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HYDROSTATIC PRESSING OF METAL POWDERS

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TABLE OF CONTENTS

ABSTRACT

Hydrostatic pressing was investigated as a method of fabricating long preforms made from uranium powder. A laboratory scale pressure vessel was constructed to evaluate the feasibility of this process. Various metal powders were loaded into flexible molds and subjected to pressure in an enclosed liquid. The high density of uranium caused the flexible molds to distort during filling and after pressing there was a tendency for the compact to adhere to the mold. Methods of minimizing these difficulties are suggested.

Uranium powder, compacted in a plastic mold at 21 tsi had a density of 12.5 g/cc. During the course of the investigation, it was found that hydrostatic pressing of other metal powders presented advantages over conventional steel die methods, especially in the ability to press experimental shapes using economical equipment. Recommendations were made for further development work on both cold and hot hydrostatic pressing.

I. INTRODUCTION

Usual powder metallurgical methods have been economically successful in the production of metal compacts of relatively small shapes. Larger sections require increased press tonnage and complex dies. In order to achieve uniform density throughout a relatively large compact, double or multiple acting dies must be employed, thus presenting a costly die machining operation. In addition, parts pressed in steel dies must be designed so that they can be ejected easily from the die. This eliminates the possibility of pressing pieces which have undercuts, reentrant angles, transverse holes or threaded sections.

Hydrostatic pressing provides a means of distributing uniform pressure to all surfaces of a compact by immersing a flexible mold containing powder in a suitable liquid, then applying a force to the enclosed liquid. Since there can be no shearing forces in a liquid, the pressure is transmitted undiminished to all parts of the mold. Eliminating the use of costly steel dies by employing cheap rubber or plastic molds permits the inexpensive evaluation of the feasibility of producing parts from various metal powders. Hydrostatic pressing can thus be considered a valuable tool in laboratory development work.

H. D. Madden[1)] and others[2,3)] have used variations of the hydrostatic pressing principle for compacting metal powders as early as 1913. This previous work however, was done using rubber molds and simple cylindrical shapes. This report describes the use of rubber and plastic molding in various shapes including simple cylinders and more complex sections.

The equipment requirements for a laboratory scale hydrostatic pressing unit are not elaborate. A steel pressing chamber with suitable closure and seal, and a method of pressure application are the essentials of a cold pressing unit. Hydrostatic pressing at elevated temperature would require a method of heating either the fluid or chamber, or both. Pressure application in this case would become a more difficult problem and special high strength, high temperature steels must be selected. The design of a hot hydrostatic system will be more thoroughly discussed in section III (B).

The purpose of this report is to relate the progress made using a laboratory scale cold hydrostatic pressing unit. The project was carried out as part of a program for the development of a method for the production of a long uranium flat-plate or tubular preform.

II. APPARATUS AND PROCEDURE

The equipment used in this investigation was designed for cold pressing only. The pressure vessel was machined from an SAE 4130 steel forging. After rough machining, the cylinder was heat treated to 25-30R_c, giving a theoretical yield strength of 50,500 psi. A scale drawing of the cylinder with dimensions is shown in Fig. 1.

The closure-seal assembly was effected by using a two-piece piston arrangement[4], machined from SAE 4340 and hardened to 50R_c (see Fig. 2.) Between the two-piston sections, an unsupported-area type gasket was used to maintain a pressure-tight seal. Combinations of neoprene rubber with teflon, copper and brass flat washers were tried and considered unsatisfactory because of extreme extrusion of the rubber. The seal design finally decided upon consisted of a 3/8-inch brass gasket with a small ridge cut to carry a rubber O-ring. (See Fig. 3). In operation, the action of the bottom section of the piston forcing the gasket against the top section caused the rubber to press outward against the cylinder walls, thus forming and maintaining a suitable seal until pressure was released. Both sections of the piston were provided with a threaded hole so that they could be removed with a screw jack.

Pressure measurement was accomplished by means of a Baldwin SR-4 strain gauge mounted on the cylinder by a 3/16-inch high pressure fitting and connected to a modified bridge strain recorder. Maximum output from the press used was found to correspond to 42,000 psi inside the chamber and this pressure was used for all experimental pressings.

The entire press assembly was mounted on a portable truck which could be placed in a 300-ton hydraulic press. Fig. 4 shows the mounted assembly in front of the press.

Both SAE 10W motor oil and water-soluble cutting fluid were used as liquid media with satisfactory results. The cutting fluid, diluted with ten parts water was ultimately chosen because it was cleaner and could be handled more easily.

One of the prime considerations in this project was the selection of a proper material for the mold to contain the powder. In general, the mold had to conform to several basic requirements. It had to be rigid enough to hold its shape within reasonable limits after being loaded with powder, but with sufficient flexibility to allow full and even compression upon the application of pressure. In addition, if the mold had any tendency to adhere to the pressed compact, it could not be strong enough to break the compact by returning to its original size after the release of pressure. Several molding materials as described below were tested, each being ultimately chosen for a particular application because of certain characteristics. Plastisols were used where strong, rigid films were needed. Latex and Gooch tubing were employed where soft elastic molds were necessary. Nuplamold was used to obtain a very soft and weak mold.

Plastisol was a liquid type molding agent consisting of a dispersion of thermoplastic resins in a system of plasticizers. It was fluid at room temperature and formed a flexible film upon the application of heat. Complete fusion occurred at 350°F and, upon cooling to room temperature, a tough, flexible film was formed conforming to the shape of the body upon which it was molded. By altering the chemical composition, it was possible to obtain variations in hardness and tensile strength. Molds were usually made by dipping.

Latex, as used in these experiments, consisted of a natural rubber dispersed in about 60% water containing a small amount of ammonia. Molds were produced by painting, spraying, or dipping. Thick coats were produced by dipping a metal form first into a coagulant, then into the latex. A soft, flexible film resulted when the water evaporated. Extra toughness could be obtained by curing the mold at 150°F for several hours.

Another plastisol compound, made by Luckey Laboratories and known as Nuplamold, was used in a few instances where casting was necessary. This plastisol was a very soft, rubbery material supplied as small pellets. It melted at 350°F and could be cast around the model. The resulting mass, when cooled to room temperature, was a soft, flexible mold which had to be used in very thick sections for any reasonable strength.

Several prefabricated tubings were used for small cylinders only. Gooch tubing is a thin, surgical rubber. Vacuum tubing is a soft reinforced rubber, usually supplied in thick sections. Polyethylene, a plastic material, was used in very thin sheeting.

The production of a cylindrical-shaped mold was a simple operation using almost any of the above molding agents. For cylinders over one-inch in diameter, plastisol was found to be most desirable because of its superior strength. In general, the method of mold preparation was as follows:

A brass or aluminum form was machined to the desired size and shape. Aluminum was preferred because its low density made it easier to handle when hot. The form was cut about six inches longer than the desired mold length to permit handling and cutting off excess.

This mold form was heated to 350°F, then dipped into liquid plastisol. In most cases a dip of one minute, giving a thickness of about 1/16-inch, was sufficient. For heavier sections, the form could be reheated and dipped again, thus building up successive layers. After dipping, the form was returned to the oven and baked at 325°F until a slight color change was noticed, indicating complete fusion. Cooling was accomplished by water quenching.

After being stripped from the form, the mold was filled with metal powder and sealed. A suitable seal was effected by cementing a rubber stopper into the top using a plastic cement.

Plastisol molds, having good rigidity, were simply filled with metal powder and placed into the pressure vessel. Other types of molds, being soft and flexible had to be suspended from a wire rack placed in the vessel.

III. DISCUSSION

A. Cold Hydrostatic Pressing

The attempt to produce a thin, rectangular plate comprised one of the efforts in the hydrostatic pressing program. Because of the large amount of uranium powder contained in a long plate (about 70 pounds) a heavy plastisol mold was decided to be the only reasonable container for the powder. As would be expected thin wall molds of a soft material deformed under the weight of the metal powder, and consequently the pressed compact was misshapen. Increasing the rigidity of the mold, by using more rigid mold materials, or by using thicker wall molds resulted in better shape and dimensional reproduction. This improvement could also be had by using a perforated metal container around the mold which served to maintain the mold configuration. The perforations or holes still permitted the hydraulic pressure to act upon the flexible mold.

At the top pressure attainable with the available facilities, a density of 12.5 g/cc was realized in the compacts. At this density uranium is pyrophoric and must be handled in a protective atmosphere. Coupled with the problem of mold adherence this made handling difficult.

Another part of this program was the attempt to hydrostatically press a hollow cylindrical uranium preform for ultimate use as a clad fuel element. Plastisol again was decided upon for a molding agent and was prepared in the usual way using an aluminum hollow cylinder as a molding form. Sealing was accomplished by using a plastic gasket cemented into both ends.

Later, the sealing process was modified for easier dry-box loading. The molding form was altered by placing three small rods on the top end. The mold thus produced had three hollow tubes leading into the powder cavity (Fig. 5). In loading, the powder was delivered to the mold through the three top tubes by means of a three-necked funnel. Sealing was then completed by melting the top of the four tubes with a soldering iron. The altered design permitted a more even distribution of powder. Fig. 5 shows a plastisol mold and a pressed cylinder.

One unique pressing problem was encountered in the hollow cylinder program. Since a constant pressure was exerted on all surfaces of the body being pressed, and less area was present on the inside surface of a hollow cylinder, the entire assembly was compacted toward the central axis. It was necessary, therefore, to obtain a mold whose inside wall would have optimum strength, namely enough to hold the powder rigid before pressing but not enough to crack the compact when pressure was released by returning to original size. A 1/16-inch mold wall was found to have both these characteristics. Again however, the adherence of uranium powder to the mold and its low density made the compacts difficult to handle.

Adherence of the compacted metal powders to the mold wall was encountered only during the pressing of uranium powder. This was partially attributed to the large particle size (-12 mesh) of the uranium. Under pressure, the soft mold material extruded between these particles and thus formed a mechanical bond. Adherence was most pronounced when soft mold materials such as latex or Nuplamold were used. The use of the plastisol molds which were harder, and the use of smaller particle sizes (-100 mesh), minimized the problem. It is believed that the use of coatings on the inner surface of the molds would also be beneficial, and aid in removal of the compact.

Cold hydrostatic pressing has definitely been proved an alternate compacting operation which could conceivably be adapted to many uses. In an experiment to compare the properties of hydrostatically pressed compacts with those pressed in a cold steel die, identically sized samples were pressed by both methods using several metal powders. Transverse green strength and density were measured, and the results are shown in Table I. This table shows that hydrostatic pressing results in general are better than those of samples compacted in steel dies, due probably to the fact that while the same unit pressure was used in both cases, friction losses were lower in hydrostatic pressing. Although no data is available concerning uranium powder cold pressed in a steel die at 21 tsi, it was found that a density of 12 g/cc was obtained when compacted in a steel die at 50 tsi as compared to 12.5 g/cc when compacted at 21 tsi hydrostatically. Observation of these compacts showed that, when compacting small particle size metal powder, the internal surface finish and contour of the mold was reproduced exactly in the sample. For example, in the event that there was an open bubble on the internal surface which was formed during the curing of the plastic or latex mold, a protuberance of the same shape was obtained on the sample. This would indicate that molds of any internal shape could be utilized to produce compacts of a complex shape and non-uniform cross section.

Hydrostatic pressing could conceivably be valuable in the compacting of intricately shaped objects, especially in the field of high temperature refractory materials which must be fabricated by powder metallurgy methods. At present, these parts are fabricated by:

a) pressing a large block
b) presintering for machining strength
c) rough machining to approximate shape
d) final sintering
e) final machining or grinding.

Hydrostatic pressing would be desirable from an economic viewpoint since a compact could be prepared close to the desired finish size so that only final sintering and grinding would be necessary.

B. Hot Hydrostatic Pressing

In conventional steel die pressing the pressure applied to the compact serves to mold the powder particles into a coherent shape by creating strong contact particle bonds. For most applications however, the cold pressed compact has insufficient strength and density and a sintering operation is required to produce the properties desired. If the heat is applied before and during the pressing operation, the process is known as hot pressing, and parts prepared in this manner may have properties superior to those prepared by cold pressing and sintering.

It is a reasonable assumption that the above principles of hot pressing as compared to cold pressing should hold true of hydrostatic compacting as well as conventional steel die pressing. In addition to the pressing of metal powders, hot hydrostatic pressing equipment should be suitable for the cladding of bare fuel elements. The use of hydrostatic pressure would provide uniform pressure on all surfaces regardless of the shape of the piece. Mold materials necessary to contain the metal powders would be dependent upon the temperature of operation required. Materials evaluated for cold hydrostatic pressing may be suitable for slightly elevated temperatures, up to 200°C. For higher operating temperatures, it will probably be necessary to use metal molds which will deform properly under pressure. This suggests that hot hydrostatic pressing using metal molds could be a method to produce clad components, in which case the desired cladding material would be used as the mold. This method of cladding could possibly be utilized for simultaneous pressing and cladding of metal powders, or for cladding of parts produced by other methods, such as by rolling or casting. Because hydrostatic pressing provides uniform pressure on all surfaces, this could be an ideal method for cladding tubes or pieces having complex shapes.

A brief analytical evaluation of the feasibility of constructing and operating such a unit has been made. The essential units of a hot, hydrostatic pressing bench-scale pilot plant are the vessel, pumping system, control and indicating instruments, valving and heating units. The major components of these elements will be discussed.

A typical process flow diagram of the unit would be similar to that shown in Fig. 6. The fluid medium is pumped into the vessel; heating is accomplished with a furnace. Temperature in the vessel is kept constant by means of a controller regulating current flow to the furnace heater elements.

The pressure vessel must withstand a design pressure of 21,000 psi at a design temperature of 1150°F. Investigation shows that Allegheny-Ludlum A-286 high strength austenitic steel is most suitable. Allowable stress is based on a value of 2/3 the stress rupture for 1000 hours, giving a safety factor of 1.5. The hot wall-type vessel is considered more favorable than other designs. Since at high temperature a plastic state of stress exists in the cylinder wall, the Bailey-Nadai[5)] theory is preferred in calculating wall thickness.

$$O.D. = I.D. \left[\frac{1}{\left(1 - \frac{\beta P}{\sigma}\right)} \right]^{1/\beta}$$

Where $\beta = 0.4$ (exponent in analytical expansion for shear stress-strain relationship)

O.D. outside diameter

I.D. inside diameter

σ allowable working stress with safety factor (39,800 psi AL-286)

P internal working pressure (21,000psi)

For an I.D. of 4.00 inches, (the I.D. of the present cylinder) the thickness required is 1.615 giving an O.D. of 7.23 inches.

The seal design in high temperature-high pressure equipment is extremely important. The design considered satisfactory is a modified Bridgman closure,[4)] used in a threaded nut or breech block. (Fig. 7). It is self-sealing due to internal pressure, intensifying the unit load and forcing the floating head against the nut, thus squeezing a triangular gasket between the head and cylinder wall. Monel seal rings should be employed, since they can be used many times without machine dressing and have greater strength and elasticity than copper.

The hydrostatic fluid to be used in hot pressing could be either water or gas. Other fluids considered for hot pressing included molten metals, organic compounds and heat treating salts. In addition to having the properties needed for this application, pumping and pressure intensification problems are simplified with water or gas. Water can be easily compressed cold, and heats readily, thus simplifying pressure intensification and heating problems. It is, however, very corrosive at high temperatures and pressures. On the other hand, a gas such as helium, nitrogen, or argon can be considered non-corrosive and relatively easy to handle, but presents a need of more elaborate equipment for obtaining the pressure desired.

Pressure intensification would be provided by the use of a hydraulic intensifier in conjunction with a low pressure pump. The intensifier considered here is a double acting Harwood model DA-10G which is capable of developing 30,000 psi, delivering fluid at a rate of one gallon per minute. If water were used as a fluid, the intensifier would deliver directly to the vessel while the use of gas would involve pumping oil to a separate closed chamber containing oil and gas. Pumping more oil into the chamber would intensify the pressure of the gas which in turn would be delivered to the pressure vessel.

Heating the assembly in either case would be done simply by using an induction heater around the vessel. Induction heating has been considered most feasible since the furnace utilizes the steel being heated as the secondary of a transformer: the primary source connected to the supply. Uniform heat distribution is the main advantage of this system since very small thermal stresses would result when only a slight temperature gradient is present in the shell. A recording controller would be sufficient for temperature control.

Valves, tubing and fittings must withstand the design temperature of this system. Due to the excessive temperature, high strength tubing must be employed. Valves and fittings are commercially available and should pose no problems in procurement.

IV. SUMMARY AND RECOMMENDATIONS

Equipment for hydrostatically compacting metal powders is described. Some difficulty was encountered in compacting uranium powders, and methods of minimizing the difficulties were indicated. Additional development work on mold materials for cold hydrostatic pressing is required in order for this process to be suitable for cold compacting uranium powder. In general, the pressed density and green strength of hydrostatically compacted metal powder was higher than for similar samples compacted in steel dies.

An analytical study of the feasibility of constructing equipment for hot hydrostatic pressing was made. It is recommended that a unit for this work be constructed in order to evaluate hot hydrostatic pressing as a method for cladding fuel elements and for compacting metal powders to full density.

TABLE I

COMPARISON OF PROPERTIES OF HYDROSTATICALLY PRESSED VERSUS COLD STEEL DIE-PRESSED SAMPLES AT 21 TSI

Metal	Pressing Method	Modulus of Rupture (psi)	Density (g/cc)
Copper	Hydrostatic	3255	6.64
	Cold Pressed	2890	6.28
Aluminum	Hydrostatic	2815	2.57
	Cold Pressed	3400	2.36
Nickel-Silver	Hydrostatic	1725	6.76
	Cold Pressed	1375	6.14
Uranium	Hydrostatic	--	12.5
	Cold Pressed	--	12.0 (1)

(1) Pressed at 50 tsi

REFERENCES

1. Madden, H. D., "Process of Preparing Billets of Refractory Material", U.S. Pat. 1,081,618, December 16, 1913

2. Lidman, W. G., and Hamjian, H. J., "Properties of a Boron Carbide-Iron Ceramal", NACA TN-2050, March 1950

3. Blackburn, A. R., and Shevlin, T. S., "Fundamental Study and Equipment for Sintering and Testing Cermet Bodies: V. Fabrication, Testing and Properties of 30 Chromium-70 Aluminum Cermets., J. Am. Ceram. Soc. 34 (11) 327-331, (1951)

4. Bridgman, P. W., "Physics of High Pressures", G. Bell and Sons, Ltd. London, 1945, p. 39

5. Buxton, W. J. and Burrows, W. R. - Trans ASME 73 575-87, (1951)

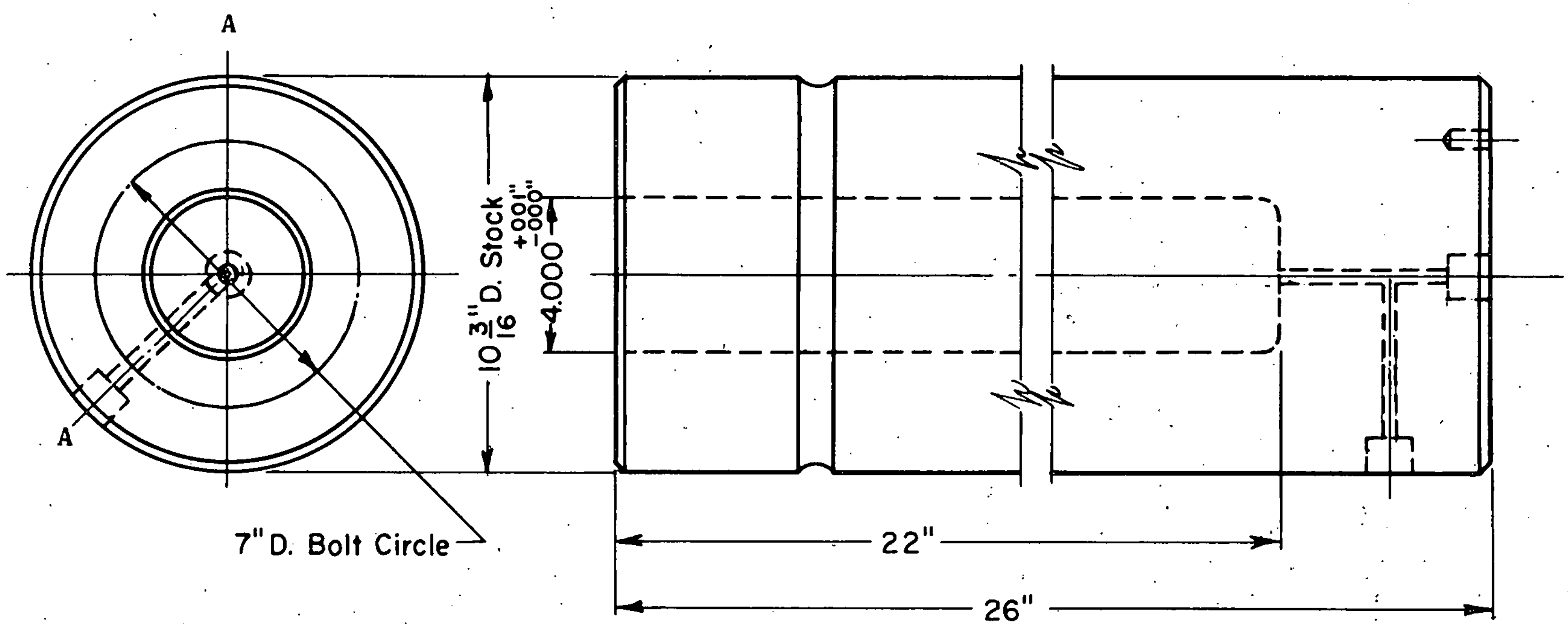

Fig. 1 - Cylindrical Test Chamber

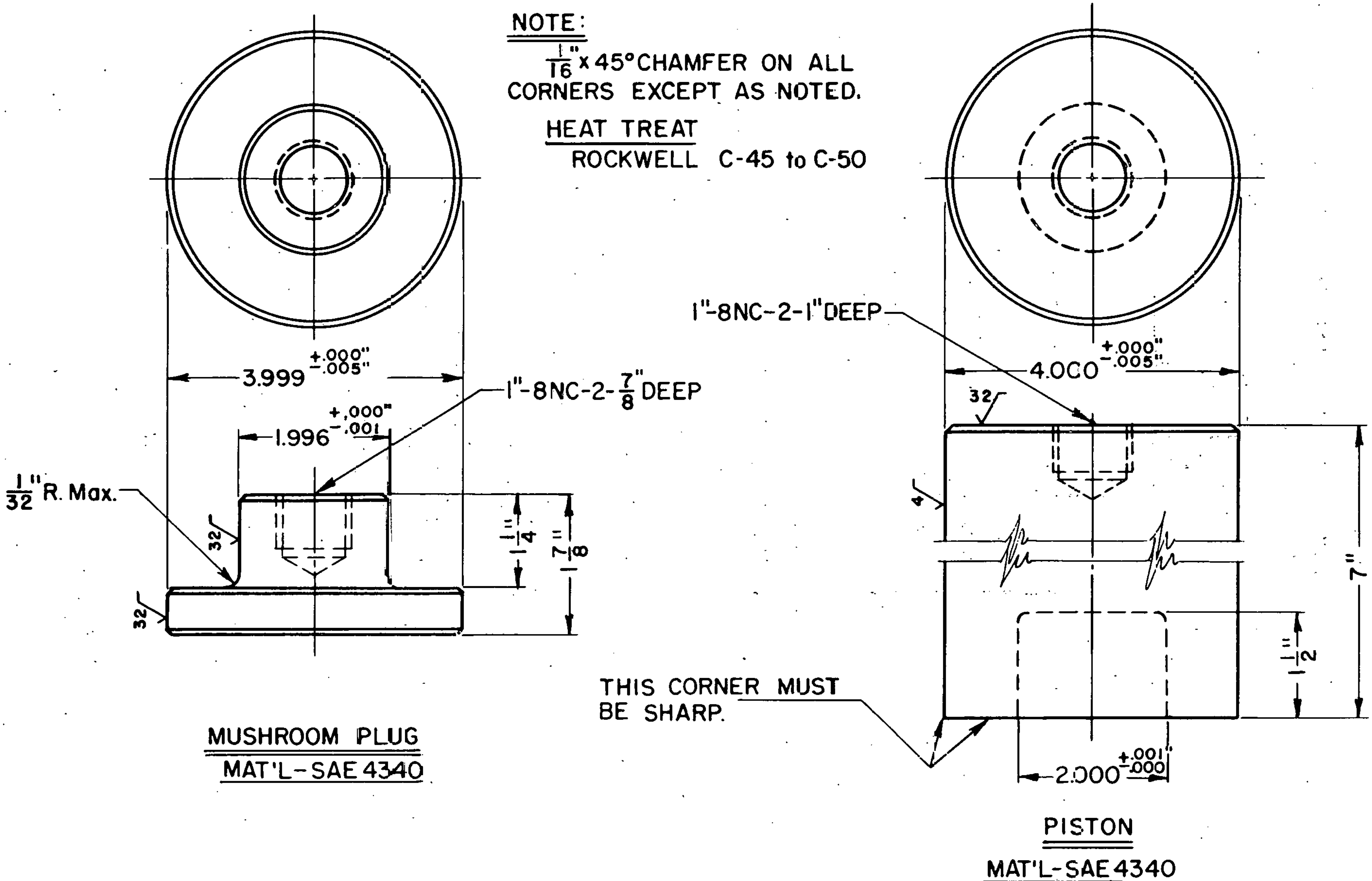

Fig. 2 - Two piece piston used as part of sealing mechanism.

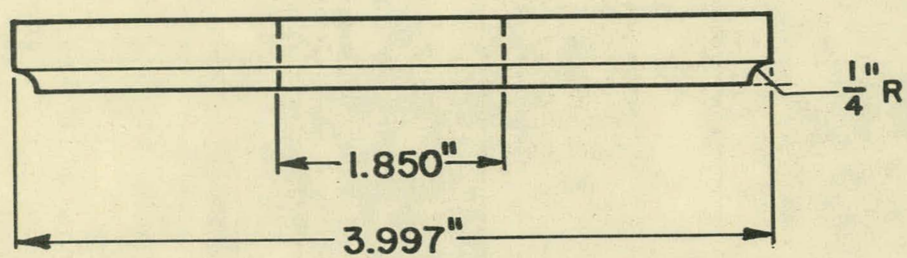

Fig. 3 - Spacer and holder for O-ring gasket for cold hydrostatic pressing unit.

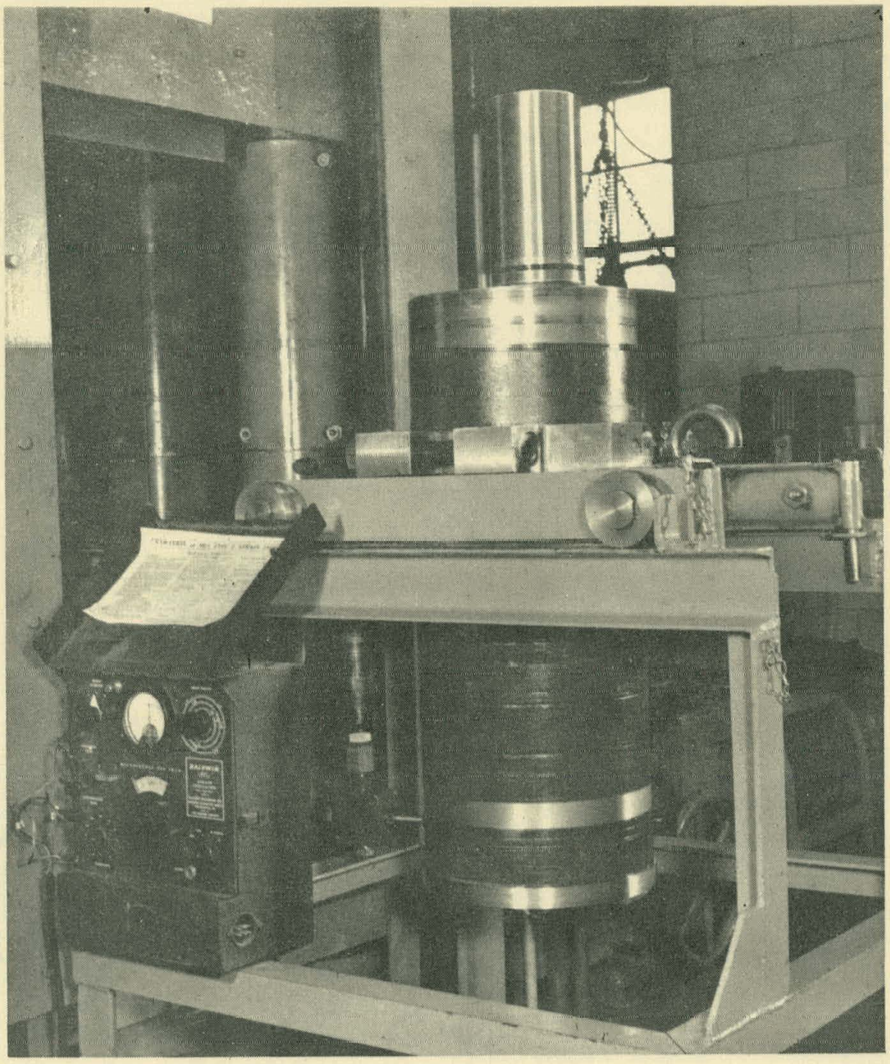

Fig. 4 - Hydrostatic pressing assembly, 300-ton press shown in background.

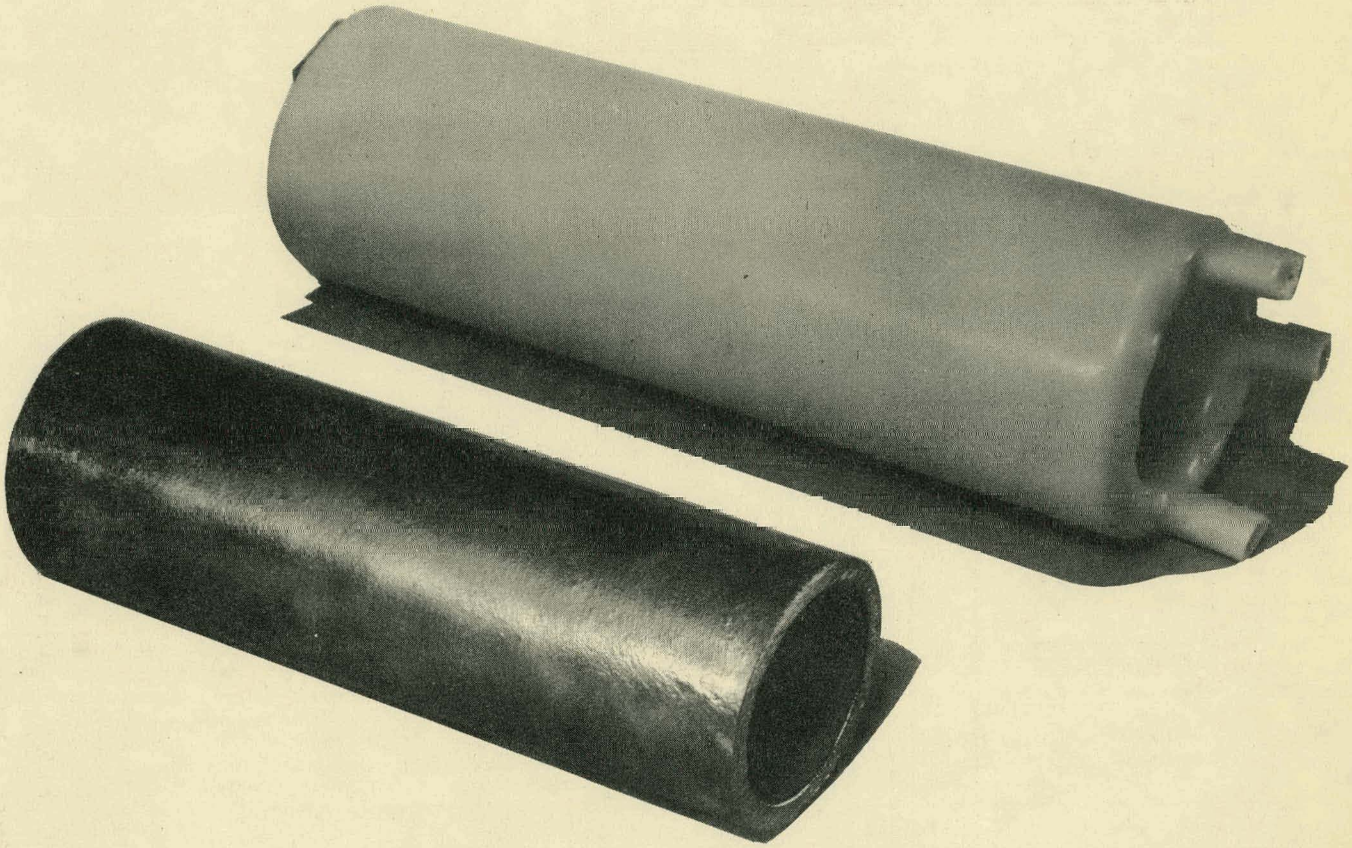

Fig. 5 - Hydrostatically compacted tube and plastic mold used in pressing. Approx. half size

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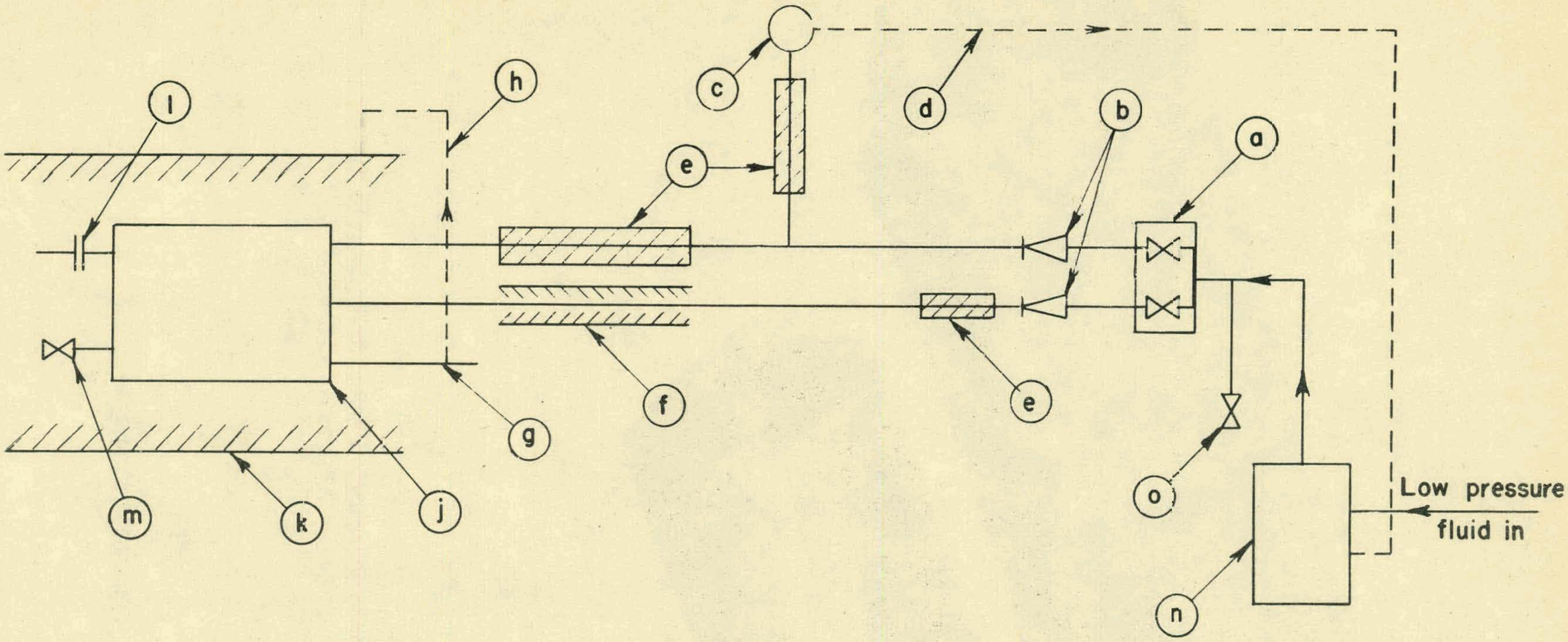


(a) combined inlet and bleeder valve

(b) check valve

(c) pressure gauge and regulator

(d) pressure regu ator sensing line

(e) jacketed cooler

(f) preheater

(g) thermocouple and controller

(h) temperature controller sensing line

(j) pressure vessel

(k) furnace for vessel

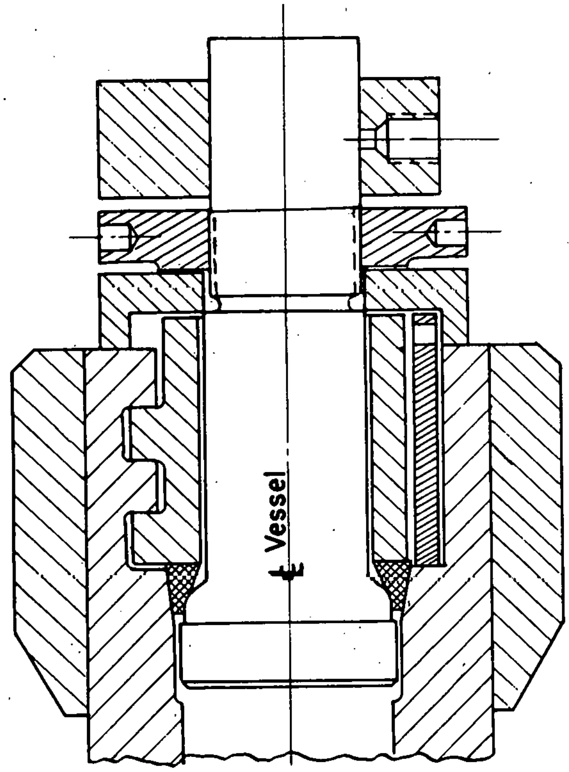
(l) safety head

(m) throt-ling valve

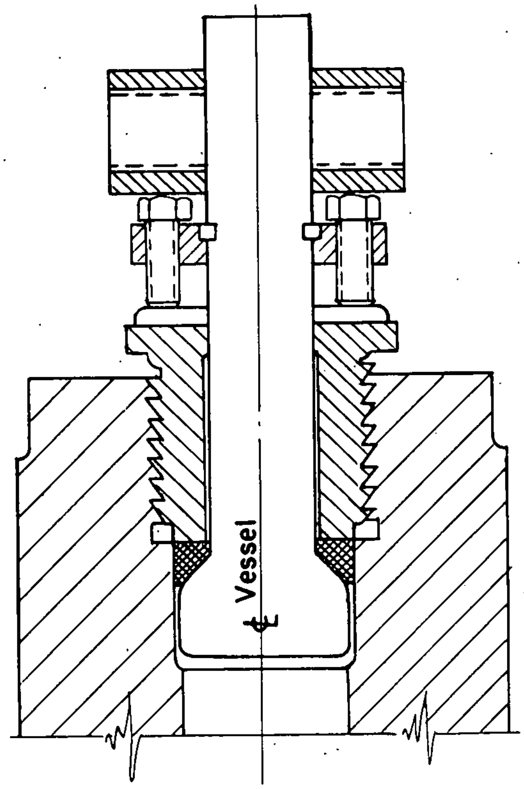
(n) pumping unit

(o) dump valve

Fig. 6 SCHEMATIC FLOW DIAGRAM



(b) Breech Type Enclosure



(a) Threaded Nut Enclosure

Fig. 7
Suggested closures for hot hydrostatic pressing chamber.

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