Indium seals for low-temperature and moderate-pressure applications

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Some ideas as to what the important factors are that will produce a reliable indium O-ring seal are discussed. Four novel indium seal designs which are based on these ideas are presented. Three of these seals have been incorporated in a Brillouin cell. They include a simple sapphire window seal, a filling line union, and a flange seal. All of them have withstood low temperatures down to 77 K and moderate pressures up to 177 bar. The fourth seal described is a Pyrex tube seal for a Raman cell.

INTRODUCTION

It would appear from the experience of Turkington and Harris-Lowe, the authors of a recent note in this Journal, that indium O-ring seals are still not completely understood despite their widespread use over a period of some 30 years.² I would like to share with your readers some thoughts as to what are the important design factors that will produce reliable indium seals. These ideas have been gathered from 29 years' experience with the design and use of such seals. Several of my successful indium seal designs which are based on these ideas will be presented in support of them. Three of these seals have been incorporated in a recent Brillouin cell that is to be used at low temperatures (down to 2.0 K) and moderate pressures (up to 82 bar). These include a simple sapphire window seal, a demountable filling line union, and a flange seal. The Brillouin cell and all its seals have been tested and have withstood pressures up to 177 bar at temperatures down to 77 K. From past experience it is known that if an indium seal is effective at 77 K it is usually reliable at helium temperatures.

I. IMPORTANT FACTORS IN THE DESIGN OF INDIUM SEALS

To make the discussion of the above-mentioned ideas more concrete, they will be discussed in terms of the demountable flange seal. Indium flange seals may be conveniently divided into three general types. Figure 1(a) shows the first type which might be called the untrapped O-ring seal. The second is shown in Fig. 1(b) and is a partially trapped O-ring seal and Fig. 1(c) shows the third which is the fully trapped O-ring seal. This classification is somewhat arbitrary and it would be quite impossible to fit all flange seals neatly into one of these three types, e.g., the shallow groove seal³ which is a fairly popular one. In this seal, the Oring is partially trapped by a groove, in one of the flanges, that is too shallow to accommodate all of the indium and consequently the excess extrudes into a thin gasket when the flanges are clamped together, the sealing action is intermediate between that of the fully trapped O-ring and the untrapped one. The meaning of this will become clear later in this section. Most flange seals, however, are variations or combinations of these three.4

For an indium O-ring to seal effectively at low tempera-

tures and moderate pressures, the indium gasket formed from the O-ring must withstand the stresses produced by the differential contraction and the applied pressure and still remain continuous and bonded to the flanges. The differential dimensional changes, due to thermal contractions, between the flange and the indium gasket are quite small and the ductility of indium, even at low temperatures, seems to be sufficient to accommodate these changes without the indium gasket rupturing. However, these same stresses may be large enough to break the "bond" between the gasket and the flanges. Thus the most important factor to consider in the design of a seal is the strength of the bond between the indium gasket and the flanges. My feeling is that the strength of the bond depends on two things: (1) the nature of the surface and (2) the maximum applied local pressure between gasket and flanges during the formation of the gasket. The nature of the surface is determined by the surface finish (see

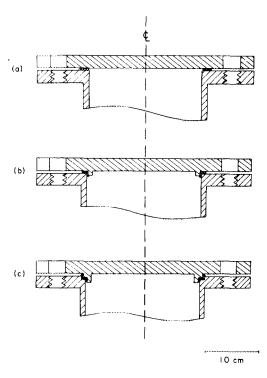


Fig. 1. Examples of the three types of indium O-ring flange seals. (a) The untrapped O-ring seal, (b) the partially trapped O-ring seal, and (c) the fully trapped O-ring seal. Note: The thickness of the indium gasket in all the figures in this article is greatly exaggerated for the sake of clarity.

Ref. 1 for a discussion of this), the material of the surface and the amount of contamination on the surface. Indium will bond strongly to many surfaces provided the surfaces, whether smooth or rough, are clean and a sufficiently large local pressure can be generated, by some means, to press the flanges onto the gasket. This statement in no way contradicts the conclusions drawn by the authors of Ref. 1 from the tests they carried out with the simple seal design that they used [similar to that shown in Fig. 1(a)] because that particular design may be capable of producing the necessary pressure for bonding the indium to only some types of surface finishes but not to all. In that design the untrapped indium O-ring is free to thin out and increase its contact area with the flanges when the bolts are tightened. This limits the amount of pressure that can be generated since any increase in force, as a result of bolt tightening, is at least partially counteracted by an increase in the contact area. No sizable increase of pressure, can be produced until the gasket stops thinning out. By which time, however, the contact area may have become so large that, for certain types of surface finishes, the bolts cannot be tightened further to produce the required local pressure for bonding before the bolts or machine screws (particularly brass ones) strip their threads or shear off their heads. The use of a smaller diameter indium wire for the O-ring to start with, coupled with the stronger steel bolts, may solve the problem for some of these more difficult surface finishes. There are limits, however, as to how small a diameter indium wire can be used before the seal becomes unreliable. A better solution, which I recommend, is to trap the O-ring; this will be discussed next.

In contrast to the untrapped O-ring design, the fully trapped O-ring seal can produce the necessary local pressure for bonding because, although the indium contact area is increasing, the relevant area for producing the pressure is not. To see how this works let me, instead of the design in Fig. 1(c), describe an equivalent configuration, that shown in Fig. 2. I have used this "ring and groove" seal many times. The seal consists of a square cross-section $(1.6 \times 1.6 \text{ mm})$ groove in the bottom flange⁶ which mates with a ring of a similar cross section in the top flange. The ring, however, is narrower and deeper than the groove by about 0.051 mm so that when the ring is centered in the groove there will be a narrow gap of 0.026 mm between the vertical sides of the ring and the groove and a horizontal gap of 0.051 mm

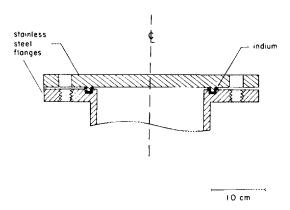


FIG. 2. The "ring and groove" indium O-ring flange seal.

between the flanges. The diameter⁷ of the ring and groove will depend on the size of the chamber to be sealed. A sufficient number of evenly spaced stainless-steel allen head bolts are used to clamp the flanges together and form the seal.

The indium O-ring lies in the space defined by the groove and the bottom of the mating ring, i.e., the trapped volume. As the bolts are tightened this volume will decrease and the indium O-ring8 in that space will deform till at some point it will completely fill the trapped volume. Past that point, the pressure on the indium will increase sharply with further bolt tightening since the indium is highly incompressible. Here, in contrast to the untrapped O-ring design of Fig. 1(a), the relevant area for pressure generation is that of the ring bottom, which is fixed, and not the total area of contact between the indium and the flanges. As the force exerted by the bolts increases, the pressure increases proportionately (at least to a first approximation). Thus the "ring and groove" acts much like a "piston and cylinder" device compressing a fluid. The pressure will eventually become great enough to extrude the indium out of the trapped volume around the bottom corners and up the very narrow vertical gaps between the ring and the groove to form a Ushaped cross-section gasket. Finally, if there is enough indium, it will extrude around the corners at the top and start filling the horizontal gap between the flanges. From then on the relevant area for pressure generation will increase as the horizontal portions of the gap are filled and the local pressure will not increase further or, at least, not as rapidly now.

The pressure needed to produce extrusion will depend on, among other things, the vertical gap width and the nature of the surface. In most cases the pressure produced by this in situ extruder is great enough to bond the indium strongly to the flange. In fact the bond is so good that it is very difficult to separate the flanges after the bolts have been removed. Further evidence of this strong bond can be seen in the bits and pieces of indium still clinging tenaciously to the flanges at many places, especially near the bottom corners and the vertical sides of the ring and groove, after the flanges have been disassembled.

I have emphasized the importance of this local pressure in the formation of a strong bond by giving it a lengthy discussion. It would be very satisfying if I could now specify this pressure precisely. Unfortunately no experiments have been done to measure this pressure for any of the various types of indium seals. But even if the pressure were known, it would be very difficult to incorporate this information in a practical design since there would still be no easy way of knowing what pressure was being generated in any new design. What is often found in the literature is some "rule of thumb" which really applies only to the particular design discussed. For example, in the case of the untrapped O-ring seal some experimenters have tried to specify this pressure indirectly by giving the number and type of clamping bolts and the final tightening torque applied, as measured by a torque wrench. This kind of specification is very crude at best and it would be very difficult to convert this information to that needed for a flange of a different diameter, especially if the clamping bolts are of a different size and type. Other experimenters

have tried to avoid this problem by using wavy washers or springs to try to maintain at low temperatures the tension in the bolts which produced a reliable seal at room temperatures. Still others have specified that the bolts be of a material that contracts more than the flanges on cooling.

In the fully trapped O-ring seal we have the means of producing the required bonding pressure for flanges of any diameter in a consistent way without having to know the value of the pressure. To see how this works let me review the ideas behind the "ring and groove" seal. In that seal the bonding pressure is created by the "piston and cylinder" action of the ring and groove on the trapped indium. In order for the indium to extrude out of the trapped volume and into the narrow vertical gaps between the ring and groove the pressure must exceed a certain minimum value, which, among other things, depends on the gap width. By choosing a suitable gap width the extrusion pressure can be made equal to or exceed the bonding pressure. From experience I have found that vertical gap widths ranging from 0.013 to 0.051 mm to be suitable. It should be noted here that the narrower the gap the higher will the extrusion pressure be but the narrower the gap the less indium sealant in the gap. Very thin indium gaskets may not be very reliable and some compromise must be struck between bonding pressure and gasket thickness. Thus by choosing a suitable gap width we can be sure that if the indium extrudes into this gap the bonding pressure is great enough to form a good seal. This might be termed the "extrusion principle" of indium seals; no knowledge of the bonding pressure is needed. The reader can easily discover other suitable gap widths for himself by a little trial and error.

Since a "ring and groove" of the same cross-sectional dimensions may be used for a wide range of flange diameters, I have standardized them. As described previously the groove cross section is a 1.6×1.6 mm square and the mating ring is 0.051 mm narrower and deeper. This gives a vertical gap width of 0.026 mm on each side of the ring. In addition, there is a horizontal gap of 0.051 mm between the flanges when they are fully mated without any indium in the groove. This horizontal gap has two useful roles. In the first place, it absorbs the excess indium that might extrude all the way up to the top and in the second place it performs a similar function as a wavy washer in maintaining tension in the bolts. A 0.76-mm indium wire (see Ref. 8) is used to make the O-ring. With this diameter indium wire some indium will extrude into the top horizontal gap between the flanges if the flanges are tightened enough; this is of no real consequence. However, where it does matter the reader could use a thinner indium wire but he should make certain that there is sufficient indium to fill the vertical gaps.

Ultimately, the flanges and bolts are the means by which pressure is applied to produce the seal. The requirements for the flanges are not too stringent (see Ref. 6); they should be thick enough that they are not permanently deformed by the clamping action of the bolts and should be strong enough to withstand the applied pressure differential with a suitable safety margin. The bolts serve two purposes. Initially they are the means by which the flanges are forced together to form the gasket and bond it to the flanges, after which they

help maintain the integrity of the seal by preventing any significant movement of the flanges relative to each other. This movement may be caused by a differential contraction on cooling or by an applied pressure differential which would tend to push them apart, as is the case in a pressure cell. The number and size of bolts needed to form the gasket are not critical so long as they are sufficient to produce an even squeeze on the O-ring (see Ref. 10). In the case of a pressure cell however, the number and type of bolts are quite critical and should be chosen to withstand the pressure differential with the required safety margin. If the flanges contract more than the bolts on cooling, the pressure on the indium gasket will be reduced by the contraction. This is a serious problem in the case of the flat flange (untrapped Oring) seal where the bonding pressure is often insufficient to make a strong bond and the pressure exerted by the bolts, through the flanges, is necessary to maintain the effectiveness of the seal; but even when the bond is strong, vibration and other mechanical shocks may cause the flanges to move relative to each other when the bolts are loosened by the differential contraction and thus opening the seal. In this type of indium seal the flange and bolts (or screws) should be made of the same material or better yet the bolts should be of a material that contracts more than the flanges on cooling. The above problem is one more reason to avoid using untrapped O-ring seals if possible. In contrast, bolts do not play as important a role in the "ring and groove" trapped O-ring seal after the gasket has been formed and the bond made. Since the bond between the indium gasket and the flanges is strong in this case, the integrity of seal does not depend on the added pressure supplied by the bolts. Also, any reduction of bolt-supplied pressure will affect only the bottom segment of the U-shaped gasket that is sandwiched between the two vertical segments which are largely unaffected. Further, because of the design the seal can withstand better any vibratory or other mechanical forces. The flanges are literally locked together by the gasket. Motion of the flanges parallel to each other is prevented by the mated ring and groove (any slack has already been taken up by the indium gasket) and, because of the excellent bond between indium and the flanges, it is extremely difficult to separate them even if one wants to (see Ref. 9). In the case of a vacuum chamber flange seal, I would not be surprised if the bolts prove to be superfluous once the seal has been formed. However, for safety, I would leave the bolts in place. Thus in the "ring and groove" seal it would be sufficient to make the bolts of the same material as the flanges with a slight pretensioning if the seal is for a vacuum can; for a pressure vessel the bolts should be pretensioned enough to prevent the pressure differential from opening the seal.

Although the manner of tightening the bolts in a "ring and groove" seal is not critical, it would be best to tighten them evenly using a criss-cross pattern.¹¹

In the design shown in Fig. 1(b) the O-ring is partially trapped by the corner on one side and the pressure required for bonding can be achieved more readily than in the design shown in Fig. 1(a), especially if the O-ring is positioned close to the corner to start with. The sealing action is intermediate between the two previous designs.

To recapitulate the main ideas: while the surface finish is one major factor in determining whether indium will bond strongly to the flange surfaces or not, the other major factor, local pressure, is the dominant one; i.e., if a sufficiently large local pressure can be applied to form the seal, a strong bond will result for most of the surfaces encountered provided they are clean. The "ring and groove" seal which uses the "extrusion principle" guarantees this strong bond without any necessity for knowing the value of the local pressure or the loading on the bolts if a suitable vertical gap width is used. As further evidence of this contention I will describe three other successful but very different seal designs which are really "ring and groove" seals in disguise. Two of these together with the "ring and groove" flange seal described above have been incorporated in a Brillouin cell which is to be used for low temperature experiments (down to 2.0 K) at moderate pressures (up to 82 bar). The third design is a seal for a Pyrex tube in a Raman cell. The surface finishes in these designs range from coarse, for the sapphire window, to mirror smooth, for the Pyrex tube.

II. SAPPHIRE OPTICAL WINDOW SEAL

Figure 3 shows the top view of the Brillouin cell and two of the four sapphire windows of the cell. The lower one is displayed fully assembled while the upper one is shown in an "exploded" form so as to exhibit more clearly the various elements that make up the seal. The sapphire window is a commercially ¹² available single-crystal circular sapphire flat 12.7 mm in diameter by 3.18 mm thick. The flat is specified to be parallel to 0.076 mm and flat to 8–10 wavelengths. The surface of the cylindrical side is a coarse ground one, similar to the sides found on unmounted single element lenses. The unsupported area of the window is 5.0 mm in diameter. Nothing special ¹³ was done to it except to degrease it careful-

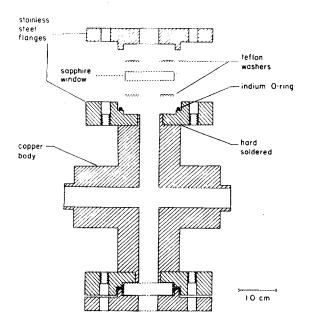


FIG. 3. Top view of the Brillouin cell with two of its four sapphire windows shown. The lower window is fully assembled while the upper is shown in an "exploded" form to exhibit more clearly the various elements that make up the seal.

ly before assembly. From the diagram it is clear that the sapphire flat and the stainless-steel flanges form an in situ extruder similar to the "ring and groove" in Fig. 2 except that the indium is in this case extruded into the gap between the window and flanges at a point in the middle of the side rather than at the bottom. With this arrangement the window is centered in the flange more readily by the extruding indium and the gap is filled more quickly. When the window is centered exactly the gap is 0.038 mm wide but will in fact vary somewhat because the centering is not perfect. The vertical gap between the flanges themselves were kept to about 0.013 mm to force more of the indium into the gap between the window and the flanges where the important part of the seal is. In this seal indium acts as both a sealant and a buffer between the window and the flanges. Two 0.051-mm-thick FEP Teflon washers prevent the faces of the windows from coming into direct contact with the flanges which could produce localized areas of high stress if they did, and that could promote cracking in the window. The edges of the sapphire flat should be bevelled enough to prevent direct contact with the flanges at their internal corners since perfectly square internal corners are difficult if not impossible to machine. Six 4-40 stainless-steel allen bolts are used to clamp these flanges together.

These windows were tested in position in the Brillouin cell. The cell was cooled down to 77 K and an internal pressure of 177 bar (the maximum obtainable from a helium gas cylinder) was applied while the outside, which was held in a vacuum, was connected to a helium leak detector with a sensitivity of 10⁻⁹ std cm³/s. No leak was detected. The internal pressure was kept at 177 bar while the cell was warmed to room temperature with still no sign of a leak. The thermal cycling was repeated twice more with the internal pressure maintained at 177 bar with no detectable leak. While the window seals have not been tested at helium temperatures, past experience has shown that a seal which is effective at 77 K will remain so at helium temperatures. For example, the "ring and groove" flange seal has previously been used in other apparatus and has proved tight even against superfluid helium.

Incidentally, in another application a pair of crystalline quartz flats sealed in similar flanges have been used successfully as windows for a far-infrared absorption gas cell. The quartz flats were 25.4 mm in diameter by 6.35 mm thick. The unsupported window area was larger in this case, about 10.0 mm in diameter. These windows have withstood pressures up to 97 bar at room temperatures and have been in use for some time now during which period they have undergone several hundred temperature and gas pressure cycles with pressures up to 34 bar and temperatures down to 126 K.

While these windows are quite easy to construct and use, a few things that would ensure their success should be emphasized. Direct contact between the windows and the flanges which can produce localized areas of high stress must be avoided. This was done by using Teflon and indium as buffers and by beveling the edges of the flats if they have not already been done by the manufacturer. Burrs on the flanges that can penetrate the indium gasket or Teflon washer and touch the flats, if any, should be removed. The stainless-steel

flanges should have a reasonably smooth finish. The Brillouin and far infrared cell flanges were machined to a finish between 0.41 and 0.82 µm which is a good but not an extraordinarily fine finish. A rougher finish would probably still be all right. Bolts should be tightened gradually and evenly using a criss-cross pattern (see Ref. 11). A torque wrench, while not absolutely necessary, would be useful for even torquing and for determining the final torque necessary for a good seal. Uneven pressure on the windows may cause cracking. Since the differential contraction between sapphire and stainless steel will in fact aid the seal in this case the tension in the bolts is not critical so long as it is enough to withstand the forces produced by the internal pressure without allowing the seal to open. In the case where the differential contraction will tend to open the seal (e.g., a metal window, for neutron or x-ray scattering experiments, which contracts more than the flanges) the bolts should be overtightened to pretension them enough to prevent the release of pressure on the indium gasket when the windows cool down. In this case the torque wrench will be needed to produce consistent results.

III. FILLING TUBE UNION

The filling tube union shown in Fig. 4 allows the Brillouin cell to be detached easily from the cryostat without the need for unsoldering the filling tube. The brass union is shown fully assembled. When the compression nut is tightened, the indium O-ring trapped between the horizontal portions of the male and female parts of the union, is extruded into the narrow annular gaps between the two parts, to form a "staircased" cross-section gasket. The female part and the compression nut were both made from 9.53-mm hexagonal brass bar stock so that only a pair of wrenches are needed for compressing the O-ring and making the seal.

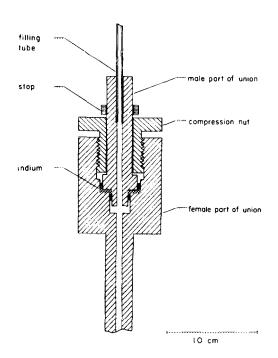


FIG. 4. The filling line union.

The dimensions of the various parts of the union are not critical, except for the annular gap, and should be chosen so that the union can sustain the maximum pressure with a suitable safety factor. The narrow annular gaps should be between 0.013 to 0.025 mm wide. The actual dimensions of the various parts will of course depend also on the construction material chosen; brass was chosen here for ease in construction and in soldering the union to a rather heavy Brillouin cell body. For greater strength, hardened beryllium-copper or stainless steel may be used.

There is a stop shown attached to the male part of the union just above the nut. This is needed for easy disassembly of the union. As mentioned previously in connection with the flange seal, parts sealed together in this trapped O-ring design are difficult to separate. 9,15 The same is true here; the bond is so strong that a pair of pliers will be needed to pull the male part out of the union if no stop is present; but with the stop in place, the compression nut will push the two portions of the union apart when it is unscrewed. The exact position of the stop on the male part of the union will be a compromise between the best position for assembly and that for disassembly. For easy assembly the nut should be slid away from the O-ring; this will allow the male part and the O-ring to be positioned in place before the threads of the compression nut must engage the threads of the female part. For disassembly the stop should be right next to the nut (as shown in Fig. 4). A ring with several set screws to lock it in place (after assembly) would work if the wall of the tube portion of the male part is thick enough to accept the screws without being weakened appreciably by them, otherwise the stop will have to be soldered in place and the compromise position will be that where the stop is right against the nut when only one or two turns of the threads of the nut are engaged with that of the female part.

IV. RAMAN TUBE CELL SEAL

Figure 5 shows the Raman cell which consists of a 4.19mm o.d. Pyrex tube with a window fused on to one end while the other is sealed to the heat sink and filling tube by a trapped indium O-ring seal. The light comes in through the window at the bottom while the light scattered by the material under investigation is collected through the side. The seal is very similar to that used for the sapphire window except that the sapphire surface sealed is coarse ground while the Pyrex surface here is mirror smooth. Four evenly spaced 2-56 stainless-steel allen head bolts are used to squeeze the copper flanges together to make the seal. The cell has been used at temperatures down to 77 K and with a pressure differential of little more than one bar. However, it would be a simple matter to design a Raman cell that would operate at a much higher pressure differential. In that case the Pyrex tube and window dimensions must be selected to allow them to operate at the higher pressures and some means must also be provided to prevent the tube from being blown out of the flanges.

V. SUGGESTIONS FOR OTHER APPLICATIONS

It has always been difficult for the low-temperature experimenter to make reliable glass-to-metal seals, especially

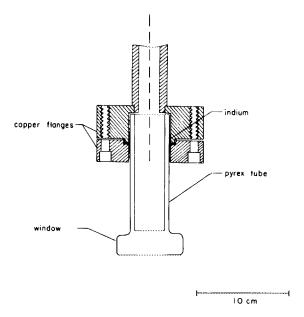


FIG. 5. The Raman cell.

ones that are easily demountable. The Raman cell tube seal can be readily adapted to solve many of these problems. For example, a thick-walled glass capillary tube has to be sealed to a metal bulb in the construction of a pycnometer that is to be used for low temperature liquid density measurements. In the past, fragile glass-to-metal seals (e.g., Kovar-to-glass, platinum-to-glass, and copper-glass Housekeeper seals) have been used for this purpose. Besides being fragile and not always reliable, the unknown nuisance volume introduced by the seals reduced the overall accuracy of the measurements. With the tube-type seal described above, the seal will be easy to make and will be very reliable. The nuisance volume introduced by the seal can be readily calculated from the gasket thickness, and the appropriate tube and flange dimensions. In fact the tube seal can be redesigned to make the nuisance volume negligible. If space permits, the flange design using bolts may be used, but if not, a compression nut together with a separate compression ring may be substituted.

In the helium film stopper for a dilution refrigerator described by Abraham and Falco, 16 an indium O-ring was used to seal the glass flat to the brass body. The face of the flat was platinized to ensure a good bond to it. A trapped Oring seal on the side of the flat, similar to the sapphire window seal would eliminate the need for platinizing the flat. Their original design is easily modified to do this. Incidentally, I have found that the indium seal made to the side of a circular flat (or window) with a "ring and groove" arrangement is far superior to that made to the face of the flat. In the first place the required bonding pressure is more easily produced and the flat is not subjected to any "bending" type stress that can produce cracking which inevitably accompanies the "face" seal when perfectly even clamping of the flanges is not achieved. In the second place, if there is a higher internal pressure which would tend to push the flat outwards, the "side" seal seems better able to withstand the

shear stress it sustains than the "face" seal the axial (longitudinal) stress it experiences.

ACKNOWLEDGMENTS

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¹R. R. Turkington and R. F. Harris-Lowe, Rev. Sci. Instrum. 55, 803 (1984).

²See Ref. 1 above for references to many of the published designs in this period.

³D. B. Fraser, Rev. Sci. Instrum. 33, 762 (1962).

⁴See K. L. Agarwal and J. O. Betterton, Jr., Rev. Sci. Instrum. 14, 520 (1974); G. K. White, Experimental Techniques in Low-Temperature Physics (Clarendon, Oxford, 1979), 3rd ed., p. 272; P. P. Craig, W. A. Steyert, and R. D. Taylor, Rev. Sci. Instrum. 33, 869 (1962), for more information on the type of seals shown in Figs. 1(a), 1(b), and 1(c), respectively.

⁵Exactly how indium adheres to a metal, glass, or other surface is not really known. Different mechanisms seem to be operating for different materials. The following statements are not based on the results of carefully designed experiments but rather on the inspections of many indium gaskets and flanges after the seals have been disassembled. For example, in the case of stainless steel the adhesion appears to be largely mechanical in nature. The "squeezing" pressure appears to have forced the indium into the surface imperfections, e.g., machine tool marks, mechanically "locking" the indium to the surface. In contrast, for brass or copper, the indium besides the mechanical locking seems to have penetrated the metal surface; this is especially true if the indium has been left clamped between the flanges for a long time. For smooth glass the adhesion may be due to a "wetting" action similar to that which takes place when indium is used to solder glass to metal. In any particular case one or more of these (or other) mechanisms may be operating and for want of a better word "bond" will be used to describe the adhesion of indium to a material surface and no particular mechanism or mechanisms is implied by its use. In any case the exact nature of the "bond" is not critical, provided it is strong, in the seal design which I will be advocating.

⁶Where suitable, I use stainless steel for the flanges because of its tarnish resistance and its great strength. However, other materials like copper or brass may be substituted if other factors demand their use. Where possible, use the same material for both flanges and the clamping bolts, but if this is not feasible and different metals must be used for the flanges, the groove should be machined in the material which contracts more on cooling; this aids the seal. If there is a requirement for both great strength and high thermal conductivity, stainless-steel flanges may be combined with copper, as is done for the Brillouin cell shown in Fig. 3, to accomplish this or hardened beryllium—copper alloy may be used instead.

⁷For example, in the Brillouin cell mentioned in the introduction the stainless-steel flanges have a "ring and groove" of 2.54-cm i.d. and a square cross section of 1.6×1.6 mm. Six evenly spaced stainless-steel allen head bolts are used to clamp the flanges.

⁸The indium O-ring used here and in the other seals described in this article is made by overlapping the beveled ends (20° bevel) of a suitable length of 0.76-mm-diam indium wire.

⁹To aid in separating the flanges, some people provide a slot between the flanges to permit a screwdriver blade to be used to pry them apart. Still others coat the flanges with silicone grease to allow easier separation. I feel that grease, in general, will degrade the bond and should be avoided. A better solution is to make two extra threaded holes diametrically opposite each other on one flange and use bolts to push the flanges apart. These holes should be located in such a way that there are no holes in line with them on the other flange. A note of caution: these extra holes should be on the same bolt circle as the clamping bolts to make sure they are outside of the "ring and groove!"

¹⁰The number of bolts required for any particular flange seal will depend on the type of bolts used and the diameter of the flange. For small flanges (e.g., the Raman tube seal flange in Sec. IV) a 2-56 bolt is suitable; for larger flanges (2.5 to 12.5 cm in diameter) a 4-40 bolt would be better.

Flanges larger than 12.5 cm may require 6-32 bolts. Bolts with the finer threads allow a more gradual clamping of the flanges than the coarser ones but the finer threaded bolts are weaker. The 2-56 and 4-40 bolts are more prone to "thread stripping," "head shearing," and "hexagonal socket rounding" than the 6-32 ones. But these smaller bolts take up much less space on a flange. For convenience, use an even number of bolts; they should be equally spaced on the bolt circle with a spacing of 1.5 to 2.5 cm. Where there is a pressure requirement, the number and type of bolts should meet this requirement first.

11 The criss-cross pattern is used in many situations where a number of bolts have to be tightened, e.g., on the wheel of a car; this pattern ensures even tightening. For convenience let us assume that there are an even number of bolts and that these have been already finger tightened. Using an allen wrench or a screwdriver, whichever is appropriate, tighten a bolt by turning it a small amount, e.g., a quarter to half a turn. Next tighten the bolt diametrically opposite to the first one by the same amount. Proceed then to the bolt adjacent to the first one and repeat the tightening; it does not matter which one is chosen. Next proceed to tighten the bolt opposite to this one. Continue in this manner by tightening alternately bolts on opposite sides of the flange until all the bolts are tight; For a vacuum chamber this would be sufficient but for a pressure cell the bolts may have to be

tightened further to ensure that the seal is not broken by the flanges moving under the pressure differential. To obtain an even compression make sure that the tightening proceeds in one direction always, say clockwise.

¹²Esco Products, Inc., 171 Oak Ridge Road, Oak Ridge, NJ 07438.

¹³Many indium seals for windows are made to one or both faces of the optical flat. To improve the bond between the window and the indium O-ring, some experimenters have deposited indium, nichrome, or platinum on the face either by vacuum deposition or by "flashing" it on.

¹⁴I. R. Dagg et al., Can. J. Phys. 63, 625 (1985). A seal based on the same principle but of a somewhat more complicated design and for use at a much higher pressure has been described by P. Mazzinghi and M. Zoppi, Rev. Sci. Instrum. 54, 1585 (1983).

15 In the case of the sapphire windows, the removal of the sapphire flats without cracking them proved to be a problem at first; however, they can be removed easily if the Brillouin cell is gently warmed with an air heat gun to the point where the indium softens and the window then blown out by nitrogen gas pressure applied through the filling tube union; the outer flange for each window should be removed before each flat is blown out after which the hole is blocked with a rubber stopper to permit the other flats to be similarly removed.

¹⁶B. M. Abraham and C. M. Falco, Rev. Sci. Instrum. 47, 253 (1976).