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ASD TR 7-886 (VII)

ASD INTERIM REPORT 7-886 (VII)
January, 1963

DEVELOPMENT OF 2400°F FORGING DIE SYSTEM

H. Nudelman
A. H. Murphy
T. Watmough
P. R. Gouwens

ARMOUR RESEARCH FOUNDATION
of
Illinois Institute of Technology

Contract: AF 33(600)-42861
ASD Project: 7-886
ARF Project B220
Interim Technical Progress Report
28 September 1962 - 27 December 1962

The selection of materials suitable for use as a forging die to operate at 2400°F is being finalized. The oxidation protection mandatory for metallic materials can be produced by a rectified viscous salt bath, but requires suitable engineering design of the die to maintain protection. Non-metallic materials without a protection system are promising provided that compressive stresses are maintained. Refrax and KT silicon carbide are most favorable. Preparation of a prototype die for heating experiments is described.

BASIC INDUSTRY BRANCH
MANUFACTURING TECHNOLOGY LABORATORY

Aeronautical Systems Division
Air Force Systems Command
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Research continued on the third phase of this program designed to evaluate new materials for use as a 2400°F forging die. The ultimate objective is the creation of a true hot-working technology for refractory metals.

Experiments have been performed to determine the feasibility of using a viscous protection coating rather than the conventional solid coatings for the refractory metals. A high-temperature barium chloride type heat-treating salt, rectified with carbon, successfully inhibits oxidation loss at 2400°F. The need for immersion does, however, dictate several restrictions on the die design in order to maintain a pool of liquid salt over the susceptible parts of the high-temperature die system. The problem of die component creep at high temperature imposes additional requirements on the compatibility of the various materials used in the die system.

Refractory materials such as titanium diboride, Refrax, and KT silicon carbide remain most promising, with the latter two excelling. Die design again is somewhat restricted in that the stresses imposed must produce die loads largely dominated by compressive stresses.

A prototype die system to be used for high-temperature heating studies has been constructed and is described in detail. Heating to 2400°F will be accomplished through electrical resistance heating elements supplemented by gas flame radiation on the die surface.

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FOREWORD

This Interim Technical Progress Report covers the work performed under Contract AF 33(600)-42861 from 28 September 1962 to 27 December 1962. It is published for technical information only and does not necessarily represent the recommendations, conclusions, or approval of the Air Force.

This contract with Armour Research Foundation of Illinois Institute of Technology, Chicago, Illinois, was initiated under ASD Manufacturing Technology Laboratory Project 7-886, "Development of 2400°F Forging Die System." It is administered under the direction of Mr. George W. Trickett of the Basic Industry Branch, Manufacturing Technology Laboratory, Aeronautical Systems Division, Wright-Patterson Air Force Base, Ohio.

Mr. Paul R. Gouwens is the project director, with Mr. A. H. Murphy, Mr. T. Watmough, and Mr. H. Nudelman principally responsible for experimental work on Phases I, II, and III, respectively. Dr. W. Rostoker and Mr. R. J. Van Thyne are serving as internal ARF consultants. All of the above are members of the Foundation's Metals and Ceramics Research Division. This report is designated as ARF-B220-21 by Armour Research Foundation.

The primary objective of the Air Force Manufacturing Methods Program is to increase producibility, and improve the quality and efficiency of fabrication of aircraft, missiles, and components thereof. This report is being disseminated in order that methods and/or equipment developed may be used throughout industry, thereby reducing costs and giving "MORE AIR FORCE PER DOLLAR."

Your comments are solicited on the potential utilization of the information contained herein as applied to your present or future production programs. Suggestions concerning additional Manufacturing Methods development required on this or other subjects will be appreciated.

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DEVELOPMENT OF 2400°F FORGING DIE SYSTEM

I. INTRODUCTION

This is the seventh interim technical report, covering the period 28 September 1962 to 27 December 1962 on Contract No. AF 33(600)-42861. This program, an extension of previous Air Force contracts AF 33(600)-35530 and AF 33(600)-35914, is designed to extend the previous development of a high-temperature die system for forging steel, which used a die temperature up to 1600°F.

The ultimate objective of the present research is the forging of refractory metals with dies operating at about 2400°F, but determination of the other limitations of the presently hot die system was also necessarily included. The detailed objectives of the present program are:

1. Evaluate the upper operating temperature limit of forging dies cast from Inconel 713C. Previously failure did not occur even at 1600°F and a load of 1000 tons.

2. Determine the minimum number of forging steps from unshaped blank to advance finished shape using the hot die system.

3. Attempt to develop a die material of metallic, ceramic, or composite metal-ceramic structure which can operate at about 2400°F, without atmospheric protection, and under loads required to hot-work refractory metals.

4. Devise methods for manufacturing die blocks using the materials developed for 2400°F applications.

5. Produce dies and forge sufficient molybdenum alloy parts to prove the process and materials developed.

Work during this past quarter has been concentrated on the third objective. The first and second phases of the research are now completed.

II. EXPERIMENTAL RESULTS

PHASE III - Development of a Die Material Suitable for Service at 2400°F

A. Selection of Metallic Materials

The criterion for acceptance of a suitable die material has been established as follows:

1. A minimum compressive yield strength of 25,000 psi at 2400°F
2. Oxidation resistance for 100 hr at 2400°F

While these standards are quite arbitrary, there is good indication that the strength requirements represent a realistic appraisal of the conditions to be met during forging of molybdenum at 3000°F.

Resistance to oxidation is the major problem and has been approached by a consideration of solid protective coatings and also by renewable viscous protective media. Previous reports document the failure of the solid coatings; however, one lot of 97% tungsten-3% molybdenum alloy samples has been submitted to Chromalloy Corporation for application of their proprietary coating. These 30 coated samples will be evaluated for oxidation resistance as soon as they are received at ARF.

Preliminary evaluation of the possibilities of affording protection against oxidation of the refractory metals by the use of salts and slags to envelope the sections has been undertaken. A sample of P4 (97W-3Mo) was submerged in a crucible containing a heat-treating salt, typically used for the heat treatment of high-speed steel. This salt is essentially barium chloride with a small amount of sodium chloride added. It is supplied by E. Houghton and is designated as Liquid Heat 1500. Oxidation tests were conducted in air, and the weight loss data are shown in Table I. These results are encouraging and further tests of TZM and TZC molybdenum alloys under the same salt have been initiated. One problem with this particular salt is its extreme fluidity and a review of the possible combinations of salts and oxides is being undertaken to obtain a spectrum of viscosities.

It is considered that for the hot die application the salt must be fairly viscous.

TABLE I

WEIGHT LOSS OF P4 ALLOY AT 2400°F

UNDER LIQUID HEAT 1500 SALT PROTECTION

Elapsed Time, hr	Sample Weight, gm	Accumulated Weight Loss, gm
0	25.4490	---
22	25.4246	0.0244
46	25.4184	0.0316
51	25.4131	0.0359

B. Selection of Nonmetallic Materials

A study was initiated to determine the practicability of a titanium diboride forging die-tungsten alloy retainer system. A thick-walled titanium diboride cylinder was ground to provide a slip fit in a thick-walled 98% tungsten-2% molybdenum cylinder with both components at room temperature. This configuration represents a half-scale approximation of the system which will ultimately be required. The forging die-retainer combination was heated in steps to 1800°F. Intervals of 200°F were used up to 1400°F, and 100°F intervals were applied thereafter. The system was held for one hour at each preselected temperature level to provide an equilibrium temperature distribution. An atmosphere of 90% nitrogen-10% hydrogen was utilized to minimize the effects of oxidation. After the 1800°F level was attained, the die-retainer combination was furnace-cooled to room temperature, separated, and examined. There was no significant dimensional change in the titanium-diboride member, but the ID of the tungsten-alloy cylinder was increased by about 0.003 in. on the diameter. This indicated that yielding and/or creep occurred during heating. This would be expected as the titanium diboride has a higher coefficient of thermal expansion than the tungsten alloy.

After attaining the 1800°F level, the combination was heated to 1900°F and subsequently to 2400°F in 100°F intervals. It was held for one hour at each temperature; the 90% nitrogen-10% hydrogen atmosphere was maintained throughout. The dimensional changes associated with this treatment were as follows:

Condition	TiB_2 Die Insert OD, in.	Tungsten Alloy Retainer ID, in.
As machined	1.7983	1.7989
After 1800°F	1.7985	1.8018
After 2400°F	1.7984	1.8000

The critical diameters of both components increased slightly as compared to their as-machined values. Oxidation of the tungsten alloy proved to be a serious problem despite the atmosphere provided. After the 1800°F exposure the tungsten was only slightly attacked. However, after heating to 2400°F the oxidation appeared excessive. The tungsten retainer and titanium diboride insert are shown in Figure 1. An oxide layer about 1/32 in. thick was built up over the entire surface area of the tungsten except where it was in contact with the titanium diboride. In addition, the free surfaces of the titanium diboride appeared to have reacted with the gaseous reaction products emanating from the tungsten alloy. From a chemical stability standpoint it would appear that this combination is impractical unless the oxidation of the tungsten can be prevented.

Prior calculations indicated that with these particular diameters, the titanium diboride would expand sufficiently to cause failure in the tungsten cylinder. These calculations were based on thermal expansion data and elastic moduli values supplied by the manufacturers which may not provide sufficient accuracy for this approach. This was verified by the calculations made after coating. On the basis of final diameter values, an interfacial pressure of about 77,000 psi was indicated at 2400°F. This pressure should have caused the tungstem alloy cylinder to deform using circumferential stress as a criterion. The increased ID of the tungsten cylinder suggested that it was deformed (yield or possibly creep) due to the expansion of the inner titanium diboride cylinder. Further work would be required to obtain a clearer understanding of these effects. However, at this stage KT silicon carbide and Refrax appear more promising, and further efforts involving titanium diboride are not currently under consideration.

A brief experiment was carried out to determine the compatibility of the tungsten with KT silicon carbide and Refrax. A cylinder of each material was hung on a tungsten rod which was heated to 2400°F in air for 15 minutes. The cylinders and rods are shown in Figures 2 and 3. Examination of the specimens did not reveal any evidence of undesirable reactive effects, but the complicating effect of pressure between the couple was not introduced.

C. Prototype Die for Heating Experiments

The machining of prototype forging die components was completed during this period. Figure 4 shows the finish-machined parts. This photo shows the lower die completely assembled and the upper die disassembled. Figure 5 is the first in a series of photos showing the assembly of the upper die. This figure shows the die retainer placed within the hold-down clamp. The clamp was cast of HT stainless steel and the retainer machined from

310 stainless plate and is for use on the prototype only. The retainer slip fits into the hold-down clamp to establish alignment. The next photo, Figure 6, shows the outer support block added. This member was cast of Inconel 713C. It is aligned by means of a slip fit of its top flange within the hold-down clamp. Figure 7 shows the middle support block in place. This piece is also of 713C and is maintained in alignment by means of a slip fit of its upper flange within the outer support block.

Figure 8 shows the final assembly of the core of the die. With the components in this position, spiral-wound Calrod heating elements are slipped into position. The slots allow the Calrod leads to extend outward. Only two Calrods are used--one around the center core and the other in the next outward ring slot. The outermost slot is filled with fiber insulation to prevent excessive heat loss in this direction.

Once the Calrods are installed, the support block shown to the left in Figure 8 is slipped into the hold-down ring to form a base. The entire assembly is then inverted and fitted into the water-cooled base plate shown to the right in Figure 8. This completes the assembly as shown in Figure 9. In the prototype assembly, die inserts have been produced to simulate the final die cavities. The pieces were produced of castable zircon refractory, as shown in Figure 10.

III. SUMMARY

Experiments have been performed to determine the feasibility of using a viscous protection coating rather than the conventional solid coatings for the refractory metals. A high-temperature barium chloride type heat-treating salt, rectified with carbon, successfully inhibits oxidation loss at 2400° F. The need for immersion does, however, dictate several restrictions on the die design in order to maintain a pool of liquid salt over the susceptible parts of the high-temperature die system. The problem of die component creep at high temperature imposes additional requirements on the compatibility of the various materials used in the die system.

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A prototype die system to be used for high-temperature heating studies has been constructed and is described in detail. Heating to 2400°F will be accomplished through electrical resistance heating elements supplemented by gas flame radiation on the die surface.

IV. FUTURE WORK

During the next work period research effort will continue on Phase III as follows:

1. Continue and extend the work on oxidation protection refractory metals utilizing combinations of salt.
2. Evaluate Chromalloy coated P4 samples for oxidation resistance at 2400°F when available.
3. Investigate potential die-retainer combinations, and develop design data for stress situations under hot forging conditions.
4. Complete and assemble prototype forging die system, and investigate heating characteristics, temperature distributions, and related effects.
5. Continue and extend the work on oxidation protection of refractory metals utilizing combinations of salts.

V. LOGBOOKS AND CONTRIBUTING PERSONNEL

Data gathered during the research work are contained in ARF Logbooks C 11166, C 11167, C 11168, C 11169, and C 11908. Foundation personnel include:

R. Corrado	A. H. Murphy
R. Domkowski	H. B. Nudelman
J. Dorcic	R. J. VanThyne
P. R. Gouwens	T. Watmough

Respectfully submitted,

ARMOUR RESEARCH FOUNDATION OF ILLINOIS INSTITUTE OF TECHNOLOGY

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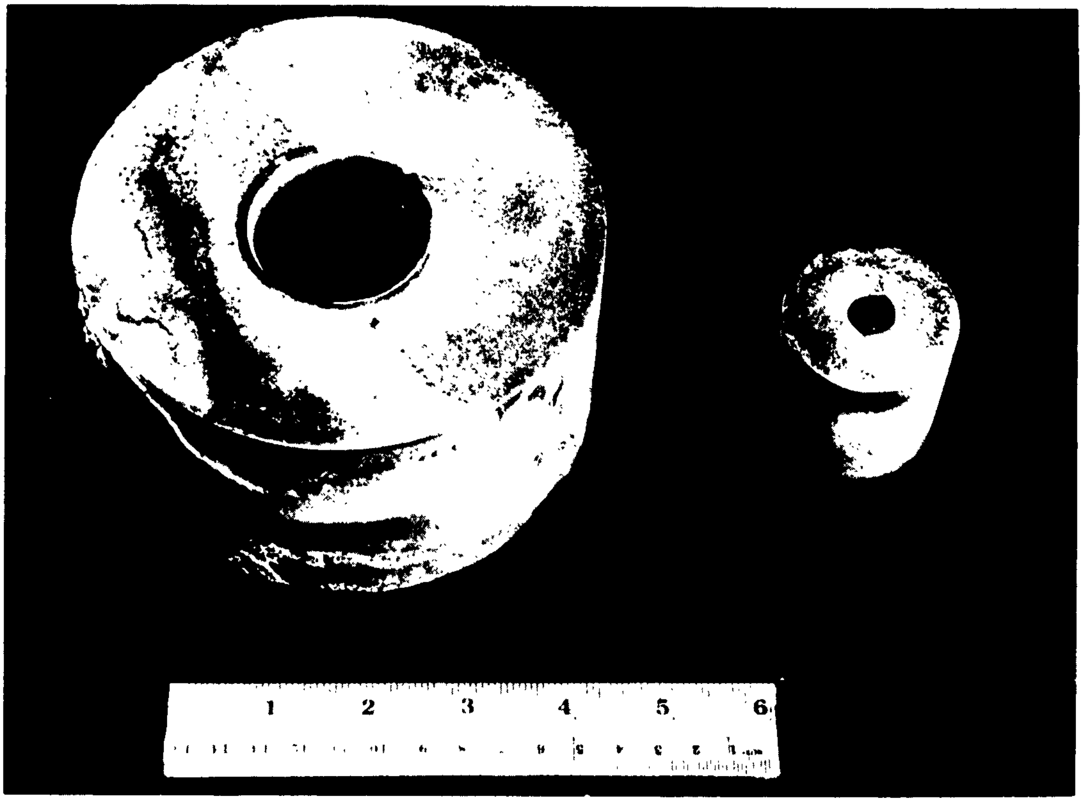

Neg. No. 24122

FIG. 1 - OXIDATION ATTACK OF 97% W-3% Mo DIE RETAINER AND TITANIUM DIBORIDE INSERT AFTER EXPOSURE TO 2400°F IN A 90% N-10% H_2 ATMOSPHERE.

Neg. No. 24119

FIG. 2 - SURFACE CONDITION OF TUNGSTEN ROD AND REFRAX CYLINDER AFTER 2400°F EXPOSURE FOR 15 MINUTES IN AIR.

Neg. No. 24120

FIG. 3 - SURFACE CONDITION OF TUNGSTEN ROD AND KT SILICON CARBIDE CYLINDER AFTER 2400°F EXPOSURE FOR 15 MINUTES IN AIR.

Neg. No. 24042

FIG. 4 - PROTOTYPE FORGING DIE COMPONENTS. LOWER DIE IS TO THE LEFT AND DISASSEMBLED UPPER DIE TO THE RIGHT.

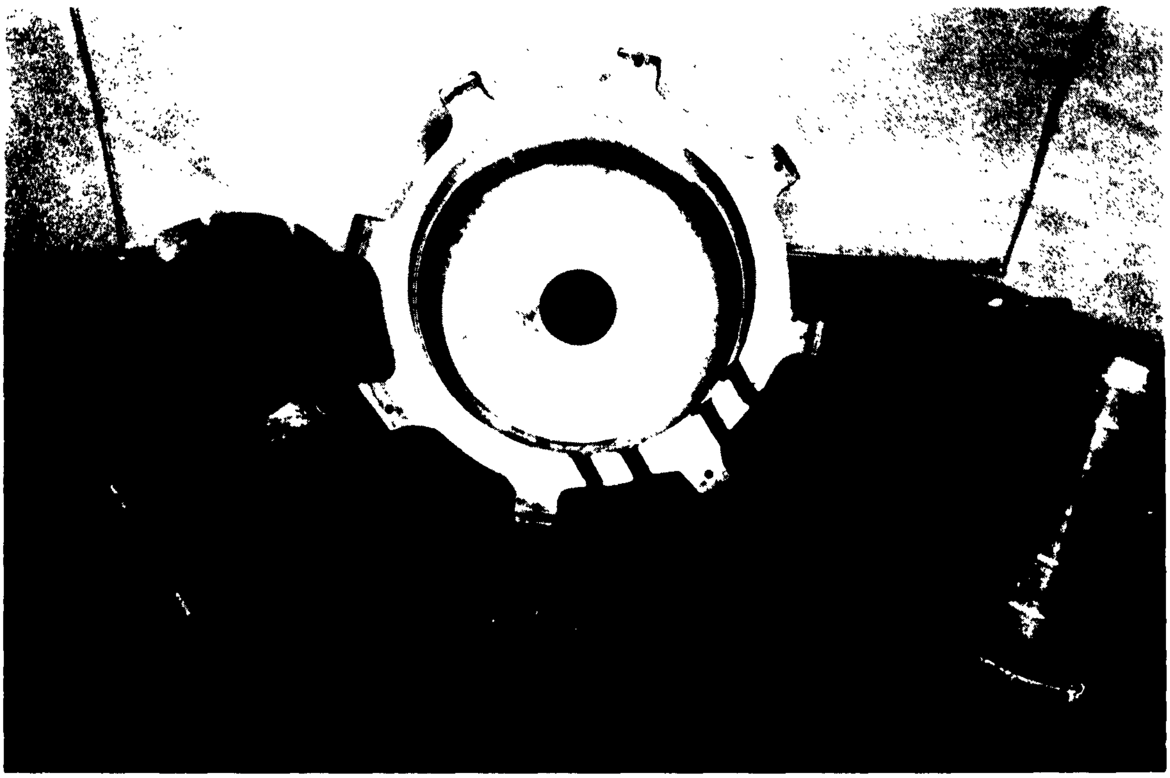

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FIG. 5 - DIE RETAINER PLACED WITHIN HOLD-DOWN CLAMP.

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FIG. 6 - OUTER SUPPORT BLOCK IN POSITION WITHIN HOLD-DOWN CLAMP.

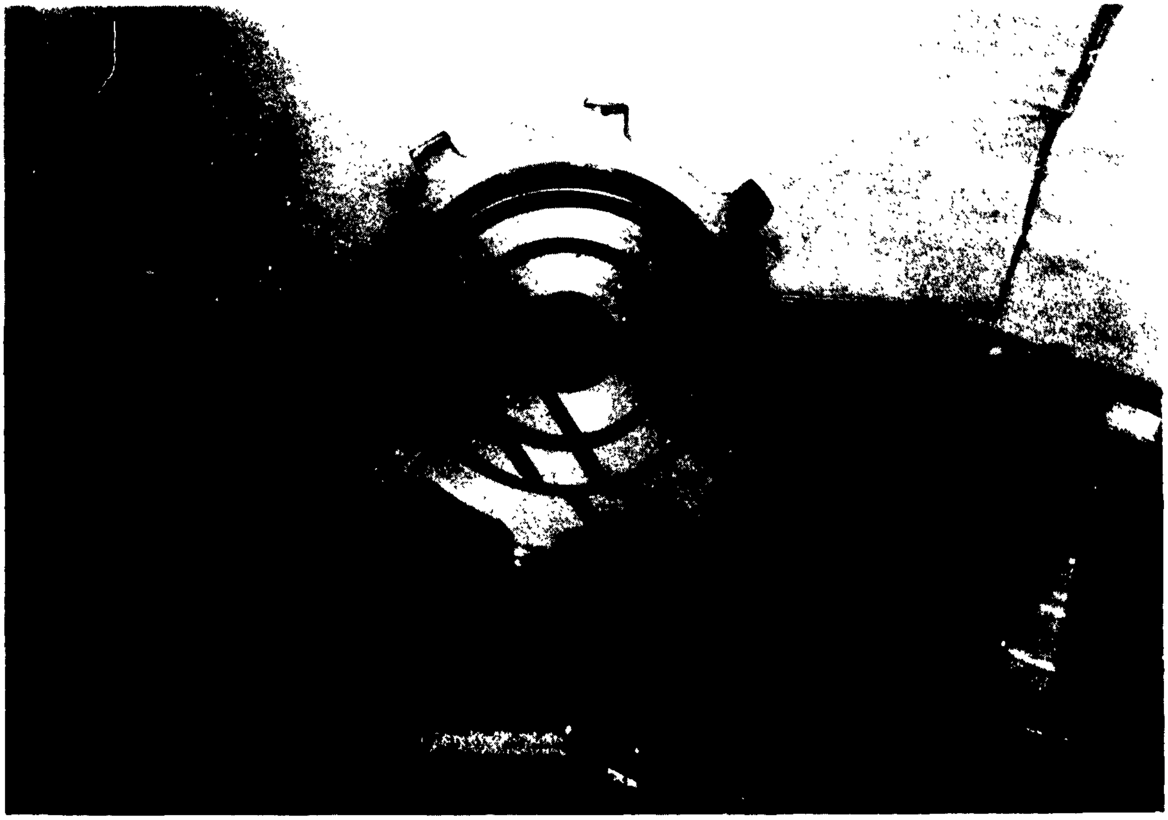

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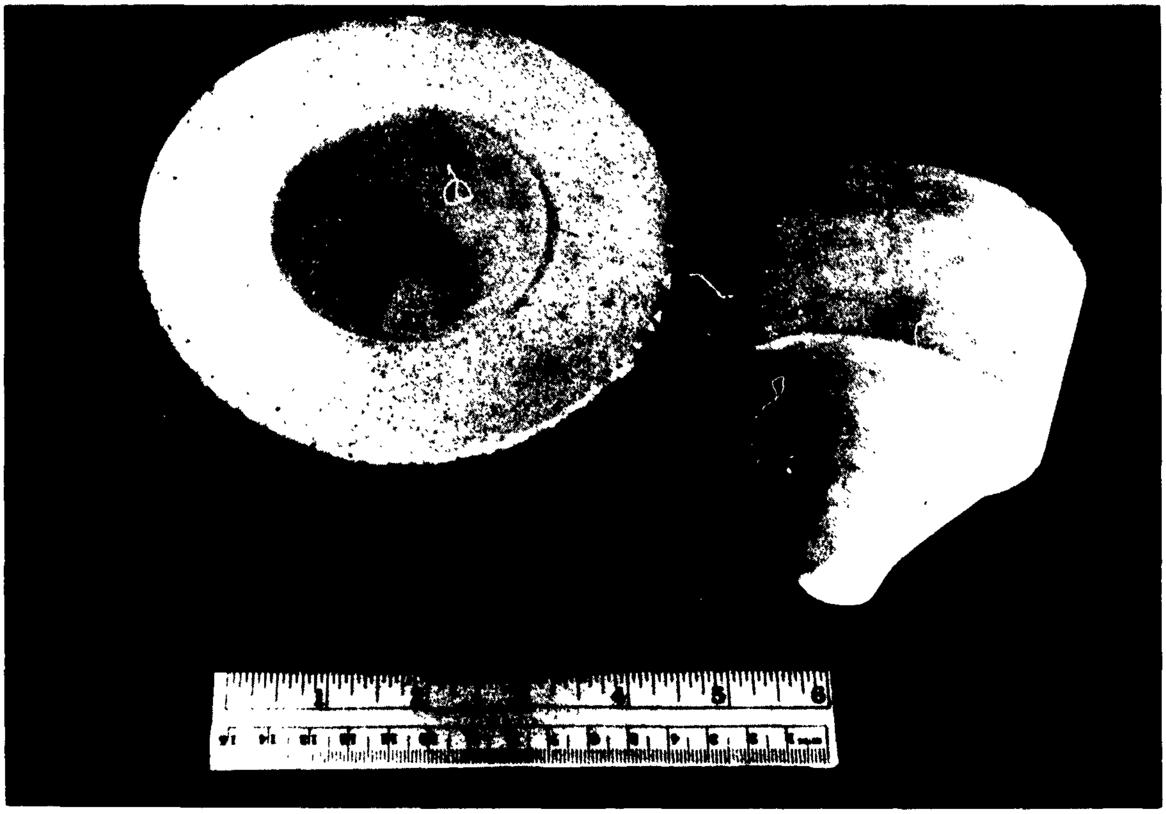
FIG. 7 - MIDDLE SUPPORT BLOCK IN POSITION WITHIN ASSEMBLY.

Neg. No. 24046

FIG. 8 - FINAL SUPPORT CORE IN PLACE WITHIN THE DIE ASSEMBLY.

Neg. No. 24047

FIG. 9 - ASSEMBLED PROTOTYPE DIE (NOT INCLUDING FINAL CAVITY PORTION.)



Neg. No. 24121

FIG. 10 - SIMULATED DIE INSERT SHAPES FOR PROTOTYPE HEATING EXPERIMENTS.

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Project 7-886

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