

FIG. 2. The influence of environment temperature  $T_3$  on measurements of the Seebeck voltage.

constant  $\Delta T'$  of  $40^\circ\text{C}$  was only  $20\ \mu\text{V}/^\circ\text{C}$  over the entire temperature range of the experiment.

Inasmuch as sizable surfaces of the sample were in contact with the heat sinks, abrupt temperature gradients with a resulting Benedicks effect<sup>11</sup> were unlikely. Ioffe<sup>12</sup> has suggested that errors might result if, when  $T_2 > T_1$ ,  $T_1 > T_1'$  and  $T_2 < T_2'$  due to thermal gradients at the contacts between the sample and the heat sinks. If such were the case, the error would be in the direction opposite from that observed because  $\Delta T_{12}'$  would be greater than  $\Delta T_{12}$ . The effect of such temperature gradients, which possibly may be present, is comparatively negligible.

If, however,  $T_1' < T_1$  and  $T_2' < T_2$  due to heat conduction through the thermocouple leads away from the thermocouple junctions and into the infinite heat reservoir of the environment at temperature  $T_3$ , then  $\Delta T_{12}' < \Delta T_{12}$  (remember that  $T_2 > T_1$  and  $T_1 > T_3$  in the experiment so that  $T_1 - T_1' < T_2 - T_2'$ ). This source of error is in the proper direction.

Finally, if the foregoing suggestion is correct, then the behavior represented by Eq. (2) should disappear when the thermocouple leads are equilibrated thoroughly with the heat sinks so that  $T_1' = T_1$  and  $T_2' = T_2$ . Experimental results with such a setup, curve C in Fig. 2, verify this conclusion. Furthermore, if  $T_3$  is made even more dissimilar from  $T_A$ , the effect should be enhanced. Again a comparison of curves B (with  $T_A = 73^\circ\text{C}$ ) and A (with  $T_A = 157^\circ\text{C}$ ) shows that such is indeed the case.

Reduction of the cross section of the thermocouple leads alone is ineffective in eliminating the errors just discussed for, unless the thermocouples are placed in deep bores, the heat transfer into, as well as out of, the thermocouple is reduced. The preferred experimental arrangement for

measuring Seebeck coefficients is one in which the thermocouples are thermally equilibrated with the heat sinks, say by leading the thermocouples in through the heat sinks. In addition  $T_3$  should be made as close to  $T_A$  as possible. The apparatus used by Brice and Wright<sup>13</sup> and that recommended by Ioffe<sup>12</sup> incorporate the first of these features. Similar precautions for measuring the thermoelectric powers of thermocells are discussed by Holtan.<sup>14</sup>

These effects can be utilized as a check on the reliability of Seebeck coefficient measuring apparatus, in particular, on thermocouple calibration and the absence of any unsuspected thermal gradients. A properly functioning apparatus may yield an increase in spread of  $|\alpha|$  values, but the average  $|\alpha|$  should remain constant as  $\Delta T$  is decreased.

\* Present address: Joseph Kaye & Company, Inc., Cambridge, Massachusetts.

<sup>1</sup> H. K. Henisch, *Elect. Commun.* **25**, 163 (1948).

<sup>2</sup> B. I. Boltaks, *Zhur. Tekh. Fiz.* **20**, 1039 (1950).

<sup>3</sup> For a reply to other of Boltaks' criticisms, see H. K. Henisch, *Proc. Phys. Soc. (London)* **B64**, 1014 (1951).

<sup>4</sup> R. A. Horne, *J. Appl. Phys.* **30**, 393 (1959).

<sup>5</sup> T. Sato, *J. Phys. Soc. Japan* **6**, 125 (1951).

<sup>6</sup> J. C. M. Li, *Trans. Am. Inst. Mining, Met. Petrol. Engrs.* **212**, 661 (1958).

<sup>7</sup> J. Savornin and A. Poggi, *Compt. rend* **238**, 656 (1954).

<sup>8</sup> J. Savornin and F. Savornin, *Compt. rend.* **236**, 898 (1953).

<sup>9</sup> G. A. Ivanov and L. I. Mokievski, *Zhur. Tekh. Fiz.* **26**, 1343 (1956); *Soviet Phys. Tech. Phys.* **1**, 1313 (1957).

<sup>10</sup> Similar difficulties have been noticed in experiments with molten salt thermocells, see, for example, B. R. Sundheim and J. Rosenstreich, *J. Phys. Chem.* **63**, 419 (1959).

<sup>11</sup> See J. Tauc, *Rev. Modern Phys.* **29**, 308, (1957), especially pp. 323-324.

<sup>12</sup> A. F. Ioffe, *Semiconductor Thermoelements and Thermoelectric Cooling* (Infosearch, Ltd., London, 1957) pp. 132ff.

<sup>13</sup> J. C. Brice and H. C. Wright, *J. Sci. Instr.* **35**, 146 (1958).

<sup>14</sup> H. Holtan, Jr., *Electric Potentials in Thermocouples and Thermocells* (Schotanus & Jens, Utrecht, 1953), p. 55.

## Method for Sealing Stainless Steel to Glass\*

J. E. BENBENEK AND R. E. HONIG

David Sarnoff Research Center, Radio Corporation of America,  
Princeton, New Jersey

(Received December 24, 1959; and in final form, January 20, 1960)

FOR some time, there has been a great need for sealing nonmagnetic metals to hard glass. While copper-to-glass seals of the "Housekeeper" type have been known<sup>1</sup> and employed for many years, they have the disadvantage that the Cu feather edge is heavily oxidized during the sealing operation. Thus it may become porous eventually and even collapse under external pressure. To avoid these difficulties, it is desirable to join stainless steel (S.S.) directly to glass. While the expansion coefficients of these materials differ considerably (S.S.:  $17 \times 10^{-6}/^\circ\text{C}$ ; hard glass:  $3-5 \times 10^{-6}/^\circ\text{C}$ ), this difference is no greater than for Cu ( $17 \times 10^{-6}/^\circ\text{C}$ ) and glass. Thus it should be possible to

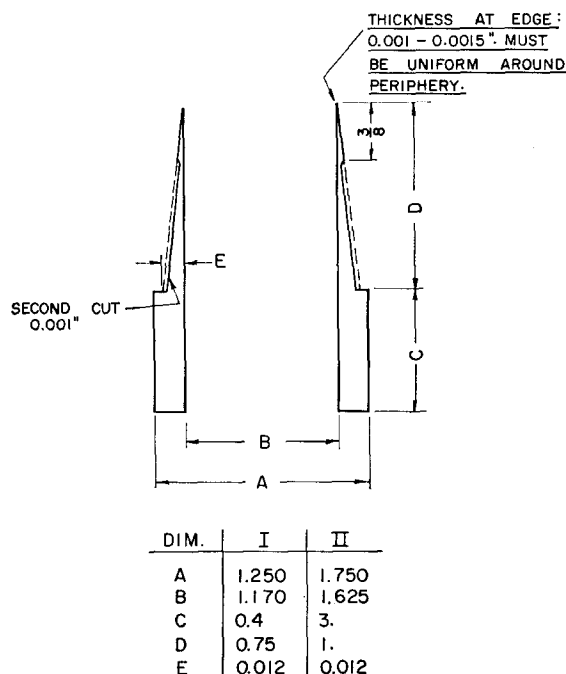


FIG. 1. Dimensions of stainless steel part for S.S.-to-glass seals (not drawn to scale—all dimensions are in inches).

make S.S.-to-glass "Housekeeper" seals, provided the greater stiffness of S.S. is taken into account. Although Pattee briefly mentions<sup>2</sup> seals of this type, he does not give information on how to make them. This note describes in detail how to produce successful S.S.-to-glass seals.

For the metal part, nonmagnetic stainless steel tubing AISI type 304 is used. To eliminate strains resulting from machining, the material is first fired in dry  $H_2$  (dew point:  $-35^\circ C$ ) at  $1065^\circ C$  for 15 min. The strain-free tubing then is machined to the dimensions shown in Fig. 1. To obtain an edge whose thickness is uniform around the periphery, a cut first must be taken from the inside. The tubing then is put on a mandrel for machining the outside. The most important feature is the second cut of about 0.001 in. depth (shown dotted in Fig. 1), which gives sufficient flexibility to the tapered end. For the edge to be smooth, it should be not less than 0.001 in. thick, but it should not be heavier than 0.0015 in. to avoid stiffness. After machining, the stainless steel part is degreased and then oxidized by firing in line  $H_2$  (dew point:  $+6^\circ C$ ) at  $1065^\circ C$  for 20 min. If it is to be welded within 2 in. of the glass seal into an outer collar, a snug fitting copper plug is inserted and the Heli-Arc welding process done before glassing. After welding, the piece may have to be remachined on the end opposite to the feather edge, to true it up. Then the assembly should be degreased, and finally vacuum fired with the copper plug at  $1000^\circ C$  for 15 min. The stainless steel part is now ready for glassing.

For sealing to stainless steel, borosilicate glass No.

7052(FN) was chosen for most applications, for the following reasons: (1) Its coefficient of expansion ( $4.6 \times 10^{-6}/^\circ C$ ) is larger than that of other hard glasses, such as Pyrex, and therefore it matches the metal somewhat better. (2) It readily wets the oxidized stainless steel. (3) It seals well to other hard glasses, including Pyrex. (4) On the other end, it can be sealed directly to molybdenum. (5) Its optical quality is high. For the sealing operation, the stainless steel tube is chucked in a lathe and wrapped to within  $\frac{1}{2}$  in. of the end to be glassed with wet asbestos paper tape to keep it cool. A piece of FN glass is shaped to overlap the exterior of the feather edge by about 0.050 in. and fused to the outside of the metal. Care is taken to point the flame on the glass rather than on the metal to prevent overheating the thin metal edge. The glass is flame-cut at a distance of  $\frac{1}{8}$  in. from the feather edge and then carefully rolled inside to complete the internal beading of the stainless steel tube. Finally, an FN glass tube is sealed to the beaded edge and the assembly flame annealed.

A number of stainless steel-to-glass seals of all sizes have been made successfully by this technique, and they have been used where nonmagnetic assemblies of this type are required. Successful seals between S.S. and Pyrex glass also have been made.

\* Presented at the Fourth Symposium of the American Scientific Glassblowers' Society, Corning, New York, May 22, 1959.

<sup>1</sup> John Strong, *Procedures in Experimental Physics* (Prentice-Hall Inc., Englewood Cliffs, New Jersey, 1945), p. 25.

<sup>2</sup> H. H. Pattee, Jr., *Rev. Sci. Instr.* **25**, 1132 (1954).

## Glass Combustion Bomb\*

RALPH L. NUTTALL, MARGARET A. FRISCH,<sup>†</sup> AND WARD N. HUBBARD  
*Chemical Engineering Division, Argonne National Laboratory,  
Lemont, Illinois*

(Received December 21, 1959; and in final form, January 18, 1960)

ONE of the major problems in bomb calorimetry is finding conditions to give complete combustion. Usually the proper conditions must be found by trial and error, and frequently a large amount of time and work are required. The number of trial combustions needed may be reduced materially by the use of a transparent bomb so that the combustion and the effects on it of varied conditions can be observed visually.

A transparent "glass bomb," apparently the first described in the literature, has been built and has proved invaluable in working out suitable combustion conditions for inorganic materials. It is possible to observe relative combustion temperatures, rates of burning, melting of sample, or combustion products, spattering, fuse wire temperature, etc., and to study the effects on these things of different gas pressures, sample arrangements and supports,