thick window disk, is leak tight to better than 1 x 10^{-10} atm cm^3/sec throughout multiple bakeouts to 150° C and after a subsequent rupture test. No problems have occurred during actual use in an NSLS x-ray beam line, either on a monochromatic branch line or a white beam branch line. The Be window assembly, with its pure Al wire gasket seal, is leak tight to better than 1 x 10^{-10} atm cm^3/sec _after_ multiple bakeouts to 250° C, however _during_ bakeout, an unexplained, temperature-dependent leak appears with a magnitude in the low 10^{-10} atm cm^3/sec range at 250° C. Additional work is required in this area.

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[14] Fel-Pro, Inc., Skokie, Illinois 60076; Type C-100.

[15] Electrofusion Corporation, 125 Constitution Drive, Menlo Park, California 94025; Type IF-1 Beryllium ingot foil.

[16] Johnson Matthey, Inc., Eagles Landing, P.O. Box 1087, Seabrook, New Hampshire 03874. Product number 10748. Such high purity wire is probably not crucial to this application, but it is readily available, and inexpensive.

[17] Metals Handbook, Ninth Edition, (American Society for Metals, Metals Park, Ohio 1979), Vol. 2, p.67.

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[21] The statement that no inert gas has appreciable solubility in any metal seems very broad and inclusive, yet we have been unable to find any original reference to substantiate this statement.

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Figure Captions

Fig. 1. Basic flange geometry considered in window system design. Male and female flanges are sealed using a metal wire gasket located in either of two places, indicated as "INSIDE SEAL" and "OUTSIDE SEAL". Appropriate choice of other dimensions permits unlimited compression of the sealing medium and storage of strain energy in the flanges to maintain the sealing force throughout thermal cycling.

Fig. 2. Photograph of an unassembled prototype Al window unit. The three major parts illustrated are, from left to right: the adapter flange, the 25 μm thick Al window disk, and the retainer ring.

Fig. 3. Scale assembly drawing of the Al window system. Eighteen high-strength stainless steel bolts are used to secure the retainer ring. Unlike the prototype illustrated in Fig. 2, the retainer ring includes a KF-50 end piece.

Fig. 4. Photograph of an assembled and an unassembled Be window unit. In the unassembled unit, the major parts illustrated are, from left to right: the adapter flange, the pure Al wire gasket, the 0.25 mm thick Be window disk, and the retainer ring. The Al gasket is formed by twisting a wire around a form, rotating the twist into the center of the ring and then cutting it short. This twist can be seen on the gasket near the assembled unit.

Fig. 5. Scale assembly drawing of the Be window system. The detail illustrates how the Al wire is trapped between the female flange and the Be window.

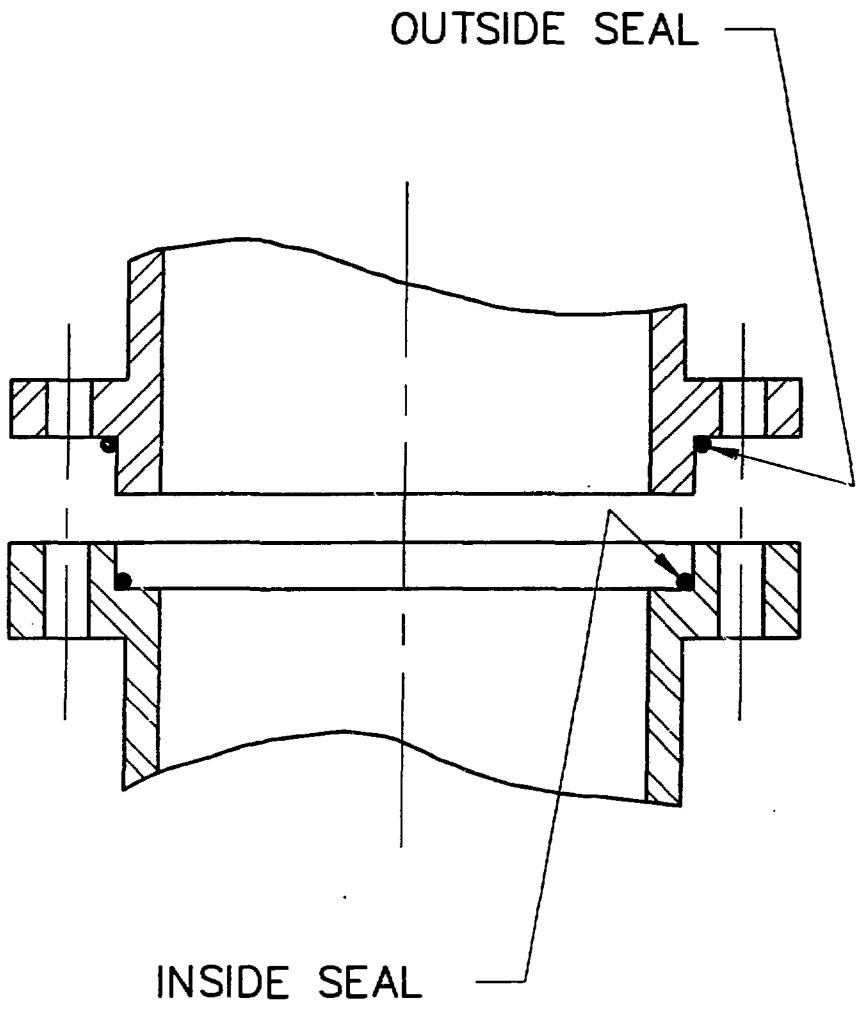
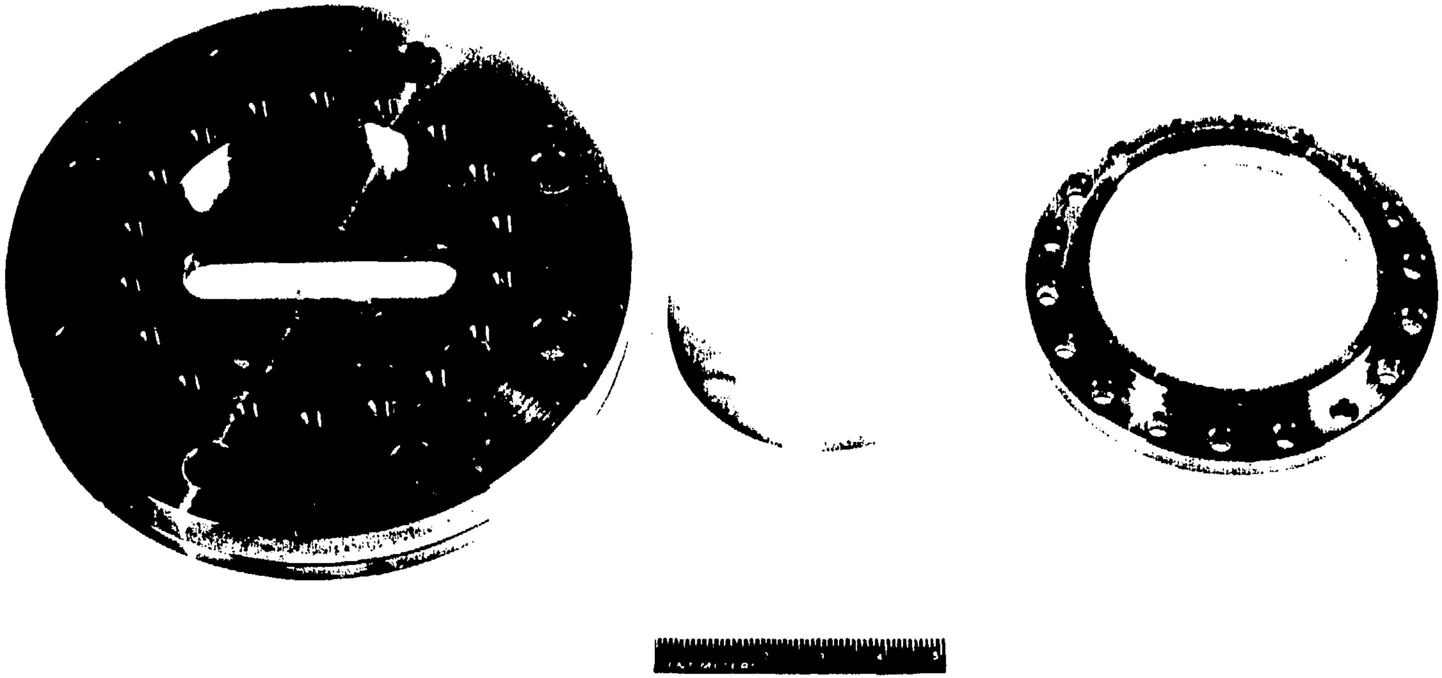


FIG. 1



F16. 2

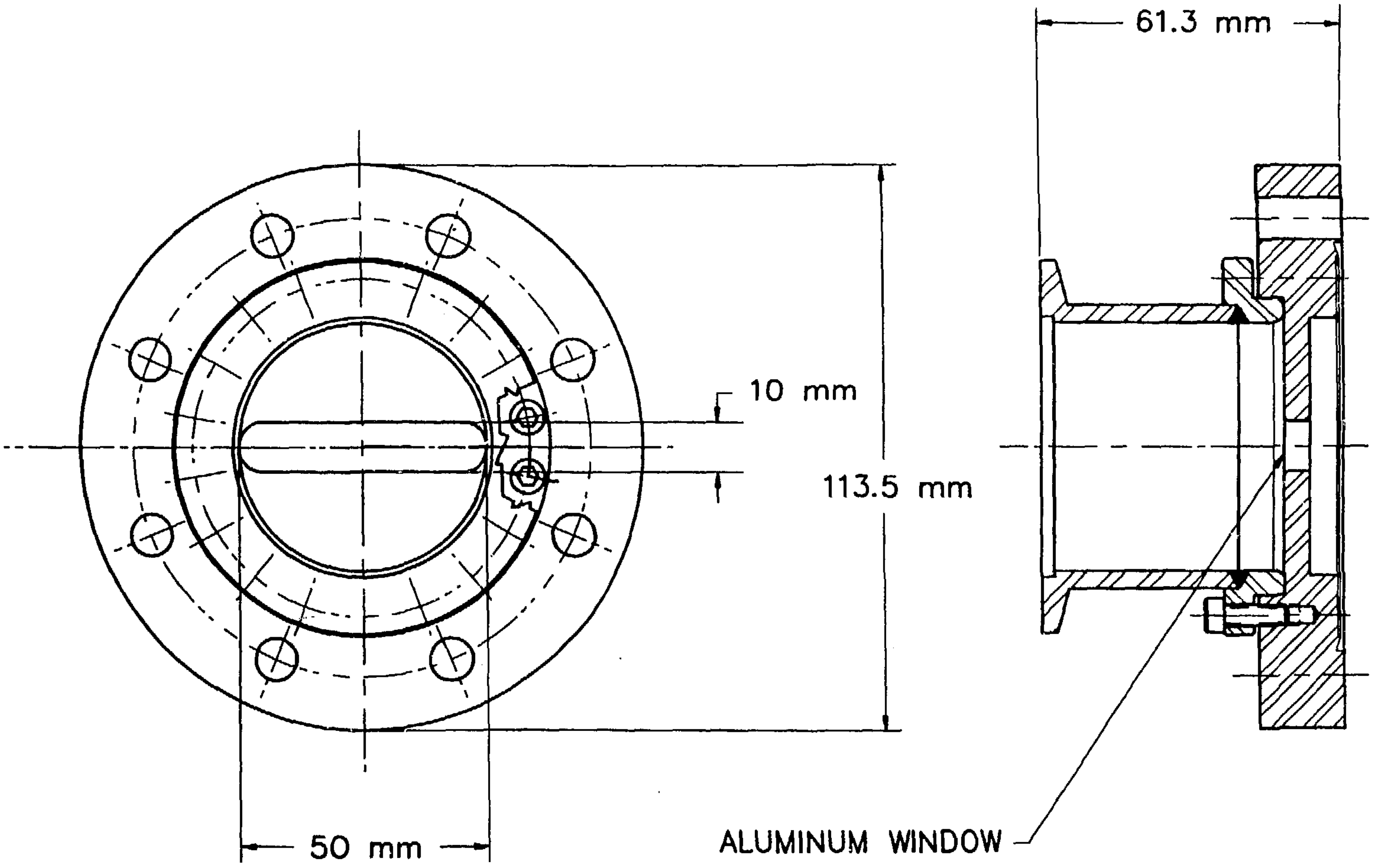
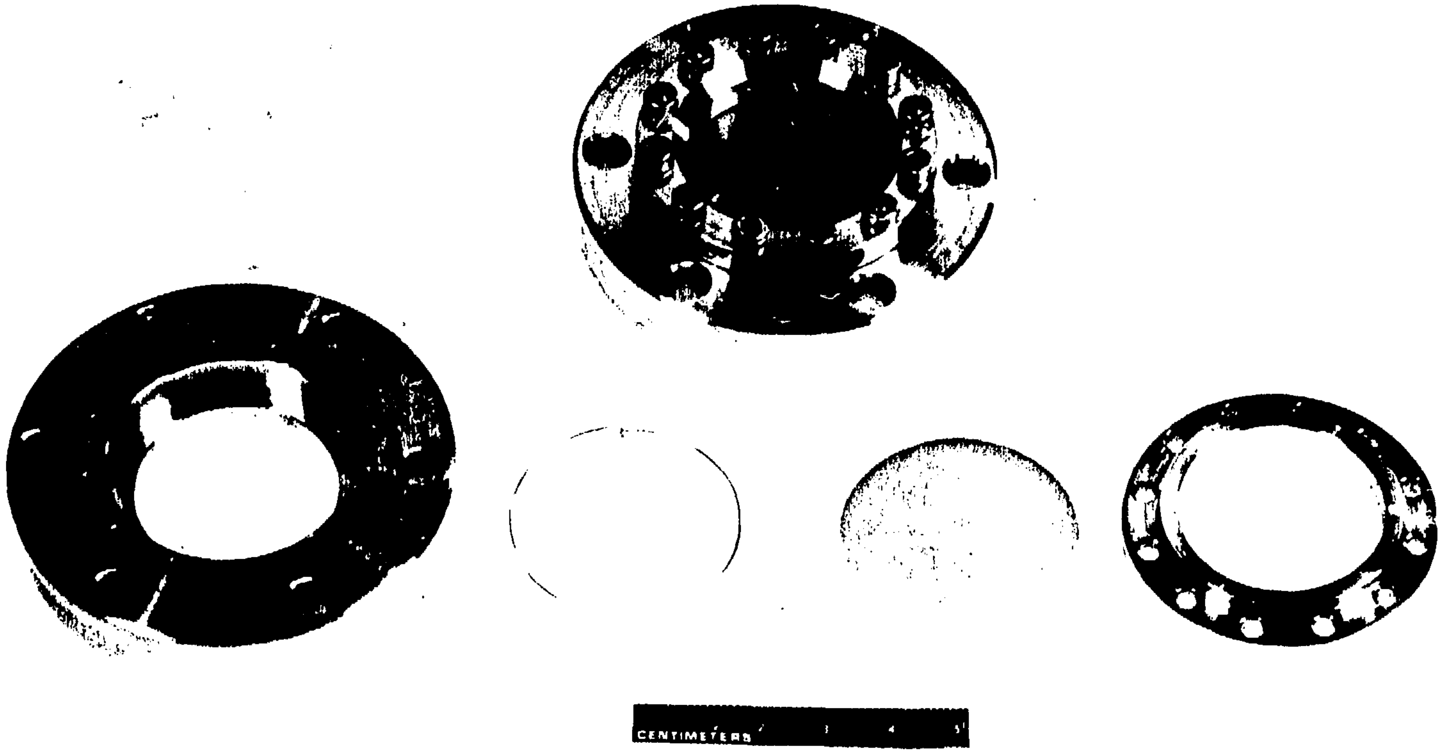
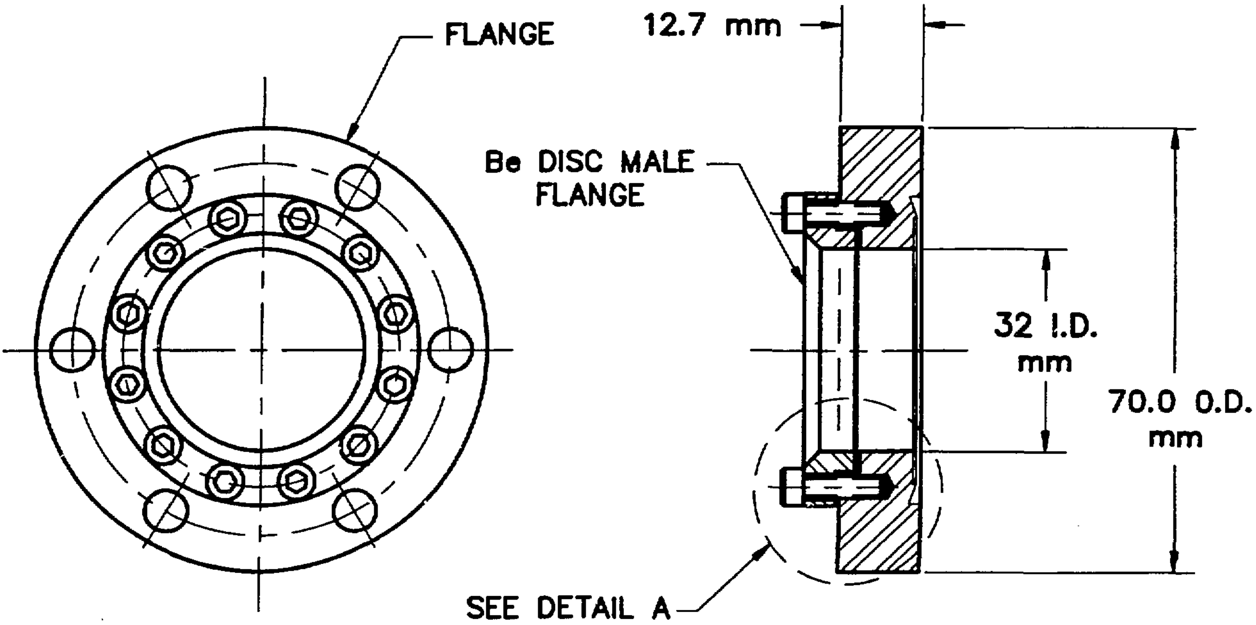
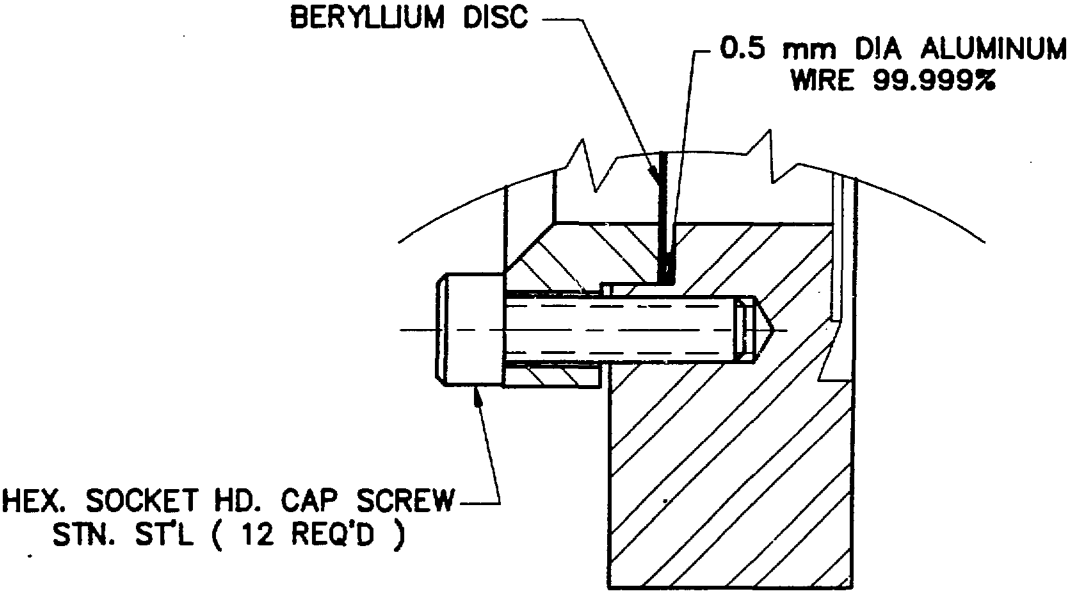


FIG. 3



F/G. 4





DETAIL "A"

FIG. 5