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Argonne National Laboratory

**DEVELOPMENT OF TECHNIQUES FOR
FABRICATION OF SMALL-DIAMETER,
THIN-WALL TUNGSTEN AND
TUNGSTEN-ALLOY TUBING**

by

**W. R. Burt, Jr., D. C. Brillhart,
and R. M. Mayfield**

RELEASED FOR ANNOUNCEMENT
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DEVELOPMENT OF TECHNIQUES FOR
FABRICATION OF SMALL-DIAMETER,
THIN-WALL TUNGSTEN AND
TUNGSTEN-ALLOY TUBING

Interim Progress Report covering the period
from Jan. 1, 1964, through Dec. 31, 1965

by

W. R. Burt, Jr., D. C. Brillhart,
and R. M. Mayfield

Metallurgy Division
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FOREWORD

This interim progress report on tungsten tubing fabrication is limited to progress of technical significance that has occurred during the reporting period. Since the intent is to report only items of significant progress, not all work completed during the period is reported. The results and data presented are preliminary and subject to change unless otherwise stated.

The issuance of this report is not intended to constitute final publication. Final results will appear in professional journals or will be published as an ANL topical report.

This is the second report on this program. Progress reports will contain a list of preceding reports to ensure continuity. Preceding report:

- 1) ANL-6806, Burt, W. R., Jr., Katiyar, H. C., and Mayfield, R. M., Development of Techniques for Fabrication of Small-diameter Thin-wall Tungsten Tubing, Interim Progress Report covering July 1, through December 31, 1963.

TABLE OF CONTENTS

	<u>Page</u>
ABSTRACT	7
I. INTRODUCTION	8
II. EQUIPMENT	9
III. STARTING MATERIALS	9
A. Tungsten	11
B. Tungsten-25 w/o Rhenium	13
C. Tungsten-30 a/o Molybdenum-30 a/o Rhenium	14
IV. PRIMARY FABRICATION	14
A. Primary Extrusion	14
1. Unalloyed Tungsten	14
2. Tungsten-25 w/o Rhenium	15
3. Tungsten-30 a/o Molybdenum-30 a/o Rhenium	16
4. Tungsten Granules	16
B. Annealing Behavior	17
1. Tungsten	17
2. Tungsten-25 w/o Rhenium	18
3. Tungsten-30 a/o Molybdenum-30 a/o Rhenium	21
4. Tungsten Granules	21
C. Secondary Extrusion	21
1. Unalloyed Tungsten	21
2. Tungsten-25 w/o Rhenium	24
D. Extrusion-core Removal	24
V. SECONDARY FABRICATION	26
A. Swaging	26
1. Tungsten	26
2. Tungsten-25 w/o Rhenium	27
B. Ductile-core Drawing	27
1. Tungsten	27
2. Tungsten-25 w/o Rhenium	28
C. Plug Drawing	28
1. Tungsten-25 w/o Rhenium	28
2. Tungsten-30 a/o Molybdenum-30 a/o Rhenium	29

TABLE OF CONTENTS

	<u>Page</u>
VI. INDUSTRIAL PARTICIPATION	30
A. Unalloyed Tungsten	30
B. Tungsten-25 w/o Rhenium.	30
C. Tungsten-30 a/o Molybdenum-30 a/o Rhenium	30
VII. DISCUSSION	32
VIII. FUTURE PLANS	33
APPENDIX: Product Evaluation	34
REFERENCES	36

LIST OF FIGURES

<u>No.</u>	<u>Title</u>	<u>Page</u>
1.	Bank of Rotary Swaging Machines	10
2.	Centorr High-vacuum, Cold-wall, Annealing Furnace	10
3.	Typical Microstructures of Conventional and "High-purity" Pressed and Sintered Tungsten Sleeves Procured from the General Electric Company	12
4.	Microstructure of a Pressed and Sintered W-25Re Sleeve Purchased from the Chase Brass Company	13
5.	Microstructure of a Pressed and Sintered Tungsten-30 a/o Rhenium-30 a/o Molybdenum Sleeve Purchased from the Chase Brass Company	14
6.	Typical Billet Assembly for Primary Extrusion of a Tungsten or Tungsten-alloy Sleeve	15
7.	Photograph of Extruded Tube-blanks after Removal of Jacket and Core	16
8.	Extrusion Dies Coated with ZrO_2 before and after Use	16
9.	Characteristic Surface Appearance of Extruded Tungsten Granules (Allied Chemical)	17
10.	Annealing Behavior of a "High-purity" Pressed and Sintered Tungsten Sleeve (GE) Extruded at 1150°C and a 4.1:1 Reduction Ratio	18
11.	Annealing Behavior of a Commercial-purity Pressed and Sintered Tungsten Sleeve (GE) Extruded at 1150°C and a 4.1:1 Reduction Ratio	19
12.	Hardness Measurements on Conventional and "High- purity" Extruded and Annealed Tungsten Sleeves (GE)	19
13.	Annealing Behavior of a Pressed and Sintered W-25Re Sleeve (Chase Brass) Extruded at 1450°C and a 3.2:1 Reduction Ratio	20
14.	Hardness Measurements on W-25Re Extruded at 1450°C and a 3.2:1 Reduction Ratio	21
15.	Annealing Behavior of Longitudinal Tube Sections of Extrusion-compacted (1400°C; 8.8:1 Reduction Ratio) Tungsten Granules	22
16.	Annealing Behavior of Transverse Tube Sections of Extrusion-compacted (1400°C; 8.8:1 Reduction Ratio) Tungsten Granules	23

LIST OF FIGURES

<u>No.</u>	<u>Title</u>	<u>Page</u>
17.	Hardness Measurements on Tungsten Granules Extruded at 1400°C and an 8.8:1 Reduction Ratio	24
18.	Tungsten-25 w/o Rhenium Extruded at 1350, 1400, and 1500°C using Nb-5Zr (D-14) Cores	25
19.	Modified Design of Hot-swaging Dies	27
20.	"Cluster" Design of Extrusion Billets for Tertiary Extrusion of W-25Re Tubing.	28
21.	Schematic Representation of Equipment Used during Plug-drawing	29
22.	Target-size Tubing of W-30a/oRe-30a/oMo Procured from Chase Brass	31

TABLE

<u>No.</u>	<u>Title</u>	<u>Page</u>
I.	Chemical Analyses of Tungsten and Tungsten-alloy Pressed and Sintered Sleeves	11

DEVELOPMENT OF TECHNIQUES FOR
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ABSTRACT

Progress in the fabrication of small-diameter, thin wall tungsten and tungsten-alloy tubing is summarized for the reporting period. Methods are described for producing tungsten or tungsten-25 w/o rhenium (W-25Re) tube-blanks by double extrusion. The starting material, generally a pressed and sintered product, is in the form of a sleeve 3.17-cm OD x 2.54-cm ID x ~10.2 cm long (1.25-in. OD x 1.00-in. ID x ~4.0 in. long). Both initial and re-extrusion processes use the filled-billet technique. The resultant tube-blanks, ~0.64-cm OD x 0.51-cm ID x 75 cm long (0.25-in. OD x 0.20-in. ID x 30 in. long), are structurally sound and suitable for secondary fabrication operations.

Secondary fabrication operations have been concentrated on the W-25Re alloy. Plug-drawing has emerged as an excellent technique, both for the reduction of the overall tube dimensions and for the removal of the longitudinal surface striations resulting from the filled-billet extrusion sequence. Plug-drawing also obviates the need for core removal at the finished-tube size (< 0.125 -in. OD). Even at larger tube sizes, core removal remains a problem, and improved methods are being sought. Crack-free swage pointing of the small-diameter W-25Re tubing is difficult unless proper dies and swaging techniques are employed. Progress in this area is presented. Sound pointing practice should permit the establishment of definitive secondary fabrication procedures for tungsten and tungsten-alloy tubing. Industrial participation in the program has been encouraged, and early results are presented.

I. INTRODUCTION

This is the second progress report on the fabrication of small-diameter, thin-wall tungsten and tungsten-alloy tubing. The initial report, ANL-6806,¹ primarily covered extrusion of unalloyed tungsten. This report covers continued extrusion development, but also stresses secondary fabrication of extruded tubing. Most of the work has been on the W-25Re alloy.

The fabrication of tungsten and tungsten alloys into small-diameter, thin-wall tubing of nuclear quality is one of the most formidable problems facing the mechanical metallurgist today. The small-diameter, thin-wall classification includes tubing of diameters 0.32 cm (0.125 in.) or less with wall thicknesses approximately 10% of the OD. Forming of these materials into tubing is difficult, not only because of the high strength of the materials involved but also because of the high ductile-brittle transition temperature (DBTT) of tungsten and its alloys. This high DBTT (from 200 to 400°C) necessitates warm-working in a temperature range that has been used with only limited familiarity heretofore.

The experimental effort involves utilization of both arc-cast and powder-metallurgy starting materials, the major emphasis being on the latter. Primary fabrication by extrusion has been the choice of most workers in the field. Although some extrusion work over a tool-steel mandrel has been reported,² most extruded tube-blanks have been produced by the "filled-billet" technique of coextruding a powder metallurgy sleeve with internal cores and external jackets of either steel or molybdenum.^{1,3} This technique provided sound extrusions but has led, in turn, to difficulties in the removal of the extrusion cores from small-diameter tube-blanks. All things considered, the primary fabrication of tungsten and its alloys has progressed quite well in the past few years.

Until recently, secondary fabrication has received little attention. Investigatory work has been carried out at various sites (both industrial firms and national laboratories) on swaging, ductile-core, mandrel, and plug-drawing. To date, little tubing has been produced in substantial lengths by any drawing technique. The most vigorous work is now being concentrated in this area.

At ANL efforts have been directed toward primary and secondary fabrication, no work being undertaken in the area of consolidation. All starting materials have been procured, mainly from commercial sources. Primary fabrication of pressed and sintered starting sleeves by double extrusion has led to the development of a reliable technique for the production of high-quality tube-blanks for secondary fabrication.

Secondary fabrication has utilized both plug-drawing and drawing over the extrusion cores. It is hoped that a continued effort on plug-drawing will result in a reliable technique for the fabrication of high-quality, small-diameter tubing.

II. EQUIPMENT

A completely integrated facility is available at ANL for processing raw materials from ingots to final products. Available for use are an electron-beam furnace, an arc furnace, two extrusion presses, two tube straighteners, three rolling mills, two drawbenches, three swaging machines, a centerless belt sander, and other miscellaneous processing and evaluation equipment. Much of this equipment has been described previously;¹ only the drawbenches, the swaging machines, and the vacuum annealing furnace are described here.

The drawbenches have 4,550- and 2,270-kg (10,000- and 5,000-lb) capacities. The larger bench can draw tubing up to 3.7 m (12 ft) long. This apparatus currently is used for room-temperature work only and will not be immediately available for hot-drawing. All secondary fabrication work on tungsten and its alloys currently is performed on the 2,270-kg (5,000-lb) drawbench, which can handle tubing up to 1.8 m (6 ft) long. A resistance furnace mounted ahead of the die is used to heat the work piece to temperatures up to 1000°C. The drawing dies are of the tungsten carbide insert type.

The Fenn two-die rotary swaging machines are shown in Fig. 1. Cast Stellite hot-working dies serve for tubing down to 0.358-cm (0.141-in.)-diam; machined tool-steel dies are employed for smaller-diameter tubing.

Annealing is performed in a Centorr high-vacuum, cold wall, resistance furnace shown in Fig. 2. This furnace incorporated a three-phase, silicon-controlled-rectifier (SCR) power supply. Temperatures up to 2100°C are obtained via a tantalum heating element surrounded by molybdenum heat shields. The power requirement at 2100°C is ~15 kVA. The uniform hot zone is approximately 7.6 cm (3 in.) in diameter by 61 cm (24 in.) long. The temperature is controlled to $\pm 20^\circ\text{C}$ at 2100°C.

III. STARTING MATERIALS

The major effort over the past two years has concentrated on fabrication of pressed and sintered tungsten and tungsten-alloy sleeves. Some work, albeit slight, was continued on the direct consolidation and compaction of tungsten granules by extrusion. This latter effort will be mentioned only briefly. Other starting materials, such as arc-cast sleeves, will be factored into the program, but at a later date.

106-8206

Fig. 1. Bank of Rotary Swaging Machines

106-8203

Fig. 2. Centorr High-vacuum, Cold-wall, Annealing Furnace

Before extrusion at ANL, all sleeves were examined by X-radiography. Of the approximately four dozen sleeves examined to date, only one or two had major defects (i.e., cracks). The quality of these sleeves has been universally high, meeting specifications in all cases.

A. Tungsten

The bulk of the work on unalloyed tungsten was with sleeves purchased from the General Electric Company, Lamp Metals and Components Dept., Cleveland, Ohio. These sleeves were 3.17-cm OD x 2.54-cm ID x 10.2 cm long (1.25-in. OD x 1.00-in. ID x ~4.0 in. long) and of two classifications as designated by GE. The first was conventional-purity tungsten (similar to the sleeves covered in ANL-6806); the second was designated "high-purity" tungsten. Typical chemical analyses for both materials are listed in Table I. The major difference in these materials was in the as-sintered density, the conventional and "high purity" being 92 and 95% of the theoretical density, respectively. Typical microstructures are shown in Fig. 3.

TABLE I. Chemical Analyses of Tungsten and Tungsten-alloy Pressed and Sintered Sleeves

Element	Analysis (ppm)				
	Allied Tungsten Granules ^a	Pressed and Sintered Conventional-purity Tungsten ^b	Pressed and Sintered High-purity Tungsten ^b	Pressed and Sintered W-25w/oRe Sleeves ^c	Pressed and Sintered W-30a/oMo-30a/oRe ^c
C	<10	<10	15	30	20
O ₂	7	21 ± 5	3 ± 3	20	20
N ₂	<1	1 ± 1	1 ± 1	10	10
H ₂	1	1 ± 1	1 ± 1	1	1
Al	<10	<6	<6	<1	<1
Si	<10	<7	<7	1	<10
Fe	<10	8	7	24	28
Cr	NA	4	4	<1	<1
Ni	<10	10	3	<10	5
Cu	<5	<3	10	<1	5
Mo	<20	62	36	<50	17.6 w/o
Mn	NA	<6	<6	NA	NA
Mg	<3	<3	6	<1	<1
Sn	NA	7	<6	NA	<1
Ca	NA	<3	31	<1	<10

(a) Supplied by Allied Chemical Company, General Chemical Division, Morristown, New Jersey.

(b) Supplied by General Electric, Lamp Metals and Components Dept., Cleveland, Ohio.

(c) Supplied by Chase Brass Co., Rhenium Division, Waterbury, Conn.

39228

150X

(a) Conventional-purity pressed and sintered tungsten (GE).
Hardness: 320 DPH (32 Rc)

43066

150X

(b) "High-purity" pressed and sintered tungsten (GE).
Hardness: 233 DPH (97 R_B)

Fig. 3. Typical Microstructures of Conventional and "High-purity" Pressed and Sintered Tungsten Sleeves Procured from the General Electric Company [Etchant: 5% KOH, 5% $K_3Fe(CN)_6$]

The tungsten granules were obtained from the Allied Chemical Company, the chemical analysis being listed in Table I. (Further information concerning this material is contained in ANL-6806.¹)

B. Tungsten-25 w/o Rhenium

The W-25Re sleeves were purchased primarily from the Chase Brass Co. of Waterbury, Conn., a few sleeves being received from GE and the Bureau of Mines at Albany, Oregon. The Chase Brass sleeves were hydrostatically pressed and sintered in hydrogen at 2600°C. The microstructure of a Chase W-25Re sleeve is shown in Fig. 4. The fine particles that are discernible in the microstructure are rhenium-rich sigma phase. Chemical analyses of this material, as received from the vendor, are included in Table I.

Fig. 4. Microstructure of a Pressed and Sintered W-25Re Sleeve Purchased from the Chase Brass Company. As-sintered hardness: 353 DPH (36 R_C). [Etchant: 5% KOH, 5% $K_3Fe(CN)_6$]

C. Tungsten-30 a/o Molybdenum-30 a/o Rhenium

The tungsten-30 a/o molybdenum-30 a/o rhenium alloy was obtained solely from the Chase Brass Company. The sintering parameters of this material were not available, but were believed to be similar to those of the binary alloy. The chemical analyses are presented in Table I; the microstructure is shown in Fig. 5.

Fig. 5. Microstructure of a Pressed and Sintered Tungsten-30 a/o Rhenium-30 a/o Molybdenum Sleeve Purchased from the Chase Brass Company [Etchant: 5% KOH, 5% $K_3Fe(CN)_6$]

IV. PRIMARY FABRICATION

The primary fabrication of pressed and sintered sleeves has been confined to double extrusion, both extrusions being of the "filled-billet" or coextrusion technique employing appropriate core and jacket materials. This method was considered to be more amenable to the high-temperature extrusion of small-diameter tubing (≤ 0.5 -in. OD) than the technique of extruding over a floating or stationary mandrel. All extrusion work was performed on a 350-ton vertical-extrusion press. (The press is described in ANL-6806.¹)

A. Primary Extrusion

1. Unalloyed Tungsten

After many trials, it was determined that a molybdenum core and a Type 304 stainless-steel jacket, coupled with a reduction ratio of 4:1

Fig. 6. Typical Billet Assembly for Primary Extrusion of a Tungsten or Tungsten-alloy Sleeve

and an extrusion temperature of 1150°C , provided the best combination for obtaining sound primary extrusions on a consistent basis. A typical billet assembly for primary extrusion is shown in Fig. 6. Higher reduction ratios than 4:1 were feasible at the extrusion temperature of 1150°C , but occasional tearing and fracture of the extruded tube-blank made a higher reduction ratio unattractive. Higher extrusion temperatures, to enable larger reduction ratios, resulted in "rippling" or severe nonuniform deformation of the tungsten tube-blank. This is probably due to the lack of adequate strength in the stainless-steel outer jacket at the higher extrusion temperatures.

Two primary extrusions of unalloyed tungsten sleeves, one of commercial purity and the other of "high purity," are shown in Fig. 7. The tube-blank size after primary extrusion was approximately 1.59-cm OD x 0.16-cm wall (0.625-in. OD x 0.062-in. wall). The tungsten primary extrusions, made with the use of stainless-steel jackets and molybdenum cores, normally were defect-free except for the usual "dog-bone" defects at the lead and tail ends. Although no detailed comparison was made, there appeared to be no significant difference in the extrudability between the conventional and "high-purity" tungsten sleeves based on press tonnages or extrusion constants.

2. Tungsten-25 w/o Rhenium

Because of the high strength and high work-hardening rate of the W-25Re alloy, this alloy was extruded at a temperature substantially higher than for unalloyed tungsten. The extrusion temperature was established at $1400 \pm 50^{\circ}\text{C}$ for the primary breakdown of the pressed and sintered sleeve, the reduction ratio being 4:1.* This extrusion temperature necessitated a change in jacket material from Type 304 SS to molybdenum. Although the cost of extrusion components is significantly higher, the alloy could not be extruded at temperatures sufficiently low to permit use of a stainless-steel outer jacket. A typical product of the primary extrusion of W-25Re is shown in Fig. 7. The relative stiffness of the alloy in contrast to pure tungsten is evident from the relatively slight amount of nonuniform deformation in the lead end of the extrusion.

*Although higher reduction ratios ($\sim 7:1$) were feasible and produced sound tube-blanks, press capacity became a limiting factor, and primary extrusions normally were made at a 4:1 reduction ratio.

42398 ~ 1/2X
(a) (b) (c)

Fig. 7. Photograph of Extruded Tube-blanks after Removal of Jacket and Core. The materials are: (a) W-25Re, (b) "high-purity" tungsten, and (c) commercial-purity tungsten.

given conditions. As in many endeavors, however, an ultimate "conspiracy of nature" developed. The ternary alloy was strongly attacked by aqua regia and other acids which were normally used to remove jackets and cores from extrusions. This problem became more pronounced for the small tube sizes. Emphasis on the W-25Re alloy has postponed additional work to overcome this problem.

4. Tungsten Granules

Direct compaction of tungsten granules by extrusion has been quite successful (see Reference 1, p. 35), although the granule size results in a rough surface. A typical

In past work, extrusion dies were machined from cast H-12 tool-steel die blanks and hardened to R_C 47-52. At extrusion temperatures above 1200°C , however, die wash became a serious problem, and it was necessary to coat the dies with zirconia (ZrO_2). Ribs were machined on the die-entrance surface to improve adherence of the ceramic coating. The dies were machined, hardened to R_C 47-52, and plasma-sprayed with zirconia. Coated dies normally were adequate for two extrusions. Die failure occurred by chipping or spalling of the ceramic coating rather than by washout of the die. Coated dies before and after extrusion are shown in Fig. 8. After use, the dies are shot-peened to remove the old zirconia and resprayed. The steel die bodies required rehardening after three to five extrusions.

3. Tungsten-30 a/o Molybdenum-30 a/o Rhenium

During the latter part of 1965, a minor effort was expended on the primary fabrication of this ternary alloy. It was feasible to extrude the ternary alloy with a stainless-steel filled billet (jacket and core) at 1100°C and extrusion ratios up to 7.4:1. The pressed and sintered ternary alloy sleeve proved easier to extrude than either tungsten or tungsten-25 w/o rhenium under the same

43275 ~ .65X
Fig. 8. Extrusion Dies Coated with ZrO_2 before and after Use. Note the chipping on the used die.

extruded tube-blank surface is shown in Fig. 9. This material was extruded at 1400°C using a molybdenum core and jacket. While this technique has received no additional effort because of concentration in other areas, it remains an attractive possibility for producing primary shapes suitable for secondary fabrication.

43082

5X

Fig. 9. Characteristic Surface Appearance of Extruded Tungsten Granules (Allied Chemical)

B. Annealing Behavior

1. Tungsten

Since primary extrusions produced tube-blanks approximately 1.59-cm OD x 0.16-cm wall thickness (0.625-in. OD x 0.062-in. wall thickness), the material was re-extruded to obtain smaller tube-blanks that could be drawn within the limited capacity of the small drawbench. In addition, it was felt that an additional extrusion and the associated "cold-working" (extrusion being at a temperature below the recrystallization temperature) might provide a material more amenable to drawing or other secondary fabrication operations.

Repeated attempts to re-extrude material from the primary extrusion resulted in catastrophic failures, even when the material was stress-relieved before re-extrusion. The only way in which sound tube-blanks could be achieved by re-extrusion was to incorporate a full recrystallization anneal after primary extrusion. Accordingly, the annealing characteristics of the various materials were studied.

The results of annealing studies on extruded tungsten tube-blanks are shown in Figs. 10 through 12. Based upon the metallographic and hardness data, annealing parameters were selected as 1 hr at 1400°C for the conventional-purity tungsten, and 15 min at 1400°C for the "high-purity" material.

2. Tungsten-25 w/o Rhenium

Based on isochronal annealing studies of as-extruded W-25Re, an anneal of 1 hr at 1500°C was selected. This treatment produced a fine-grained, recrystallized structure of low hardness. The resultant microstructure was quite uniform, with no evidence of coarsening due to secondary recrystallization. The microstructures of annealed samples are shown in Fig. 13, and the corresponding hardness profile is illustrated in Fig. 14.

Fig. 10. Annealing Behavior of a "High-purity" Pressed and Sintered Tungsten Sleeve (GE) Extruded at 1150°C and a 4.1:1 Reduction Ratio [Etchant: 5% KOH, 5% $K_3Fe(CN)_6$]

Fig. 11. Annealing Behavior of a Commercial-purity Pressed and Sintered Tungsten Sleeve (GE) Extruded at 1150°C and a 4.1:1 Reduction Ratio [Etchant: 5% KOH, 5% $K_3Fe(CN)_6$]

Fig. 12

Hardness Measurements on Conventional and "High-purity" Extruded and Annealed Tungsten Sleeves (GE)

40122 150X

As extruded
553 DPH (52 R_c)

40213 150X

15 min at 1400°C
513 DPH (50 R_c)

40214 150X

15 min at 1500°C
490 DPH (48 R_c)

40215 150X

15 min at 1600°C
444 DPH (45 R_c)

40216 150X

15 min at 1700°C
432 DPH (44 R_c)

40341 150X

1 hr at 1400°C
494 DPH (48 R_c)

40359 150X

1 hr at 1500°C
429 DPH (43 R_c)

Fig. 13. Annealing Behavior of a Pressed and Sintered W-25Re Sleeve (Chase Brass) Extruded at 1450°C and a 3.2:1 Reduction Ratio [Etchant: 5% KOH, 5% $K_3Fe(CN)_6$]

Fig. 14. Hardness Measurements on W-25Re Extruded at 1450°C and a 3.2:1 Reduction Ratio

are shown in Figs. 15 and 16 for longitudinal and transverse sections, respectively. The most striking characteristic was the apparent nucleation of recrystallized grains at the center of the individual granules. This was due to the use of a "seed" as the starting material in granule production by the Allied Chemical Company. Evidently this "seed" acted as an excellent nucleating agent, and the resultant structure was vastly different from the uniform grain size exhibited after the annealing of the pressed and sintered sleeves. Corresponding hardness profiles are shown in Fig. 17.

C. Secondary Extrusion

1. Unalloyed Tungsten

After annealing, the products of primary extrusion were canned in Type 304 SS (the original molybdenum extrusion cores were retained) and extruded at 1150°C. A 7.4:1 reduction ratio produced a nominal 0.64-cm (0.25-in.)-OD tube-blank for secondary fabrication. Jacket and core materials were removed by etching in aqua regia before secondary fabrication.

3. Tungsten-30 a/o Molybdenum-30 a/o Rhenium

An annealing study of this alloy is underway. Based upon work conducted at the Chase Brass Company, a heat treatment of 1 hr at 1500 or 1600°C will probably yield a fine-grained, recrystallized structure suitable for further fabrication. Information from the Chase Brass Company has indicated that the recrystallized state is the most ductile (in comparison with heavily stress-relieved material) for both primary fabrication by swaging and secondary fabrication by drawing.

4. Tungsten Granules

As-extruded material was annealed in vacuum at temperatures up to 2000°C. The microstructures

40332 150X

As extruded Hardness: 516 DPH

40356 150X

1 hr at 1600°C Hardness: 509 DPH

4108C 150X

1 hr at 1700°C Hardness: 446 DPH

41082 150X

1 hr at 1800°C Hardness: 417 DPH

41165 150X

1 hr at 1900°C Hardness: 410 DPH

41167 150X

1 hr at 2000°C Hardness: 391 DPH

Fig. 15. Annealing Behavior of Longitudinal Tube Sections of Extrusion-compacted (1400°C; 8.8:1 Reduction Ratio) Tungsten Granules [Etchant: 5% KOH, 5% $K_3Fe(CN)_6$]

40355 150X

As extruded Hardness: 532 DPH

40358 150X

1 hr at 1600°C Hardness: 483 DPH

41081 150X

1 hr at 1700°C Hardness: 464 DPH

41163 150X

1 hr at 1800°C Hardness: 433 DPH

41164 150X

1 hr at 1900°C Hardness: 396 DPH

41166 150X

1 hr at 2000°C Hardness: 380 DPH

Fig. 16. Annealing Behavior of Transverse Tube Sections of Extrusion-compacted (1400°C; 8.8:1 Reduction Ratio) Tungsten Granules. Transverse sections of annealed tubes are shown. [Etchant: 5% $K_3Fe(CN)_6$]

Fig. 17. Hardness Measurements on Tungsten Granules Extruded at 1400°C and an 8.8:1 Reduction Ratio

reduction ratio. Subsequent efforts to reproduce this extrusion failed, complete breakup of the W-25Re tubing being exhibited. It was necessary, therefore, to return to the higher extrusion temperature and molybdenum jackets. These conditions produced sound tube-blanks for secondary fabrication. Annealing characteristics of re-extruded tubing were similar to those of tubing after the primary extrusion.

D. Extrusion-core Removal

The most difficult problem associated with the production of sound extruded tube-blanks has been the removal of the extrusion core. To date, both the cores and jackets have been removed by dissolution in aqua regia. The reaction is slowed down appreciably by the gaseous reaction products, and core dissolution in small-diameter tubing is tediously slow. Efforts to alleviate this difficulty have been made at Nulcear Metals, Inc.,³ and elsewhere by techniques of acid drilling. Although such efforts have helped to a limited extent, there is as yet no established technique for rapid removal of a molybdenum core.

It was originally expected that a core might be found that would be amenable to elongation in tension, the accompanying reduction in area being sufficient to loosen the core. Unfortunately, experience has shown that the large degree of mechanical locking produced during primary extrusion (up to 20% of the tube-wall thickness may be interlocked with the extrusion core in random longitudinal striations) precluded any reasonable stretching method.

2. Tungsten-25 w/o Rhenium

As with unalloyed tungsten, a secondary extrusion at a reduction ratio of 7.4:1 was most satisfactory. The extrusion temperature was 1400°C, the same as in the primary extrusion. The annealed section from the primary extrusion was canned in molybdenum for re-extrusion. Once again, the jacket and core of these re-extruded products were removed in an aqua regia bath before secondary fabrication. The resulting re-extruded tube-blanks had nominal diameters of 0.64 cm (0.25 in.) and wall thicknesses of 0.064 cm (0.025 in.).

The first secondary extrusion of W-25Re attempted at ANL was accomplished with a Type 304 stainless-steel jacket and extrusion parameters of 1150°C and a 10.4:1

Two extrusion cores other than molybdenum have been used with unalloyed tungsten: Type 304 SS and TZM. The TZM alloy was too hard and led to tears in the tube-blank; the Type 304 SS core was not rigid enough to support the tube-blank during postextrusion cooling. This resulted in tube cracking, which most likely occurred when the tube was compressed by the Type 304 stainless-steel jacket during cooling. The steel jacket has a coefficient of thermal expansion approximately twice that of tungsten and causes high residual hoop (circumferential stresses to be developed in the tungsten tube-blank). Since a stainless-steel core is substantially weaker than molybdenum at elevated temperatures, it is reasonable to presume that the tube cracked as a result of the lack of internal support by the stainless-steel core.

To alleviate the difficulties associated with extrusion core removal, several extrusions of W-25Re were attempted using Nb-5Zr (D-14) cores. It was hoped that this alloy would prove amenable to removal by reaction with hydrogen gas. Typical extruded products are shown in Fig. 18. The

extrusion temperature was increased from 1350 to 1500°C to obtain a sound tube-blank. At temperatures less than 1500°C, the D-14 alloy has insufficient plastic behavior commensurate with sound tube-blank extrusions.

Micro 43192

.75X

1350°C

1400°C

1500°C

Fig. 18. Tungsten-25 w/o Rhenium Extruded at 1350, 1400, and 1500°C using Nb-5Zr (D-14) Cores

The product of the hydrogen reaction with the niobium alloy had a marked tendency to swell and led, in these initial investigations, to splitting of the tungsten-rhenium tubing during hydration. Two runs were made, one of 2 hr at 900°C and the other of 2 hr at 700°C. Reaction will be attempted in the future by directing a stream of hydrogen on the end of the tube-blank rather than by merely suspending the tube in a hydrogen atmosphere. This technique should preclude the diffusion of hydrogen through the tube wall and subsequent reaction in confined areas in the center of the tube. If the reaction can be localized at the end of the niobium-base core, the product should flake off without the damaging results obtained in the earlier experiments.

V. SECONDARY FABRICATION

Secondary fabrication involves the reduction of a tube-blank obtained by primary fabrication (extrusion in this case) to a smaller size. This size reduction may be accomplished by various methods, foremost of which are ductile-core drawing, moving-mandrel drawing, plug (or stationary mandrel)-drawing, and swaging. Ductile-core drawing involves the insertion of a ductile core (rod) into the tube before drawing. The tube and core are then reduced, much the same as a rod. The core provides internal support and helps to deter cracking and ID folding. The fundamental drawback is in the area of core removal, which can become complicated, particularly for long sections of tubing.

Moving-mandrel drawing is accomplished by inserting a rod of hard, nondeforming material into the tube. Again, both the tube and the mandrel are simultaneously drawn through a die. However, in contrast to ductile-core drawing, the mandrel does not deform and a large degree of tube-wall thinning can be realized. The difficulty of mandrel removal after drawing is analogous to that of ductile-core drawing. This technique has not been attempted at ANL.

In plug-drawing, the plug (or mandrel) is held stationary in the die. The tubing is supported on the inner surface during deformation but is pulled off the plug at the die exit. A large degree of inner surface working and improvement can be obtained by this method, along with any desired degree of wall-thinning (ironing). There are no plug-removal difficulties.

Swaging may be used either to reduce the overall tube dimensions or to point tubing prior to drawing. In work at ANL, swaging was confined primarily to pointing tubes for drawing.

A. Swaging

1. Tungsten

Tungsten swaging was carried out, for the most part on commercial material of rather low quality. Swaging temperatures were varied between 700 and 1200°C using cast Stellite hot-swaging dies patterned after dies used for hot-swaging tungsten rod. The dies featured a 32° entrance angle, a blade length twice the die diameter, and a 120° exit relief angle. Swaging was carried out over bare and jacketed tungsten with various core materials. In general, the surface quality obtained was poor. Although swaging might be considered as a satisfactory method of fabrication under certain controlled conditions, it was decided to use this method only for the pointing of tubing before drawing. Even in pointing, it was difficult to obtain satisfactory swaged points free of minor cracks that could propagate rapidly on further working. Most failures in tungsten tube drawing could be traced

back to propagation of a crack from the swaged point into the tube. The rapid crack-propagation characteristics led, in great part, to the substitution of W-25Re for pure tungsten.

2. Tungsten-25 w/o Rhenium

Progress in plug-drawing, up to the time of writing, had been slowed by difficulties in pointing. Information from Chase Brass Company indicated that a significantly higher-quality swaged point could be obtained by reducing the blade length of the dies. The modified die design is shown in Fig. 19. Further variations in die design are being investigated at ANL.

Fig. 19

Modified Design of
Hot-swaging Dies
(All dimensions in
inches)

A'	B'	C'	D'	E'	F'	G'
DIE DIA.	MAX. DIA	BLADE	DEPTH	RELIEF	RADIUS	1/2 OF
$\pm .005$	$120\% \times A'$	LENGTH	ENTRY	DIA.	$1/2 - B'$	A'
.294	.353	.175	3/32	5/8	.177	.147

B. Ductile-core Drawing

1. Tungsten

Attempts were made to draw tungsten tubing with various ductile-core materials at temperatures from 500 to 700°C. This technique proved to be extremely sensitive to the core material employed. Large differences in the thermal coefficients of expansion between the core material and tungsten tube invariably led to tube cracking, either in the predraw heating or during postdraw cooling. Drawing with a molybdenum core at a temperature of 700°C appeared most feasible, and several short lengths of target-size (0.2-cm, 0.080-in.-OD) tubing were produced in this manner.

The removal of the core presented an even more difficult task. Mechanical removal by stretching generally led to splitting of the brittle tungsten. It was found most feasible to remove the molybdenum cores by etching, severely limiting the tube length. The gaseous products evolved during etching were not removed with sufficient rapidity to consider this technique as a final method of tube production.

2. Tungsten-25 w/o Rhenium

Program requirements to date have mainly required 0.2-cm (0.080-in.)-OD material with the molybdenum extrusion core still in place. Two methods of secondary fabrication were amenable to the production of this stock. The first technique involved drawing re-extruded and annealed tubing to the final size without decoring, starting with a re-extruded 0.64-cm (0.25-in.)-OD tube-blank. The drawing temperature was 700°C. This alloy was annealed after each 30% reduction in area to prevent longitudinal cracking. As was expected, the external surfaces were smooth from the ironing action of the dies, but the internal surfaces were heavily striated. The striations (existent on as-extruded material) were not ironed by the ductile molybdenum core.

A second technique, more expensive but faster than the above, was employed periodically. It consisted of a tertiary extrusion to approximately 0.24-cm (0.095-in.) OD and drawing to the 0.20-cm (0.080-in.)-OD target size. To make this approach less costly, extrusion billets incorporated a "cluster" design in which four 0.64-cm (0.250-in.)-diam tubes (with cores) were extruded in a single molybdenum billet. This "cluster" design is shown in Fig. 20. The tube sections were heat-treated for 1 hr at 1500°C before the tertiary extrusion. A primary consideration was the introduction of ovality in the extruded product. The tubes on the perimeter of the circle were no more out of round than the centrally located tube. That is, a tube-blank extruded to a 0.229-cm (0.090-in.) nominal diameter was actually elliptical in cross section, with axes approximately 0.224 x 0.234 cm (0.088 x 0.092 in.) as an extreme case. The tubes were extruded sufficiently oversize so that subsequent drawing to the 0.20 cm (0.080-in.)-diam target size would eliminate the ovality.

Fig. 20. "Cluster" Design of Extrusion Billets for Tertiary Extrusion of W-25Re Tubing

C. Plug-drawing

1. Tungsten-25 w/o Rhenium

Plug-drawing of W-25Re was considered feasible because of the substantially lower working temperatures that had been reported for W-25Re in comparison with unalloyed tungsten.

Accordingly, draw plugs were fabricated from "Clarite" (T-1) high-speed tool-steel rods hardened to 58-62 R_C. The carbide drawing dies featured a 30° entrance angle. Plugs and dies were matched to provide

for 10% reductions in area while maintaining a diameter-to-thickness ratio (D/t) of 10:1. The drawing operations were conducted at 700°C using a resistance heating furnace. A schematic drawing of the plug-drawing arrangement is shown in Fig. 21.

Macro 43257

Fig. 21. Schematic Representation of Equipment Used during Plug-drawing

Although the working temperature of 700°C did not prove to be as low as had been anticipated, the plug-drawing technique proved feasible. Two 10% reductions by plug-drawing were sufficient to lower the striation level (or defect level as discussed further in the appendix) to less than 10% of the wall thickness.

The lower limit of plug size has not, as yet, been determined. To date, W-25Re tubing has been successfully drawn over 0.287-cm (0.113-in.)-diam plugs. At no point in this work has plug wear or softening become a problem. The plugs appear to hold up under drawing temperatures as high as 750°C, although this obviously will be a function of exposure time. Plug-drawn tubes were annealed (1 hr at 1500°C) after each 10% reduction in area. Hopefully, greater reductions can be achieved between anneals, especially at the smaller sizes. However, it is reasonable to assume that lower reductions in area between anneals will be required in plug-drawing as opposed to ductile-core drawing to avoid overloading the tube- and/or plug-holding mechanism.

2. Tungsten-30 a/o Molybdenum-30 a/o Rhenium

No work has been done on secondary fabrication of the W-30a/oMo-30a/oRe alloy. Unfortunately, this alloy has not been commercially available in high-quality form, and emphasis will be switched to the ternary alloy when drawing parameters for W-25Re are better defined.

VI. INDUSTRIAL PARTICIPATION

A. Unalloyed Tungsten

The most significant industrial contribution to the procurement of wrought, unalloyed tungsten tubing has been by the General Electric Company, Lamp Metals and Components Dept., Cleveland, Ohio. ANL purchased 7.6 m (300 in.) of 0.20-cm (0.080-in.)-OD tubing at a cost of \$5.90/cm (\$15.00/in.). The material was of high quality and had a defect level less than 10% of the wall thickness when subjected to X-ray and pulsed-eddy-current analyses.

B. Tungsten-25 w/o Rhenium

The bulk of the industrial effort on W-25Re was conducted at Nuclear Metals, Inc.³ Much of the research was of a developmental nature, very little target-size tubing being produced. NMI has relied exclusively upon extrusion as a fabricating method with only slight contributions to secondary fabrication by drawing. While good progress was made, the tubing was inconsistent in quality.

C. Tungsten-30 a/o Molybdenum-30 a/o Rhenium

Early in 1965 the Chase Brass Company supplied ANL with 3 m (10 ft) of 0.20-cm (0.080-in.)-diam wrought W-30a/oMo-30a/oRe tubing. This material was of extremely high quality, defects being less than 10% of the wall thickness in depth. Both the OD and ID surfaces were free from striations typical of most extruded products. Subsequently, ANL purchased 91 m (300 ft) of this material at \$165.00/m (\$50.00/ft).

The fabrication procedure developed at Chase Brass consisted basically of a series of draws over nondeformable drill-rod mandrels at 500°C. After each draw (a reduction in area of 5 to 10%), the material was straightened by reeling and the mandrel pulled out. The alloy was then recrystallized by annealing for 20 min at 1650°C in a hydrogen furnace, a new, smaller mandrel inserted, and the process repeated.

Unfortunately, the 91-m (300 ft) order was not of high quality. Nondestructive testing disclosed numerous cracks. In addition, there were numerous OD and ID gouges. Less than 30% of the shipment was acceptable on a basis of a 10% defect level. Photomicrographs typical of this tubing are shown in Fig. 22.

42839

200X

42840

200X

42841

200X

Fig. 22. Target-size Tubing of W/30a/oRe-30a/oMo Procured from Chase Brass

VII. DISCUSSION

During the reporting period, tungsten-alloy tubing has been fabricated by double-extrusion, followed by warm drawing to sizes in the vicinity of 0.203 cm (0.080 in.). (Work on tungsten was suspended earlier, and little new in the way of fabrication successes can be reported.)

Both primary and secondary extrusions of the W-25Re alloy have been carried out at 1400°C by the filled-billet technique with molybdenum components. Additional work on the compatibility of extrusion components, extrusion dies, and working temperature to improve the extruded tube quality would seem unwarranted. The tube-blanks currently produced were of good quality and suitable for subsequent secondary-fabrication operations. More work is required in extrusion-core removal, however, if present length restrictions are to be overcome.

Warm-drawing of the W-25Re alloy from an extruded tube-blank to the finished target size of 0.20 cm (0.080 in.) in diameter has been accomplished by ductile-core drawing with the molybdenum core from extrusion retained in place. Until core-removal methods are improved, a severe restriction is placed upon finished tube length. The smaller the finished-tube diameter, the shorter the length from which the core can be easily removed under existing practice. This technique has been used as an expedient, and it is not recommended for high-quality finished tube production since no opportunity is provided to work the internal surface and remove characteristic striations. If high-quality internal surfaces are required, a drawing technique such as moving mandrel or plug drawing is necessary.

Plug-drawing of W-25Re tubing in the range of 600 to 700°C has progressed to the point where feasibility has been established. Solutions to the pointing problem appear to be forthcoming. With this problem minimized and with a suitable tooling inventory, the plug-drawing approach should be emphasized. This method holds much promise. Working of the internal surface is necessary to eliminate striations and produce the internal surface quality ultimately desired. Although some investigators have indicated success with moving mandrel drawing, the severity of the subsequent reeling operation (to remove the mandrel) has introduced defects that presently preclude this fabrication technique, at least at small diameters.

The capability of industry to supply high-quality, small-diameter tubing of tungsten and tungsten alloys appears somewhat in doubt at present. Progress has been made in this area in the last year, but not enough. Numerous industrial efforts are being directed to this problem,⁴ but the work at this stage (end of 1965) is largely of a developmental or "best-effort" nature and some distance away from high-quality specification procurement of large quantities.

VIII. FUTURE PLANS

Future work at ANL will continue largely in the area of secondary fabrication for the W-25Re alloy until definitive processing schedules can be established and evaluation quantities of tubing material can be made available. Work on tungsten tubing will be reactivated later using some of the technologies developed for the tungsten-rhenium alloy. Effort on both the primary and secondary fabrication operations will be required. Fabrication efforts on the W-Mo-Re ternary alloy will be continued. Efforts will be concentrated on core materials and their removal at appropriate steps in the processing sequence. The secondary fabrication of this alloy is not expected to be as difficult as either the tungsten or the tungsten-rhenium alloy. It will likely follow previously developed patterns, hopefully with some simplification.

Fabrication economics will eventually be considered. Simplified and/or improved fabrication processes could significantly reduce costs with no compromise in product quality.

APPENDIX

Product Evaluation

Product evaluation by nondestructive testing has seen great progress during the reporting period. Radiographic, pulsed-eddy-current, and ultrasonic techniques have all been employed.⁵

The starting sleeve, 3.18-cm OD x 2.54-cm ID x 10.2 cm long (1.25-in. OD x 1-in. ID x 4 in. long), a pressed and sintered powder product (~94% of theoretical density), has presented some problems in nondestructive testing. Visual and radiographic means have been used with satisfactory results. Ultrasonic techniques have not proven feasible for this material for reasons that are not yet fully understood. The minute voids in the sintered product may cause the problem, but more work will be required to resolve the difficulties. Generally, a small ring section from one end of the as-sintered sleeve is provided by the vendor, and metallographic studies on this section have provided necessary data on the size, shape, and distribution of metallographic phases. Cracks or other defects in the sleeve are detected by visual means (or other penetrant techniques). Such large cracks would generally be detected by the radiographic inspection procedure. In general, the pressed and sintered sleeves have been of excellent quality.

The extruded tube-blanks resulting from either primary or secondary extrusion also presented problems for the nondestructive-test specialist. The 0.16-cm (0.062-in.) wall thickness of the primary extrusion proved too thick for the pulsed-eddy-current method, which is generally restricted to approximately 0.064 cm (0.025 in.). The surfaces, both external and internal, contained extrusion striations that made application of the ultrasonic method difficult.

In the early stages of process development, considerable effort was expended to suitably prepare extruded tube-blanks in a manner that would permit ultrasonic as well as other means of definitive testing. By ultrasonic, radiographic, and visual means, the primary-extrusion operation was judged acceptable. Primary extrusions were examined only periodically once the quality was established.

The above-mentioned difficulties in nondestructive testing of the primary extrusion were, in some cases, compounded by the presence of the extrusion core. With the core in place it was practically impossible to determine the nature of the internal surface by radiographic or ultrasonic analysis.

After secondary extrusion to a tube size of approximately 0.64-cm OD x 0.064-cm wall (0.25-in. OD x 0.025-in. wall), the tube quality could be examined in a definitive manner by pulsed-eddy-current techniques. The extrusion striations present on extruded products presented some problems

in both eddy-current and radiographic inspection and made quality-control interpretations complicated. The defect level as determined by these two means was between 10 and 20% of the wall thickness. These so-called defects were the longitudinal striations--not cracks, tears, or sharp notches. With the existence of such a surface condition it became difficult to determine whether there were other real defects (cracks, tears, etc.) that might cause later problems in subsequent fabrication operations.

The striations on the external surface were easily removed by belt-sanding. The internal surface was difficult to condition except by further working operations such as plug or mandrel drawing. Consequently the same procedure was utilized at this stage as after the primary-extrusion operation. Typical tubing was suitably surface-conditioned so that nondestructive testing could indicate that no heavy wall defects (other than the striations) were being generated by the secondary-extrusion operation.

In subsequent secondary-fabrication operations, the striations on the external surface were further reduced by drawing. The internal surface was improved through the use of plugs or mandrels, the former being favored. The use of a ductile core provided little internal surface improvement. Processed tubing was subjected to both radiographic and pulsed-eddy-current inspection. The radiographic sensitivity was good, but its results were confirmed by pulsed-eddy-current testing. The radiographic inspection proved invaluable for the detection of gradual changes in wall thickness, porosity, large cracks, and remaining extrusion striations.

For finish-drawn tubing with reasonably smooth surfaces, the pulsed-eddy-current technique easily spotted defects both above and below the 10% level (10% of the wall thickness). In the case of a 0.020-cm (0.008-in.) wall tube of small OD, this represented a defect level of 0.002 cm (0.0008 in.) or less.

Generally speaking, nondestructive-testing techniques satisfactorily determined the quality of the finished tubing products to prescribed levels such as 10% of the wall thickness. During the reporting period, finished tubing of this quality level was produced. Process development has aimed at this level, but in the early stages it was unrealistic to expect that all tubing produced will meet this criterion.

With the nondestructive methods well established, it now remains to improve the processing techniques so that a high-quality product can be produced routinely.

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