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AN ELECTRONIC BALANCE FOR WEIGHING FOAMS AT CRYOGENIC TEMPERATURES\*

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ABSTRACT

A commercial electronic balance was altered to weigh objects in a cryogenic environment containing combustible fluids. The balance was used to measure the mass gain of open cellular foams touching the surface of liquid hydrogen. The mass gain rate is a function of the foam wicking characteristics (a function of the foam structure). Weighing the empty and liquid filled foam sample in the ullage above the liquid, and the foam submerged in the liquid, gives sufficient information to allow a free volume or porosity to be determined for the foam. These tests are especially important for foams too weak to withstand the surface tension forces of ambient temperature liquids. Details of the design of the cryogenic balance and some results for several types of foams are presented in this paper.

INTRODUCTION

A desired fuel pellet for the inertial confinement fusion (ICF) research experiment is a hollow spherical shell of liquid fuel.<sup>1</sup> An outer hollow spherical shell contains the liquid fuel, but in spheres of the desired size the liquid slumps to the bottom of the shell in 1-g gravity. A method being investigated to support a thin uniform layer within the outer shell consists of lining the container with a rigid foam matrix. Liquid condensed from an initial gas fill or added to the cold target through a tube fills the foam and is held in it by surface tension. Characteristics of the foam important for this use include small pores that will readily wick low surface tension cryogenic fluids, the ability to survive low temperatures, a high liquid capacity compared to the foam volume and easy machinability that allows fabricating to the close tolerances required in this application.

APPARATUS

We investigated the low temperature characteristics of foams in liquid hydrogen or liquid deuterium by suspending them from a modified

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electronic balance. Weighing the foam samples in low temperature environments allowed us to measure the wicking characteristics with the two hydrogen isotopes and determine the mass of liquid held by a known volume of foam.

The electronic balance, shown in figure 1, was constructed to weigh the foam samples in liquid hydrogen. The electronic balance sensing unit was sealed into a chamber maintained at ambient temperature that communicated with the hydrogen in the cryostat ullage volume via a vertical tube. The balance mechanism is from a commercially available balance capable of weighing 200 g to 0.001 g. The electronics associated with the balance were separated from the sensing unit and installed in a purged box to eliminate ignition sources in the experimental area. An extension cord connected the electronics to the sensing unit. The vertical tube penetrates into the inner test Dewar. The electronic balance was suspended from a water actuated cylinder that could move the balance mechanism vertically. Moving the balance allowed us to weigh the foam in the ullage without a liquid fill, with one end of the foam sample touching the liquid surface and wicking liquid and with the sample submerged in the liquid and full in the ullage. From these measurements the wicking rate, the quantity of liquid held by the foam and the foam material density could be calculated. Liquid surface tension influenced the submerged weight unless the sample mechanism D was completely submerged to above the hook at C in figure 1.

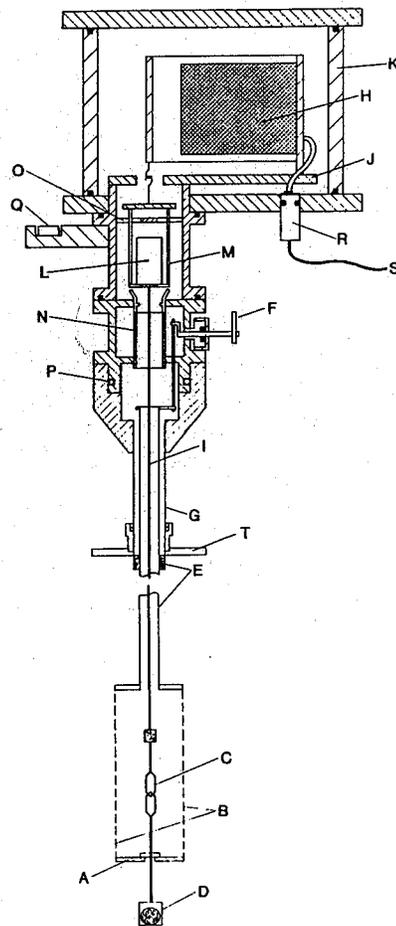


Fig. 1. The electronic balance sensing unit modified for use in liquid hydrogen.

In figure 1, the foam and cage D are shown lifted from the hook C allowing the balance to be zeroed. The foam is raised by a pneumatic cylinder via the bell crank F, the tube E, the chains B, and the washer A. Vertical movement is allowed by the double O-ring slip seal in flange T and the 25 mm O.D. tube G from the electronic balance sensing unit. The sample is suspended from the balance H by a small chain I in the tube E. The balance H is mounted on a cantilevered plate J so that, should gas tight enclosure K flex when pressurized, the leveling of the balance is unaffected. The enclosure is capable of operating with up to one MPa internal pressure. The tungsten reference weight L, used to monitor the balance calibration, is shown placed on its suspension cage M. This cage replaces a section of the suspension I. Some swivel joints in this suspension allows flexing but not rotation,. A pneumatic-cylinder-driven bell crank, similar to F but not shown in the drawing raises N which lifts the reference weight L off the suspension and clamps it against the fixed top support O. A gas tight seal R is provided for the electrical leads S leading to a remote digital readout.

Two adjustments were designed into the balance. The O-ring seal at P, connecting the microbalance assembly to the support tube G, is a gland seal rather than a flange seal so the instrument can be leveled according to the bull's eye level Q while G is held vertical. The flange T is leveled as required to maintain the suspension wire C centered in tube E by moving the mating flange on the cryostat. The mating flange is flexibly connected to the cryostat with a bellows; and is leveled using three threaded standoffs screws.

The lightest foam sample weighed 0.3 grams and there was an inaccuracy of 0.3 % in its mass due to the sensitivity of the balance.

The cryostat used in the foam tests consists of a liquid nitrogen shielded liquid hydrogen container with an internal test container. The internal container can be filled directly with liquid hydrogen or filled with liquid deuterium condensed by the liquid hydrogen in the hydrogen container. Windows in the cryostat permitted lighting, viewing and photographing the foam during the tests.

#### TEST PROCEDURES

The first foams tested were silica aerogel foams.<sup>2,3</sup> These aerogel foams had pores that ranged in size from 5 to 10 nm and a free volume of about 95% of the total volume of the foam. The combination of small pore size that produced relatively large forces from surface tension and of the weak interstitial material caused this foam to shatter into powder when touched by room temperature liquids.

The initial tests were qualitative. The main interest was whether the foam would wick liquid hydrogen and survive, and whether the foam would hold the liquid when it was suspended in the ullage space. Fortunately, the silica based aerogel was clear enough that with a proper back lighting the wicking liquid interface was visible as it moved up the foam sample. Figure 2 is a picture of the liquid interface in an aerogel foam sample 2 cm long and 2 cm in diameter. The liquid deuterium surface is faintly visible near the bottom of the top foam sample, while the wicking liquid interface is near the top of the sample. The wicking interface was horizontal unless one of the holding cage wires was too close to the foam and the liquid wicked up between the wire and the foam. When the liquid did wick up the side, a complex interface formed because liquid wicked in from the side as well as the bottom.

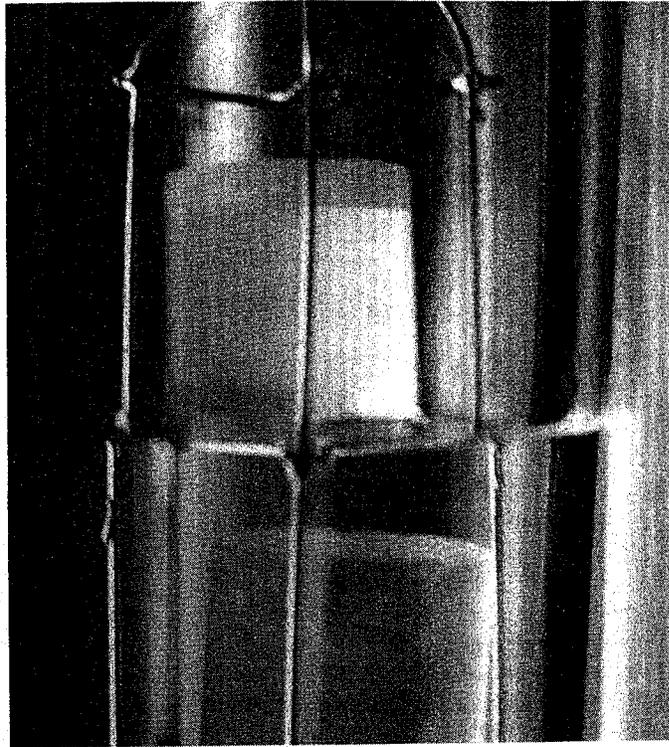


Fig. 2. Silica aerogel foam wicking liquid deuterium. The top sample is nearly full and is opaque. The lower sample is submerged and clear.

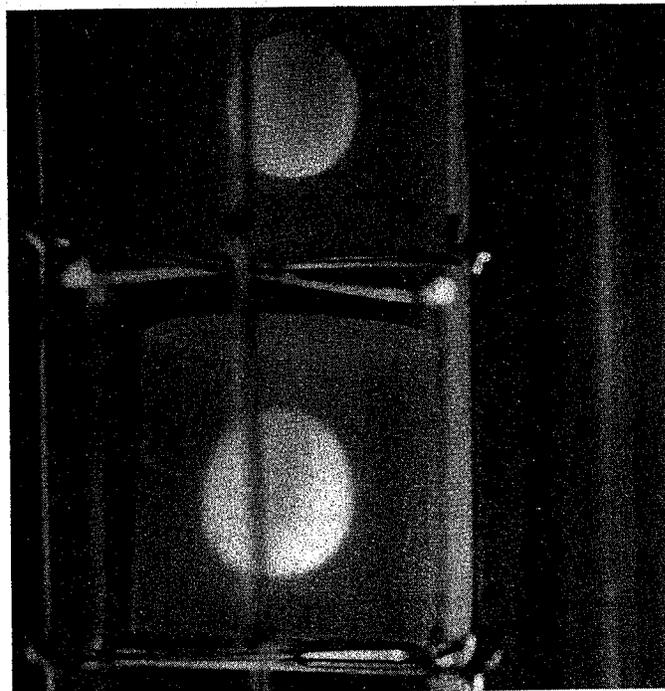


Fig. 3. Silica aerogel foam warming and evaporating the contained liquid deuterium. The white sphere is the remaining deuterium.

When the liquid hydrogen wicking interface reached the top of the sample, just filling with liquid, the sample would remain temporarily opaque. After a short time, the foam would become clear. The top sample in figure 2 shows the opaque quality while the submerged lower sample is clear. The foam remained clear until it warmed and liquid began evaporating. As the liquid evaporates from the sample it first turns white and the edges of the sample become clear while the portion containing liquid remains white and opaque. A photograph of the remaining opaque white spheres is shown in figure 3. We do not believe the remaining sphere is solid deuterium since the temperature and pressure in the test chambers are always above the triple point.

The procedure followed to obtain wicking characteristics of the foams was to fill the cryostat with the test liquid, weigh the sample in the ullage before it picked up any liquid, weigh the sample when the bottom of the sample touched and wicked the liquid, and finally weigh the full sample submerged in the liquid and in the ullage space above the liquid.

### RESULTS

Figure 4 shows some typical wicking data for a variety of foams. These curves are based on weight gain-versus-time measurements made as the foam samples wicked full of liquid. The weight gains were converted to volumes and then to interface heights in the foam, based on the liquid density, the foam's free volume, and the assumption that the wicking liquid interface was horizontal. The curves on figure 4 show interface velocity or slope of the filling curve plotted against the height of the interface. The disparities in the starting or minimum wicking height result from variations in how far the foam sample is initially submerged to begin the wicking test. If the sample is dipped very far into the liquid, time is needed before a horizontal interface

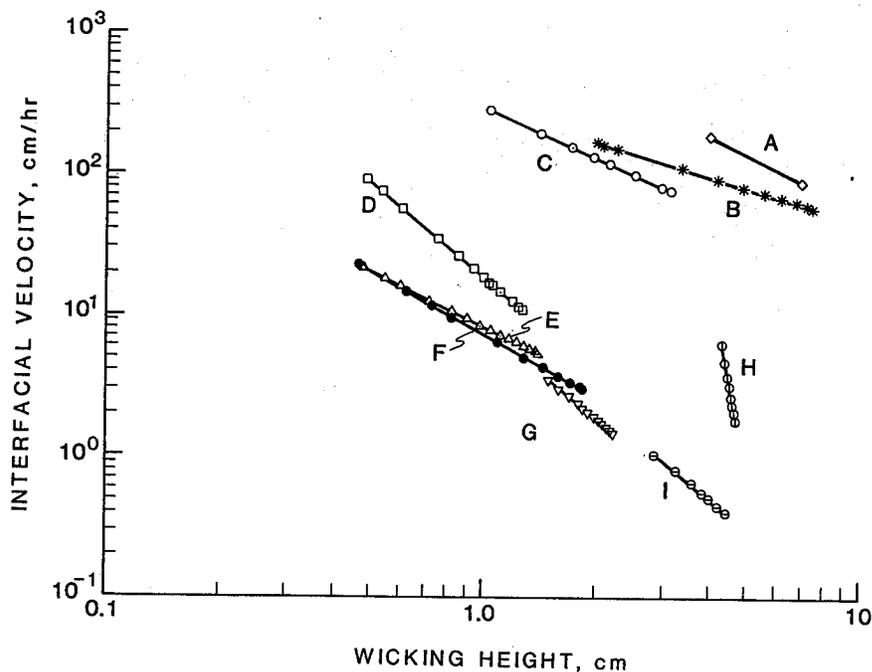


Fig. 4. Liquid interface velocities for various foams.

Table 1. Physical characteristics of the foams shown in figure 4

Foam	Foam Type	Test Fluid	Foam Density mg/cm <sup>3</sup>	Free Volume percent
A	TPX	LH <sub>2</sub>	40	95
B	Carbon	LH <sub>2</sub>	43	90
C	Polystyrene	LH <sub>2</sub>	65	95
D	Resorcinol	LH <sub>2</sub>	111	89
E	Polyethylene	LH <sub>2</sub>	107	89
F	Silica Aerogel	LD <sub>2</sub>	101	95
G	Polyethylene	LH <sub>2</sub>	107	95
H	Polystyrene	LH <sub>2</sub>	65	95
I	Polyethylene	LH <sub>2</sub>	107	89

is established and the wicking proceeds from a fixed area. During this time the slope of the wicking curve changes rapidly depending on how far the sample end is submerged into the liquid. These initial data cannot be readily interpreted and are not included on figure 4. The abrupt change in slope when the sample filled is also not shown by the curves.

Most of the foams tested were manufactured specifically for use in ICF experiments. Thus, every attempt had been made to manufacture a foam with small pore sizes and yet a 90% or higher free volume to hold the maximum liquid. Table 1 shows some of the physical characteristics for the foams tested. The free volume is the fraction expressed as a percentage of the foam filled with liquid, based on the weight of a foam sample full of liquid in the ullage. Foams E, G, and I are the same foams but the curves are for three different tests. The difference in slope between foams E and I is due to the short duration of test E. The wicking rate had probably not yet stabilized from the initial submersion. Curve G represents the wicking rate with the wicking liquid subcooled by slightly pressurizing the ullage with helium.

#### CONCLUSIONS

These measurements show that an electronic balance with a zeroing feature can accurately weigh samples in liquid hydrogen. The balance has been used to measure the liquid hydrogen and liquid deuterium wicking rates into various foams and to demonstrate that the foams retained the liquid while suspended in the ullage above the liquid.

The foams tested in liquid hydrogen display widely varying wicking rates. Most foams, except for one polystyrene sample (H), wick well enough to be used in ICF targets. The aerogel foams with densities less than 100 mg/cm<sup>3</sup> tended to fracture when filled with liquid deuterium but no damage has ever been observed (in the other foams during the low) temperature wicking tests.

#### REFERENCES

1. D. H. Darling and R. A. Sacks, "Wetted foam capsules for direct drive ICF reactor application," Trans. Am. Nucl. Soc., Vol. 52 (1986) p. 257-258.

2. W. J. Schmitt, R. A. Grieger-Block, and T. W. Chapman, "The preparation of acid catalyzed silica aerogel," Chemical Engineering at Supercritical Fluid Conditions, Ed. Michael E. Paulaitis, Ann Arbor Science (1982), p. 445-460.
3. C. W. Price, W. G. Halsey, and S. C. Sanders, "Structural characterization of silica aerogel using specific surface area measurements," Report, Lawrence Livermore National Laboratory, 1985.