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A VACUUM HOT PRESS FOR USE IN A GLOVE BOX

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ABSTRACT

This note describes a simple hot press suitable for use in either a vacuum or a controlled atmosphere. The press can be easily set up in a glove box.

1. INTRODUCTION

In an effort to produce dense compacts of fine-grained beryllia, it was decided to use vacuum hot pressing for a comparative study of the fabrication of various highly sinterable BeO powders. Because of the toxic nature and finely divided form of the powder, it was desirable to carry out all the operations (the preparation and handling of the powder, the loading of the die, etc.) under glove box conditions.

This note describes a hot press which can be operated to a temperature of 1300°C in vacuo or in any desired atmosphere. The dimensions of the press are such that it can be operated easily in the confined space of a Harwell Mark II glove box.

2. DESCRIPTION

The arrangement of the press is shown in Figure 1. It consists of an outer casing of silica with water-cooled brass top and base plates cemented in position with epoxy resin. Both plates have holes to allow the die and support to pass freely through.

The base plate of the casing sits on a solid platen which is also water cooled, and the two are vacuum sealed by means of neoprene O-ring. The platen has provision for the vacuum line and the thermocouple connection.

The graphite die and stainless steel support sit on the platen. A Pt v. Pt 13% Rh thermocouple passes through the support, and is sealed into the bottom platen with an epoxy resin The measuring junction is located in the graphite die alongside the sample.

The top plate on the silica has an O-ring vacuum seal on which the bottom plate of a stainless steel bellows is placed. The stainless steel ram and screwed end-piece for connecting to the hydraulic ram are one section and form the top plate of the bellows. The bellows have an overall length of 3 inches with fourteen convolutions and compress to 1-1/4 inches before any pressure is exerted on the silica.

3. OPERATION

To set up the press, the bottom platen is placed on the base of the press and connected to the vacuum system, and the stainless steel support is placed in position with the thermocouple passing through it. The graphite die is then loaded and placed on top of the support. The silica

tube is placed over the die and the hydraulic ram placed in position. The top section of the ram, which is connected to the stainless steel bellows, is screwed to the hydraulic ram and a minimal pressure of 100 lb/in^2 is exerted. The clamps to hold the bellows to the brass top plate are then placed in position and the chamber evacuated

The bellows are clamped to the brass top plate before the vacuum is applied to prevent contraction of the bellows under reduced pressure and consequent rupture of the O–ring seal.

The bellows should never be completely contracted; otherwise pressure is transmitted to the silica. This can be prevented by using a plunger of suitable length. In the work on BeO fabrication, plungers of alumina tubing and of various lengths are used depending on the thickness of compact required.

The press is used in a Harwell Mark II glove box and all controls, including those to the vacuum system to the hydraulic press, and to the recirculating cooling water, are located outside the box. The overall length of the vacuum press is 15 inches and with the aid of locating holes in the platen and the stainless steel support, no difficulty has been experienced in setting up in the glove box.

Heating of the specimens is carried out by a high frequency induction furnace, the work coil being the only component in the box. Under typical conditions the vacuum obtained is better than 1×10^{-3} mmHg. In all the work on BeO the sample was outgassed at 400 °C for 15 minutes before any pressure was exerted. Without any insulation between the die and the silica wall, sample temperatures to 1300 °C are easily achieved, although the temperature of the silica must not rise above 1200 °C.

After heating, pressing, and cooling, the clamps are released slowly, and air is admitted to the press.

Although present experience has been with vacuum operation of the press, it would be a simple matter to operate under any desired atmosphere.

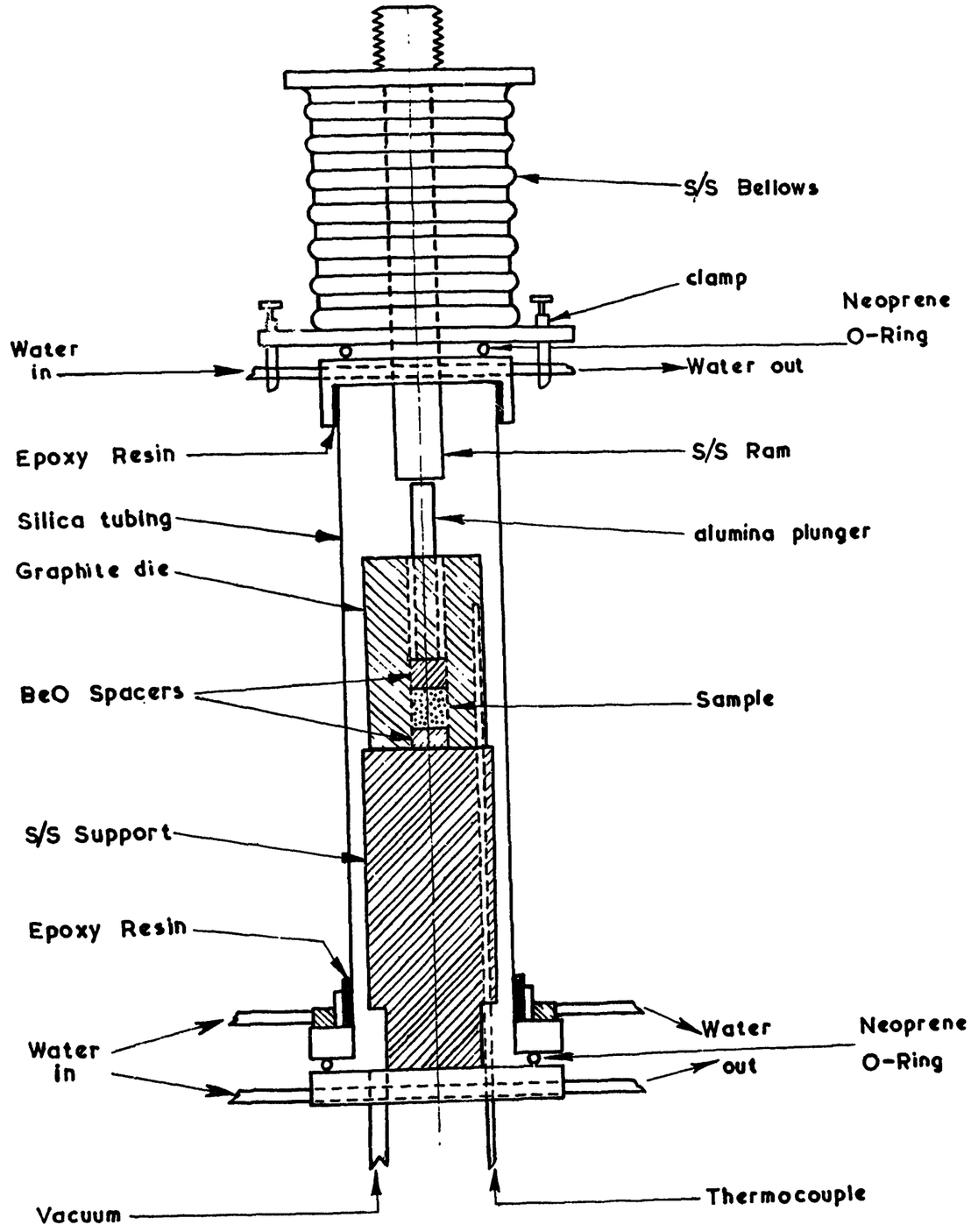

FIGURE 1 HOT VACUUM PRESS

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