NOTES

BRIEF contributions in any field of instrumentation or technique within the scope of the journal should be submitted for this section. Contributions should in general not exceed 1000 words.

Reusable pressure seal for low temperature use requiring a small annular space

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A reusable, low-temperature, super leaktight joint sealed with epoxy is presented which increases the available working area over that provided by a coventional bolted O-ring system. The seal is usable at arbitrarily low temperatures and was tested with a pressure of 70 bar with no apparent deterioration in performance. The simple method of assembly and disassembly is described and the results of various tests are discussed.

Reusable pressure seals have generally consisted of either rubber O-rings, solder, soft metal (Indium) seals, or medium hard metal (gold or copper) seals. Choice of seal depends on specific needs.[1] Obviously, rubber cannot be used at low temperatures where it is no longer elastomeric, but it is easier and cheaper to make and break than the metal seals. Soft metal seals cannot be used for high pressure and medium-hard metal seals are both expensive and difficult to fabricate. All of these seals require some manner of compressing the sealing flanges using screws or clips, placing size and/or geometry constraints on the system. Presented here is a method of constructing a super leaktight seal that maximizes available working space, is usable down to arbitrarily low temperatures, and can withstand relatively high pressure.

This design was motivated by a solid ^{3}He experiment consisting of an ultrasound/NMR cell contained in a silver pressurizable housing, itself contained in a superconducting magnet. This placed restrictions on the outside diameter, the wall thickness of the cell, and materials used for the seal. Capillary fill lines, electrical leads, and optical fibers also had to be installed in the cell and routed outside while maintaining a super leaktight seal which could withstand 35 bar while being resealable and reusable. The delicate nature of the cell eliminated the possibility of using solder as a sealant. The first inclination was to use a gold O-ring seal: although expensive and difficult to make, they are often used at low temperatures when superconducting materials should be avoided. A conventional pair of flanges with appropriate screws on the perimeter is used to compress the gold and make the seal [see Fig. 1(a)]. This requires a certain annular space in order to use a screw size that is adequate to withstand the pressure and provide a proper seal. This method of sealing is also not reusable unless flanges are made of hard materials such as berylium copper, since the gold deforms the surface of softer (silver or copper) flanges when compressed.

The screw-type cell with epoxy seal shown in Fig. 1(b) greatly reduces the annular space needed for the seal. It can withstand pressures exceeding 1000 psi for a copper container of the dimensions shown, it remains leaktight to arbitrarily low tempertures, and it can be reused and resealed with a turnaround time of one day. The cell itself consists of a threaded container and an associated threaded cap. The cap is designed to form a fairly tight metal-to-metal seal when screwed into the container; a groove cut around the circumference of the cap is then filled with epoxy to form a leaktight seal [Fig. 1(b)]. The epoxy actually seals the cell, while the threads are used to provide the strength needed to withstand pressure. The cell tested was constructed of copper, with 3/8 in.-24 threads. A .06 in. $\times$ .06 in. notch was cut around the circumference of the cap leaving enough copper above the threads to provide a metal-to-metal seal with the cell. The body was made in a similar way with the threads being cut to correspond with the cap but with the notch being omitted. The cell was then cleaned with trichloroethylene, acetone, and methanol to remove any oils or solvents left from the machining. This is very important to ensure that the epoxy forms a leaktight seal. Before assembling the cell it is also advisable to lubricate the threads. Oil of some kind can be used if it does not create a problem in the cell; but the groove must be cleaned again to remove any excess oil before applying epoxy. In our particular case, to avoid oil contamination, dry graphite was used. If no form of lubrication is used, the "dry" copper has a tendency of binding together and can completely hinder removal of the cap. If this occurs, soaking the cell in a low-viscosity lubricant will free the cap, but at the cost of contaminating the cell.

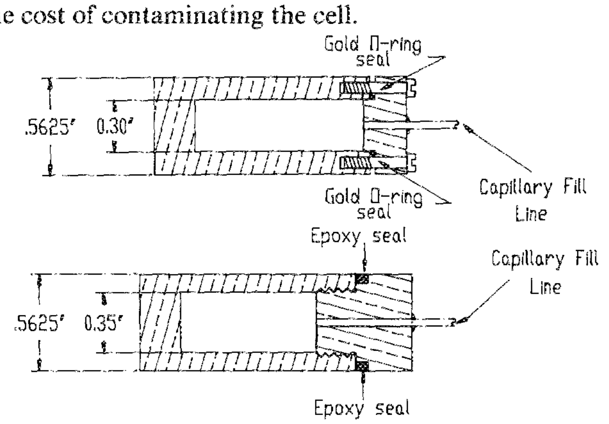

FIG. 1. Two types of vacuum seals: (a) conventional O-ring seal, (b) epoxy seal.

Assembly of the cell is straightforward. With the threads lubricated and the groove clean, simply finger tighten the cap and apply the epoxy to the groove. Emerson and Cumming,[2] 2850 FT was used on our cell because it has an expansion coefficient that closely matches that of copper. Other epoxies could be used, but separation and leakage will occur if the cell is to be heated or cooled and the expansion coefficients are too dissimilar. A low viscosity hardener[2] 24LV was used with the 2850 FT so that the epoxy would flow into the groove easily and fill any voids or irregularities. The cell was then allowed to slowly spin overnight, to insure a uniform seal. After the epoxy hardens, excess epoxy may be machined off if desired, but this is not necessary.

Reuse of the cell is accomplished by removing the epoxy and the cap, cleaning, and resealing. The epoxy can be easily removed using an organic acid, Ecostrip.[2] In order to avoid contamination of the cell, a wick was used (Fig. 2) to apply the Ecostrip to the epoxy seal. This avoided immersion of the cell, possibly exposing its interior to the solvent. The wick used was a piece of fiberglass cloth wrapped around the cell and was secured with a copper clip. The ends were then placed in a small amount of Ecostrip. This setup supplies a continuous source of solvent and provides a wicking action that actually removes the epoxy from the groove. For our cell, total removal required an overnight sitting using about 5 cm^3 of Ecostrip. The cap can then be easily removed and the cleaning process repeated in preparation for resealing.

The conventional cells which motivated this design used a gold O-ring compressed by eight screws [see Fig. 1(a)]. No combination of gold thickness or screw size proved satisfactory for our purpose. Larger screws limited the diameter of the gold wire and weakened the walls of the cell and smaller screws compromised the strength of the joint. The screw-type cell, on the other hand, has been successfully tested under various extreme conditions. Initially it was pres-

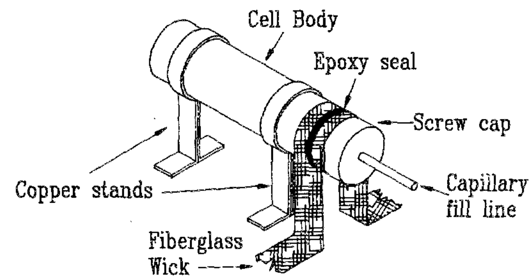

FIG. 2. Apparatus for removal of epoxy. The entire apparatus sits in a tray holding a small amount of Ecostrip, and is covered with a large beaker to reduce evaporation.

sure tested to 1000 psi at 300 K and then leak checked with 550 psi of ^{4}He. The cell was then cooled to 77 K and again leak checked to 550 psi of ^{4}He. Additional similar tests were made after disassembling and reassembling the seal three times using the techniques described above. Then, the cell was immersed in superfluid ^{4}He and found to be leak tight when evacuated. Finally, the cell was opened, resealed and again found to be leak tight in superfluid ^{4}He.

It is hoped that this note will enable the reader to use the simple techniques described here for achieving a low temperature, high pressure seal that allows certain freedoms in designing the parameters for an experiment. Although we merely mention the particular constructional parameters used for our own cell, we can easily foresee many varieties of applications for which this technique could be used.

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[1]See for example: G. K. White, *Experimental Techniques in Low Temperature Physics* (Oxford University, Oxford, 1959); R. C. Richardson and E. N. Smith, *Experimental Techniques in Condensed Matter Physics at Low Temperatures* (Addison–Wesley, New York, 1988).
[2]2850 FT, 24LV, and Ecostrip are trademarks of Emerson and Cumming, Canton, MA 02021.