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Reaction Bomb for Use at Elevated Temperatures and Pressures

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In the preparation of the trihalides of zirconium and hafnium from the corresponding tetrahalides,[1] it was necessary to devise a system in which the entire reaction container could be maintained at an elevated temperature and pressure. This was accomplished in the design of the bomb shown in Fig. 1, and described below.

For our purposes 8–18 stainless steel had satisfactory properties with respect to corrosion. The stainless steel body was 9 in. long, and 2 in. in diameter, with an internal cavity 8 in. deep and 1 in. in diameter. The top face was tapped to take eight $\frac{1}{4}$-28 Allen head cap screws. The head consisted of two parts, *B* and *C*, which sealed the bomb and also formed the valve system. The outer part *B* had a flanged face the same diameter as the body of the bomb, and was also tapped to take the cap screws. A recess was cut in the face of *B*, 6/32 in. deep and 1 in. in diameter, in which was seated the terminal end of insert *C* when the bomb was sealed. Both joined surfaces at *D* and *E* were sealed by using sheet copper gaskets ($\frac{1}{32}$ in. thick) in conjunction with matching groves and ridges which were machined on the gaskets and surfaces of the bomb parts. The terminal end of valve stem *C* was $\frac{1}{16}$ in. smaller in diameter than the cavity of the bomb, so that the stem could be moved into the bomb cavity during evacuation or back filling. The valve stem *C* had a hole drilled lengthwise to within a $\frac{1}{16}$ in. of the terminal end, at which point it was joined with a hole perpendicular to the first. A leak-proof seal between the valve stem and the sidewall of the head was effected by means of an "O" ring at point *F*. The retaining screw *G* prevented the stem from rotating about its axis, and permitted a limited linar displacement of the valve stem. The hex-nut *H* fixed the valve stem in position.

An example of a typical procedure follows. Upon loading the reactants into the body of the bomb, the outer copper gasket and bomb head are fitted into place and tightened securely by means of the capscrews. With the retaining screw loosened slightly, and the valve open, the bomb is evacuated. While still evacuating, or back-filling with gas, the hex-nut is turned so as to bring the sealing surfaces tightly together onto the inner copper gasket. With the hex-nut and retaining screw secured the bomb is ready for operation. The bomb is then placed in a tube furnace to such a depth that the valve stem is outside the furnace and therefore considerably cooler than the body of the bomb. The bomb described has been heated to 700°C without undue degradation of the rubber "O" ring.

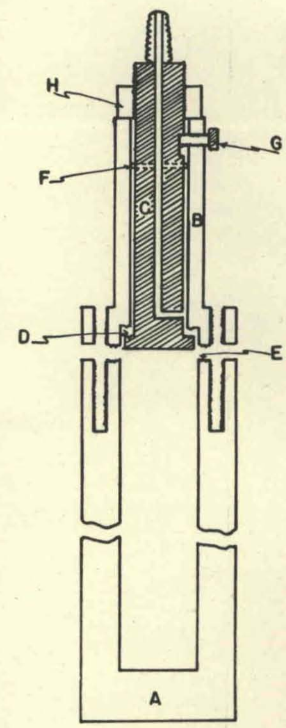

Fig. 1.

Gas samples may be conveniently taken during the course of the reaction. We feel that such a bomb may be conveniently adapted to the preparation of a variety of compounds, particularly if a more corrosion-resistant liner and gasketing material are used. This would vary with the preparations.

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Teflon as a Pressure Medium

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TEFLON has been used in a number of different ways in high pressure systems. Weir[1,2] has investigated its compressibility, mechanical properties, and transition phases at room temperature up to 10 000 atmospheres. Swenson[3] investigated the possibility of the use of Teflon as a pressure transmitting medium in the liquid helium range. His results showed that Teflon as a pressure medium is of marginal utility at very low temperatures. The purpose of this paper is to report on the use of Teflon as a pressure transmitting medium at elevated temperatures and pressures.

The use of Teflon as suggested in this note is quite similar to a similar use of indium and silver chloride described by Bridgman.[4]

The flow rate of Teflon is insensitive to pressure and temperature, permitting its use in pressure vessels without regard to liquid tight closures. It is possible to use a simple steel die, Fig. 1, as the pressure vessel, thus providing an economical experimental apparatus at high pressures and temperatures in laboratories otherwise not equipped for high pressure experimentation. Such an apparatus permits the measurement of irreversible changes in solids and powders at pressures up to at least 10 000 atmospheres, and temperatures up to at least 300°C, and time intervals up to at least one week.

The temperature in the steel die, shown in Fig. 1, was controlled by heated platens in a 20-ton hydraulic press. The stress in the Teflon was assumed to be equal to the force on the press divided by the area of the plunger, less friction. The friction was measured by observing the force necessary to move the hull on the plunger under load and temperature.

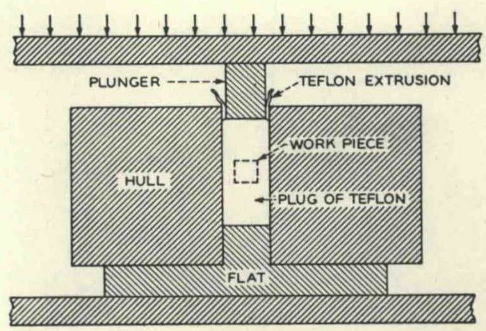

FIG. 1. Typical pressure experiment.

For slowly reversible changes, this system is useful because the specimen can be removed and cooled to room temperature in approximately 10 sec. The chief utility of this system is that it will give quick results at very little cost, which may justify the expense of building a more elaborate high pressure facility.

The setup shown in Fig. 1 was used to measure reversible and irreversible density changes in glass.[5] It has also been used to find the inversion temperature of GeO_2 powder between its two phases as a function of pressure.

[1] C. E. Weir, J. Research Natl. Bur. Standards **46**, 207 (1951).
[2] C. E. Weir, J. Research Natl. Bur. Standards **50**, 95 (1953).
[3] C. A. Swenson, Rev. Sci. Instr. **25**, 834 (1954).
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[5] O. L. Anderson, J. Appl. Phys. **27**, 943 (1956).

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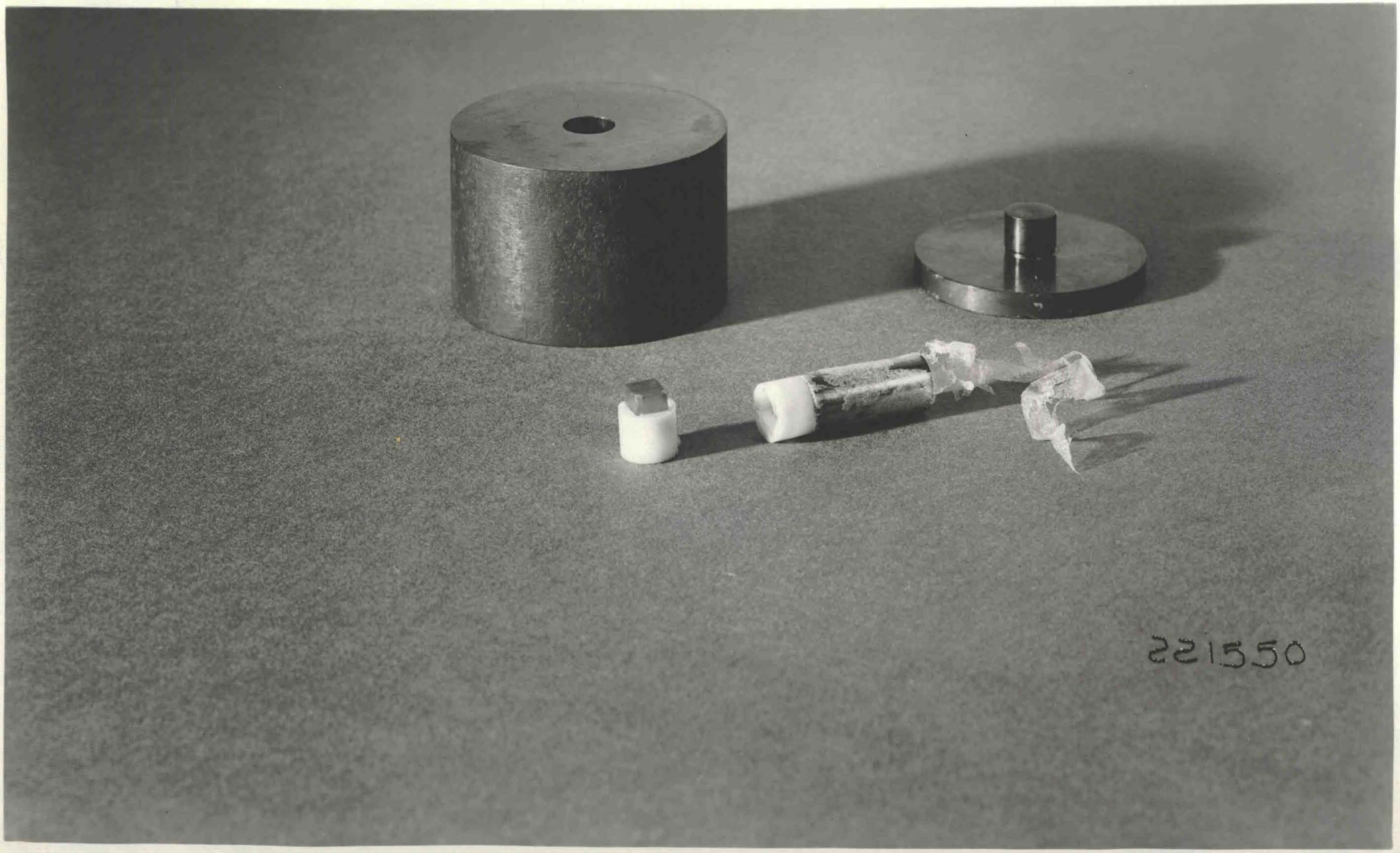

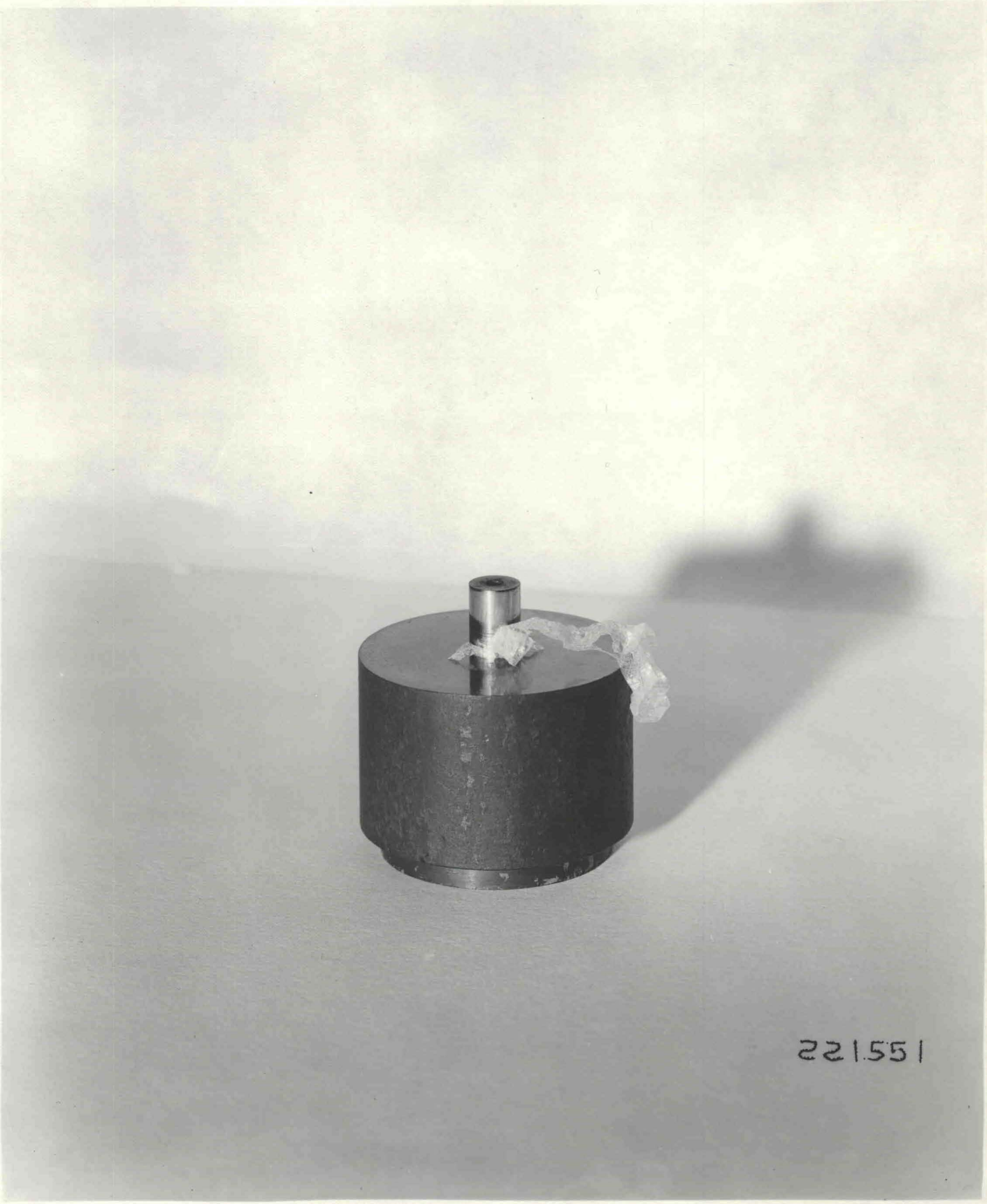

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