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DISCLAIMER

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ABSTRACT

An alumina-filled casting epoxy (trade name Stycast) was found to reliably seal to thin stainless steel edges and 1 mm diameter stainless steel feedthrough pins. All seals retained their integrity after typically 50 cycles from -196°C to 20°C and 10 cycles from -196°C to +100°C. Several chambers were constructed using the epoxy both as a vacuum-tight wall and as an electrical insulator for a large number of multi-kV feedthroughs. The largest of these was 18 cm in diameter and contained 125 feedthrough pins. The chambers were immersed in a freon-11 bath at -105°C, filled with liquid xenon, and tested for the presence of electronegative impurities. After degassing, the contamination levels measured were slightly higher than those we commonly observe for chambers constructed only of glass and metal.

INTRODUCTION

This work demonstrates the possibility of using an alumina-filled casting epoxy (trade name Stycast)[1] for large multi-feedthrough low temperature vacuum seals. Previously, this material had been used successfully for small (3 mm diam) vacuum seals at liquid helium temperatures,[2] and is in common use for similar applications. The primary advantage of epoxy is that it can be easily cast into almost any desired configuration with a minimum of skill and technology. By comparison, the construction of ceramic and glass to metal seals of similar complexity involves considerably more skill, delay, and expense.[3] However, the latter have superior mechanical properties, much lower outgassing rates, and can be baked at 300°C while epoxies cannot be subjected to temperatures much above 150°C.

PRINCIPLES OF CONSTRUCTION

The thermal expansion of Stycast 2850 Ft. in the temperature range from -200°C to 70°C is similar to that of aluminum (see Table I). Above 70°C, however, the thermal expansion of the epoxy is much greater than aluminum. It is thus not surprising that our attempts to plug large (4-12 cm diam) tubes of aluminum, brass and stainless steel (in the manner described in Ref. 2 for small seals) failed after repeated cycling from --196°C to +100°C. Hairline cracks developed between the epoxy and the inside of the tube, possibly because the extrusion of the epoxy during the warm portion of the cycle caused excessive tensile stress to occur at the bond during the cold portion of the cycle. These cracks appeared whether the tube wall was 1.5 mm or 0.13 mm thick.

We then turned to the approach pioneered in 1923 by W. G. Housekeeper in the construction of metal-to-glass seals.[4] As he stated it: "The method consists in providing a large surface of contact between the glass

and the metal, and in so proportioning the metal that the stresses resulting from the difference in coefficients of expansion are less than the ultimate strength of the joint between glass and metal." In applying this method to our Stycast-to-metal seals, we developed the following rules:

(1) All cylindrical tubes (sleeves) to be imbedded in the epoxy were thin walled (0.13 mm) and imbedded to a depth of at least 5 mm.

(2) To allow for flexure, the sleeves were also thin walled outside the epoxy for a distance of at least 5 mm from the point where they entered the epoxy.

(3) All feedthrough pins were small in diameter (≤1 mm).

(4) To reduce the chance that the imbedded metal edge would initiate a crack in the epoxy and to reduce hoop stress, the edge was surrounded by ∿10 mm of epoxy.

(5) For good bonding, all relevant metal surfaces were abraded.

PROCEDURES AND RESULTS

The Stycast 2850 FT (Blue) epoxy resin was warmed to 60°C to reduce its viscosity and then thoroughly mixed with 5% (by weight) of catalyst 11 for approximately 30 min. with a stirrer mounted in a drill press.[1] At this temperature the pot life is 4 hours. The mixture was vacuum degassed in a bell jar for 45 min., poured into the machined polyethylene mold, and cured at 70°C for 24 hours. The mold supported all metal pieces during curing. A post cure at 100°C for 1 week improved the hardness at elevated temperatures and reduced outgassing.

Samples similar to the chamber shown in Fig. 1 withstood 50 cycles from 20°C to -196°C (liquid nitrogen temperature) and 10 cycles from 100°C to -196°C without developing a leak at the thin metal sleeve seals or at the 1 mm diam stainless steel feedthroughs.[5] The first portion of

the cycle consisted of a one hour lowering of the sample into a 1 m tall dewar containing a 20 cm layer of liquid nitrogen and included the full immersion of the sample. The second portion of the cycle consisted of a one hour warmup to room temperature. An optional third portion consisted of a one-half hour warmup to 100°C.

One useful property of Stycast epoxy is the possibility of installing new feedthrough pins after the epoxy has cured. A hole can be drilled through the cured epoxy and after a stainless steel pin is positioned, new epoxy may be poured around it to result in a vacuum tight feedthrough. We found that the bond between old and new Stycast is not a plane of weakness and appears to be as strong as the bulk material.

Two chambers were constructed using Stycast epoxy and tested by filling them with liquid xenon to measure the fraction (σ) of free electrons attached to impurities per mm of electron drift. This measurement does not determine the total concentration of impurities but the capture of electrons specifically by electronegative impurities. Since the chambers were cooled by immersing them in a bath of freon-11 ($C\,Cl_3\,F$) at -105°C, a low value of σ is additional proof of the absence of leaks.[6]

The first chamber was constructed using a 7 cm diam flange and had 11 feedthroughs. Upon filling with liquid xenon, we measured $\sigma = 0.10 \pm 0.02$ mm^{-1} at 2 kV/cm. (This value is somewhat higher than the range $\sigma = 0.02 - 0.05$ mm^{-1} we routinely obtain at that field in metal and glass chambers.) After an operating period of 16 hours, the same value of σ was obtained, demonstrating a lack of deterioration of liquid purity. Voltages of 7.5 kV were applied without significant leakage pulses.

A second, larger chamber was constructed using an 18 cm diam flange and had 125 feedthroughs (Figs. 1 and 2). Initially at 2 kV/cm the value $\sigma = 2.4 \pm 0.3$ mm^{-1} was measured. After two weeks of degassing at

100°C (both in an open oven and on the vacuum system) and periodic measurements, the value $\sigma = 0.11 \pm 0.02$ mm^{-1} was finally obtained. We tentatively attribute our difficulties with the large chamber to its greater complexity and the fact that the internal epoxy wall was machined after curing. This produced a cratered surface (due to small bubbles within the epoxy) that possibly contained absorbed gases.[7]

Some potential applications of this technique in the field of particle detection are (1) liquid xenon multi-wire chambers for the efficient detection and accurate localization of 0.1-5 MeV gamma rays[8-9] and (2) liquid argon (or heavy hydro-carbon) multi-plate electromagnetic shower counters for >100 MeV gamma rays.[10-12]

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References

1. Manufactured by Emerson Cuming, Inc., 604 West 182nd Street, Gardena, CA 94247. The cost is approximately $4.10 per kg in 8.4 kg containers (specific gravity 2.3). For additional information, see Technical Bulletin 7-2-7A, available from the above address. Note: This bulletin recommends the Blue preparation for high voltage applications.

2. K. S. Balain and C. J. Bergeron, Rev. Sci. Instr. 30, 192 (1959).

3. Reliably producing many ceramic to metal (kovar) seals in a single structure requires considerable care and skill. Cleanliness, surrounding atmosphere, wetting agents, time, and temperature are all very critical parameters. This has been amply verified by our own attempts to construct ceramic chambers in configurations similar to that shown in Fig. 1.

   One alternative possibility is a recently available "machineable glass" (Corning Glass Works, Corning, N. Y. 14830). It is expected that this material will be used extensively due to its excellent machineability and the fact that its thermal expansion closely matches available sealing glasses and nickel-iron alloys.

4. W. G. Housekeeper, Am. Inst. Elect. Eng. 42, 870 (1923). Discussion and diagrams are also in Strong, Procedures in Experimental Physics, pp. 25-26 (Prentice-Hall, Inc., Englewood Cliffs, N. J., 1966).

5. All seals were leak checked using a helium mass spectrometer having a sensitivity of 1×10^{-8} cc/sec.

6. In liquid xenon a 1 ppm O_2 contamination results in a σ value of approximately 1 mm^{-1} at 2 kv/cm [our measurements]. In gases, it has been found that haloginated compounds are much more electronegative than O_2. For example, SF_6 captures electrons at a rate $\approx6\times10^4$ times greater than O_2. [Robert Bins, McDonald Douglas

Research Lab., St. Louis, Mo., private communication (1971)].

Assuming that C Cl_3 F is 10^3 times more electronegative in liquid xenon than O_2 our measurement of σ = 0.1 at 2 kV/cm sets an upper limit on the C Cl_3 F contamination at 1 part in 10^{10}.

7. After this chamber had been degassed, we measured the outgassing rate R in μℓ/sec per cm^2 of exposed Stycast.

[Note: 1 μℓ is the number of molecules (3.5×10^{16}) required to fill a 1 liter volume to a pressure of 1 μm Hg]. At 100°C we found $R\approx2\times10^{-4}$ μℓ/sec/cm^2 and at 20°C $R\approx2\times10^{-6}$ μℓ/sec/cm^2. This latter value is lower than those measured for the elastomers commonly used in vacuum systems (Kel-F, Viton, etc.) [A. Roth, Vacuum Sealing Techniques (Pergamon Press, New York, 1966)].

8. H. Zaklad, S. E. Derenzo, R. A. Muller, and R. G. Smits, IEEE Trans. Nucl. Sci. NS 20(1), 429 (1973).

9. H. Zaklad, S. E. Derenzo, T. F. Budinger and L. W. Alvarez, Lawrence Berkeley Laboratory Report LBL-3000 (1974), Proceedings of the First World Congress on Nuclear Medicine, p. 362, Tokyo, Japan, Oct. 1974.

10. J. Engler, B. Friend, W. Hofmann, H. Keim, R. Nickson, W. Schmidt-Parzefall, A. Segar, M. Tyrrell, D. Wegener, T. Willard, and K. Winter, Nucl. Instr. Meth. 120, 157 (1974).

11. G. Knies and D. Neuffer, Nucl. Instr. Meth. 120, 1 (1974).

12. W. J. Willis and V. Radeka, Nucl. Instr. Meth. 120, 221 (1974).

13. Araldite 501 is a casting epoxy made by Ciba Products Co. See R. J. Corruccini and J. J. Gniewek, U. S. National Bureau of Standards Monograph 29 (1961) for thermal expansion data from 0°K to 300°K. The expansion value at 70°C used in Table I was determined by linear extrapolation.

TABLE I

Differential thermal expansion ($\Delta L/L$)[a]
(parts per million)

	-196°C	-77°C	20°C	70°C	120°C
Aluminum[b]	- 5125	-3290	-1175	0	+1215
Yellow Brass[b]	- 4495	-2735	- 975	0	+ 975
Stainless Steel AISI 304[b]	- 3615	-2280	- 820	0	+ 820
Stycast 2850 FT[c]	- 5050 ± 400	-3100 ± 400	-1400 ± 200	0	+3300 ± 500
Araldite 501[d]	-12720	-8550	-3300	0	-

[a] Arranged so that $\Delta L/L \equiv 0$ at 70°C, the curing temperature of Stycast.

[b] From the American Institute of Physics Handbook, Section 4, edited by D. E. Gray (McGraw-Hill, New York, 1972).

[c] Our measurements.

[d] A "typical" unfilled casting epoxy (See Ref. 13.)

Figure Captions

Fig. 1. Simplified schematic of low temperature liquid xenon chamber with 5 rows of 25 feedthrough pins (4 rows shown), edge sleeve seal to flange and central sleeve seal to pumpout-fill tube. All metal components are stainless steel AISI 304 except for copper gasket used to seal flanges.

Fig. 2. Bottom view of upper flange assembly sketched in Fig. 1.

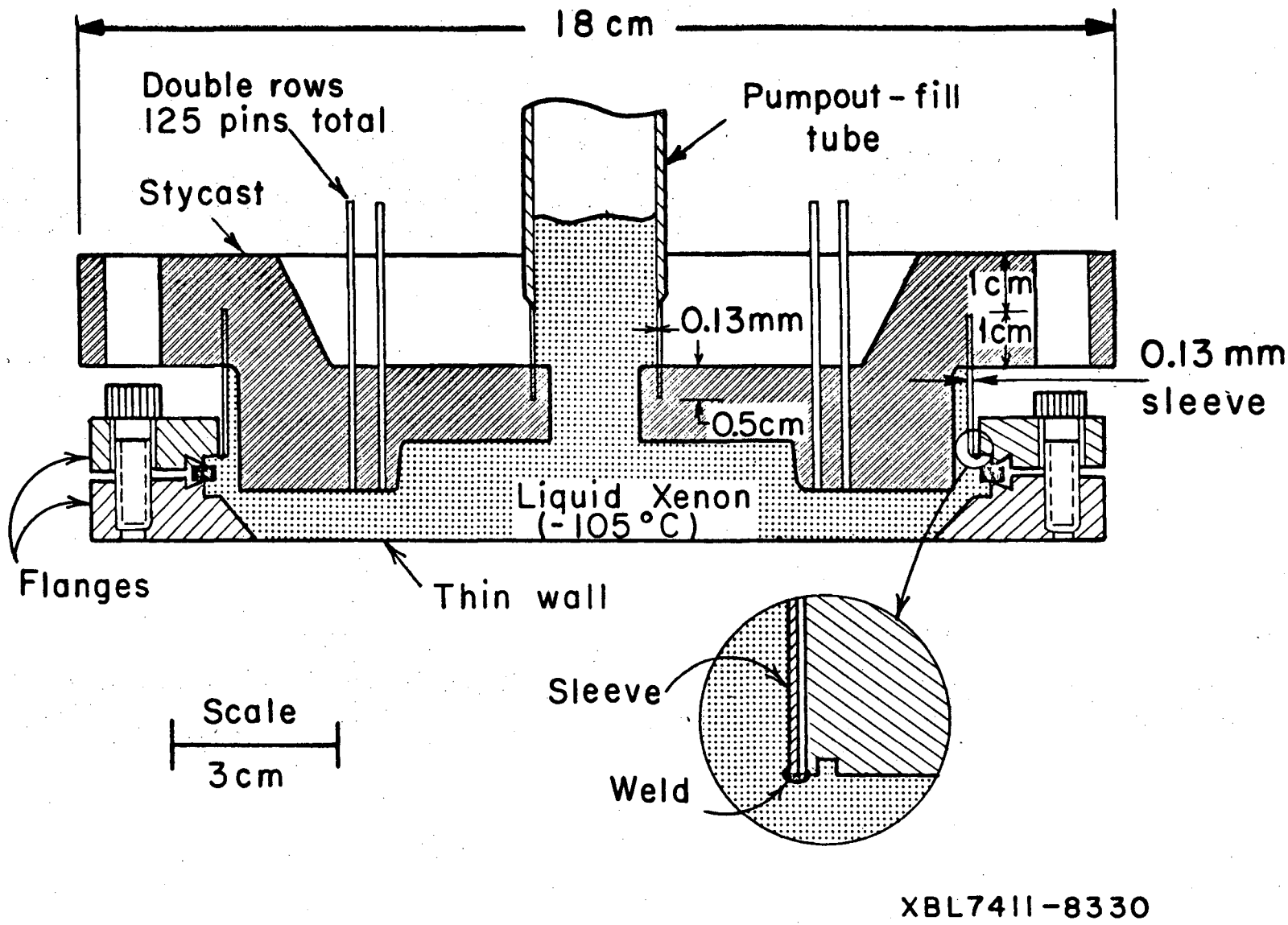
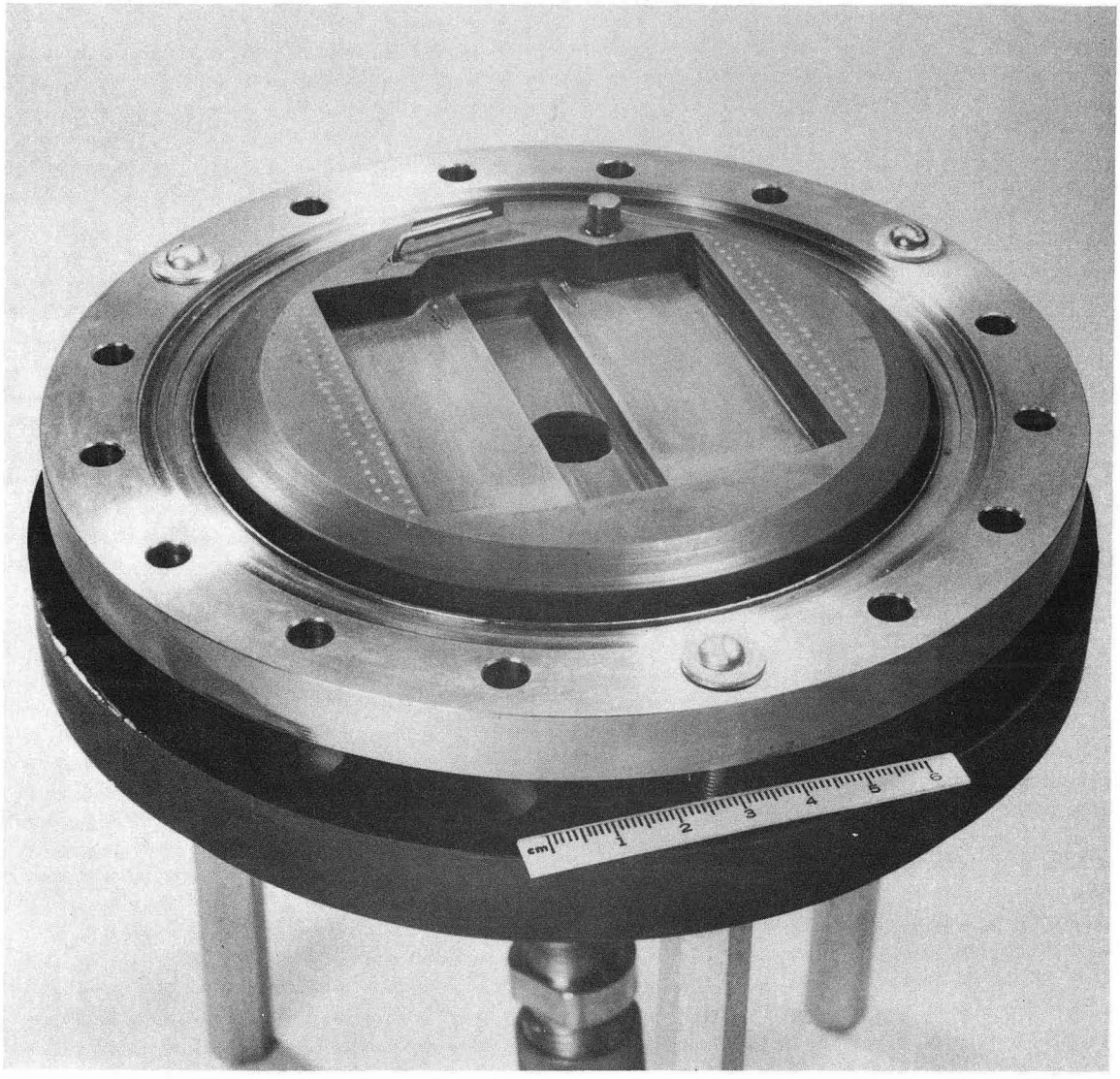

18 cm
Double rows
125 pins total
Pumpout - fill
tube
Stycast
0.13mm
1cm
1cm
0.13 mm
sleeve
0.5cm
Liquid Xenon
(-105 °C)
Flanges
Thin wall
Sleeve
Weld
Scale
3cm
XBL7411-8330

Fig. 1.



CBB 7411-7517

Fig. 2.

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