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HOT PRESSES FOR GLOVE BOX AND MANIPULATOR CELL USE

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ISOTOPES DEVELOPMENT CENTER

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Isotopes Division

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ABSTRACT

Two hot presses were designed to fabricate dimensionally accurate compacts: a miniature press for research and development operations in glove boxes and a larger press for production applications in manipulator cells. The miniature hot press is designed to withstand very rapid heating and cooling cycles as well as rapid pumpdown or purging. Self-alignment is incorporated in the pressing column, and packed insulation in the furnace is eliminated to achieve compactness and simplicity. The miniature press can be readily disassembled and reassembled in a glove box through the access ports. Although it was designed primarily for use with radioactive materials, it is also useful for general development work where easy operation, small sample size, and rapid heating-cooling cycles are desired. Samples in the range of 1/4 to 1/2 in. in dia can be compacted in the miniature press.

The production hot press was designed to fabricate sources in the range of 3/4 to 3 in. in dia. The production press has a self-aligning pressing column and an induction-heated furnace.

Both presses have been operated at 1800°C. Graphite dies or graphite dies lined with refractory metals or alumina have been used in all applications.

INTRODUCTION

In most applications where radioisotopes are used as radiation or heat sources, dimensionally accurate compacts fabricated from source materials with known physical properties are highly desirable. The desired properties include high density, high melting point, high thermal stability, high thermal conductivity, and good mechanical properties. These properties greatly affect the efficiency of a source and also the shield and heat transfer characteristics.

The fabrication methods applicable to radioisotope sources can be divided into three groups: (1) cold forming, (2) hot forming, and (3) melt forming. The hot-forming method has two distinct advantages over the other two methods--the dimensional accuracy of the compact formed and the adaptability of the method to a wide variety of materials. The hot-forming method also has the advantage that two materials can be reacted to produce the desired compound as the compact is being formed (reactive hot pressing). Cold-forming methods are sensitive to a number of process variables which are not amenable to precise control with radioactive materials. Particle size, for example, is impossible to control if self-heating of a powder raises the temperature above that required for sintering.

The hot-forming method consists essentially of the simultaneous application of heat and pressure; the pressure can be applied uniaxially (hot pressing and hot swaging) or multiaxially (isostatic pressing). Hot pressing--the application of pressure to a sample in a heated die--was chosen for the present work because of its simplicity and safety.

Several hot presses are described in the literature.[1-3] McClelland and Smith[4] describe a hot press which they designed for use as a research tool. The two hot presses described in this paper are designed for two applications: the miniature press for research and development operations in glove boxes (and with minor modifications, for use in a manipulator cell) and the larger press primarily for production in manipulator cells.

EQUIPMENT

Miniature Hot Press

The miniature hot press (Figs. 1 and 2) consists of three components: (1) the heater, which includes a carbon resistance element and variable-voltage, high-amperage power supply; (2) the die column, which includes the die assembly and the hydraulic cylinder for applying pressure to the die plunger; and (3) the containment shell, which provides insulation and contains the vacuum or other atmosphere.

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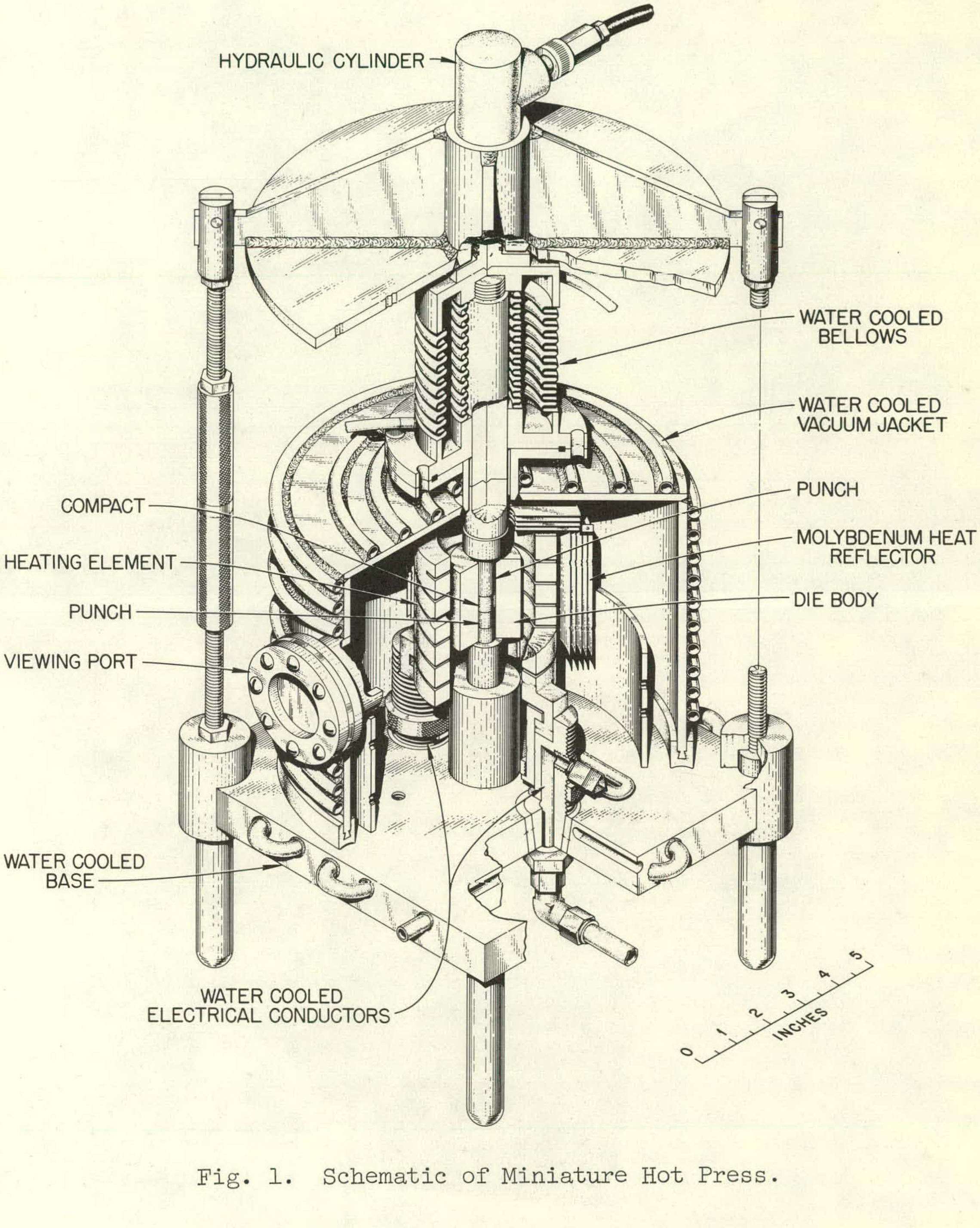

Fig. 1. Schematic of Miniature Hot Press.

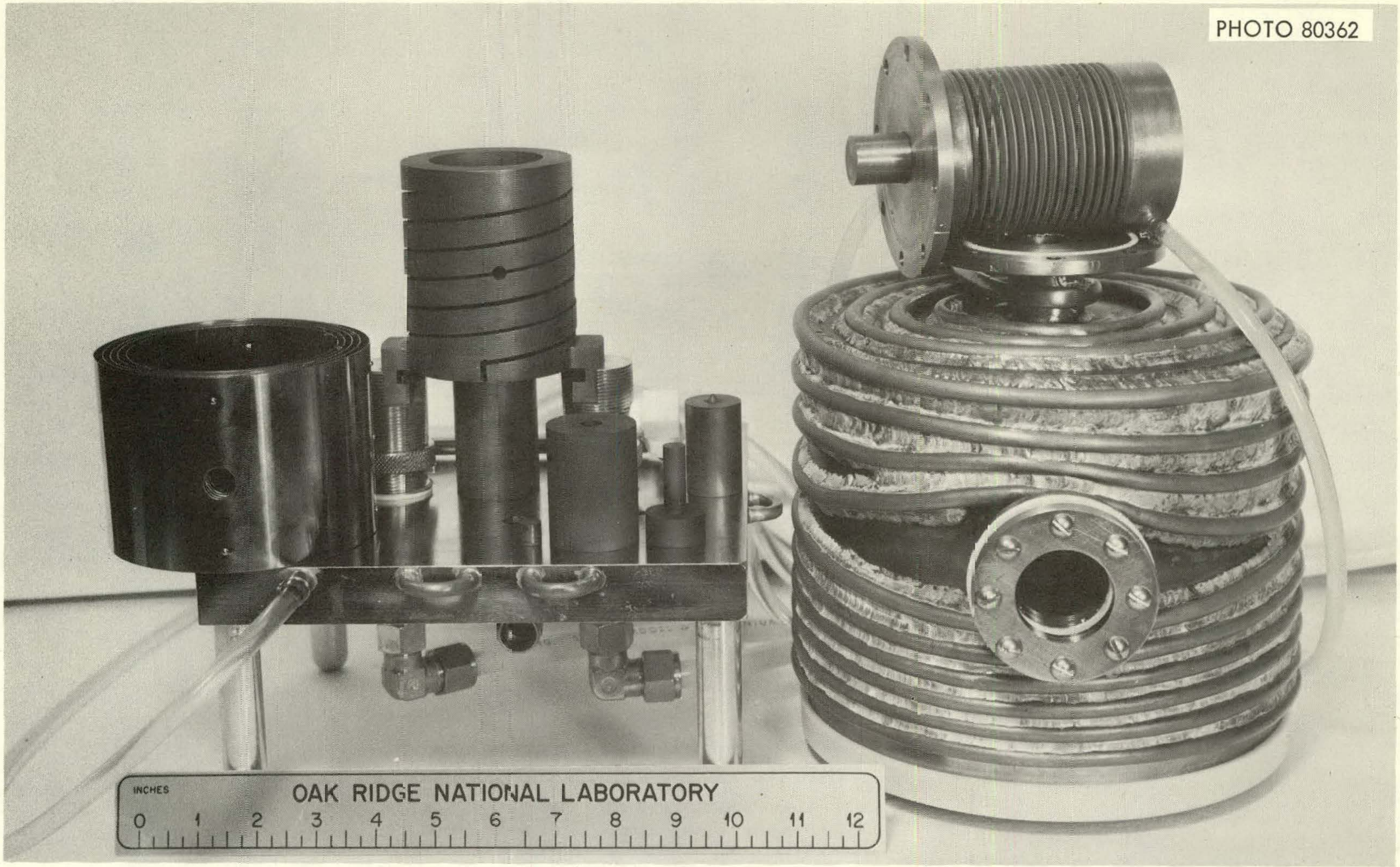

Fig. 2. Disassembled Miniature Hot Press.

Containment Shell

The containment shell assembly has three main parts: the base plate, the cover vessel, and the molybdenum heat reflector, which is the only insulation. Since the greatest heat loss occurs through the die column, packed-type insulation would only increase the size of the press without significantly reducing heat loss. The molybdenum heat reflector consists of five concentric sheet-metal cylinders assembled with molybdenum wire. Each cylinder contains holes in line with the quartz window of the cover vessel which form a viewing port for observation. During operation, the reflector assembly is placed around the graphite heating element where it is supported by the annular projections at the bottom of the heating element. Thorium oxide disks are placed between the reflector assembly and the graphite projections to provide electrical insulation.

The cover vessel is constructed of stainless steel and is water-cooled by an external copper coil which is attached by brazing. The bottom of the cover vessel is sealed to the base plate with a neoprene rubber channel gasket and is aligned by two interior pins that mate with two blind holes in the base plate. A sealed quartz window on the front of the cover vessel allows temperature measurement by optical pyrometry. The top of the cover vessel has a flange for the attachment of a sealed push rod to transmit force to the die plunger. A vacuum seal is attained around the push rod by two concentric bellows that are sealed by solder. Water is passed between the two bellows to provide cooling.

In addition to maintaining containment, the base plate of the shell serves as a base for assembly and alignment and provides connections for vacuum or purge. The heating element, die assembly, and cover vessel are attached directly to the base plate.

Die Column

The die column consists of the base plate, die support, die body, bottom die plunger, top die plunger, graphite push rod, and brass push rod. The die column components are self-aligning with the aid of centering projections and mating indentations. The brass push rod does not center but is free to position itself to prevent misalignment. The mechanism which provides hydraulic force to the bellows-sealed push rod consists of a Blackhawk Model R-618 hydraulic cylinder mounted on a reinforced circular plate. The flange that contains the hydraulic cylinder is secured to the base plate by four adjustable threaded rods.

Heater

The die assembly is heated by a graphite resistance element, fabricated from bar-stock ATJ graphite, which consists of a cylinder milled to produce two concentric spiral conductors with origins and terminations spaced radially at 180°. Two annular projections at the bottom of the heating element fit into clamps on two water-cooled, threaded current posts for electrical contact. Tapered sections of the posts are fitted with tapered Teflon sleeves to furnish electrical insulation as well as a vacuum seal where the posts pass through the base plate. Below the base plate, the posts provide water and electrical connections.

Power for the heating element is provided by a 600-amp transformer and rectifier with a 400-v variable input to the primary. The power requirement (3300 w at 1500°C) is not considered excessive for a furnace of this size.

Production Hot Press

The production hot press (Figs. 3, 4, and 5) consists of three components: (1) the frame assembly, in which the furnace can be tilted for loading and which contains the hydraulic ram; (2) the furnace, which includes the radio-frequency work coil, the graphite susceptor, and the furnace insulation and shell; and (3) the die column, which includes the die assembly, plungers, and push rods.

Frame Assembly

The frame assembly is designed so that the furnace (and die assembly) can be tilted to a horizontal position for loading and unloading the press. This arrangement reduces the vertical reach required of the manipulators during operation of the press. Views of the furnace in the horizontal and

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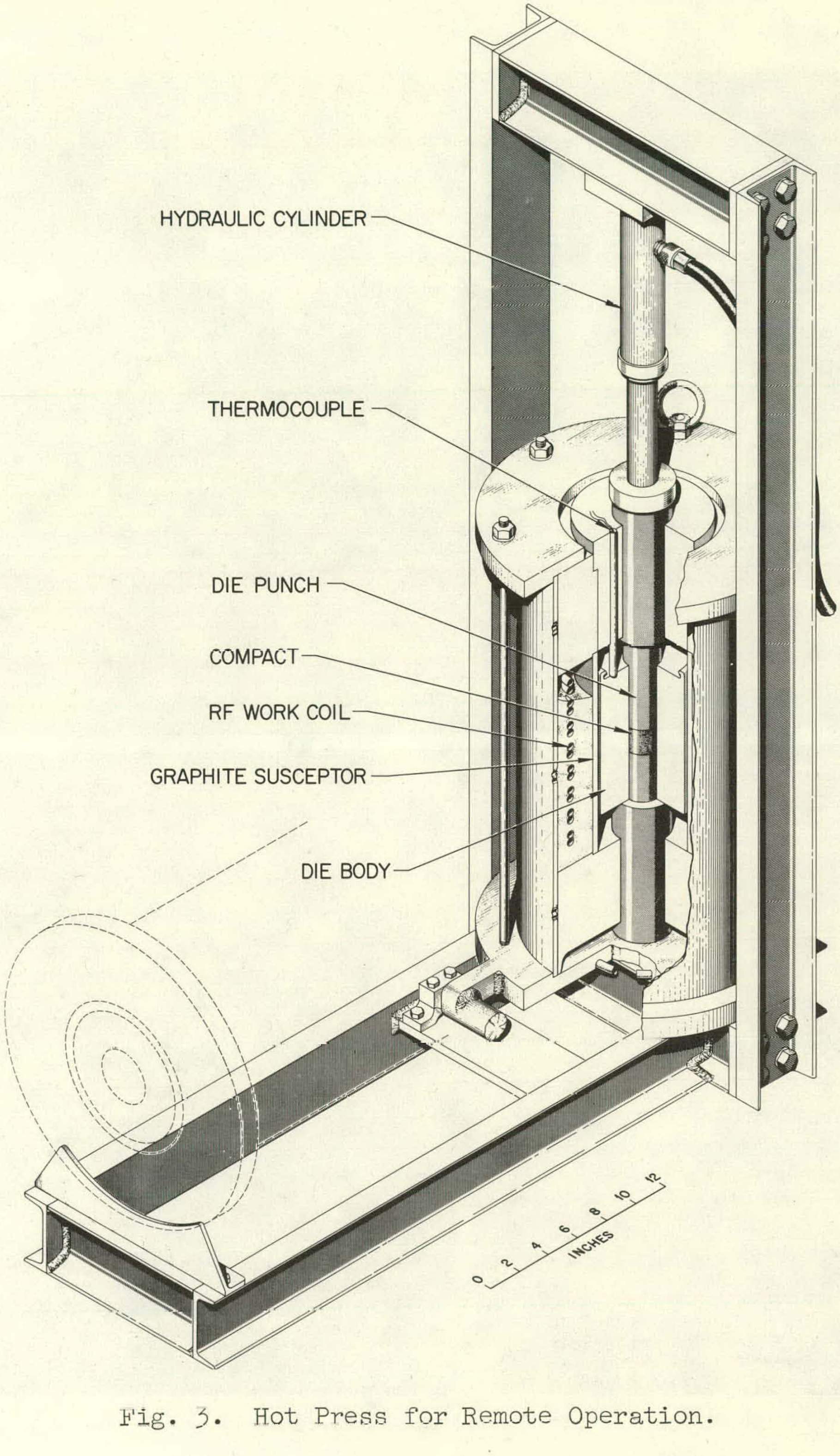

Fig. 3. Hot Press for Remote Operation.

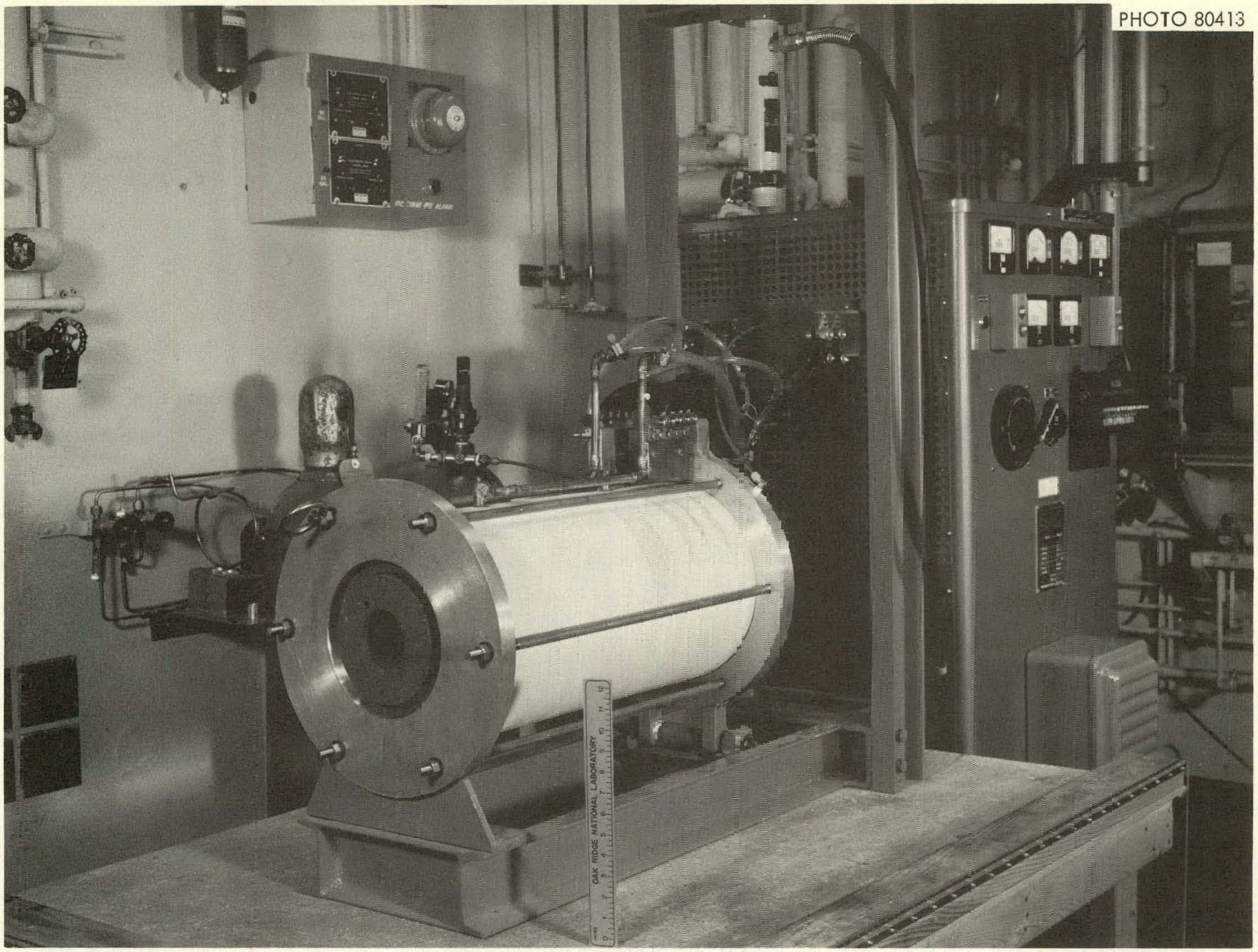

Fig. 4. Hot Press in Horizontal Position.

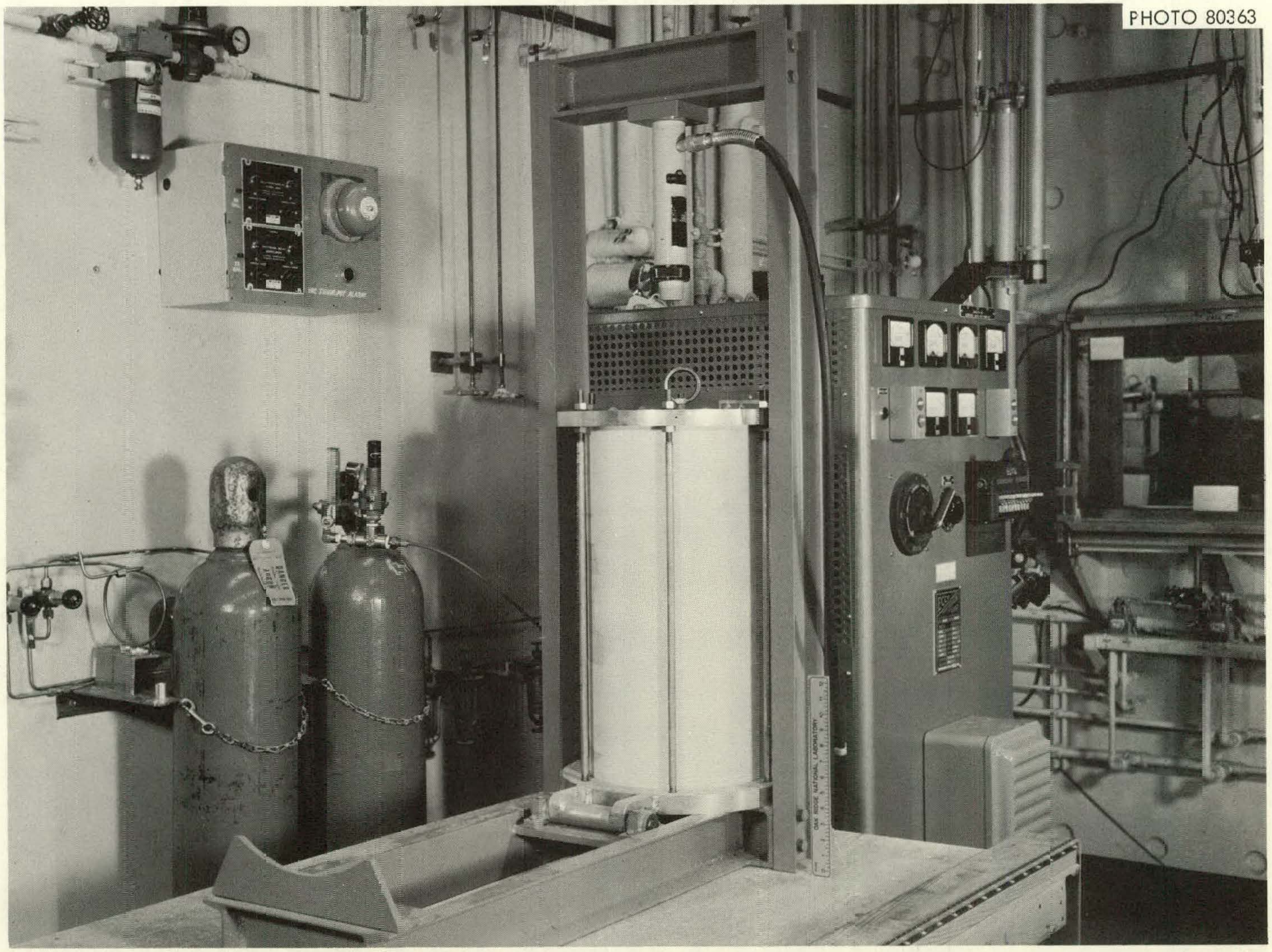

Fig. 5. Hot Press in Vertical Position.

vertical positions are shown in Figs. 4 and 5, respectively; pressing operations are carried out in the vertical position.

Furnace

Electric power is supplied to the furnace by a 20-kw, high-frequency (10,000 cycle) motor generator unit. The radio-frequency conductor is a dual spiral coil, 9-1/2-in. ID by 10-5/8-in. height, constructed of 3/8-in.-dia copper tubing. A graphite susceptor, 6-7/8-in. OD x 6-1/8-in.-ID x 12-in. height, converts the high-frequency electrical energy to heat. Heavy duty flexible leads prevent disconnection of the electrical leads when the unit is moved from the vertical to the horizontal position.

The temperature is measured with a Pt-10% Rh thermocouple inserted in the high temperature zone. The jacket of the furnace is fabricated from a nonconductor to prevent coupling to the work coil; the jacket also confines the carbon insulating material. The furnace assembly is equipped for purging the heated zone with inert gas.

Die Column

The compacting die consists of a high-density graphite body and two graphite punches. The 6-in.-dia by 8-1/2-in.-high graphite body is equipped with a flange on the top for convenient removal from the hot press furnace. The punches are right cylinders; the long punch is 4 in. and the short punch is 2-7/8 in. The pressing force to the die assembly is supplied at the top of the frame by a 10-ton hydraulic ram with a 6-in. travel and spring return. The total force required to produce the compact is determined by the inside diameter of the die body. With a 2-1/4-in. ID, compacting pressures up to 4000 psi are used. The maximum inside diameter that has been used is 3 in.

OPERATION

Both the miniature hot press and the production press have been used to compact a variety of materials. Graphite dies or graphite dies lined with refractory metals (W, Mo, and Pt) or alumina have been used in all applications. A typical alumina-lined die assembly for the production press is shown in Fig. 6. Sufficient space at room temperature must be allowed between the alumina liner and the graphite die body to provide a 1-mil clearance at operating temperature. Similar alumina-lined dies are used with the miniature press. The miniature press will compact samples in the range of 1/4 to 1/2 in. in dia and the production press in the range of 3/4 to 3 in. in dia.

The highest temperature at which the presses have been operated is 1800°C. The limit on the maximum temperature in the miniature press is determined by the effectiveness of the heat shielding and water cooling. The heat shielding for this apparatus was designed for operation at $\leq$2000°C in order to permit relative ease of assembly and disassembly. Similar carbon resistance furnaces with more elaborate heat shields have been operated at temperatures up to 2700°C. The power required to attain a given operating temperature in the miniature press is shown in Fig. 7. The heating cycle requires 16 min, and the press cools in 13 min. The temperature limit of the production press is fixed by the 20-kw power supply. A hot-pressing cycle with the production press requires ~8 hr.

The miniature press can be operated either in an inert atmosphere or in a vacuum. A small mechanical pump maintains an internal pressure of a few torr. The production press can be operated at atmospheric pressure with an argon purge.

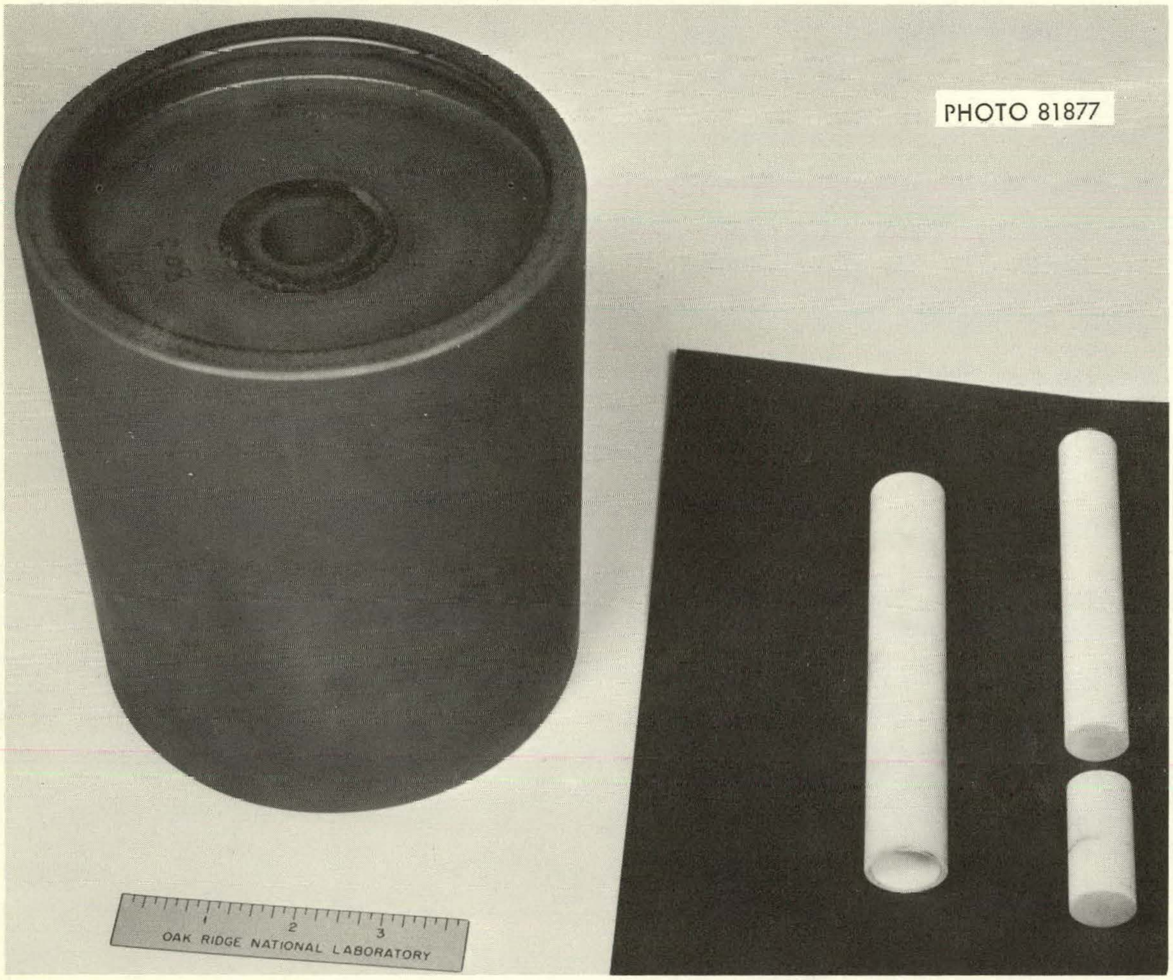

Fig. 6. Alumina-Lined Die Assembly.

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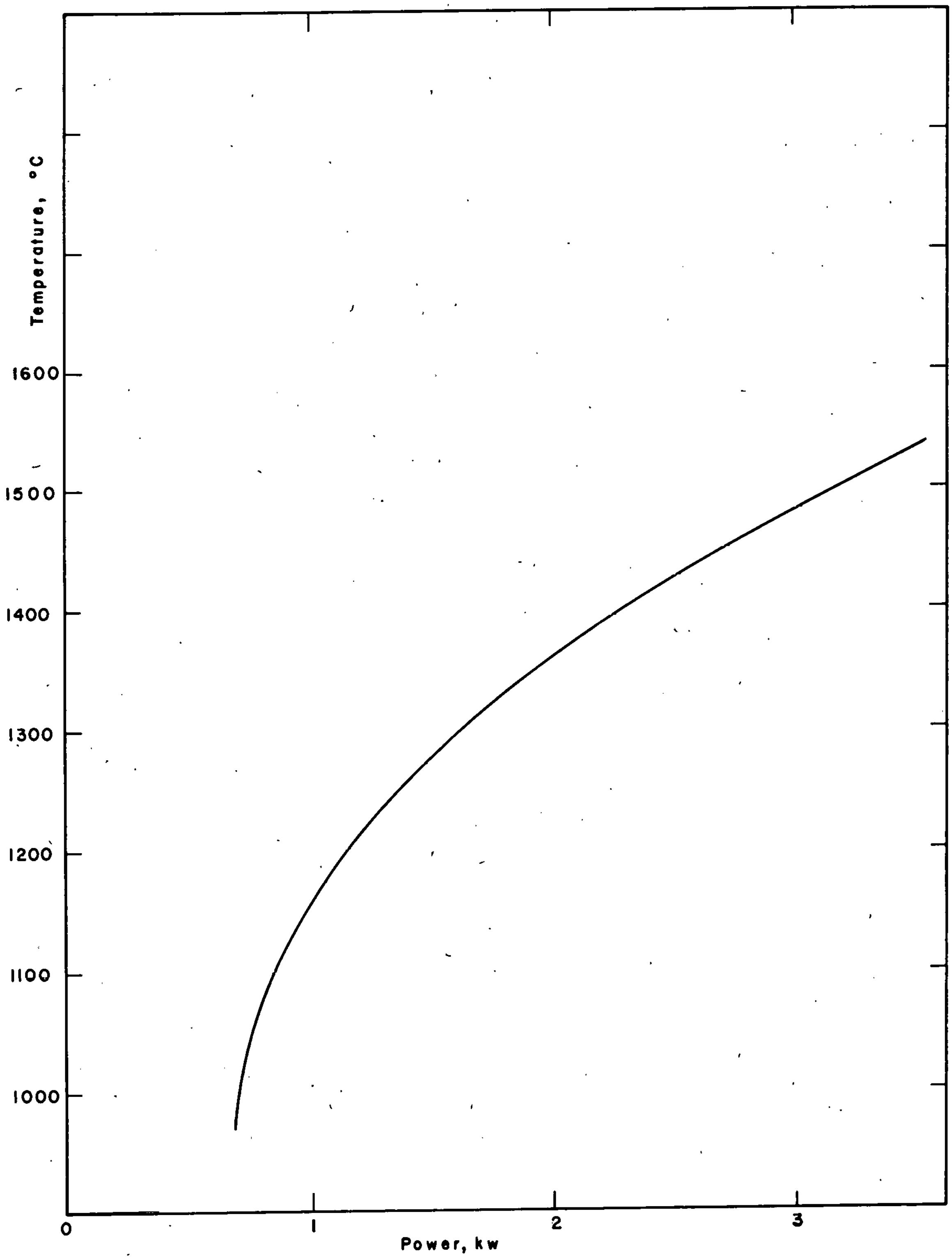

Fig. 7. Power Curve for Hot Press Heating Element.

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