A new sample cell for neutron scattering at high and low temperatures

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We describe a simple, inexpensive sample container for neutron scattering at nonambient temperatures. The sample cell works equally well with reactive glasses, crystalline powders, or high vapor pressure liquids. Demonstrated performance has been obtained at temperatures from 10 K to 350 °C.

We present in this communication the design of a new sample container to be used for neutron scattering at nonambient temperatures. The sample cell can be used with reactive glasses, crystalline powders, and high vapor pressure liquids. In its present form, the container is intended for use with samples that will not attack vanadium or stainless steels. Demonstrated performance over the temperature range 10–623 K has been obtained. The design accommodates either standard 10% scattering samples or highly absorbing samples involving lithium-6, cadmium, or boron.

The so-called "sample can problem" has been a continuing source of concern at Argonne National Laboratory's Intense Pulsed Neutron Source (PNS). The problem is one shared by other steady-state and pulsed-source neutron facilities. Many of the instruments at the IPNS require a cylindrical sample geometry, 1/4–1/2-in. diameter and 1/2–2-in. long. Thin-wall vanadium tubing is the material of choice. What has been needed is a vacuum-tight, inexpensive, commercially available assembly that can hold reactive solids and liquids and that can be used above and below room temperature. The sample container should have a demountable closure so that it can be reused.

Sample cell designs that incorporate elastomer O rings fail at low temperatures. Therefore, we have machined grooved miniflanges out of vanadium, aluminum, and stainless steel that use indium as the O-ring material. Unfortunately, we have not been able to make reliable electron-beam welded joints between thin-wall vanadium tubes and any of the metal flanges (including the vanadium flanges). The beam-welded joints invariably leak after thermal cycling. Therefore, we have resorted to bonding the vanadium tubes to the flanges with a flexible epoxy cement. These procedures are costly and time consuming. Moreover, the seals will not withstand elevated temperatures. Such assemblies are useful only for solids at low temperatures or for noncorrosive liquids at room temperature. The cans are not only expensive to fabricate, but their utility is constrained by the chemical reactivity and physical properties of epoxy and indium. The design presented below obviates all of these difficulties and meets the requirements stated earlier.

High-purity vanadium tubes with one end closed (COE) are purchased[1] in 1/4-, 3/8-, and 1/2-in. diameters. The seamless tubes have been deep drawn to a 0.006-in. wall thickness from high-purity, electron-beam consolidated vanadium;[2] and they are 2.0–2.5-in. long. It is convenient to purchase the tubes in the longest anticipated lengths and then to cut them as required using a wire spark saw. Specifications for the 3/8-in.-diam tube are 0.375 ± 0.004-in. o.d. with a 0.006 ± 0.002-in. wall thickness and RMS 63 surface finish. The tubes are free of oxide inclusions, and they have a helium-leak-rate porosity $< 5 \times 10^{-11}$ atm cc/s.

The most direct way to close the open end of a COE vanadium tube would be to use an ordinary compression fitting to form a hermetically sealed, cylindrical sample cell. However, commercially available compression fittings will crush thin-wall vanadium tubing when the fitting is torqued enough to form a vacuum-tight seal. The key to a successful connection is the use of an internal sleeve to support the vanadium wall (see Fig. 1). The tube fitting is a standard 316 stainless-steel Swagelock[3] cap and nut with 316 stainless-steel front and back ferrules. The reinforcing sleeve is a commercially available Cajon[4] XOA adapter (304 stainless steel) that is intended for a completely different purpose. The outer diameter of the sleeve is turned down a few thousandths of an inch so that it just slips into the vanadium tube. The tube seats against the collar of the sleeve. The compression fitting is made up the first time by tightening the nut approx. one turn (not the 1-1/4 turns recommended by the manufacturer for standard wall-thickness tubing). Subsequently, the cap can be removed and reliably resealed with only a slight additional rotation. Samples are routinely loaded in a helium-gas atmosphere for subsequent rapid thermal equilibration.

A blind 5-40 tapped hole in the cap is used to fasten the sample can to a heat sink or for suspending it in an oven. The top of the cap is lightly coated with thermal joint compound.[5] Silicone grease works well if only low-temperature measurements are to be made. All nonambient temperature measurements are carried out in an evacuated sample-chamber environemnt.

The new sample assembly described here has been cycled repeatedly to 77 K in liquid nitrogen and to 350 °C in an oven. The Swagelock cap has been sealed and resealed with no loss in integrity (as verified by helium-leak tests). The container has been used at 10 K, but cycle tests to determine lifetime have not been done at this temperature. It is expected that no long-term problems will arise at liquid-helium temperature based on the successful tests at liquid-nitrogen temperature.

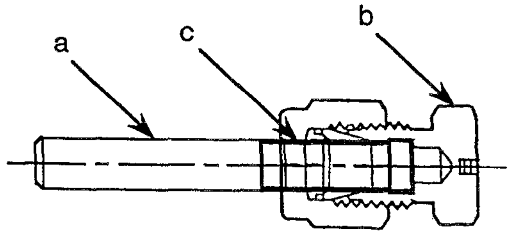

FIG. 1. Schematic outline of the sample cell assembly. (a) Thin-wall vanadium tube. (b) Swagelock cap. (c) Cajon internal reinforcing sleeve.

For high-temperature measurements, the stainless-steel front ferrule is lightly sprayed with a solid film lubricant[6] and then burnished with a soft cloth. The male threads on the cap are treated in a similar manner. This assembly can most probably be run above 350 °C, but the upper temperature limit has not been explored.

For nonambient temperature measurements, stainless-steel front and back ferrules are used. For room-temperature measurements, a Teflon front ferrule can be used if desired. The Swagelock fitting will hold pressures well above the working pressures of the thin-wall vanadium tubing. Obviously, the maximum pressure for the container assembly is a function of the vanadium tube wall thickness and the operating temperature.

For neutron scattering measurements with solid samples that have a high absorption cross section, it is convenient to achieve the "ideal 10% scattering sample" by inserting a smaller diameter vanadium tube into the container and then filling the annulus with the powdered crystal or glassy specimen.

Other thin-wall tubing such as aluminum or titanium/zirconium "null matrix" alloy can be considered within the framework of the design presented here. Also, Swagelock fittings and ferrules of other metals with differing chemical and thermal properties are commercially available. Several of these combinations are under investigation.

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[1] B-J Scientific Products, Inc., 1240 S. W. Alandale Avenue, Albany, Oregon, USA.

[2] Teledyne Wah Chang Albany, P. O. Box 460, Albany, Oregon, USA. Typical oxygen contents are 150–250 $\mu g/g$.

[3] Swagelock Co., Solon Ohio, USA. For a 3/8-in. o.d. tube, Cat. No. SS-600-C.

[4] Cajon Company, 9760 Shepard Road, Macedonia, Ohio, USA. For a 3/8-in. o.d. tube, Cat. No. 304-6-XOA.

[5] Wakefield Engineering, Inc., Wakefield, Massachusetts, USA.

[6] Lubri-Bond A (a molybdenum disulphide/graphite aerosol). E/M Corporation, P. O. Box 2400, West Lafayette, Indiana, USA.