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A SOFT X-RAY PHOTOIONIZATION DETECTOR

By

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SUMMARY

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A simple ceramic radiation detector used in solar and astrophysical observations from rockets and satellites is the direct-current gas-filled ionization chamber. The detector consists of a ceramic shell incorporating a beryllium window and containing Xenon fill gas. The sensitivity is determined by the ion-pair yield of Xenon, along with Xenon's spectral absorbtion characteristics, and the transmission properties of beryllium. The fabrication technique for these detectors is described, along with some observed operating characteristics.

I. INTRODUCTION

One of the simplest radiation detectors used in solar and astrophysical observations from satellites is the direct-current gas-filled ionization chamber. The observer, by selecting combinations of window materials and fill gases may have at his disposal a spectrally selective detector with a fairly narrow pass-band. Such a detector requires only a voltage source and a high impedance electrometer to yield a direct measurement of those radiation fluxes which fall within its particular pass-band.

In the past, the Astrophysics Branch of the Goddard Space Flight Center has developed a reliable technique applicable to the construction, evacuation, and gas filling of these detectors. Previously such detectors have employed synthetic cleaved LiF, CaF or BaF_2 as window materials and have been filled with gases such as Nitric oxide, acetone vapor, and carbon disfulfide. Such combinations have convenient spectral pass-bands in the 1150 to 1350 Å region[1-2].

As interest in solar X-rays increased it became desirable to extend these techniques to the development of detectors employing thin aluminum or beryllium foil windows filled with one of the noble gases. We describe the fabrication techniques for these chambers, along with some observed characteristics.

II. ION CHAMBER SHELL

The ion chamber shell is identical to that described by Stober, Scolnick, and Hennes[1]. Briefly, the shell is fused of high density alumina whose basic profile is shown in Figure 1. The interior surface is sequentially coated with molybdenum manganese (metalizing), a flash plating of nickel, a plating of copper, and a sintered gold plate. Brazed into the unplated rear face of the shell are a Kovar central pin electrode, a guard ring and connector, and a copper gas-fill tube which also serves as the electrical connection to the plated-shell electrode.

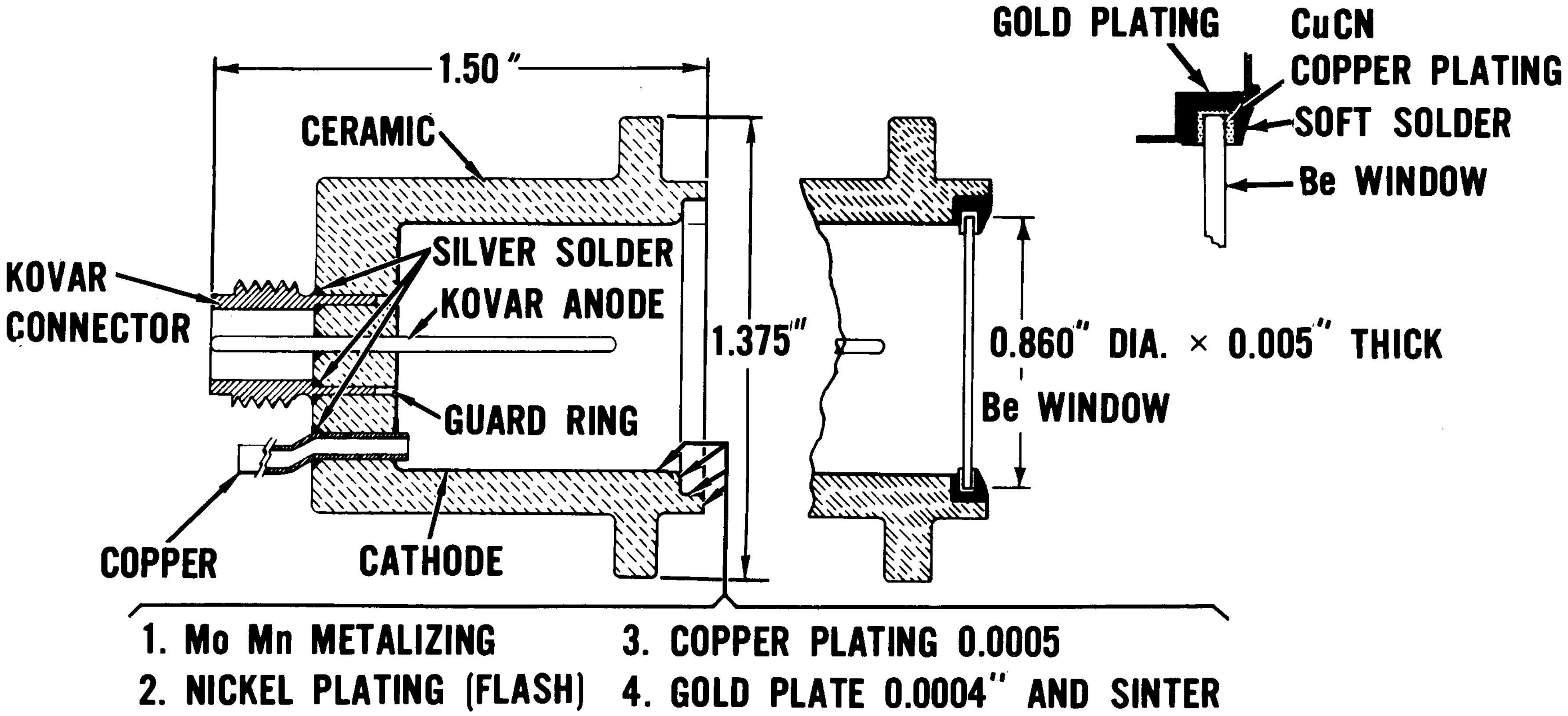
III. WINDOW MATERIAL

The window material used in the detector is QMV* hot-rolled beryllium foil made by the Brush Beryllium Company. The window is .860" in diameter, chosen to fit the shell seat. The window thickness is .005" as shown in Figure 1.

Although it would certainly be desirable at times for the observer to employ thinner windows, this does not seem to be currently practical for the following reasons: Thinner foil cannot support a 14.7 psi pressure differential over a .860" diameter disk without additional mechanical support on both sides. This would be necessary during the evacuation and gas-fill procedure as well as in flight applications. Such supports are very difficult to assemble and reduce the effective window area

*Registered Trade Mark

Figure 1. NASA ION CHAMBER



about 50%. Secondly, the quality of beryllium foil available is poor. Microphotographs of a cross-section of foil exhibit deep voids which often extend through the foil,creating pin holes, which cause gas leakage[3]. These voids also substantially reduce the mechanical strength of the material, all of which makes thinner foil undesirable for permanently sealed detectors unless high selectivity of window discs is possible. Whenever possible, thin foil disks should be stored in a dessicator box as protection against minute traces of chlorine in the air. Chlorine will etch a beryllium surface and a thin window could be ruined by this process.

IV. CHAMBER ASSEMBLY

The beryllium windows are bonded to the ceramic shell with 60-40 lead-tin solder,(Figure 2.) This is accomplished by preparing a special soldering surface around the periphery of the window disk, as solder will not ordinarily adhere to a beryllium surface. This surface is provided by masking the central area of the window and electroplating a copper surface around the exposed edge. The copper surface is then used for the solder seal to the gold plated seat of the ceramic shell.

This technique is simple and appears to be reliable. Be-windowed ion chambers were assembled by this method and leak tested successfully with a He mass spectrometer exhibiting a

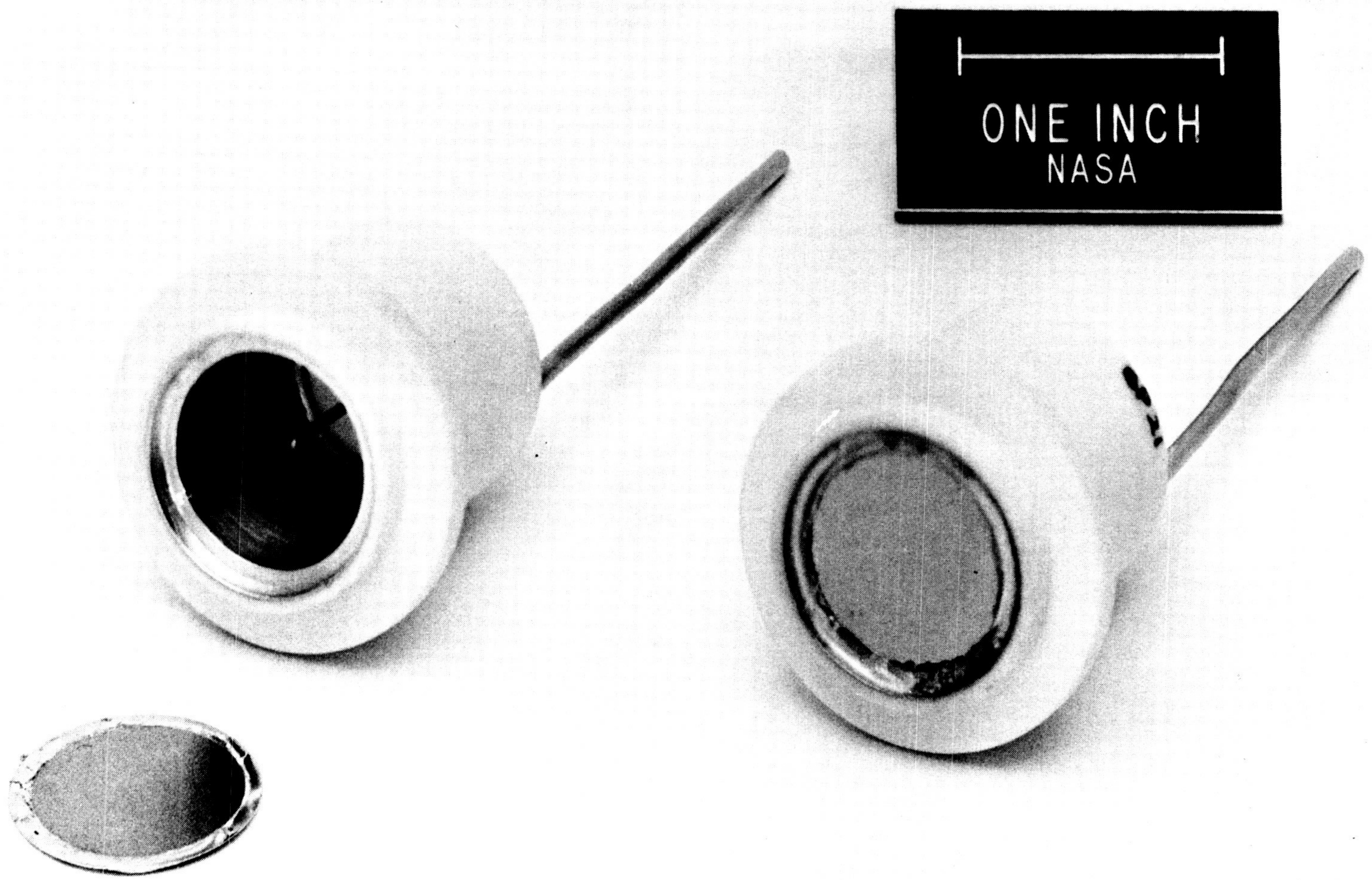


Figure 2. ION DETECTOR ASSEMBLY

maximum sensitivity of 2×10^{-10} cc/sec at 1 atm. The chambers were then evacuated to 2×10^{-7} mm Hg pressure, and back-filled with the desired gas.

The preparation and plating procedure of the foil disks is as follows:

1. Ultrasonically clean the Be disks in an acetone bath for thirty (30) minutes. Remove the disks and allow them to dry.
2. Apply the masking clamp. This clamp, similar to a "C" clamp, has a circular teflon pad for each jaw, and masks off the effective window area from the plating solution. The pad leaves an exposed edge of approximately 3/32" in width.
3. Dip the window into an all-purpose alkali plating cleaner, agitating the window while in solution.
4. Water rinse and dip into a 15% HCL solution for about two seconds and again water rinse. This etches the plated surface with helps to assure a stronger plated bond.
5. Immediately place the window in a cyanide copper solution of 3.5 oz/ga. of CuCN and 6 oz/gal. of Rochelle salt. Give the window a high current strike at 6 volts for about two seonds. This initiates the plating action before any oxidation can occur on the plated surface which, in turn, would cause a flaky copper plate.

6. Plate the window at a slower rate with two volts for five to ten minutes. The slower rate assures uniformity over the peripheral area and yields a surface which will not flake.

7. Remove the window from the plating bath, water rinse, and dry, the window is now ready for soldering.

The plating currents depend upon the area to be plated. In this case currents of about 50 ma and 20 ma respectively were used. This particular CuCN solution has a current plating range of 20 amps/sq. ft. to 60 amps/sq. ft.

Several plating solutions were tested and CuCn proved most effective. Copper acid solution attacked the beryllium; nickel and silver did not plate with a strong band to the Be; and gold plated unevenly, leaving pin spots of exposed Be over the plated surface.

The ceramic shell is prepared for mounting by an ultrasonic bath in clean acetone for thirty minutes followed by a thirty minute reinse in ethyl alcohol. This cleans the ceramic and plated surfaces thoroughly of grease and absorbed water. The shell is preheated to about 200° F on either a hot plate or in an oven. The shell's window seat is then fluxed and tinned with 60-40 lead-tin solder.

The shell is next cycled through the acetone/alcohol cleaner to thoroughly remove any rosin residue. Particular notice should be taken of the pin-guard ring area, as this is the shortest electrical leakage path in the shell itself. The shell is hot air dried and is ready for the window mounting.

The beryllium window is prepared in a similar manner. Once again the center portion of the window is masked off for protective purposes. The copper plated edge is fluxed and thoroughly tinned. Care should be taken to assure thinning of the entire copper surface. This avoids mounting problems such as exposed plated copper flashing over the effective window area thereby ruining the window's transmission properties. Thoroughly clean the masked assembly in an alcohol bath before removing the window from the assembly, and once again in a clean solution after window removal. Care must be taken at all times to avoid contaminating the window. Perspiration, for instance, could render the window ineffective.

The flux used throughout the above procedure is an alcohol-rosin flux which is acid free. Beryllium surfaces are immediately attacked by acid, and the fumes would dull the gold cathode of the shell. Both exposures measurably decrease the efficiency of a chamber and should always be avoided. The effective window area should at all times be protected from acid fumes, baths, and foreign matter.

The window and shell are now ready for assembly. The shell is mounted in a vertical position and slowly heated with a hydrogen flame. When the solder flow temperature is reached, the window is dropped onto its seat. The window's edge will quickly reach temperature and a smooth flow pattern should appear around the assembly's periphery. The flame may then be removed, allowing the solder to harden. Care should be taken to avoid an excess of solder. This could run down the walls of the cathode rendering the assembly useless.

After the chamber has cooled to room temperature, a thin coating of Hysol 1200 epoxy is applied to the soldered seam to assure a leak-free joint. It may be noted that although the copper-beryllium-solder-gold band was leak tight in most cases, the epoxy seal coat improved the reliability and increased the fabrication yield. After the epoxy has cured, the chamber is ready for leak testing and gas filling.

V. LEAK TESTING AND GAS FILLING

In all cases the assembled chambers have been found fit for gas filling if they pass the following test:

The chambers are affixed to a He mass spectrometer whose sensitivity is about 2×10^{-10} cc/sec of He at 1 atm. The chamber is evacuated and its internal volume exposed to the spectrometer detector tube. The spectrometer's electrometer signal detector is set to the most sensitive scale.

At this point a rough check of all seams is performed with a He gas jet. If the chamber exhibits no gross leaks, a plastic bag filled with He is placed over the chamber and tied together. If after a five minute exposure to this arrangement no He ion signal is obtained, the chamber is considered fit for gas filling. It is worth noting that excessive atmospheric-vacuum cycling should be held to a minimum thus avoiding window fatigue or rupture.

Leaks may be stopped by applying epoxy. However, care should be taken in the area between the Kovar anode and the guard ring. (See Figure 1). For instance, if signals in the 5×10^{-13} ampere range are expected to be detected then an epoxy resin such as Hysol 1200 could not be used, because a high electrical surface leakage path is developed across the epoxy which can contribute an error in the signal as high as 50% of full scale sensitivity when operating the chamber at 45 volts and at a slightly elevated temperature. If epoxy must be applied to this area, then one must be used whose dielectric properties are high, such as Armstrong C-7 with activator W. In a number of cases the ceramic to the rear electrical connector seal and the fill tube developed minute holes either through rough use of through defective ceramic to metal fusion. Armstrong C-7 should be used around the welds.

These ion chambers may be filled with either Argon or Xenon. Argon renders a sharper spectral absorption cut-off than Xenon thereby shaping a narrower pass-band. In the case of the type chamber used on OSO-1, the procedure was as follows: The chamber was affixed to a vacuum cycling system capable of reaching an ultimate pressure of 1 x 10^{-7} mm Hg. The chamber is evacuated first with a roughing pump and then a LN_2 baffled oil diffusion pump to an ultimate system pressure reading of about 2 x 10^{-6} mm Hg. The chamber is then warmed to about 170° F to enhance outgassing of the chamber internal surfaces. This is continued for 48 hours after which the system pressure will be about 3 x 10^{-7} mm Hg. The heat is then removed and after the chamber assumes room temperature, it is ready for admittance of the fill gas.

Research-grade Xenon is available in lecture bottles with impurities less than one part in 10^6. Therefore, double-freeze distillation techniques may be ignored, as the principal source of contamination of the gas will probably be from the system's walls during the gas transfer process.

When a system pressure of about 1200 mm Hg is reached, the lecture bottle is valved off. The trapped gas is then frozen with LN_2 into a glass bottle which is valved to the system. When an equilibrium system pressure is reached, lecture bottle gas is once again admitted to the system and frozen into the trap bottle. The trap bottle is then valved off and the system's residual gas is pumped away. After a system pressure of about 2 x 10^{-7} mm Hg. or less is obtained, the system is valved off.

Gas from the trap bottle is then allowed into the evacuated ion chamber until a pressure of about 760 mm Hg. is reached. The filled chambers are then valved off from the system. The chamber fill stem is now sealed off with a commercial pinch-off tool, snipped from the system, and the stem tip immersed in solder to assure the firmness of the cold weld joint.

VI. DETECTOR CHARACTERISTICS

The spectral pass-band of these detectors is determined by the transmission properties of beryllium combined with the photoabsorption characteristics of Xenon. Referring to S.J.M. Allan's data on the mass absorption coefficients of beryllium[4], a typical transmission curve for .005" thick Be is traced in Figure 3. Also shown is the absorption curve for Xenon under the pressure and path length conditions as defined by the ion chambers[5]. These two curves define a window approximately 8 Å wide which is ideally 100% efficient at its peak.

The energy loss per photon per ion pair created is chosen as the number most descriptive of the sensitivity of a gas detector. In this case Xenon renders an average energy loss for each created ion pair[5] of 22 ev or 2.8×10^{10} ion pairs/absorbed erg. If, for example, one used an electrometer capable of sensing a current of 1×10^{-13} amperes an energy flux of 1.4×10^{3} photons/cm^2 sec. could be measured at 6 Kev. This assumes that photon interaction with the gas results only in

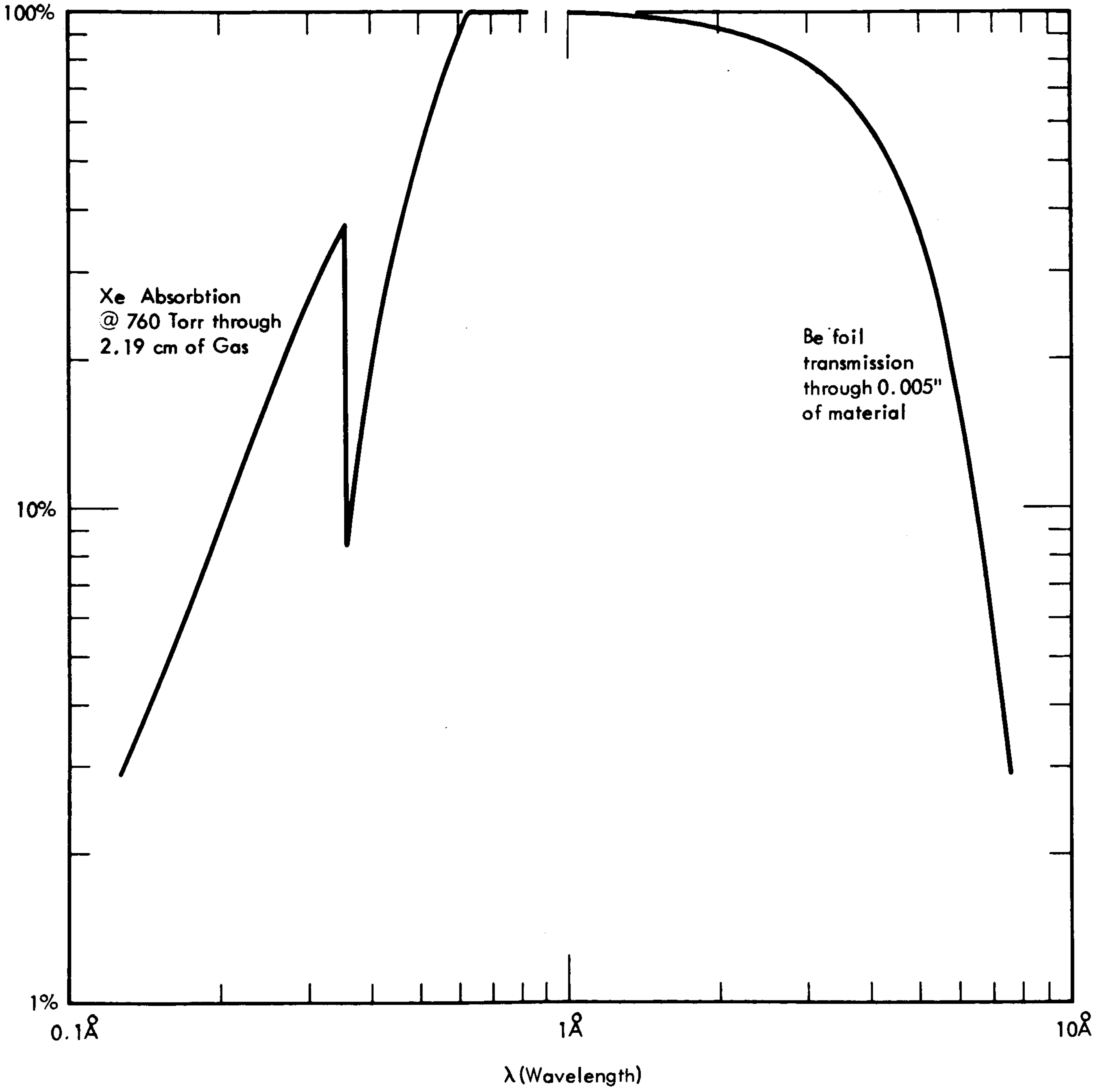


Figure 3. Spectral Passband of a Beryllium Windowed Xenon filled Ion Detector

photoionization, that all photons are absorbed, and all formed ion pairs are collected before re-combination occurs.

To date, the electrometer's current sensitivity limits the photon sensitivity of the detector at 6 Kev. The low shell-to-anode diameter ratio prevents any practical increase in sensitivity when attempting to operate the detector as a gas-gain device at gas pressures in the order of an atmosphere.

VII. APPLICATIONS

This detector, as described, has been used in several solar rocket probes, and was also flown in the pointed section of OSO-I, the latter being most fruitful in data yield for both quiet and active solar periods.

When operating this detector as a solar sensor, extreme caution should be exercised concerning interpretation of the intensity and spectral distribution of the input energy. The reader is referred to W. A. White's presentations on solar X-rays for further discourse on this problem[6,7].

VIII. ACKNOWLEDGEMENTS

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