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FORMING OF URANIUM IN THE GAMMA

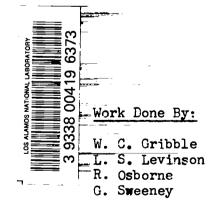
PHASE TEMPERATURE RANGE

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METALLURGY AND CERAMICS

Report Written By: L. S. Levinson W. C. Gribble J. M. Taub D. T. Doll UNCLASSIFIED VERIFIED UNCLASSIFIED

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METALLURGY AND CERAMICS

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ABSTRACT

It has been found relatively easy to form uranium in the gamma phase temperature range by hot pressing, forging, or extrusion. The metal is quite plastic and flows readily to form a shape.

Several temperatures from 800°C to 1000°C were investigated. No forming difficulties were experienced with the metal at the several temperatures concerned.

The major difficulty in gamma phase hot pressing or extruding was associated with the tools. Metals or ceramics were not successful as tools for one or more reasons concerned with: lack of hot strength, reaction with the uranium, failure in thermal shock, and tendency to spall. Graphite was found to be the best material available, but it is not entirely satisfactory because of low strength.

Uranium formed in the gamma phase possesses some refinement of grain structure as compared with as-cast metal; however, the grain size is guite large.

No physical properties of the gamma phase formed metal were determined.

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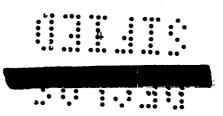
Introduction

The development of fabrication technique for uranium metal has been of great interest to the Los Alamos Scientific Laboratory and an extensive amount of work has been performed on this problem.

Most of the experimental work reported thus far has been confined to alpha phase uranium. In the alpha phase region (room temperature to 660°C) uranium has an orthorhombic structure and by using suitable techniques the metal can be formed by the commonly used techniques of rolling, forging, swaging, wire drawing, deep drawing, etc. (1). The reports indicated in reference (1) reveal some of the fabrication problems which have been encountered with alpha phase uranium and indicate the techniques which have been employed in order to obtain satisfactory pieces.

The beta phase of uranium possesses a complicated structure which is quite brittle and not amenable to normal fabricating techniques. As a rule, this phase is deliberately avoided if fabrication of the metal is required.

The final allotropic form of uranium metal exists between 770°C and the melting point of uranium. This form is the gamma phase, which possesses a body-centered cubic structure. Gamma phase uranium metal is extrememly soft and plastic and will deform easily at relatively low pressures. However, there are certain disadvantages to gamma



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phase fabrication which tend to counteract, or at least make difficult to use, the advantages of high plasticity of the metal in this region. Uranium oxidizes and burns if exposed to air at elevated temperature, so that an inert atmosphere or a vacuum must be used to protect the metal. Uranium is also a very active metal and will react with most die materials at temperature in the gamma region. Consequently a number of problems must be solved before the fabrication of gamma phase uranium can be considered a production technique.

It is the purpose of this paper to present some of the preliminary work on gamma phase uranium fabrication which has been carried on at the Los Alamos Scientific Laboratory. Needless to say much work still remains to be done on the problem but the information presented herein may be of some interest to those working on uranium fabrication problems.

Apparatus

All of the experiments described in this paper were carried out in dies mounted within a water cooled induction coil, which was powered by a 20 EN Ajax spark-gap high frequency convertor. The die and induction coil were mounted either in a 30 ton Watson-Stillman power driven hydraulic press or a 20 ton Elmes hand operated hydraulic press. Figure 1 shows the die assembly mounted in the 20 ton Elmes press. Figure 2 reveals a cross section through the die and coil assembly, in this case graphite being the die material. The mica insulation was to prevent arcing between the induction coil and the die.

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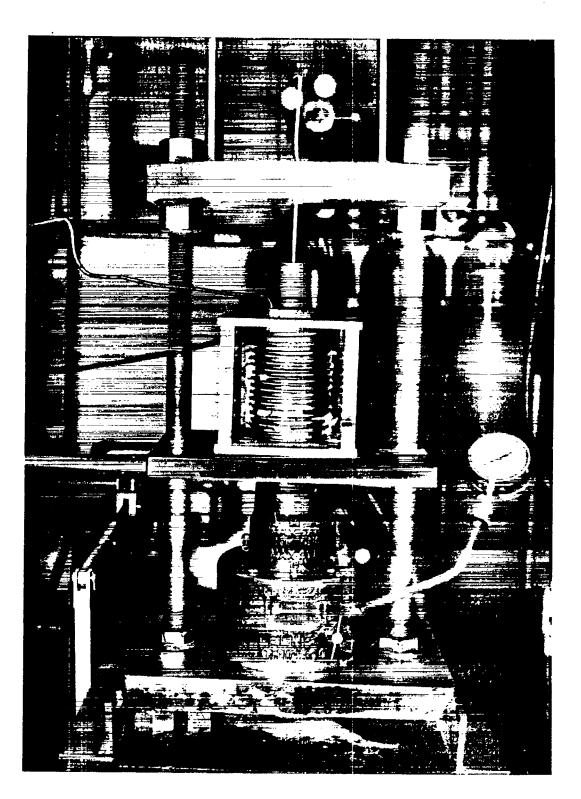


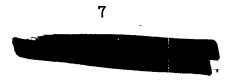
Figure 1. 20 Ton Elmes Hydraulic Press with

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Hot Pressing Die Assembly.







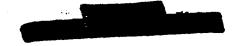
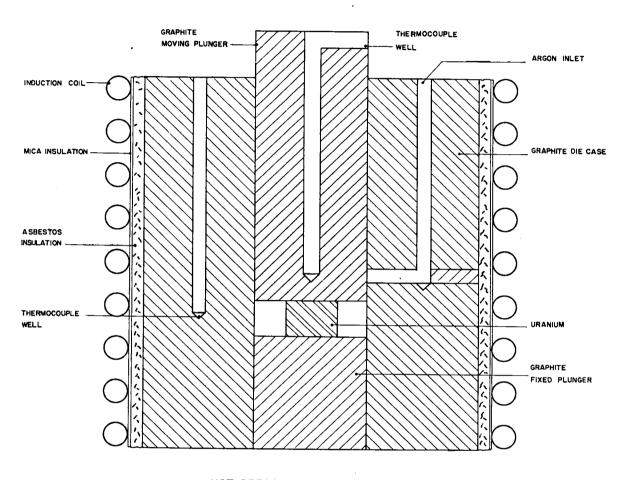
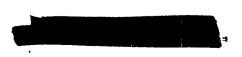


Figure 2. Hot Pressing Die Assembly.



HOT PRESSING DIE ASSEMBLY



The asbestos insulation helps to minimize radiation losses from the hot die and also prevents the induction coil from overheating.

Temperatures were measured by means of Chromel-Alumel thermocouples inserted into the die assembly, the temperatures being measured with a potentiometer.

The inert atmospheres used were either argon or helium gas which was piped directly from the tank through a manifold and to the die assembly. No attempts were made to dry or purify the tank gas. A line pressure of 1.5 psi was used to maintain the inert atmosphere within the die assembly.

Procedure

The die assembly was inserted into the induction coil and the calculated amount of metal to make the desired part was placed within the die. The plunger was then assembled in the die and the protective atmosphere introduced into the loaded die. The necessary thermocouples were also positioned as desired. The entire die assembly was then heated by induction until the desired temperature (800-1000°C) was obtained and the pressure was applied to the die plunger by the hydraulic press. After pressing, the assembly was permitted to cool to room temperature at which time the die was disassembled. In cases where the uranium was formed about a graphite core it was necessary to remove the core by chipping or drilling. The formation of a thin film or flash of uranium between moving parts of the die also created difficulty when attempting to remove the pressed pieces and occasionally



it was necessary to break open the die casing in order to remove the formed piece of uranium.

Plasticity of Uranium in the Gamma Phase

Uranium metal becomes plastic to a remarkable degree when heated into the gamma region. In order to obtain more information concerning the properties of gamma uranium, a number of experiments were carried out, the results of which indicated two things, namely (a) how much deformation could gamma uranium take, and (b) the relative values of plasticity and deformation pressures, compared to lead at room temperature. In these experiments in closed dies uranium was pressed, rolled and extruded.

In the pressing experiment to determine the flow of uranium a cylinder three inches in diameter by four inches high and containing a centrally located cavity 0.250 inches in diameter by 0.750 inch long was pressed in a graphite die to a diameter of 3.5 inches at a temperature of 1000°C and at a pressure of 2700 psi. The purpose of such a pressing was to determine whether or not the metal would flow sufficiently to close up the center cavity.

The pressed cylinder was radiographed and no evidence of a cavity was found. The cylinder was then sectioned and again no trace of a cavity was found, the only evidence being the presence of a fine line where the cavity closed up but did not weld because of the oxidized surfaces. The results of this experiment indicated that the metal would flow readily at the temperature and pressure used.

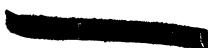
A rather unique rolling experiment was performed in the special graphite die shown in Figure 3. In this die, the rolls were merely polished cylinders of graphite which were free to rotate but were not driven. The friction of the uranium passing through the rolls caused them to rotate. All of the pressure required for the operation was transmitted to the uranium through a graphite plunger. At a temperature of 1050°C and a pressure of 3350 psi it was possible to obtain a reduction in area of the uranium of 75 percent. The photograph in Figure 4 shows the rolled section. The end of the section reveals the shape of the original billet before rolling.

The extrusion process was used as the basis for determining most of the plasticity characteristics of gamma phase uranium. Sachs and Eisbein (2) have shown that straight lines which pass through the origin are obtained when pressure P of the extrusion process is plotted against the logarithm of the extrusion ratio A/a. The formula $P = C \log (A/a)$ is used, where C is a constant related to the resistance of the material to plastic deformation. Experimentation has been carried out to determine C for uranium at various temperatures in the gamma region.

Direct and indirect extrusion techniques were employed in the experiments, the indirect extrusion technique having the advantage of requiring less pressure and being somewhat easier to measure for pressure determination.

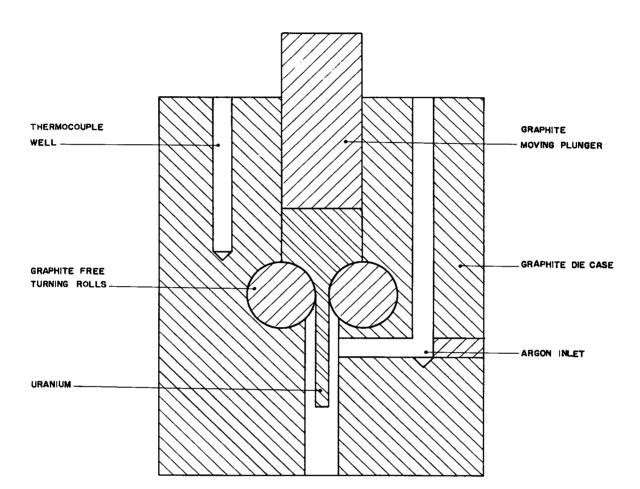
In the direct extrusion technique, the die is located at one

Figure 3. Rolling Die Assembly



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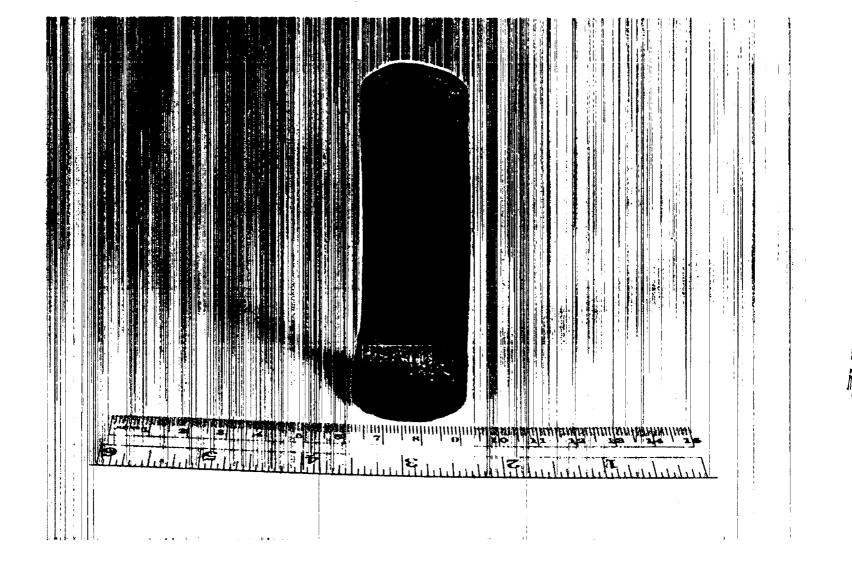




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Figure 4. Uranium Rolled in Gamma Phase Reduction in Area, 75% Pressure on Plunger, 3350 P.S.I. Temperature, 1050°C

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end of the billet container and the metal flows through the die traveling in the same direction as the ram which is exerting the pressure. In the case of the indirect extrusion technique, the die is attached to the end of a hollow ram and the metal flows through the die in a direction counter to the movement of the pressing ram. The friction encountered in indirect extrusion is less than in direct extrusion, thus accounting for the lower extrusion pressures. Figure 5 shows an exploded view of an indirect extrusion die assembly, and Figure 6 is an exploded view of a direct extrusion die assembly.

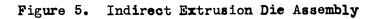
The extrusion dies were constructed of National Carbon Company Grade CS-312 graphite, a fine grained, relatively high strength graphite. The uranium metal used in the experiments was in the form of machined cylinders 1.375 inches in diameter by one inch long.

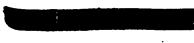
The extrusion rate used in all experiments was 0.01 inch per minute and this low rate was used in order to obtain the maximum possible extrusion ratio, that is, the ratio of initial to final areas. The maximum ratio at this rate, using graphite dies, was found to be 120 to 1.

Some work was performed using lead at room temperature in order to have some results with which the plasticity of uranium could be compared. Lead is usually considered as being quite plastic but does not appear so when compared to gamma phase uranium. As an example, when lead was extruded at an extrusion ratio of 100, a

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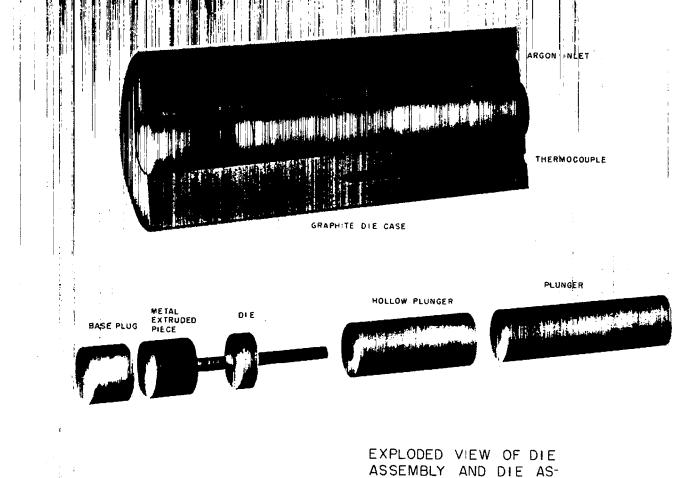


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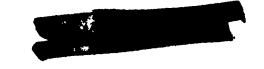
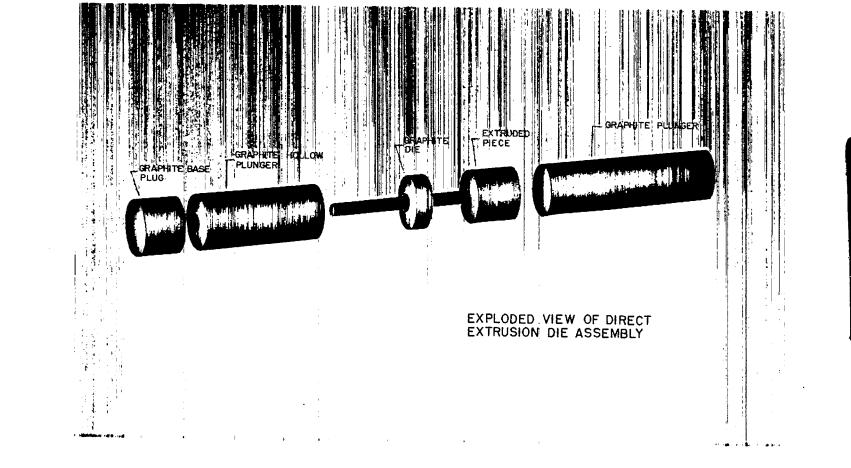


Figure 6. Direct Extrusion Die Assembly



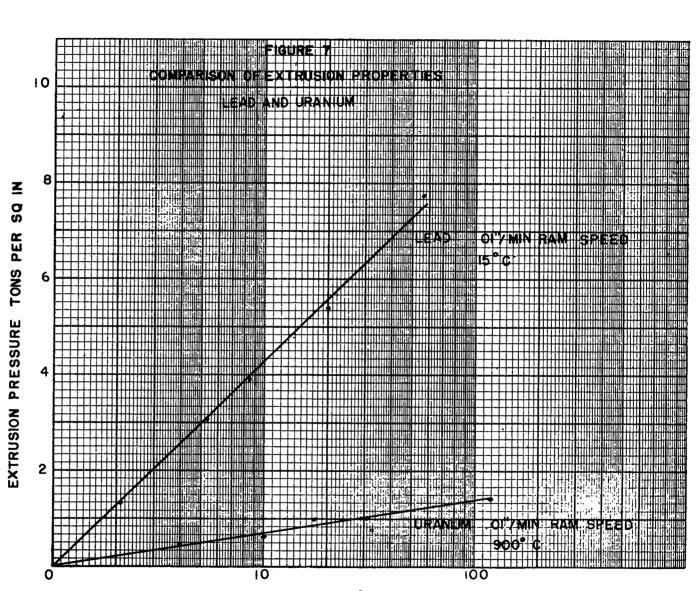
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pressure of 22,000 psi was required (3). At the same extrusion speed and extrusion ratio, gamma uranium required only 3,000 psi.

A further comparison of lead and gamma uranium is shown in Figure 7. The data for the lead curve was taken from C. E. Pearsons' "The Extrusion of Metals", Wiley, 1944. The original data were taken at an extrusion speed of 0.10 inch per minute whereas the uranium speed was 0.01 inch per minute. The extrusion pressure varies with the rate of extrusion and to make a comparison the data for lead were reduced 36 percent, (4) the pressure correction given by Pearson for a speed factor of ten. The "C" factor, which is the resistance of the metal to deformation by extrusion, was found to be 1.87 tons per square inch for lead and only 0.33 ton per square inch for uranium extruded at 900°C. This would indicate that gamma uranium is only one-sixth as difficult to extrude as lead.

The effect of temperature of the gamma uranium on extrusion pressure is indicated in Figure 8. The results indicated that in the temperature range 800°C to 1000°C there is not a marked increase in the plasticity of the metal. The "C" values for uranium at temperatures of 800°C, 900°C and 1000°C are 660, 610 and 570 psi, respectively. Figure 9 is a photograph of uranium extruded at 900°C at two different extrusion ratios.

The results of a plastic flow study, in which a static load was applied for five minutes to uranium cylinders 0.50 inch in diameter and 0.41 inch high, is shown in Figure 10. The edges of the pieces



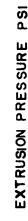
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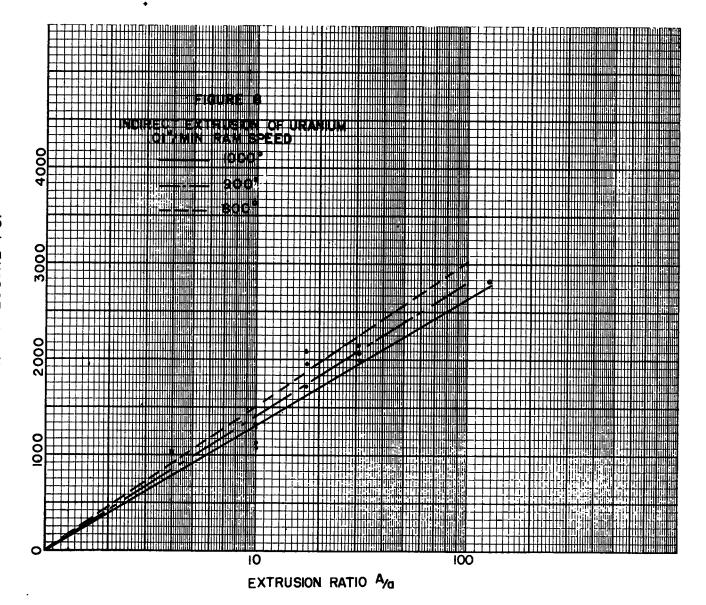
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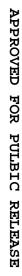
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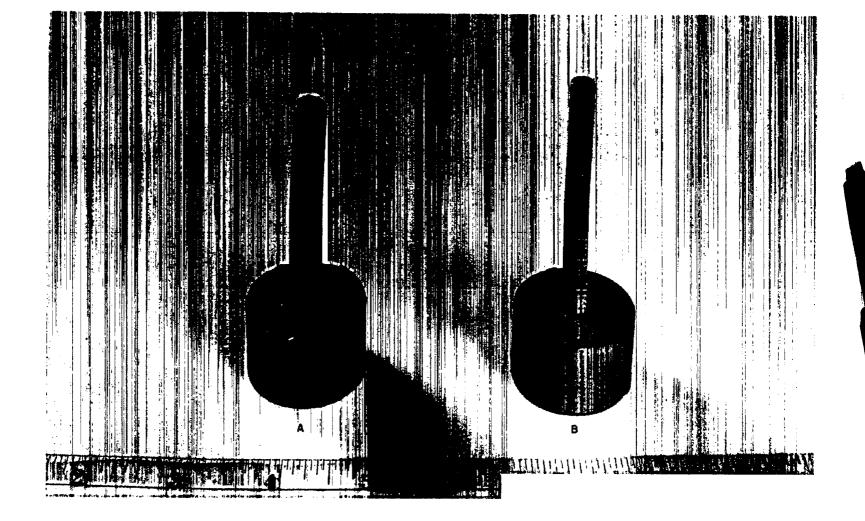
Figure 9. Uranium Extruded in the Garma Phase.

	Ratio (A/a)	Temperature ^o C
Λ	17.5	9 00
В	3 1	900

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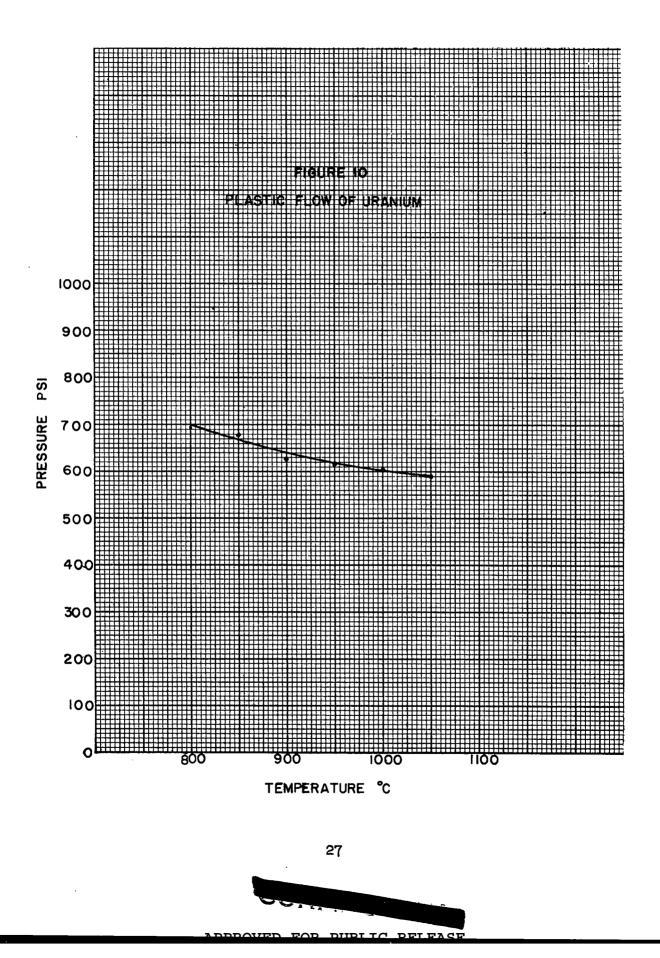






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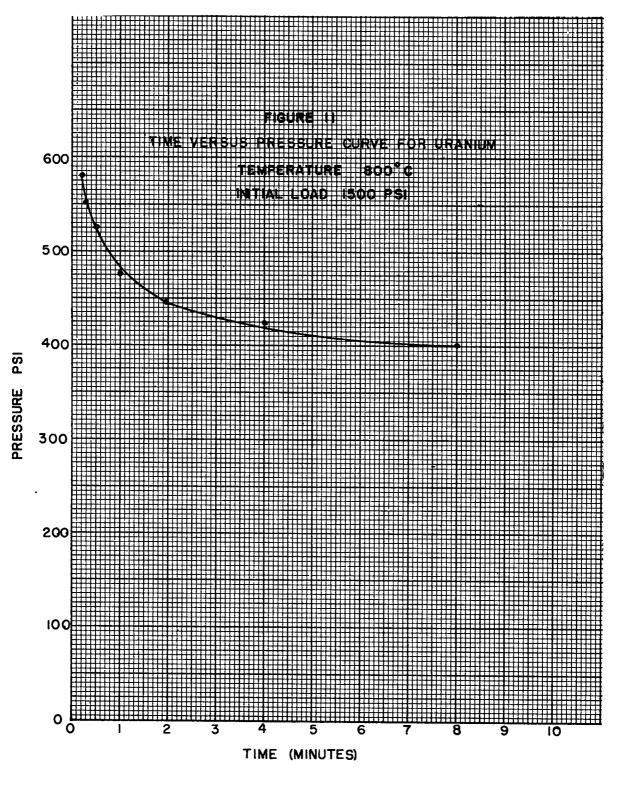
were not restrained by the die wall, and the metal was allowed to flow until an equilibrium condition was reached. The uranium cylinders flowed somewhat more at the higher temperatures, and so the unit load is lower at the high temperature end of the curve. Figure 2 shows a view of the assembled die.

Figure 11, "Time Versus Pressure Curve for Uranium", was carried out to determine the time necessary for complete flow of gamma uranium in the pressing die. Uranium cylinders, 0.50 inch in diameter and 0.41 inch high, were pressed with a load, initially 1500 psi at 800°C.

Die Materials

The die materials necessary for gamma phase uranium working must be considered from the standpoint of the type of operation and the method of use. The material needed for a die which is normally maintained at room temperature and in contact with the gamma uranium for only a short period, such as in a forging operation, may be quite different from the material necessary for an operation in which the uranium is heated within the die and the materials are in contact for a relatively long interval of time. Inasmuch as the first type of operation has had very little investigation no definite conclusions can be reached at this time. Most of the work described herein has been done with closed dies using the second technique described above and the results described were determined from that procedure.

It was apparent, after a number of tests had been made on







various materials, that the desirable characteristics of a die material to be used for gamma phase uranium working would be the following:

- 1. Easily fabricated material.
- 2. High resistance to thermal shock.
- 3. Does not react with uranium.
- 4. Possesses good mechanical strength at 1000°C.
- 5. Die surfaces capable of being polished.

The die materials which have been used fall into the categories of metals, ceramics and graphite. None of the materials tested thus far possessed all of the characteristics indicated above and only one material, namely, graphite, showed any signs of satisfactory performance and this occurred only when the best grade of graphite was used. The following summary indicates very briefly the die materials used and the results of the test.

Metals

1). Ferrous Materials. Die steels of various types and stainless steels have all failed as a die material because of reaction with gamma phase uranium. The reaction is quite vigorous and ruins both the die and the uranium piece. Coatings on the steel surfaces have not been very successful.

2). Stellite (chromium-cobalt-tungsten). Failed by reaction with uranium.

3). Hastelloy (nickel-molybdenum). Failed by reaction with

uranium.

4). Tungsten. Reacted with uranium but not to the extent of the other metallic inserts.

5). Tantalum. Reacted in manner similar to tungsten.

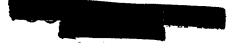
6). Molybdenum. Failed by reaction with uranium.

7). Tungsten Carbide. This material has been used for die inserts and extrusion dies with some success but the results have been somewhat erratic. The early experiments were carried out with material containing nine percent cobalt as the binder and failure occurred by both reaction with uranium and by spalling of the die. The reduction of the amount of binder to three percent decreased the uranium reaction but did not effect the spalling phenomenom.

Tungsten carbide is both difficult and expensive to fabricate and consequently is not too available for experimental work. However, if the working of gamma uranium on a production basis were to be considered, it probably would be possible to develop a tungsten carbide die which would be quite satisfactory.

Ceramics

Over a period of time a number of ceramics have been used as die inserts for uranium fabrication studies. Among the materials tested have been beryllium oxide, magnesium oxide, mullite $(Al_2O_3 - SiO_2)$, steatite (magnesium silicate), alsimeg (aluminum-magnesium silicate), zirconium silicate and aluminum oxide. The ceramic materials, as a class, are subject to failure by thermal shock and by



spalling. These materials are also difficult to fabricate and do not possess the necessary fabrication flexibility exhibited by the nonceramic materials. Beryllium oxide appeared to be the most promising of the ceramic materials but it also was subject to failure by spalling and by thermal shock.

The experimental work on ceramics has not been carried out to the fullest extent but the materials do not show great promise as die materials and it is unlikely that much additional work will be done on this phase of the program.

Graphite

The most suitable die material obtained thus far has been graphite. It fulfills the requirements for a die material to a greater degree than any material tested thus far. It should be emphasized, however, that only the best grades of graphite, which possess a fine grained, dense structure and have relatively high tensile and compressive strength, are satisfactory. National Carbon Company grades CS-312 and ATL fall within this category.

Graphite is easy to fabricate with standard machine tools, it has excellent resistance to thermal shock and does not react with uranium to any extent at the temperatures employed. The low strength of the graphite, as compared to metals, is the major objection to the use of this material. The best grade of graphite used in this work had a tensile strength of 1000 to 1500 psi and a compressive strength of approximately 6000 psi.

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Graphite abrades easily with an increasing roughning of the surface as the die is used and a corresponding change in die dimensions. The plastic gamma phase uranium will extrude into the pores and fissures of the material and create enough friction so that surface flow of the uranium becomes very difficult. Surface oxidation, primarily of the outside of the die casing, does occur but not to an objectionable extent.

Die Design

The factors affecting die design must necessarily be limited to our experience in which graphite was the die material used. Consequently, if a material other than graphite were to be considered for the forming die the information on die design given herein may not necessarily apply.

When using graphite as the die material, the die design must necessarily take into account the low tensile strength of the graphite. In particular the hoop stresses set up in the outer fibers of the die casing must be kept very low if cracking is to be avoided. In most cases this means that a relatively thick wall is required for the die casing. Under normal circumstances if a die material such as steel were to be used, the thickness of the casing would not be very important because the forming pressures required for gamma uranium are very low and usually well below the yield strength of most metals.

The extrusion die design which has been found to be most satisfactory consists of the simplest possible design, namely, a flat

surface on the entrance side of the die and a sharp corner leading into the die opening. Dies with a bell mouth (either straight taper or a radius) break because of the hoop stress imposed on the graphite die. No relief on the exit side of the die was used.

Diametral clearances between plungers and the die casing should be between 0,002 inch and 0.005 inch. If a press fit is to be used the interference should be between 0.005 inch and 0.003 inch, depending on the diameters involved. Excessive interference naturally will cause cracking.

If it is necessary that the die plunger be pushed into the uranium to form an interior surface or a hole, this part of the die should be designed so that it will be considered expendable. Uranium, upon cooling from the gamma region, will shrink around the die plunger and prevent its removal. The portion of the die remaining in the uranium piece must be chipped or drilled out.

The die assembly, both casing and plunger, should contain holes for the necessary thermocouples and for the introduction of the inert atmosphere. The holes should be spaced so that they do not fall on a diametral line which would result in a weak section through the assembly.

Microstructure

Uranium which has been heated and worked in the gamma region exhibits a large grained structure, the average grain size being approximately 0.3 mm. Figure 12 indicates the structure obtained in



Plate No. J-111-1-2 Mag. 250% No etch: Polarized light Gamma pressed (970°C) hemispherical section.

Figure 12

Photograph of the structure of uranium hot pressed in the gamma temperature range.

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uranium which was pressed at 970°C.

The same type of structure is found in both pressed and extruded pieces and is similar to the "as-cast" uranium structure observed in Figure 13. No grain refinement and little, if any, orientation was observed in the pressed or extruded pieces. Figure 14 shows the microstructure of uranium extruded with various extrusion ratios and at various temperatures in the gamma region.

Summary

Although the scope of this investigation was quite limited and the forming techniques were restricted almost entirely to closed die procedures, the following conclusions may be reached at this time:

1. Uranium in the gamma phase can be extruded, rolled and pressed.

2. The plasticity of gamma phase uranium is very great, the resistance to deformation being approximately one-sixth that of lead at room temperature. The maximum extrusion ratio obtained was 120 to 1. using graphite dies.

3. The most promising die material tested was fine grained, high strength graphite.

4. The micro-structure of gamma worked uranium consisted of large grains, not unlike an as-cast structure, but showing some evidence of strain.

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Plate No. J131-2-1Mag. 250XLongitudinal SectionNo etch: polarized lightSample #P3-2A.Normal purity Tu, as cast.

Figure 13

Photomicrograph of cast uranium.

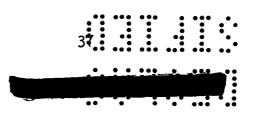




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Chromic acid acetic acid etch; b	right	field	
gamma extruded 900°C; extrusion	ratio	(A/a)	118



Plate No. J75-C4 Mag.: 250X Chromic acid acetic acid etch; bright field gamma extruded 1000°C; extrusion ratio (A/a) 4

Figure 14

Photomicrographs of uranium extruded at several temperatures and ratios.

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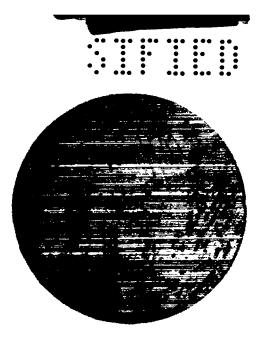


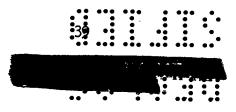
Plate No. J75-B3	Mag.:	250X	
Chromic acid acetic acid etch; 1	bright	field	
Gamma extruded 800°C; extrusion	ratio	(A/a)	4



Plate No. J75-C3 Mag.: 250X Chromic acid acetic acid etch; bright field Gamma extruded 800°C; extrusion ratio (A/a) 30

Figure 14 (Continued)

Photomicrographs of uranium extruded at several temperatures and ratios.





References

1. LA Reports on Alpha Phase Working.

LA-1150, Deep Drawing of Uranium by Gribble, Levinson, Taub, Doll.

LA-1178, Forging of Uranium by Anderson, Taub, Doll.

LA-1180, Rolling of Uranium by Deutsch, Hanks, Taub, Doll.

- 2. Sachs and Eisbein. Powder Consumption and Mechanism of Flow . in the Extrusion Process. Mitt. Material, S.16, 1931, 67-96.
- C. E. Pearsons "The Extrusion of Metals", John Wiley and Sons.
 C. E. Pearsons, Ibid.

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