NASA CR-54316
AI-65-12

FINAL REPORT
DEVELOPMENT
OF A
MAGNEFORMING PROCESS
FOR THE
FABRICATION OF THIN-WALL TUNGSTEN CYLINDERS

By

L. ABRAHAM, N. H. KATZ, AND E. C. SUPAN

Prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

CONTRACT NAS 3-5211

ATOMICS INTERNATIONAL

A DIVISION OF NORTH AMERICAN AVIATION, INC.

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Prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
JANUARY 13, 1965
CONTRACT NAS 3-5211
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ATOMICS INTERNATIONAL
A DIVISION OF NORTH AMERICAN AVIATION, INC.

ABSTRACT

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Tungsten powders were consolidated into thin-wall cylinders in a die which compacted the powders at elevated temperature, by virtue of the differences in thermal expansion between the die body and mandrel materials. Feasibility of using this consolidation method as a cylinder preforming process was demonstrated, and densities up to 75% of theoretical were obtained. Consolidated cylinders were formed at 1800°C and further densified by Magneforming.

Magneforming, a high-energy-rate metal-forming technique, was adapted to high-temperature, vacuum-atmosphere operating conditions. Special tooling and methods were developed to enable metal-forming operations to be carried out at temperatures as high as 1427°C. Presintered thin-wall tungsten cylinder preforms were densified an additional 5 to 10% by this method, but cracked during a subsequent leaching step, which is required to release the cylinders from the Magneform workpiece assembly.

Equipment, methods, and results are described, and process recommendations are made.

CONTENTS

TABLES

FIGURES

FIGURES

DEVELOPMENT OF A MAGNEFORMING PROCESS FOR THE FABRICATION OF THIN-WALL TUNGSTEN CYLINDERS

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SUMMARY

The ultimate objective of this program was the development of a magnetic pulse forming method (Magneforming) for the fabrication of thin-wall tungsten cylinders having a density >98% of theoretical. The scope of the work undertaken in Phase I, which is described in this report, was limited to the feasibility demonstration of novel processing methods for tungsten powder consolidation, final forming, and densification into thin-wall cylindrical shapes. The specific objectives of this program include the concurrent development of a powder consolidation and predensification process, and a final-forming-and-densification high-temperature Magneforming process. At the conclusion of the time period allotted for the Phase I work, some process refinements still remained to be made, in order to meet all of the objectives.

The tungsten powder consolidation method selected for development in this program was based on the principle of differential thermal expansion characteristics of the structural components of a consolidation die. The feasibility of this novel method of tungsten powder consolidation into thin-wall cylindrical shapes was demonstrated, and the results are encouraging. Further work is required, however, to increase the compact density and to completely eliminate density gradients in the cylinder wall. Additional process parameter studies, relating to powder particle sizing and powder loading techniques, are needed to resolve the remaining process difficulties. However, support of this program was terminated by NASA.

Best results were obtained with a consolidation die which used graphite as the die body material in conjunction with an alumina mandrel. The graphite-alumina combination permitted powder consolidation temperatures as high as 1800°C (3272°F), and resulted in generally well-formed, relatively strong, thin-wall, predensified tungsten cylinders. Density of 1-1/4 in. OD cylinders did not exceed 75% of theoretical. Smaller sized cylinders, produced earlier in the

program, had densities as high as 92%. An alternate consolidation die materials combination, TZM molybdenum alloy with stainless steel, was found to be less satisfactory.

Several attempts were made to produce thin-wall cylinders by ambient-temperature Magneform compaction of tungsten powder, followed by sintering. Compacted cylinders were successfully formed by this method; however, cylinders produced in this manner generally exhibited cracking or end defects, or actual breaking away of end segments. While experiments to date have shown this process to be a promising alternate method for tungsten powder consolidation into thin-wall cylindrical shapes, an extensive development effort is required to resolve all of the processing difficulties.

Magneforming, a high-energy-rate metal-forming technique, is conventionally used under ambient temperature conditions. For the purpose of final-forming and densifying sintered tungsten, thin-wall cylinders, it was required that the Magneforming method be adapted to high-temperature vacuum (or inert) atmosphere operating conditions. At working temperatures as high as 1427°C (2600°F), operational performance of this high-temperature Magneforming system was excellent, and resulted in good sizing of the tungsten cylinder to a mandrel assembly. However, densification of the tungsten cylinders was considerably lower than that required by the product specification; and cracking of the cylinders occurred during a subsequent leaching operation, which is used to separate the tungsten cylinders from the workpiece assembly. The cracking is presumably the result of a stress condition.

Development of the high-temperature Magneforming process was brought to a point where most of the major special tooling and materials selection problems were successfully resolved. While progress, from the initial concept to the present stage of development, is very encouraging, a final demonstration of process feasibility, specific to the densification of sintered tungsten cylinders, requires additional process parameter studies. The reasons for the cracking of tungsten cylinders during leaching from the workpiece assembly must be determined, and the problem eliminated. It is felt that further Magneforming experiments, in the 1650°C (3000°F) working range, will achieve full densification of the thin-wall tungsten cylinders and may avoid the cracking problem by improvement in the mechanical properties of tungsten. Refinements in the design of the hot swaging assembly should result in further improvement in the high-temperature Magneforming process.

I. INTRODUCTION

The cylinder fabrication method described in this report employs novel processing concepts which are particularly applicable to the fabrication of thin-wall, seamless, tungsten cylinders which are required by the NASA-Lewis Research Center. The ultimate objective of the process development under this contract was to develop a magnetic pulse forming method (Magneforming) for the fabrication of thin-wall tungsten cylinders. Processing variables and equipment designs, necessary to the production of high-density cylinders, were to be established; and the final product was to meet the following requirements:

a) The cylinders must be highly densified, to at least 98% of theoretical.

b) Wall thickness of the cylinders must be uniform within 0.001 in. for a specimen size of 1-1/4 in. OD by 0.020 in. wall by 1-1/2 in. long.

The contract objective was to be achieved in two phases. In Phase I, which is summarized in this report, the fabrication process was to be developed for high-temperature forming and densification operations, applicable to thin-wall tungsten cylinders. If this were successfully achieved, the subsequent Phase II work included final development of the fabrication process for W-base composite cylinders. However, work on Phase II was not initiated because of termination of the program by NASA.

The reference thin-wall tungsten cylinder specimen size used for development purposes in Phase I was 1-1/4 in. OD by 0.020 in. wall by 1-1/2 in. long. The specific objectives of Phase I included the concurrent development of a powder consolidation and predensification process and a final-forming-and-densification high-temperature Magneforming process.

This report describes the design and development work undertaken to demonstrate the feasibility of novel processing methods for tungsten powder consolidation, final forming, and densification into the thin-wall cylinders.

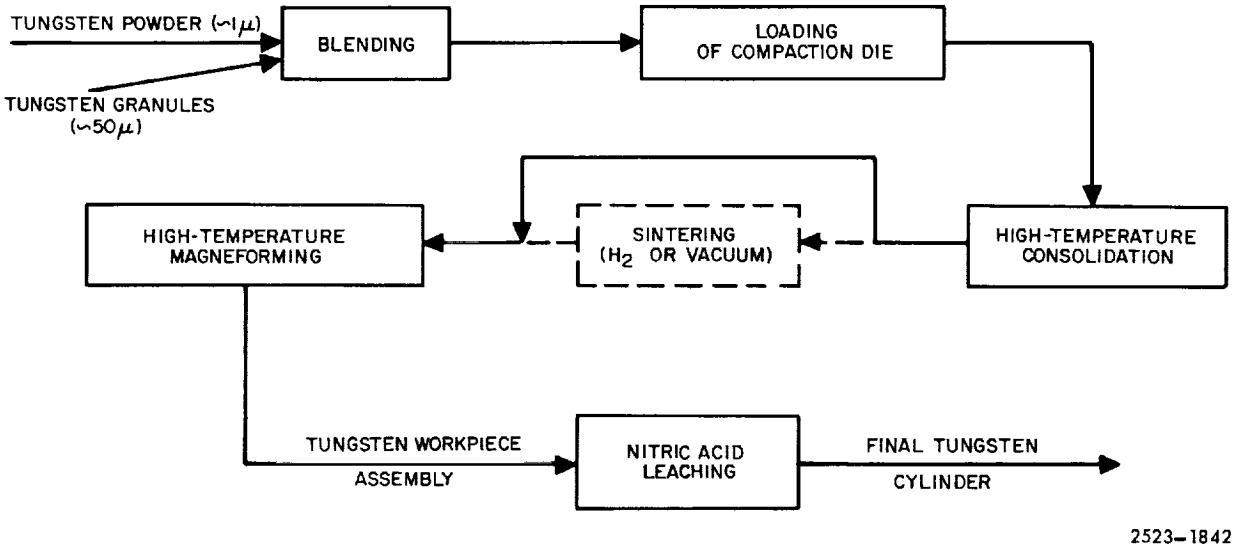


Figure 1. Process Flow Diagram

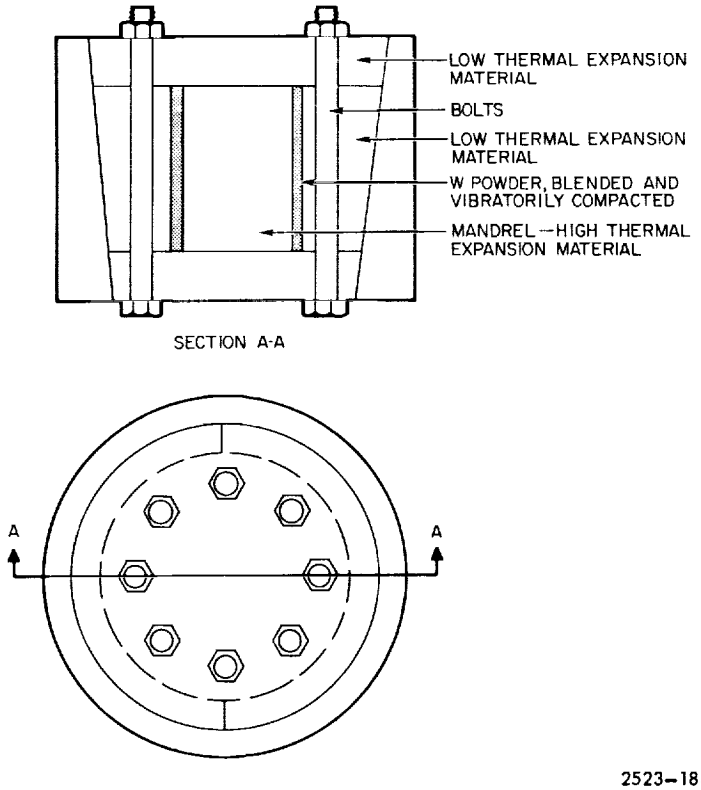


Figure 2. Differential Thermal Expansion Fixture

II. FABRICATION PROCESS

A simplified flow diagram of the overall fabrication process is given in Figure 1. A blend of coarse (~50μ) and fine (~1μ) tungsten particles was used in this program because of the NASA requirements on the final product. The blended powders are loaded into an annular consolidation die by vibratory compaction on a fixed frequency mechanical vibrator, followed by a final tamping step. The die surfaces, which form the annular space provided for powder consolidation, are precoated with a parting agent, such as ZrO_2 slurry.

The powder consolidation die is based on the principle of differential thermal expansion characteristics of structural materials. The cylindrical mandrel of this die (see Figure 2) is fabricated from a structural material having a coefficient of thermal expansion which is high, relative to that of the material from which the die body is made.

The loaded compaction die is transferred to a vacuum atmosphere furnace, where it is heated to a temperature of 1500 to 1800°C (2732 to 3272°F) for 2 to 4 hr. This high-temperature consolidation step forms a sintered, predensified, thin-wall cylinder having a wall thickness somewhat greater than that ultimately desired. The predensified cylinder is removed from the consolidation die, and may optionally be given an additional sintering treatment (in either hydrogen or vacuum atmosphere) at a temperature of ~ 1750°C (~ 3182°F).

The final fabrication process step is a high-temperature magnetic pulse swaging (Magneforming) operation, where the tungsten cylinder is final-formed and densified under radial pressures of ~ 50,000 psi. In the Magneforming operation, the sintered cylindrical tungsten compact is positioned over a refractory ceramic mandrel and tubular refractory metal heating element, and surrounded by an electrically conductive tube. The tube (or "driver") constitutes the primary workpiece for magnetic pulse forming, and pressure is transmitted to the predensified tungsten cylinder by the collapsing of the driver (as a result of the magnetic pulses). The entire assembly is enclosed in a quartz or Vycor envelope which is evacuated prior to initiating the heating and Magneforming cycle. The tubular heating element is brought to the desired operating temperature [> 1200°C (> 2192°F)] by resistance heating, and magnetic pulse forming is applied immediately after the tungsten cylinder reaches the required operating

temperature. Normally, heating to temperature and Magneforming is accomplished in 8 to 10 sec. The formed and densified thin-wall tungsten cylinder is finally separated from both the collapsed driver and the ceramic mandrel-heater tube assembly by a chemical leaching operation.

The operation of Magneform* is based upon the discharge of electrical energy from a capacitor bank through a coil. The collapsing magnetic field causes eddy currents to be induced into a conductive workpiece placed inside the coil. This results in a repulsive force between the coil and the workpiece, due to the interaction of their magnetic fields, in accordance with Lenz's law. The coil must be rigidly constructed, to withstand the force equal and opposite to that exerted on the workpiece. Maximum forming pressures are typically of the order of 50,000 psi, in a time lapse of 10 to 100 μsec.†

The energy output of the Magneform machine used in this work is continuously adjustable from 0 to 6 kj. An adjustable autotransformer controls the maximum voltage charge (E) on the capacitor bank, and the stored energy is proportional to E^2. The machine is designed to automatically charge and discharge in a full-power operational sequence of about 6 sec, with less time required for lower voltage settings.

The Magneform machine is equipped with a standard 4-in. ID work coil. In order to concentrate the compressive forces, a "field shaper" is inserted into the work coil, which effectively localizes the induced currents in the workpiece over a short length (usually, 2 in. or less). The field shaper is essentially a single-turn coil which is electrically insulated from both the work coil and the workpiece. For the experimental effort of this program, three field shapers were fabricated from Type 6061-T6 aluminum alloy, as shown in Figure 3.

The diameter (D) is selected to minimize the annulus between the field shaper and the conductive workpiece, for maximum swaging performance through inductive coupling. The effective swaging length is 2 in.

If the resistivity of the workpiece exceeds about 15 $\mu\Omega$-cm, a high conductivity driver material should be placed between the field shaper and the workpiece, to increase the swaging force. Fully annealed copper and aluminum are the better driver materials (based on electrical conductivity and cost). If the driver must

*Equipment manufactured by General Atomic Division of General Dynamics Corp.
†These values are stated by the manufacturer of Magneform equipment.

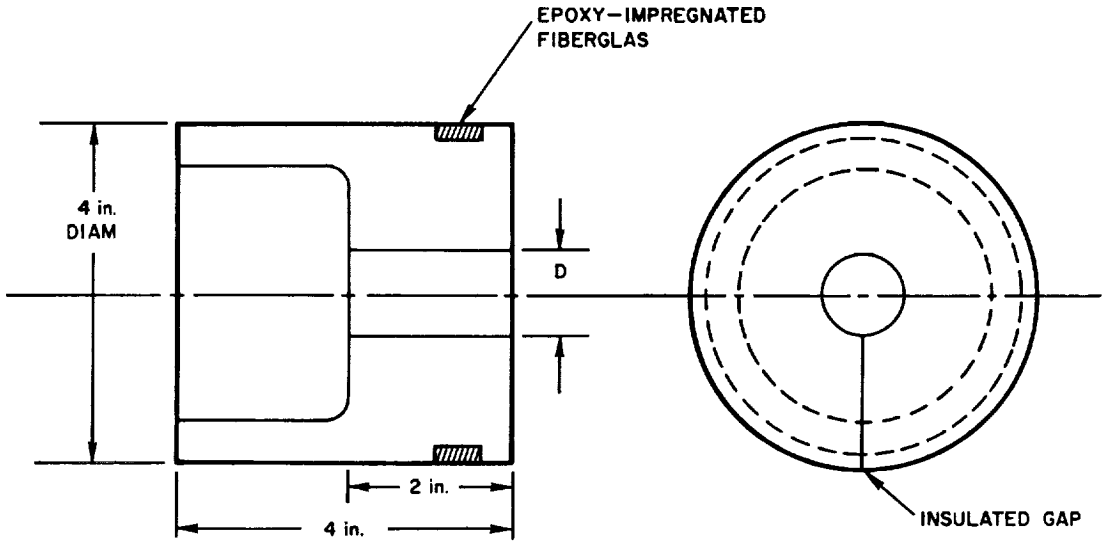


Figure 3. Field Shaper for Magnetic Pulse Forming (Type 6061-T6 aluminum alloy)

undergo severe deformation during swaging, aluminum is the more efficient driver material, because of its lower strain-hardening characteristics; otherwise, copper is better than aluminum, because of its higher conductivity.

In the specialized case of hot swaging, overheating of the driver should be avoided, because of the associated increase in its resistivity. Obviously, temperatures sufficient to melt the driver must also be avoided.

The Phase I work scope included the design, construction, and operation of special tooling required for

a) The powder consolidation and predensification step

b) The final-forming-and-densification high-temperature Magneforming step,

as well as the development of the required process parameters for the overall fabrication process. The many special tooling design and materials selection problems to be resolved in Phase I included areas such as:

a) Determining suitable high-temperature material combinations, for use as structural materials for the "thermal differential expansion" powder consolidation die

b) Developing a suitable method of heating the preformed tungsten cylinder to the desired Magneforming temperature, and providing a thermal barrier for the driver

c) Selecting suitable materials to be used in the Magneforming assembly (i.e., materials for the driver, the heating element, and the mandrel)

d) Selecting suitable materials and methods for accomplishing release of the thin-wall tungsten cylinder from both the powder consolidation and Magneforming tooling.

III. EXPERIMENTAL WORK

A. POWDER CONSOLIDATION

1. Differential Thermal Expansion Die Method

A novel approach to the forming of hollow cylinders was conceived specifically for the fabrication of thin-walled tungsten cylinders. The method consists of consolidating tungsten powders in a unique fixture, designed around the principles of differential thermal expansion characteristics of structural materials. The fixture, illustrated in Figure 2, is essentially a die which contains a cylindrical mandrel fabricated from a structural material having an appreciably higher thermal expansion coefficient than the die. Tungsten powder is loaded into an annular space between the die and the mandrel, and the entire assembly is raised to an elevated temperature. The die and mandrel cause a radial compacting pressure to be exerted on the powders, by virtue of their different coefficients of thermal expansion. The compact formed may be sintered subsequently, to achieve further densification.

All of the powder consolidation experiments (summarized in Table I) performed in this program may be categorized into five groups, based on major changes made in process procedures, as required to produce a complete tungsten cylinder. The experimental groups will be defined here, and discussed in detail in subsequent sections.

Group I: (Initial Experiments) Direct Loading of Powders into TZM Die

Group II: Metal Sleeves – TZM die

Group III: Low Expansion Sleeves – TZM Die

Group IV: Low Expansion Sleeves – Graphite Die

Group V: Graphite Die – Al_2O_3 Mandrel

The major problem areas associated with the development program are identified here, and their numbers are used with the respective experiments listed in Table I:

1. Low compact strength after warm consolidation; difficult to handle
2. Adherence of tungsten compact to die wall
3. Adherence of tungsten compact to mandrel

TABLE I

WARM CONSOLIDATION EXPERIMENTS
(Sheet 1 of 3)

Group	Experiment	Sleeves		Mandrel	Wash Coating	Consolidation		Post Consolidation Treatment				Results	Problem Areas
								Presintering		Sintering			
		Outer	Inner			Temperature (°C)	Time (hr)	Temperature (°C)	Time (hr)	Temperature (°C)	Time (hr)		
I	WC-1	None		SS-316	Molykote X-15	750	2					Compact adhered to die walls. Could not be removed intact.	1, 2, 3
I	WC-2	None		SS-316	Molykote X-15	750	2					Compact adhered to die walls. Could not be removed intact.	1, 2, 3
I	WC-3	None		SS-316	Stabilized ZrO_2	750	2	1400	4	2200	6	Portion of compact adhered to mandrel. Compact distorted severely after sintering.	2, 3, 4, 5
I	WC-4	None		SS-316	Stabilized ZrO_2	1000	2					Compact adhered to die walls. Could not be removed.	2, 3,
II	WC-5	SS	Cu	SS-316	Stabilized ZrO_2	750	2					Assembly of outer sleeve, compact, inner sleeve, and mandrel was easily removed, but mandrel could not be removed from assembly.	
II	WC-6	SS	None	SS-316	Stabilized ZrO_2	750	2					Mandrel could not be removed.	
III	WC-7	ATJ Graphite	None	SS-316	Stabilized ZrO_2	750	2	1400	4	1750	4	Small portion of compact adhered to mandrel. Remainder of compact was good. Compact had cracks after sintering.	3, 4, 5
III	WC-8	ATJ Graphite	None	SS-316	Stabilized ZrO_2	750	2					Portion of compact adhered to mandrel, resulting in a nonuniform compact. Could not be sintered.	1, 3, 4
III	WC-9-11	Graphite	None	SS-316	Stabilized ZrO_2	750	2					Portion of compact adhered to mandrel, resulting in a nonuniform compact. Could not be sintered.	1, 3, 4
III	WC-12	Graphite	None	Polished SS-316	ZrO_2 used on all parts except mandrel	750	2	1400	4	1750	4	Small portion of compact adhered to mandrel. ID of compact machined to remove defect region. Compact, sintered around graphite tube, was found to have cracked.	3, 4, 5
III	WC-13	Graphite	None	Polished SS-316	ZrO_2 used on all parts except mandrel	750	2	1400	4	1750	4	Results were very similar to WC-12	3, 4, 5
III	WC-14	Graphite	None	Polished Cr-Plated SS	ZrO_2 used on all parts except mandrel	750	4					Compact adhered to mandrel. Could not be removed intact.	1, 3, 4
III	WC-15	Graphite	None	Polished SS-316	ZrO_2 used on all parts except mandrel	750	4					Compact adhered to mandrel. Could not be removed intact.	1, 3, 4
IV	WC-16	Graphite	None	SS-316	Stabilized ZrO_2	1300	2					Portion of mandrel fused. Tungsten powder was well compacted, but adhered to mandrel and could not be removed intact.	3, 4
III	WC-17	Graphite	Graphite	SS-316	Stabilized ZrO_2	750	2	1400	4	1600	4	Mandrel easily removed. Compact had cracks which coincided with cracks in inner sleeve. Compact retained shape after sintering, but had many cracks.	4, 5
IV	WC-18	Graphite	None	SS-316	Stabilized ZrO_2	1100	2					Compact adhered to mandrel. Could not be removed intact.	3, 4
IV	WC-19	Graphite	Graphite	SS-316	Stabilized ZrO_2	1100	2			1600	4	Mandrel easily removed. Compact ID had several surface cracks which coincided with cracks in inner sleeve. Compact sintered without inner sleeve. Retained shape and cracks along ID surface. Considered potential reference procedure.	5

TABLE I

WARM CONSOLIDATION EXPERIMENTS
(Sheet 2 of 3)

Group	Experiment	Sleeves		Mandrel	Wash Coating	Consolidation		Post Consolidation Treatment				Results	Problem Areas
								Presintering		Sintering			
		Outer	Inner			Temperature (°C)	Time (hr)	Temperature (°C)	Time (hr)	Temperature (°C)	Time (hr)		
V	WC-20	Graphite	None	Al_2O_3	ZrO_2 on graphite parts only	1100	2					Compact adhered to Al_2O_3 mandrel and could not be removed intact.	3, 4
V	WC-21	Graphite	Graphite	Al_2O_3	ZrO_2 on graphite parts only	1600	1					Mandrel was easily removed. Compact had only one crack, which coincided with crack in graphite sleeve. Tungsten powder was well compacted and could be handled. Compact did not distort. Considered as potential reference procedure.	5
V	WC-22	Graphite	None	Al_2O_3	ZrO_2	1600	2			1800	4	Composite cylinder was formed at 1600°C. Could not be removed from mandrel. Tungsten cylinder plus Al_2O_3 mandrel were sintered at 1800°C in vacuum. There was some evidence of a reaction between tungsten and die components, and some reduction of the Al_2O_3 mandrel. However, a complete uncracked cylinder was obtained. The sintering temperature is considered to be excessive, for the materials used in this experiment.	3
V	WC-23	Graphite	None	Al_2O_3	ZrO_2	1650	2					Compact was easily removed from die and mandrel. The only apparent defect was some small cracks near one end of the cylinder. The cylinder had considerable strength, and could be handled without difficulty.	
V	WC-24	Graphite	None	Al_2O_3	ZrO_2	1650	2			1750	4	The cylinder was removed without difficulty and was complete. No defects could be observed.	
V	WC-25*	Graphite	None	Al_2O_3	ZrO_2	1650	2					Two compacts were prepared. Compacts were intact on OD, but could not be removed from mandrel	3
V	WC-26*	Graphite	None	Al_2O_3	ZrO_2	1700	1					Ta end shields used prevent reaction with graphite. Ta appeared to react with graphite, and adhered to compacts. Compacts could not be removed.	3
V	WC-27*	Graphite	None	Al_2O_3	ZrO_2	1650	2					Some fusion observed in tungsten compacts which is unexplainable at this time.	3
V	WC-28*	Graphite	None	Al_2O_3	ZrO_2	1450	2					Two compacts adhered to mandrel. Third was removed after slitting axially. Compact was submitted for further densification by Magneforming.	3
V	WC-29	Graphite	None	Al_2O_3	ZrO_2	1650	2					Compact adhered to mandrel.	3
V	WC-30	None		Al_2O_3	ZrO_2	1650	2					Compact adhered to mandrel.	3
V	WC-31	None		Al_2O_3	ZrO_2	1700	2					Compact adhered to mandrel.	3
V	WC-32	None		Al_2O_3	ZrO_2	1800	2					Compact easily removed from mandrel. Some pits on surface.	
V	WC-33	None		Al_2O_3	ZrO_2	1650 plus 1800	2 2					Severe reaction between mandrel and graphite. Compact was distorted.	

TABLE I

WARM CONSOLIDATION EXPERIMENTS
(Sheet 3 of 3)

Group	Experiment	Sleeves		Mandrel	Wash Coating	Consolidation		Post Consolidation Treatment				Results	Problem Areas
								Presintering		Sintering			
		Outer	Inner			Temperature (°C)	Time (hr)	Temperature (°C)	Time (hr)	Temperature (°C)	Time (hr)		
V	WC-34†	None		Al_2O_3	ZrO_2	1800	2					Compact cracked during removal.	3
V	WC-35†	None		Al_2O_3	ZrO_2	1800	2					Compact adhered to mandrel.	3
V	WC-36	None		Al_2O_3	ZrO_2	1800	2					Compact adhered to mandrel.	3
V	WC-37†	None		Al_2O_3	ZrO_2	1800	2					Compact adhered to mandrel.	3
V	WC-38†	None		Al_2O_3	ZrO_2	1800	2					Compact easily removed from mandrel.	
V	WC-39	None		Al_2O_3	ZrO_2	1800	2					Compact easily removed from mandrel.	
V	WC-40	None		Al_2O_3	ZrO_2	1800	2					Compact adhered to mandrel.	3
V	WC-41	None		Al_2O_3	ZrO_2	1800	2					Compact removed from mandrel. Some lamination near one end.	
V	WC-42	None		Al_2O_3	ZrO_2	1850	2					One-half of compact adhered to mandrel. Compact could not be removed intact.	3
V	WC-43	None		Al_2O_3	ZrO_2	1800	2					Compact released from mandrel and was found to have laminated, due to uneven powder compaction during initial loading.	
V	WC-44	None		Al_2O_3	ZrO_2	1800	2					Compact easily released from mandrel.	
V	WC-45	None		Al_2O_3	ZrO_2	1800	2					Compact easily released from mandrel.	
V	WC-46	None		Al_2O_3	ZrO_2	1800	2					Compact easily released from mandrel.	
V	WC-47	None		Al_2O_3	ZrO_2	1800	2					Compact was removed from mandrel, but exhibited some lamination near one end.	
V	WC-48§	None		Al_2O_3	ZrO_2	1800	2					Compact adhered to mandrel and was cracked during removal.	
V	WC-49**	None		Al_2O_3	ZrO_2	1800	2-1/2					Compact was removed from mandrel, but exhibited concavity around the mid-section.	
V	WC-50††	None		Al_2O_3	ZrO_2	1800	2-1/2					Same as WC-49. Compact could not be removed without being cracked.	
V	WC-51§§	None		Al_2O_3	ZrO_2	1825	2-1/2					Compact adhered to mandrel.	3
V	WC-52	None		Al_2O_3	ZrO_2	1825	2-1/2					Incomplete run. No compacts.	
V	WC-53***	None		Al_2O_3	ZrO_2	1825	2-1/2					Compact exhibited slight bow at mid-section.	

*Multiple cavity die.
†Run simultaneously in same furnace.
§Powder was vibrated and tamped into die cavity, then pressed from top at 3000 psig in a Pasadena press. A 2% stearic acid – alcohol binder was added to the powder.
**Powders cold pressed at both ends at 3000 psig.
††Powders cold pressed at both ends at 6000 psig.
§§Powders cold-pressed by Magneform, prior to consolidation.
***Powders cold-pressed at both ends at 8000 psig.

Problem Areas
1. Low compact strength after warm consolidation. Difficult to handle.
2. Adherence of tungsten compact to die wall.
3. Adherence of tungsten to mandrel.
4. Nonuniform compact, after removal of mandrel.
5. Compact cracked or distorted during sintering.

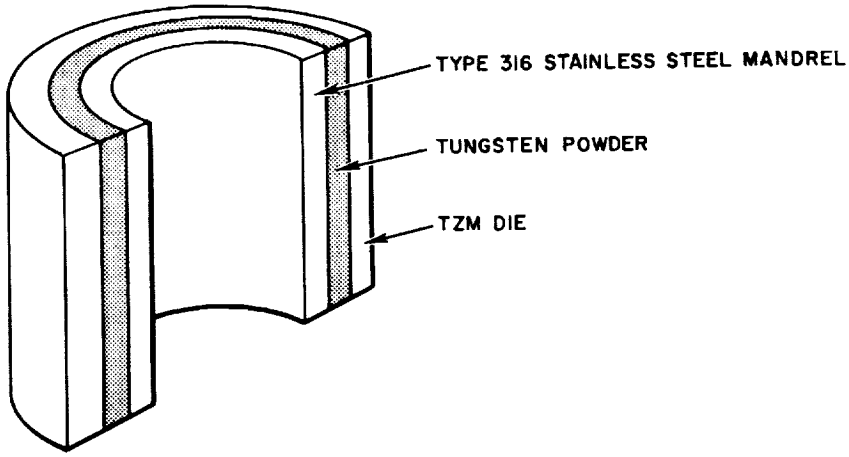
4. Nonuniform compact, after removal of mandrel

5. Compact cracked or distorted during sintering

a. Group I: (Initial Experiment) Direct Loading of Powder into TZM Die

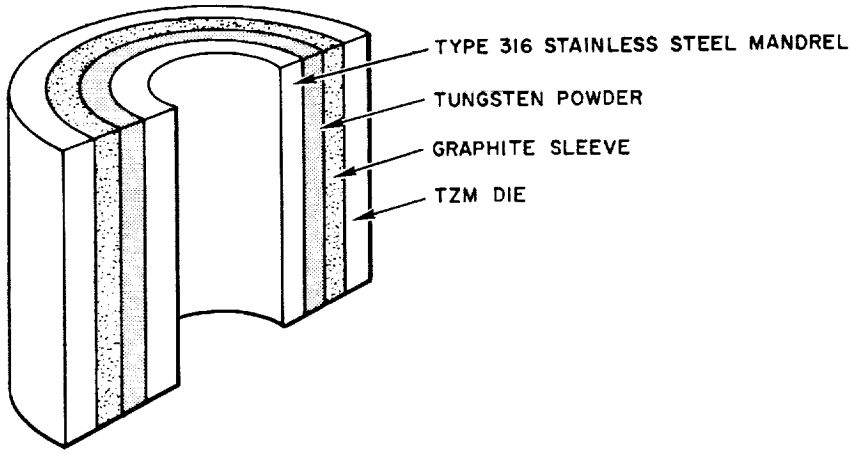
The first group of powder consolidation experiments was conducted, using TZM as the die material and Type 316 stainless steel as the mandrel material, assembled as illustrated in Figure 4a. The die was designed as a 4-segment split-type die (see Figure 5), to facilitate removal of the compact after consolidation. The die components are shown in Figure 6a. The mandrel was so designed that a 0.029-in. wide die-mandrel annulus was obtained when the mandrel was correctly positioned within the die cavity. Prior to filling the annulus with powder, the die components and mandrel were thoroughly coated with a parting agent composed of MoS_2 or ZrO_2 stabilized with MgO. The MoS_2 was applied as a fine spray, while the ZrO_2 was applied as an aqueous wash and subsequently oven dried. Tungsten powders, 4 to 7 μ, were added, without binder, to the annulus volume while the die-mandrel assembly (shown in Figure 6b) was vibrated (at a fixed frequency of 60 cps, with variations made in the amplitude until packing was complete). The vibrated powders were tamped, using a plastic tube as the ram, to eliminate any compaction density gradient which might have been introduced during the initial loading. The entire assembly, consisting of the die, mandrel, compacted powders, and die end plates bolted in place (see Figure 6c), was heated at a rate of 50°C/min, in vacuum, to 750°C (1382°F) and maintained for 2 hr, followed by cooling in vacuum. A 750°C (1382°F) limitation was originally imposed on the die, to avoid the possibility of excessive high pressure cracking the die. The 750°C (1382°F) temperature was considered to be a "warm" level, as compared to the much higher temperatures usually required to achieve sintering of tungsten.

The parameters used and the results obtained for the Group I consolidation experiments are given in Table I. In two of the experiments (WC-1 and WC-2), where MoS_2 was used as the parting agent on the die and mandrel, the tungsten powder adhered to either the die wall or mandrel, making removal of a tungsten cylinder, intact, impossible. Portions of the compact removed were found to have low green strength, which indicated that subsequent handling of the compact would be a sensitive operation. In the third experiment (WC-3), a ZrO_2 wash was substituted for MoS_2. This facilitated removal of the mandrel after warm



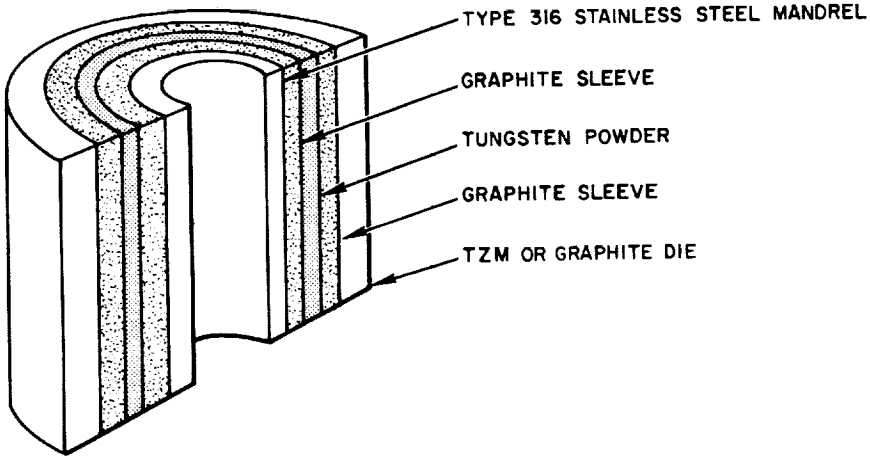
a. Initial Assembly

2523-1845



b. Graphite Outer Sleeve Added

2523-1846



c. Graphite Inner and Outer Sleeves Added

2523-1847

Figure 4. Powder Consolidation Die Experimental Assemblies

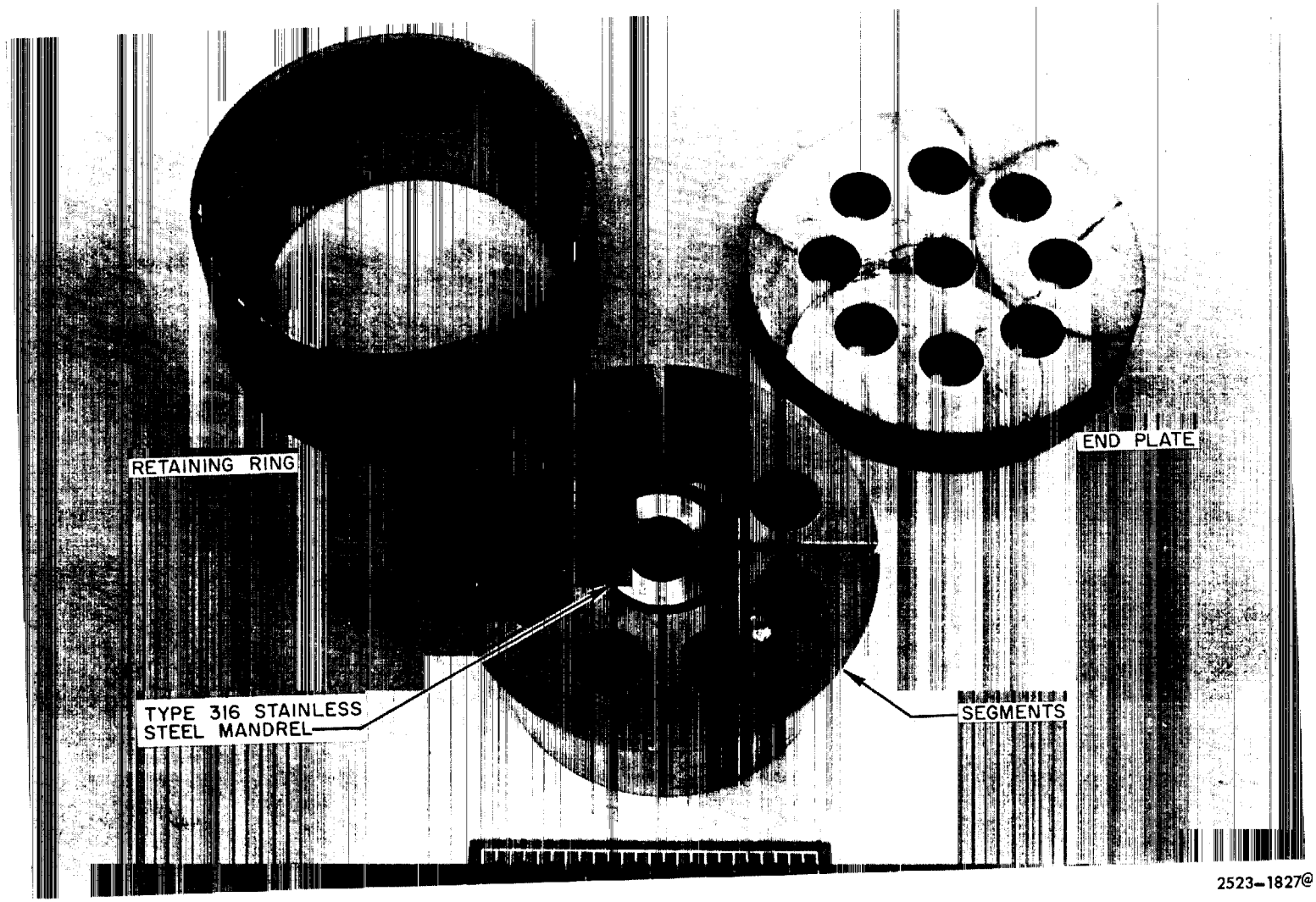
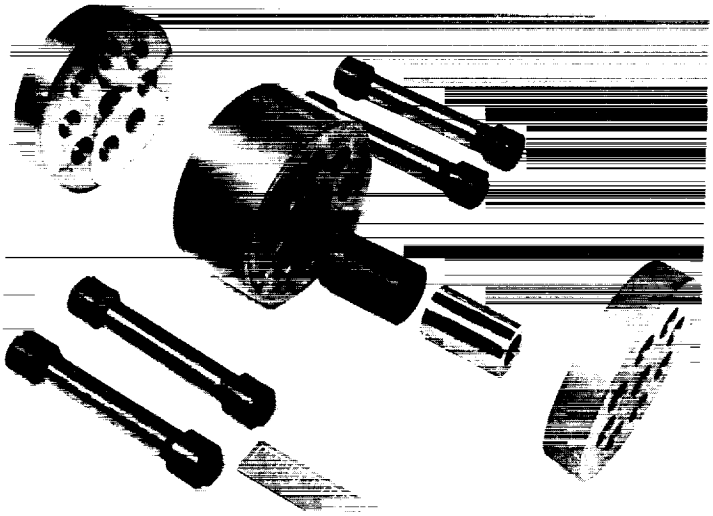


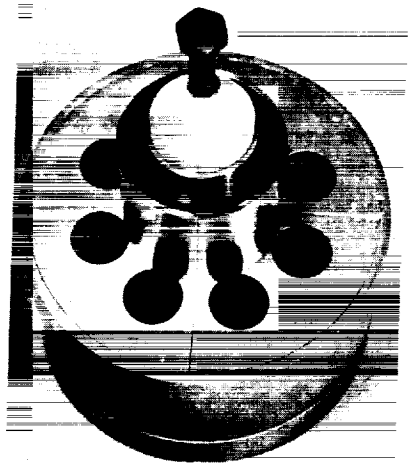
Figure 5. Four-Segment Split-Type TZM Die



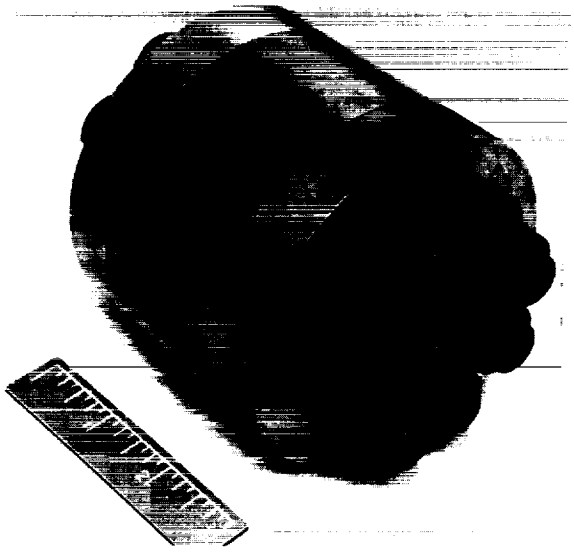
2523-1821

a. Components

b. Prepared for Final Loading (Type 316 stainless steel mandrel positioned within funnel above die cavity)



2523-1828



2523-1826

c. Assembly, Ready for Warm Consolidation Treatment

Figure 6. TZM Die

consolidation; however, a small portion of the compact did adhere to the mandrel. The die containing the tungsten compact, but not the mandrel, was given a presintering treatment at 1400°C (2552°F) for 4 hr in hydrogen, to increase the green strength. Following this treatment, the die was disassembled and the compact was removed intact. Raising the warm consolidation temperature to 1000°C (1832°F) in Experiment WC-4 appeared to strengthen the green compact, but did not prevent the tungsten from adhering to the mandrel.

In each experiment, it was evident that, after the warm consolidation treatment, the mandrel had thermally expanded against the powder and die, and had plastically deformed, first axially and then radially, as expected. The tungsten powder was definitely compressed, and the mandrel was found to have contracted away from the compact. Additional shrinkage and strengthening of one compact (WC-3) was obtained by sintering at 2200°C (3992°F) for 6 hr. However, this treatment also resulted in severe distortion of the compact, because it had a portion missing prior to being sintered.

b. Group II: Metal Sleeves – TZM Die

The ineffectiveness of a ZrO_2 wash as the parting agent on the TZM die wall and stainless mandrel, and the low green strength of the warm consolidated compact, prompted the introduction of metal sleeves as parting agents and support members. In two experiments (WC-5 and WC-6) performed in this group, thin-wall copper and/or stainless steel tubes were selected as the ID and/or OD sleeves, respectively. The process parameters and results obtained for WC-5 and WC-6 are given in Table I. The metal sleeves allowed easy removal of the mandrel-powder compact-sleeve assembly from the TZM die. However, all efforts to remove the mandrel from the assembly were unsuccessful. It was evident that, on cooling, the stainless steel outer sleeve had contracted around the compact-mandrel assembly, and prevented separation of the mandrel and compact.

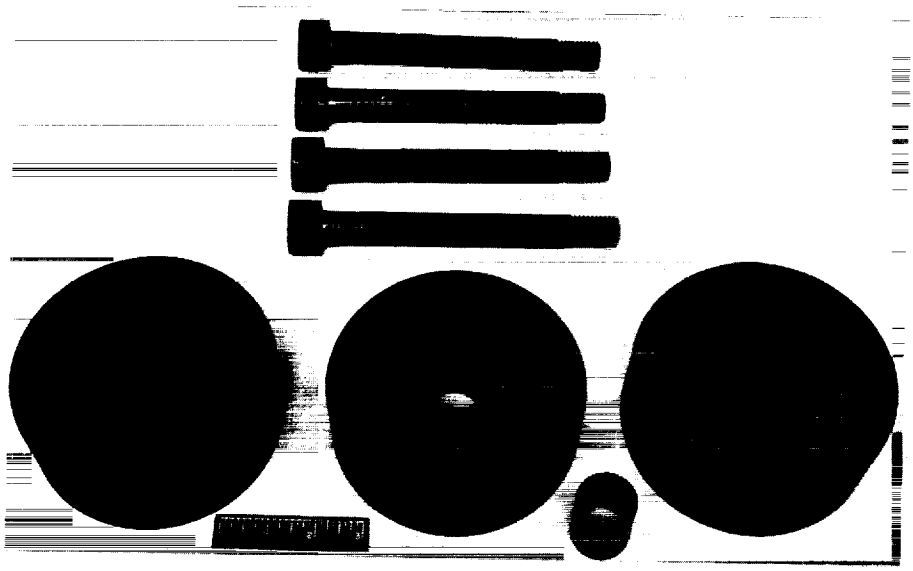
c. Group III: Low Expansion Sleeves – TZM Die

The next approach to effect a release of the mandrel and compact after warm consolidation was the use of a low-expansion sleeve material. Graphite was selected, as it was readily available and easily fabricated into thin-wall, 0.005 in. sleeves. The initial experiments in this group used only outer sleeves

of graphite, as illustrated in Figure 4b. This resulted in easy removal from the die, and allowed the mandrel to be separated from the compact. However, the problems of partial adherence of the compact to the mandrel and low green strength were still present. The relatively weak compact was usually cracked during removal of the mandrel. Inner graphite sleeves were introduced in Experiment WC-17. The thin-wall, 0.002 to 0.005 in. sleeves were slip-fit over the Type 316 stainless steel mandrel, prior to loading of the powders. All graphite parts used in each experiment were completely coated with ZrO_2 wash before tungsten powder was loaded into the annulus. A complete assembly, illustrating the use of inner and outer sleeves, is shown in Figure 4c. The inner graphite sleeve accomplished release of the compact from the mandrel. However, a new problem appeared – the introduction of a crack along the ID of the compact, which coincided with a crack along the inner graphite sleeve. The thin-wall sleeve, although flexible to some extent, cracked during warm consolidation, due to the expansion of the Type 316 stainless steel mandrel. The process parameters and results obtained for Group III experiments are given in Table I.

d. Group IV: Low Expansion Sleeves – Graphite Die

Results of Group III experiments indicated that the graphite inner sleeve had to be abandoned or replaced with another low expansion material (such as Mo) which could deform plastically during warm consolidation. As an expedient, since Mo or other suitable tubing was unavailable for immediate use on the project, an alternate solution was decided upon. Graphite was substituted for the TZM die body. This enabled powder consolidation to be carried out at sufficiently high temperatures to impart a high green strength to the compact, and thus make the removal from the die a less delicate operation. The die, a single-cavity, nonsegmented type, was made from ATJ graphite, and is shown in Figure 7. The warm consolidation temperature was raised from the usual 750 to 1300°C (1382 to 2372°F), which was initially considered to be the upper use limit for Type 316 stainless steel in this group of experiments. As a portion of the mandrel was found to have fused at 1300°C (2372°F), the temperature had to be reduced to 1100°C (2012°F) for the remaining experiments in this group. In each case, the major problem was adherence of the compact to the mandrel. As was done in the previous experiments where graphite parts were used, each part was completely coated with ZrO_2 wash, prior to use. Group IV process parameters and results are given in Table I.



2523-1811

Figure 7. Graphite Components for a Single-Cavity, Nonsegmented Powder Consolidation Die

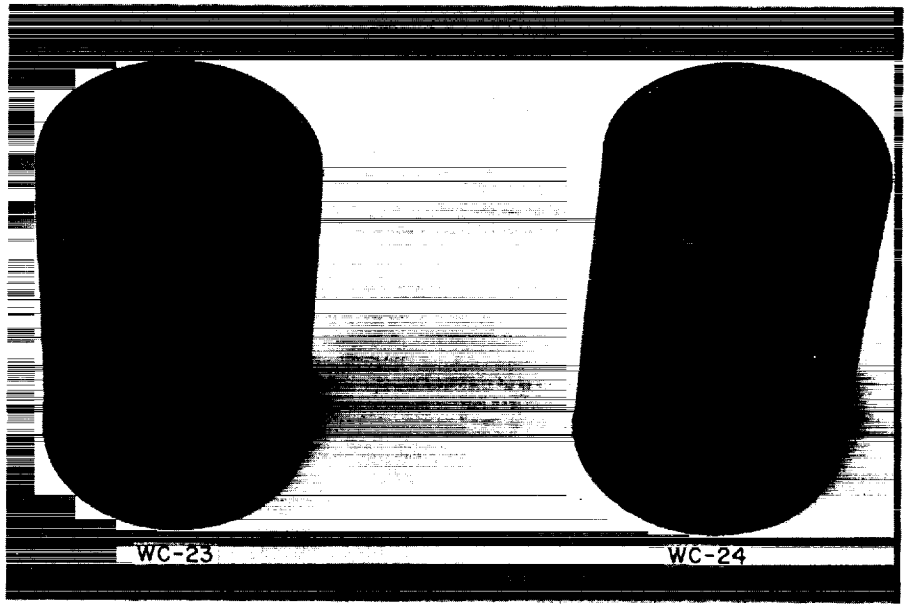
2523-1823

Figure 8. Graphite Die, with Al_2O_3 Mandrel Positioned Within the Die Cavity

e. Group V: Graphite Die – Alumina Mandrels

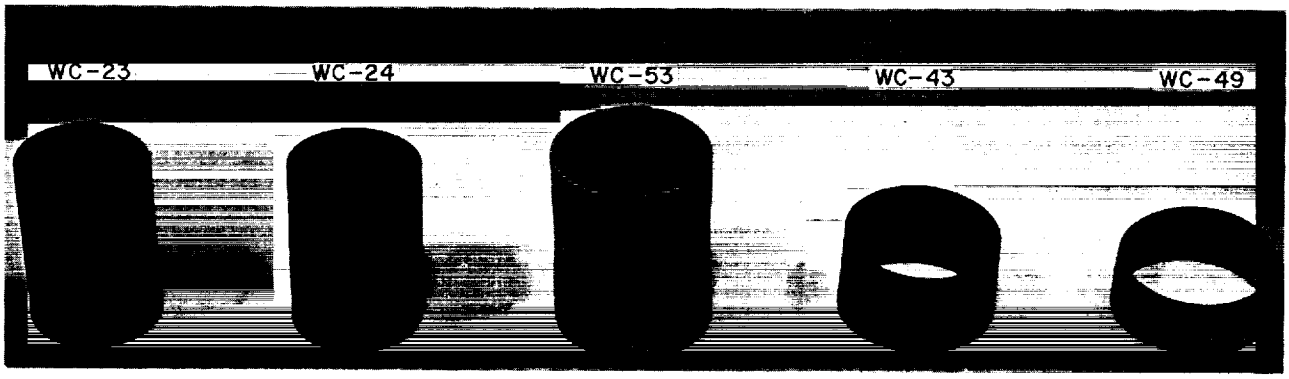
The 1100°C (2012°F) consolidation temperature limit, imposed by the use of a stainless steel mandrel, necessitated a change in materials. Al_2O_3 was selected, on the basis of its relatively high thermal expansion at elevated temperatures, high compressive strength, and thermodynamic stability. Al_2O_3 mandrels were prepared from 1-in. OD tubing and cut to 1.80 in. lengths. An assembly, consisting of an Al_2O_3 mandrel in a graphite die, is shown in Figure 8. Consolidation temperatures were eventually raised to the 1600 to 1700°C (2912 to 3092°F) range, during the initial experimentation. The first complete cylinders which were obtained from Experiments WC-23 and WC-24 appeared to be in a quasi-sintered condition after the consolidation treatment. WC-24 was given an additional H_2 sintering at 1750°C (3182°F) for 4 hr. Both cylinders are shown in Figure 9. The tungsten powder used to fabricate these cylinders was prepared by blending < 1, 1 to 10, and 30 to 60 μ particle sizes. A density determination, based on specimen geometry and weight, indicated the as-sintered density to be ~92% of theoretical. This value was considered approximate, because an accurate geometry-based volume determination was not possible, due to the wall thickness varying from 0.031 to 0.039 in. The diameter (OD) varied from 1.057 to 1.063 in.

To expedite the fabrication of cylinders for Magneform swaging experiments, an attempt was made to scale up production to six compacts per experiment, by the use of a multiple-cavity die. At the same time, the compact diameter was increased from the 1 in. nominal development size to 1.25 in., to conform with the specification requirements of the contract. The scaleup in size introduced geometrical changes in the graphite die and Al_2O_3 mandrel. Al_2O_3 mandrels, machined from 1-1/4 in. OD tubing, were found to have properties which differed from the Al_2O_3 used in earlier experiments. This necessitated a change in the powder consolidation temperature from 1650 to 1800°C (3002 to 3272°F). The process parameters for the multiple-cavity die required an additional development effort, which could not be carried out until a later date. Powder consolidation experiments were therefore continued, using the single-cavity graphite die with a 1.250 in. diameter mandrel and an annulus thickness of 0.033 in. Compacts produced in this die were complete and without cracks. However, they were found to have considerably lower density than the 1 in. compacts produced earlier in the program. Densities, as



2523-1839

Figure 9. Cylinders Obtained from Experiments WC-23 and WC-24



2523-1840

Figure 10. Representative Tungsten Cylinders Resulting from Group V Experiments (WC-23 and WC-24, nominal 1 in. diameter; WC-53, WC-43, and WC-49, nominal 1-1/4 in. diameter)

TABLE II

ESTIMATED RADIAL STRESSES DURING POWDER COMPACTION

Assembly	Temperature (°C)	Radial Stress (tsi)*
TZM Die - Stainless Steel Mandrel	750	~20
	1000	~30
Graphite Die - Al_2O_3 Mandrel	1500	~25
	1800	~33

*Based on vibratory – loaded powder bulk density estimated at ~50%

TABLE III

COMPACT DIMENSIONS AS A FUNCTION OF CONSOLIDATION AND SINTERING TREATMENTS

Compact Dimension	As Compacted (in.)	After Consolidation (in.)	After Sintering (in.)	After Consolidation and Sintering (in.)
OD	1.086	1.082	1.078	
ID	1.000	1.002	0.999	†
Length	1.800	1.800	1.79	
OD	1.335			1.298
ID	1.243	†	†	1.232
Length	1.800			1.757

†Treatment not applicable

determined by oil immersion, were initially in the range of 47 to 55% of theoretical. The low densities were found to be caused by low initial powder compaction densities, resulting from the use of powder mixtures containing only submicron particles which tended to agglomerate during vibration of the die. To eliminate this problem area, the all-submicron powder mixture was replaced with a mixture containing 1, 4 to 7, and 30 to 60 μ particles. At the same time, the powders were cold pressed in the die, using a ring-type ram at pressures up to 25 tsi. The increase in compact density and strength was very evident. Compact densities after consolidation were increased to 75% of theoretical. The process parameters and results obtained for Group V experiments are given in Table I. A representative sampling of tungsten cylinders, produced from Group V Experiments, is shown in Figure 10.

The radial stress between the die body and mandrel, introduced by virtue of differential thermal expansion, has been estimated for those experiments where tungsten powder was consolidated in (1) a TZM die having a Type 316 stainless steel mandrel, and (2) a graphite die having an Al_2O_3 mandrel. The stresses are given in Table II for two representative temperature levels.

Some typical data, showing dimension changes of powder compacts formed with graphite dies and Al_2O_3 mandrels, as a function of consolidation and/or sintering treatments, are given in Table III. The "as compacted" dimensions correspond to the OD of the mandrel and the ID of the die.

2. Cold Magneform Compaction of Tungsten Powder

Early in this program, a limited effort was made to fabricate tungsten cylinders by cold Magneform compaction of tungsten powder followed by sintering. This was necessitated by the need for a suitable tungsten workpiece in the hot Magneform swaging studies.

The tungsten powder was cold compacted between a seamless metallic retaining sleeve and a ceramic mandrel. An aluminum driver was used with an intermediate layer of Cerrobend,* which separated the metallic sleeve from the driver, and acted as a pressure transmitting medium during swaging. The

*Cerrobend is an alloy with a nominal composition of 50.0 Bi – 26.7 Pb – 13.3 Sn – 10.0 Cd, and a melting point of 70°C (158°F).

aluminum driver was subsequently separated from the tungsten (after swaging) by melting out the Cerrobend. Well-formed green tungsten compacts were obtained by this method. Seamless tantalum or mild steel tubing were used as powder retaining sleeves, in conjunction with alumina or fired Grade A lava* mandrels. Best results were obtained with the steel tube – alumina mandrel combination. After a presintering step at 1200°C (2192°F), the alumina mandrel could be separated easily from the tungsten cylinder. The steel retaining sleeve was subsequently etched away in dilute nitric acid, and the thin-walled cylinder was then sintered at ~1600°C (2912°F). All cylinders produced in this manner exhibited minor wall cracking and considerable end defects, which lead to breaking away of end segments. It is felt that these defects can be attributed to fill-density variation or expulsion of tungsten powder during the Magneforming step. Further work is required to develop this powder consolidation method.

B. HIGH TEMPERATURE MAGNEFORMING OF PREDENSIFIED CYLINDERS

Early experiments were aimed at obtaining satisfactory heating of the cylindrical tungsten workpiece by resistance heating. A temperature of 1204°C (2200°F) was considered as a probable absolute minimum for hot swaging tungsten. Heatup trials were initially made on stainless steel tubing, as a simulated workpiece. A butt-type electrical contact with the tubular workpiece proved to be unsatisfactory, because of severe arcing at the contacts and excessive heat losses at the ends of the workpiece by conduction through the heavy copper electrodes. In order to overcome this problem, a separate resistance heater was designed for satisfactory heating of the workpiece itself.

The use of a dc rectifier welding unit proved to be inadequate as a power supply for resistance heating, because of improper voltage control. This was replaced by a low voltage, 3 kva transformer (six taps, 2 to 4 v range). The heating cycle was synchronized with the Magneform swaging pulse by a common switch in conjunction with variable time-delay-relays. This was necessitated by a time lapse of about 6 sec in the Magneform operational sequence of charging and discharging its capacitor bank for swaging. The heating cycle was set for 8 sec, with the Magneform pulse following about 0.5 sec later. The circuitry

*Lava is a naturally occurring aluminum silicate.

also allowed for independent operation of either the heater or the Magneform machine. In this manner, any number of 8-sec heating cycles could be used prior to swaging.

1. Hot Swaging Through a Thermal Barrier

Experimental efforts were continued with the hot swaging assembly shown in Figure 11. Good electrical contacts with the copper electrodes were made with a tubular 0.010-in. wall steel heater, by means of spring steel retaining rings. The tungsten workpiece was contained in the annulus between the alumina mandrel and the heater. An aluminum driver tube was separated from the heater by a thermal barrier of Alundum powder which was retained at the ends by fired lava sleeves and Fiberfrax tape. The entire assembly was enclosed in a Vycor tube and evacuated with a mechanical roughing pump through the passage drilled through one of the electrodes. It was intermittently back-flushed with argon.

A number of heatup trials were conducted with this assembly, but in the absence of a tungsten workpiece. Thermocouples were attached to the outsides of both the heater and the driver, and the temperatures were recorded. Heater temperatures up to 982°C (1800°F) were obtained with an insignificant temperature rise in the aluminum driver over several 8-sec heating cycles. Substitution of up to five layers of 1-mil tantalum foil, in place of the steel heater, gave temperatures to about 1093°C (2000°F). Heat losses to the thermal barrier were considered excessive, although higher temperatures could have been obtained if higher voltages had been available with the power supply used.

Several hot swaging trials were conducted on the heater alone and in conjunction with a green tungsten powder compact as a workpiece (sintered tungsten cylinders were not available from the other phase of the project at this time). The swaging performance was generally good, but the temperature of ~1100°C (~2013°F) was too low to achieve a high quality, hot tungsten powder consolidation.

Magnesium zirconate powder was originally used as the thermal barrier material, but this was replaced by alumina powder, because of severe vacuum outgassing difficulties during the heatup trials. Alumina was an excellent mandrel material, and was not damaged by thermal shock or by the swaging operation. Both mullite and fired lava were tried out as mandrel materials, but both were

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