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TRITIUM/DEUTERIUM GAS CELL

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ABSTRACT

A tritium/deuterium gas cell has been designed as a neutron target to generate a high-energy "neutron source", $15 \leq E_n \leq 30$ MeV, when bombarded with deuterons up to 12 MeV. The secondary neutrons emitted at 0° from the reactions ${}^2\text{H}(d,n){}^3\text{He}$ and ${}^3\text{H}(d,n){}^4\text{He}$ are used to study neutron scattering differential cross sections from materials of interest. The resolution needed for these measurements required a monoenergetic neutron beam with an energy spread $\Delta E \sim 100$ keV. This is not possible to achieve at the same source strength with a standard tritium/deuterium loaded solid target, because of the large differential energy spread of the incident deuterons through the target (about 160 keV compared to 30 keV in the gas cell filled to 1 atm.). The present gas cell, 3 cm long and with a 7 μm Mo window, produces 9.3×10^7 or 2.4×10^7 neutrons when filled with one atmosphere of deuterium or tritium respectively, for 1.0 μA deuteron current.

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TRITIUM/DEUTERIUM GAS CELL

The gas cell is a stainless steel (SS-316) tube, 0.95 cm ID, 3 cm long, with 0.038 cm (0.015") wall thickness. The end of the cell, designed to stop up to 20 MeV deuterons, is a 0.051 cm (0.020") thick gold cap, which is laser welded to the cell. The gas window is at the present time a Mo foil about 7.6 microns thick (0.00030"). A discussion on the preparation of this foil and Havar foils used in the past will follow.

The gas cell is sealed on the vacuum end of the cell by the entrance foil. The outer edge of the foil, 0.064 cm wide and appreciably thicker, 250 microns (0.010") than the center of the foil seats on an indium gasket that rests on a ledge. The sealing of the entrance foil is achieved in four steps. 1) The first indium gasket is placed in the barrel and positioned on the ledge. 2) The foil is positioned on the gasket to form the gas tight seal for the cell. 3) A second indium gasket is placed on the rim of the foil to form the vacuum seal. 4) The piston P is placed in the barrel compressing the gaskets, (0.041 cm thick, 0.953 cm OD, 0.792 cm ID) against the ledge, foil and piston. (See Fig. 1.) A compression nut fastens the barrel of the cell to the beam tube shown in Fig. 2. The gas cell is electrically isolated from the beam tube by a NEMA G10 threaded piece positioned at the end of the tube.

The cells are filled with up to two atomspheres of gas. (They have been tested up to four atms.) The filling line is a SS-304 tube, 0.10 cm OD, 0.036 cm ID, and 35 cm long connected to a steel needle/bellows valve (Nupro SS-2H) mounted on the Rotator Plate to be discussed later. Because of safety requirements at LLNL, the T cell filling line is attached to two valves in series, while only one valve is used for the D cell. The pressure in the cells is measured at the valves assemblies using mini-transducers (Statham

Pressure Transducer, Model PA 822-170) coupled to Doric digital readers (S/N 31021) calibrated between 0.0 and 60 psi.

In order to minimize the presence of impurities in the gas, such as ^3He from the tritium β decay, and C, N, O, commonly found in the gas from commercially purchased gas bottles, a Uranium-Tritide furnace (UTF) is used, where the tritium is stored as a compound, uranium tritide, UT. Similarly, a Uranium-Deuteride furnace is used when filling the deuterium cell. Both furnaces, the UTF and the UDF have been build at Lawrence Berkeley Laboratory.³ Each cell has 18 g of U and can absorb as much as 3 liters of tritium (or deuterium respectively). In order to release the T or D gas the UTF (or UDF) must be heated up to 350°C.

BEAM TUBES

The beam tube is a conical stainless steel tube, 30.5 cm long, 2.54 cm base diameter tapering to 1.27 cm diameter at the end meeting the NEMAG10 insulator on the gas cell. A Ta collimator, 0.272 cm ID and a negative 300 volt bias ring are inserted in the beam tube immediately behind the gas cell. Both, the Ta collimator and the bias ring (see Fig. 2) are electrically isolated from the tube by a Nylatron housing. The present configuration has two parallel beam tubes mounted diametrically opposite to each other on a rotating plate, allowing gas-in/gas-out measurements to be carried out without the need to empty the filled gas cell. An alignment collimator 0.792 cm OD, 0.635 cm ID, immediately behind each beam tube has been installed in the Rotator Plate to assure that the tubes are accurately positioned into the beam line after rotation of the plate. The plate has also two port windows, 1.25 cm diameter, at 90° from the cells and at the same radial distance. These windows, a quartz and a pyrex one, allow alignment of the collimators ahead of the cells in the beam line of the accelerator, and focusing of the beam (quartz) before entering the gas cell. Windows and gas cells are successively positioned into the path of the beam by rotation of the plate. Rotation is achieved under vacuum conditions $\sim 4 \times 10^{-6}$ Torr, using a 200 step DC motor controlled by a stepping motor power supply.

ENTRANCE FOILS

In the choice of materials to be used as a thin entrance window to the hydrogen gas cell, the two most serious problems are:

a) The material must not suffer hydrogen embrittlement. This requirement eliminates a large number of metal foils, Ta, Pt, etc., transition metals in general, which otherwise would be very attractive because of their strength and heat desipation.

b) The material must be a good heat conductor to diffuse the heat developed at the beam spot, and it must have mechanical strength to endure alternate heating and cooling cycles. This last requirement eliminates good heating conducting materials such as Au, Cu, Ni, Fe, etc., which are too soft to tolerate the mechanical changes as a result of the temperature changes under relative intense beams ($>1 \mu\text{A}$).

Molybdenum, tungsten and compounds such as Havar have been successfully tried.¹ Tungsten, although an excellent choice because of its properties, cannot be obtained free of defects when very thin foils ($\leq 0.0007 \text{ cm}$) are required. In addition to the Mo foil presently being used for the gas window, Havar foils have been used in the past with only partial success. The main problem with Havar has been the lack of reproducibility of the method used to mount the entrance foil so that a leak-tight gas cell is not obtained routinely.

Havar Window: The Havar foils $6.3 \mu\text{m}$ thick were sandwiched between two In gaskets (0.025 cm thick, 0.952 cm OD, 0.722 cm ID) when compressed against the ledge in order to seal the cell. The first In gasket was positioned on the ledge followed by the Havar foil and the second In gasket. The cell was assembled by compressing the In-Havar-In layers against the ledge with relatively moderate compression. The rate of success in obtaining a tight

cell was less than 20%. Variation in the thicknesses and widths of the In gasket, in the use of different epoxies and other sealants (Epoxi Patch #0151, Hardman Epoxy, Double Bubble, Hardman Urethane, etc.) to attach the foil to the piston or to the ledge, failed to improve the success rate.

It became clear that the source of the leaks was the radial deformation of the Havar. Radial striations developed on the Havar foil as a result of the indium gaskets being pushed toward the center of the circular opening when compressed by the piston.

To minimize this effect, a Cu ring of equal dimensions to the In ring was electro plated to the Havar on the side facing the inside of the gas cell. It was assumed that the increased rigidity of the Havar foil would preclude radial deformation. This approach, however, was unsuccessful. Similarly, a laser beam welding of the Cu ring onto the Havar did not succeed either. In both cases the Cu did not adhere uniformly to the Havar over the whole area of contact.

The next approach was to replace the Cu ring by a SS-316 ring and furnace braze to the Havar foil. The brazing method succeeded in giving a leak free foil, but due to the high temperature used in the process, foil embrittlement took place. The gas cell leaked slowly during the beam on periods and the leak stopped with the beam off.

Molybdenum Window: The Mo entrance foil has proven the most successful of all the different approaches. Starting with a rather thick, 0.013-0.025 cm Mo foil, 0.952 cm diameter, its central region, 0.825 cm diameter is chemically etched^{2,3} (45% HNO₃, 5% H₂SO₄) to a thickness of around $7.0 \pm .6 \mu\text{m}$ (see sketch 6). The assembly of the cell proceeds as described earlier, by sandwiching the foil between the two In gaskets which this time press onto the thicker edge of the foil preventing its wrinkling under compression.

SUMMARY

After several trials with different materials for the entrance window of the gas cell (tungsten, Havar and molybdenum) as well as different schemes for mounting the foils we have succeeded in obtaining a leak free gas cell. A molybdenum foil chemically etched to reach the desired thickness and mounted on the cell between two indium gaskets has consistently resulted in a tight gas cell. The cell filled up to two atmospheres of deuterium gas remains tight under bombardment with a deuterium beam of intensity as high as $2\mu\text{A}$.

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FIGURE CAPTIONS

- Fig. 1 Layout of the gas cell assembly and piston compressing the molybdenum window on the cell.
- Fig. 2 Enlarged view of the end section of the beam tube where the cell is mounted.

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Figure 1

Figure 2