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DEVELOPMENT OF GAMMA-PHASE HOT-PRESSING OF URANIUM

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ABSTRACT

The hot-pressing method for forming uranium is described in detail, giving data on temperatures, pressures, die materials, tolerances, etc. This method is primarily of interest for forming very simple shapes, such as solid, or thick-walled hollow hemispheres, etc., and is not feasible for moderately complicated shapes, such as octants of spheres and other shapes of low symmetry. It is not a high-production method, and cannot compete with precision casting where many pieces are required.

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DEVELOPEMENT OF GAMMA-PHASE HOT-PRESSING OF URANIUM

INTRODUCTION

By gamma phase hot-pressing of uranium is meant pressing in a suitable die in the temperature range of 770°C to the melting point, the range in which uranium is in the soft, ductile, high-temperature gamma modification. In practice there is no need to exceed 950°C, and most pressing is done close to 850°C.

Both ordinary uranium and uranium-235 have been formed by hot pressing. Though there is no difference in behavior, greater care is necessary in handling "25" to avoid loss, even recoverable temporary loss; hence particular attention must be paid to avoiding oxidation.

Hot pressing has been used as an alternative to casting for certain simple shapes such as small discs and solid and hollow hemispheres. The main advantage of hot pressing over casting is the elimination of hot tops with consequent hold-up of material, and of much machining with consequent large temporary and a little permanent loss. The product, moreover, is of greater density and free from blowholes. Another advantage of hot pressing over casting is the shorter time of preparation of the graphite die used, compared to the time required to produce an investment (silica) mold for precision casting. This advantage naturally holds only when one or two pieces are required. When many pieces are necessary, the time involved favors casting. Hot pressing of certain very simple shapes may allow greater dimensional accuracy than is readily possible by casting, except after prolonged trial and error. Lastly, in hot pressing, one does not have to contend with feeding a casting and hence run the risk of having shrinks (depressed areas) which may cause rejection.

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HOT-PRESSING PROCEDURE

Fig. 1 shows a typical die, ordinarily made of machined graphite pieces. The metal of the necessary volume to fill the die cavity is placed in the die, an inert gas, either argon or helium, is passed into the die to sweep out air and maintain a neutral atmosphere, and the die itself with the metal inside is heated to the pressing temperature of about 850°C. High-frequency induction heating is usually most convenient. The die is placed on the bed of a hydraulic press, and when the thermocouple inserted in the punch or die wall indicates the correct temperature, the punch is forced into the die by the press, and held for a few minutes at a pressing corresponding to 1000 to 4000 pounds per sq. in. of projected area. The die and contents are then allowed to cool to about 100°C or below with argon maintained to keep out air. At this temperature, the amount of oxidation occurring on removing the piece is negligible.

The very low pressure required is made possible by the exceedingly low deformation resistance of uranium in this temperature range. It is this low pressure which makes possible the use of graphite for the dies.

It has been found convenient to use a water-cooled copper plate under the graphite die to prevent heating the press bed and to speed up die cooling to some extent, although a stream of air is mainly relied upon for this purpose.

VARIABLES AND LIMITATIONS OF HOT PRESSING

Effect of Blank Shape

By blank is meant the slug of metal placed in the die to be pressed to the desired shape. It has been determined that the blank shape is relatively

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unimportant as long as it is chunky and in one piece. Preferably, folding of the blank should not occur, as this is apt to cause a lamination in the final pressing. Similarly, an extremely irregular piece with re-entrant sections, etc., should be avoided if maximum smoothness is desired.

When cylinders are pressed out to flat discs, it has been found advantageous to round the corners to secure good surface finish of the flat surfaces.

However, irregular blanks, such as the ones with holes drilled in them, have been pressed out, filling up the holes so that it is difficult to see where the holes were.

<u>Dimensional Tolerances</u>

The dimensional accuracy possible in hot pressing naturally depends considerably upon the size and shape of the piece being pressed. In the case of a solid cylinder of 1 to 2" diameter and height, perhaps the easiest of all shapes, an accuracy of diameter of about .003" is possible. In the case of solid or thick walled hemispheres up to about 2.5" diameter, the corresponding accuracy is about .003 to .005". Accuracy is limited by die wear which is serious in the case of graphite, and also by the accuracy of machining graphite which is not as good as in the case of steel. In the case of large pieces up to 6" diameter, metal shrinkage plays an important part, and the reproducibility of contraction between 850°C and room temperature is a limiting factor. Another important point is die finish or smoothness. Obviously, a comparatively rough surface, characteristic of most grades of graphite, does not allow as close dimensions as in pressing in polished steel dies.

Shape Limitations

It is very difficult to press objects which have a high ratio of surface area to thickness. A case in point is the pressing of a thin-walled hemisphere of 2" OD and .080" wall thickness. This represents a limiting shape. Such shells have been pressed but with rather poor dimensional tolerances, at a unit pressure of 5100 pounds per sq. in. which fractured the die.

Apparently frictional effects in these thin-walled shapes are very important, and limit metal flow considerably, hence requiring comparatively high pressures.

Another difficult operation is flattening out a flat wedge of metal of perhaps 1/4" average thickness, but where the thickness varies somewhat from one side to the other. In the case of a metal ring about 4 1/2" OD, 3" ID, and 1/4" thick, it was found impossible to make the thickness uniform when the maximum to minimum ring thickness varied on opposite sides by about .030". Here again, metal flow was apparently inhibited by friction of the die walls around the ring.

Theoretically, by the use of specially shaped blanks, pieces could be made with cored holes by the use of graphite inserts, and drilling or chipping out the graphite cores after pressing. The only work done along this line was the ring described above. It would probably be impractical in most cases, especially with small holes, because of the fragility of the cores and the difficulty of preparing a suitable blank.

Generally speaking, hot pressing is limited to rather simple shapes, free of undercuts and asymetric sections.

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Scrap Production, Including Flash and Oxidation

In most cases, flash (metal squeezed between die and punch walls) is not important in a well-made die with good fit between the component parts.

Oxidation is more a source of concern, together with rough surfaces. Obviously, in a die with poor surface finish, there is more surface area to be oxidized, and also a rough surfaced die allows more air to enter, because the fit between punch and die is poor.

It has often been considered that another source of oxidation, particularly in the case of the coarser-grained graphites, is air absorption on the surface of the die. This point has never been thoroughly checked. However, it has been shown that very fine-grained grades of graphite not only produce a smooth surface, but show much less oxidation. This, however, may be because a smooth surface does not form as much oxide as a rough surface.

Depending upon the size of the piece being pressed, the loss due to oxidation (most of which is recoverable) is nearly always less than 1 per cent, and with pieces weighing 300 to 600 grams, the loss is usually of the order of 0.5 per cent. The per cent loss naturally depends on the ratio of surface to volume.

Gas purity of the argon or helium is a very important factor in oxidation. For minimum loss, the gas is purified of oxygen, nitrogen, and water vapor by passing it over uranium chips heated to 600°C.

Pressing Temperature

While any temperature over 720°C (the beta-to-gamma transformation point)

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should be hot enough, in practice it has been found that a temperature of 850°C is satisfactory. Because of some temperature lag between die and metal, and because the thermocouple junction cannot be embedded in the metal itself, it is necessary to exceed the minimum possible temperature by a safe margin to avoid pressing during the beta-gamma transformation. This has frequently happened when attempting to press at or about 800°C, with the result that part of the metal flows, while other portions do not. Successful pressings have been made in the range from 850° to 925°C with little noticeable difference in behavior.

Fig. 2 shows the effect of varying temperature in pressing uranium in a flat faced cylindrical die, in which the metal was pressed but not "coined". That is, the metal was not forced out to the die wall, but allowed to flow edgewise freely except for surface friction. The pressure on the original samples, (0.50" in diameter and about 0.42" high) was 1500 pounds per sq. in. The same load was maintained until flow ceased, when the final area was measured and the final pressure computed.

The results show that in the range between 850°C and 950°C, the final pressure or amount of deformation of the samples varied very little.

<u>Effect of Forming Pressure</u>

As indicated in the discussion on part tolerances, different pressures are required for different shapes. The pressure appears to depend largely upon the ratio of surface area to thickness. In the case of a 2"-OD hollow hemisphere with .080" wall, over 5000 pounds per sq. in. were required. About the same was required for a 1.5"-OD sphere [illegible] wall. On the other hand, a 1.5"

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solid hemisphere only required about 1800 pounds per sq. in. to fill the corners sharply. Short solid cylinders require about the same pressure.

At this stage of the art, actual trial is necessary to determine minimum pressure. The pressures quoted refer to the projected area of the part, i.e., the OD circle area in the case of a hemisphere. Also, these pressures are those required to completely form or "coin" the part. If a cylinder should be reduced to half its height in a die whose inside diameter is greater than the OD of the pressed part, the pressure required would be considerably under 2000 pounds per sq. in. The cylindrical surface would also be barrel shaped.

The pressures applied in a graphite die must not exceed the low strength of graphite, which varies with different grades of the material. Generally speaking, 5000 pounds per sq. in. is about the limit for the ordinary AGR grades used in most of this work. The die walls must be fairly heavy, about one inch thick or more, to withstand the hoop tension set up in the hollow die during the final stages of "coining-out" or completely filling the die.

When some of the smoother grades of graphite are used, or when smooth die inserts are employed, the required pressure is somewhat less because of reduced friction. This is particularly important in pressing thin sections.

Pressing Time

Fig. 3 shows the effect of time in pressing uranium cylindrical shapes about 0.50" in diameter by .369 to .402" high. It will be noted that after about 2 minutes, the deformation does not change appreciably. The initial pressure was 1500 pounds per sq. in. and the temperature was 800°C. As in the experiments

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where temperature was the controlled variable, the die was large enough compared to the samples so that coining did not occur. The samples were compared, as before, on the basis of "free flow".

DIE MATERIALS

General Requirements

The material for hot pressing dies should have the following characteristics:

1. Ease of shaping (casting or machining)
2. Smooth surface
3. Non-reactive with uranium
4. Thermal shock resistance
5. Mechanical strength at 900°C
6. Ability to stand prolonged heating at 900°C

Early in the work, it appeared that if metal or ceramic inserts (die and punch facing) could be used, much smoother surfaces and closer dimensional tolerances could be held; and if a castable ceramic was employed, complex shapes could easily be molded. This first conclusion has been proved correct by several experiments with both types of inserts.

However, there seem to be almost insurmountable difficulties in the continued use of non-graphitic dies. After one, or at the most, two or three pressings with die inserts they have invariably failed for one reason or another. The metals alloy with uranium, either sticking so badly that the insert has to be chiseled away from the pressing, or bits of metal are picked up on the insert

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or pressing, thus ruining the pressing and the insert simultaneously.

In the case of ceramic inserts, failure is apparently due to thermal shock, or thermal shock plus the loading stress.

At the present stage of the art, the most likely solution to the problem is very fine grained graphite, when available.

Metal Inserts

Various metals and alloys used as inserts will be discussed very briefly individually.

As might be anticipated, ordinary grades of tool steel, including "Crocar" containing 13 per cent chromium, alloy very badly with uranium at 850°C in the clean atmosphere required to prevent oxidation of the pressing. Deliberately oxidizing the steel first improves matters slightly, but the attack still persists. The proprietary special high temperature alloys, Stellite, Hastelloy, and Steodite, were no better than common steel.

A high chromium-high nickel steel with considerable additions of tungsten and molybdenum, now being used in gas turbines, super-chargers and the like, was also tried. It has very high strength at elevated temperatures. A stress of 5000 pounds at 900°C causes hardly measurable deformation for short periods of time, but it was found to react very rapidly with uranium. Two dies were made of this material as a substitute for graphite, and it appears to be suitable for this application if die inserts of some other non-reactive material could be used for the surfaces in contact with the uranium.

A tungsten plate stood up as well as any metallic material with the possible exception of tungsten carbide, but it reacted in spots. Tantalum sheet

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was not as good as tungsten. Molybdenum alloys very readily with uranium, and has no value for this purpose.

Sufficiently large pieces of silver-tungsten alloy (Elkonite) were not available for a good test, but on the basis of a small sample test, alloying was hardly noticeable. This is possibly worth following up.

For a time, cobalt-bonded tungsten carbide appeared to offer the most promise of any insert material, as several tests looked very good, with exceedingly smooth surfaces. However, more exhaustive tests showed a marked tendency of some carbide samples to spall, and in some cases, there was a slight tendency for the uranium to stick in spots to the carbide. This difficulty, plus that of fabrication of the carbide into some of the shapes that might be required, caused further work in this direction to be dropped.

Ceramic Inserts

Magnesia is quite susceptible to thermal shock and proved to be too brittle. Added MgF_2 made a denser, smoother structure, but the cracking tendency was worse.

Hot pressed BeO was as successful as any ceramic insert, but the tendency to crack away near edges and sharp corners could not be avoided.

Mullite (Al_2O_3), Steatite (magnesium silicate), and Alsimag (aluminum-magnesium silicate) were all found to be too susceptible to thermal shock. All samples of alundum cements tested were quite porous and gave a rough surface. They also cracked in use.

Zircon (Zirconium silicate) has high resistance to thermal shock. The

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tested samples were of a rather coarse structure, and were not fired very high. There seemed to be some reaction with the uranium which caused surface flaking of the ceramic. A finer-grained, higher-fired zircon might behave reasonably well.

After the exploratory work on the metal and ceramic die materials, attention was again turned to graphite, with the idea of using finer-grained material than the normal AGR grades.

Several types of fine grained graphite were tested, and found to yield much improved surfaces over the AGR. The AGMT and C-15 grades of graphite (National Carbon Company) looked promising, and C-17 carbon was good, but difficult to machine. A disadvantage of the C-15 and C-17 materials is that they are available in plates only, rather than long rounds.

Another advantage of the finer-grained grades is a somewhat higher strength than that obtained with the coarser-grained grades.

While the tests on metal and ceramic inserts were not exhaustive, further work along these lines appeared unwarranted. The tests on the fine-grained graphite were very promising, and compared with the difficulty and time required to prepare either metal or die inserts, it seemed as though the better grades of graphite provided the best solution to the problem. With graphite, machining a moderately complicated die is rapid and easy compared with using a ceramic insert where a mold must first be made, then careful fired to avoid cracking, and followed probably by a difficult grinding or finishing operation to insure smooth surfaces and tolerances. Even if a ceramic piece could be

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pressed smooth enough in a steel die, shrinkage on firing will always be difficult to compensate for accurately.

EVALUATION OF THE GAMMA HOT PRESSING PROCESS

The gamma hot-pressing process was originally useful for the preparation of small discs and hemispheres (solid and hollow) of "25".[1] It was a satisfactory method for this early work when the shapes were very simple and the amount of material limited, so that metal was not held up as in casting risers.

However, as the shapes required became more complicated, the cost of dies and time of pressing renders the hot-pressing method unfeasible, and the smaller cubes and larger sphere segments for criticality tests were made by investment casting. Centrifugal casting was the natural method to use in making sample rings, but hot pressing is returning to favor as the best method of shaping heavy hemispherical parts, either solid or hollow.

It appears probably that from the standpoint of work for the project, gamma hot-pressing has seen its heyday and will soon be an obsolete method, except possibly for an occasional piece for special measurements, or for the 25 implosion gadget, if used.

It is also used as a step in the preparation of sheet and foil. Much time can be saved in rolling by starting with a comparatively thin hot pressed plate instead of a thick casting.

1) See LA Report 198, Jan. 13, 1945

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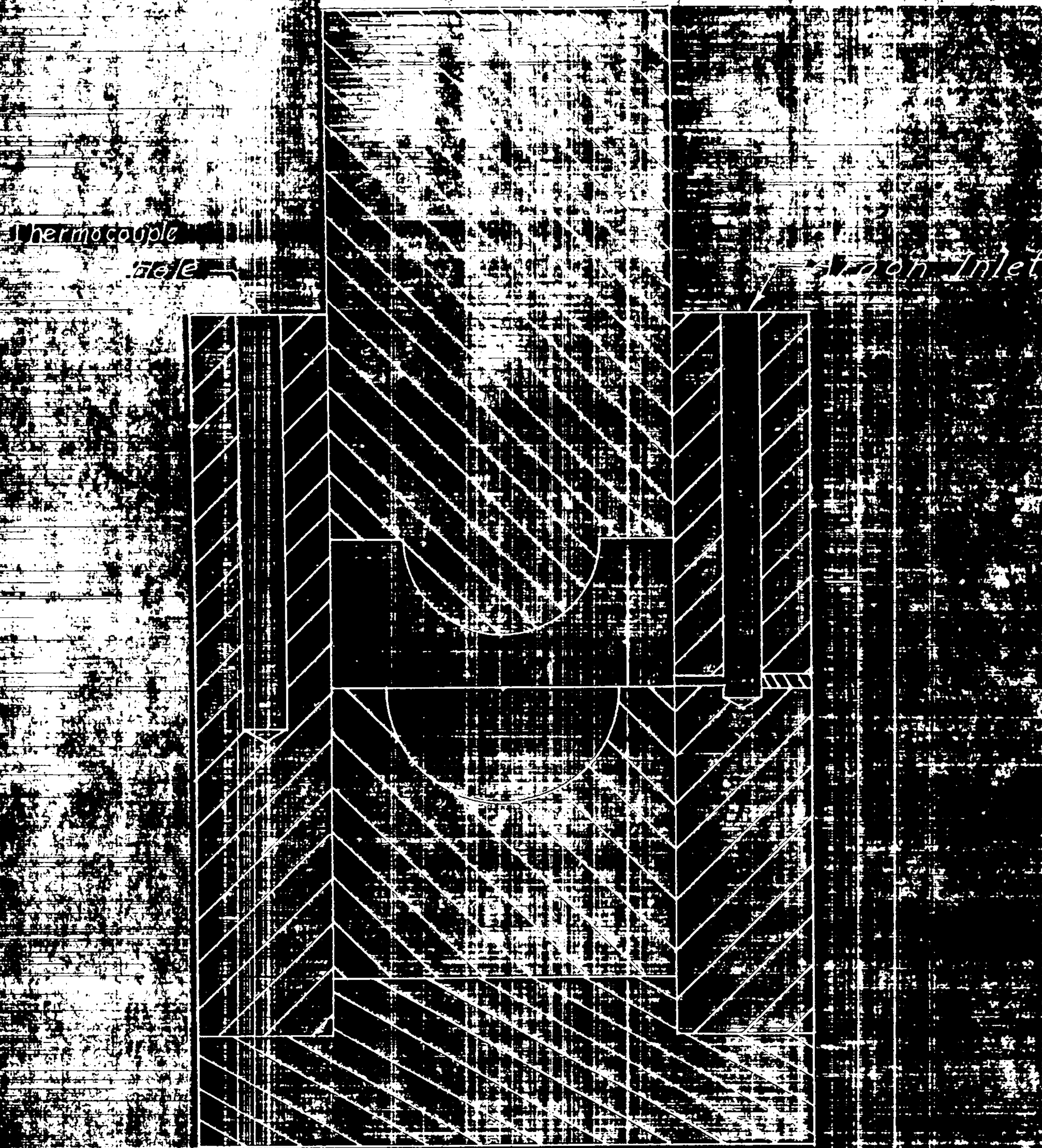


Fig I - Hot-Pressing Die

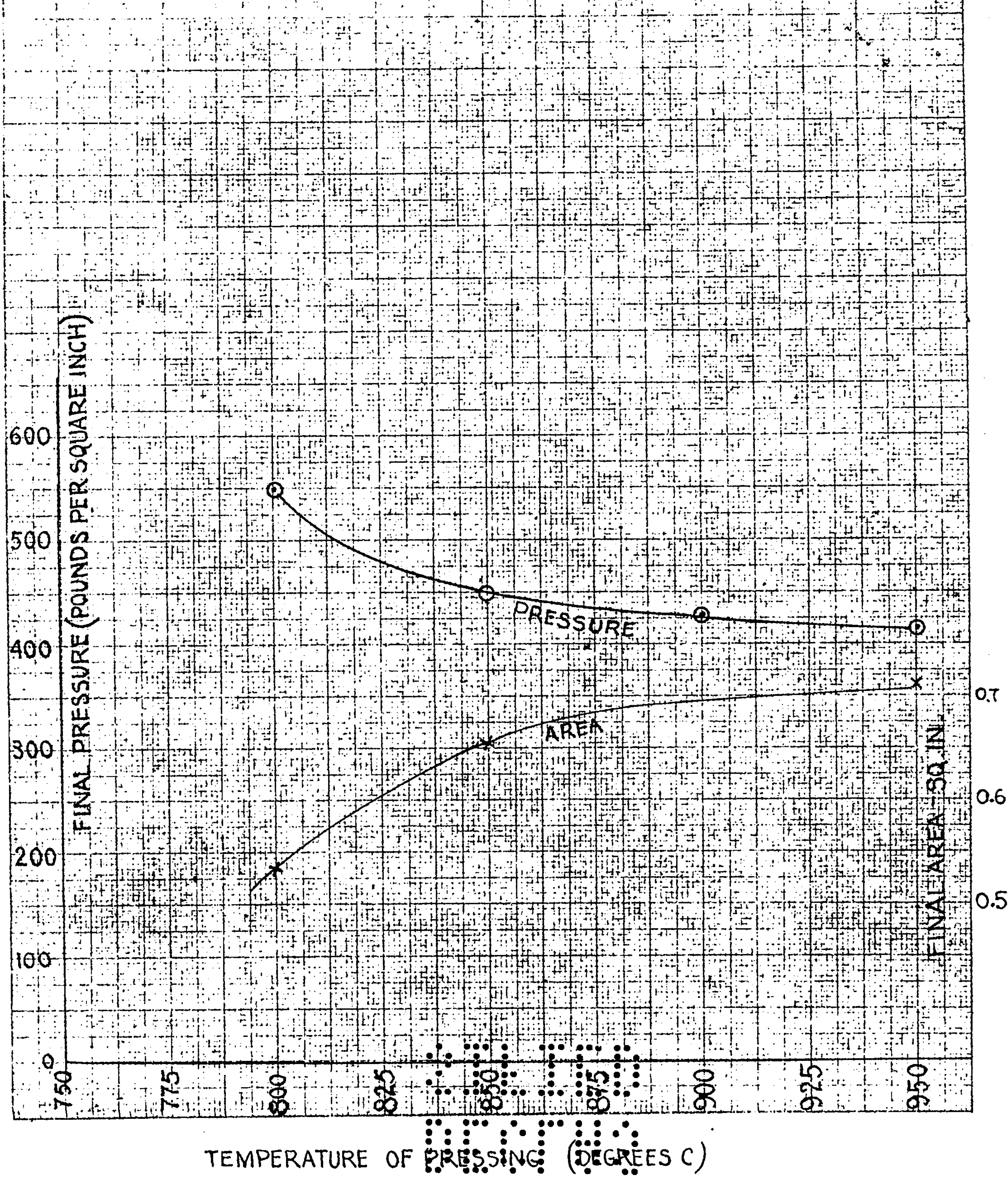
FIG-2

TEMPERATURE VS. FINAL PRESSURE AND AREA FOR HOT PRESSED TUBALLOY

TOTAL LOAD = 295 POUNDS

INITIAL AREA = 0.196 SQ. IN.

INITIAL LENGTH = 0.42 IN.



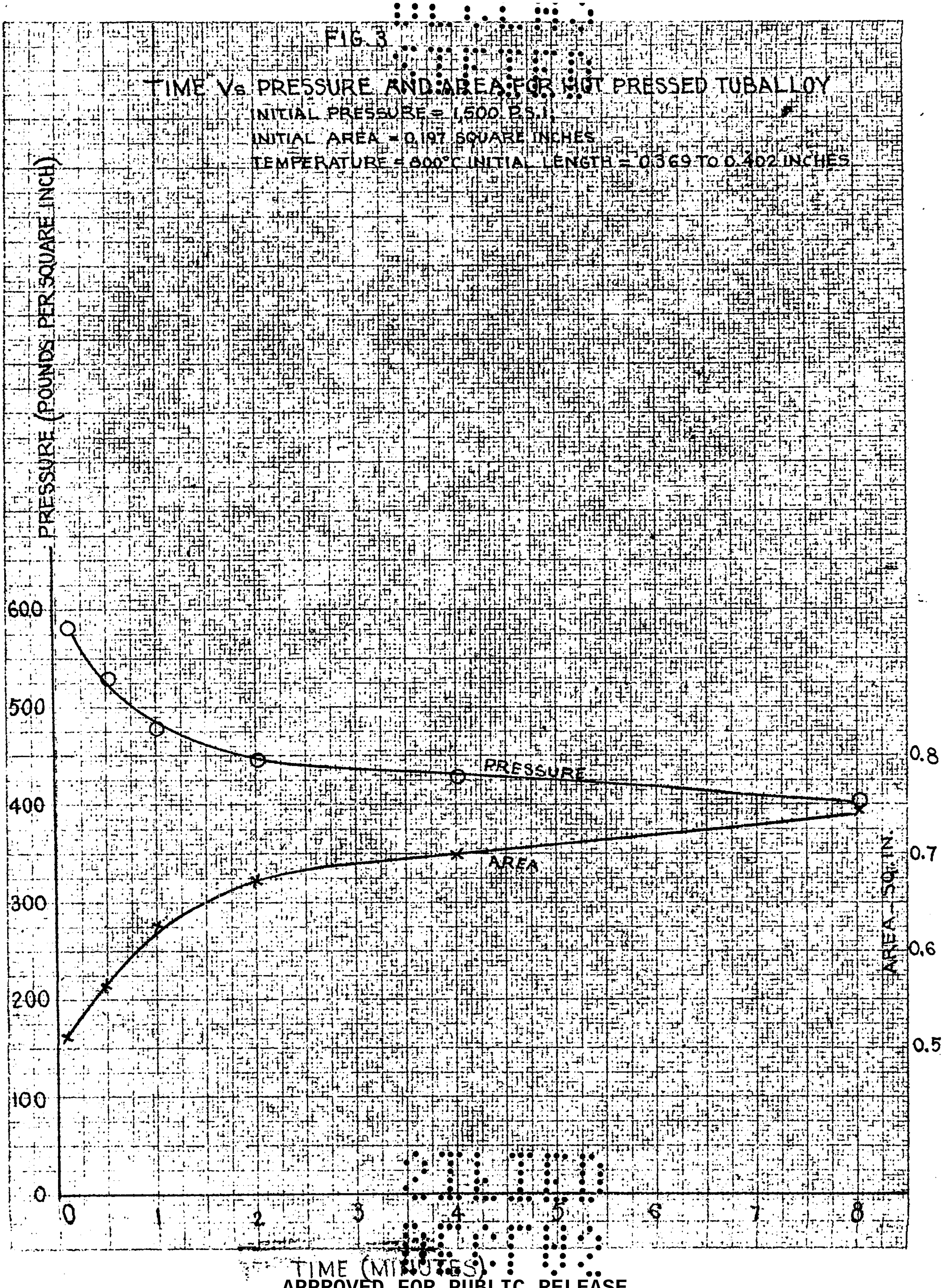


FIG. 3
TIME Vs PRESSURE AND AREA FOR HOT PRESSED TUBALLOY
INITIAL PRESSURE = 1,500 P.S.I.
INITIAL AREA = 0.197 SQUARE INCHES
TEMPERATURE = 800°C INITIAL LENGTH = 0.369 TO 0.402 INCHES
PRESSURE (POUNDS PER SQUARE INCH)
600
500
400
300
200
100
0
PRESSURE
AREA
AREA - SQ. IN.
0.8
0.7
0.6
0.5
0
1
2
3
4
5
6
7
8
TIME (MINUTES)

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