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AEC RESEARCH AND DEVELOPMENT REPORT

PHASE I - SUMMARY REPORT FABRICATION OF TUNGSTEN-URANIUM DIOXIDE HONEYCOMB STRUCTURES (U)

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Prepared For

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
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UNION CARBIDE

UNION CARBIDE CORPORATION
NUCLEAR DIVISION

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By

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ABSTRACT

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Tungsten-20 volume percent uranium dioxide honeycomb structures have been successfully made by two fabrication methods. One method involved stacking of individual unsintered hexagonal tubes, followed by isostatic pressing and sintering. The other method formed the unsintered honeycomb as a unit which was then sintered. The sintered structures, consisting of a close array of 1-1/2 in. long hexagonal channels, had densities ranging from 94 to greater than 97 percent of theoretical. In addition to honeycomb fabrication, two methods are described for cladding the interior honeycomb surfaces to prevent uranium dioxide loss during high temperature operation. Conf RD Author

FABRICATION OF TUNGSTEN-URANIUM DIOXIDE HONEYCOMB STRUCTURES

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FABRICATION OF TUNGSTEN-URANIUM DIOXIDE HONEYCOMB STRUCTURES

By D. E. White and E. M. Foley

Oak Ridge Gaseous Diffusion Plant
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SUMMARY

This report describes the first phase of work undertaken to determine the feasibility of producing tungsten-uranium dioxide fuel elements for use in a nuclear reactor for space vehicle propulsion. This element is a honeycomb type structure 1-1/2 inches long with a circular exterior approximately 2 inches in diameter and having 163 hexagonal-shaped, internal channels 1/8-inch across flats. For the purpose of this study, natural tungsten with a uniform fuel loading of 20 volume percent natural uranium dioxide was used.

Both tungsten and tungsten-uranium dioxide honeycomb elements with 19 internal hexagonal channels have been successfully fabricated by stacking individual hexagonal tubes which were then isostatically pressed and sintered. Densities greater than 97 percent of theoretical were obtained for four unfueled tungsten units and densities greater than 94 percent were obtained in two tungsten-uranium dioxide composites that were supplied to NASA for evaluation. The feasibility of forming the honeycombs in one step has also been partially demonstrated by forming a seven-channel unfueled tungsten element with a circular exterior. After sintering, units formed in this manner had densities greater than 97 percent of theoretical. Although the honeycomb produced by both methods were smaller than the 163 channel units ultimately desired and some difficulties in bond quality and dimensional control existed, it appears that both of these methods can be scaled up to produce units of the desired size and shape. This will be attempted in the second phase of this program.

Cladding all surfaces of the honeycomb with a thin layer of tungsten may be necessary to prevent uranium dioxide loss during high temperature operation. Either of two methods appear feasible for cladding the channel walls. Individual hexagonal tubes and 19 channel honeycomb units have been successfully gas-plated with tungsten by hydrogen reduction of tungsten hexafluoride. It also appears feasible to form thin wall, hexagonal, unfueled tungsten tubes which could be inserted inside the channels of unsintered honeycombs and isostatically pressed and sintered in place. However, the second phase of this program will rely upon the use of tungsten-coated UO_2 particles as the primary method of fuel retention.

In the course of this work, studies have shown that sintering in hydrogen reduces the UO_2 loss by a factor of 30 as compared to vacuum sintering.

Carbon analyses have indicated that by proper selection of sintering atmospheres, times, and temperatures the residual carbon in the final product is less than 50 parts per million. Addition of thorium dioxide to the fuel element may be necessary to refine the sintered grain size. Approximately 2 to 3 w/o thoria appears to be sufficient.

INTRODUCTION

In its search for a small, lightweight nuclear reactor suitable for space propulsion, the National Aeronautics and Space Administration is considering a thermal reactor design in which the fuel elements have hollow channels through which a propellent gas (hydrogen) is flowed, heated to a high temperature and exhausted at a high velocity to produce thrust. One such reactor design makes use of fuel elements containing enriched uranium dioxide dispersed in tungsten which is enriched in the W-184 isotope.

A honeycomb structure 1-1/2 inches long with 163 hexagonal shaped interior channels and a circular exterior approximately 2 inches in diameter is one design of fuel element stages that is being considered. The hexagonal channels are to be 1/8-inch across flats and in close array with a uniform web 0.020-inch thick (see figure 1). These elements are to be at least 98 percent of theoretical density with the uranium dioxide dispersed uniformly throughout the interior and there should be no exposed uranium dioxide on the surface.

The work discussed in this report was the first phase of work undertaken to determine the feasibility of producing this type of structure using natural tungsten and a uniform loading of 20 volume percent natural uranium dioxide. Although this study was limited to a uniform fuel loading, the actual reactor elements will require honeycombs with fuel loadings varying radially and perhaps circumferentially in the range of 10 to 30 volume percent. Any method developed to demonstrate the feasibility of fueled honeycomb fabrication should be adaptable to variable fuel loadings. For this reason this study was limited to two approaches. One approach was the forming of individual hexagonal tubes and joining the unsintered tubes into a honeycomb structure. This method is very attractive due to the ease of achieving variable fuel loadings. Extreme care, however, is necessary to form straight, uniform, individual, hexagonal tubes in order to ensure complete bonding of all surfaces. The other approach was to form the entire honeycomb as a unit. Although producing a variable fuel loading is perhaps more difficult with this method as compared to the stacked tube method, the problems of bonding are not as serious.

Attempts were made to eliminate exposed uranium dioxide on the surface by cladding the inside of the honeycomb channels with a 0.0015-inch thick layer of tungsten. Two methods were again considered to achieve this cladding. One method was gas plating by hydrogen reduction of tungsten hexafluoride. Another method consisted of forming thin wall

(0.003-inch) tungsten hexagonal tubes, inserting these inside the hexagonal channels of the honeycomb, and sintering the structure together.

In addition to work on honeycomb fabrication and cladding, sintering studies were also made on methods of achieving high density, methods of reducing uranium dioxide loss during sintering, and methods of reducing carbon contamination. The results of these studies are discussed separately, and the data obtained are presented in graphical and tabular form in the appendix. Photomicrographs of metallurgical cross sections and photographs of some of the configurations produced are also included in the appendix.

MATERIALS

Tungsten Powders

Three different commercially produced tungsten powders with average particle sizes ranging from 1.3 to 1.8 microns (measured by Micromerograph) were used to form these tubes. Properties of these powders are given in the appendix in tables I through III and photomicrographs of these powders with their Micromerograph particle size distributions are also included in the appendix in figures 2 through 7.

Uranium Dioxide Powder

The uranium dioxide powder (produced at ORGDP) used had a screen size of -230/+400 mesh (63 to 37 microns). The properties of this powder are summarized in table IV. Microscopic examination indicated an average particle size of about 28 microns; the Micromerograph distribution, however, indicated that this powder was approximately 9 microns in average diameter (see figure 8). As shown by the cross-sectional photomicrograph in figure 9, the particles contain some internal porosity and are stratified. This stratification may cause the particles to break up in the Micromerograph deagglomerator and thus indicate a smaller particle size.

SINTERING STUDIES

Tungsten Powder Selection

Individual hexagonal tungsten tubes were formed using the three tungsten powders described in tables I, II, and III. These tubes were first sintered for various times at temperatures ranging from 800°C. to 1200°C. in moist* hydrogen followed by 15 minutes in dry** hydrogen. The results

*Electrolytic cylinder hydrogen bubbled through water.

**Electrolytic cylinder hydrogen.

are summarized in table V. These data show that during the low temperature sintering, type A-1 tungsten powder sintered at a faster rate as is characteristic of a smaller powder. It is noted that the low temperature cycle resulted in a decrease in density during early stages of sintering as predicted from theory.

These tubes were then further sintered at various conditions ranging from 1 hour at 1600°C. in hydrogen to 1 hour at 2350°C. in vacuum. The different low temperature sintering steps (summarized in table V) affected the densities very little after high temperature sintering. All the results for a given powder have been averaged and are presented in table VI. With one exception, the results obtained on tubes made with the type A-1 tungsten were equivalent to those for tubes made with the type A-2 powder; at 1600°C in hydrogen for 1 hour, the type A-1 powder appeared to have sintered more rapidly than either of the type A-2 or A-3 powders. This posed no particular advantage for the A-1 powder. The A-2 powder did, however, consistently sinter more rapidly than the A-3 material. On the basis of these data and for reasons of availability, the A-2 tungsten powder was adapted for most of the honeycomb fabrication. Typical cross-sectional photomicrographs of tubes made from the three tungsten powders under various sintering cycles are shown in figures 10 through 12.

Residual Carbon

Hexagonal tungsten tubes made from the various tungsten powders previously described were also used for carbon analysis. The results of these tests are summarized in table VII. Some carbon is introduced during tube forming, and these data show that high temperature vacuum sintering of tubes which have been previously sintered at a low temperature does not reduce the residual carbon content to an acceptable level (< 50 ppm.). A preferable method seems to be further sintering in hydrogen at elevated temperatures. Carbon analyses on the order to 35 ppm. were also obtained on tubes with uranium dioxide dispersed in the tungsten.

Uranium Dioxide Loss

A study was made to determine the sintering conditions at which tungsten-uranium dioxide structures and/or tungsten cladding could be consolidated without loss of uranium dioxide. To accomplish this, unclad tungsten hexagonal tubes containing 20 volume percent uranium dioxide were sintered in atmospheres of hydrogen, helium, and vacuum at temperatures ranging from 1800°C. to 2350°C. and for times varying from 1 to 4 hours. These data are presented graphically in figure 13 and show that hydrogen sintering at 1800°C. for times up to 4 hours produced little, if any, uranium dioxide loss. Vacuum sintering and sintering in helium at pressures below atmospheric, however, did produce substantial losses of uranium dioxide from unclad tubes. It was also determined that weight loss on sintering (especially in the case of hydrogen sintering) was not a measure of the loss of uranium dioxide. For example, the uranium dioxide loss as calculated from weight loss of tubes sintered in hydrogen at 1800°C. for 4 hours was 7.3 percent. Chemical analysis for uranium

dioxide showed the real loss to be only 0.8 percent. One possible explanation for this difference is a reduction or volatilization of tungsten oxide.

Thoria Additions

Investigation has shown that the addition of finely divided thorium dioxide to tungsten powder results in a much finer tungsten grain size when sintered at high temperatures (1). As shown by the photomicrographs in figures 14 through 17, additions of 3 to 5 percent 2.2-micron thorium dioxide blended with tungsten or tungsten-uranium dioxide mixtures in a Waring blender were very effective in preventing formation of large grain tungsten. Density measurements were also obtained on these materials and these data are tabulated in tables VIII and IX.

Since good dispersion of thorium dioxide and uranium dioxide throughout the tungsten matrix is essential, small amounts (1% - 2%) of finely divided carbon were added to facilitate mixing in a Waring blender. Hydrogen sintering at 1880°C. for 4-1/2 hours was effective in reducing the total carbon content of the tungsten-uranium dioxide-thorium dioxide samples to 7 parts per million. Thus, it appears that ThO_2 can be satisfactorily added if the addition of thorium dioxide to actual fuel elements is necessary to control the grain size of the tungsten matrix.

FABRICATION STUDIES

Hexagonal Tubes

Hexagonal tubes were formed in the "green" state from both tungsten and tungsten-uranium dioxide materials. Since allowances were made for shrinkage during sintering, the as-formed unsintered tubes measured 0.169-inch across the inside flats and had a wall thickness of 0.016-inch. After sintering to a density of 95 percent of theoretical or greater the inside diameter (across flats) was 0.125-inch and the wall thickness 0.014-inch. Typical hexagonal tubes are shown in the photograph in figure 18 of the appendix. In figure 19 typical cross sections of unsintered tungsten and tungsten-uranium dioxide tubes are shown.

Honeycomb Fabrication

Stacked Tube Method. To determine the feasibility of joining individual tubes to form a honeycomb structure, twenty-eight unsintered hexagonal tubes made with the Type A-1 tungsten powder were joined together using a cementing technique. The tubes were cut approximately 2-1/4 inches in length, joined together, and sintered for 1 hour in hydrogen (electrolytic cylinder H_2, atmospheric pressure) at 1200°C. followed by 30 minutes in ultrapure hydrogen at 1900°C. and 1 hour in vacuum ($\sim 5 \times 10^{-6}$ Torr) at 2370°C. These tubes contained known variations in straightness, and this test pointed out some of the difficulties to be expected in forming a honeycomb by this method. As shown by the photographs in figure 20, void areas were left at the three-tube junctions even in cases where the tubes

matched very well in the unsintered state. Attempts to overcome this latter difficulty by mixing tungsten powder with the cementing material to fill the small void areas before sintering were also unsuccessful. The tungsten powder added in this manner had a much lower unsintered density than the tubes, and cracks developed at the junctions due to uneven shrinkage during sintering.

It was decided to combine isostatic pressing with the cementing techniques to bond the individual tubes completely. To test this approach, a silicone rubber pressing boot was molded to fit a 19-tube array. As shown by the photograph in figure 21, the mold is composed of hollow hexagonal fingers sealed at one end. The unsintered individual tubes were slipped on these fingers to form the hexagonal array also shown in figure 21. This assembly was then treated to soften the surfaces of the tubes and then completely sealed using the hexagonal rubber disk.

The sealed boot was isostatically pressed at pressures ranging from 72,000 to 82,000 psi. The pressure system (see figure 22) consists of an air-driven hydraulic pump that can generate pressures up to 16,000 psi. and a pressure intensifier with a ten-to-one ratio. The samples were pressed in a vessel with a chamber 2 inches in diameter and 9 inches deep. Pressures were read on precision gauges.

This isostatic pressing technique was successfully used to fabricate tungsten-uranium dioxide and pure tungsten honeycombs. Figure 23 shows two of the tungsten-uranium dioxide honeycombs fabricated from tubes made with the type A-2 tungsten powder and 20 volume percent uranium dioxide. These units were isostatically pressed at approximately 80,000 psi. and then sintered for 75 minutes at 1200°C. and for 180 minutes at 2000°C. in hydrogen. Based on a tungsten density of 19.3 g./cu.cm. and a uranium dioxide density of 10.9 g./cu.cm., these two fueled honeycombs had densities greater than 94 percent of theoretical. Although some good tube bonding was noted, there was considerable evidence of incomplete bonding at many three-tube junctions of one honeycomb (4794-32-A) in particular. This honeycomb was formed using an alternative mold design in which the hollow figures were circular instead of hexagonal, and thus good pressure distribution was not achieved at the corners.

The four unfueled, 19-channel honeycombs shown in figure 24 were formed by isostatically pressing tubes made with the type A-2 tungsten powder. Two of these units were pressed at 78,000 psi., and the other two at 82,000 psi. The four honeycombs were sintered in hydrogen at 1200°C. for 75 minutes, followed by 6-1/2 hours at 2400°C. in vacuum. Densities greater than 97 percent were obtained on all of these units. In most cases, good bonding of the tubes was attained. As with all the honeycomb samples, shrinkage occurred in a uniform manner with no apparent distortion imposed by the sintering step. Channel distortions present in the unsintered stacks were faithfully preserved through the final sintering operation.

Although the stacked tube-isostatic pressing method appears very promising for producing the honeycomb units, several variables remain to be optimized

in Phase II of this program. Some of these include determining the amount of cementing and softening material to be added and the time necessary to compact and join the tubes. Refinements in the pressing boot are also needed to eliminate channel distortion. Methods to improve bonding of the tungsten-UO_2 tubes are also required.

Entire Unit Forming Method. Another method investigated for fabricating tungsten-uranium dioxide honeycombs eliminates joining requirements with the entire unit being formed at one time. Equipment was designed to produce a sintered product 1.89 inches in outside diameter with 163 interior channels (as shown in figure 1). Sufficient tolerances were included in the designs to allow for shrinkage of the sintered structure.

Difficulties were experienced in assembly of machined parts for the equipment to form the 163-channel honeycomb as a unit and solutions to these problems were not found until late in the program period. In the short time remaining in Phase I, it was decided to concentrate the remaining effort on the stacked tube-isostatic pressing method previously described. However, equipment was assembled late in the program period to produce a simple seven-channel honeycomb of unfueled tungsten with most of the techniques to be used for the 163-channel unit. Although there was some misalignment of the forming pins, the results were very encouraging and clearly demonstrated that the shape of the forming unit is accurately reproduced in the formed piece. The photographs in figure 25 show several sintered units and a cross section of a typical three-channel junction. In this cross-sectional photomicrograph the effect of the misaligned pins is clearly evident.

Although these units were made with type A-2 tungsten powder, no appreciably greater difficulties are expected from uranium dioxide dispersed in the tungsten powder. The two units trimmed to 1-1/2 inch lengths were sintered in hydrogen for 75 minutes at 1200°C. followed by 5-1/2 hours at 2400°C. in vacuum. These units had densities of greater than 97 percent of theoretical. The unit trimmed to approximately 4-inch length was sintered in hydrogen for 75 minutes at 1200°C. followed by 2 hours at 2000°C. in hydrogen. This unit had a density of 96.2 percent of theoretical. A limited study of this process will continue in Phase II of the program.

Flat Sheet Fabrication

Flat sheets approximately 1 inch by 5 inches by 0.025 inch which contain 20 volume percent uranium dioxide dispersed in a tungsten matrix have been successfully formed using the same techniques employed in forming the hexagonal tubes. No difficulties were encountered. Six plates as shown in figure 26 were supplied to NASA for further evaluation.

TUNGSTEN CLADDING STUDIES

Gas Plating

Gas plating by hydrogen reduction of tungsten hexafluoride has been used to tungsten-clad the internal surfaces of single hexagonal tungsten-uranium dioxide tubes and isostatically bonded 19-tube assemblies. A mixture of hydrogen and tungsten hexafluoride was passed through the structures which were heated to approximately 900°F. The tungsten hexafluoride was reduced to metallic tungsten which was deposited on the heated structure surfaces. Exact conditions for the desired uniform 0.0015-inch cladding have not yet been established for either configuration; however, continuous claddings almost as smooth as the substrate were formed on the 19-tube assemblies with only minor thickness variations from tube-to-tube and with a linear thickness range from 0.0020 to 0.0028-inch over a 1.5-inch length. Over a 5-inch length on a single tube, the thickness increased from 0.002-inch at the inlet to 0.006-inch at the outlet. This linear variation in plate thickness can be controlled by varying the gas velocity and/or the temperature during the plating run. Either method requires several runs to establish empirically the correct conditions for any configuration. These claddings were applied to the substrate material prior to final sintering. Differences observed in the final densities of clad and unclad honeycombs (compare figures 23 and 27) may be a reflection of the difference in sintering time.

As shown by the photomicrographs in figures 27 through 29 good metallurgical bonds were achieved between the plate and substrate of both tubes and honeycombs by heat treating the plate at temperatures above 1800°C. in hydrogen. There was occasional evidence, however, of uneven shrinkage of plate and substrate with subsequent cracks in the plate, particularly around uranium dioxide particles. Stronger bonds and elimination of cladding discontinuities may be achieved by more complete sintering of the substrate prior to plating. This increased consolidation of the tungsten matrix may also better protect the uranium dioxide particles from possible fluorine attack during the plating operation.

Flat plates of tungsten-uranium dioxide up to 1 inch wide, 6 inches long, and 0.025-inch thick have been clad with tungsten by the gas plating technique using a fluid bed of fused alumina particles to suspend and support the plates. Smooth, even deposits ranging from 0.001- to 0.002-inch in thickness were formed over all faces and edges by a double plating procedure; e.g., reversing the pieces in the bed after about half the plate was formed. Although good metallurgical bonding between plate and substrate was obtained by subsequent heat treating (2 hours at 1980°C. in hydrogen), uneven shrinkage of plate and substrate resulted in severe warping of the piece and in some areas cracking of the cladding. The cladding was also disrputed by vaporization of uranium dioxide and/or uranium dioxide-hydrogen fluoride reaction products. A more complete consolidation of the substrate prior to plating may eliminate the warping and cracking during subsequent heat treatment.

Thin Wall Tubing

An alternative method of cladding with thin wall tungsten tubes has also been briefly investigated. In this approach unsintered, thin wall (0.003-inch) tubes would be inserted in the channels of the unsintered honeycomb and bonded together by sintering. Partially sintered (1200° to 1600°C.) thin wall tubes could also be inserted in the honeycomb channels, and the differential shrinkage during sintering used to produce bonding. In a simulated test of the latter technique, a sintered hexagonal tube was placed inside a partially sintered (1200°C.) tube, and the composite was resintered. The results of this test were encouraging in that partial bonding between the two tubes was obtained (see figures 30 and 31).

An attempt was also made to clad a tungsten-uranium dioxide hexagonal tube on both the inside and outside by use of two sizes of thin wall tungsten tubes. In this case one unsintered thin wall tungsten tube was inserted inside an unsintered tungsten-uranium dioxide tube, and this assembly was then inserted in a larger, unsintered, thin wall tungsten tube. The assembly was then sintered for 75 minutes at 1200°C. in hydrogen followed by 4 hours at 1880°C. in hydrogen. The resultant cladding was approximately 0.004-inch thick. As shown by the photomicrograph in figure 32 some bonding did occur, but the bonding was by no means complete. The use of an isostatic pressing technique similar to that used for the honeycomb fabrication, or the inclusion of the thin wall tube at the time the honeycomb itself is pressed may eliminate this problem.

Although the results obtained were encouraging, this method of cladding was not actively pursued due to the apparent early success of the gas plating method and the desire to use precoating of the UO_2 particles as the primary method of fuel retention in this type of structure.

CONCLUSIONS

From the data presented, several general conclusions can be drawn. These are:

1. With the application of isostatic pressing techniques, the stacked tube method appears to be quite feasible for honeycomb fabrication. This method is particularly attractive because of the ease of varying the uranium dioxide concentration both radially and circumferentially.

2. The direct forming method of producing a honeycomb is very promising in view of the results obtained on an experimental seven-channel unit. One advantage of this method is that it eliminates joining requirements of the stacked tube method since the entire unit is formed at one time.

3. If cladding of the honeycomb surfaces with a thin layer of tungsten is necessary to prevent loss of uranium dioxide at high temperature,

either of two methods appear feasible.

(a) Gas plating by hydrogen reduction of tungsten hexafluoride has been used to deposit a uniform tungsten coating on the interior of individual tubes and 19-channel honeycomb assemblies.

(b) Preliminary studies indicate that by using isostatic pressing techniques, thin wall tungsten tubing can be used to clad individual tubes and the channel surfaces of honeycomb assemblies.

4. A study of sintering atmospheres, times, and temperatures indicated that tungsten and tungsten-uranium dioxide tubes and honeycombs can be fabricated and sintered so that the final units contain less than 50 parts per million residual carbon.

5. Hexagonal tubes and flat sheets have been successfully formed from tungsten and tungsten-uranium dioxide dispersions by the same method.

6. Hydrogen sintering at a pressure of one atmosphere greatly reduces the uranium dioxide loss over that obtained by sintering in vacuum at the same time and temperature.

7. Investigations have shown that finely divided thorium dioxide dispersed in tungsten and tungsten-uranium dioxide composites results in a much finer tungsten grain structure when sintered at high temperatures.

ACKNOWLEDGEMENTS

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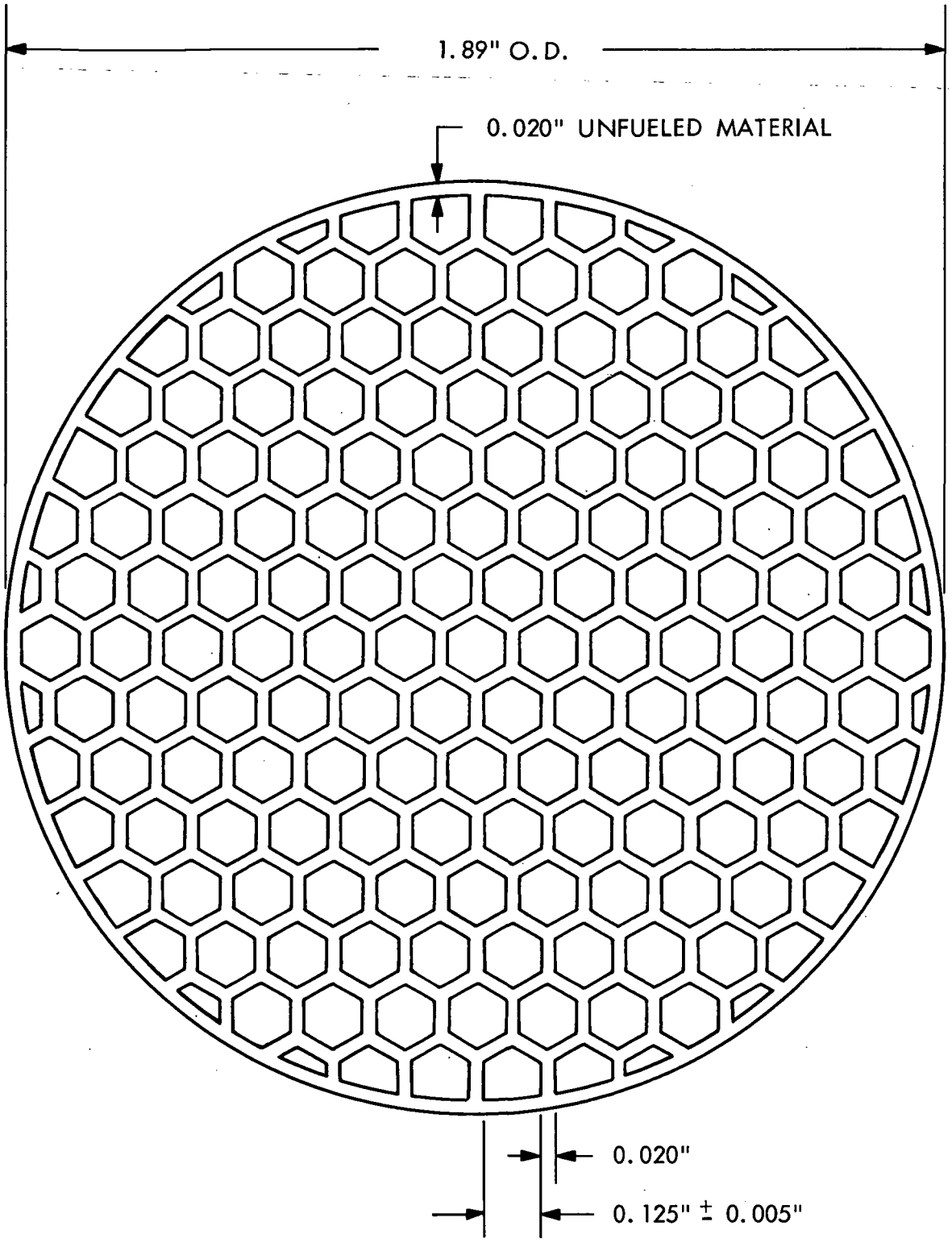
APPENDIX



HONEYCOMB FUEL ELEMENT CROSS-SECTION

Figure 1

Table I

PROPERTIES OF TYPE A-1 TUNGSTEN POWDER

IDENTIFICATION: Type A-1 Tungsten Powder ORGDP No.: 4538-58-1

SIZE:

Micromerograph (50%): 1.3 microns

Fisher Sub Sieve Size: 0.58 microns at 77.0 % porosity

X-ray Crystallite Size: > 0.1 microns (alpha tungsten)

SURFACE AREA:

By Nitrogen Adsorption: 1.68 sq. m./g.

DENSITY:

Tapped: 3.39 $g./cm.^3$

Free Flow: 2.21 $g./cm.^3$

True Density: _____ $g./cm.^3$

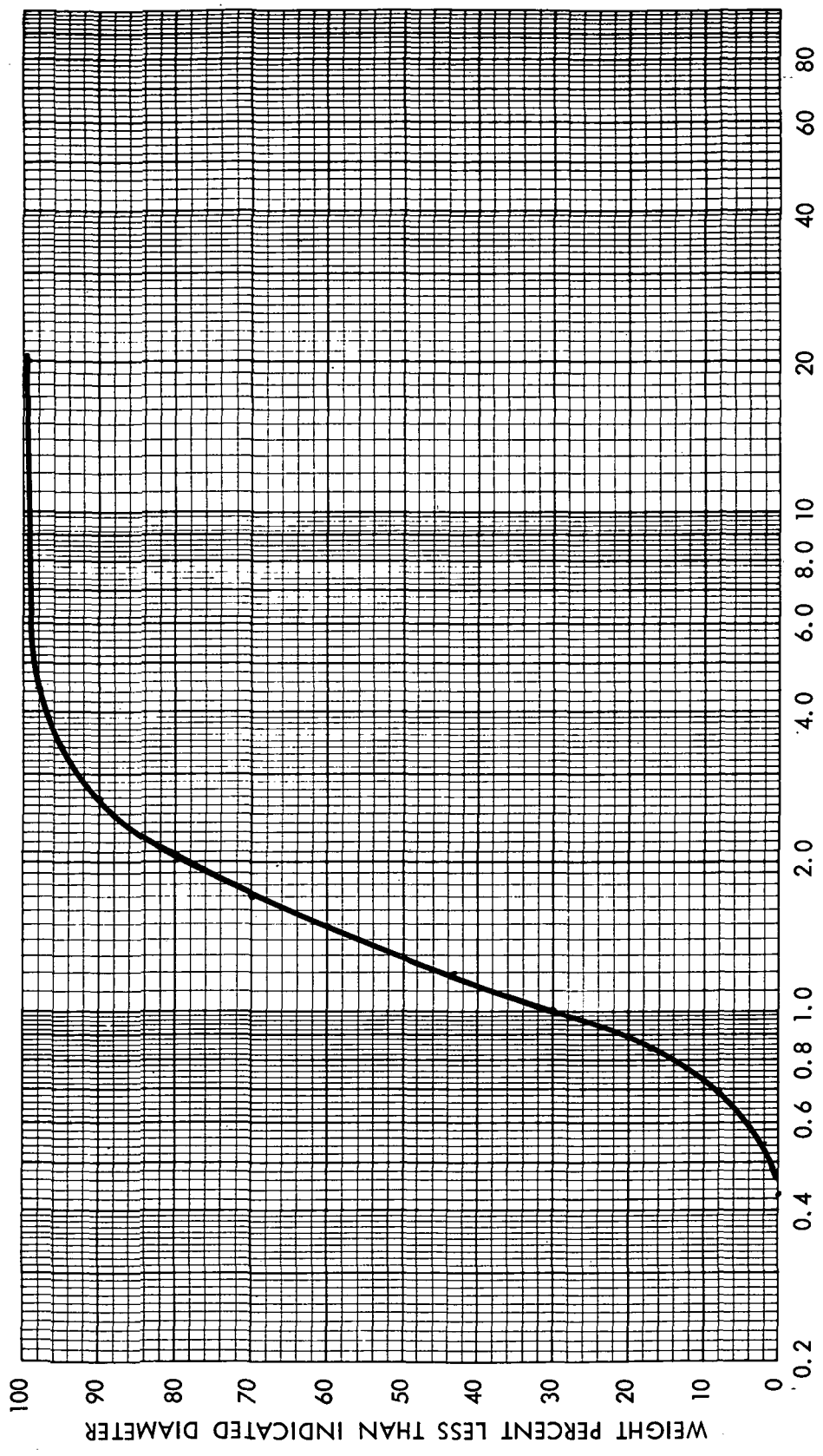
Handbook Density: 19.3 $g./cm.^3$

CHEMICAL ANALYSES:

Carbon: 82 ppm. Sulfur: 56 ppm. Fluoride: _____ %

Spectrochemical: (Parts per million)

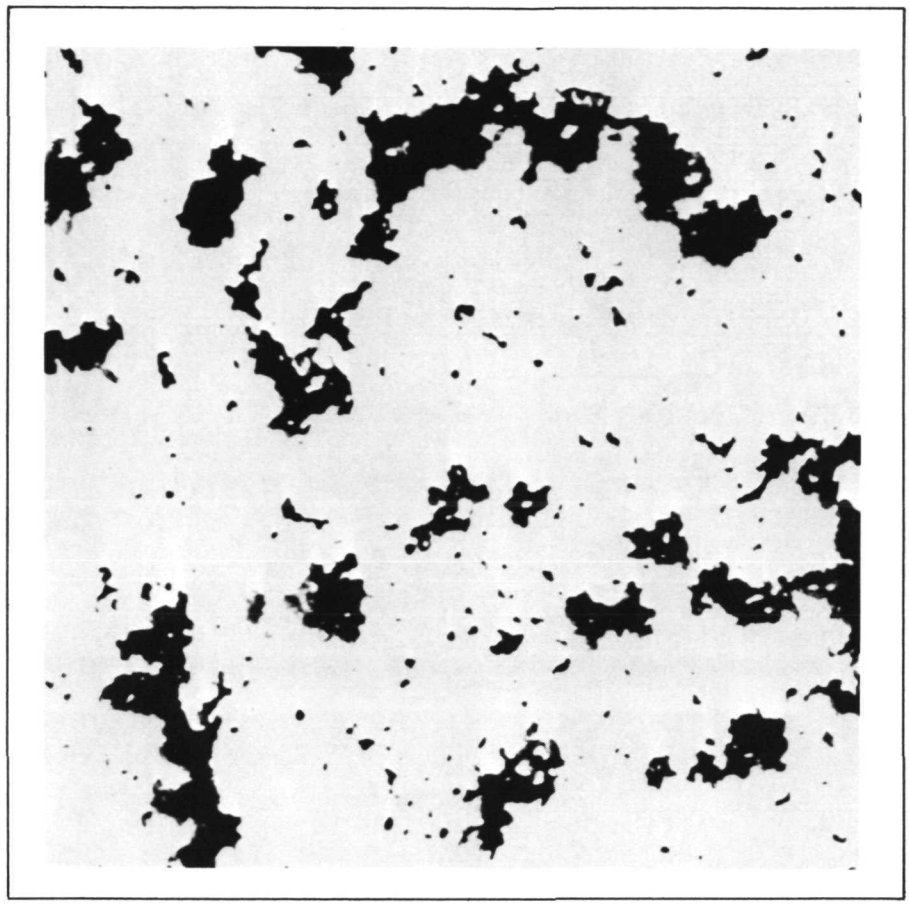
Ag	< 1	Cr	<10	Mn	<10	Si	< 3
Al	5	Cu	< 1	Mo	<30	Sn	<10
Ca	40	Fe	30	Ni	<10	V	<10
Co	<10	Mg	< 1	Pb	<100	Zr	<30



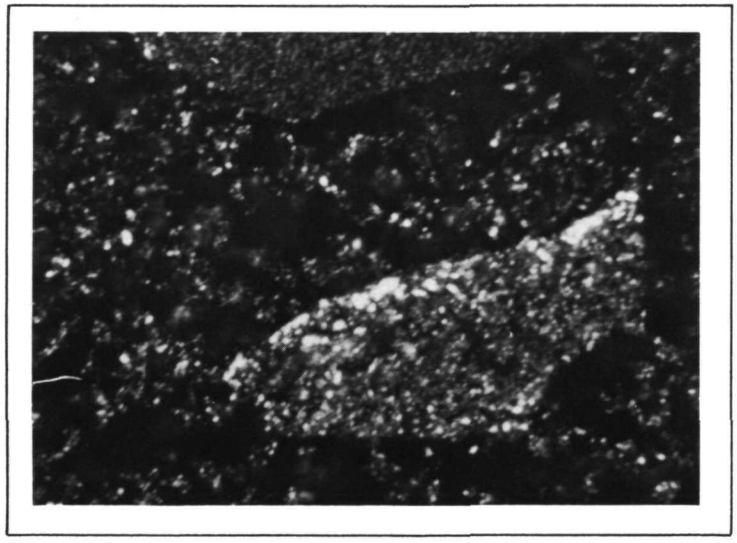
MICROMEROGRAPH PARTICLE SIZE DISTRIBUTION
OF TYPE A-1 TUNGSTEN POWDER

Figure 2

POWDER PHOTOMICROGRAPHS



Light Shadowgraphs at 500X



Mounted and Polished Cross Section at 500X

PHOTOMICROGRAPHS OF
TYPE A-1 TUNGSTEN POWDER
Figure 3

Table II

PROPERTIES OF TYPE A-2 TUNGSTEN POWDER

IDENTIFICATION: Type A-2 Tungsten Powder ORGDP No.: 4502-38

SIZE:

Micromerograph (50%): 1.5 microns

Fisher Sub Sieve Size: 0.64 microns at 76.7 % porosity

X-ray Crystallite Size: _____ microns

SURFACE AREA:

By Nitrogen Adsorption: 1.52 sq. m./g.

DENSITY:

Tapped: 3.42 $g./cm.^3$

Free Flow: 2.05 $g./cm.^3$

True Density: 19.2 $g./cm.^3$

Handbook Density: 19.3 $g./cm.^3$

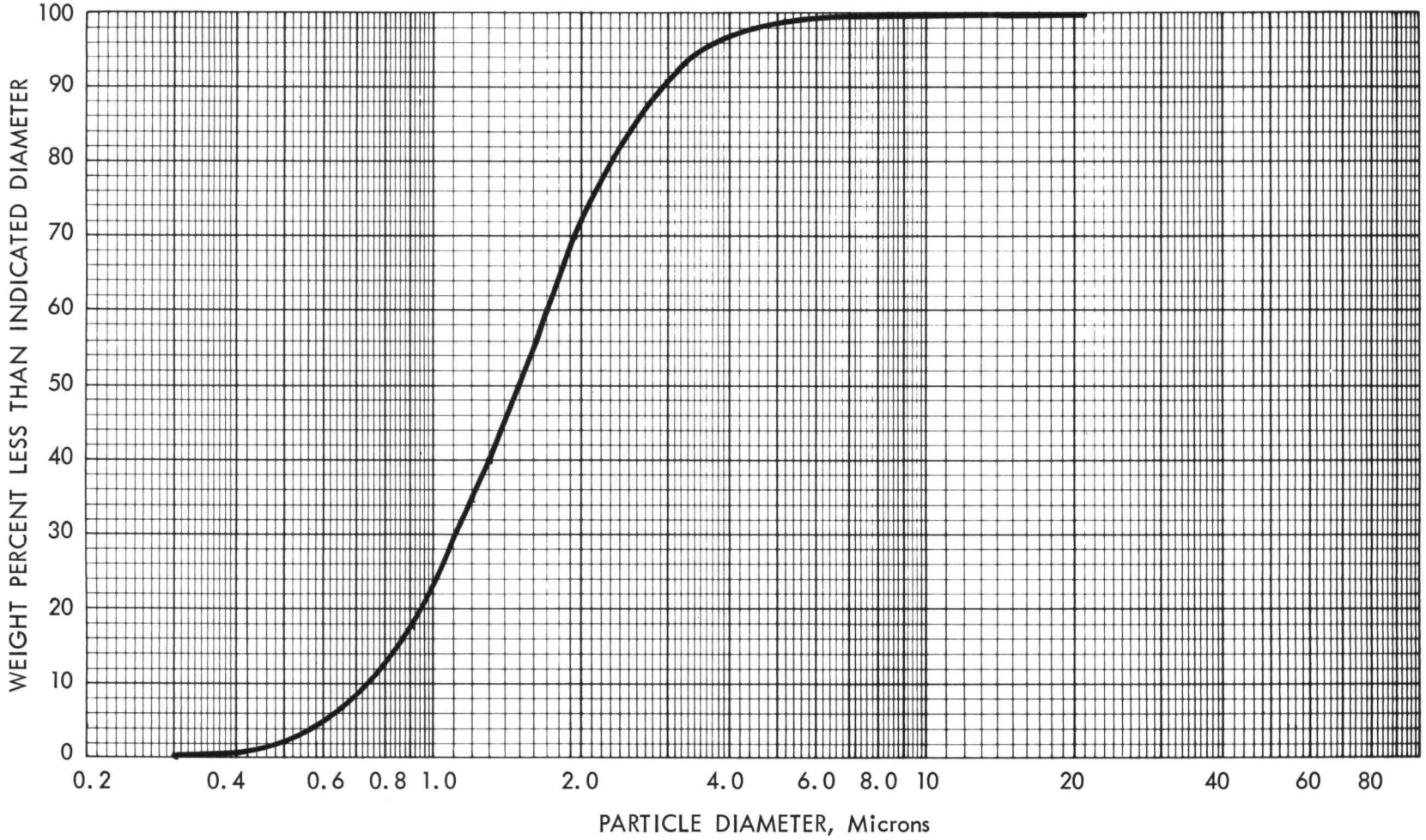
CHEMICAL ANALYSES:

Carbon: 70 ppm. Sulfur: <50 ppm. Fluoride: _____ %

Gained weight in Hydrogen.

Spectrochemical: (Parts per million)

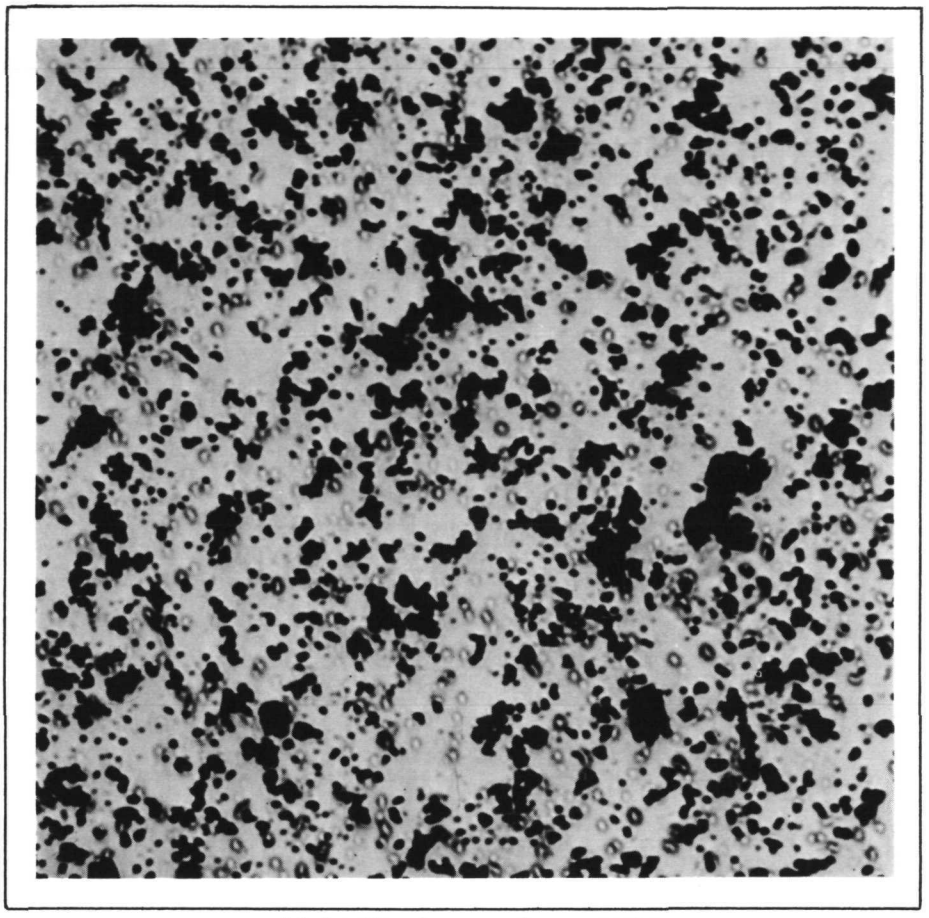
Ag		Cr	10	Mn	1	Si	10
Al	5	Cu	1	Mo	10	Sn	
Ca		Fe	100	Ni	50	V	
Co		Mg	20	Pb		Zn	



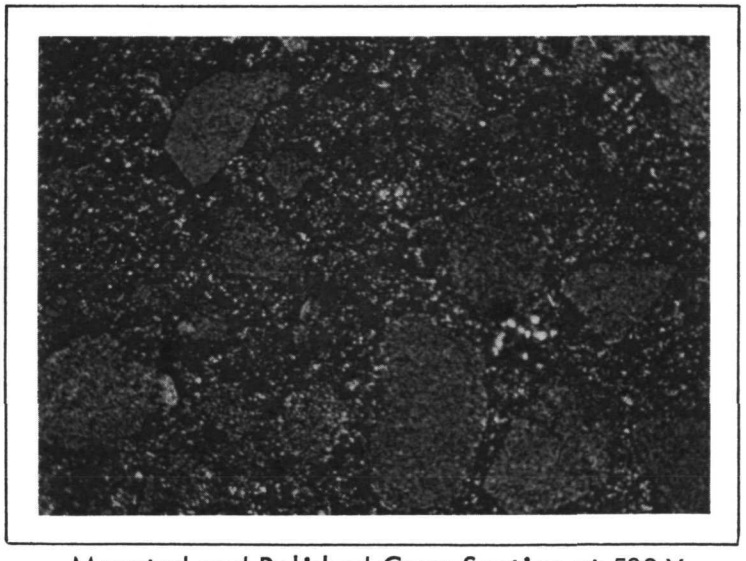
MICROMEROGRAPH PARTICLE SIZE DISTRIBUTION
OF TYPE A-2 TUNGSTEN POWDER

Figure 4

POWDER PHOTOMICROGRAPHS



Light Shadowgraphs at 500 X



Mounted and Polished Cross Section at 500 X

PHOTOMICROGRAPHS OF
TYPE A-2 TUNGSTEN POWDER

Figure 5

Table III

PROPERTIES OF TYPE A-3 TUNGSTEN POWDER

IDENTIFICATION: Type A-3 Tungsten Powder ORGDP No.: 4502-39

SIZE:

Micromerograph (50%): 1.8 microns

Fisher Sub Sieve Size: 1.26 microns at 76.8 % porosity

X-ray Crystallite Size: _____ microns

SURFACE AREA:

By Nitrogen Adsorption: 0.55 sq. m./g.

DENSITY:

Tapped: 3.81 g./cm.^{3}

Free Flow: 2.28 g./cm.^{3}

True Density: 19.2 g./cm.^{3}

Handbook Density: 19.3 g./cm.^{3}

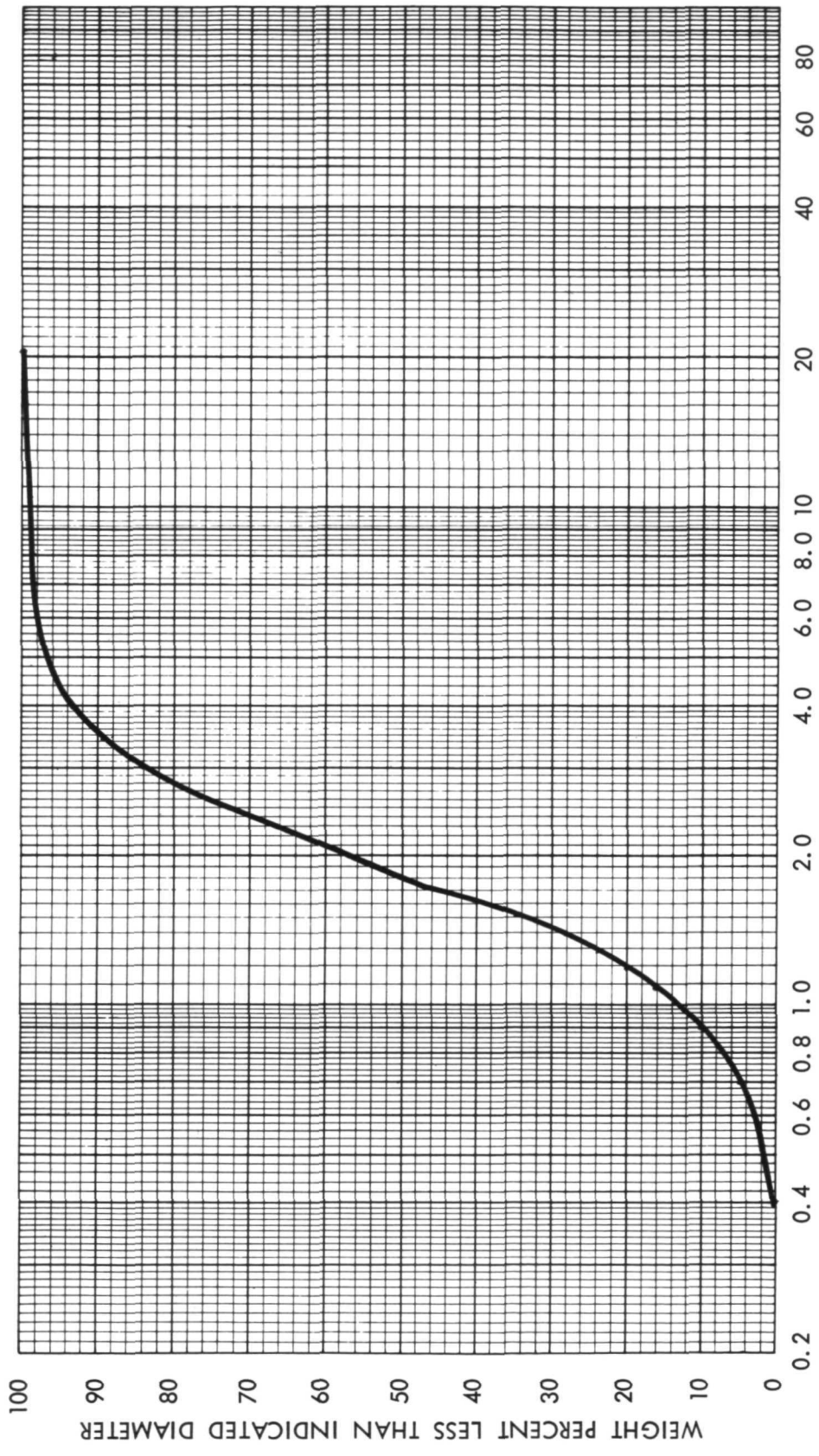
CHEMICAL ANALYSES:

Carbon 80 ppm. Sulfur: <50 ppm. Fluoride: _____ %

Gained weight in Hydrogen.

Spectrochemical: (Parts per million)

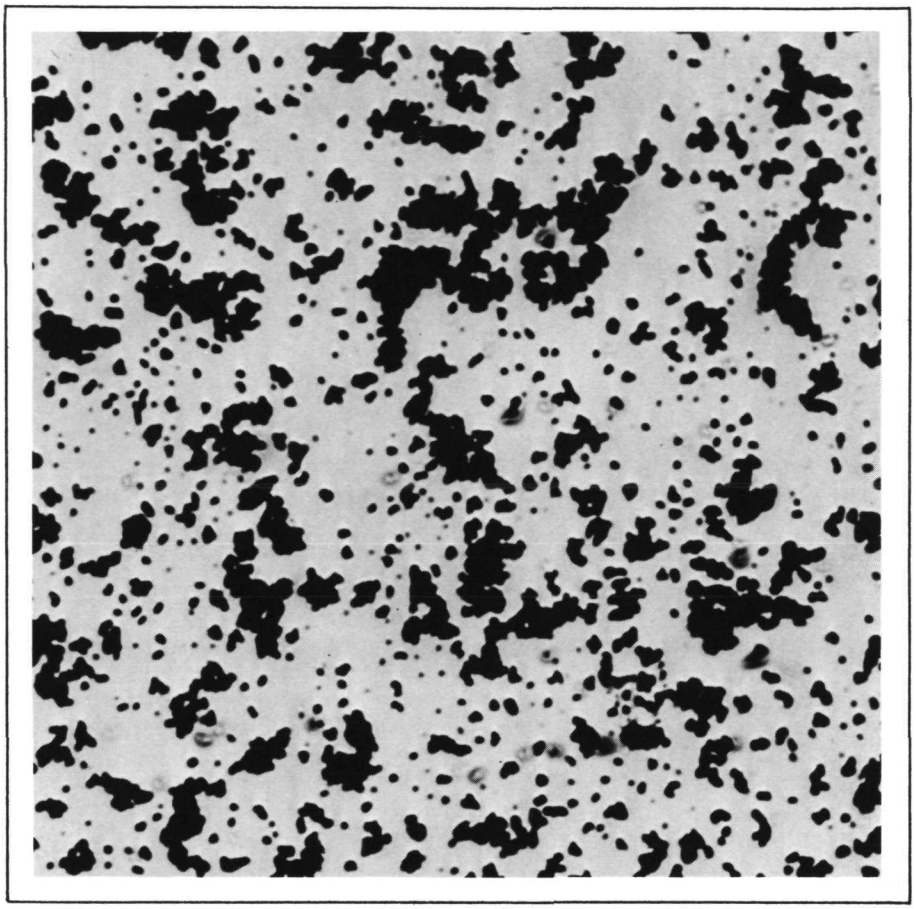
Ag		Cr	10	Mn	1	Si	1
Al	5	Cu	1	Mo	50	Sn	
Ca		Fe	100	Ni	5	V	
Co		Mg	10	Pb		Zn	



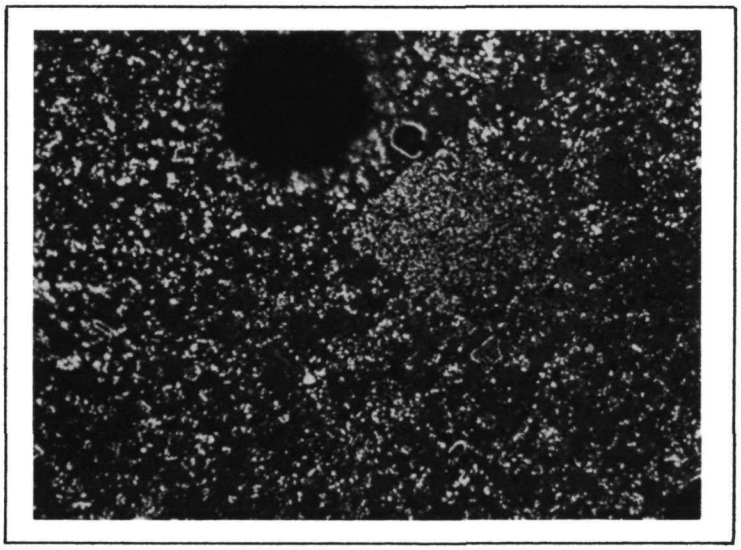
MICROMEROGRAPH PARTICLE SIZE DISTRIBUTION
OF TYPE A-3 TUNGSTEN POWDER

Figure 6

POWDER PHOTOMICROGRAPHS



Light Shadowgraphs at 500 X



Mounted and Polished Cross Section at 500 X

PHOTOMICROGRAPHS OF
TYPE A-3 TUNGSTEN POWDER
Figure 7

Table IV

PROPERTIES OF TYPE B-1 URANIUM DIOXIDE POWDER

IDENTIFICATION:, Type B-1 Uranium Dioxide Powder ORGDP NO. 4784-11

SIZE:

Micromerograph, (50%): 9.1 microns

Fisher Sub Sieve Size: - microns at - % porosity

X-ray Crystallite Size: - microns

Microscopic: 28 microns Sieve Analysis: -63/+37 microns

SURFACE AREA:

By Nitrogen Adsorption: - sq. m./g.

DENSITY:

Tapped: - $g./cm.^3$

Free Flow: - $g./cm.^3$

True Density: - $g./cm.^3$

Handbook Density: 10.9 $g./cm.^3$

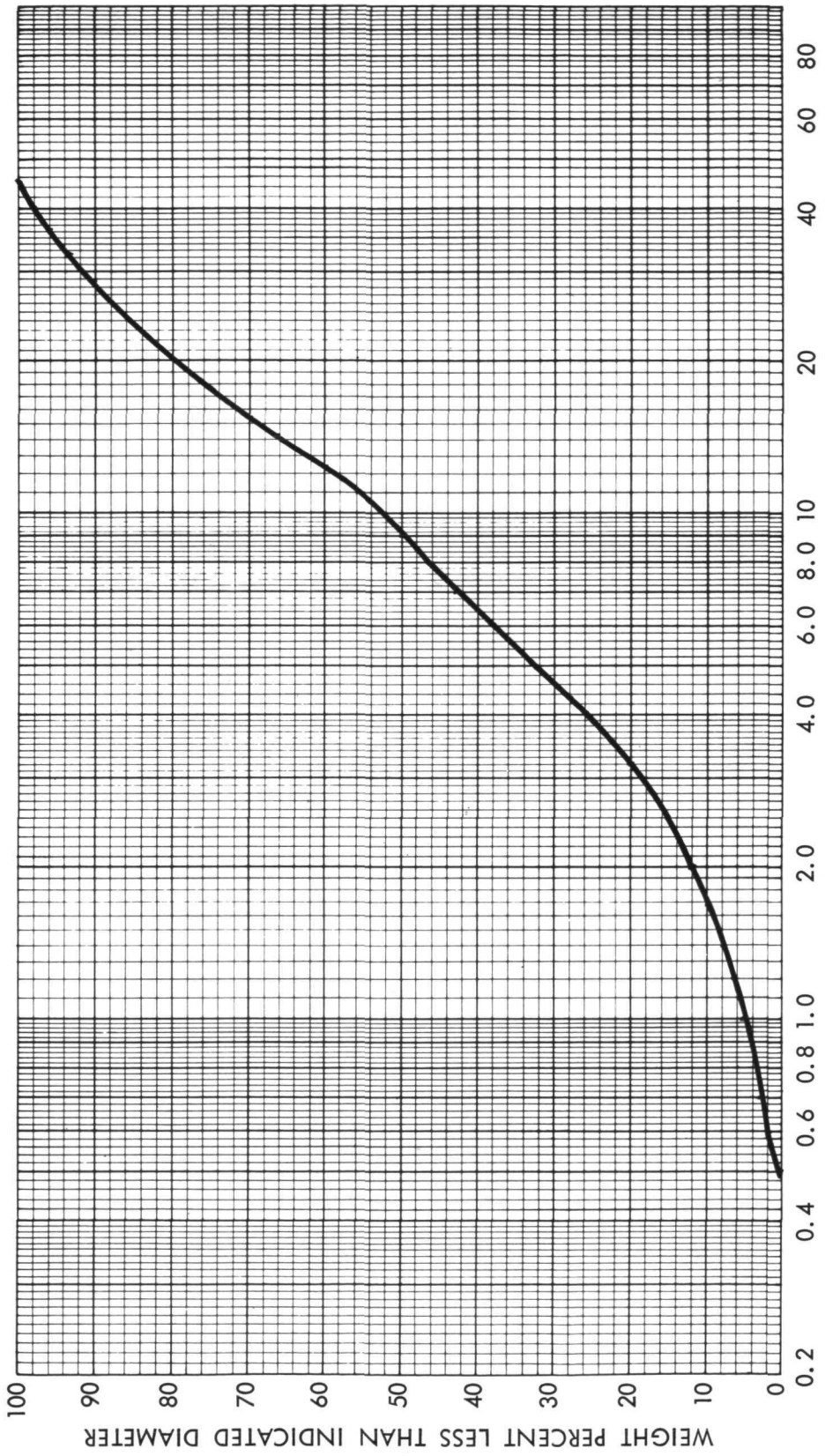
CHEMICAL ANALYSES:

Carbon: - % Sulfur: - % Fluoride: - %

Oxygen/Uranium Ratio: 2.04

Spectrochemical: (Parts per million)

Ag	-	Cr	-	Mn	-	Si	1.5
Al	-	Cu	1	Mo	-	Sn	-
Ca	< 1	Fe	20	Ni	-	V	-
Co	-	Mg	1	Pb	-	Zn	-
K	-	Li	-	Na	20	B	-
As	-	Au	-	Ba	-	Be	-
Bi	-	Cd	-	Ge	-	In	-
Tl	-						



MICROMEROGRAPH PARTICLE SIZE DISTRIBUTION
OF TYPE B-1 URANIUM DIOXIDE POWDER

Figure 8

CROSS SECTION PHOTOMICROGRAPH OF
TYPE B-1 URANIUM DIOXIDE POWDER (500X)

Figure 9

Table V

PROPERTIES OF LOW TEMPERATURE SINTERED TUNGSTEN TUBES
MADE FROM COMMERCIAL TUNGSTEN POWDERS

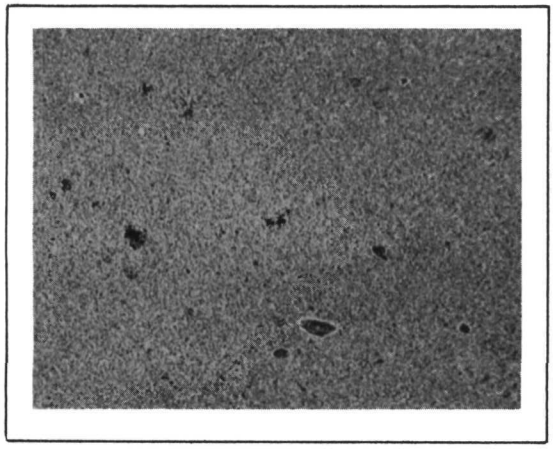
Tungsten Powder	Temp., °C.	Time, min.	Unsintered Density, %	Sintered Density, %	Change in Density
Type A-1	1000	75	44.1	45.4	1.3
Type A-1	1000	135	44.1	44.6	0.5
Type A-1	1000	195	44.1	46.8	2.7
Type A-1	1200	75	44.1	54.2	10.1
Type A-1	1200	135	44.1	56.3	12.2
Type A-1	1200	195	44.1	56.7	12.6
Type A-1	Multiple Steps*		44.1	54.3	10.2
Type A-2	1000	75	42.0	40.5	- 1.5
Type A-2	1000	135	42.0	40.9	- 1.1
Type A-2	1000	195	42.0	42.0	0.0
Type A-2	1200	75	42.0	47.1	5.1
Type A-2	1200	135	42.0	49.1	7.1
Type A-2	1200	195	42.0	49.4	7.4
Type A-2	Multiple Steps*		42.0	46.3	4.3
Type A-3	1000	75	42.0	41.6	- 0.4
Type A-3	1000	135	42.0	41.4	- 0.6
Type A-3	1000	195	42.0	40.8	- 1.2
Type A-3	1200	75	42.0	43.3	1.3
Type A-3	1200	135	42.0	43.1	1.1
Type A-3	1200	195	42.0	43.2	1.2
Type A-3	Multiple Steps*		42.0	42.7	0.7

*2 hours at 800°C., plus 1 hour at 1000°C., plus 1 hour at 1200°C. in moist hydrogen followed by 15 minutes at 1200°C. in dry hydrogen.

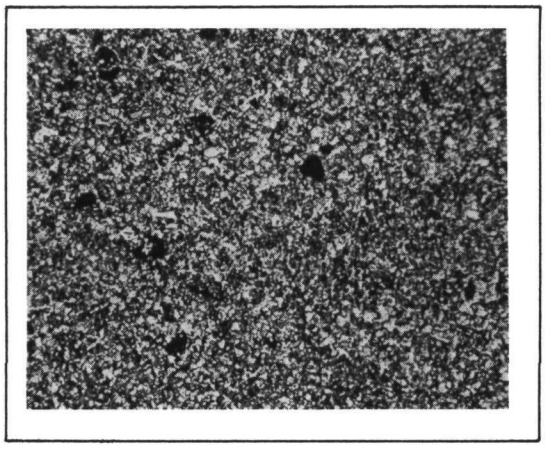
Table VI

DENSITIES OF SINTERED TUNGSTEN TUBES

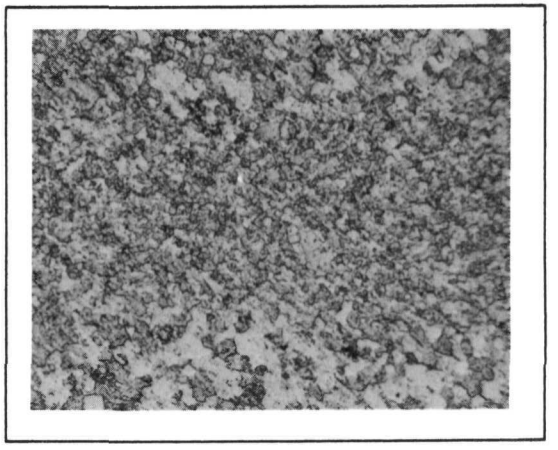
Tungsten Powder	1 hour 1600°C. Hydrogen	1 hour 1800°C. Hydrogen	1 hour 2000°C. Vacuum	1 hour 2350°C. Vacuum
Type A-1	85.8%	89.5%	91.8%	94.3%
Type A-2	79.5	89.3	92.2	94.5
Type A-3	77.3	79.6	87.7	93.1



1200°C. - 75 min. - H_2

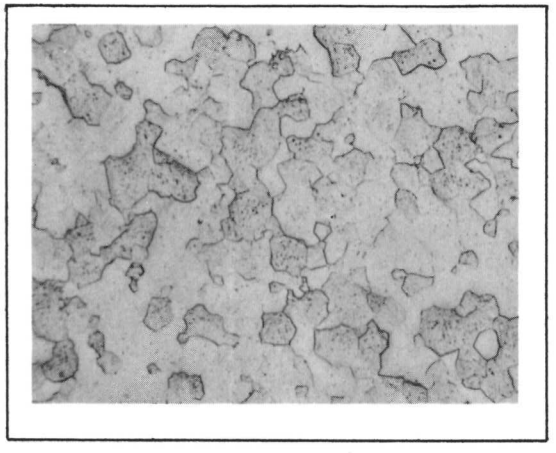


1200°C. - 75 min. - H_2
1600°C. - 60 min. - H_2

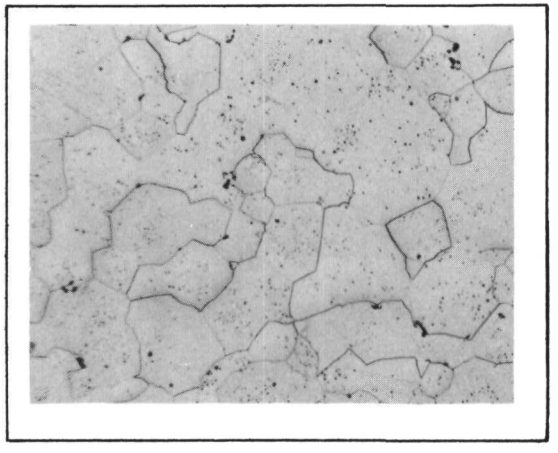


1200°C. - 75 min. - H_2
1800°C. - 60 min. - H_2

Magnification - 200X



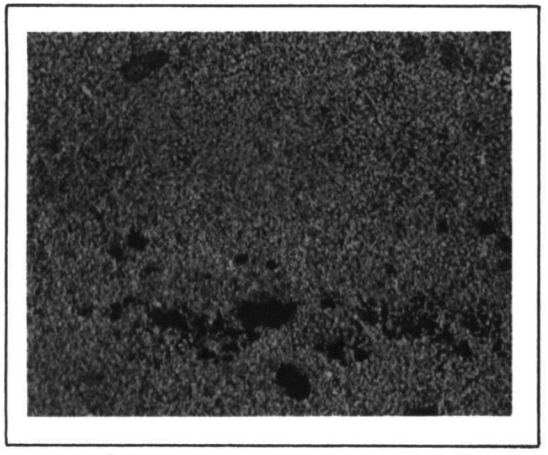
1200°C. - 75 min. - H_2
2000°C. - 60 min. - Vac.



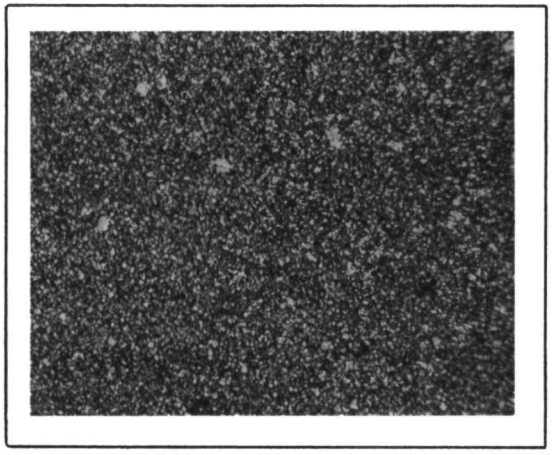
1200°C. - 75 min. - H_2
2350°C. - 60 min. - Vac.

CROSS-SECTION PHOTOMICROGRAPHS OF HEXAGONAL TUBES
MADE WITH TYPE A-1 TUNGSTEN POWDER

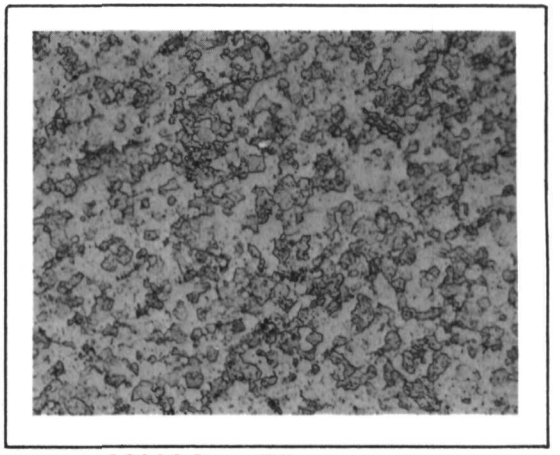
Figure 10



1200°C. - 75 min. - H_2

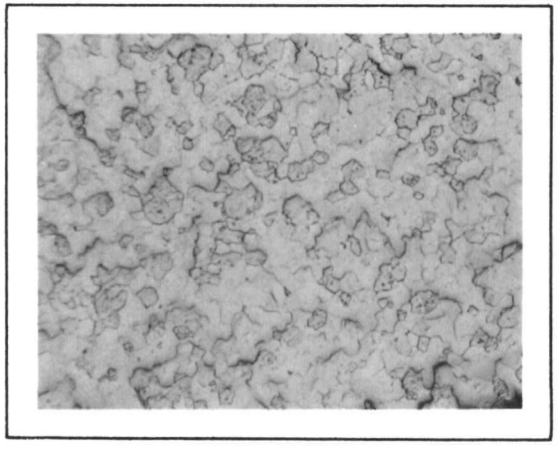


1200°C. - 75 min. - H_2
1600°C. - 60 min. - H_2

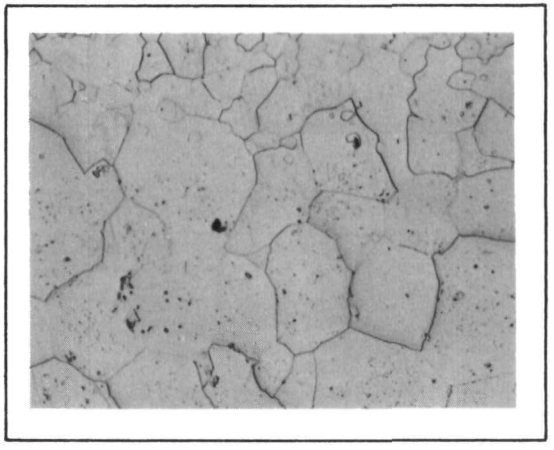


1200°C. - 75 min. - H_2
1800°C. - 60 min. - H_2

Magnification - 200X



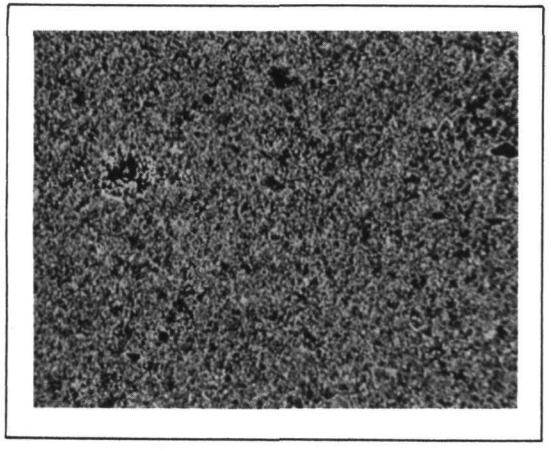
1200°C. - 75 min. - H_2
2000°C. - 60 min. - Vac.



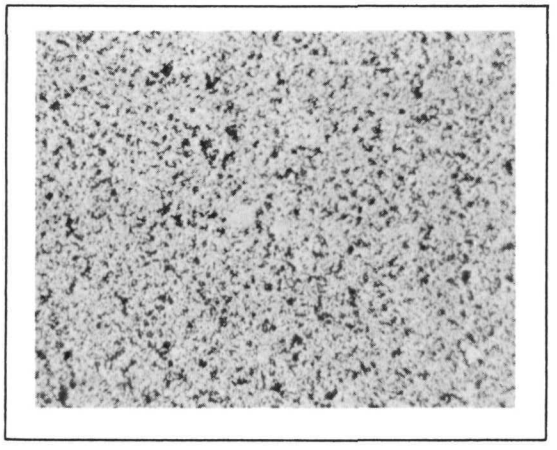
1200°C. - 75 min. - H_2
2350°C. - 60 min. - Vac.

CROSS-SECTION PHOTOMICROGRAPHS OF HEXAGONAL TUBES MADE WITH TYPE A-2 TUNGSTEN POWDER

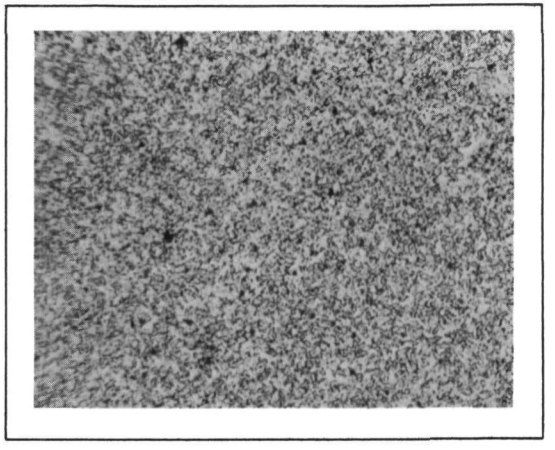
Figure 11



1200°C. - 75 min. - H_2



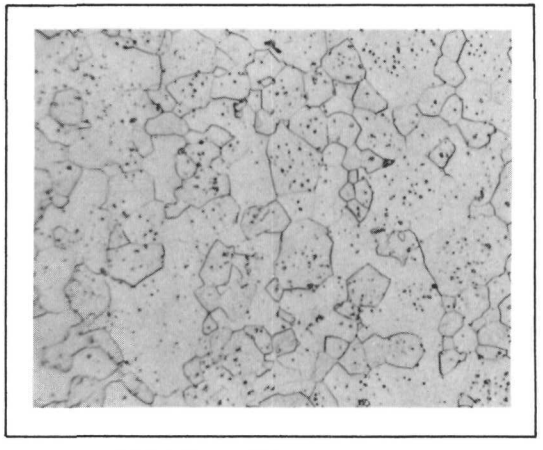
1200°C. - 75 min. - H_2
1600°C. - 60 min. - H_2



1200°C. - 75 min. - H_2
1800°C. - 60 min. - H_2

Magnification - 200 X

1200°C. - 75 min. - H_2
2000°C. - 60 min. - Vac.



1200°C. - 75 min. - H_2
2350°C. - 60 min. - Vac.

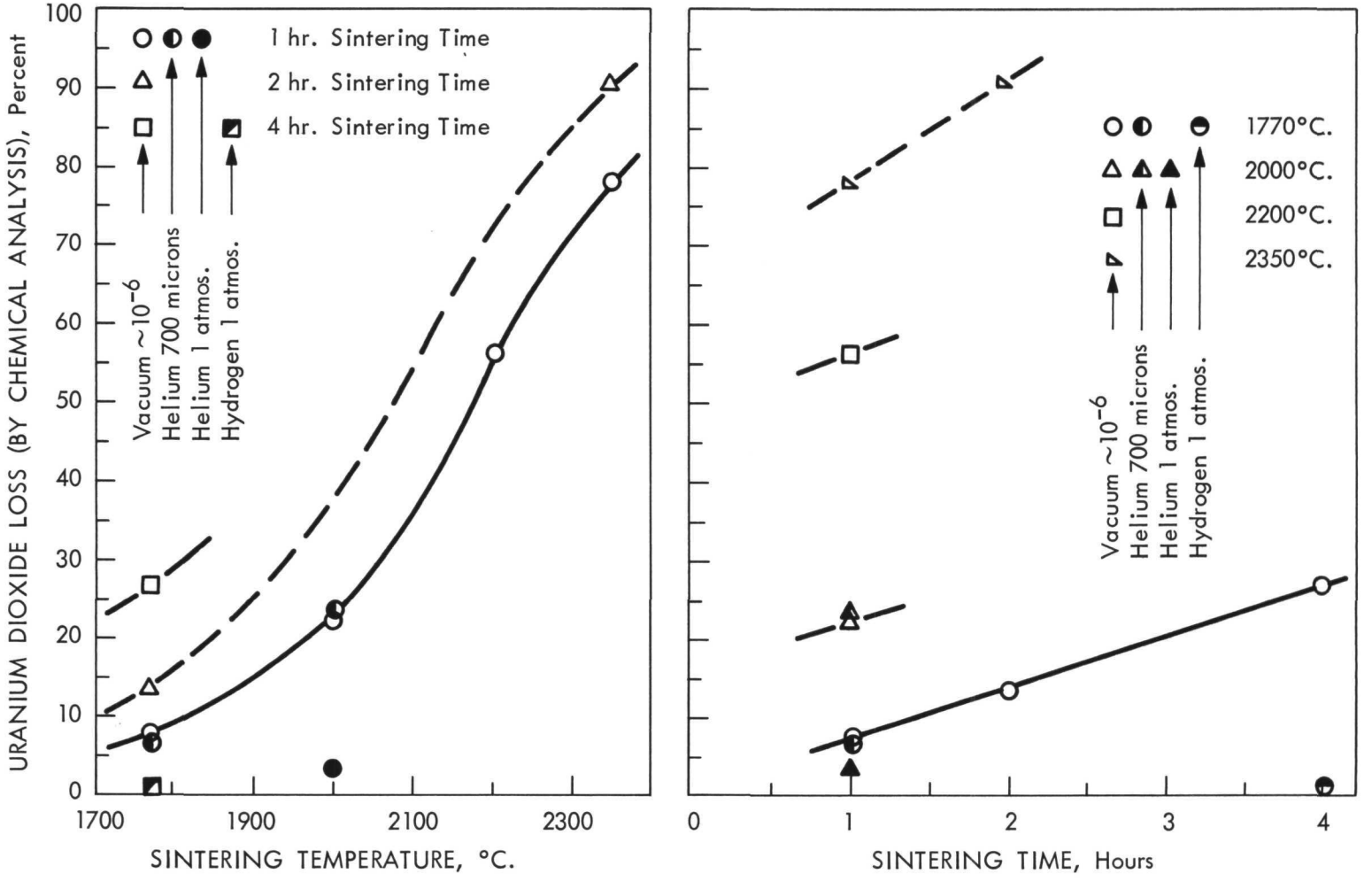
CROSS-SECTION PHOTOMICROGRAPHS OF HEXAGONAL TUBES MADE WITH TYPE A-3 TUNGSTEN POWDER

Figure 12

Table VII

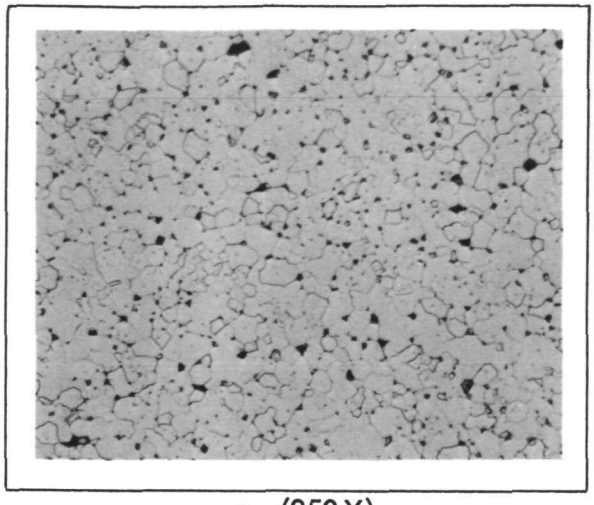
CARBON ANALYSES OF SINTERED TUNGSTEN TUBES

Tungsten Powder	Powder Carbon Content, ppm.	Carbon Content, ppm. After Initial Sintering 1200°C., 195 min., H_2	Carbon Content, ppm. After Further Sintering, 60 min. 1600°C. - H_2	2350°C. - Vac.
Type A-1	82	150	18	400
Type A-2	70	700	21	613
Type A-3	80	22	31	122

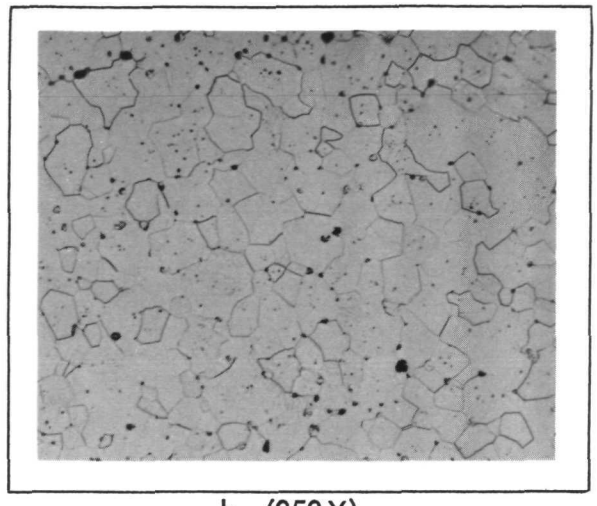


URANIUM DIOXIDE LOSS AS A FUNCTION OF
SINTERING TEMPERATURE, TIME, AND ATMOSPHERE

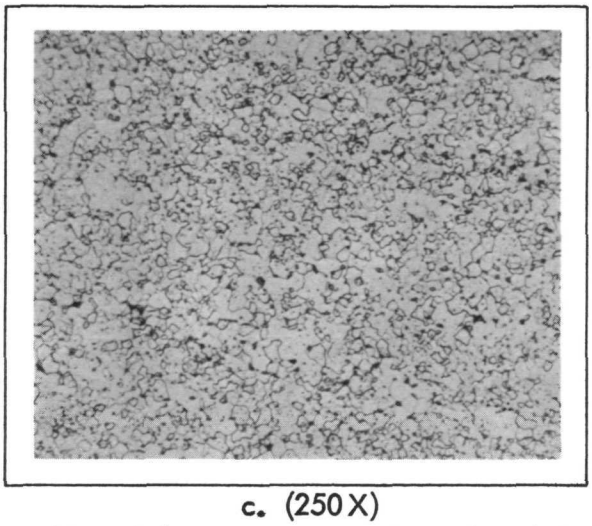
Figure 13



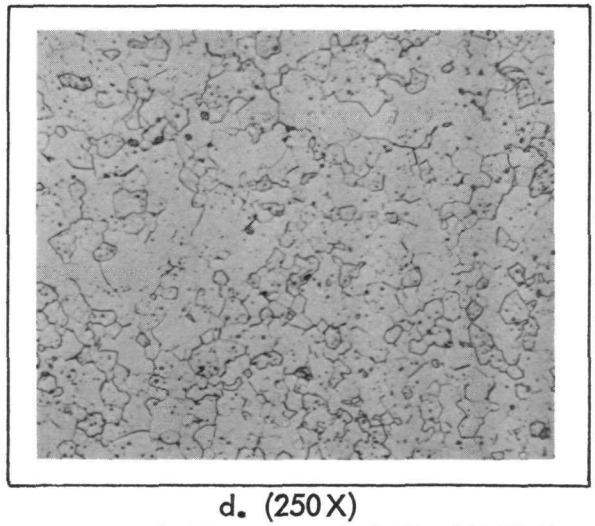
a. (250X)
Material: 100% Type A-1 Tungsten Powder
1800°C. - 60 min. - H_2
Density: 91.3%



b. (250X)
Material: 100% Type A-1 Tungsten Powder
1800°C. - 60 min. - H_2
2300°C. - 60 min. - Vac.
Density: 94.1%



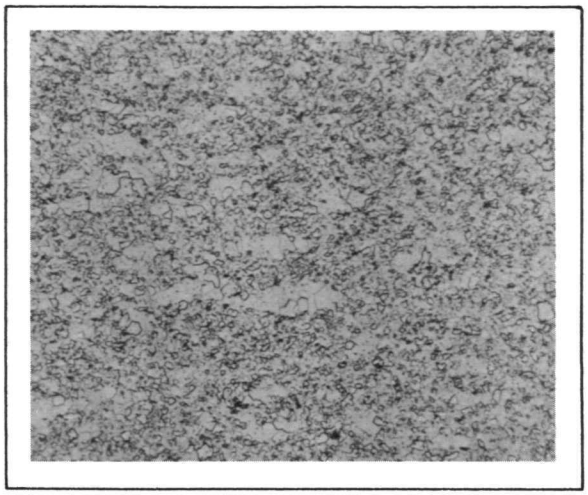
c. (250X)
Material: 99% Type A-1 W - 1% ThO_2
1800°C. - 60 min. - H_2
Density: 93.7%



d. (250X)
Material: 99% Type A-1 W - 1% ThO_2
1800°C. - 60 min. - H_2
2300°C. - 60 min. - Vac.
Density: 95.3%

EFFECT OF THORIA ON SINTERING OF TUNGSTEN

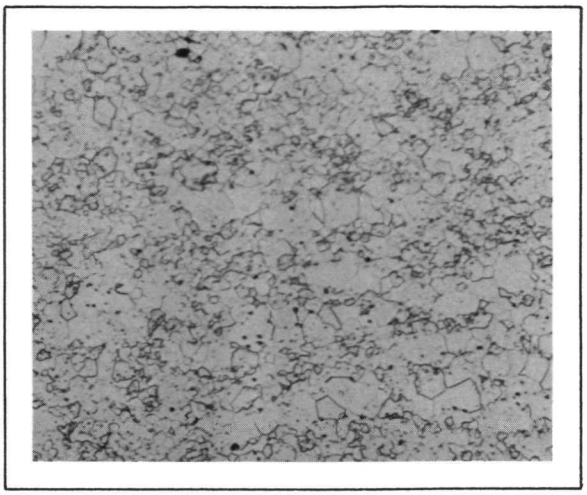
Figure 14



a. (250X)

Material: 97% Type A-1 W - 3% ThO_2
1800°C. - 60 min. - H_2

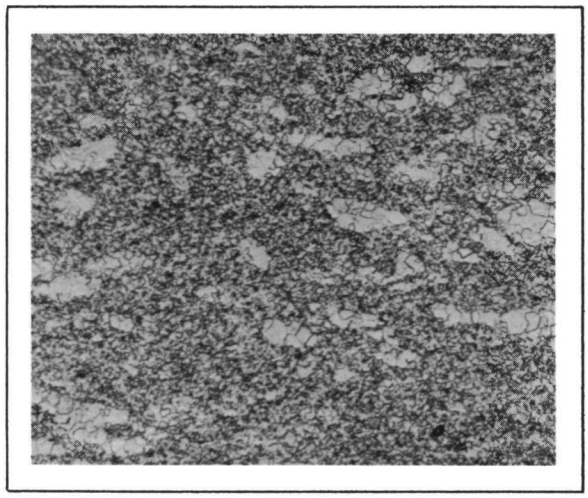
Density: 92.7%



b. (250X)

Material: 97% Type A-1 W - 3% ThO_2
1800°C. - 60 min. - H_2
2300°C. - 60 min. - Vac.

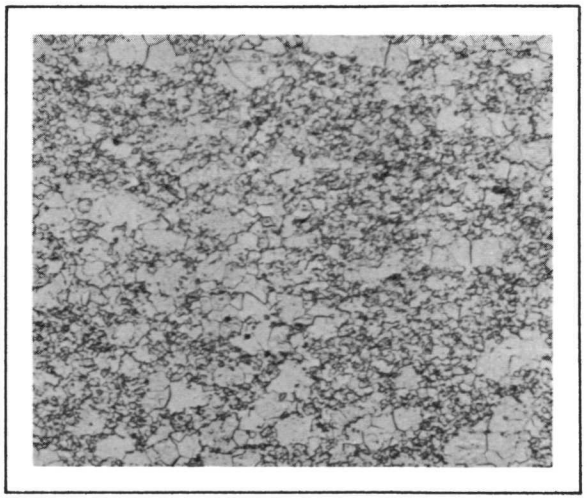
Density: 95.4%



c. (250X)

Material: 95% Type A-1 W - 5% ThO_2
1800°C. - 60 min. - H_2

Density: 91.8%



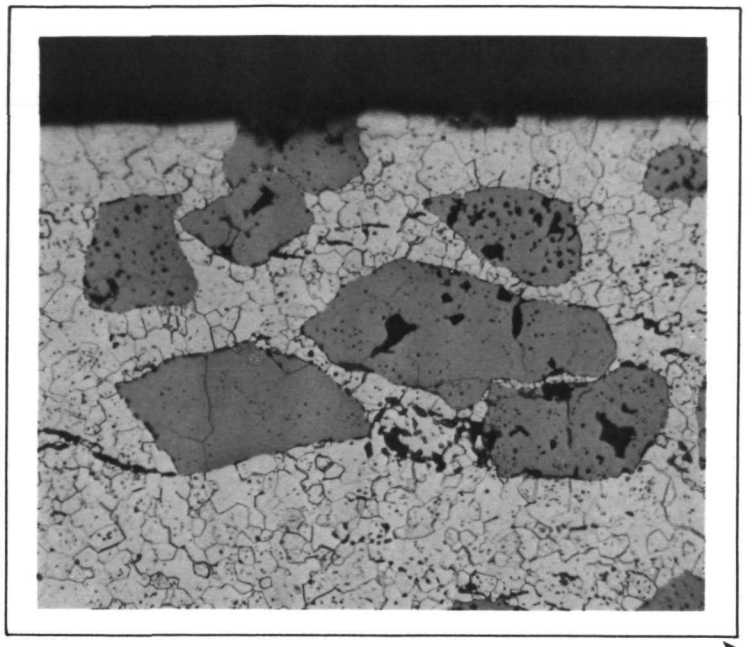
d. (250X)

Material: 95% Type A-1 W - 5% ThO_2
1800°C. - 60 min. - H_2
2300°C. - 60 min. - Vac.

Density: 96.8%

EFFECT OF THORIA ON SINTERING OF TUNGSTEN

Figure 15



a. (250 X)

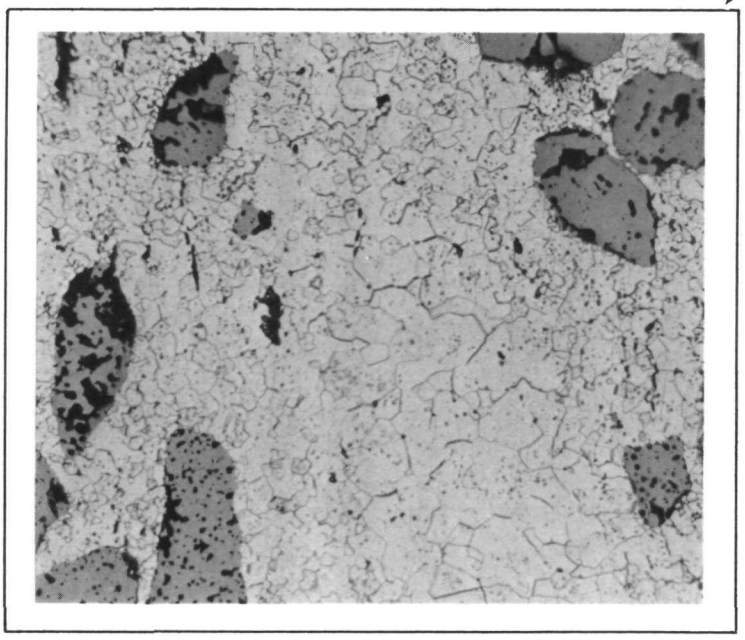
Material:

88.0 w/o Type A-1
Tungsten Powder
12.0 w/o Type B-1 Uranium
Dioxide Powder

Density: 89.0%

Sintering:

1880°C. - 1 hr. - H_2
1995°C. - 3-1/2 hr. - He



b. (250 X)

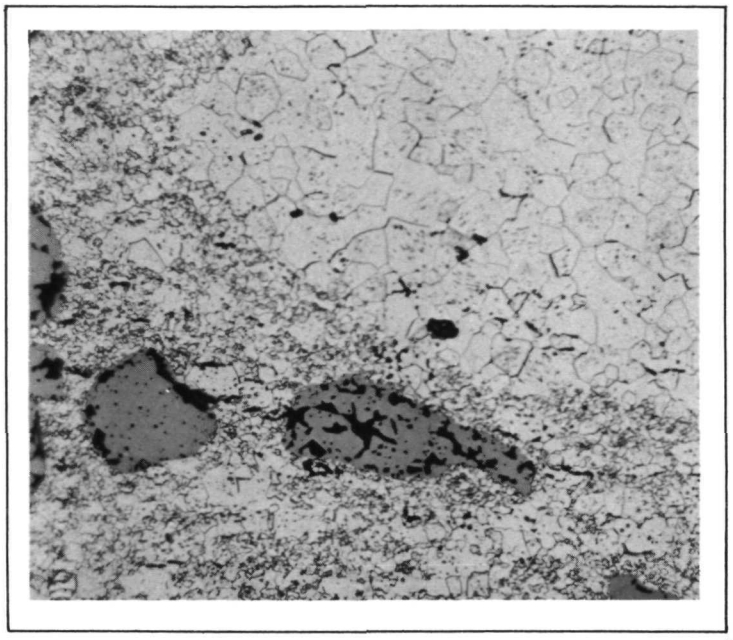
Material:

87.0 w/o Type A-1
Tungsten Powder
12.0 w/o Type B-1 Uranium
Dioxide Powder
1.0 w/o 2.2-micron ThO_2

Density: 88.3%

EFFECT OF THORIA ADDITIONS ON
SINTERING OF TUNGSTEN-URANIUM DIOXIDE

Figure 16



a. (250X)

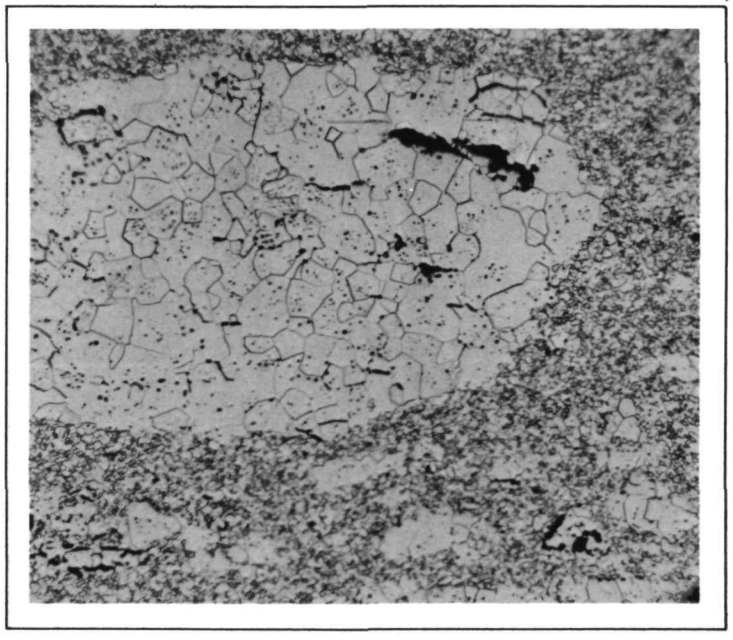
Material:

85.5 w/o Type A-1 Tungsten Powder
12.0 w/o Type B-1 Uranium Dioxide Powder
2.5 w/o 2.2-micron ThO_2

Density: 88.9%

Sintering:

1880°C. - 1 hr. - H_2
1995°C. - 3-1/2 hr. - He



b. (250X)

Material:

84.0 w/o Type A-1 Tungsten Powder
12.0 w/o Type B-1 Uranium Dioxide Powder
4.0 w/o 2.2-micron ThO_2

Density: 87.0%

EFFECT OF THORIA ADDITIONS ON
SINTERING OF TUNGSTEN-URANIUM DIOXIDE

Figure 17

Table VIII

PROPERTIES OF SINTERED COMPACTS MADE FROM TUNGSTEN-THORIA POWDER MIXTURES

Thoria Addition, %	Mixture Theoretical Density, g./cu. cm.	Sintering Conditions			Density, %		
		Temperature, °C.	Time, min.	Atmosphere	Physical Dimensions	Mercury Displacement	Inert Gas Displacement
0	19.3	1800	60	H_2	91.3	91.3	91.6
		1800	60	H_2	95.0	94.1	95.4
		2300	60	Vacuum			
0.2	19.3	1800	60	H_2	90.8	91.7	93.6
		1800	60	H_2	95.0	93.8	96.9
		2300	60	Vacuum			
0.5	19.2	1800	60	H_2	91.1	91.7	95.3
		1800	60	H_2	97.5	95.3	95.9
		2300	60	Vacuum			
1.0	19.1	1800	60	H_2	93.0	93.7	94.8
		1800	60	H_2	98.0	95.3	96.8
		2300	60	Vacuum			
3.0	18.8	1800	60	H_2	91.6	92.7	93.9
		1800	60	H_2	97.7	95.4	99.2
		2300	60	Vacuum			
5.0	18.4	1800	60	H_2	93.7	91.8	93.4
		1800	60	H_2	98.4	96.8	98.0
		2300	60	Vacuum			

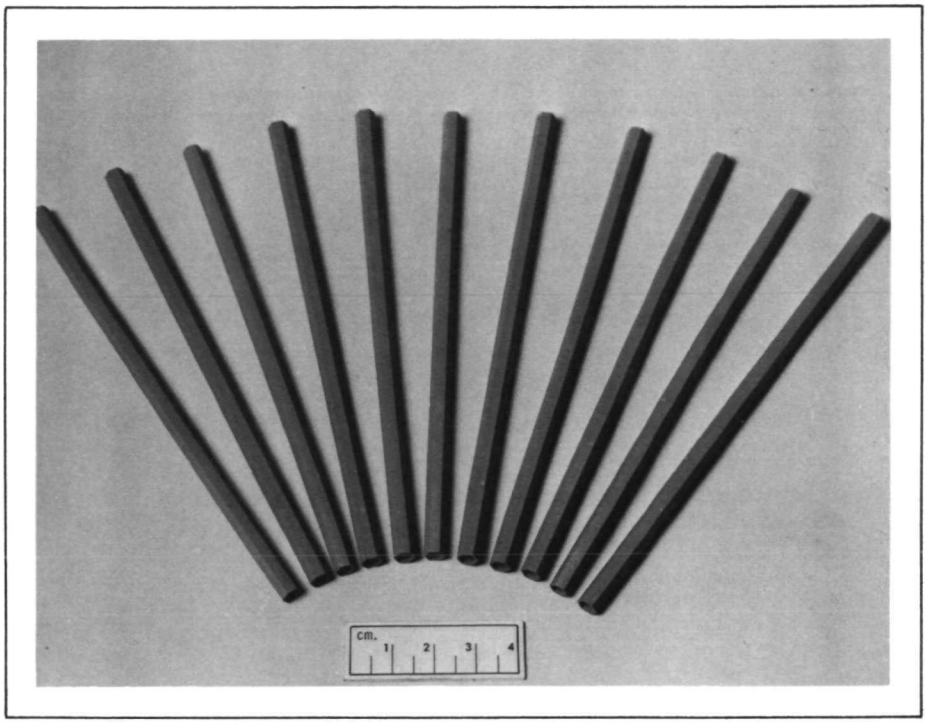
*Compacted at 85,500 psi.

Table IX

DENSITIES OF SINTERED COMPACTS MADE FROM
TUNGSTEN-THORIA-URANIUM DIOXIDE POWDER MIXTURES

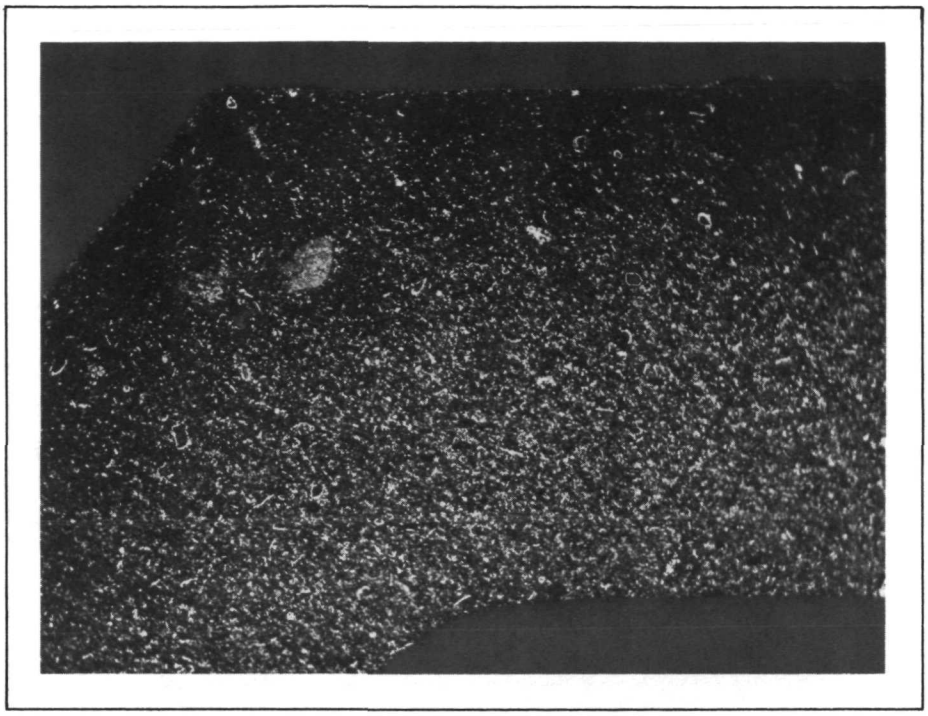
Composition			Mercury Immersion Density,* %	
Type A-1 Tungsten, %	UO_2, %	ThO_2, %	1880°C. -1 hr. -H_2 1995°C. -3-1/2 hr. -He	1880°C. -4-1/2 hr. -H_2
88	12	0	89.0	91.7
87.5	12	0.5	89.6	91.1
87	12	1	88.3	90.0
85.5	12	2.5	88.9	91.5
84	12	4	87.0	90.7

*Densities are calculated using theoretical densities of the pure materials and assuming the original ratios are present in the sintered product.



PHOTOGRAPH OF TYPICAL HEXAGONAL TUBES

Figure 18



Material:
Type A-1 Tungsten Powder

Density: 44.1%

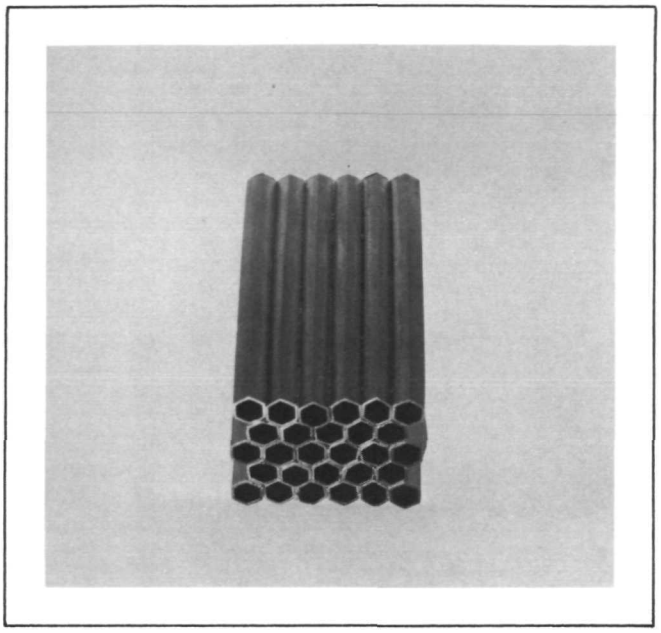
Unsintered Tungsten Hexagonal Tube (150X)

Material:
Type A-1 Tungsten Powder
Type B-1 Uranium Dioxide Powder
Density: 47.5%

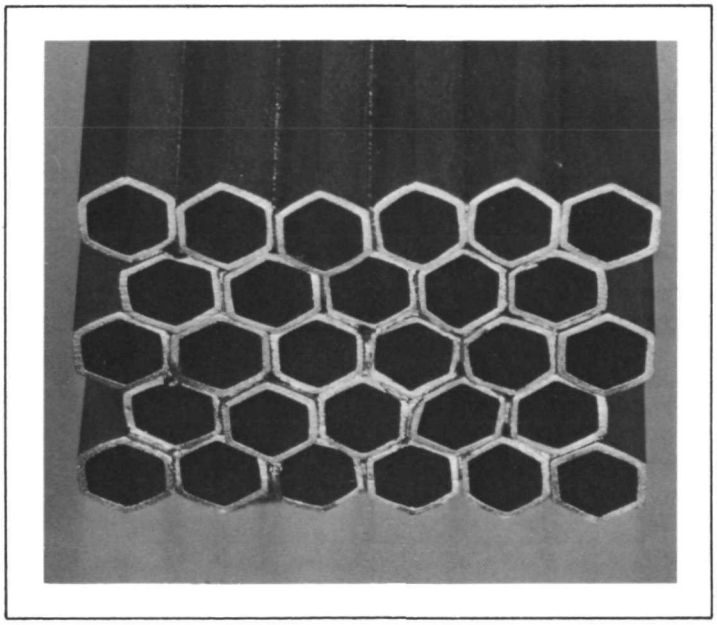
Unsintered Tungsten-Uranium Dioxide Tube (150X)

CROSS-SECTION PHOTOMICROGRAPHS OF TYPICAL UNSINTERED TUNGSTEN AND TUNGSTEN-URANIUM DIOXIDE HEXAGONAL TUBES

Figure 19



a. (1X)



b. (3X)

Material: Type A-1 Tungsten Powder

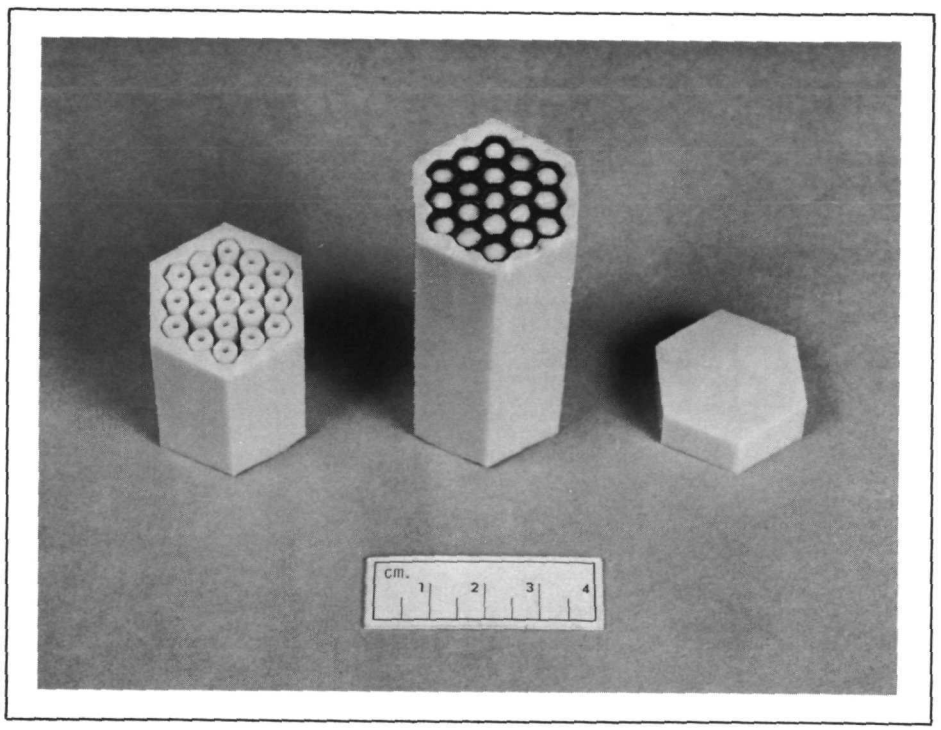
1200°C. - 60 min. - H_2

1900°C. - 30 min. - H_2

2370°C. - 60 min. - Vac.

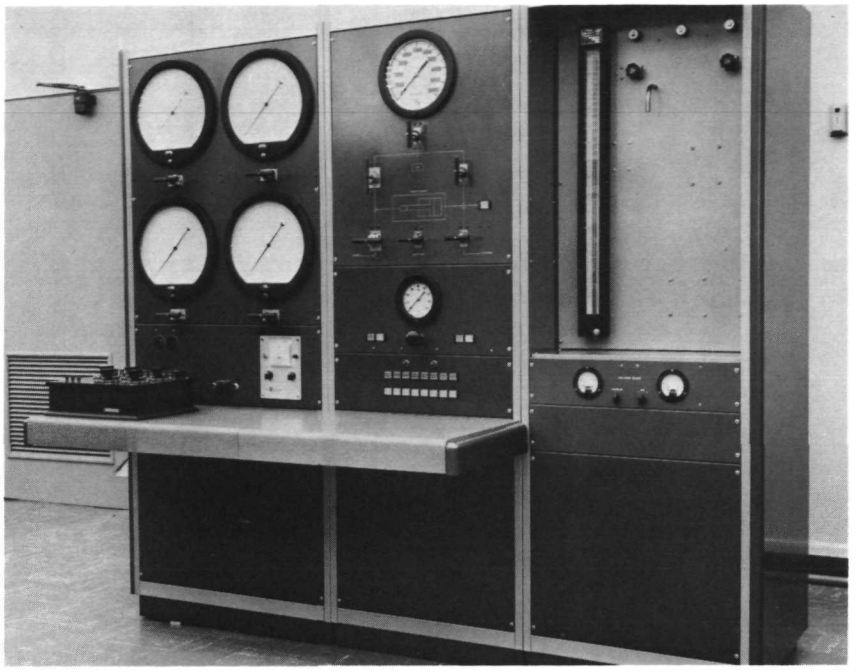
PHOTOGRAPHS OF SINTERED TUNGSTEN HONEYCOMB - STACKED TUBES METHOD

Figure 20

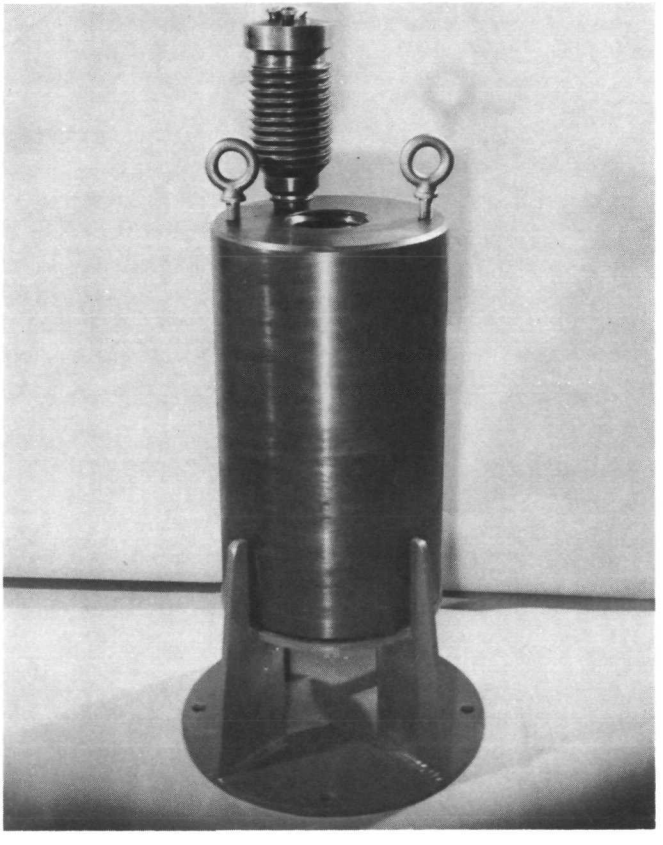


PHOTOGRAPH OF RUBBER ISOSTATIC PRESSING BOOTS AND INSERTED TUBES

Figure 21



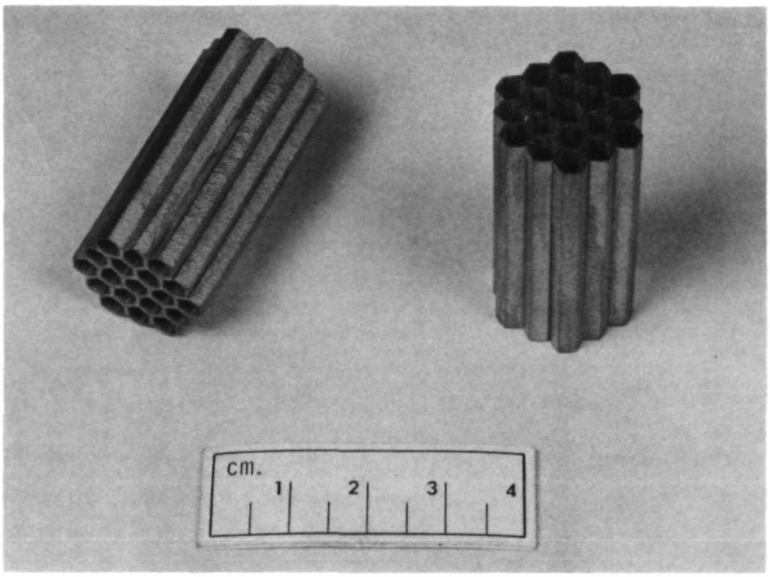
a. High Pressure System



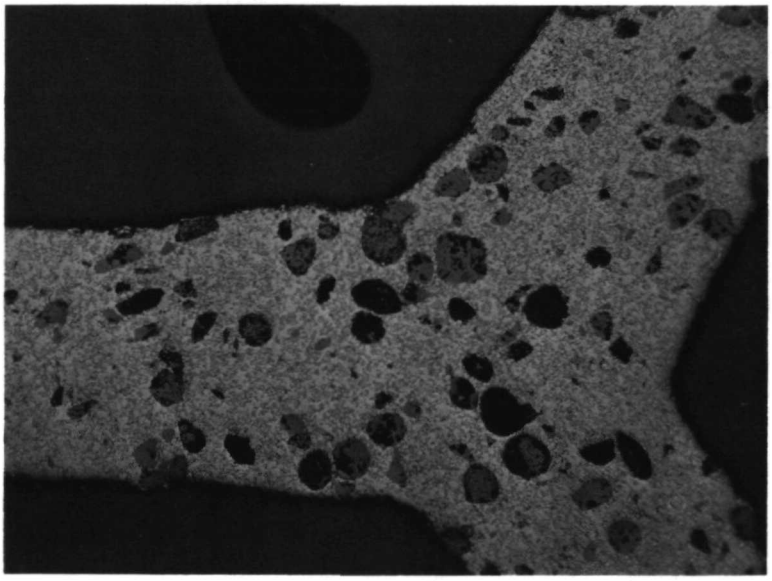
b. Isostatic Pressure Vessel

EQUIPMENT USED FOR ISOSTATIC PRESSING OF STACKED TUBES

Figure 22



a. Tungsten-UO_2 Honeycombs



b. Tube Junction (50X)

Material:

Type A-2 Tungsten Powder
20 v/o - Type B-1 Uranium Dioxide Powder

Pressing:

(4794-27) - 79,500 psi
(4794-32-A) - 80,000 psi

Sintering:

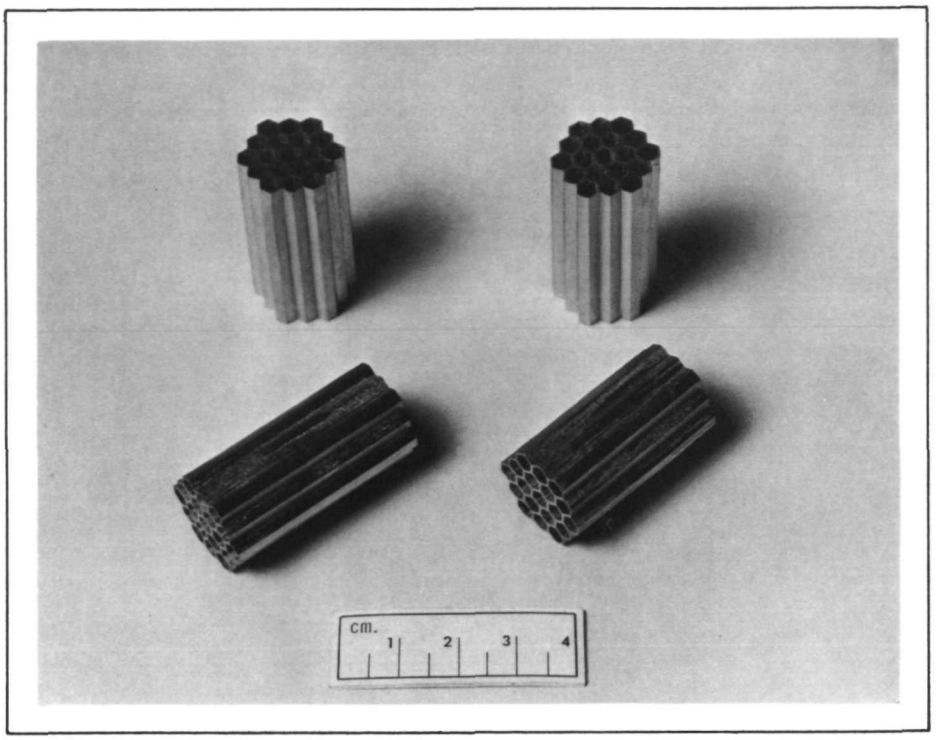
1200°C. - 75 min. - H_2
2000°C. - 180 min. - H_2

Density:

(4794-27) - 94.6%
(4794-32-A) - 94.1%

ISOSTATICALLY PRESSED STACKED TUBE
TUNGSTEN-URANIUM DIOXIDE HONEYCOMBS
(4794-27, 4794-32-A)

Figure 23



Material: Type A-2 Tungsten Powder

Pressing:

(4794-38) (4794-39) 78,000 psi

(4794-40) (4794-41) 82,000 psi

Sintering:

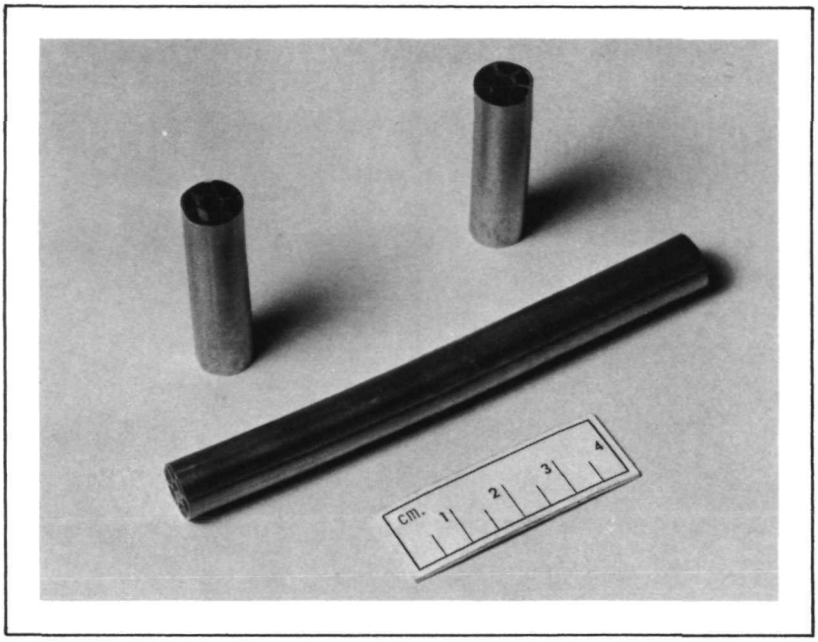
1200°C. - 75 min. -H_2
2400°C. -390 min. -Vac.

Density:

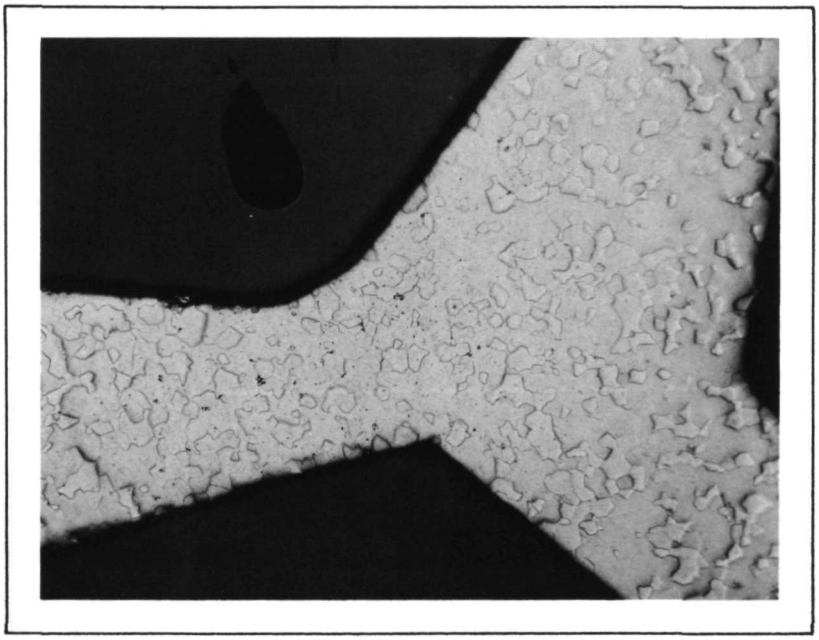
(4794-38) - 98.0%
(4794-39) - 98.0%
(4794-40) - 97.9%
(4794-41) - 98.2%

ISOSTATICALLY PRESSED STACKED TUBE TUNGSTEN HONEYCOMBS
(4794-38, 4794-39, 4794-40, 4794-41)

Figure 24



a. Seven Channel Honeycombs



b. Typical Channel Junction (50X)

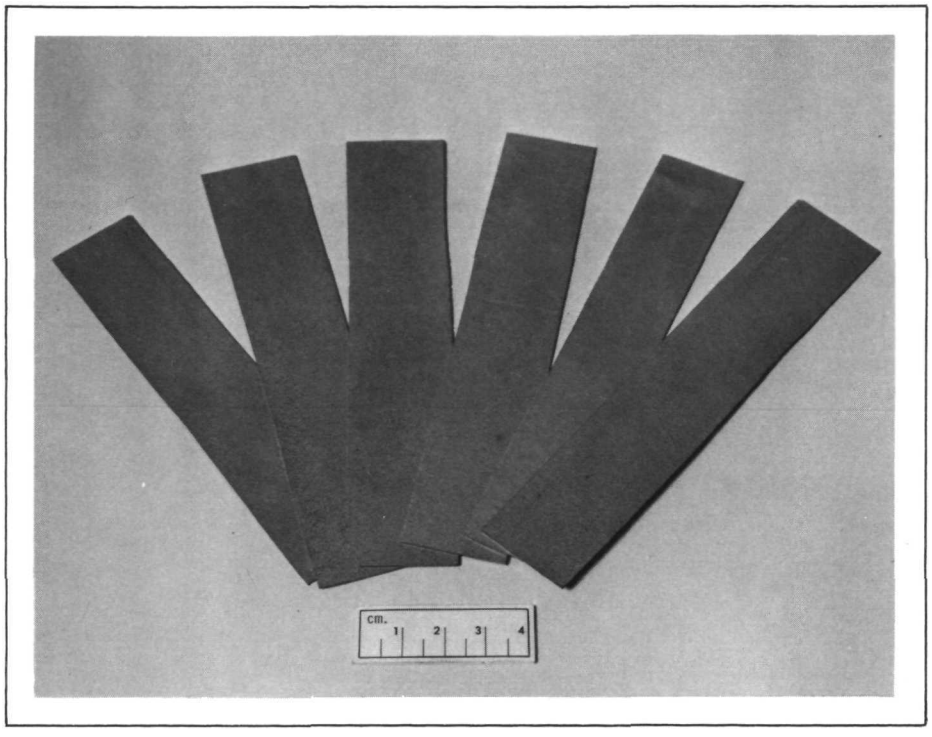
Material:
Type A-2 Tungsten Powder

Sintering:
(a) 1-1/2 in. Units:
1200°C. - 75 min. - H_2
2400°C. - 330 min. - Vac.
(b) 4 in. Unit:
1200°C. - 75 min. - H_2
2000°C. - 120 min. - H_2

Density:
(a) 1-1/2 in. Units:
97.9%; 98.0%
(b) 4 in. Unit:
96.2%

SEVEN CHANNEL HONEYCOMB FORMED AS A UNIT
(4794-49-1, 4794-49-2, 4794-50-1)

Figure 25



Material:

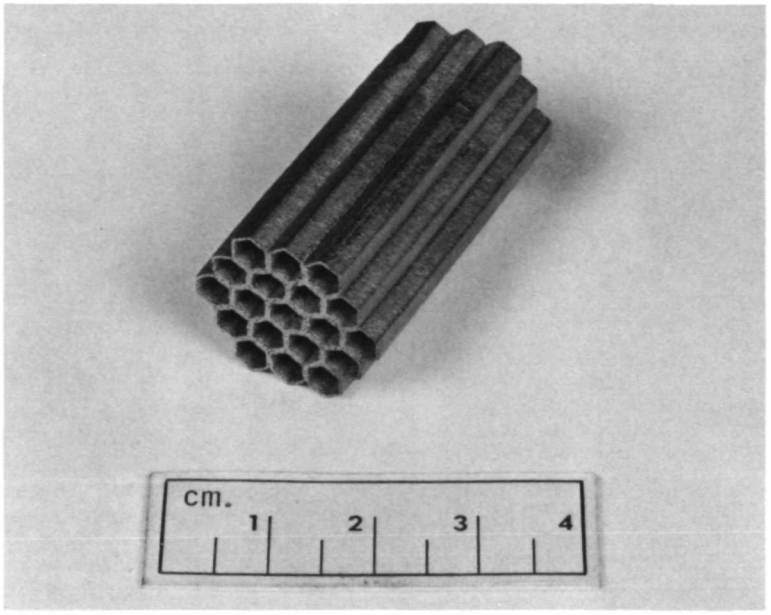
Type A-2 Tungsten Powder
20 v/o Type B-1 Uranium Dioxide Powder

Sintering:

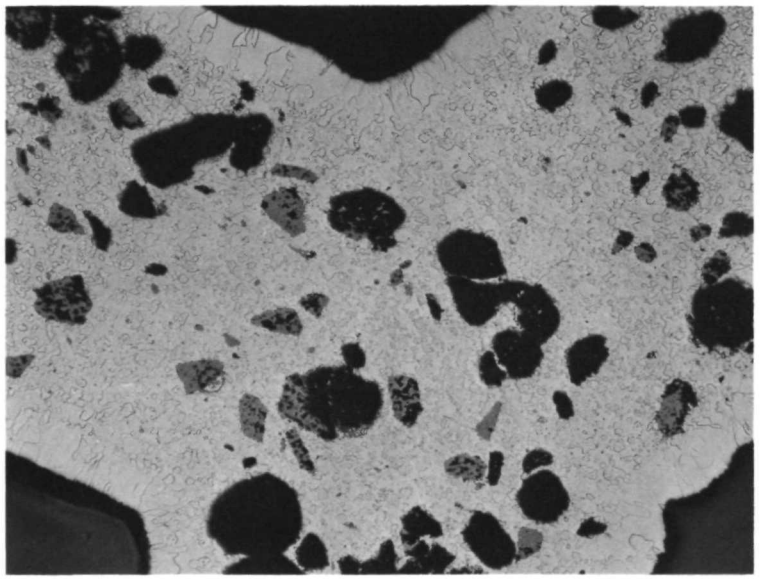
1200°C. - 75 min. - Hydrogen
1600°C. - 80 min. - Hydrogen
2025°C. - 180 min. - Hydrogen

TUNGSTEN-URANIUM DIOXIDE FLAT SHEETS
(4794-22-2-A thru F)

Figure 26



a. Tungsten Clad Tungsten-UO_2 Honeycomb



b. Typical Tube Junction (50X)

SUBSTRATE:

Material:

Type A-1 Tungsten Powder
Type B-1 Uranium Dioxide Powder

Pressing: 72,000 psi

Sintering:
1200°C.- 75 min. - H_2
1600°C. - 60 min. - H_2

PLATING CONDITIONS:

910°F. - 1 hour
65 - 75 scfh Hydrogen
87 cc/min. WF_6

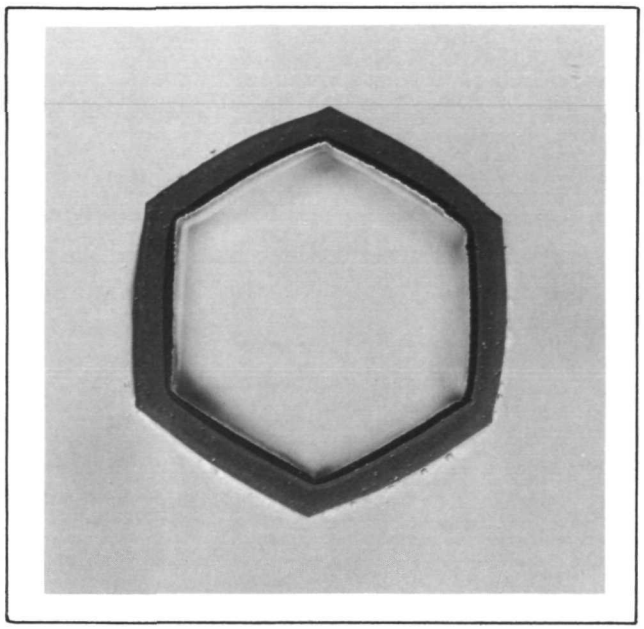
RESINTERING:

2000°C. - 120 min. - H_2

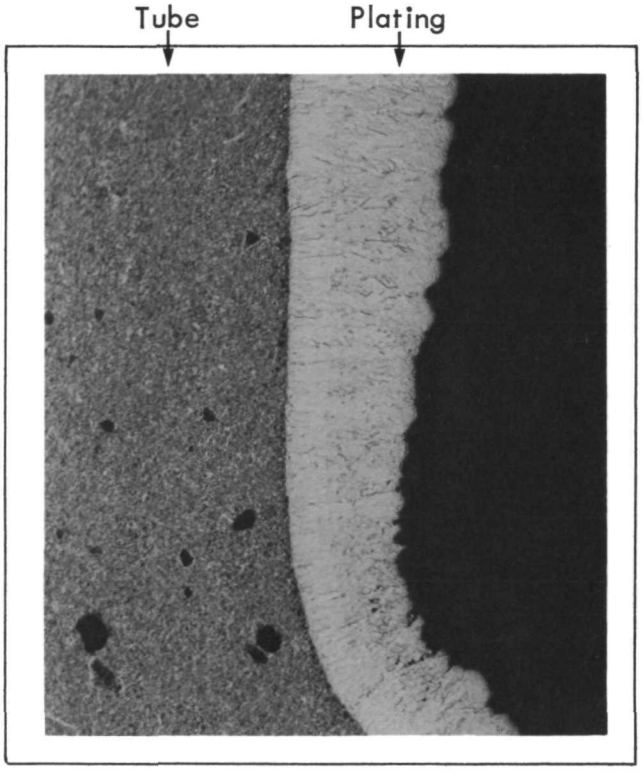
DENSITY: 91.5%

TUNGSTEN CLAD ISOSTATICALLY PRESSED STACKED TUBE
TUNGSTEN-URANIUM DIOXIDE HONEYCOMB
(4794-32-C)

Figure 27



a. (10X)



b. (250X)

Material: Tungsten

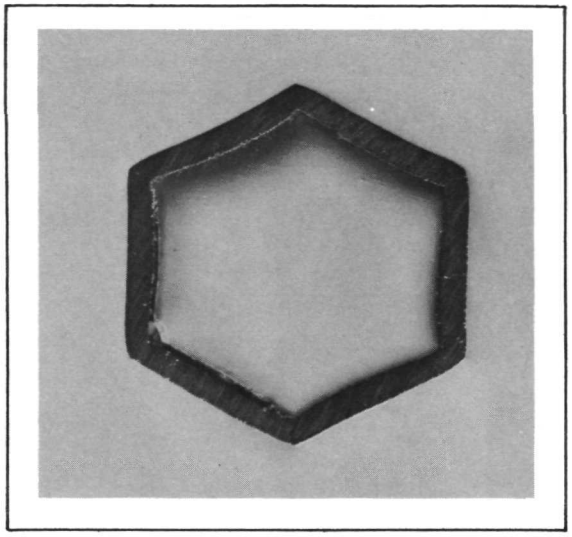
1200°C. - 60 min. - H_2

Plating Conditions:

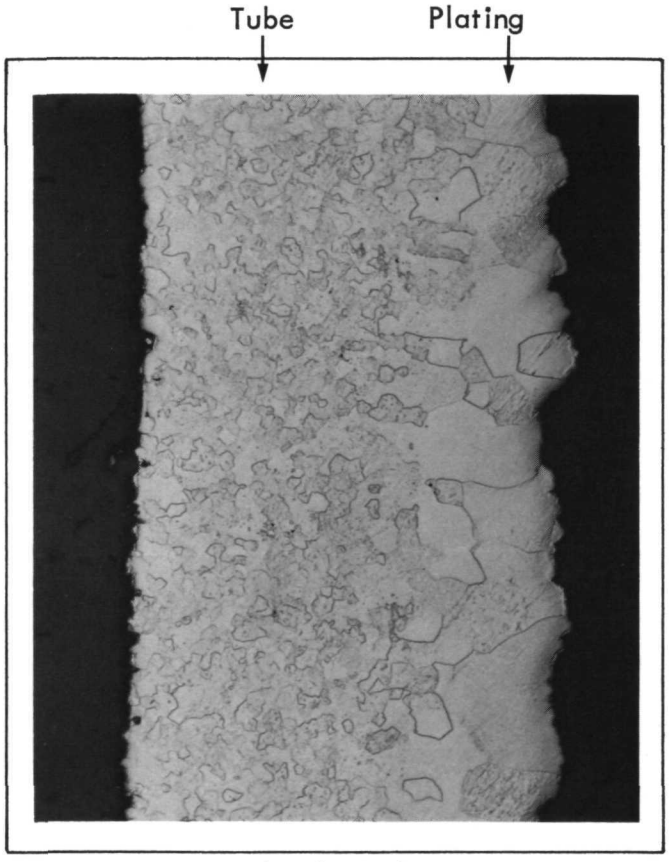
900°F.
14 scfh Hydrogen
150 cc./min. WF_6

TUNGSTEN CLADDING ON PARTIALLY
SINTERED TUNGSTEN HEXAGONAL TUBE

Figure 28



a. (10X)



b. (150X)

Material: Tungsten

1200°C. - 60 min. - H_2

Plating Conditions:

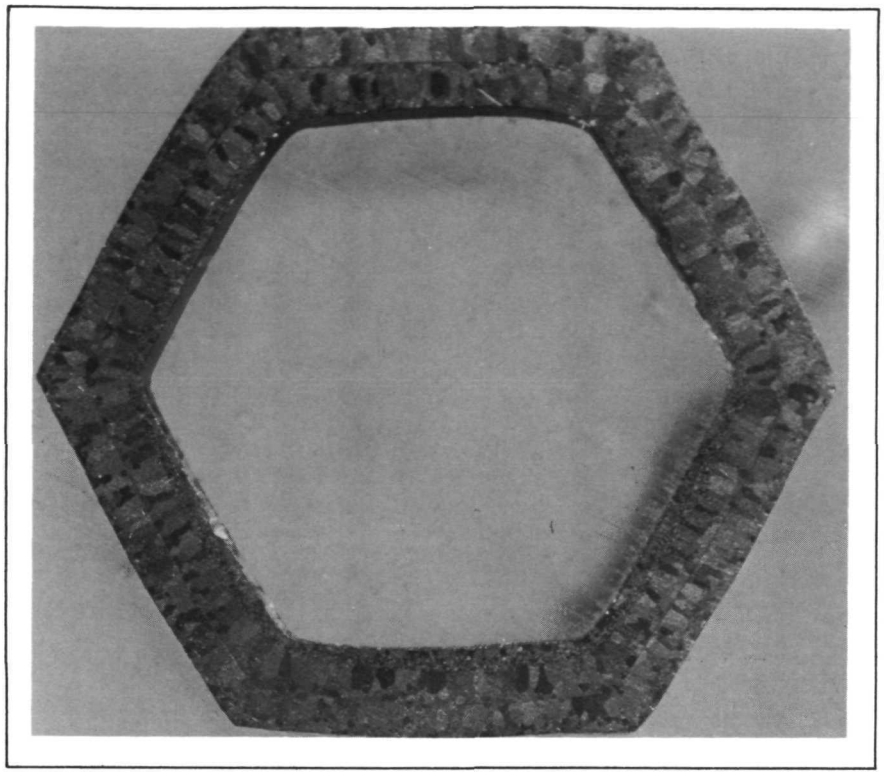
900°F.
14 scfh Hydrogen
150 cc./min. WF_6

Heat Treatment:

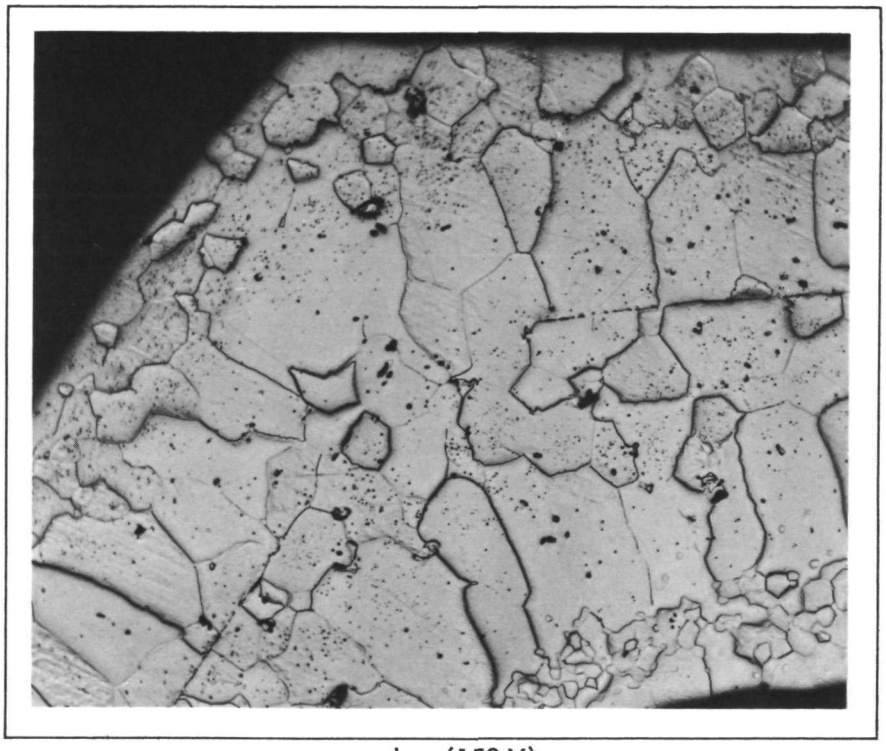
1860°C. - 270 min. - H_2

HEAT TREATED TUNGSTEN CLADDING ON
TUNGSTEN HEXAGONAL TUBE

Figure 29



a. (20X)



b. (150X)

SINTERED BONDED TUBES

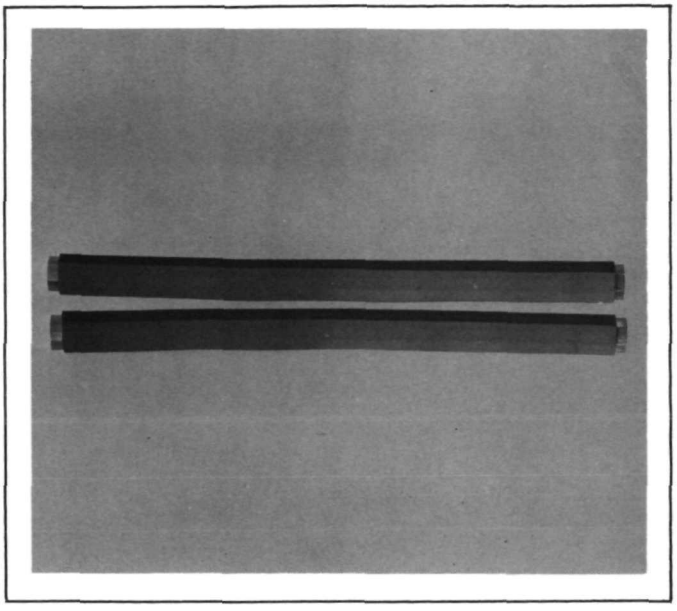
Figure 30

Material:
Type A-1 Tungsten Powder

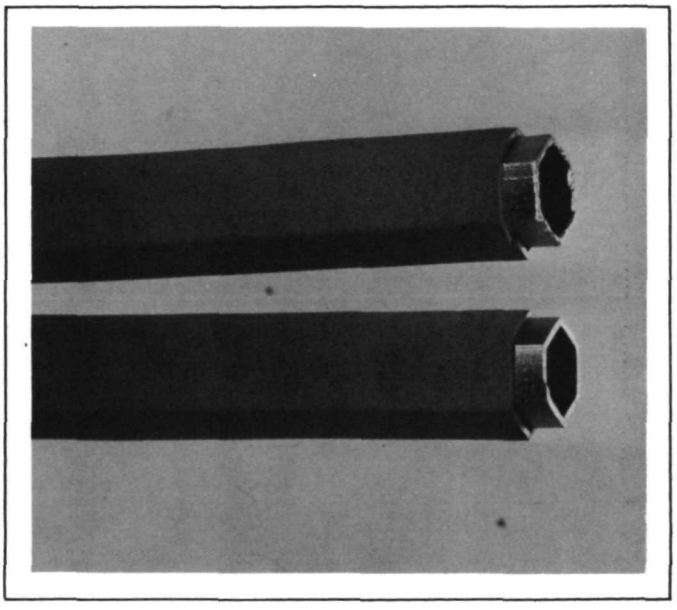
Inner Tube:
1200°C. -60 min. -H_2
1860°C. -15 min. -H_2
2300°C. -30 min. -Vac.

Outer Tube:
1200°C. -60 min. -H_2

Combined Tubes:
1912°C. -30 min. -H_2
2350°C. -60 min. -Vac.



a. (IX)



b. (3X)

Material:

Inner Tube: Type A-1 Tungsten Powder

1200°C. - 60 min. - H_2
1860°C. - 15 min. - H_2
2300°C. - 30 min. - Vac.

Outer Tube:

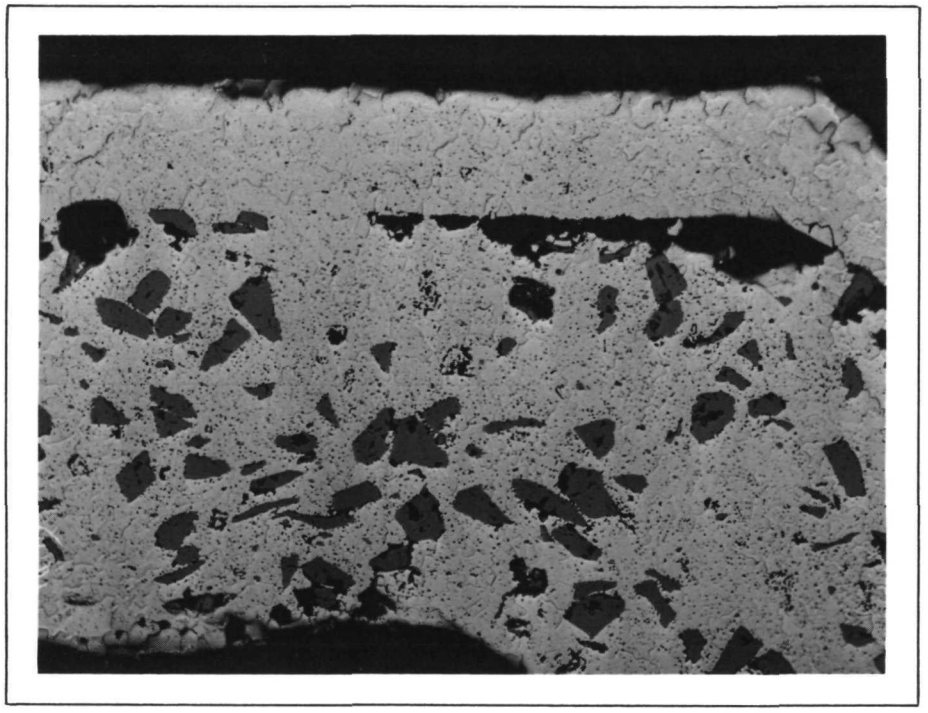
22.6 w/o Type B-1 UO_2 and
77.4 w/o Type A-1 Tungsten
Unsintered

Combined Tubes:

1200°C. - 60 min. - H_2

SINTERED TUNGSTEN TUBES INSIDE LOW TEMPERATURE
SINTERED TUNGSTEN-URANIUM DIOXIDE HEXAGONAL TUBES

Figure 31



150X

PHOTOMICROGRAPH OF THIN-WALL
CLAD TUNGSTEN-URANIUM DIOXIDE HEXAGONAL TUBE

Figure 32

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