Improved indium seal for LiF windows of resonance lamps for use at low temperatures

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(Received 17 October 1988; accepted for publication 28 November 1988)

A method of attaching crystalline LiF windows to glass lamps has been developed. The resulting seals have good vacuum properties and can be cooled to cryogenic temperatures while maintaining their vacuum integrity. The technique uses dual, indium O rings to provide a vacuum seal for a LiF window on a resonance lamp. It is appropriate for resonance lamps that produce line spectra. LiF crystals must be cooled to transmit Lyman-β radiation that can optically pump atomic hydrogen. This indium seal has other applications such as windows for VUV lasers. Details of the construction, assembly, and performance of the seal are described. A discussion of the merits and applications of the window seal is presented.

INTRODUCTION

For some applications, such as the optical pumping of atomic hydrogen,[1] it is necessary to produce Lyman-β radiation that occurs at 1026 Å. Currently there are no appropriate laser sources at this wavelength, although resonance lamps of hydrogen or deuterium can be made to produce useful fluxes of narrow-line radiation. One problem in making such a lamp is that no solid window material transmits 1026 Å radiation at room temperature. Hunter and Malo[2] found that when LiF crystals were cooled, they would transmit well at 1026 Å. A rf-excited lamp with a LiF window cooled by liquid nitrogen has been constructed.[3] It produced an intense flux of Lyman-β radiation. However, a reliable technique for attaching the LiF window to the pyrex body of the lamp was an ever present problem. This proved a barrier to scaling the lamp to larger size and greater flux intensity.

There are several important characteristics that a useful resonance lamp must have. It should have a discharge that produces high-intensity hydrogen resonance radiation. The radiation should have low self-reversal and narrow spectral width to better match the absorption profile of the atomic gas or beam. A high-frequency discharge is preferable for this reason. The discharge must dissociate the molecular hydrogen to atomic hydrogen. So the discharge must not be exposed to metals since this will cause the hydrogen to recombine efficiently to molecular hydrogen. The lamp should be made of low vapor pressure materials and be free of contaminants. Any materials which might form a film on the surface of the lamp might lead to an increase in the recombination rate of the atomic hydrogen. Also a film on the LiF window would lower the transmission of the window in the VUV. The discharge in the lamp must be close to the window to reduce any self-reversal of the hydrogen lines. The electrode structure of the lamp must be arranged so that the field lines which drive the discharge are parallel to the surface of the LiF window. This reduces ion bombardment of the surface of the window that can cause color centers to form. Color centers in LiF degrade transmission in the VUV. There should be some effective way to shield the lamp to prevent unwanted resonance lines and rf radiation from leaking from the lamp region into an experimental region. There must be good thermal cooling of the LiF window since the transmission in the VUV decreases rapidly as the temperature of the LiF crystal increases.

The requirements for a seal in such a lamp are therefore stringent. It must resist repeated cooling to liquid nitrogen temperatures. It must provide good thermal conductivity. It must be compact since the window needs to be close to the discharge of the lamp to avoid self-reversal of the resonance line. The materials in the seal must have a low vapor pressure. Also these materials must be compatible with LiF which is hygroscopic and brittle.

The material for the seal that meets all these requirements is indium. The first approach for constructing a lamp involved brazing the LiF window to the pyrex bulb with indium.[3] The pyrex body was partially covered by a gold film. This technique has long been known.[4,5,6] Although the approach was successful, the following properties frustrated attempts to scale the lamps to larger sizes. A large vacuum oven is required to braze the seals of the lamps. The gold film can outgas in the brazing process and contaminate the window surface. Sometimes several attempts are required to braze the seals. Finally, windows can only be used once. This is prohibitive since the larger LiF windows are costly.

Another approach is to use an indium compression seal. Initially the configurations that were considered were inconvenient since these seals were too bulky. The surfaces of the mating elements needed to be of high quality.[7] The assembly put too much stress on the fragile LiF window.

A way to circumvent these problems has been provided by Lim in a recent article.[8] The primary criterion for a successful indium compression seal is that large local pressure should be produced around the indium. Lim calls this the "extrusion principle." Also the mating surfaces should have a smooth finish. However with a higher pressure on the seal a lower quality of the surface finish can be tolerated. Finally

0034-6748/89/030467-04$01.30

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the surface should be clean and free from contaminants such as oil. These insights and a synthesis of the designs put forth by Lim have lead to a window seal for a resonance lamp that is described here. The seal is elastic at cryogenic temperatures and deforms to accommodate the thermal expansion of the fragile LiF windows. It provides thermal conductivity to cool the window and allows the window to be removed and reinstalled. Also it is compact and does not interfere with the functioning of the lamp.

I. CONSTRUCTION DETAILS

The lamp assembly is shown in cross section in Fig. 1. The indium seal is shown compressed. In this assembly two seals are made. One is along the rim of the LiF window and the other is along the circumference of the pyrex bulb. The six main components of the seal are the LiF window, the copper cooling block, the pyrex bulb of the lamp, the inner flange, the outer flange, and the middle ring. A detailed view of the indium seal is shown in Fig. 2.

The copper cooling block is the basic support structure for the lamp and seal. It is made of a single piece of OFHC copper to reduce outgasing. The block is 19.0 mm (0.750 in.) thick and can be attached to a liquid nitrogen cooled trap in a vacuum chamber. There is a hole 14.1 mm (0.555 in.) deep and 38.4 mm (1.510 in.) in diameter in the rear of the block to accommodate the pyrex bulb and the electrode structure of the lamp. The aperture through the copper block for the flanges is 28.6 mm (1.126 in.) in diameter and 3.96 mm (0.156 in.) deep. Twelve holes were drilled and tapped on a 33.3 mm (1.312 in.) bolt circle for the flanges. There is a recess for the outer flange 38.4 mm (1.510 in.) in diameter and 0.99 mm (0.039 in.) deep. The copper was electrolytically coated with gold[9] to reduce surface corrosion when exposed to air.

The pyrex bulb of the lamp was made of 1.5-mm wall tubing selected from stock for roundness. The cross section is nearly elliptical with the major axis being 25.1 mm (0.987 in.) and the minor axis 25.0 mm (0.983 in.). A thick-walled 6-mm capillary with a 1-mm inner diameter was fused on one end. The other end was cut perpendicular to the axis of the tube and lapped flat. The length of the bulb excluding the capillary is 31.8 mm (1.25 in.). The length of the capillary is 9.52 mm (0.375 in.).

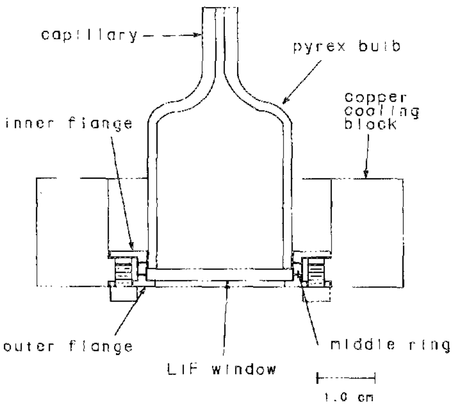

FIG. 1. Cross section of the window, indium seal, and flanges as viewed from the side. The indium seal is shown in the compressed configuration. Only the screws attaching the outer flange to the copper block are shown.

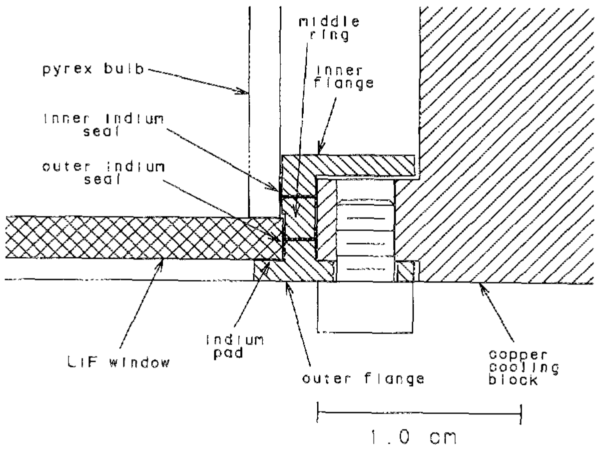

FIG. 2. Detail cross section of the indium seal.

The LiF windows are commercially available[10] and were unmodified for use in this seal. They are nearly round and 25.45 mm (1.002 in.) in diameter. The thickness is 2.06 mm (0.081 in.).

The arrangement of the stainless-steel flanges is the heart of this design. The sealing flanges consist of an outer flange, a middle ring, and an inner flange. The purpose of having two flanges is that when the pyrex bulb or window is changed, the copper block does not need to be modified. Only the flanges and the middle ring need to be changed.

The outer flange and the middle ring make the indium compression seal for the LiF window. The outer flange has a ring projection that fits between the copper block and the LiF window. This ring is the plunger for the compression seal of the window. The height of the ring is 1.02 mm (0.040 in.) and the width is 1.55 mm (0.061 in.). The outer diameter of the ring was machined to slip fit in the copper block so that a 0.0005-in. gap remained. The inner diameter of the ring was custom machined for the particular LiF window that was used so that a 0.0015-in. gap remained between the window and flange. Similarly the lower portion of the inner diameter of the middle ring was machined to have a 0.0015 in. clearance with the window. However when the middle ring was inserted, it sometimes rotated and caught against the sides of the copper block. So the outer diameter of the ring was made to fit loosely in the copper block. The outer diameter was 28.55 mm (1.124 in.) so a 0.001-in. gap remains between the ring and the copper block. The overall height of the middle ring, including the part around the window and the pyrex bulb, is 2.01 mm (0.079 in.). The part that has the inner diameter for the window is 1.22 mm (0.048 in.) high. When the indium seal is compressed, the indium extrudes between the LiF window, the outer flange, and the middle ring and make the seal for the LiF window. A certain amount also extrudes between the outer diameter of the middle ring and the copper block. This provides a place for the excess indium to go. There is a support lip for the LiF window on the outer flange.

The inner flange and the middle ring make the compression seal for the pyrex bulb. There is a ring projection of the inner flange that fits between the copper block and the pyrex bulb. This ring is the plunger for the compression seal of the pyrex bulb. The height of the ring is 1.02 mm (0.040 in.) and the width is 1.73 mm (0.068 in.). The outer diameter of the ring was machined to slip fit in the copper block so that a 0.005 in. gap remained. The inner diameter of the ring was machined to fit the particular pyrex bulb that was used. Because the bulb was out-of-round a 0.002–0.003-in. gap remained between the pyrex bulb and the inner flange. The top of the middle ring has the same inner diameter as the inner flange. When the arrangement is compressed the indium extrudes between the pyrex bulb, the middle ring, and the inner flange to make the seal for the pyrex bulb. Care was taken with the inner dimensions of the middle ring so that when the flanges were fully compressed, the edge of the inner diameter does not press against the LiF window and crack the window. When the lamp was assembled, there was a minimum clearance of 0.006 in. between the LiF window and the edge on the inner diameter of the middle ring.

The flanges have six holes on a 33.32-mm (1.312 in.) bolt circle. They were held in place with 4–40 stainless-steel screws. The outer and inner flanges were bolted to the copper block using alternate holes of the copper block for reasons to be discussed later. When the flanges were fully compressed a minimum distance of 0.003 in. remained between the bottom of the inner flange and the top of the copper block. The stainless-steel flanges, LiF window, and copper block contract and expand at greatly different rates when cooled or heated. This gap provides a way for continuous tension to be maintained on the flanges by the screws when the seal is cooled or heated. Care was taken with all dimensions so that no excess stress was put on the window when the flanges were fully compressed.

II. ASSEMBLY

When the bolts are tightened a double seal is simultaneously formed. Care is needed in assembling the flanges so that a good seal will result. All the components except the window were cleaned in an ultrasonic bath. Indium tends to adhere best to clean surfaces. The windows were not cleaned to avoid contaminating or damaging the surfaces and lowering the transmission in the vacuum ultraviolet.

The indium O ring was formed by extrusion through a small hole in a stainless-steel plunger similar to a recently reported design.[11] Both ends of an appropriate length of indium were cut at a 20° bevel and laid atop one another. The diameter of the O ring was chosen so that the sealing space would be filled. If too much indium is used, the indium will extrude between the window and the pyrex bulb into the lamp interior when the seal is compressed. An O-ring diameter of 1.58 mm worked well.

Before the seal was assembled the screws were lubricated with molybdenum disulfide. This lubricant prevents galling of the threads and has a low vapor pressure. First, the outer flange was screwed in place. Three thin pads of indium 0.001 in. thick were put on the lip of the flange for the LiF window to rest on. This prevented stresses that could crack the LiF window during later stages of assembly. The window was lowered into place and an indium O ring placed around it. The middle ring and inner flange were inserted and lightly screwed down. This caused the indium to distribute uniformly in the first seal and also to hold the window in place. There are twelve holes in the copper block. The inner flange is screwed to the copper block with the alternate holes not used by the outer flange. The inner flange was then removed. The pyrex bulb was inserted and another indium O ring was laid around it. The inner flange was then again mounted and lightly screwed down. At this point the indium of both seals was uniformly distributed. The screws on the inner flange were now further tightened. They were tightened in a crisscross fashion to be certain that the indium is uniformly distributed. First one screw is tightened a quarter turn. Then the screw on the opposite side of the flange is tightened a quarter turn. Then this is repeated for the screw next to the first one and the screw opposite to it. The tightening of alternate screws proceeds in a clockwise fashion until all the screws are tightened on the inner flange. While the screws were tightened, the progress of the seal was monitored by observing the interior of the window and pyrex bulb. As the seal was compressed the extruding indium centers the window and the pyrex bulb in their respective flanges. When the seal was properly formed, a shiny ring of indium appeared around the inner surface of both the pyrex bulb and the edge of the window. This showed that the seal was formed properly. Also any evidence of window cracking was monitored.

III. PERFORMANCE

After assembly, the seal was tested for vacuum integrity and its ability to survive thermal cycling. The lamp was attached to a pump station by the capillary tube. The lamp was evacuated by a 2-ℓ/s ion pump. The end pressure was 2×10^{-5} Torr. This corresponds to a leak rate of 4×10^{-5} mm ℓs^{-1}. Although this is not ideal, it is certainly adequate for this application. This lamp is intended to be used in a high-vacuum, atomic-beam apparatus. The lamp will contain less than 1 Torr of gas, be located directly over a 2000-ℓ/s cryopump, and be required to operate in 10^{-7}–10^{-8}-Torr vacuum. Although it is not necessary for this application, it is certainly possible to improve this seal.[8] In particular, one could grind the pyrex bulb so that it is round, not elliptical, for the seal with the inner flange. Further, the outer diameter of the middle ring could be increased to reduce the gap between it and the copper block. This would prevent the indium from extruding between the middle ring and the copper cooling block. The pressure on the indium seal around both the LiF window and the pyrex bulb would increase and so improve the seal.

The lamp was attached to a liquid nitrogen Dewar in a vacuum chamber and cooled to less than 80 K several times. When the lamp was removed, it was again evacuated on the pump station. The end vacuum was the same as before cooling. This shows that thermal cycling does not affect the seal. Although the LiF window and pyrex bulb have much different thermal expansion coefficients than the stainless steel flanges, the seal is not seriously affected since the indium accommodates any expansion or contraction that occurs.

Occasionally it is necessary to change the window of the lamp. This design allows this to be done in a convenient manner. However, since the indium tenaciously adheres to the flanges, it is necessary to force them apart. The design allows this to be done easily also. The screws in the flanges are first removed. Then longer screws, about one inch in length, are put in their place. When fully screwed in, they push out the opposing flange on the other side. Occasionally the outer flange must be gently heated to separate the LiF window from it. If a window is replaced, it is only necessary to make a new outer flange and middle ring. Similarly, if a pyrex bulb is replaced, only a new inner flange and middle ring need to be made.

IV. DISCUSSION

The seal described here solves the previous problems of attaching a fragile, crystal window to a VUV lamp. This lamp allows the use of larger windows than in previous lamps. It can be conveniently scaled to a row of lamps. Such an arrangement has been constructed with two lamps side-by-side. In the actual configuration of the lamp, the rf discharge is driven by two electrodes on opposite sides of the pyrex bulb. With this geometry the electric field lines are nearly parallel to the LiF window. This inhibits the formation of color centers that degrade the VUV transmission of the windows. The lamp is filled with a helium-hydrogen mixture that is fed into the lamp through the capillary after passing through the copper cooling block to precool the gas. The ratio of hydrogen to helium can be varied to minimize self-reversal while maintaining a stable discharge and intense resonance lines.

This indium seal provides good thermal conductivity between the LiF window and the copper-cooling block. Since LiF itself has a large thermal conductivity, large intensities from the lamp are possible without overheating the window. If the window heats, the temperature of the window may rise so that the LiF crystal no longer transmits in the VUV. The materials that are used in this seal are all suitable for use in ultrahigh vacuum systems. There is therefore no possibility of contamination of the surface of the window, interior of the pyrex bulb, or the vacuum apparatus that contains the lamp. There is no metal in contact with the discharge. If there were, the metal would cause the atomic hydrogen to recombine into molecular hydrogen. Also the metal surface might sputter in the discharge and coat the window. Since this seal is flat, the discharge can be close to the window. This reduces the self-reversal of the resonance lines. The windows can be conveniently changed. This may be necessary if color centers are formed in the LiF crystal. This reduces down time and expense.

The lamp seal described above may prove useful in a variety of ways. Precision measurements involving the $2S^{1/2}$ metastable state in hydrogen need an intense, cold source. This seal makes possible the construction of lamps that can be used to effectively optically pump an intense, ground-state hydrogen beam.[12] Such an intense metastable beam would be of interest in precision optical measurements[13] as well as measurements of fundamental quantities.[14] In addition the window seal may find application in a window for VUV lasers such as the free-electron laser.

ACKNOWLEDGMENTS

I would like to thank C. C. Lim, W. L. Williams, and T. J. Whitaker for helpful discussions. I would also like to thank C. Fehrenbach for assistance at the initiation of this work. I gratefully acknowledge the excellent work done by the machine shop in the Department of Physics of the University of Michigan. A portion of this research was supported by the National Science Foundation, Grant No. PHY-8218744.

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