Laboratory method for making small alumina crucibles

1961 J. Sci. Instrum. 38 372

(http://iopscience.iop.org/0950-7671/38/9/423)

temperature with a precision of the order of 0·01° c. It eliminates the possibility of thermo-elements ever touching the water level or the zone of temperature gradient.

References

A.S.T.M., 1955 *Book of ASTM Standards*, Part 5, ASTM Designation E 77-52T (American Society for Testing Materials, Philadelphia), p. 1373.

BANSAL, T. D., 1961, *Vigyan Pragati*, **10**, 179.

HALL, J. A., 1955, *Quatrième Rapport du Comité Consultatif de Thermometrie au Comité International des Poids et Mesures*, Session de 1954 (Gauthier Villars, Editeur; Imprémeur Libraire, Paris), p. T44.

STIMSON, H. F., 1945, *J. Wash. Acad. Sci.*, **35**, 201.

THOMAS, J. L., 1934, *J. Res. Nat. Bur. Stand.*, **12**, 323.

Laboratory method for making small alumina crucibles

by I. LEVANONE, Ministry of Defence, Scientific Department, Tel-Aviv, Israel

MS. received 4th January 1961, *in revised form 13th March* 1961

The technique generally adopted in the laboratory for the vacuum evaporation of powdered materials requires small refractory crucibles made from fired ceramic cement. However, the powdered material is likely to be contaminated by impurities originating from the crucible material at the high temperatures usually required. An example is the vacuum evaporation of pure zinc sulphide—for luminescence purposes—at a temperature exceeding 1400° c. It would be advisable in such cases to use crucibles made from pure materials, which contain no bonding cements and which can be melted to a crucible form. A suitable material that complies with these requirements is crystallized powdered alumina, which can be formed into the required crucible shape by the technique described here.

The raw material (RR Alundum in crystalline form, 90–120-mesh (Norton Co., U.S.A.)), its surface roughly levelled (Fig. 1), is placed in a porcelain beaker and the

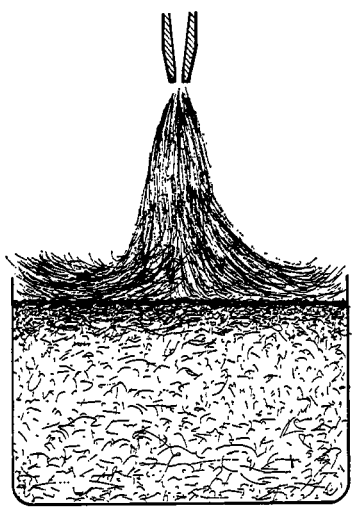

Fig. 1. Gentle heating of powdered alumina, using an oxy-gas blow-lamp.

surface gently heated by an oxy-gas blow-lamp, thus producing a sintered surface layer (which will prevent subsequent blow-out of the alumina powder). The gas flow into the blow-lamp is then increased considerably until a very high-temperature flame is obtained.

This flame when concentrated at one point on the sintered surface will form, in a short time, a large drop of melted alumina. The gas flow is then adjusted to give a thin short flame and the burner is held as close as possible to the centre of the drop. The crucible shape will be formed by the jet flow (Fig. 2). Toward the end of the process the nozzle is brought to the surface level of the crucible.

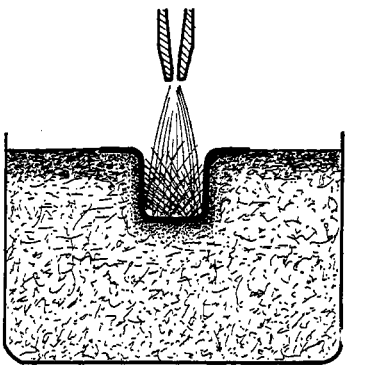

Fig. 2. Formation of crucible shape.

Owing to the high temperature the nozzle has to withstand, it is advisable to use one made of stainless steel. If a brass nozzle is used it might be damaged during the process and the crucible itself would be contaminated.

With a normal glass blow-lamp the crucible obtained has a diameter of 10 to 15 mm, a height of 15 to 20 mm and 1 to 2 mm thickness. If an oxy-acetylene blow-lamp is used, larger dimensions may be obtained.

The wall thickness can be controlled by the duration of the heating. With some experience and using alternative jets, more complex shapes can be produced as shown in Fig. 3.

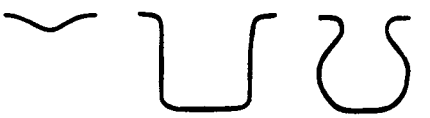

Fig. 3. Varying crucible shapes.

Large edges are normally formed in the heating process; they may have to be trimmed with a hard grinding stone.

Crucibles obtained by this method proved to be most satisfactory for the evaporation of zinc sulphide and have been actually used for hundreds of evaporation processes. From a practical point of view it may be noted that the crucible can be cleaned by boiling acids without damage and that it withstands temperatures up to 2000° c.