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An Inexpensive Target Cell for the Study of Liquids and Gases by X-Ray Scattering*

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IN this note we shall describe a new target cell which has been constructed and used in the study of liquids and gases by x-ray scattering. The cell we shall describe is primarily for use at temperatures at and below 77 K although it could be used at room temperature as well.

An ideal target cell to study liquids and gases by x-ray scattering should serve to contain the sample but provide no scattering itself. Because of its low atomic number, beryllium is an ideal substance to use in such a chamber. Obtaining beryllium in thin walled cylinders is a difficult and expensive procedure. The target cell we shall describe here is extremely inexpensive and for certain applications nearly as good as beryllium. Another advantage of the design we shall present is that it is demountable.

We have made target cell bodies from both Mylar and Kapton.¹ The choice depends both on the experiment to be done and on the scattering properties of the materials. Scatter of Cu K_α x rays from thin films of these materials shows pronounced structure in certain angular ranges (see Figs. 1 and 2). Although Kapton is less subject to catastrophic failure and radiation damage than is Mylar, its pronounced scattering (especially at small angles) makes it somewhat less useful as a construction material. Mylar targets have been used extensively by the author in small angle scattering studies of superfluid helium.^{2,3}

The basic design used here is patterned after one given by Bearden *et al.*,⁴ but differs from it in several important aspects. A completed target chamber consists of a Mylar (for example) cylindrical shell which is seamed down one

FIG. 2. Transmitted intensity for Kapton. Again, we have eight $25\ \mu$ thick sheets and one step = 0.049° . The intensity of Figs. 1 and 2 can be compared to see the relative intensity.

side. The ends are sealed by the use of nylon plugs—one end is sealed completely while the other threads onto an appropriate target mount which has an access hole to facilitate filling of the target. Figure 3 shows a completed target chamber.

The addition of brass struts increases the reliability of the target cell during the course of an experiment. They prevent the target from collapsing should a reverse pressure accidentally be applied to the cell.

For applications below 77 K the target mount is made from copper. The nylon-copper joint provides a vacuum tight seal⁵ at temperatures below 77 K. This seal is brought about by the difference in expansion coefficients of these materials and is superfluid tight below 2.18 K. The joint need only be finger tip tight at room temperature. The target mount can be attached to a holder or refrigerator using an indium seal.²

At low temperatures the use of aluminized Mylar (aluminum coating $\approx 750\ \text{\AA}$) in place of uncoated Mylar aids in reducing the radiative heat transfer between the target and the cryostat walls.⁶

Construction begins with a rectangular sheet of aluminized Mylar (for example) $25.4\ \mu$ thick. The aluminum coating is stripped back about 3 mm from three edges by painting on a 0.5M solution of NaOH. The Mylar is then washed in distilled water. After drying, the edges which will come in contact with the epoxy⁷ are roughened with No. 600 emery cloth.

The Mylar cylinder is formed with the aid of a brass mandrel. The mandrel is first wrapped with $12.7\ \mu$ thick Mylar whose surface has been coated with a nonstick film⁸ to ensure that the epoxy will not adhere to it. We find this procedure to be much easier and more reliable than the slotted mandrel approach used in Ref. 1. The

FIG. 1. Transmitted intensity as a function of scattering angle for Cu K_α x rays scattered from a foil made from eight sheets of $25\ \mu$ thick Mylar. One step corresponds to a scattering angle of 0.049° . The structure-free region from 2 to 10° makes Mylar a useful construction material for small angle scattering work.

FIG. 3. This is a close to scale representation of a 16 mm diam target cell. The various parts as described in the text can be seen in their proper positions. The refrigerator base and centering hole are specific to previous work by the author.³ It should be pointed out that thermal gradients can be greatly reduced by including copper foil in the construction (remembering, of course, the path of the beam).

Mylar rectangle is next wrapped about the covered mandrel and a seam made with epoxy along the 1 mm of overlap. The cylinder is then again wrapped with 12.7 μ coated Mylar to ensure that the seam is uniform and flat. At this point, the partially constructed target chamber is cured.

After cure, the external coated 12.7 μ Mylar is removed exposing the seamed cylinder. The base core sheet of coated Mylar is next slipped from the mandrel along with the seamed cylinder. The coated core sheet is given a half-twist from the ends and the seamed cylinder pops free and slips off the coated core sheet. The seamed cylinder is examined for uniformity and creases.

It is now ready for the nylon end pieces. The nylon collars (Fig. 3) are slipped over the cylinder and, using the same epoxy liberally, the end caps are inserted. Using more epoxy, the collars are pushed into place over the end caps so that the Mylar cylinder is between the end caps and the collars. The collars are turned so the slots line up and the brass strengthening struts are set in place—again with the same epoxy. The target is now completed and ready for the oven.

The entire target is now wrapped with 12.7 μ Mylar and inserted into a large brass tube. This ensures that the completed cylinder with end caps has axial symmetry. This entire unit is now cured. After cure, the completed

target chamber slips from the brass tube and the thin, coated Mylar sheet which has kept the target from adhering to the brass tube unwraps easily.

A testing apparatus which allowed visual observation of the target under various conditions of internal pressure near 4 K was also constructed. Using this apparatus the vacuum integrity of the target cell and superfluid seal was established. No cell of this design has ever shown a leak either in the test rig or in the experimental cryostat. The brass struts allow reverse pressures of an atmosphere with no adverse effects. One case of a 16 mm Kapton cell is worth mentioning. Reverse pressures up to 60 Torr showed no visible denting of the cell. Pressures above 100 Torr or so resulted in a "caving in" of the sides, but no creasing. Returning the pressure differential between the inside and outside of the target to zero slowly caused the target to "pop" back to its cylindrical shape when the reverse pressure had fallen to about 50 Torr. Attempts to rupture this cell and others made from Mylar by increasing the internal pressure from vacuum (zero differential pressure) to 760 Torr in less than 1 sec failed every time. In short, these cells have been shown to be mechanically reliable and tolerant to misuse.

We have made target cells 5 cm long and 9.6, 16, and 25.4 mm in diameter with equal success. Several precautions will guarantee reliable results. The Mylar must be free from pinholes. Since Mylar is porous to helium gas at room temperature, it can only be leak tested with helium gas at 77 K or below. Thus, the Mylar should be carefully inspected at the beginning of construction. Secondly, the epoxy must be allowed to cure properly. Finally, the "step cut" in the nylon end plug which seals to the copper target mount must be sharp and free from burrs and scratches. This is of particular importance in low temperature applications.

We would like to thank T. von Hooydonk for suggesting the mould release and other technical assistance.

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¹ Both Mylar and Kapton are registered trademarks of the E. I. du Pont de Nemours Company.

² R. B. Hallock, Ph.D. thesis, Stanford University, Stanford, Calif. (1969) (University Microfilms, Ann Arbor, Mich.).

³ R. B. Hallock, Phys. Rev. Lett. **23**, 830 (1969).

⁴ T. H. Moss, C. F. Kellers, and A. J. Bearden, Rev. Sci. Instrum. **34**, 1267 (1964).

⁵ This method of sealing has been used before. See, for example, H. D. Cohen, Ph.D. thesis, Stanford University (1966) (University Microfilms, Ann Arbor, Mich.).

⁶ One can also obtain Kapton with an aluminum coating.

⁷ We have found several epoxies to be useful at low temperatures: Hysol adhesive R8-2038 and Hysol hardener H8L-3466 (Hysol Division, The Dexter Corp., Olean, N. Y.); Narmco base resin 3135 and Narmco curing agent 7111 (Narmco Materials Division of the Whittaker Corp., 600 Victoria St., Costa Mesa, Calif.).

⁸ RF-300 mould release available from Resin Formulators Co. is most useful in coating the core sheet as it leaves no particulate residue. Fluoro-glide,⁴ available from Chemplast, Inc., 150 Day Rd., Wayne, N. J., works well but tends to leave a Teflon powder residue.