TANTALUM ALLOY TUBING DEVELOPMENT PROGRAM

F. S. Turner

FOREWORD

This final report covers the work performed under Contract AF 33 (657)-11261 from 1 July 1963 to 31 March 1967. The manuscript was released by the author on 31 August 1967 for publication as an AFML Technical Report.

This contract with Allegheny Ludlum Steel Corporation, Brackenridge, Pennsylvania, was initiated under Manufacturing Methods Project 8-108, "Tantalum Alloy Tubing Development Program". It was accomplished under the technical direction of Mr. Kenneth L. Love of the Metallurgical Processing Branch, MATB, Manufacturing Technology Division, Air Force Materials Laboratory, Wright-Patterson Air Force Base, Ohio.

F. S. Turner, Research Metallurgist, Extruded Products Research and Technology Section, Research Center, Allegheny Ludlum Steel Corporation was responsible for the execution of the work and preparation of this report. Others who cooperated in the effort were: E. G. Flynn, Supervising Metallurgist in charge of the Extruded Products and Research and Technology Section, and P. A. Santoli, Senior Research Metallurgist, Extruded Products Research and Technology Section. The major efforts during Phases III and IV of the program were carried out at Superior Tube Company. Mr. L. C. Shaheen, Development Metallurgist, was responsible for the work performed at Superior Tube Company, and is a major contributor to the report. The elevated temperature tensile and stress rupture testing was conducted at Metcut Research Associates in Cincinnati, Ohio, under the supervision of William J. Stross, Assistant Supervisor, Materials Testing Laboratory.

This project has been accomplished as a part of the Air Force Manufacturing Methods Program. The primary objective of the Air Force Manufacturing Methods Program is to develop, on a timely basis, the manufacturing processes, techniques and equipment for use in economical production of USAF materials and components. This program encompasses the following technical areas:

Metallurgy - Rolling, Forging, Extruding, Casting, Fiber, Powder.
Chemical - Propellant, Coating, Ceramic, Graphite, Nonmetallics.
Fabrication- Forming, Material Removal, Joining, Components.
Electronics- Solid State, Materials and Special Techniques, Thermionics.

Suggestions concerning additional Manufacturing Methods development required on this or other subjects will be appreciated.

This technical report has been reviewed and is approved.

CHARLES H. NELSON, Acting Chief
Manufacturing Technology Division
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ABSTRACT

Tantalum Alloy Tubing Development Program

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A process has been developed to produce tantalum alloy tubing in two alloys, Ta-10W and T-222 (Ta-9.6W-2.4HF-.01C) to each of three sizes; .500-inch OD by .062-inch wall, .375-inch OD by .062-inch wall, and .250-inch OD by .020-inch wall. The extrusion method was selected as the most economical and practical approach to producing tube hollows for subsequent tube reducing and drawing. Both the Ta-10W and T-222 alloys were extruded to 2.062-inch OD by 1.440-inch ID tube hollows from a 5-inch liner system on a 2200-ton extrusion press. Short billets of the Ta-10W alloy were successfully extruded unclad from a temperature of 2800F. Because of the additional frictional forces present, a temperature of 3100F was required for longer billets. Because initial extrusions of the T-222 alloy indicated that this alloy required considerably more pressure to extrude, a molybdenum cladding technique was utilized to reduce pressures and enable us to extrude from lower temperatures. Excellent extrusions of the T-222 alloy were made using this technique from a temperature of 3000F. The main problem is in post extrusion conditioning. The tubes were roll straightened cold and conditioned by OD centerless grinding and ID boring. Many stress corrosion type cracks developed during this stage of processing. The material should be annealed or at least stress relieved between extrusion and the various conditioning operations to avoid stress cracking and extreme care should be taken in selecting coolants and lubricants.

The conditioned tube hollows were then tube reduced to 7/8-inch OD by .109-inch wall in two steps and then cold drawn to each of the finished sizes in each of five temper conditions; 1/4 hard, 1/2 hard, full hard, full hard + stress relieved, and fully annealed. Both alloys displayed good drawability with area reductions up to 62 percent without intermediate annealing. It was demonstrated that the T-222 alloy can be processed through the same schedule as Ta-10W and provide comparable results in the finished tubing. The tubing was evaluated in terms of all the routine non-destructive and destructive tests. Flaring, flattening, bend and tensile tests indicated that both alloys exhibit good ductility for their respective strength levels even with some degree of cold work. Extensive tensile and stress rupture tests were conducted at temperatures of 2100F, 2400F and 2700F. In both alloys the best combination of strength and ductility at 2400F was obtained by material in the stress relieved condition. The properties obtained for stress relieved Ta-10W were ultimate tensile strength 45 to 49 ksi, .2% yield strength 27 to 37 ksi, % elongation 20 to 25. The properties obtained for the stress relieved T-222 alloy were ultimate tensile strength 56 to 63, .2% yield strength 44 to 50 ksi, % elongation 30 to 79. In general, the strengths obtained with the T-222 alloy were 15 to 20 ksi higher than the Ta-10W, while elongations are comparable. Tubing from both alloys was successfully welded in an inert atmosphere using a tungsten electrode. While strengths were comparable to those obtained on the base metal, ductility of the welds was considerably lower than that of the base metal.

To demonstrate reproducibility of the process, a second lot of tubing in each alloy was processed to each of the three finished sizes and finished in the stress relieved condition. The tubing produced during this phase was checked for dimensional tolerances and spot checked for elevated temperature tensile and stress rupture properties at 2400F. Excellent correlation was obtained with the properties obtained on the test lot sequence tubes.

This abstract is subject to special export controls and each transmittal to foreign governments or foreign nationals may be made only with prior approval of the Manufacturing Technology Division, MATB, Air Force Materials Laboratory, Wright-Patterson Air Force Base, Ohio 45433.

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SECTION I

INTRODUCTION

One of the major components of advance nuclear aerospace units is tubing of a satisfactory quality to meet power generation requirements involving liquid metals. Large quantities of small diameter thin wall tubing are required in the boilers, radiators and turbine nozzles in both nuclear propulsion and nuclear auxiliary power units. The size and weight of the radiators varies inversely as the fourth power of the radiator temperature, so that small gains in temperature can result in large weight reductions. Consequently, tantalum alloys, by virtue of their excellent elevated temperature properties and their unique corrosion resistance to molten alkali metals, have considerable potential for use in this application.

Recognizing this potential, the Aeronautical Systems Division of the United States Air Force awarded to Allegheny Ludlum Steel Corporation, Contract AF 33(657)-11261, a "Tantalum Alloy Tubing Development Program." The specific objective of the program is to provide processing mechanisms and sequences for the production of defect-free, fabricable and weldable tantalum alloy tubing displaying improved high strength and corrosion resistance characteristics. The program involves four chronological phases:

Phase I - State-of-the-Art Analysis

Phase II - Development of Tube Hollows

Phase III - Development of Tube Production Process

Phase IV - Production of Tubing

This report is the final report on this program and summarizes the efforts under all four phases. During the program tubing was produced in both the Ta-10W and T-222 (Ta-9.6W-2.4Hf-.01C) alloys to each of three sizes: .500-inch OD by .062-inch wall, .375-inch OD by .062-inch wall and .250-inch OD by .020-inch wall.

The tube reducing and cold drawing was conducted at Superior Tube Company. The elevated temperature tensile and stress rupture tests were conducted at Metcut Research Associates.

SECTION II

Phase I - State-of-the-Art Analysis

A. Introduction

Under Phase I of the program a comprehensive State-of-the-Art Analysis was conducted. Since items such as raw materials, ingot consolidation, initial ingot breakdown, etc., were thoroughly covered in the State-of-the-Art Analysis conducted for previous contracts involving tantalum alloys(1),(2), it was requested by the Air Force personnel that special emphasis be placed on applications for tantalum alloy tubing and the metallurgical problems and difficulties unique to these applications. However, since the optimum factors in tube production are closely interrelated and a selection of one cannot be made without careful consideration of its influence on all the other factors, the survey was made as comprehensive as possible.

In conducting the survey use was made of a questionnaire, personal visits or contacts and an extensive search of both the domestic and foreign literature.

The questionnaire (see Appendix A), which consisted of the following sections:

Section I - Interests, Applications and Requirements for Tantalum Alloy Tubing

Section II - Raw Materials

Section III - Consolidation of Raw Materials

Section IV - Melting Techniques

Section V - Ingot or Pressed and Sintered Billet Evaluation

Section VI - Initial Ingot Breakdown

Section VII - Post Extrusion Operations

Section VIII - Tantalum Alloy Data Sheets

was mailed to 108 industrial concerns, research organizations and Government agencies known or believed to have applications for or experience in the production of tantalum alloy tubing. Sixty replies were received, but only 22 of those contained useful information.

The American Society for Metals, Information Searching Service, was contracted to perform a retrospective complete file search for the period back to 1958. A total of 67 abstracts were obtained from this search.

A manual search was made of the period prior to 1958 but no significant references were located. The foreign technology phase of the analysis was conducted by World Progress in Engineering, Incorporated.

B. Interests, Applications and Requirements

The survey indicated that only a few organizations had current needs for tantalum alloy tubing and these were for small amounts for standard items such as heating susceptors and heat exchanger tubing. Besides these common items most of the present requirements are experimental in nature and are used in various tests assimilating future applications. The greatest potential application for tantalum alloy tubing is in advanced nuclear aerospace propulsion and power generation systems, especially in the nuclear auxiliary power generation systems involving liquid metals.

Our space vehicles currently get their power from solar or chemical batteries which are neither light weight nor inexpensive. Obviously, because of the great cost and difficulty of launch, a basic parameter for evaluation and comparison for any component to be used in space is its weight. Figure 1 illustrates the specific weight of various power generation systems in pounds per kilowatt as a function of electrical power output in kilowatts. This figure clearly shows that if we are to achieve maximum power output at minimum specific weight for future spacecraft,emphasis should be on the reactor turbo-generator system.

Two reactor turbine generator systems under consideration for the more advanced power plants are the Rankine cycle and the Brayton cycle. The Rankine cycle is a standard turbine generator system in which heat is generated in a compact nuclear reactor. In this system the water normally used as the cycle working fluid is replaced by the more efficient liquid alkali metal. The liquid metal is heated by the reactor, is converted into a vapor which is used to drive the turbine which in turn drives a generator to produce the electrical power required. The vapor is then condensed to a liquid and pumped back to the reactor. Since, in space, cycle waste heat must be reflected by radiation, a radiator must be used in conjunction with the condenser. The most important consideration in selecting metals for components of this system is their compatibility with the liquid metals used as the cycle fluid. The refractory metals, by virtue of their unusual corrosion resistance to molten alkali metals, will be required for this application. Maximum temperatures will vary between 1800F-2400F and stress will be in the neighborhood of 6000 psi.

The Brayton cycle uses a neutral gas such as helium, argon or neon rather than a liquid alkali metal as the cycle fluid. In this system the temperature of the gas going into the turbine is about 2500F. The peak temperatures in the radiator will be about 2000F to 2200F. While the Brayton cycle eliminates the corrosion problem, the size of the radiator required is about ten times that required for the Rankine cycle.

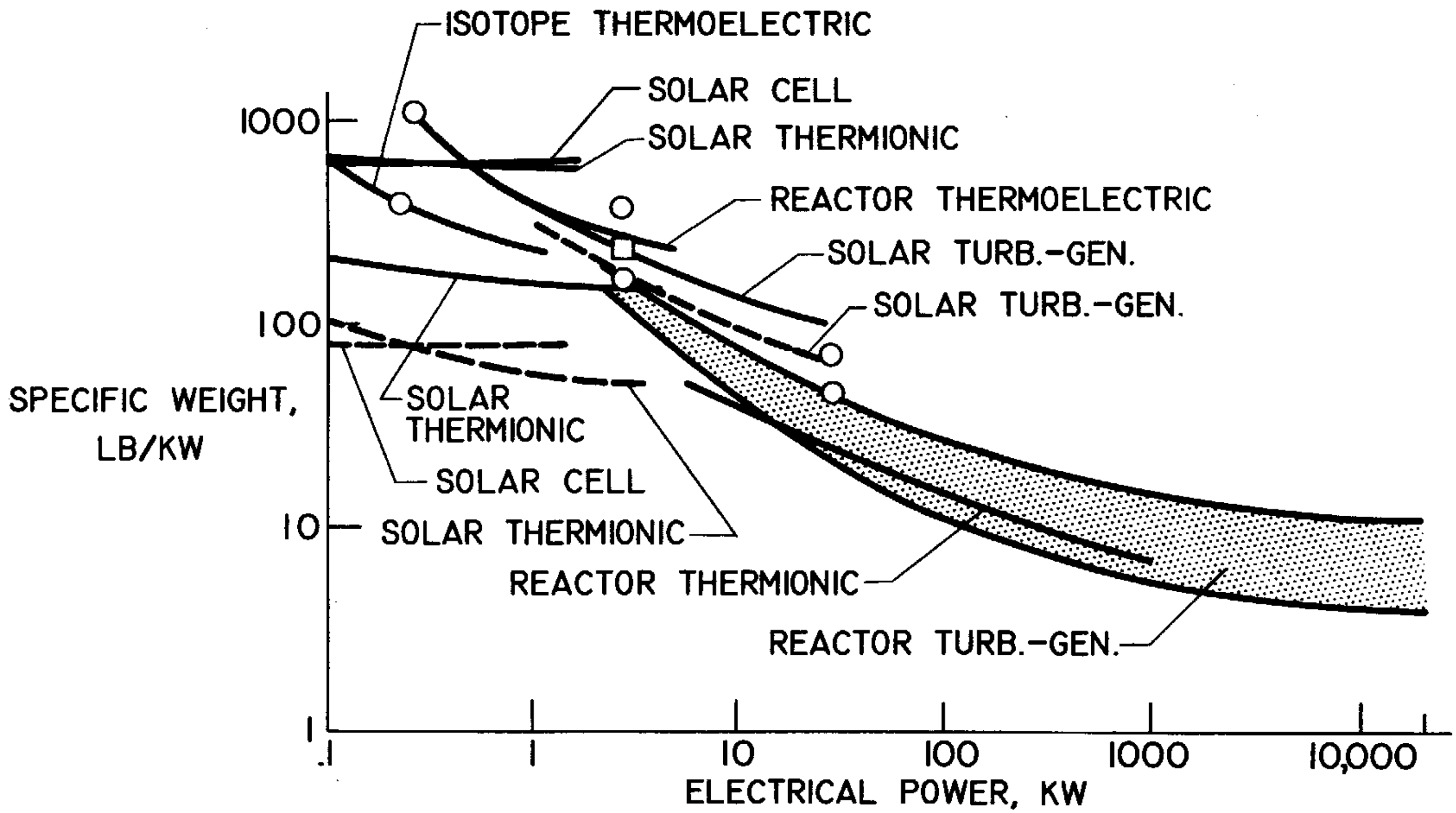


Figure 1

Estimated specific weights of power generator systems

(Courtesy of NASA)

In reactor turbine generator systems the radiator alone accounts for 40 to 60 percent of the total weight. Figure 2 is a schematic drawing of a manned vehicle of this general type. Large quantities of small diameter thin wall tubing are required in the boilers and radiators. Ault(3) estimates that for a 1000 kilowatt system,the boiler, if made of 1/4-inch diameter tubing, would require 3300 feet of tubing and the radiator, if likewise made of 1/4-inch diameter tubing, would require about 55,000 feet. Since the size of the radiator varies inversely as the fourth power of the radiator temperature, operating temperatures must be raised to reduce specific weights if greater powers are to be realized from these systems. It is at the higher temperatures that tantalum alloys would have the advantage over all other materials under consideration.

Tantalum alloy tubing also has found applications as "thimbles" or capsules for the molten plutonium fuel in Lamprey I fast reactor experimental core facility at Los Alamos Scientific Laboratory. This reactor is part of a program to determine under what conditions and forms plutonium can be used as fuels in power reactors. Another application listed was as thermionic diode material in a thermionic converter system. In order to produce comparable power output the thermionic systems must operate at higher temperatures than the turbine-generator system. At the temperatures required it may be necessary to employ a liquid metal cooling loop to extract heat from the anode and carry it to a radiator. The Jet Propulsion Laboratory of California Institute of Technology reports that if this liquid metal cooling system is necessary,then large quantities of tantalum alloy tubing could be required.

Small quantities of tantalum alloy tubing are also required in the chemical industry for fluid transfer and in conventional shell and tube type heat exchangers and, more particularly, as bayonet heaters.

C. Availability and Property Requirements

At the time the survey was conducted at least three producers listed tantalum alloy tubing as items in their commercial product line. However, only experimental quantities had actually been produced and insufficient data was available on its properties and behavior to adequately judge its quality. The users also reported wide differences in the quality of commercial tubing from batch to batch and from fabricator to fabricator. They reported that they had received tubing of excellent quality from one vendor and the next shipment from the same vendor was of inferior quality. The defects present were, for the most part, laps and seams and small trapped impurities found on the OD and ID surfaces of the tubing.

Obviously, from the nature of the applications mentioned previously, the most important property of the tantalum alloy tubing is corrosion resistance to its service environment, whether it be liquid alkali metals, liquid plutonium, acid salts, or rocket engine exhaust products. Other important properties, as cited in the replies to the questionnaire, are

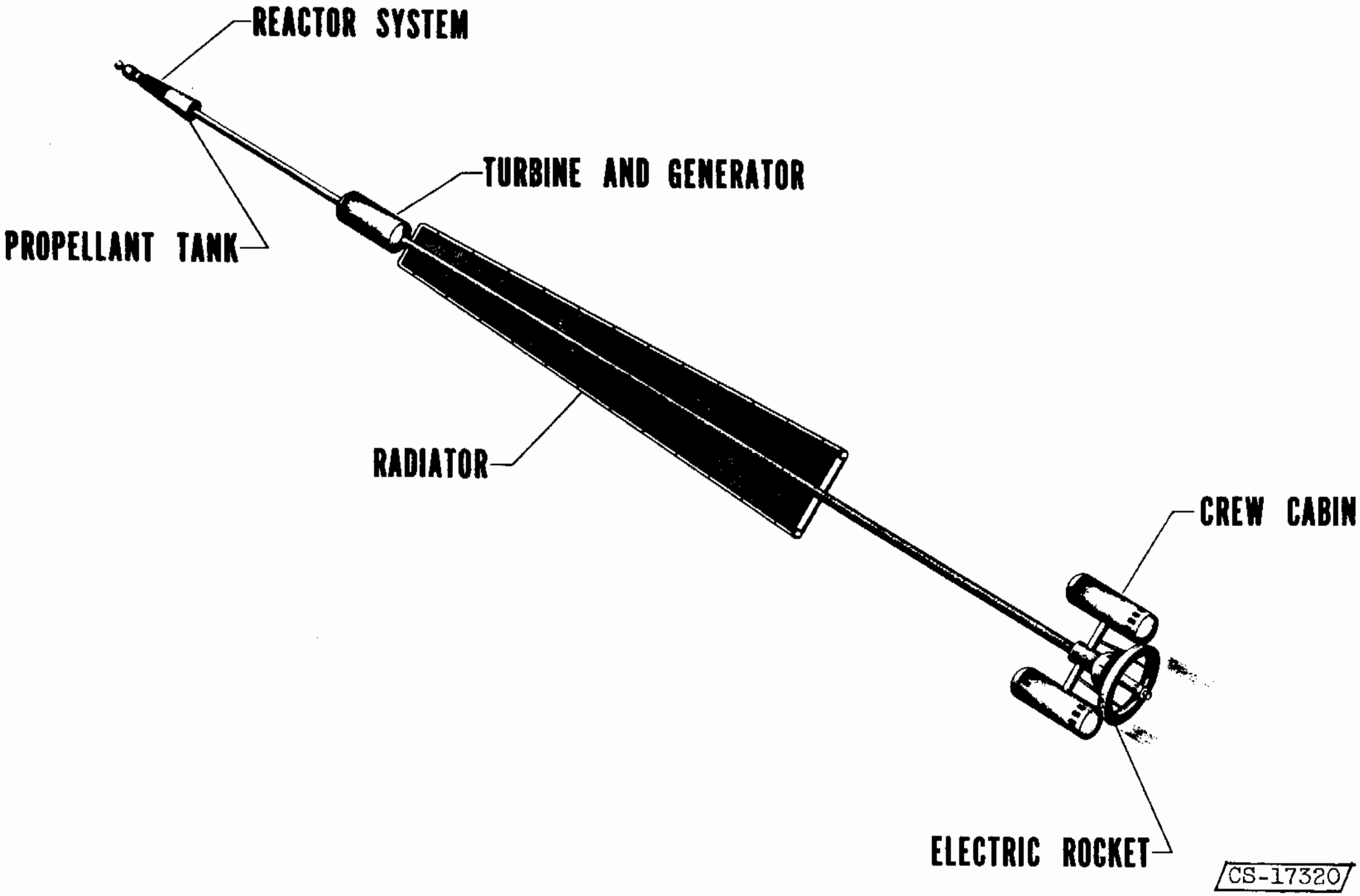


Figure 2

Electric Space Vehicle

(Courtesy of NASA)

bendability, weldability, and metallurgical stability over a period of years at temperatures between 2000F-2200F. Since it is believed that tubing will fail by bulging or creep longitudinally rather than fracturing, 10,000 hour creep data is also required.

The main difficulty in the production of tantalum alloy tubing is in obtaining high quality tube hollows. Of the many methods of obtaining tube hollows, direct extrusion offers the most economical and practical means. For this reason, our program concentrated on the extrusion method of producing high quality tube hollows.

SECTION III

Phase II - Development of Tube Hollows

A. Introduction

Under Phase II of the original contract Allegheny Ludlum was to develop the direct extrusion method of producing high quality tube hollows in two alloys, Ta-10W and Ta-30Cb-7.5V. However, in December of 1963, a supplemental agreement was received from the Air Force which modified the contract by substituting the experimental alloy T-222 (Ta-9.6W-2.4 Hf-.01C) for the Ta-30Cb-7.5V alloy. It was our intention to extrude a 2.060-inch OD by 1.440-inch ID tube hollow from a 5-inch diameter liner system. This allows .030-inch on a side for clean-up to the 2-inch OD by .250-inch wall desired as a starting size for tube reducing. Two 6-inch long and three 11-inch long billets of each alloy were extruded. Because of the very limited number of extrusions being made on this contract, there was no allowance for experimentation and the general extrusion technology such as minor tooling design, lubrication, speed, etc., was essentially the best that was developed on our previous tantalum alloy extrusion development program, Contract AF 33(600)-42396.

B. Tantalum Alloy Extrusion Billet Material

Since melting and fabrication of extrusion billets was beyond the scope of this program, and since reliable technology to produce sound and reproducible ingots had been developed on previous government contracts(2),(3) wrought, fully machined extrusion billets were ordered from Wah Chang Corporation. Two 6-inch long and three 11-inch long billets were ordered in each alloy. The billets were machined to $4.850 {}^{+.015}_{-.000}$-inch OD by $1.550 {}^{+.000}_{-.015}$-inch ID with a 1-inch nose radius on one end and a 63 RMS or better surface finish. In addition, the billets were ordered to conform to the following specification:

1. Certified billet soundness in accordance with the ultrasonic procedure described in Ordnance Specification OS-9426-A, Acceptance Level 1.
2. Billet concentricity ± 3 percent.
3. Free of injurious defects such as seams, pipes, cracks, scale, nonmetallic inclusions and segregation which adversely affect extrudability.
4. Billet ends checked by dye penetrant method.

5. Certified chemical analyses on turnings from the end face of one billet from each heat, with spot checks on other faces.

Chemical Specifications

	Ta-10W	T-222
W	9-11%	9-10%
Hf		2.0-2.8%
C	50 ppm max.	75-150 ppm
O	75 ppm max.	50 ppm max.
N	50 ppm max.	50 ppm max.
H		10 ppm max.

6. Spectrochemical analysis for trace element to be run on ingot.

7. Recrystallized microstructure with uniformly fine grain size, preferably equal to or smaller than ASTM No. 3.

1. Ta-10W Alloy

All five billets of Ta-10W alloy were obtained from Heat Number 62026 - Ta-10W - S3B. This heat was double electron-beam melted followed by consumable arc melting into an 8-8-1/2-inch diameter water cooled copper crucible. Chemical analyses runs on samples from the top and bottom portions of this heat are presented in Table 1. The hardness was in the range of 192-207 BHN with an average of 199 BHN.

The conditioned ingot was forged hot from a temperature of 2200F to a 5-1/4-inch diameter round. The forged billet was then given a recrystallization anneal of four hours at 2800F. The billet was then cut and machined into finished extrusion billets. A photograph of one of the 11-inch long billets is shown in Figure 3. No visible defects were revealed by dye penetrant tests and metallographic examination revealed a grain size of ASTM 3.5.

The extrusion billets were ultrasonically inspected at Allegheny Ludlum by the contact method using a 5 MC barium titanate crystal. Standardization was on a 2-inch peak to peak using a 3-0300 block (3/64-inch defect). The five billets were completely inspected around the periphery and length. No ultrasonic indications were noted.

TABLE 1

Chemical Analysis
Tantalum-10 Tungsten Ingot*
Heat Number 62026 - Ta-10W-S3B

Composition in Percent

Element	Top	Bottom
Ta	Balance	Balance
W	9.5	9.4

Impurity Content ppm

Element	Top	Bottom
Al	<20	<20
C	<30	<30
Cb	680	840
Cd	< 5	< 5
Co	<10	<10
Cr	<20	<20
Cu	<40	<40
Fe	<40	<40
H	2.5	2.7
Mg	< 20	< 20
Mn	< 20	< 20
Mo	200	200
N	35	55
Ni	< 20	< 20
O	< 50	<50
Pb	< 20	< 20
Si	<40	< 40
Sn	<20	< 20
Ti	< 40	< 40
V	< 20	< 20

*As Certified by
Wah Chang Corporation.

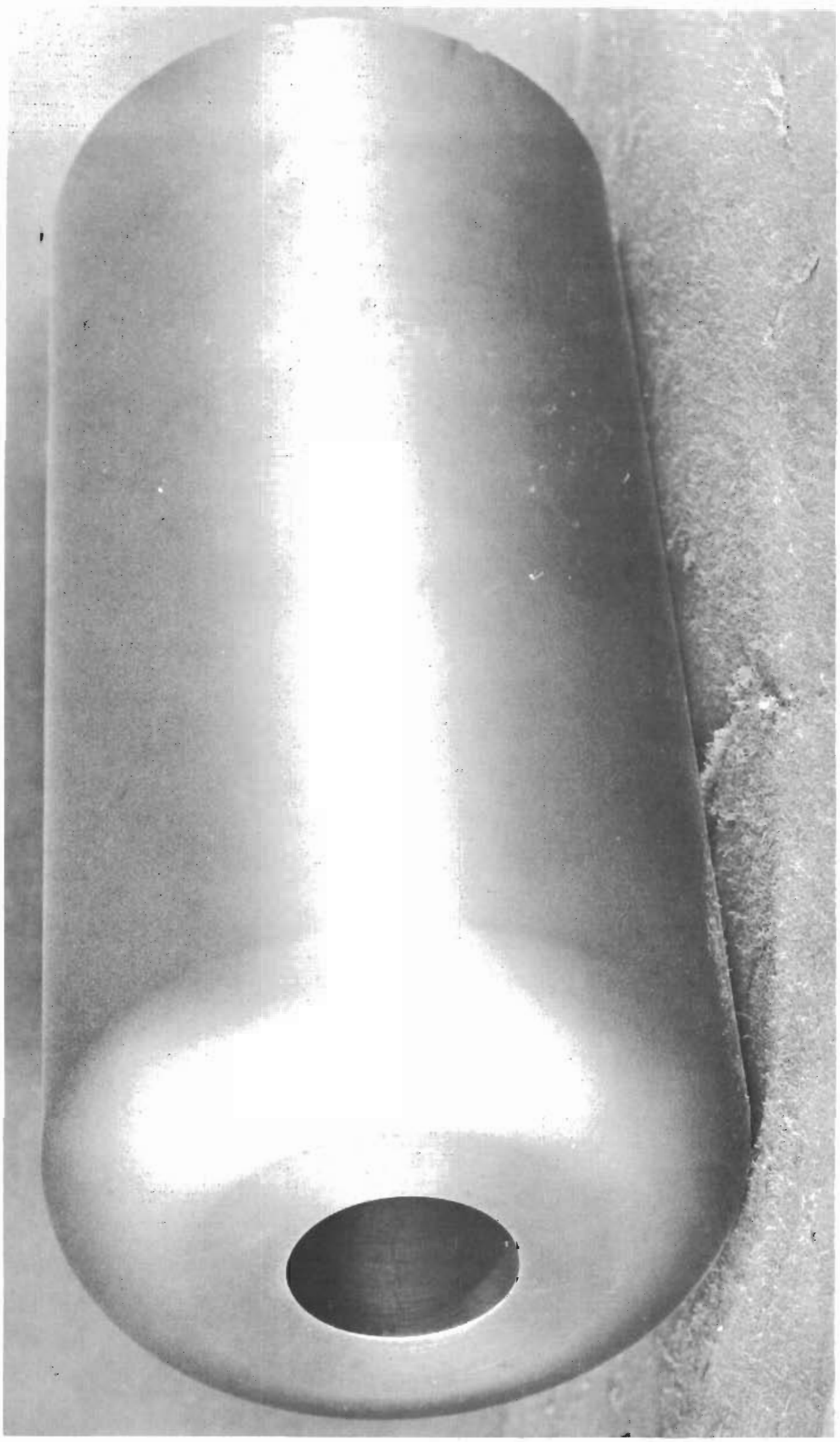


Figure 3

Ta-10W Extrusion Billet 4.850-inch O.D. by 1.55-inch I.D. by 11-inch long

2. T-222 Alloy

At the time the T-222 alloy was inserted into this program only one 3-inch diameter, 22-pound billet of the T-222 had been melted. Wah Chang Corporation encountered some difficulty in scale-up melting the T-222 alloy. The first ingot produced for use on this program was off analysis and had to be remelted. The 8-inch diameter remelt ingot (Heat No. 65032) was found to be ultrasonically sound and the chemical analysis reported by Wah Chang is shown in Table 2. All of the T-222 billets used on this program were from this heat. The ingot hardness was in the range of 248-269 BHN with an average of 260 BHN. Initial breakdown of the cast structure was accomplished by extrusion from a 9-1/4-inch diameter liner on the Canton Drop Forge and Manufacturing Company's 5,500-ton press. The 8.4-inch diameter billets were canned in low carbon steel and heated in a salt bath for one hour at 2300F prior to extrusion. Following extrusion the billets were press forged to 5-1/8-inch diameter, cut and machined into finished hollow extrusion blanks 4.850-inch OD by 1.550-inch ID. These billets were then given a vacuum anneal of four hours at 2800F at Fansteel Metallurgical Corporation. The finished billets were received at Allegheny Ludlum Steel Corporation on July 7, 1964. Upon receipt they were examined ultrasonically using the same procedure used on the Ta-10W billets. No ultrasonic indications were detected.

A 1/4-inch thick disc of material taken from the same heat and processed fully with the billets was supplied for metallographic examination. Figure 4 is a photograph of the macroetched disc. Metallographic examination revealed a partially recrystallized microstructure. A hardness traverse was made on this disc. Hardnesses ranged betwen 272 and 276 VHN with an average of 274 VHN. Ingot hardness as reported by Wah Chang was 260 BHN which is equivalent to 274 VHN.

C. Extrusion Tooling

The extrusions were performed on a Lake Erie production press of a modified Schloeman design. The press is rated at 2200 tons. For this program a 5-inch diameter liner system was designed which is essentially a scale-up of the 3-7/8-inch liner tooling system used on previous Air Force refractory metal extrusion programs at Allegheny Ludlum. This tooling arrangement is shown in Figure 5, along with the principal dimensions of each item. The full capacity of 2200 tons was utilized to give, in effect, a liner pressure of 224,000 psi. For the extrusion of the tube hollow, a unit billet pressure of 246,000 psi was in effect at the maximum liner capacity. The extrusion of the 2.060-inch OD by 1.550-inch ID tube hollow from this 5-inch diameter liner system is a reduction ratio of 10.6:1. From this relatively high reduction ratio in association with this unique, high performance tooling system, extrusion lengths in excess of ten feet were possible. As compared to the previous methods of obtaining tube hollows such as trepanning or drilling bar stock, obvious economic advantages can be realized with this capability.

TABLE 2

Chemical Analysis
T-222 Ingot*
Heat Number 65032 - Ta-9W-2.5Hf-0.01C

Composition in Percent

Element	Top	Bottom
Ta	Balance	Balance
W	9.4	9.4
Hf	2.7	1.9
C	0.14	0.13

Impurity Content ppm

Element	Top	Bottom
Al	< 10	< 10
Cb	< 300	< 300
Co	< 5	< 5
Cr	< 10	< 10
Cu	< 20	< 20
Fe	< 20	20
H	2.6	1.9
Mo	< 10	< 10
N	17	17
Ni	< 10	< 10
O	< 50	< 50
Si	20	20
Ti	< 20	< 20
V	40	15

*As Certified by
Wah Chang Corporation

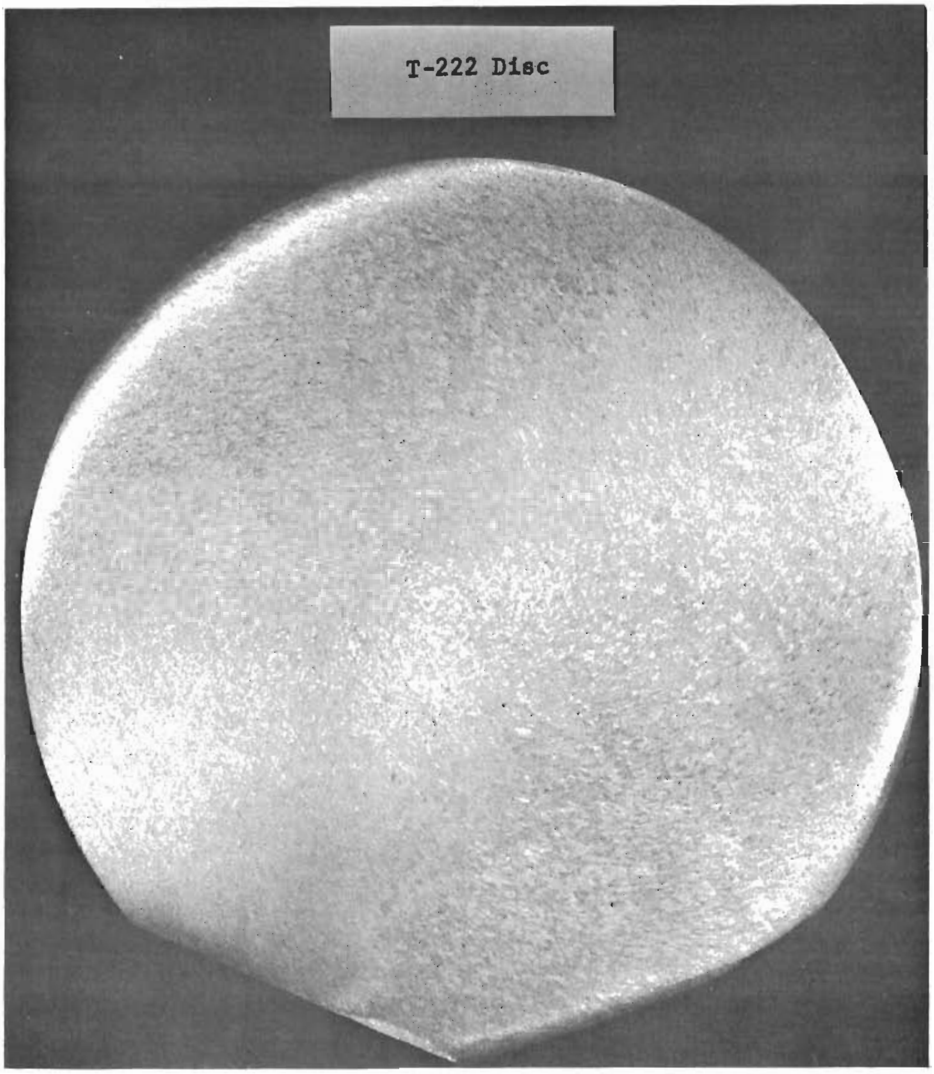
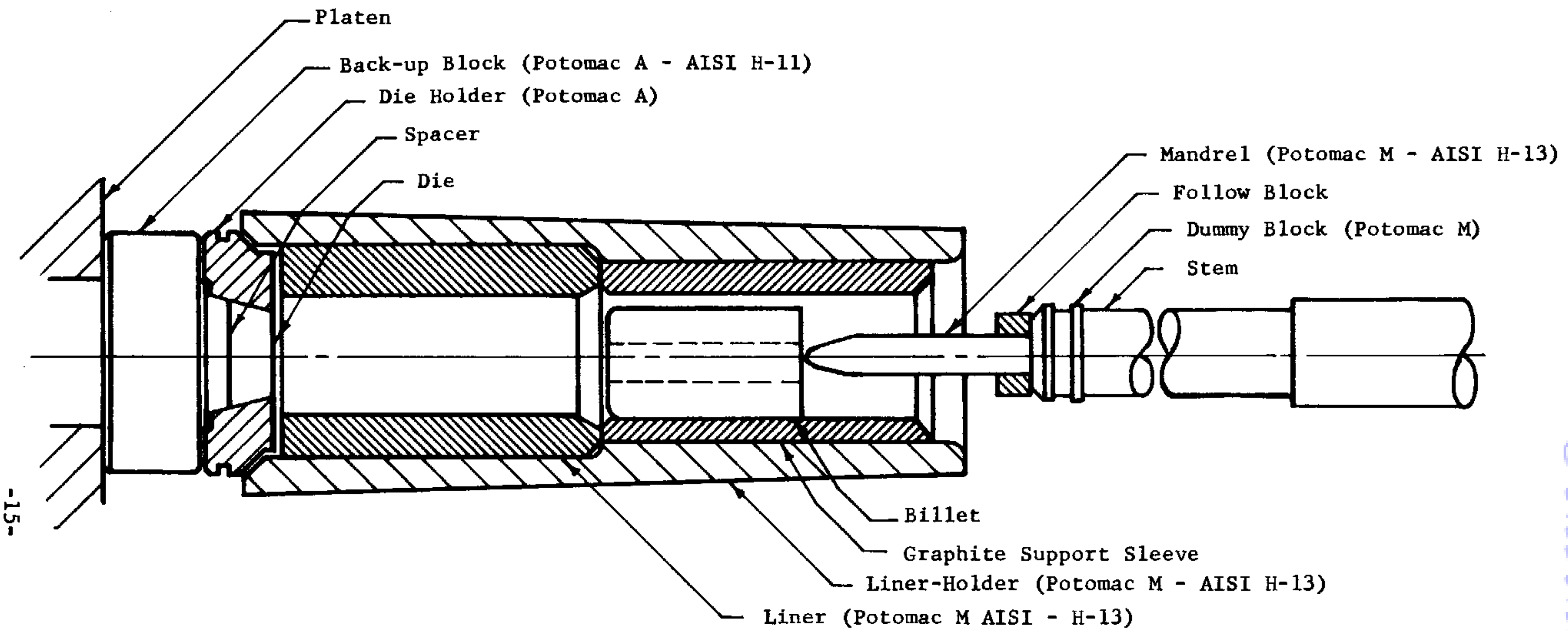


Figure 4

1X Macroetched Disc of T-222 Alloy



Name	NOMINAL SIZE O.D.*	 I.D.*	Length*
Backup Block	10	2-1/2	4-3/8
Die Holder	10.020	5.375 min.-6.360 max.	3
Stem	4.900 min.-5.625	--	35-7/8
Dummy Block	4.980	--	2-1/4
Liner Holder	10.020 min.-11.538 max.	7-1/2 min.-8-1/2 max.	29
Liner	8-1/2	5	15.020

*Dimensions given in inches.

Figure 5

Tooling Arrangement for the Extrusion of Tube Hollows

1. Liner Design and Materials

A liner, liner-holder assembly was used for this program. The inner, working liner was constructed of Potomac M AISI H-13 alloy heat treated to Rockwell C 53-56. The 15-inch long liner was assembled into the liner holder with an interference fit of 0.010-inch. The liner-holder was made of AISI H-13 alloy heat treated to Rockwell C 48. A graphite support sleeve was utilized in the back end of the liner-holder to support the billet and facilitate easier billet loading.

The liner, liner-holder assembly was placed in the container on a taper with an included angle of three degrees. The container is made of SAE 6145 steel heat treated to a hardness level of Brinell 245-275.

The container temperature was maintained at 500F primarily to avoid excessive loss of strength in the tooling components.

2. Stem Design and Materials

For this program a special stem was designed which could withstand an applied compressive stress of up to 240,000 psi, which relates to a liner pressure of 224,000 psi. The stem was constructed so that the high stress (240,000 psi) portion of the stem would be confined within the liner-holder. The stress in the larger diameter back end of the stem was below 200,000 psi. A cast brass ring, riding on the back end of the stem, seats against the container during extrusion in order to prevent stem fragments from coming out in the event of an explosive type failure. The stem was fabricated from prime, freckle-free, consumable electrode vacuum melted Potomac M (H-13). A special heat treatment was used to give the high stress portions considerably more strength than normally provided for stems.

3. Minor Tooling Design

The dies used had a 90-degree included angle conical entry with a constant entry radius of 5/16-inch and a constant land width of 1/4-inch. The dies were made of consumable electrode vacuum melted Potomac M (AISI H-13) hot work tool steel heat treated to Rockwell C 48-50. The ID surface of the dies were coated with a thermal barrier of zirconia. Detail drawings of the die design before and after coating are shown in Figures 6 and 7.

Three mandrel designs were used: 1) a conventional straight mandrel, 2) a stepped mandrel, and 3) a modified stepped mandrel. The stepped mandrel was of a design such that the front portion of the mandrel was of a diameter considerably less than the main working length. This, in effect, would mean that the first few inches of the extrusion would be of a lower extrusion ratio than the rest and therefore should require less pressure at the start of extrusion. The modified stepped mandrel is essentially a straight mandrel designed to a specific length such that the billet would start to extrude on the radius.

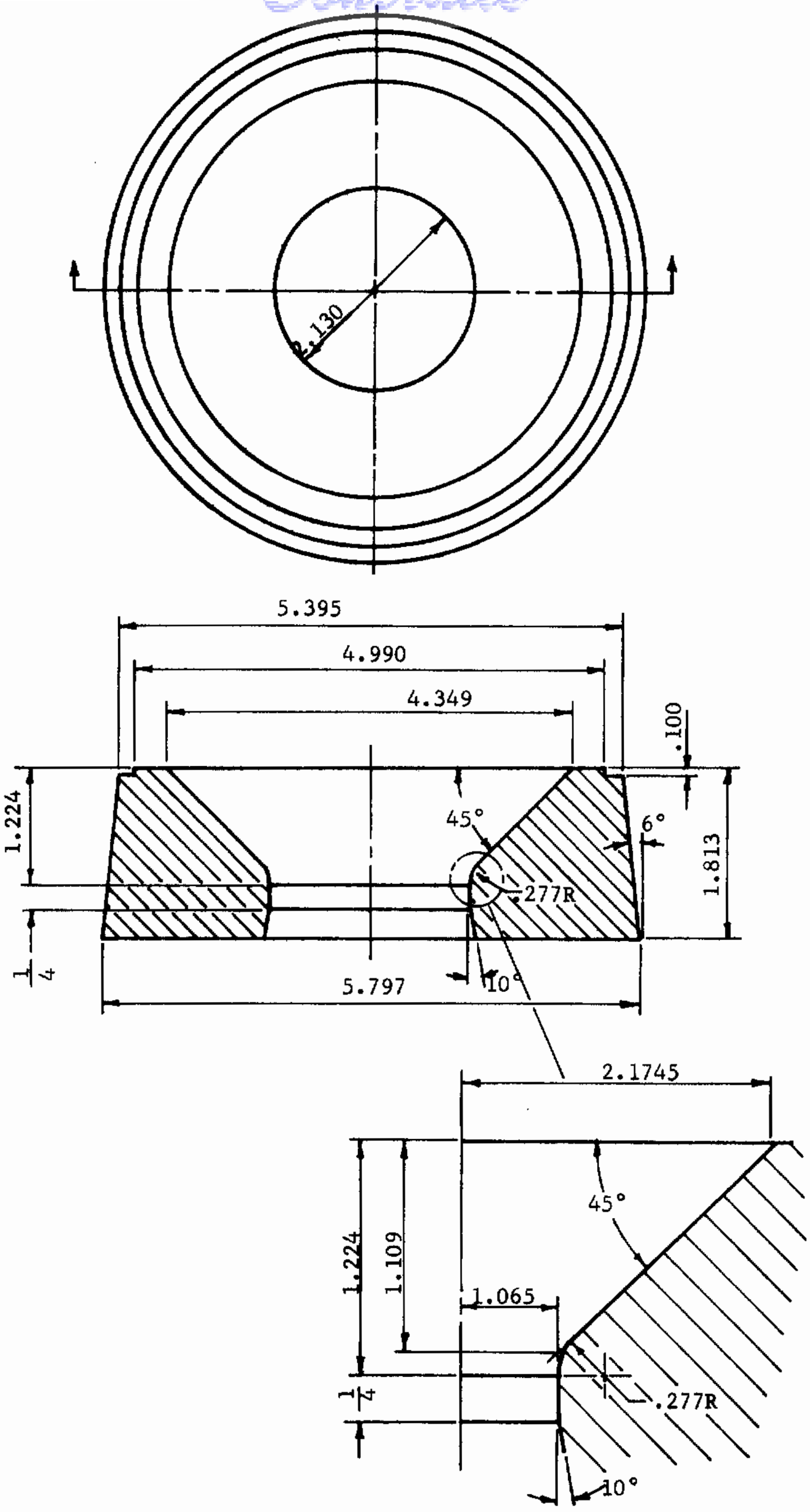


Figure 6

Die Design B-0601 Before Coating

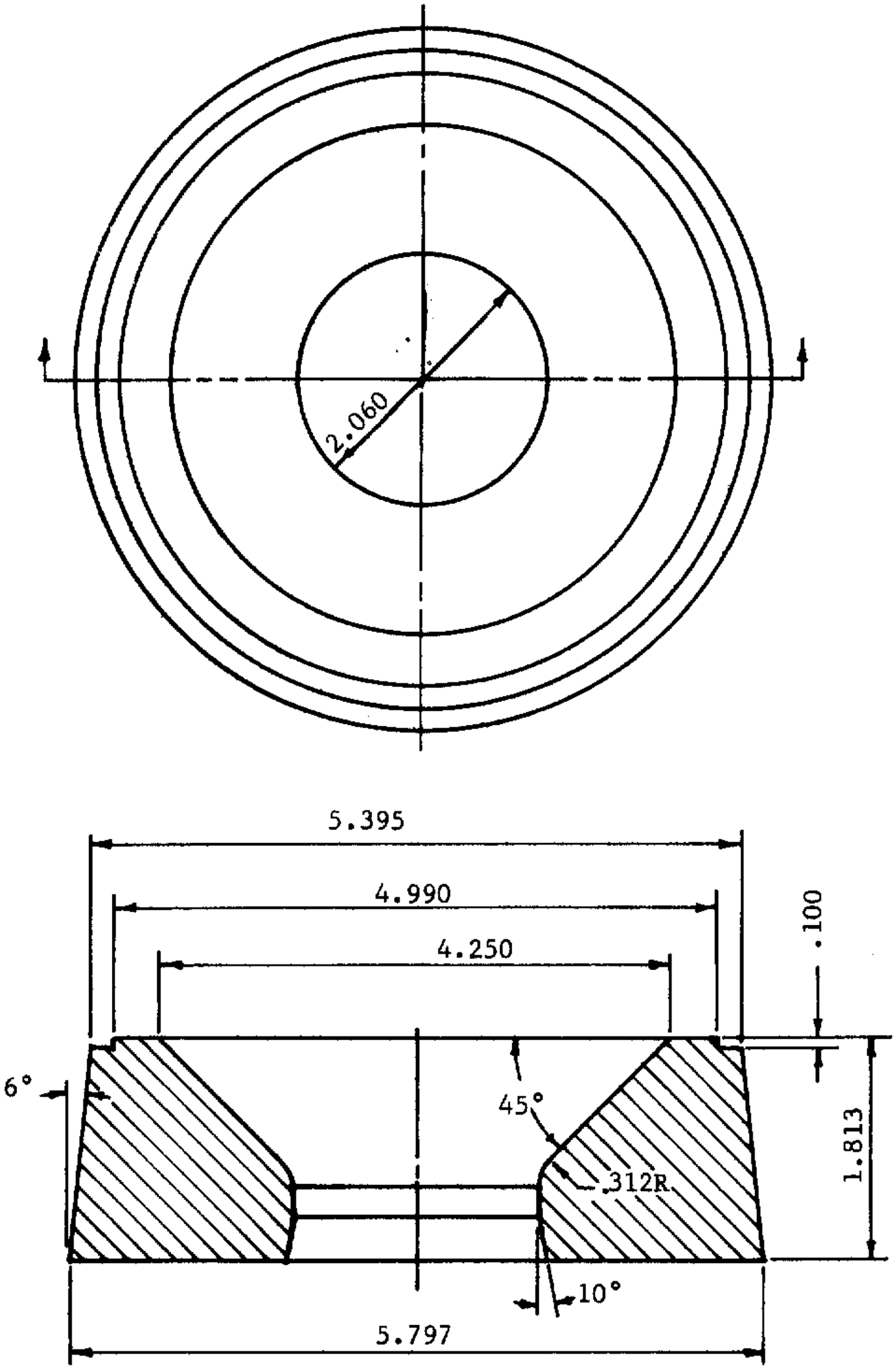


Figure 7

Die Design B-0601 After Coating

The mandrels were made of consumable electrode vacuum melted Potomac M (AISI H-13) hot worked tool steel heat treated to Rockwell C 48-50. The mandrels were likewise coated with a thermal barrier of zirconia.

Zirconia coated corner cones were used to bridge the gap between the die and the liner preventing material from extruding into this space. The corner cone also offers thermal protection to the front corner of the liner preventing wear of this corner.

The zirconia coating on the dies, mandrels and corner cones was applied by the oxyacetylene flame spray solid rod feed process. Surfaces to be coated were prepared by grit blasting to a 500-600 RMS finish and pre-coating with a .003 to .005-inch thick coating of a nickel-chromium alloy. Surfaces were coated with approximately .060-inch thick coating to achieve a .035-inch thick coating after clean-up by grinding and polishing with silicon carbide wheels and abrasive paper. Figure 8 contains photographs of a zirconia coated die and mandrel.

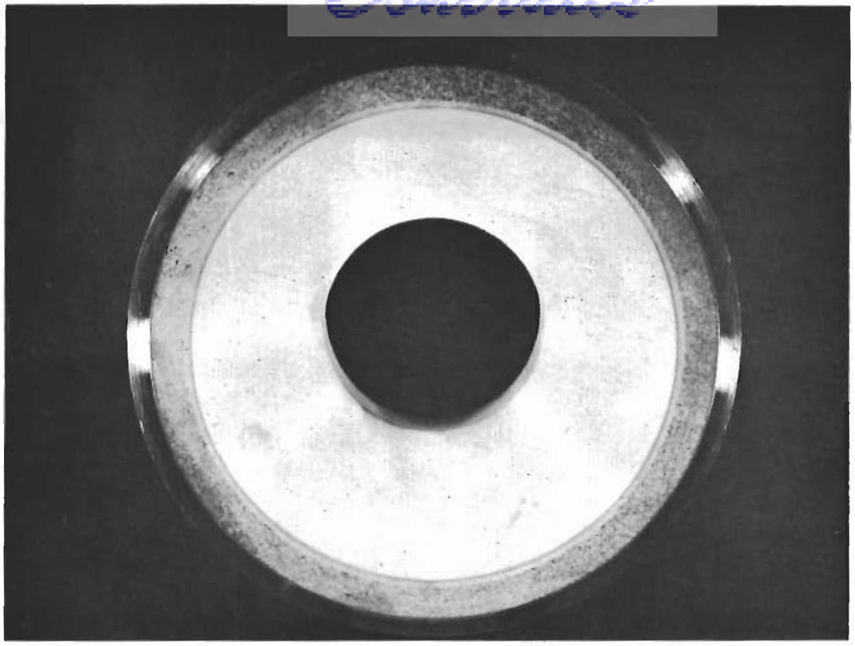
D. Extrusion of Tube Hollows

All billets were extruded using the previously described 5-inch diameter tooling system and utilizing the full 2200-ton capacity of the press. The billets were induction heated in an argon atmosphere. Temperature was controlled by means of a Land Radiation Pyrometer. Upon retraction from the induction coil the billets were immediately rolled in glass while en route to the automatic loading mechanism of the press. The pre-set, no load ram speed was 1000 inches per minute. Mild steel pushouts were used to insure that all of the tantalum alloy was extruded. One solid round extrusion of stainless steel was made prior to the tantalum alloy extrusion to check press alignment and tooling performance.

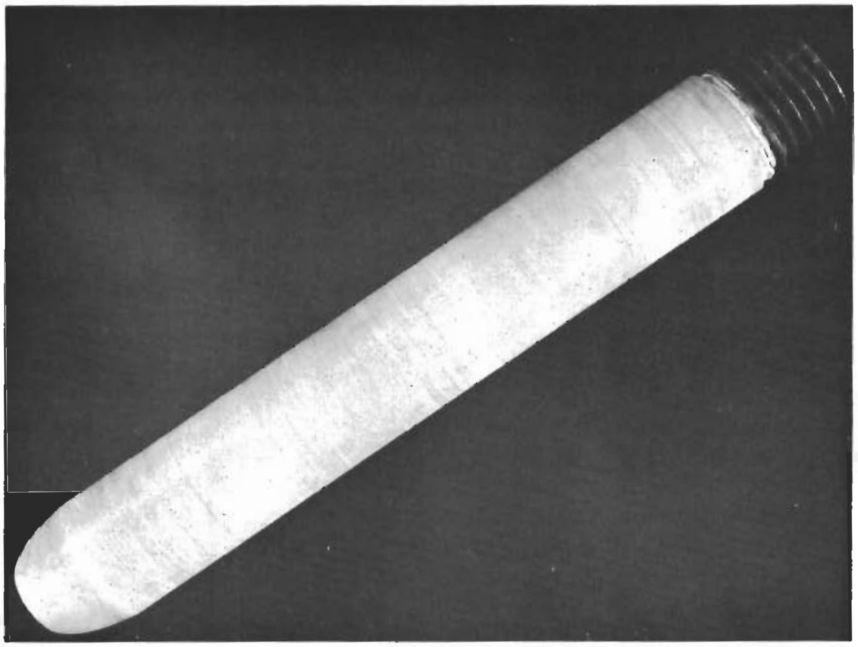
The ten tantalum alloy extrusions were conducted in three campaigns. The extrusion data is presented in Table 3.

In the first campaign two 6-inch long billets of both the Ta-10W and the T-222 alloys were extruded. The first billets of both alloys were extruded from a temperature of 3000F using a straight mandrel. The first Ta-10W billet (Extrusion No. 2) required almost full pressure during initial upset but the run pressures were considerably lower. Post extrusion examination of the die used revealed that the zirconia coating had eroded away in the land area. However, little or no metallic wash was evident. The cause of this erosion could be the presence of a highly abrasive tantalum oxide on the surface of the billet. Being the first billet heated in the coil on this date, the billet required a comparatively long time (24 minutes) to heat to temperature.

The first T-222 billet (Extrusion No. 3) required full pressure during the entire run. In this instance, metallic die wash had occurred and the mandrel sucked-off during extrusion at the approximate junction between the tantalum alloy and the low carbon steel pushout. These phenomena can very likely be



2.060-inch I.D. Zirconia Coated Die 45° Conical Entry
Mag. 1/2X (approx.)



Mag. 1/2X (approx.)

Figure 8

Zirconia Coated Extrusion Mandrel
1.440-inch diameter by 8-inch working length

TABLE 3

EXTRUSION DATA

Extrusion Sequence	Tantalum Alloy	Temp. (°F) From Coil	Time to Temp. (min.)	Total Transfer Time (sec.)	Lubrication			Extrusion Pressure		Extrusion Constant	
					OD	Die	ID	Upset (ksi)	Run (ksi)	Upset (ksi)	Run (ksi)
1	Steel Extrusion to Check Tooling										
2	Ta-10W	3000	24	17	AL-31-45	AL-N-56	AL-31-45	244.0	212.0	103.0	90.0
3	T-222	3000	11	22	AL-31-45	AL-N-56	AL-31-45	244.0	239.0	103.0	101.5
4	Ta-10W	2800	8	20	AL-31-45	AL-N-56	AL-31-45	160.0	196.0	81.5	83.0
5	T-222	3000	9	19	AL-31-45	AL-N-56	AL-31-45	203.5	239.0	105.5	101.6
6	Ta-10W	3000	30	24	AL-31-45	AL-N-56	AL-46-35	224.0	217.0	95.0	92.0
7	Ta-10W	3100	14	25	AL-46-35	AL-N-56	AL-46-25	224.0	224.0	95.0	95.0
8	Ta-10W	3100	15	26	AL-31-45	AL-N-56	AL-46-25	224.0	224.0	95.0	95.0
9	T-222	3000	37	35	AL-27-45	AL-N-56	AL-26-35	170.0	174.0	72.0	73.0
10	T-222	3000	13-1/2	32	AL-27-45	AL-N-56	AL-26-35	174.0	164.0	73.0	69.5
11	T-222	3000	12-3/4	31	AL-27-45	AL-N-56	AL-26-35	178.0	174.0	75.5	73.0

attributed to the extremely high unit pressures realized.

The second billets of each alloy, Extrusion Nos. 4 and 5, utilized a stepped mandrel to lower the initial resistance to deformation and insure that sufficient pressure was available to start the extrusion. Since the run pressures on the first Ta-10W billet extruded were considerably below press capacity, it was decided to extrude the second billet (Extrusion No. 4) from a temperature of 2800F. Both the initial upset and run pressures were lower than those recorded for the billet extruded from 3000F using a conventional type mandrel. Post extrusion examination of the die revealed that the zirconia coating was intact with no evidence of die wash.

Since both the initial upset and run pressures of the first T-222 billet were near press capacity, it was decided not to lower the temperature of the second T-222 billet extruded (Extrusion No. 5). The pressure data as seen in Table 3 shows that the stepped mandrel was very effective in reducing initial upset pressure. However, the run pressures were still very high. It is believed that these high pressures were responsible for the rather severe die wash that occurred.

Results of this first extrusion campaign indicated that the use of a stepped mandrel was very effective in reducing initial upset pressure. However, it was felt that the sharp step tended to wipe the ID lubricant from the billet resulting in poor ID surfaces. Consequently, the mandrels used during the second and third extrusion campaigns were essentially straight mandrels with front radius increased and designed to a length so that the material would start to extrude on the radiused portion, thus accomplishing the same objective as a stepped mandrel.

As a means of cutting down contamination following extrusion, the tube hollows from the second and third campaigns were extruded into an argon flooded steel pipe.

During the second extrusion campaign it was planned to extrude the remaining three 11-inch long billets of each of the two alloys. While the best Ta-10W extrusion accomplished during the initial campaign was made from a temperature of 2800F using a stepped mandrel, it was decided that because of the additional frictional forces present with the longer billets, the first long Ta-10W billet (Extrusion No. 6) should be extruded from a temperature of 3000F using the previously described modification of the stepped mandrel design. The pressures required were higher than those previously experienced with the Ta-10W alloy and extensive die wash was evident on the back half of the extrusion. In view of this the last two extrusions of the Ta-10W alloy (Extrusion Nos. 7 and 8) were made from a temperature of 3100F. This increase in temperature did not lower the pressures required but the quality of the extrusion was improved.

The initial campaign had indicated that the T-222 alloy required considerably more pressure to extrude than the Ta-10W alloy at the same temperature. This indicated, therefore, that the T-222 billets, if extruded in a like manner, probably should be extruded from a temperature of 3200F or higher. Previous experience of other extruders of the alloy had indicated that the T-222 alloy tends to be hot short when worked at temperatures in excess of 3000F. Therefore, as a means of lowering pressures and enabling us to utilize lower extrusion billet temperatures, the T-222 billets were clad in .030-inch thick molybdenum and equipped with a 1-inch thick pressed and sintered molybdenum nose plug. Figure 9 is a photograph of the molybdenum clad billet.

The three molybdenum clad T-222 billets were extruded from a billet temperature of 3000F during the third extrusion campaign. Since the billets were clad in molybdenum the glass lubricants used were those developed for the extrusion of molybdenum alloys and are identified by glass code number in Table 3. The pressures, as can be seen in Table 3, were considerably lower than those obtained with the unclad billets extruded from the same temperature - approximately 175,000 psi maximum for the clad billets versus 244,000 psi maximum for the unclad billets.

E. As Extruded Dimensional and Surface Evaluation

A review of the as extruded dimensional data and surface condition is presented in Table 4. The die orifice for all extrusions was 2.060-inch diameter.

Figure 10 is a close-up view of the as extruded OD surface of Extrusion No. 2. The tiny cross tears evident were localized in the front portion of the tube. While dimensional run-out from the front to back was only .025-inch on the diameter, the diameter of the tube at the front end indicates that the zirconia coating broke up immediately. There was no evidence of metallic pick-up however.

An actual size close-up view of the as extruded surface of Extrusion No. 3 (T-222, 3000F) is shown at the top of Figure 11. Dimensional run-out was .060-inch on the diameter. Again, it is evident that the zirconia coating broke up immediately. Metallic die wash had occurred in this instance and this is evident as metallic pick-up on the OD surface.

Extrusion No. 4 (Ta-10W) extruded from 2800F displayed an excellent OD surface. This is shown in the bottom photograph of Figure 11. Dimensional run-out over the 51 inches of extruded length was .013-inch maximum. Post extrusion examination of the die revealed that the zirconia coating was intact with no evidence of die wash.

OD dimensional run-out on Extrusion No. 5 (T-222) exceeded .100-inch and the OD surface was similar to that shown for Extrusion No. 3 (Figure 11) with large amounts of metallic pick-up on the OD surface.

Figure 9

Molybdenum Clad T-222 Alloy
Extrusion Billet

TABLE 4

AS EXTRUDED DIMENSIONAL DATA

Extrusion Number	Tantalum Alloy	Length (Inches)	Weight (Pounds)	Outside Diameter		As Extruded OD Surface Condition
				Front	Back	
2	Ta-10W	34	40	2.110	2.135	Good in front-light striations on back
3	T-222	42	54	2.105	2.178	Good in front-light striation in back
4	Ta-10W	51	58	2.040	2.053	Good
5	T-222	43	58	2.110	2.220	Light to heavy striations front to back
6	Ta-10W	75	108	2.110	2.330	Fair-Striated in back end
7	Ta-10W	106	110	2.037	2.081	Excellent Front-striated in back half
8	Ta-10W	96	108	2.038	2.112	Excellent Front-striated in back half
9	T-222	74	83	2.030	2.046	Excellent clad surface
10	T-222	101	103	2.040	2.047	Excellent clad surface
11	T-222	103	105	2.040	2.046	Excellent clad surface

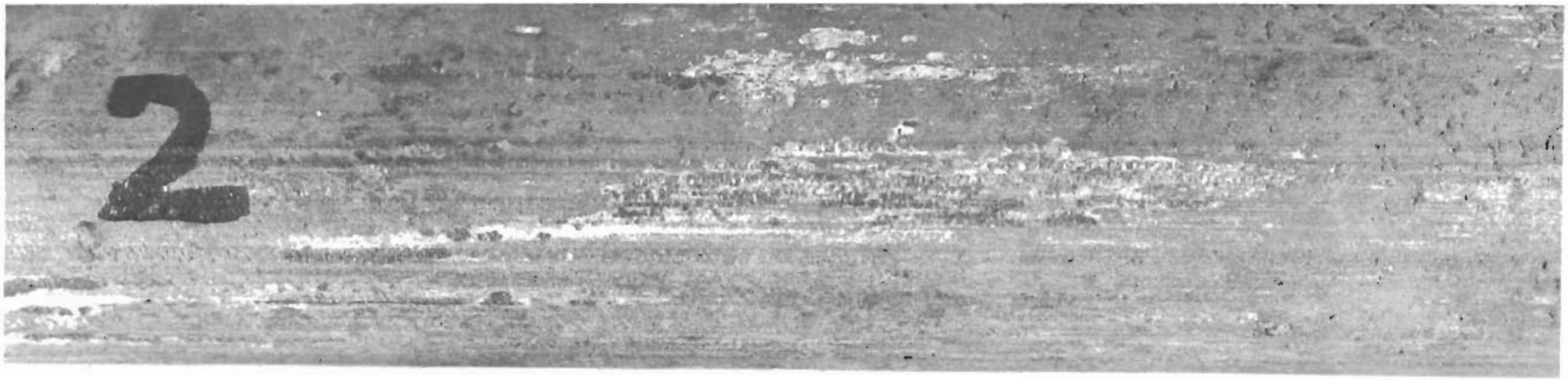


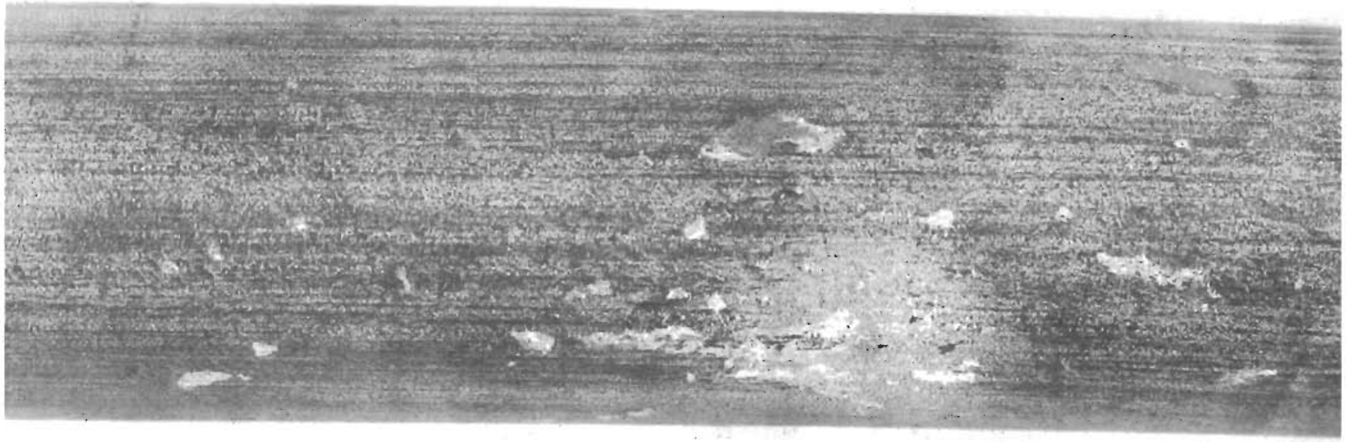
Figure 10

Close-up View of the As Extruded O.D. Surface of Extrusion No. 2

Ta-10W Alloy
Extrusion Temperature - 3000F

Close-up View of the As Extruded O.D. Surface of Extrusion No. 3

T-222 Alloy
Extrusion Temperature - 3000F



Close-up View of the As Extruded O.D. Surface of Extrusion No. 4

Ta-10W Alloy
Extrusion Temperature - 2800F

Figure 11

Extrusion No. 6 (Ta-10W), the first 11-inch long billet, was extruded from a temperature of 3000F. The pressures required were higher than those previously experienced for this alloy and extensive die wash was evident on the back half of the extrusion. In view of this, the last two extrusions of Ta-10W were made from a temperature of 3000F. The front half of Extrusion Nos. 7 and 8 were excellent with longitudinal striations developing about midway in the 9-foot length. This can be seen in Figure 12, which shows the as extruded surface of Extrusion No. 7. The surfaces of Extrusion No. 8 were similar.

The as extruded surfaces of the three clad T-222 extrusions were excellent. There was no tearing or stripping of the cladding and the cladding was evenly distributed over the entire surface area. Figure 13 is a close-up view of the as extruded surface at the back end of Extrusion No. 9. There was no evidence of die wash. The die opening was 2.060-inch and the outside diameter at the back end of all three tubes was 2.046 to 2.047 inches.

F. Post Extrusion Evaluation

Following extrusion, all of the unclad tube hollows were grit blasted on the OD and ID to remove the glass lubricant and any loose oxide scale. The molybdenum clad T-222 extrusions were pickled in a 25 percent HNO_3 solution to remove the cladding. Figure 14 shows the OD surface of the declad extrusions. A matte OD surface finish was revealed. Inspection of the ID's revealed a surface finish, in general, similar to that of the OD. However, it appeared that a reaction had occurred between the tantalum alloy and the molybdenum cladding in localized areas and the ID surfaces were not good in these areas.

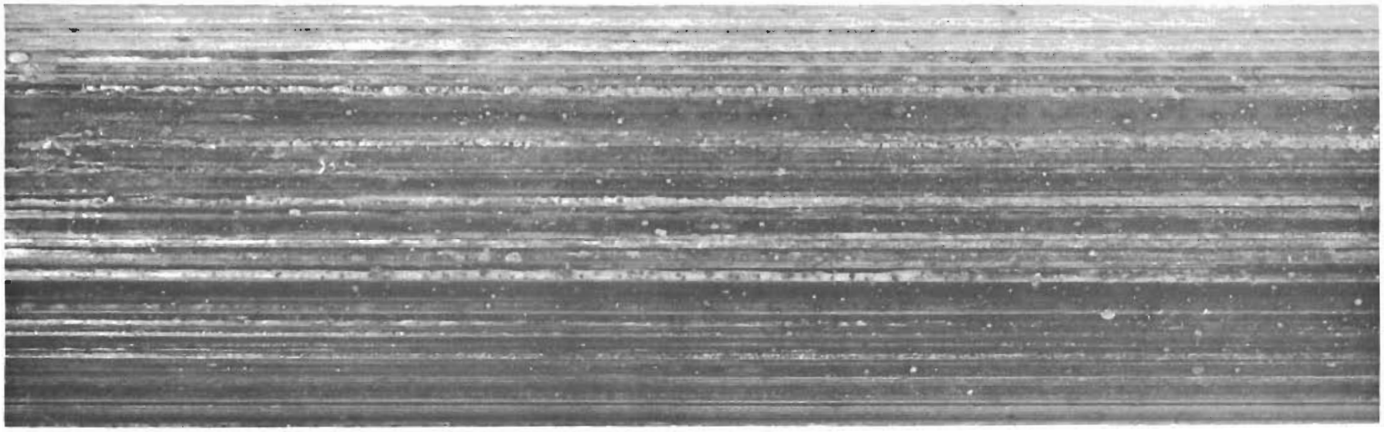
All extrusions were straightened on a Medart Roll Straightener. The Ta-10W extrusions were straightened cold, while a temperature of 500F was used to straighten the long T-222 extrusions. Samples were cut from the tube hollows produced during the initial campaign for evaluation in terms of hardness, room temperature tensile tests, metallographic examination and analysis for interstitial elements carbon, nitrogen, oxygen and hydrogen. Samples were also obtained to determine the stress relief and recrystallization temperatures.

1. Room Temperature Tensile Data

Room temperature tensile data is shown in Table 5. All specimens were tested in the as extruded condition at a strain rate of .005 inch/inch/minute to yield and .05 inches/inch/minute to fracture. The data shows that the room temperature tensile properties, both strength and ductility, are higher for the T-222 alloy. The slightly lower strengths shown for the nose samples, as compared to the back end, of Extrusion Nos. 4 and 5, can be attributed to the use of a stepped mandrel which results in the front end being extruded at a lower reduction ratio than the remainder of the tube hollow. In comparing the properties of the Ta-10W extrusions, it

Front

Middle



Back

Figure 12

Ta-10W Extrusion No. 7
Extrusion Temperature - 3100F
Length - 8 feet 6 inches

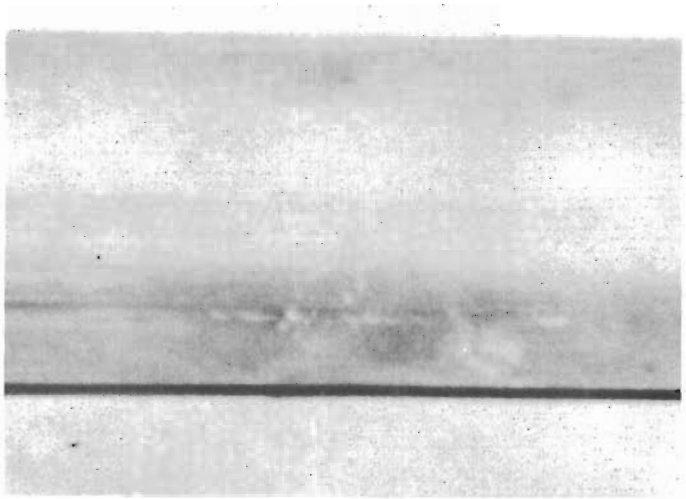


Figure 13

Close-up View of the As Extruded Surface of Extrusion No. 9 Back End

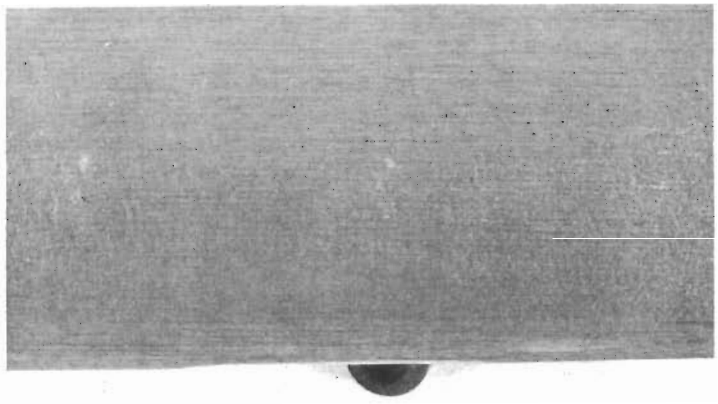


Figure 14

Surface Finish of T-222 Tube Hollow
Following Removal of Molybdenum Cladding

TABLE 5

ROOM TEMPERATURE TENSILE DATA

Extrusion Sequence Number	Alloy	Position	Ultimate Tensile Strength (psi)	.2% Yield Strength (psi)	.02% Yield Strength (psi)	% Elong.	% Reduction of Area
2	Ta-10W	Nose	116,417	102,487	88,308	13.1	29.9
		Nose	110,101	96,969	88,383	10.4	28.3
		Back	117,010	102,577	91,752	16.0	29.9
		Back	111,956	97,282	86,413	13.3	26.0
3	T-222	Nose	132,338	130,348	129,352	20.0	55.2
		Nose	132,019	129,310	128,078	19.2	55.7
		Back	132,000	128,000	125,000	16.8	45.5
		Back	135,643	131,188	129,950	20.8	39.1
4	Ta-10W	Nose	97,772	82,920	65,594	20.8	55.4
		Nose	97,512	72,139	50,995	19.2	60.6
		Back	110,784	96,078	85,784	15.2	53.9
		Back	106,467	92,039	83,333	22.4	52.7
5	T-222	Nose	124,861	99,447	81,491	15.2	51.3
		Nose	121,890	105,721	90,796	22.5	53.7
		Back	127,586	120,689	108,374	23.2	51.2
		Back	128,855	119,402	99,502	15.2	60.2
6	Ta-10W	Nose	113,432	98,258	94,527	16.9	29.9
		Back	131,592	127,363	104,477	7.2	7.5*
7	Ta-10W	Nose	111,442	100,746	87,064	13.6	29.9
		Back	114,925	102,487	95,024	17.6	41.8
8	Ta-10W	Nose	112,437	98,258	70,646	15.2	45.8
		Back	101,990	93,283	84,577	19.2	48.2

*Crack

will be noted that the lower strengths and higher ductilities were obtained on the extrusion made from the lower temperature 2800F.

2. Metallographic Examination and Hardness Traverse

A metallographic study and hardness traverse were made to determine the depth of contamination of extruded material. Figures 15 through 18 are plots of the hardness versus distance from edge of samples for Extrusion Nos. 2 through 5, respectively. Photomicrographs of the specimens in the areas where the hardnesses were taken are also shown in the figures. The greatest contamination depth determined by this method was .030-inch on the first Ta-10W billet extruded. This billet had required 24 minutes to heat to 3000F. The least depth was .004-inch on the Ta-10W billet heated to 2800F in 8 minutes.

3. Determination of Recrystallization Temperatures

To determine the recrystallization temperatures of the materials, micro specimens of each alloy were heat treated in an R.D. Brew Vacuum Furnace at temperatures in the range of 1800F to 3000F for one hour in a vacuum of less than 2×10^{-5} Torr. Figure 19 shows the micros and hardnesses obtained on the Ta-10W specimens. Figure 20 shows the micros and hardnesses obtained on the T-222 alloy specimens. Metallographic examination and hardness results revealed that the Ta-10W alloy was completely recrystallized at 2400F. Only incipient recrystallization, comparable to the as extruded structure was evident at temperatures up to 2200F. The T-222 alloy was about one-third recrystallized at 1800F with a gradual increase in the amount of recrystallization until completion at 3000F. Some worked structure was still evident at 2800F.

4. Chemical Analysis of Extruded Tube Hollows

Samples from Extrusion Nos. 2, 3, 4 and 5 were analyzed for the interstitial elements carbon, nitrogen, oxygen and hydrogen. Results of these are compared to the ingot analysis as reported by Wah Chang Corporation in Table 6.

G. Post Extrusion Conditioning

Superior Tube Company, our subcontractor to perform the tube reducing and drawing under Phases III and IV, required a finished tube hollow 2.00-inch OD by .250-inch wall thickness with an OD and ID surface finish of 125 RMS or better. The three to four-foot long extrusions made during the initial run were too short for conditioning on our production OD centerless grinding and ID honing and polishing equipment. It was necessary to condition these tubes by machining on a lathe. While machining produced excellent surfaces on the OD, this method of conditioning the ID's was

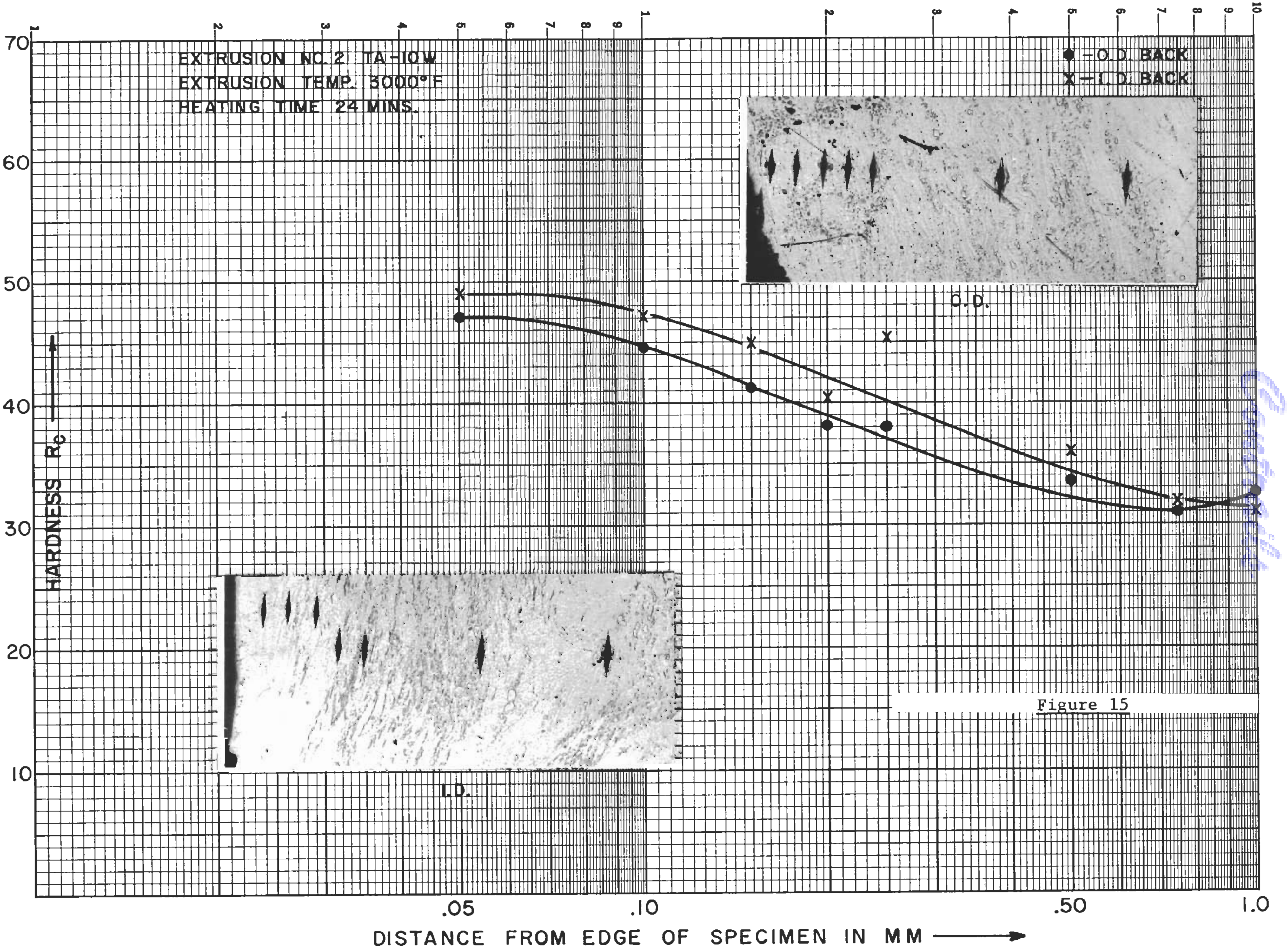

EXTRUSION NO. 2 TA-10W
EXTRUSION TEMP. 3000°F
HEATING TIME 24 MINS.
● - O.D. BACK
X - I.D. BACK
O.D.
I.D.
HARDNESS R_c
70
60
50
40
30
20
10
.05
.10
.50
1.0
DISTANCE FROM EDGE OF SPECIMEN IN MM

Figure 15

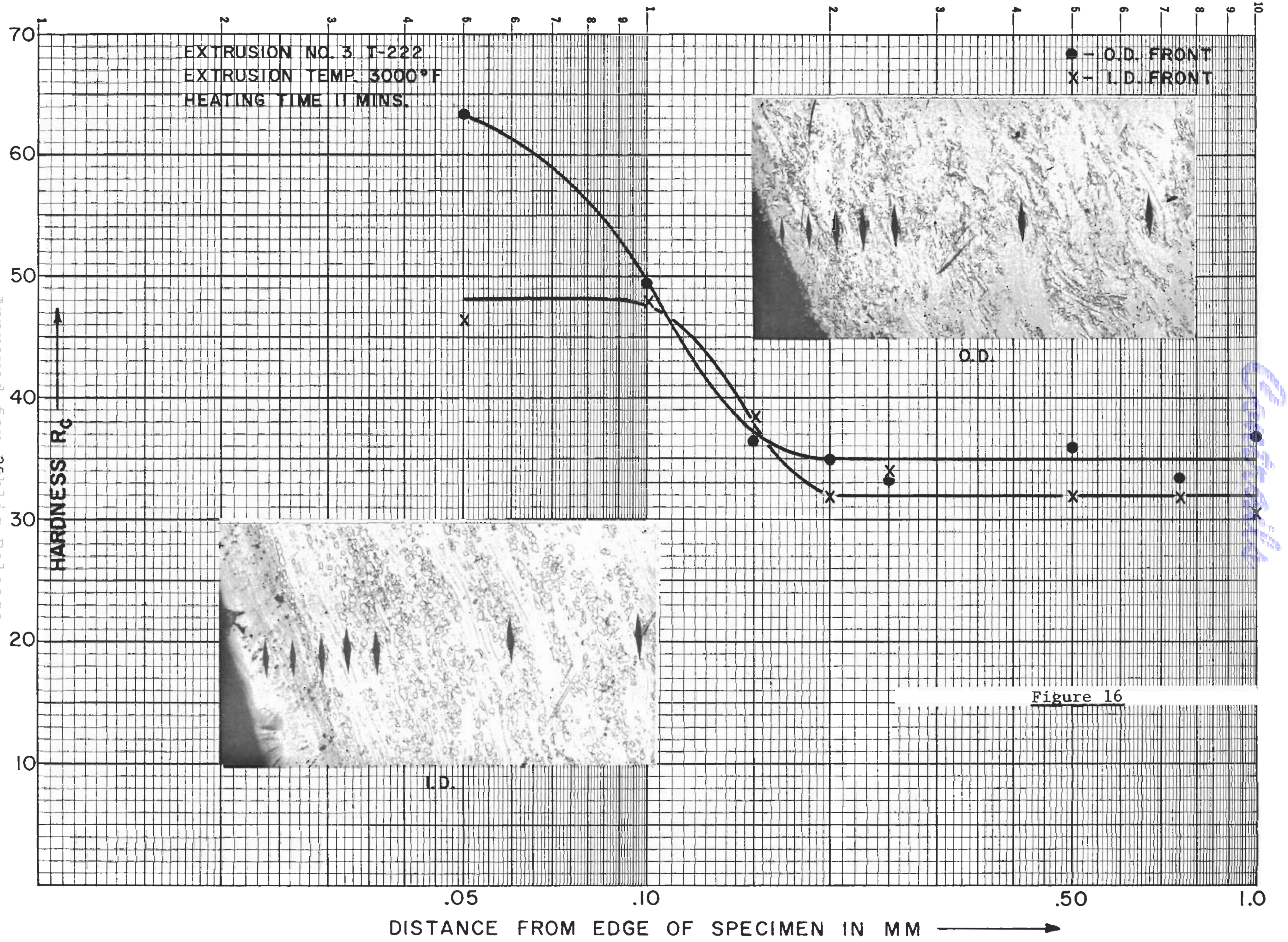
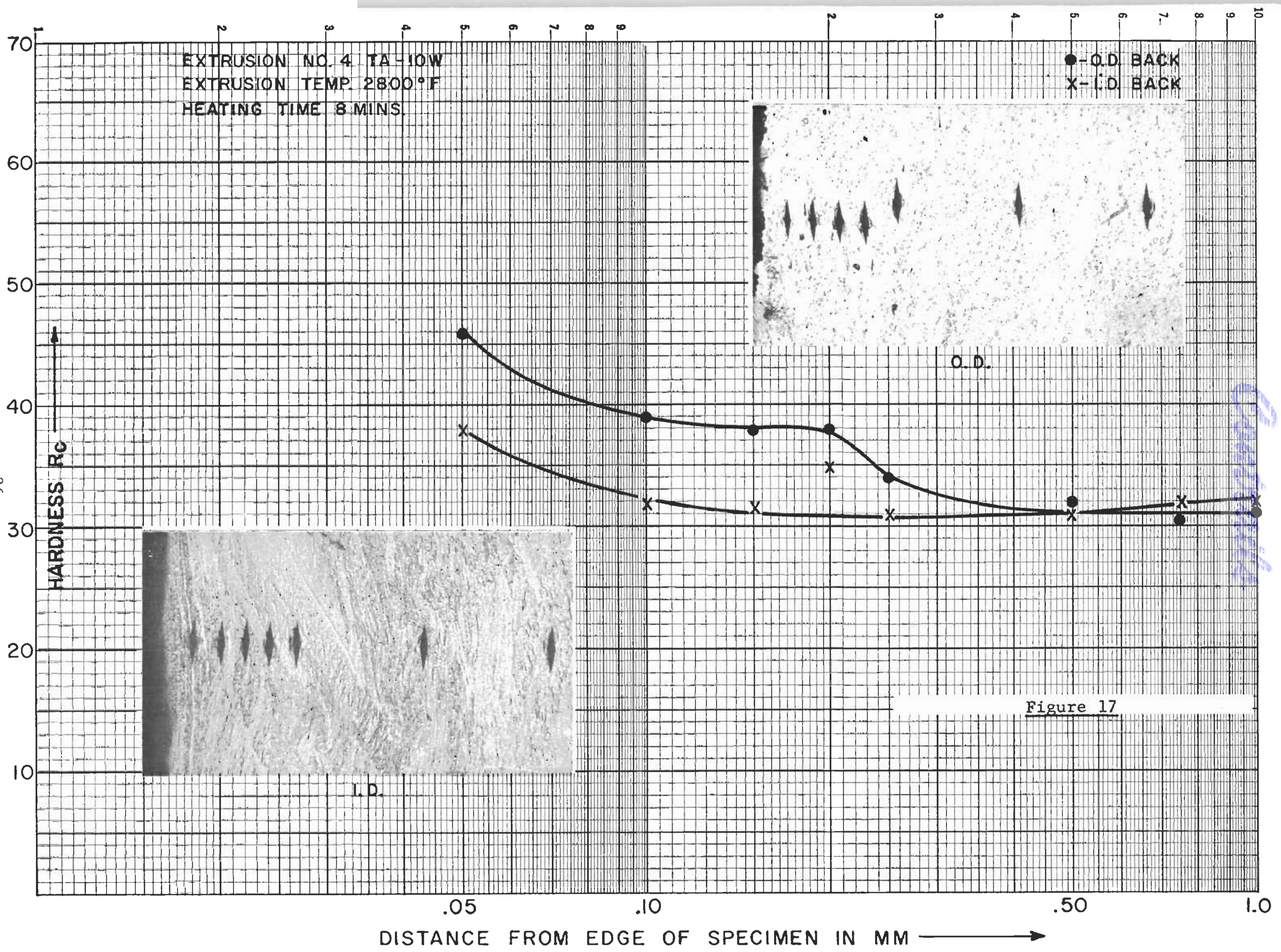

EXTRUSION NO. 3 T-222
EXTRUSION TEMP. 3000°F
HEATING TIME 11 MINS.
● - O.D. FRONT
X - I.D. FRONT
O.D.
I.D.
HARDNESS R_c
70
60
50
40
30
20
10
.05
.10
.50
1.0
DISTANCE FROM EDGE OF SPECIMEN IN MM

Figure 16



EXTRUSION NO. 4 TA-10W
EXTRUSION TEMP. 2800°F
HEATING TIME 8 MINS.

Figure 17

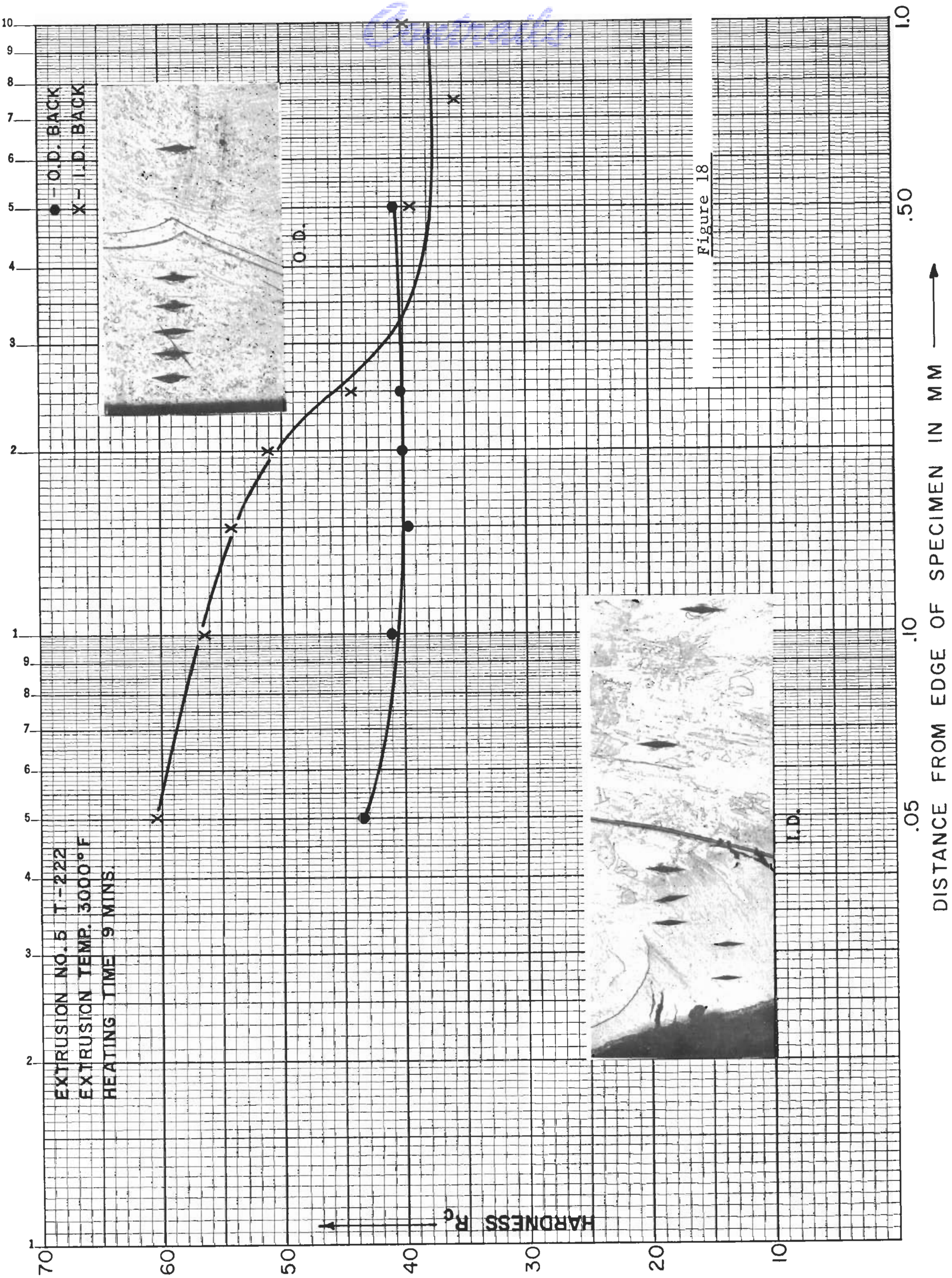
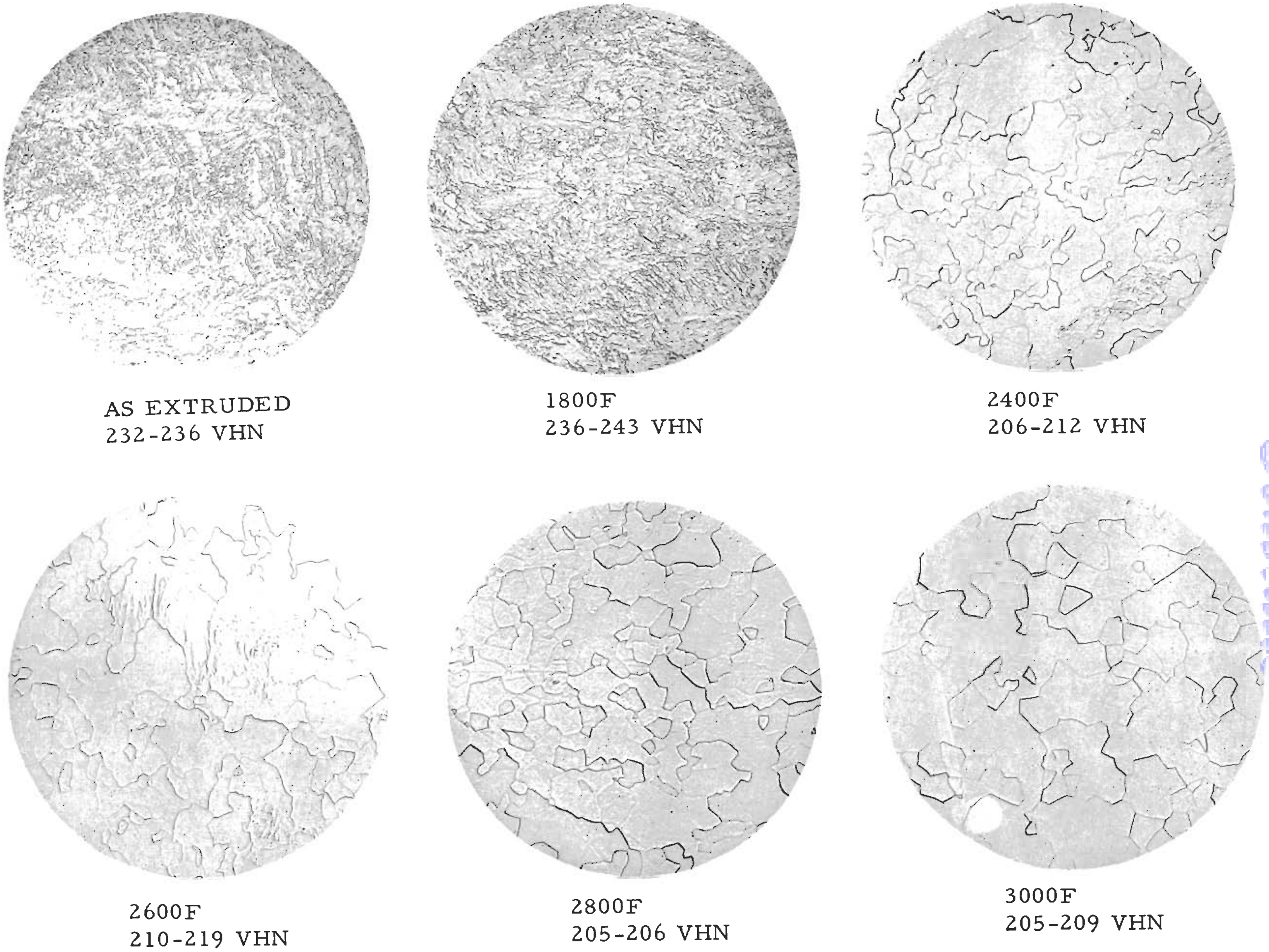

● - O.D. BACK
X - I.D. BACK
O.D.
I.D.
EXTRUSION NO. 5 T-222
EXTRUSION TEMP. 3000°F
HEATING TIME 9 MINS.
HARDNESS R_c
DISTANCE FROM EDGE OF SPECIMEN IN MM
70
60
50
40
30
20
10
.05
.10
.50
1.0
1
2
3
4
5
6
7
8
9
10

Figure 18



AS EXTRUDED
232-236 VHN

1800F
236-243 VHN

2400F
206-212 VHN

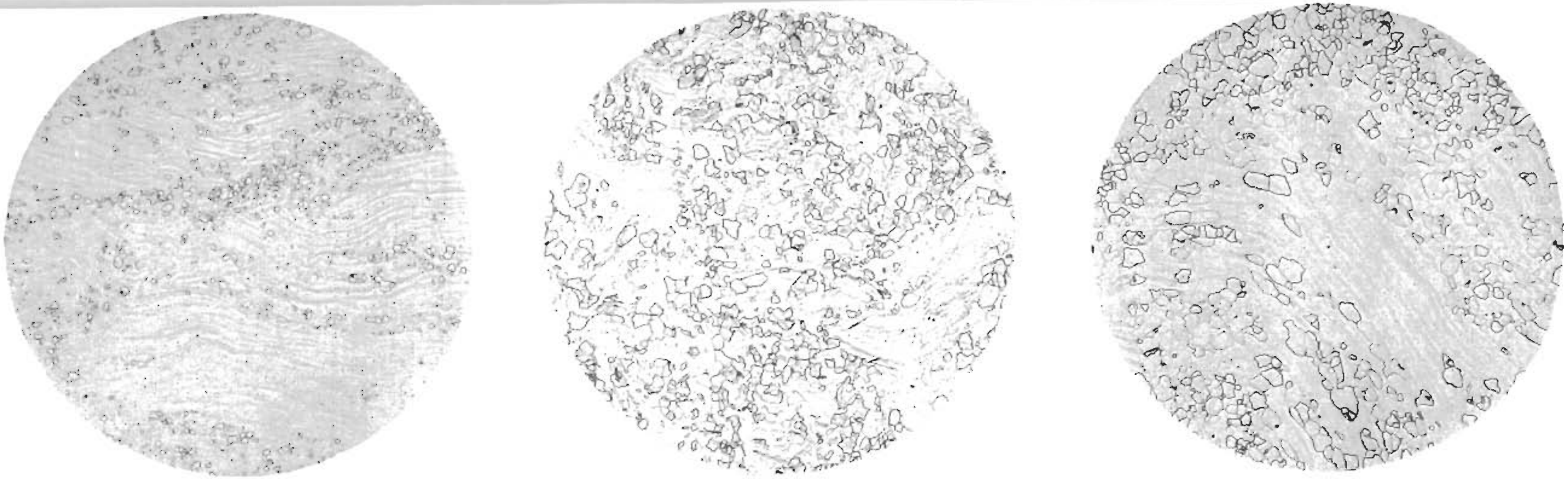
2600F
210-219 VHN

2800F
205-206 VHN

3000F
205-209 VHN

<u>Figure 19</u>

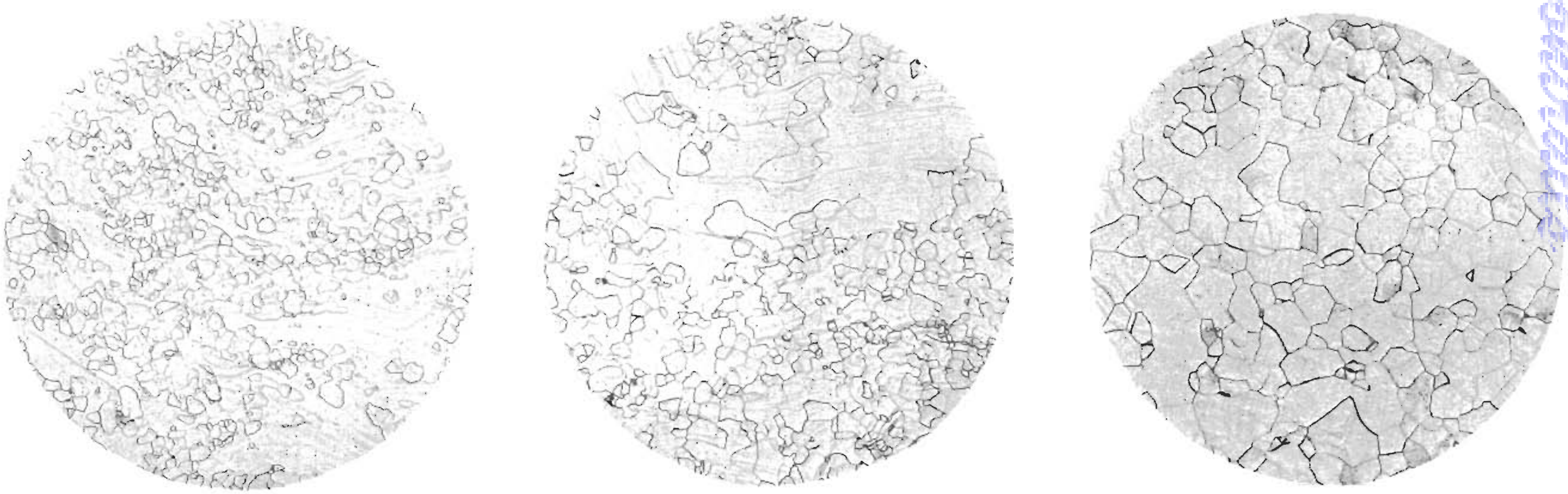
DETERMINATION OF RECRYSTALLIZATION TEMPERATURE
TA-10W ALLOY



AS EXTRUDED
306-309 VHN

1800F
279-306 VHN

2400F
279-285 VHN



2600F
279-283 VHN

2800F
292-297 VHN

3000F
287-294 VHN

Figure 20

DETERMINATION OF RECRYSTALLIZATION TEMPERATURE
T-222 ALLOY

TABLE 6

CHEMICAL ANALYSIS FOR INTERSTITIAL ELEMENTS

	Element	Ingot Analysis	Extrusion No. 2	Extrusion No. 4
Ta-10W Heat No. 62026-Ta-10W-S3B	Carbon	< 30 ppm	200-210 ppm	180 ppm
	Oxygen	< 50 ppm	78 ppm	14 ppm
	Nitrogen	35-55 ppm	3 ppm	8 ppm
	Hydrogen	2.5-2.7 ppm	14 ppm	4 ppm

	Element	Ingot Analysis	Extrusion No. 3	Extrusion No. 5
T-222 Heat No. 65032-Ta-9W-2.5 Hf-0.01C	Carbon	130-140 ppm	300 ppm	230-250 ppm
	Oxygen	< 50 ppm	10 ppm	12 ppm
	Nitrogen	< 10 ppm	7 ppm	7 ppm
	Hydrogen	1.9-2.6 ppm	8 ppm	6 ppm

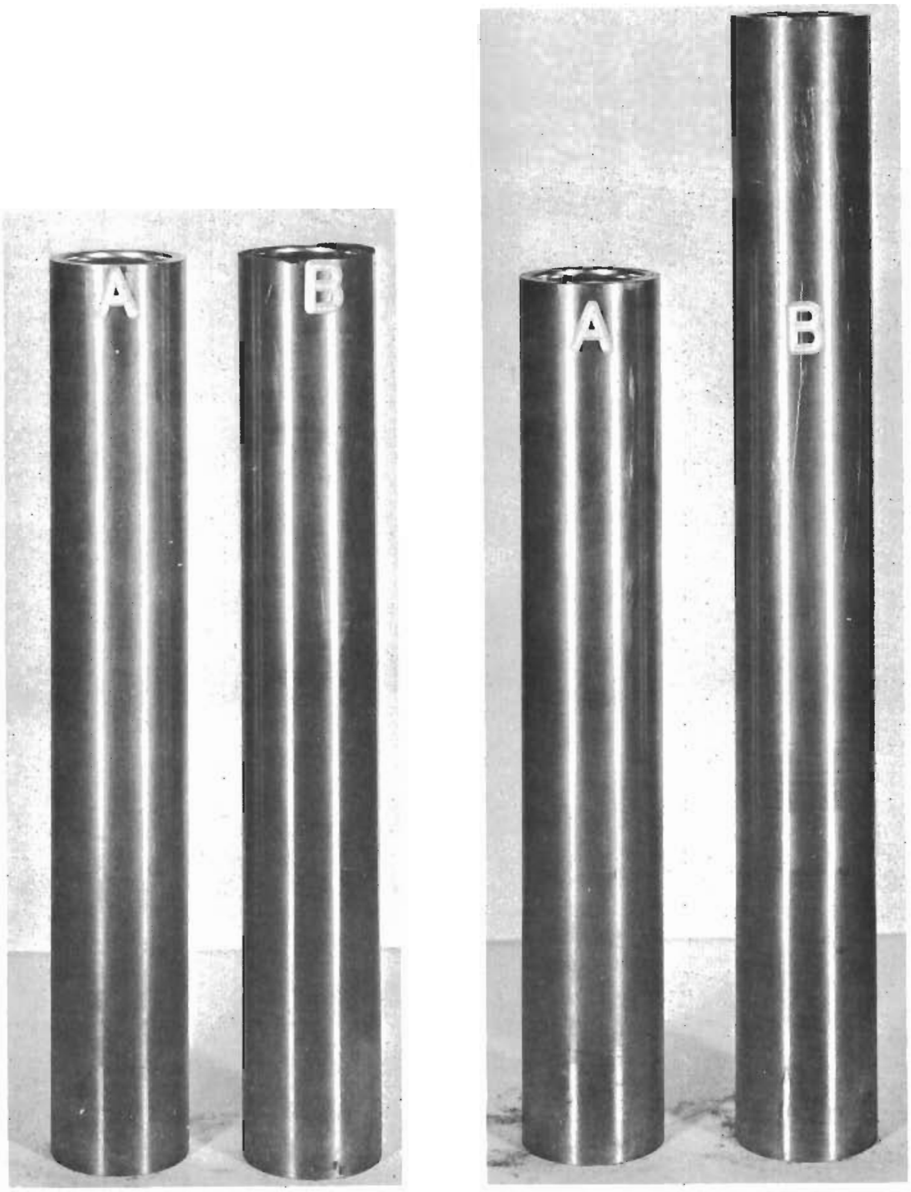
unacceptable because many machine marks, gouges and smear-type defects were produced. It was necessary to polish the ID surfaces following machining. OD surface readings were in the range of 20-40 RMS in the longitudinal direction and 10-20 RMS in the transverse direction. Figure 21 is a photograph of the two initial T-222 extrusions following conditioning. It was necessary to cut the extrusions into two pieces for machining.

The three longer tube hollows of each alloy were centerless ground on the OD to a surface finish of about 50 RMS. It was found that the ID polishing belt did not remove sufficient material to adequately clean-up the ID's. Consequently, the ID's were bored at Babcock and Wilcox Company, Tubular Products Division. Babcock and Wilcox reported that the material was very difficult to ID bore and excessive tool wear was encountered.

All of the conditioned tube hollows were inspected by ultrasonic examination and dye penetrant tests. Some small sonic indications were noted at random in various areas of the tubes and, in most cases, these could be associated with visible marks (i.e., machine marks, handling scratches, etc.) on the tubes. A few gross ID defects were present which we were aware of and which we had decided would be cut out at a later stage in the processing, when less material would be lost. Since no gross indications other than those visible on the ID surface were detected, the tubing was judged to be of good ultrasonic quality.

The OD dye penetrant tests confirmed the ultrasonic test results with no evidence of microcracks or other surface defects, other than minor scratches or machine marks.

The tube hollows were to be supplied to Superior Tube Company in the fully annealed condition. For economic reasons all of the tube hollows were vacuum annealed at Superior Tube Company at a temperature of 3100F for approximately ten to twelve minutes at temperature. The vacuum at the beginning of the annealing cycle was 5×10^{-5} Torr while at the end of the cycle the vacuum was 2×10^{-5} Torr. Some checks were made to ascertain that this shorter time at a higher temperature would be adequate for the T-222 alloy, and while we realized it might slightly over-anneal the Ta-10W alloy, it was felt that this would not present a problem at this tube size. Figure 22 shows the typical microstructures of both the Ta-10W and T-222 alloys following vacuum annealing. Following the anneal both alloys displayed a grain size of ASTM 5-7.

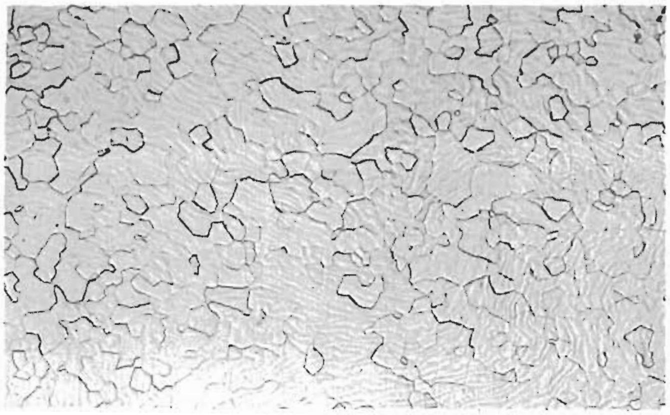


Extrusion No. 3

Extrusion No. 5

Figure 21

Finished Tube Hollows of T-222 Alloy



Ta-10W

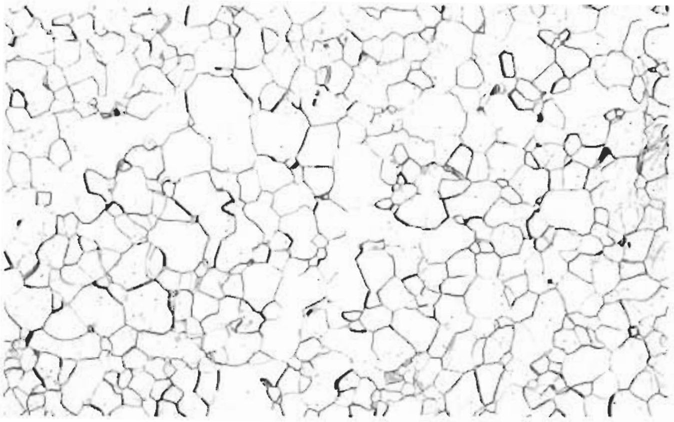


Figure 22

Typical Microstructures Following Vacuum Annealing

Mag. 100X
Etchant: 100cc H_2O, 50cc HNO_3, 5cc HF, 25 grams ammonium fluoride

SECTION IV

Phase III - Development of Tube Production Process

A. Introduction

Phase III of the program had a two-fold objective:

1. To develop the optimum process for tube reducing and drawing of sample quantities of each alloy to each of three sizes:

   .500-inch OD by .062-inch wall
   .375-inch OD by .062-inch wall
   .250-inch OD by .020-inch wall

   Material from both alloys for evaluation under this phase was tempered and tested in the 1/4 hard, 1/2 hard, full hard, full hard plus stress relieved, and fully recrystallized condition.

2. To evaluate the acceptability of the finished tubing and establish tentative specification requirements as to tolerances, quality and mechanical properties. Material from this test lot sequence was completely evaluated in terms of all the below listed non-destructive and destructive tests:

   a) ultrasonic and eddy current tests
   b) hydrostatic tests
   c) flaring, flattening and bending tests
   d) room temperature tensile tests
   e) burst testing
   f) analysis for interstitial elements carbon, oxygen, nitrogen and hydrogen
   g) metallographic examination
   h) surface roughness determination

   In addition extensive elevated temperature tensile and stress rupture tests were conducted on tubing from both alloys in each of the three sizes and in all five temper conditions. Tests were conducted at temperatures of 2100F, 2400F and 2700F.

B. Tube Inspection

Following vacuum annealing all of the tube hollows were subjected to Superior Tube Company's raw material inspection. This inspection included metallographic examination, Vidigage, ultrasonic and dye penetrant inspection. Essentially, those tube hollows which had been conditioned by machining passed this inspection, while those which had been centerless ground and ID bored were rejected as having defects in excess of .006-inch deep.

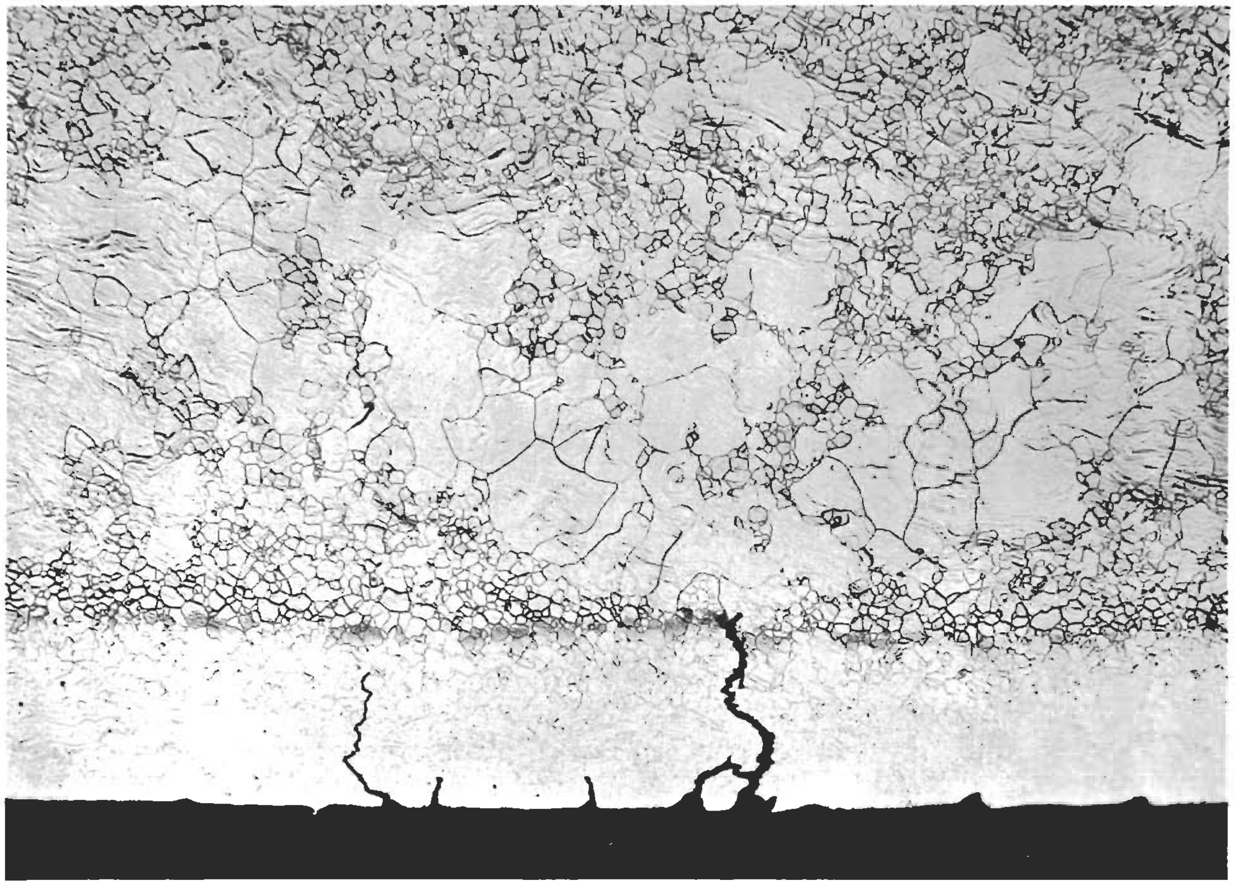
1. Metallographic Examination

Considerable concern was generated when metallographic examination at Superior Tube of a sample from one end of Tube Number 10 (T-222) revealed some small cracks and a light etching surface area to a depth of .012-inch. This is shown in Figure 23. Also present on this same sample was an area of large grains located about .012-inch from the outer edge. It was felt that this area might be a contamination or diffusion layer that could impede tube reducing. Since we were uncertain as to whether this was a general condition present on all of the tubes, it was decided that a thorough metallographic examination should be conducted on each of the tube hollows. Consequently, rings were cut from both ends of each of the tube hollows and the entire ring was examined metallographically at Allegheny Ludlum. This study revealed that neither the light etch area nor the band of large grains were present on any of the other samples. Microexamination of samples from Tube Number 10 in the declad, as extruded condition prior to centerless grinding or vacuum annealing revealed that there was no surface contamination layer present, nor was there a diffusion layer formed between the tantalum alloy and the molybdenum cladding. This can be seen in Figure 24. The sample examined at Superior Tube Company had been taken from the area immediately adjacent to a 1/2-inch diameter hole which had been drilled to suspend the tube in the vacuum furnace. Since this light etch area and large grain band were not detected on other sections taken from the same ring of Tube Number 10, it was felt that the heat generated in drilling the hole may be a likely cause of this phenomena.

2. Ultrasonic and Dye Penetrant Inspection

The ultrasonic and dye penetrant inspection revealed the presence of two conditions: 1) the presence of severe ID gouges which we were aware of and which we had decided would be cut out at a later stage when less material would be lost, and 2) the presence of cross cracks located sporadically over the entire length of the three longer Ta-10W tubes (Tube Nos. 6, 7 and 8) and T-222 (Tube No. 10).

Superior Tube felt that the defects in Tube No. 8 (Ta-10W) and Tube No. 10 (T-222) precluded the production of quality tubing unless additional OD conditioning was performed prior to tube reducing. The cracks in these two tubes were about .010-inch deep. While most of the defects were removed by spot grinding, these two tubes were recenterless ground over the entire length to remove .012 to .014-inch from the OD surface. These were dye penetrant checked at Allegheny Ludlum immediately following the regrind operation and appeared free from cracks. However, re-inspection at Superior Tube Company again revealed numerous cross cracks.



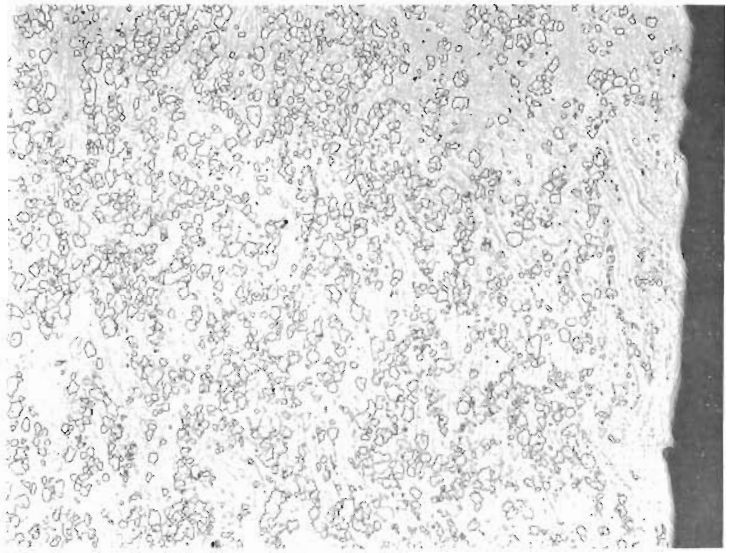
Mag. Approx. 100X

Figure 23

Showing Light Etching Surface and OD Cracks to a Depth of .012-inch

Etchant: 20 grams $NH_4 (HF)_2$ dissolved in 80 cc H_2O + 60 cc HF with 40 cc H_2O

Photograph Courtesy of Superior Tube Company



Mag. 100X

Figure 24

T-222 - Tube No. 10

Microstructure of tube Following Decladding and Prior to Centerless Grind or Vacuum Anneal. Note Absence of Light Etch Area or Microcracks

Etchant: 100 cc H_2O, 50 cc HNO_3, 5 cc HF, 25 grams ammonium fluoride

While some of the tubes had stress cracks present in the as extruded condition, these were generally very shallow and should have been removed in conditioning as considerable material was removed from the OD of each tube hollow. Furthermore, no surface cracking whatsoever was evident on the as extruded T-222 tube hollows which had been extruded using the molybdenum cladding technique. Obviously, the cracking detected during the inspection at Superior Tube Company occurred or propagated during the operations conducted between extrusion and vacuum annealing. These operations included roll straightening and centerless grinding.

A metallographic investigation was conducted to characterize the cracks and to determine the specific cause of them. Examination of the cracks at higher magnification (see Figure 25A) revealed that the cracks were transgranular. The cracks also seemed to be branched, which is typical of stress corrosion type cracking. The crack shown in Figure 25B, taken at a magnification of 750X, shows evidence of chemical attack.

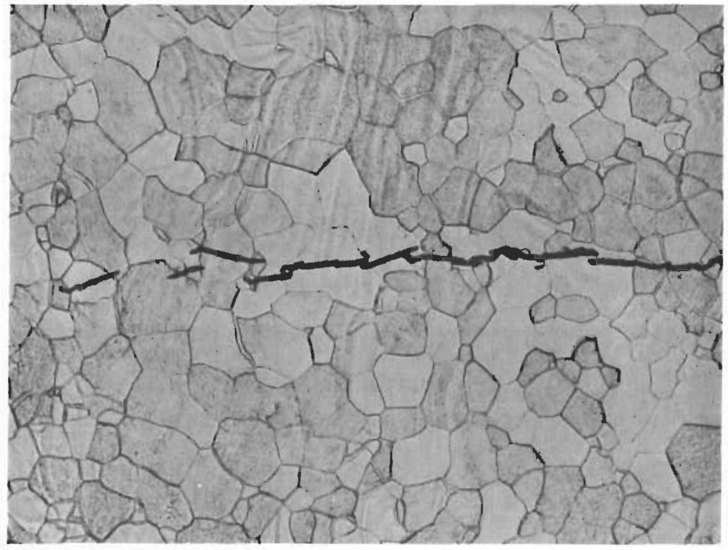
The only possible corrosive atmosphere which the tubes came in contact with was the coolant used during the centerless grinding operation. This coolant was a water soluable emulsifying cutting oil. It is described by the producer as a naphthenic base oil which is highly alkaline and contains inactive sulfur compounds with some naphthenic acid added as an emulsifier. A Fansteel Technical Data Bulletin states that tantalum is corrosive resistant, except in HF, strong alkalis and free SO_3. The coolant used contained two of these exceptions mentioned; namely, a strongly alkaline solution and free SO_3 present upon breakdown of the inert sulfur compounds. Based on these findings, it was theorized that the cracks were caused by a combination of high residual stresses built up during extrusion, roll straightening and centerless grinding and corrosive attack by the coolant used during grinding. This could be avoided in the future.

3. Vidigage Tests

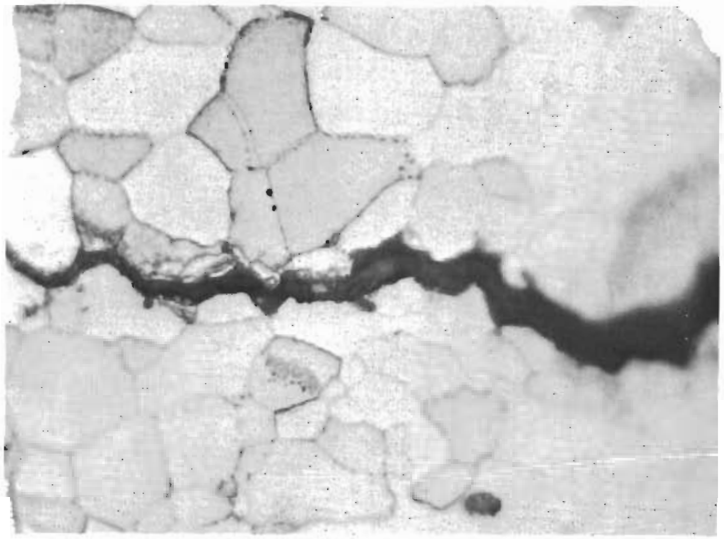
The Vidigage results, as shown in Table 7, indicated that the wall thickness variation was generally within the .025-inch total spread, which is considered to be commercial. While Tube No. 8 (Ta-10W) was badly eccentric, particularly at one end, the other tubes were satisfactory. The eccentricity on Tube No. 8 was due at least in part to excessive conditioning time on the ID belt polisher.

C. Tube Reducing

All of the tube hollows (except Nos. 8 and 10) were tube reduced to 7/8-inch OD by .109-inch wall in two steps. The initial reduction was from 2.00-inch OD by .250-inch wall to 1-3/8-inch OD by .156-inch wall (a 57 percent reduction). With the exception of Tube No. 7 (Ta-10W), the tubes generally tube reduced successfully. Tube No. 7 (Ta-10W) had severe cross cracks propagate at the front end during this initial tube reducing pass. This is shown in Figure 26.



Mag. 250X A



Mag. 750X B

Figure 25

Cracks on OD of Extruded Tube Hollow

No. 10 (T-222) Following Straightening, Centerless Grinding and Vacuum Annealing

Etchant: 100 cc H_2O, 50 cc HNO_3, 5 cc HF, 25 grams ammonium fluoride

TABLE 7

Vidigage Inspection Results for Ta-10W Tube Hollows

Tube No.	Wall Thickness (inches) Min.	Max.	Spread
2	.225	.252	.027
4A	.234	.252	.018
4B	.235	.244	.009
6	.228	.260	.032
7	.232	.250	.018
8	.204	.298	.094

Vidigage Inspection Results for T-222 Tube Hollows

Tube No.	Wall Thickness (inches) Min.	Max.	Spread
3A	.246	.260	.014
3B	.239	.253	.014
5A	.232	.252	.020
5B	.232	.252	.020
9 (8 pcs.)	.225	.251	.026
10	.230	.255	.025
11 (3 pcs.)	.230	.260	.030

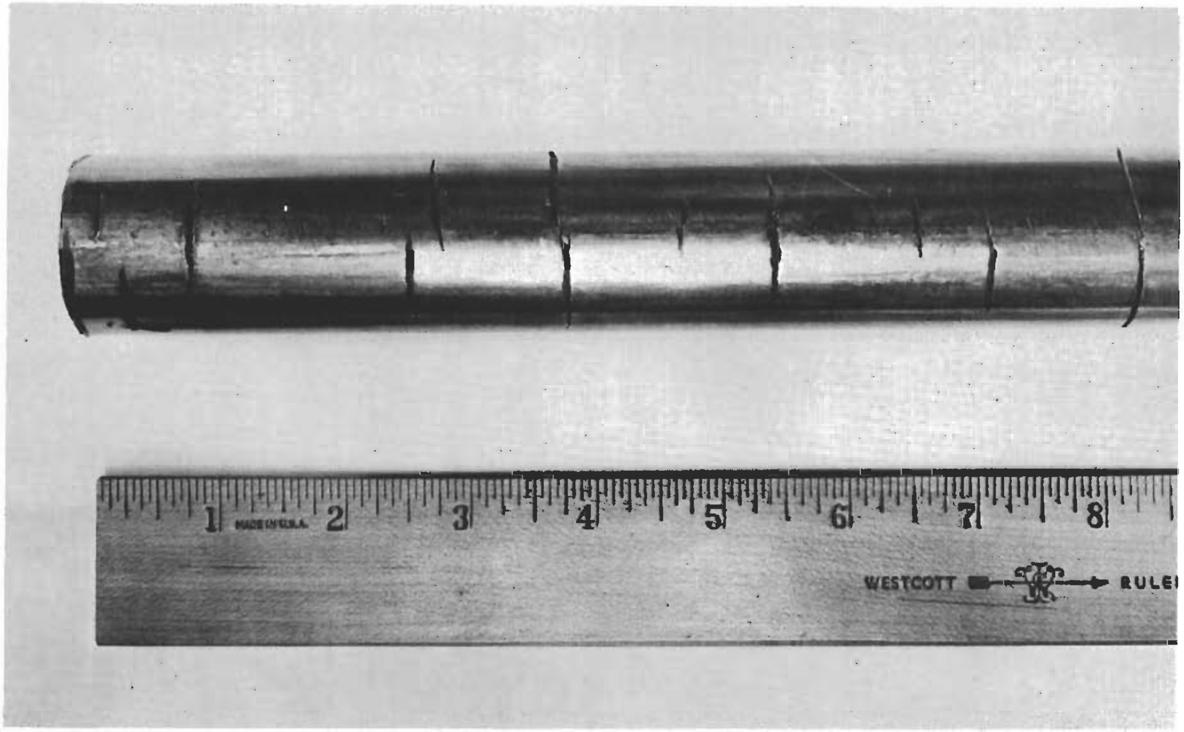


Figure 26

Ta-10W Tube No. 7 After Tube Reducing From 2.00-inch OD by .250-inch Wall to 1-3/8-inch OD by .156-inch Wall

The Ta-10W tubes were then stress relieved at 1900F for one hour and the T-222 tubes were annealed for one-fourth hour at 2800F, defective material was cropped and the tubes were given a second tube reducing pass to 7/8-inch OD by .109-inch wall. The tubes reduced very satisfactorily and Superior Tube reported that the T-222 tubes, having been fully annealed, worked more easily than the Ta-10W tubes. The results of the two tube reducing passes are summarized in Table 8. The end splitting reported is considered to be normal for both of these alloys. Most of the end splitting was minor and only a few inches of stock were lost on these tubes.

Tube No. 8 (Ta-10W) and Tube No. 10 (T-222) were reconditioned by machining. Tube No. 10 was tube reduced and numerous cracks were observed on the OD surface. A total of 28.4 pounds of acceptable material at 7/8-inch diameter was realized from a tube which weighed 81.5 pounds prior to tube reducing. Tube No. 8 was also tube reduced but severe cracking occurred and no acceptable material could be salvaged.

D. Cold Drawing

The 7/8-inch OD by .109-inch wall tubes from extruded Tube Hollow No. 6 (Ta-10W) and Tube Hollow No. 11 (T-222) were selected for the test lot sequence and divided into three sections for processing to the three finished sizes of .500-inch by .062-inch wall, .375-inch by .062-inch wall, and .250-inch by .020-inch wall. The pieces were all given a recrystallization anneal of one-fourth hour at 2800F. The pieces which were to be drawn to the .500-inch by .062-inch wall were drawn to .759-inch by .096-inch wall and the pieces to be drawn to the .375-inch by .062-inch wall and the .250-inch by .020-inch wall were drawn to .743-inch by .094-inch wall. Following this operation each of the test lots was divided into five equal parts. These five lots were identified as follows:

Lot No. 1 - Annealed temper
Lot No. 2 - Stress relieved temper
Lot No. 3 - 1/4 hard temper
Lot No. 4 - 1/2 hard temper
Lot No. 5 - Full hard temper

Tables 9 and 10 summarize, respectively, the processing sequence and results for cold drawing of the test lot tubes of Ta-10W and T-222 to each of the finished sizes. Figure 27 is a photograph of the T-222 tubes drawn to .500-inch OD by .062-inch wall.

Superior Tube Company reported that both alloys displayed good drawability and area reductions up to 62 percent were successfully accomplished without intermediate annealing. While minor in process surface conditioning was required, this is not unusual for tantalum alloys. Superior Tube reported that despite the defects in the starting material, the basic material is superior to that normally obtained from both the standpoint of drawability and formation and propagation of defects during drawing. Tantalum alloy material previously processed by Superior Tube was prone to the formation of OD defects during drawing.

TABLE 8

Processing Data
Tube Reducing to 7/8-inch OD by .109-inch wall

Operation	Size	% Reduction	Rockwell Hardness	Comments
Ta-10W Tube Hollows				
Anneal			B 94/96	15 min. @ 3100F
Inspect				
Tube Reduce	1-3/8-inch OD by .156-inch W	57		See Note 1
Stress Relieve				1 hour @ 1900F
Crop Defective Material				
Tube Reduce	7/8-inch OD by .109-inch W	56	B 93	See Note 2
Anneal				15 min. @ 2800F
T-222 Tube Hollows				
Anneal			B97/C25	15 min. @ 3100F
Inspect				
Tube Reduce	1-3/8-inch OD by .156-inch W	57		See Note 3
Anneal				15 min. @ 2800F
Crop Defective Material				
Tube Reduce	7/8-inch OD by .109-inch W	56		See Note 4
Anneal			B 103	15 min. @ 2800F

TABLE 8 (Continued)

NOTE 1: Tube reducing results for Ta-10W tubes for 1-3/8-inch reduction based on visual examination are as follows:

Tube No. 2	5 cross cracks along length of tube
Tube No. 4A	Tube reduced without difficulty
Tube No. 4B	Tube reduced without difficulty
Tube No. 6	Tube reduced without difficulty
Tube No. 7	Serious cross cracks evident on OD surface
Tube No. 8	Not tube reduced

NOTE 2: Tube reducing results for Ta-10W tubes for 7/8-inch reduction based on visual examination are as follows:

Tube No. 2	5 cross cracks from above - to be cropped
Tube No. 4A	Tube reduced without difficulty
Tube No. 4B	Tube reduced without difficulty
Tube No. 6	Minor end splitting
Tube No. 7	Minor end splitting
Tube No. 8	Not tube reduced

NOTE 3: Tube reducing results for the T-222 tube for the 1-3/8-inch reduction based on visual inspection are as follows:

Tube No. 3A	Serious end splitting
Tube No. 3B	Tube reduced without difficulty
Tube No. 5A	Minor end splitting
Tube No. 5B	Minor end splitting
Tube No. 9	7 of 8 pieces tube reduced - 4 had ID defects
Tube No. 10	Not yet tube reduced
Tube No. 11	Minor end splitting

NOTE 4: Tube reducing results for the T-222 tubes for 7/8-inch reduction based on visual inspection are as follows:

Tube No. 3A	Tube reduced without difficulty
Tube No. 3B	Tube reduced without difficulty
Tube No. 5A	Minor end splitting
Tube No. 5B	Tube reduced without difficulty
Tube No. 9	All pieces tube reduced without difficulty - ID defects still present
Tube No. 10	Not yet tube reduced
Tube No. 11	Tube reduced without difficulty

TABLE 9

Processing Data-Test Lot Sequence
Ta-10W - Tube No. 6

Operation	Size (inch)	% Red.	Rockwell Hardness	Lot No.(1)	Remarks
Starting Size: 7/8-inch OD by .109-inch W					Finish Size: .500-inch OD by .062-inch W
Layout	7/8 by .109	--	B-93		
Draw	.759 by .092	27	B102/103		
Inspect					No visible defects
Anneal			B100/103	1,2,5	2800F - 1/4 hour
Inspect				1,2,5	No visible defects
Draw	.648 by .074	31	C25/28	1,2,3,4,5	
Anneal			B-94	4	2800F - 1/4 hour
Inspect				4	Minor OD surface defects
Condition					
Draw	.549 by .062	29		1,2,3,4,5	
Anneal				3	2800F - 1/4 hour
Inspect				3	Minor OD surface defects
Condition					
Sink	.500 by (.062)	11		1,2,3,4,5	
Anneal				1	2800F - 1/4 hour
Stress Relieve				2	1800F - 1 hour
Starting Size: 7/8-inch OD by .109-inch W					Finish Size: .375-inch OD by .062-inch W
Layout	7/8 by .109		B-93		
Draw	.743 by .094	27	B102/103		One deep lap and several minor OD defects
Inspect					
Condition					

TABLE 9 (Continued)

Operation	Size (inch)	% Red.	Rockwell Hardness	Lot No.(1)	Remarks
Starting Size: 7/8-inch OD by .109-inch W			**Finish Size**: .375-inch OD by .062-inch W		
Sink	.638 by (.094)	14		1,2,3,4,5	
Anneal			B93/94	1,2,3,5	2800F - 1/4 hour
Inspect					Minor OD surface defects Numerous ID laps (scale)
Condition					
Draw	.548 by .074	30	C-23	1,2,3,4,5	
Anneal			B92/93	4	2800F - 1/4 hour
Inspect					No visible OD defects
Draw	.449 by .062	32		1,2,3,4,5	
Anneal			B92/94	3	2800F - 1/4 hour
Inspect				3	Minor OD defects
Condition					
Sink	.375 by (.062)	16		1,2,3,4,5	
Stress Relieve				2	1800F - 1 hour
Anneal				1	2800F - 1/4 hour
Starting Size: 7/8-inch OD by .109 W			**Finish Size**: .250-inch OD by .020-inch W		
Layout	7/8 by .109		B-93		
Draw	.743 by .074	27	B102/103		
Inspect					No visible defects
Draw	.650 by .075	30	C26/28	1,2,3,4,5	
Inspect				1,2,3,4,5	No visible defects
Draw	.570 by .060	28	C-30	1,2,3,4,5	
Anneal			B-95	1,2,3,4,5	2600F - 1/4 hour
Inspect				1,2,3,4,5	No visible defects
Draw	.521 by .048	26	C-25	1,2,3,4,5	

TABLE 9 (Continued)

Operation	Size (inch)	% Red.	Rockwell Hardness	Lot No. (1)	Remarks
Starting Size:	7/8-inch OD by .109-inch W		**Finish Size**:	.250-inch OD by .020-inch W	
Inspect				1,2,3,4,5	No visible defects
Draw	.476 by .038	26.5		1,2,3,4,5	
Inspect				1,2,3,4,5	No visible defects
Draw	.410 by .030	31.5		1,2,3,4,5	
Anneal				1,2,3,4,5	2800F - 1/4 hour
Inspect				1,2,3,4,5	No visible defects
Draw	.348 by .024	31.5		1,2,3,4,5	
Anneal				4	2800F - 1/4 hour
Inspect				1,2,3,4,5	ID laps
Draw	.305 by .021			1,2,3,4,5	
Inspect					OD-OK ID - laps
Anneal				3	2800F - 1/4 hour
Sink	.252 by (.021)			1,2,3,4,5	
Anneal				1	2800F - 1/4 hour
Stress Relief				2	1800F - 1 hour

NOTE (1): Lot No. 1 - Annealed Temper
Lot No. 2 - Stress Relieved Temper
Lot No. 3 - 1/4 Hard Temper
Lot No. 4 - 1/2 Hard Temper
Lot No. 5 - Full Hard Temper

TABLE 10

Processing Data-Test Lot Sequence
T-222 - Tube No. 11

Operation	Size (inch)	% Red.	Rockwell Hardness	Lot No.(1)	Remarks
Starting Size: 7/8-inch OD by .109-inch W **Finish Size:** .500-inch OD by .026-inch W					
Layout	7/8 by .109		B-103		Minor OD indications
Draw	.759 by .092	27	C-34		
Inspect					Circumferential defects
Condition					
Anneal			C-27	3,5	2800F - 1/4 hour
Draw	.678 by .074	31	C34/35	1,2,3,4,5	
Anneal			C-27	1,2,4	2800F - 1/4 hour
Inspect				1,2,3,4,5	One lap
Condition					
Draw	.549 by .062	29		1,2,3,4,5	
Anneal				3	2800F - 1/4 hour
Inspect				1,2,3,4,5	Long OD defects on one end of one tube (Tube from Lot No. 5)
Condition					
Sink	.500 by (.062)	10		1,2,3,4,5	
Anneal				1	2800F - 1/4 hour
Stress Relieve				2	1800F - 1 hour
Starting Size: 7/8-inch OD by .109-inch W **Finish Size:** .375-inch OD by .062-inch W					
Layout	7/8 by .109		B-103		One deep cross crack
Draw	.743 by .094	27	C-35		
Inspect					One deep cross crack
Condition					
Anneal			C-27	1,2,4	2800F - 1/4 hour

TABLE 10 (Continued)

Operation	Size (inch)	% Red.	Rockwell Hardness	Lot No.(1)	Remarks
Starting Size:	7/8-inch OD by .109-inch W		**Finish Size**:		.375-inch OD by .062-inch W
Sink	.638 by (.094)	14		1,2,3,4,5	
Anneal			C25/26		2800F - 1/4 hour
Inspect					ID laps - OD all right
Draw	.548 by .074	30	C-32	1,2,3,4,5	
Anneal			C25/26	1,2,4	2800F - 1/4 hour
Inspect					No visible defects
Draw	.449 by .062	32		1,2,3,4,5	
Anneal			C-26	3	2800F - 1/4 hour
Inspect				1,2,3,4,5	Minor OD defects
Condition					
Sink	.375 by (.062)	16		1,2,3,4,5	
Anneal				1	2800F - 1/4 hour
Stress Relieve				2	1800F - 1 hour
Starting Size:	7/8-inch OD by .109-inch W		**Finish Size**:		.250-inch OD by .020-inch W
Layout	7/8 by .109		B-103		Minor OD indications
Draw	.743 by .094	27	C-35		
Inspect					No visible defects
Draw	.650 by .075	30	C-35	1,2,3,4,5	
Anneal			C-27	1,2,3,4,5	2800F - 1/4 hour
Inspect					1 long deep defect - 1 minor defect
Condition					
Draw	.570 by .060	28	C-35	1,2,3,4,5	
Inspect					No visible defects
Draw	.521 by .048	28	C35/36	1,2,3,4,5	

TABLE 10 (Continued)

Starting Size: 7/8-inch OD by .109-inch W **Finish Size:** .250-inch OD by .020-inch W

Operation	Size (inch)	% Red.	Rockwell Hardness	Lot No.(1)	Remarks
Anneal			C-26	1,2,3,4,5	2800F - 1/4 hour
Inspect				1,2,3,4,5	Minor OD surface defects
Condition					
Draw	.476 by .038	26.5	C34/35	1,2,3,4,5	
Inspect					No visible defects
Draw	.410 by .030	31.5		1,2,3,4,5	
Anneal				1,2,3,4,5	2800F - 1/4 hour
Inspect				1,2,3,4,5	No visible defects
Draw	.348 by .024	31.5		1,2,3,4,5	
Anneal				4	2800F - 1/4 hour
Inspect				1,2,3,4,5	ID laps
Draw	.305 by .021			1,2,3,4,5	
Inspect					OD-OK ID - Laps
Anneal				3	2800F - 1/4 hour
Sink	.252 by (.021)			1,2,3,4,5	
Anneal				1	2800F - 1/4 hour
Stress Relieve				2	1800F - 1 hour

NOTE (1): Lot No. 1 - Annealed Temper
Lot No. 2 - Stress Relieved Temper
Lot No. 3 - 1/4 Hard Temper
Lot No. 4 - 1/2 Hard Temper
Lot No. 5 - Full Hard Temper

Figure 27

Test Lot Sequence T-222 Tubing at Finished Size of .500-inch OD by .062-inch Wall

Tubing from Extrusion No. 6

The yield results as shown in Table 11 were comparable to those normally attained for these sizes and quantities. Because of the extensive destructive testing required, the table indicates finished weight in addition to the weight actually shipped to Allegheny Ludlum for elevated temperature tensile and stress rupture testing.

E. Testing of Test Lot Sequence Tubing

The test lot sequence tubing produced during Phase III of the program was evaluated in terms of all the routine non-destructive and destructive tests listed in Section IV-A on Page 16.

1. Ultrasonic and Eddy Current Tests

The ultrasonic and eddy current test results are shown in Table 12. While the final inspection results showed no trend with regard to quality versus size, the quality of the T-222 is generally better than that for the Ta-10W alloy. This is a reflection on the comparative quality of the raw material. Results also show that the T-222 could be subjected to the same processing sequence as Ta-10W and provide comparable results in the finished tube.

2. Metallographic Examination

Table 13 shows the results of the metallographic examination of the finished tubes. Each specimen was evaluated in terms of microhardness, precipitate reading, grain size determination, structure and defects. The metallographic examination revealed that the ultrasonic and eddy current test indications were primarily due to ID laps. This condition is illustrated in Figure 28, which reveals an ID lap to a depth of .020-inch in the .500-inch OD by .062-inch wall Ta-10W tubing.

3. Vidigage Tests

Final wall dimensions, as reported in Table 14, indicate that the percent variation in the finished tubing is generally consistent with that of the raw material. Since the material from each size was cut from one original tube hollow, it would be expected that the over-all variation for a given size would be less than the over-all variation in the starting tube.

4. Room Temperature Tensile Tests

Room temperature tensile properties of the test lot sequence tubes are presented in Table 15. The data was obtained on the tubes from both alloys in all three sizes and each of the five temper conditions. Comparative results are shown graphically in Figures 29, 30 and 31. Both alloys exhibited satisfactory ductility for their respective strength levels. Since elongation values are normally lower in thinner sections, the elongation values for the .250-inch OD by .020-inch wall sizes were not included with Figure 31. Surprisingly, Ta-10W had a higher work hardening rate than T-222. This is illustrated in Figure 32. The percent increase in

TABLE 11

Yield Results

Alloy	Size	Layout Weight (lbs)	Finished* Weight (lbs)	Finished* Yield (%)	Shipped Weight (lbs)	Shipped Yield (%)
Ta-10W	.500" x .062"	24.6	17.0	69.0	14.2	57.7
T-222	.500" x .062"	27.4	16.3	59.5	14.3	52.2
Ta-10W	.375" x .062"	19.0	12.7	66.8	9.9	51.8
T-222	.375" x .062"	21.4	12.6	61.2	10.6	49.5
Ta-10W	.250" x .020"	14.9	9.3	62.4	7.3	49.0
T-222	.250" x .020"	16.8	9.0	53.5	7.0	41.7
Ta-10W	Over-all	58.5	39.0	66.7	31.4	53.7
T-222	Over-all	65.6	37.9	57.8	32.9	50.2

*Finished yield figures represent weight prior to sampling for destructive tests.

TABLE 12

Ultrasonic and Eddy Current Test Results(1)

Alloy	Size	Temper	Ultrasonic Indications	Eddy Current Indications
Ta-10W Tube No. 6	.500" x .062"	Annealed	ID indications entire tube	Not tested(2)
		Stress Relieved	ID indications entire tube	Not tested(2)
		1/4 hard	2 indications	Not tested(2)
		1/2 hard	No indications	Not tested(2)
		Full hard	ID indications entire tube	Not tested(2)
Ta-10W Tube No. 6	.375" x .062"	Annealed	No indications	No indications
		Stress Relieved	One indication	No indications
		1/4 hard	No indications	No indications
		1/2 hard	3 indications	3 indications
		Full hard	No indications	1 indication
Ta-10W Tube No. 6	.250" x .020"	Annealed	1 indication	1 indication(3)
		Stress Relieved	4 indications	4 indications
		1/4 hard	No indications	No indications
		1/2 hard	2 indications	No indications
		Full hard	1 indication	1 indication
T-222 Tube No. 11	.500" x .062"	Annealed	No indications	Not tested(2)
		Stress Relieved	No indications	Not tested(2)
		1/4 hard	No indications	Not tested(2)
		1/2 hard	No indications	Not tested(2)
		Full hard	1 indication	Not tested(2)
T-222 Tube No. 11	.375" x .062"	Annealed	No indications	No indications
		Stress relieved	No indications	No indications
		1/4 hard	No indications	No indications
		1/2 hard	No indications	No indications
		Full hard	No indications	No indications
T-222 Tube No. 11	.250" x .020"	Annealed	1 indication	1 indication
		Stress relieved	1 indication	1 indication
		1/4 hard	1 indication	1 indication
		1/2 hard	No indications	1 indication
		Full hard	No indications	No indications

(1) Notch Standard 1/2" long x .004" deep - .500" x .062" size
1/2" long x .002" deep - .250" x .020" size

(2) Eddy current test not generally performed on tubing of this size because of high background noise level.

(3) Ultrasonic and eddy current indications not in same area.

TABLE 13

Metallographic Results
Finished Tubes - Test Lot Sequence

Alloy	Size	Temper(1)	Hardness(2)	Precipitate Reading(3)	Grain Size	Structure	Defects OD	Defects ID
Ta-10W	.500 x .062W	Annealed	B94-98	C-1	ASTM 6	Recrystallized	None	None
		Stress Relieved	C-32-33	C-4 to C-5	ASTM 7	Very Elongated Grains	None	Laps to .004
		1/4 Hard	C-29	C-1	ASTM 6	Slightly Elongated	None	1 Lap to .0065
		1/2 Hard	C-29-30	C-1	ASTM 6	Elongated	None	1 Lap to .0015
		Full Hard	C-34-35	C-1		Fibrous	1 Lap .0008	1 Lap to .012
Ta-10W	.375 X .062W	Annealed	C-20-21	C-1	ASTM 6	Recrystallized-Very Slightly Elongated	None	1 Lap to .008
		Stress Relieved	C-34	Mid Wall C-1 Band of C-4 on OD & ID	-	Fibrous	None	1 Lap to .001
		1/4 Hard	C-34-37	C-1	ASTM 6	Slightly Elongated	None	1 Lap to .020
		1/2 Hard	C-32-36	C-1	ASTM 6.5	Elongated	None	1 Lap to .022
		Full Hard	C-36-37	C-1	-	Fibrous	None	1 Lap to .005
Ta-10W	.250 x .020W	Annealed	B-96	C-1	ASTM 6.5-7	Recrystallized	None	1 Lap to .0015
		Stress Relieved	C-32-33	C-3 to C-4	-	Fibrous	None	None
		1/4 Hard	C-31-32	C-1	ASTM 6.5-7	Very Slightly Elongated	None	None
		1/2 Hard	C-34-35	C-1	ASTM 6.5-7	Elongated	None	1 Lap to .005
		Full Hard	C-35	C-1	-	Fibrous	None	None
T-222	.500 x .062W	Annealed	C-24-25	C-3	ASTM 6.5	Recrystallized	None	Laps to .003
		Stress Relieved	C-37-42	C-4 to C-5	ASTM 6.5-7	Very Slightly Elongated	None	Laps to .002
		1/4 Hard	C-37-39	C-3 to C-4	ASTM 6.5	Recrystallized-Very Slightly Elongated	None	Laps to .002
		1/2 Hard	C-39	C-4	ASTM 6.5	Elongated	None	Laps to .0025
		Full Hard	C-39	C-4 to C-5	ASTM 6.5	Elongated	None	Laps to .005
T-222	.375 x .062W	Annealed	C-28-31	C-2 to C-3	ASTM 7	Equiaxed	None	None
		Stress Relieved	C-40-41	C-5	ASTM 7	Elongated	None	1 Crack to .0004
		1/4 Hard	C-40	C-2 to C-3	ASTM 7	Slightly Elongated	1 Crack to .005	1 Crack to .0025
		1/2 Hard	C-38-39	C-3 to C-4	ASTM 7-8	Elongated	None	1 Lap to .0025
		Full Hard	C-40-41	C-4	ASTM 7-8	Very Elongated	None	1 Lap to .0025
T-222	.250 x .020W	Annealed	C-29-30	C-5	ASTM 7.5	Equiaxed	None	None
		Stress Relieved	C-40-41	C-6	ASTM 7-8	Very Elongated	None	None
		1/4 Hard	C-40	C-4	ASTM 7	Very Slightly Elongated	None	None
		1/2 Hard	C-37-40	C-4 to C-5	ASTM 6.5-7	Slightly Elongated	None	None
		Full Hard	C-41	C-6	ASTM 7-8	Very Elongated	None	None

(1) Annealed = 15 min. @ 2800F
Stress Relieved = 1 hour @ 1800F
1/4 Hard = Annealed + 1 draw pass
1/2 Hard = Annealed + 2 draw passes
Full Hard = Annealed + 3 draw passes

(2) Hardness taken in Knoop and converted to Rockwell

(3) C-1 = No precipitate C-6 = Large amount of precipitate

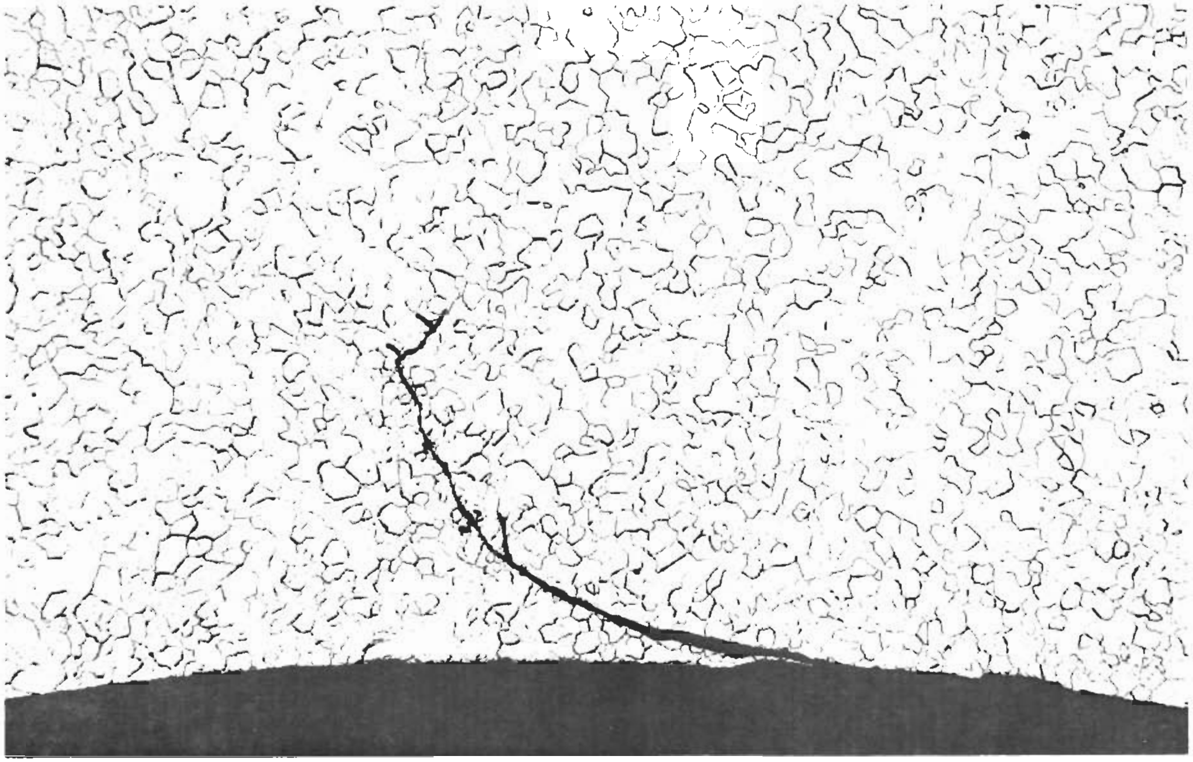


Figure 28

Transverse Section Showing ID Lap
to a Depth of .020-inch in Finished Tube

.500-inch OD by .062-inch Wall

TABLE 14

Variation in Wall Thickness for Finished Tubing

Alloy	Size	Wall Dimensions Min.	Wall Dimensions Max.	Wall Dimensions Spread	Spread on Tube at 2" x .250"
Ta-10W Tube No. 6	.500" x .062"	.0587"	.0616"	.0029" (4.8%)	.032" (13%)
	.375" x .062"	.0575"	.064"	.0065" (10.8%)	.032" (13%)
	.250" x .020"	.0200"	.0208"	.008" (4%)	.032" (13%)
T-222 Tube No. 11	.500" x .062"	.0560"	.0633"	.0073" (12.1%)	.030" (12%)
	.375" x .062"	.060"	.0635"	.0035" (5.8%)	.030" (12%)
	.250" x .020"	.0183"	.0217"	.0034" (17%)	.030" (12%)

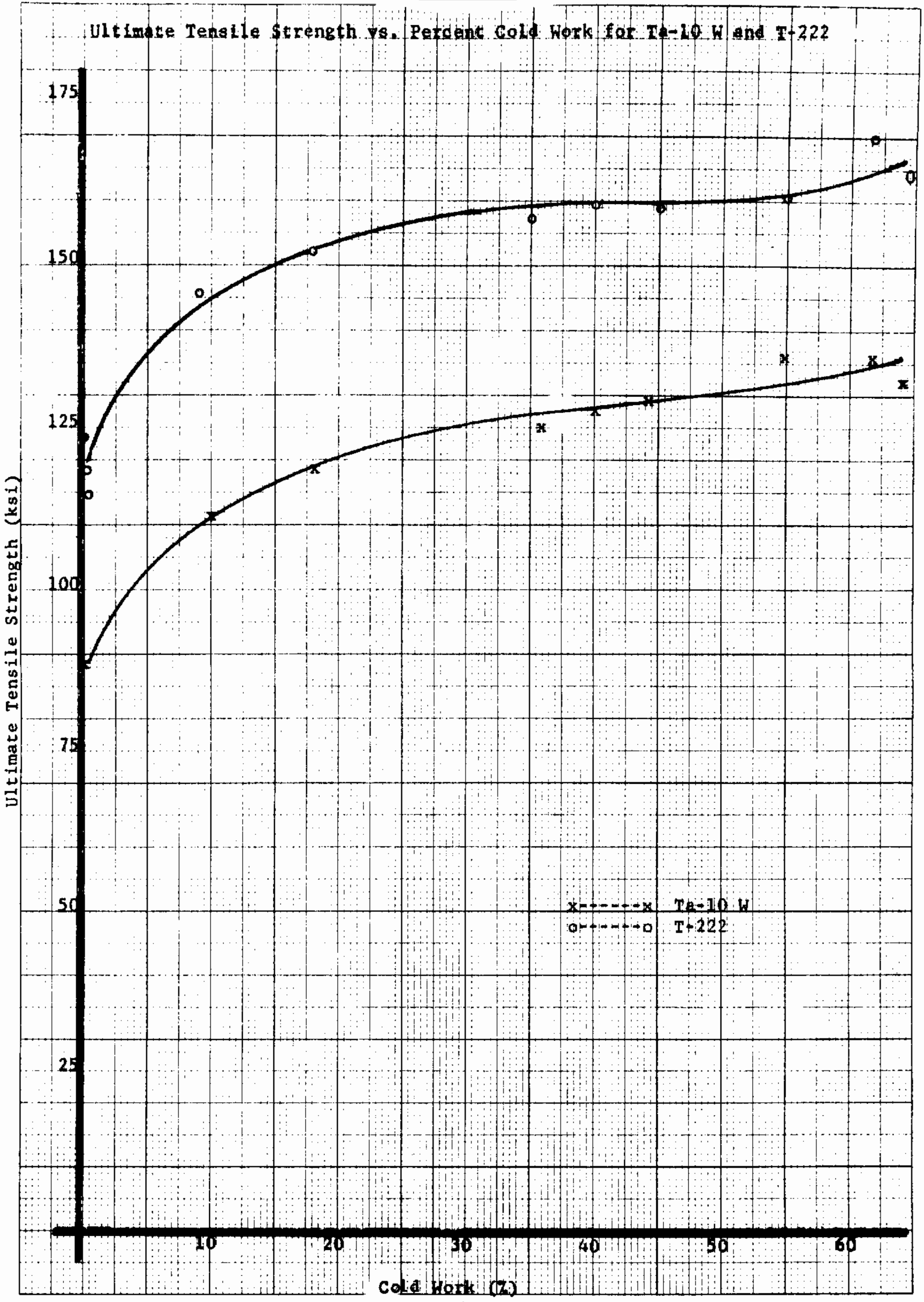
TABLE 15

Room Temperature Tensile Properties Finished Tubes - Test Lot Sequence

Alloy	Size	Temper(1)	Tensile Yield Strength(ksi)	Ultimate Tensile Strength(ksi)	Percent Elongation in 2"
Ta-10W	.500 x .062W	Annealed	74.3	89.6	38
		Stress Relieved	125.1	131.5	21
		1/4 Hard	110.4	112.1	15
		1/2 Hard	123.0	125.0	13
		Full Hard	129.3	136.4	11
	.375 x .062W	Annealed	71.7	87.0	34
		Stress Relieved	113.1	121.8	20
		1/4 Hard	113.8	118.8	14
		1/2 Hard	123.2	127.6	12
		Full Hard	128.5	132.5	12
	.250 x .020W	Annealed	73.5	88.0	30
		Stress Relieved	116.8	126.9	14
		1/4 Hard	114.8	118.7	8
		1/4 Hard	123.0	127.1	7
		Full Hard	134.0	136.7	8
T-222	.500 x .062W	Annealed	109.1	123.1	25
		Stress Relieved	153.6	161.2	16
		1/4 Hard	142.9	145.1	13
		1/2 Hard	153.7	157.6	13
		Full Hard	155.3	160.8	13
	.375 x .062W	Annealed	108.3	118.1	26
		Stress Relieved	151.2	160.9	16
		1/4 Hard	146.9	152.2	12
		1/2 Hard	152.1	159.5	11
		Full Hard	157.0	164.0	10
	.250 x .020W	Annealed	108.0	115.0	24
		Stress Relieved	148.2	157.0	13
		1/4 Hard	146.4	152.2	7
		1/2 Hard	152.3	160.0	8
		Full Hard	161.1	170.3	8

(1) Annealed = 15 min. @ 2800F
Stress Relieved = 1 hour @ 1800F
1/4 Hard = Annealed + 1 draw pass
1/2 Hard = Annealed + 2 draw passes
Full Hard = Annealed + 3 draw passes

Figure 29



Ultimate Tensile Strength vs. Percent Cold Work for Ta-10 W and T-222

Figure 30

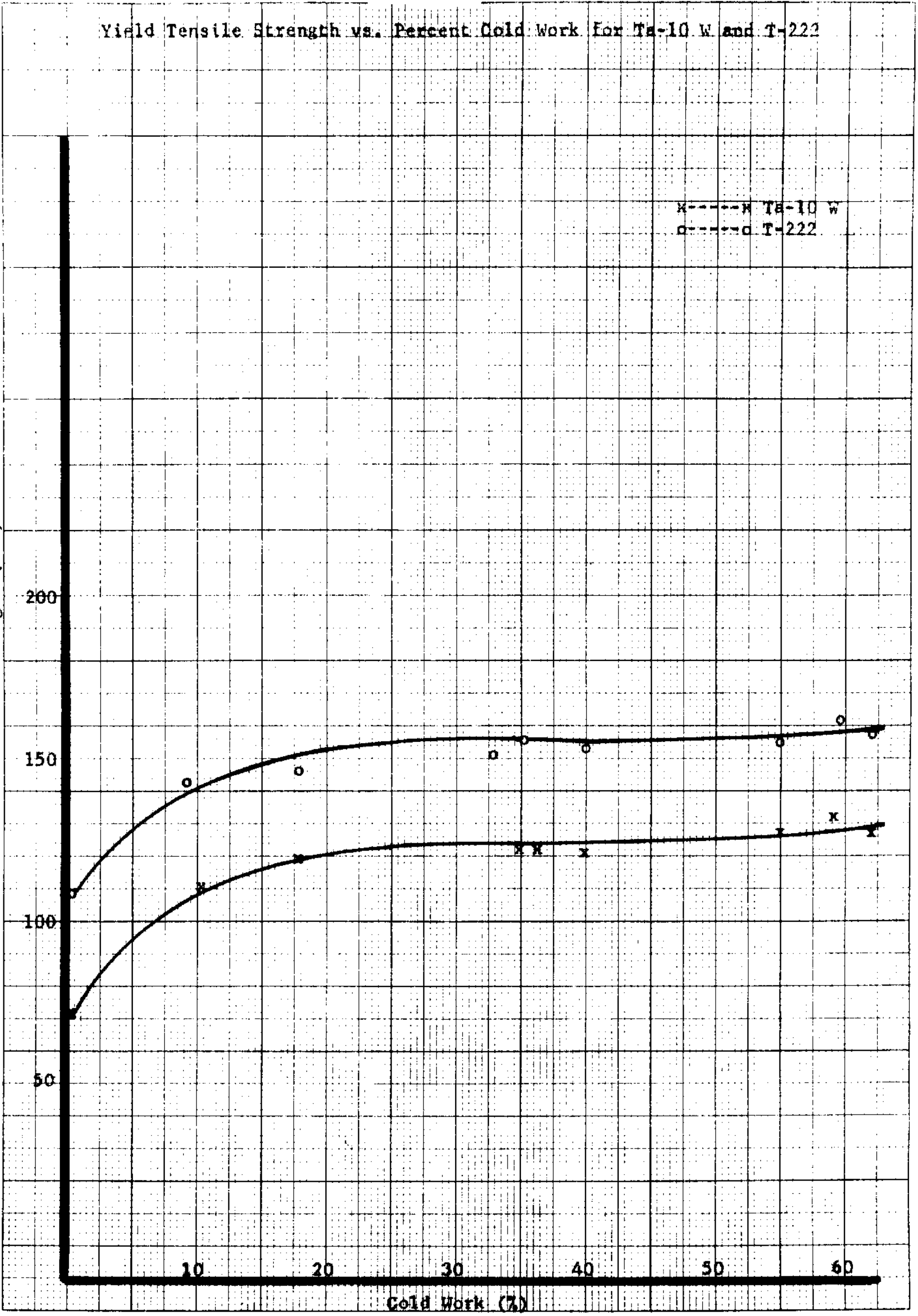

Yield Tensile Strength vs. Percent Cold Work for Ta-10 W and T-222
x------x Ta-10 W
o------o T-222
Yield Strength (ksi)
200
150
100
50
10
20
30
40
50
60
Cold Work (%)

Figure 31

Elongation vs. Percent Cold Work for Ta-10 W and T-222

.500" x .062" and .375" x .062" Sizes

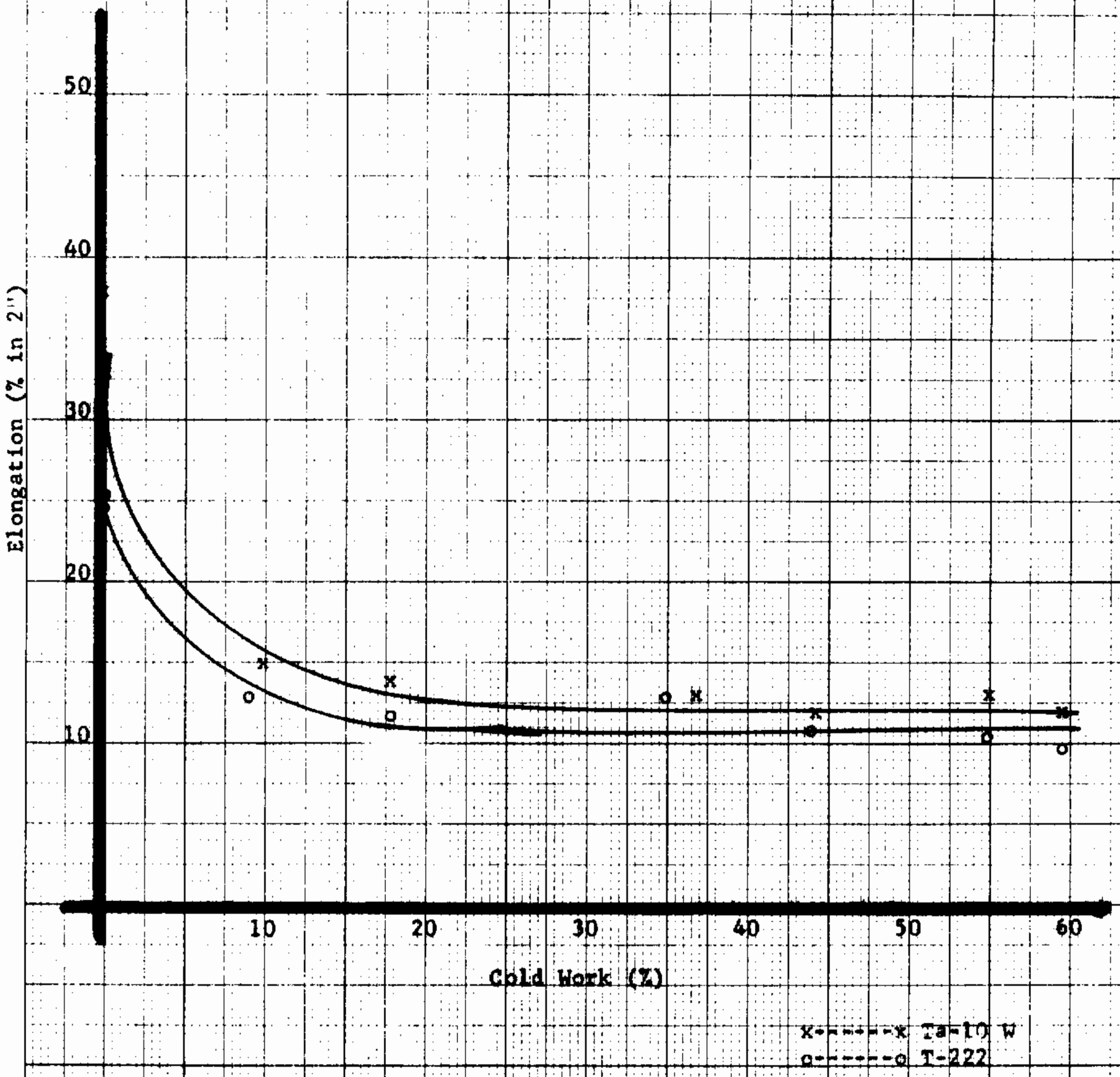
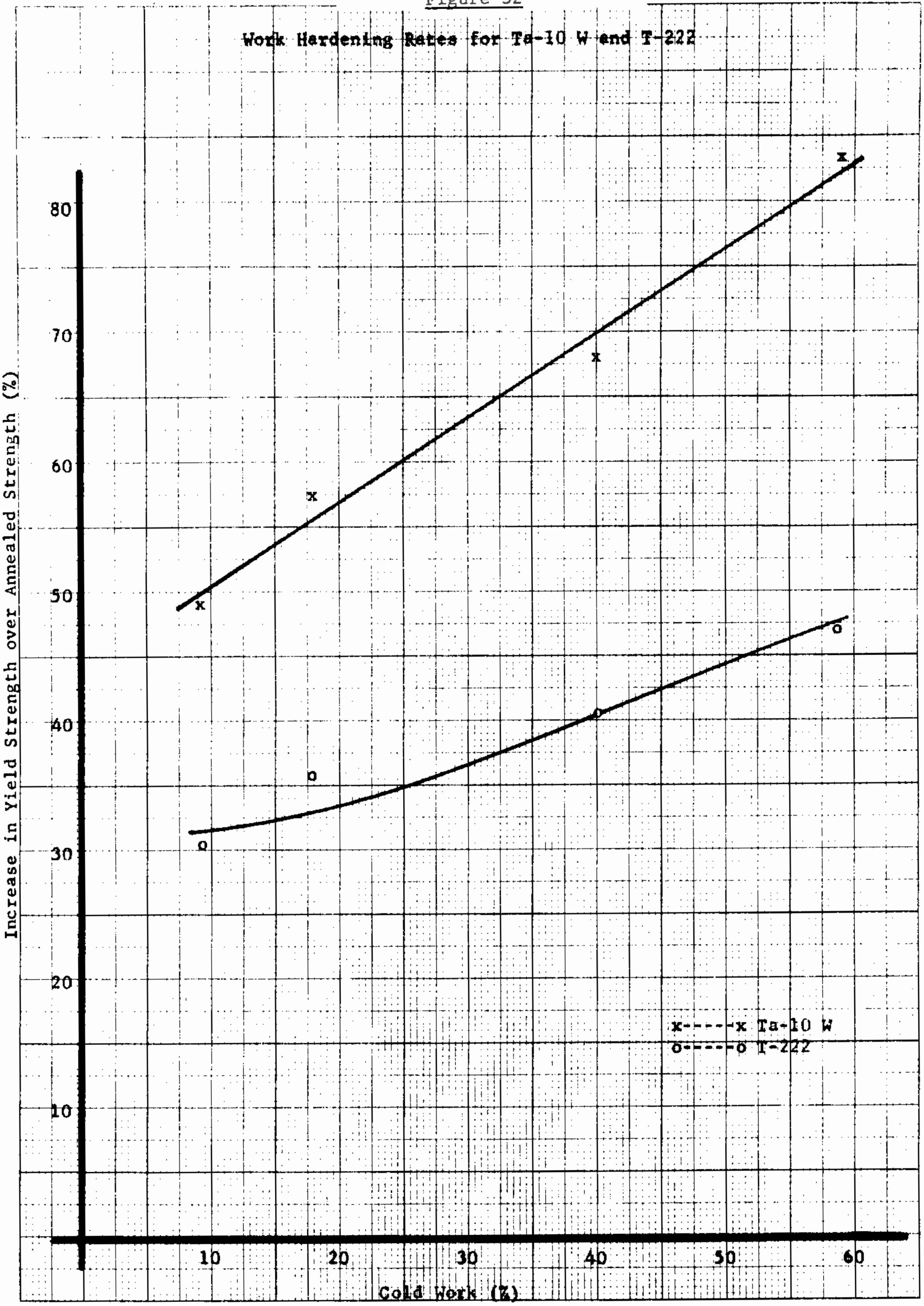


Figure 32


Work Hardening Rates for Ta-10 W and T-222
Increase in Yield Strength over Annealed Strength (%)
80
70
60
50
40
30
20
10
10
20
30
40
50
60
Cold Work (%)
x-----x Ta-10 W
o-----o T-222

yield strength with cold work is greater for the Ta-10W alloy than for the T-222. In the annealed condition, the yield strength of Ta-10W is 68 percent of that for the T-222, whereas, after 55 percent cold work, it is 83 percent of the yield strength for T-222.

5. Flaring, Flattening and Bend Tests

Flaring, flattening and bend tests indicated that both alloys exhibit good ductility even with some degree of cold work. As shown in Table 16, the two alloys appear to be equivalent with respect to flaring and flattening. However, some unexplainable inconsistencies are evident. In the 1/2 hard temper the T-222 flares to a greater degree than the Ta-10W, a fact which might have been explained by its lower work hardening rate; however, the situation was reversed for the full hard temper. All tempers of each alloy were successfully bent 180 degrees around a 3/4-inch diameter mandrel.

6. Hydrostatic, Burst and Leak Tests

The hydrostatic and burst test results are presented in Table 17. The burst properties were generally consistent with the tensile properties. In the annealed condition the burst pressure for Ta-10W was 76 percent of that for the T-222, whereas, in the full hard condition, the burst strengths were equal. The hydrostatic tests were performed only on the .250-inch OD by .020-inch wall tubes as the strength for the two larger sizes exceeded the capability for testing.

All tubes were helium leak tested successfully.

7. Chemical Analyses

Chemical analyses for the interstitial elements carbon, oxygen, hydrogen and nitrogen were conducted on the finished tubes of both alloys. The .500-inch by .062-inch and .375-inch by .062-inch wall tubes were checked in the annealed condition, while the .250-inch by .020-inch wall, which involved the most operations, were analyzed in all five temper conditions. The analyses are shown in Table 18.

8. Surface Roughness Determination

OD surface roughness determinations were made on finished tubes from each alloy in all three finished tube sizes. Typical surface readings are presented in Table 19. It will be noted that the surface finish of the T-222 tubes is slightly better than the Ta-10W and as would be expected, the smaller diameter tubing, because of the additional cold drawing performed on it, displayed slightly better surfaces.

TABLE 16

Flaring and Flattening Evaluation(1)

Alloy	Temper	Flare (increase in Diameter		Flatness	
		Test 1	Test 2	Test 1	Test 2
Ta-10W	Annealed	32%	44%(2)	3X Wall	2.5X Wall
	Stress Relieved	8%	8%	11X Wall	11X Wall
	1/4 Hard	40%	25%	6X Wall	4.5X Wall
	1/2 Hard	7%	7%	6X Wall	- (3)
	Full Hard	4%	4%	11.5X Wall	9.5X Wall
T-222	Annealed	46%(2)	46%(2)	3X Wall	2.5X Wall
	Stress Relieved	10%	0	11.5X Wall	11X Wall
	1/4 Hard	41%	34%	9X Wall	3.5X Wall
	1/2 Hard	40%	37%	11X Wall	9X Wall
	Full Hard	0	0	12X Wall	10X Wall

(1) Tests performed on .250" x .020" sizes only.

(2) Specimen buckled - flared end did not fail.

(3) Specimen failed - did not flatten.

TABLE 17

Hydrostatic and Burst Test Results(1)

Alloy	Temper	Hydrostatic Proof Test (psi)	Burst Pressure (psi)
Ta-10W	Annealed	9,000	19,000
	Stress Relieved	14,000	23,000
	1/4 hard	14,300	23,000
	1/2 hard	15,300	24,000
	Full hard	16,600	30,000
T-222	Annealed	13,500	25,000
	Stress Relieved	18,400(2)	18,400(2)
	1/4 hard	18,000	28,000
	1/2 hard	19,000	30,000
	Full hard	20,000	30,000

(1) Strength of .500" x .062" and .375" x .062" sizes exceeded capability for testing. Test performed only on .250" x .020" Wall sizes.

(2) Tube failed during hydrostatic test.

TABLE 18

Chemical Analysis on Finished Tubing

Alloy	Size	Temper	Interstitial Analysis (ppm)(1)			
			C	O	H	N
Ta-10W	.500" x .062"	Annealed	20	220	<5	8
Ta-10W	.375" x .062"	Annealed	<20	140	<5	8
Ta-10W	.250" x .020"	Annealed	24	160	<5	20
Ta-10W	.250" x .020"	Stress Relieved	<20	150	<5	8
Ta-10W	.250" x .020"	1/4 hard	40	280	<5	10
Ta-10W	.250" x .020"	1/2 hard	40	120	5	17
Ta-10W	.250" x .020"	Full hard	24	110	<5	20
T-222	.500" x .062"	Annealed	120	130	<5	28
T-222	.375" x .062"	Annealed	120	110	<5	12
T-222	.250" x .020"	Annealed	120	190	<5	8
T-222	.250" x .020"	Stress Relieved	125	230	<5	44
T-222	.250" x .020"	1/4 hard	90	270	5	35
T-222	.250" x .020"	1/2 hard	120	160	<5	8
T-222	.250" x .020"	Full hard	110	230	<5	48

Raw Material(2)

	C	O	H	N
Ta-10W	<20	120	<5	10
T-222	115	110	<5	27

(1) Analyses for O, H and N performed by National Spectrographic Laboratory, Cleveland, Ohio. Carbon analysis performed at Superior Tube Company.

(2) Sample taken by STC at 1-3/8" OD x .156" Wall - annealed condition.

TABLE 19

Surface Roughness of Finished Tubes

Alloy	Tube Size (inches)	Surface Finish RMS	
		Longitudinal Direction	Transverse Direction
Ta-10W	.500" x .062" Wall	17-24	16
	.375" x .062" Wall	17-21	18
	.250" x .020" Wall	15-20	17
T-222	.500" x .062" Wall	16-20	16
	.375" x .062" Wall	13-17	15
	.250" x .020" Wall	11-14	12

9. Weldability Tests

Weldability tests were conducted on the .250-inch by .020-inch wall tubes of both alloys. Specimens were machined to square the ends, degreased, and butted together in a special rigging inside a welding chamber. The chamber was evacuated to below one micron of Hg, purged and filled with helium and the specimens were successfully welded using a tungsten electrode. Clean, bright, crack-free welds were obtained. Figure 33 is a photomicrograph showing the structure and hardness of the weld area, heat affected zone and the base metal. Room temperature tensile tests were conducted on the welded specimens. Specimens were tested at a strain rate of .005 inches/inch/minute through the .2 percent yield point and .25 inches/inch/minute to fracture. The data obtained is presented in Table 20. All specimens ruptured in the weld area. While the tensile strengths, as compared to the room temperature tensile properties reported in Table 15, are only slightly lower than the base metal the ductilities are considerably lower.

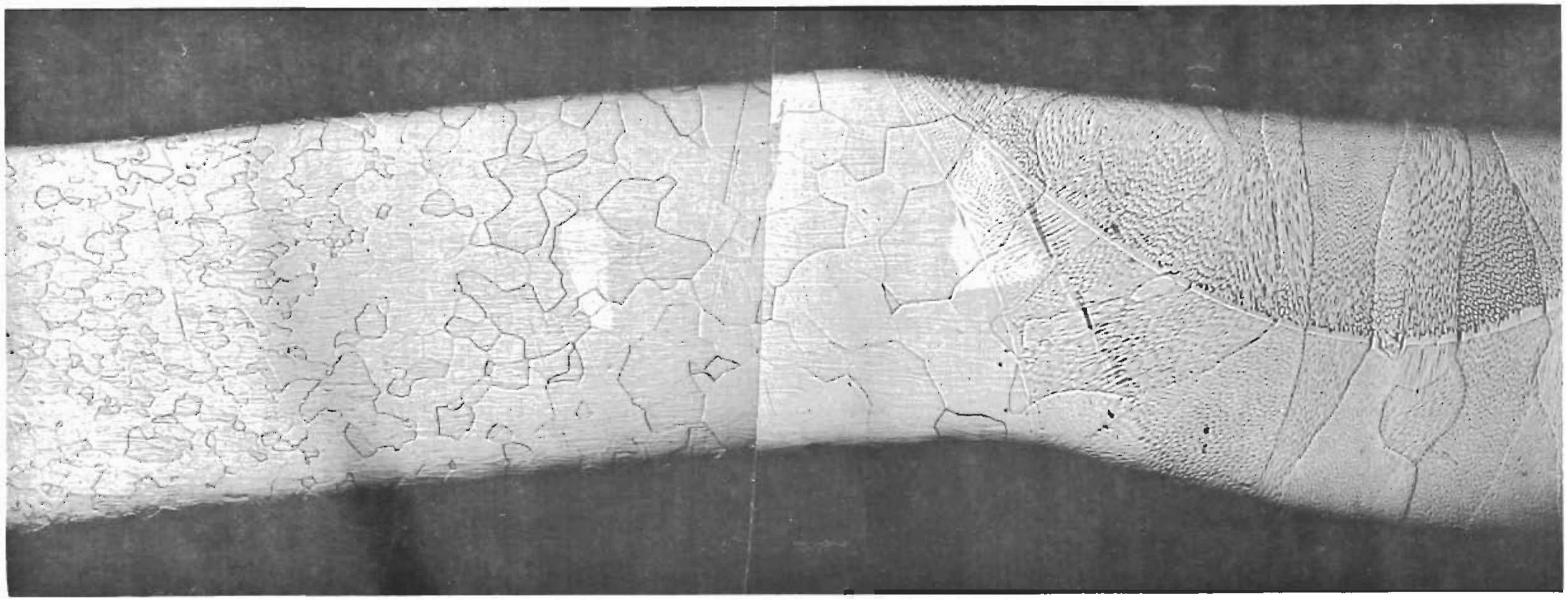
10. Elevated Temperature Tensile and Stress Rupture Tests

Elevated temperature tensile and stress rupture tests were conducted at Metcut Research Associates on material from both alloys, in all sizes and temper conditions. The tests were conducted at temperatures of 2100F, 2400F and 2700F.

a. Testing Procedures

It was originally intended that all three tube sizes would be tested in the full section. However, the high strength levels found in the initial tests on the 1/4-inch diameter tubes indicated that it would be impossible to test the 1/2-inch and 3/8-inch OD by .062-inch wall tubes in this manner. It was mutually decided by representatives of Metcut Research Associates, Allegheny Ludlum and the Air Force that in order to reduce the cross-sectional area of the test specimen parallel flats would be machined in the two larger diameter tubes prior to testing. The 1/4-inch diameter tubes were tested in the full section. Figure 34 contains drawings of the test specimens from all three tube sizes. The same specimen design was used for both the tensile and stress rupture tests.

In both the tensile and rupture furnaces, specimens are heated to the normal test temperature in about fifteen minutes. After a fifteen minute stabilization period, tensile tests were run at controlled strain rates. All specimens were tested at a strain rate of .005 inch/inch/minute through the 0.2 percent yield point. The head rate to failure for all specimens except those from the 3/8-inch and 1/4-inch OD tubes of the T-222 alloy was .01 inch/minute. The head rate to failure for the specimens from the 3/8-inch OD and 1/4-inch OD T-222 tubes was .05 inch/minute. In starting the rupture tests, the load was applied gradually and continuously over a ten to fifteen-second interval.



Base Metal
Hardness 260 DPH

Heat Affected Zone
Hardness 220-223 DPH

Weld
Hardness 240-243 DPH

Figure 33

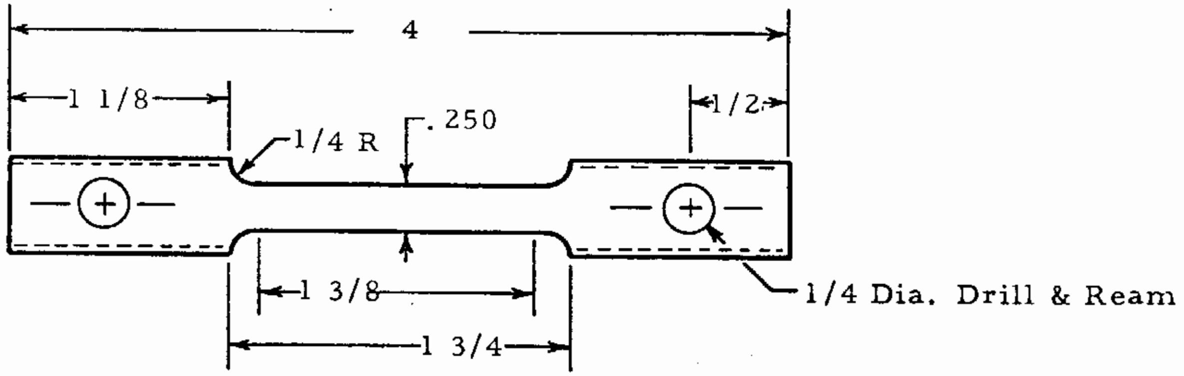
Composite Photograph Showing Microstructure of Welded Ta-10W Tube .250-inch OD by .020-inch Wall

TABLE 20

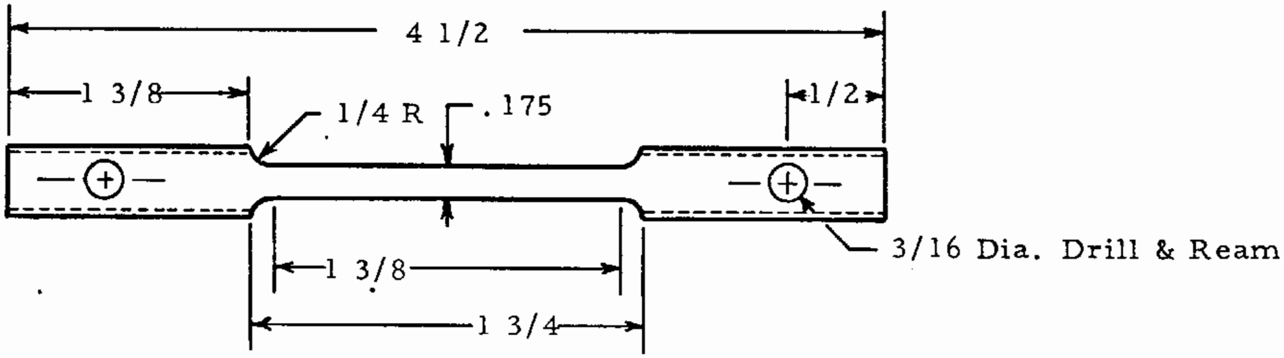
Room Temperature Tensile Tests on Weldability Specimens from .250-inch OD by .020-inch Wall Tantalum Alloy Tubes

Alloy	Condition	UTS (ksi)	.2% YS (ksi)	Ex 10^{-6}	Elong. (%)
Ta-10W	Annealed	68.1	68.1*	28.1	.6
		79.5	72.9	28.8	6.0
	1/4 Hard	92.3	81.7	31.7	4.0
T-222	Annealed	100.4	98.9	31.9	4.0
		100.2	98.6	28.3	4.0

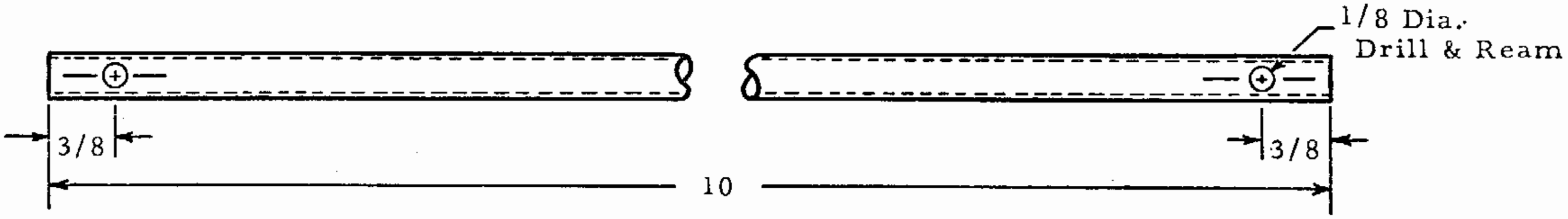
*NOTE - Broke at Yield



1/2-inch OD by .062-inch W Tube Specimen



3/8-inch OD by .062-inch W Tube Specimen



1/4-inch OD by .020-inch W Tube Specimen

Figure 34

Drawings of Tensile and Stress Rupture Test Specimen

b. Test Results

Ta-10W Alloy

The results of the elevated temperature tensile tests are presented in Table 21. The aim properties for the Ta-10W alloy at 2400F, as specified by the contract, were 38,000 psi ultimate tensile strength and 27,000 psi .2 percent yield strength. These properties were met or exceeded by the material from all three sizes in the full hard, 1/2 hard and stress relieved conditions. At 2400F, the properties of the material in the 1/4 hard were only slightly lower than the aim properties, while at 2700F the material in the 1/4 hard condition generally displayed strengths higher than those obtained on material in any of the other conditions. The material in the fully annealed condition had the best ductilities as measured by percent elongation. However, this was at a considerable sacrifice in strength. At 2400F the material in the stress relieved condition displayed the best combination of strength and ductility. The properties obtained for Ta-10W at 2400F on material in the stress relieved condition are:

Ultimate Strength - 45 to 49 ksi
.2% Yield Strength - 27 to 37 ksi
% Elongation - 20 to 25

Stress rupture data for the Ta-10W alloy is presented in Table 22. Stresses were calculated to yield rupture lives in the neighborhood of 20 hours. At 2400F this was about 18 ksi for the Ta-10W material in the stress relieved condition.

T-222 Alloy

Because of the experimental nature of the T-222 alloy at the start of this contract no aim properties were specified. The results of the elevated temperature tensile tests on the T-222 alloy tubes are presented in Table 23. Again, the best combination of strength and ductility at 2400F was obtained by the material in the stress relieved condition. The properties obtained were in the following range:

Ultimate Tensile Strength - 56 to 63 ksi
.2% Yield Strength - 44 to 50 ksi
% Elongation - 30 to 79

In both alloys there was good correlation among the data obtained from all three tube sizes. Figure 35 is a bar graph comparing the properties obtained at 2400F on the 1/4-inch OD by .020-inch wall tubes from each alloy in all five temper conditions. This graph shows that the strength obtained on the T-222 alloy is 15 to 20 ksi higher than the Ta-10W alloy, while elongations are comparable.

TABLE 21

High Temperature Tensile Data
Ta-10W Alloy

Size	Condition	Test Temp (°F)	Vacuum[a] torr x 10^{-5}	UTS (ksi)	.2% YS (ksi)	Elong. (%)
1/4" OD x .120" W	Hard	2100	.8	59.6	41.4	16
		2100	1.0	58.0	38.6	16
		2400	.6	44.9	27.5	20
		2400	.5	42.1	27.6	20
		2700	1.0	20.9	16.4	60
		2700	1.0	22.1	17.1	57
	1/2 Hard	2100	.6	51.9	35.7	15
		2100	.7	51.6	39.5	16
		2400	.7	40.6	27.9	24
		2400	1.0	40.6	27.2	22
		2700	1.0	23.4	17.3	35
		2700	1.0	23.3	16.8	35
	1/4 Hard	2100	.7	42.3	35.5	28
		2100	.6	42.9	34.5	25
		2400	.7	33.8	26.7	36
		2400	1.0	35.2	26.2	35
		2700	1.0	26.7	18.9	44
		2700	1.0	27.2	17.8	38
	Stress Relieved	2100	4.0	57.9	37.6	15
		2100	3.0	56.7	37.5	16
		2400	2.0	45.9	26.4	20
		2400	1.0	45.4	26.1	23
		2700	.8	20.8	16.6	56
		2700	2.0	22.1	16.1	62
	Annealed	2100	1.0	34.3	20.4	45
		2100	3.0	33.8	20.0	42
		2400	3.0	25.8	17.6	41
		2400	2.0	28.1	17.9	53
		2700	2.0	21.8	16.1	58
		2700	3.0	22.2	16.7	63
3/8" OD x .062" W	Hard	2100	0.3	58.6	47.3	11
		2100	0.1	63.2	53.3	8
		2400	0.6	44.2	33.2	16
		2400	0.2	46.8	34.1	15
		2700	2.0	22.1	16.9	54
		2700	1.0	21.3	15.9	49

TABLE 21 (Continued)

High Temperature Tensile Data Ta-10W Alloy

Size	Condition	Test Temp (°F)	Vacuum[a] torr x 10^{-5}	UTS (ksi)	.2% YS (ksi)	Elong. (%)
3/8" OD x .062" W	1/2 Hard	2100	0.8	55.0	46.8	18
		2100	0.7	52.2	43.2	21
		2400	1.0	43.6	29.8	34
		2700	2.0	23.6	17.8	59
		2700	2.0	23.0	17.7	61
	1/4 Hard	2100	1.0	41.3	38.2	28
		2100	2.0	40.8	36.6	29
		2400	2.0	31.8	27.9	37
		2400	0.9	32.8	28.4	40
		2700	4.0	26.0	20.6	45
		2700	3.0	26.5	19.8	31
	Stress Relieved	2100	2.0	59.6	49.1	14
		2100	3.0	63.1	49.3	13
		2400	2.0	44.8	33.6	25
		2400	2.0	45.8	37.4	21
		2700	2.0	22.6	16.1	49
		2700	2.0	22.7	16.1	49
	Annealed	2100	2.0	34.4	20.9	37
		2100	0.6	33.9	21.2	38
		2400	2.0	28.3	18.9	38
		2400	3.0	27.7	18.1	40
		2700	6.0	22.4	15.5	43
		2700	7.0	22.0	15.4	43
1/2" OD x .062" W	Hard	2100	0.1	63.8	56.6	12
		2100	2.0	64.0	52.7	9
		2400	3.0	46.8	37.8	19
		2400	3.0	49.6	35.1	20
		2700	3.0	21.6	15.6	61
		2700	4.0	22.6	16.2	58
	1/2 Hard	2100	3.0	49.6	42.5	16
		2400	3.0	41.4	31.0	27
		2700	4.0	29.8	18.9	34
	1/4 Hard	2100	1.0	38.8	35.7	28
		2400	2.0	31.3	26.0	43
		2700	3.0	26.0	18.9	38

TABLE 21 (Continued)

High Temperature Tensile Data
Ta-10W Alloy

Size	Condition	Test Temp (°F)	Vacuum[(a)] torr x 10^{-5}	UTS (ksi)	.2% YS (ksi)	Elong. (%)
1/2" OD x .062" W	Stress Relieved	2100	2.0	60.2	46.2	15
		2400	3.0	49.4	33.8	20
		2700	3.0	21.8	15.9	56
	Annealed	2100	2.0	35.8	23.2	38
		2400	0.4	28.7	18.9	47
		2700	0.3	22.0	15.3	59

(a) Vacuum measured at test temperature.

TABLE 22

Elevated Temperature Stress Rupture Data Ta-10W Alloy

Size	Condition	Test Temp °F	Vacuum(a) torr x 10^{-5}	Stress (ksi)	Rupture Life (hrs)	Elong. (%)
1/4" OD x .020" W	Hard	2100	3.0	33.0	34.8	30
		2100	2.0	34.0	30.6	28
		2400	0.8	18.5	12.8	50
		2400	0.9	17.0	23.8	50
		2700	0.03	8.0	169.6	46
		2700	0.2	10.0	54.2	52
	1/2 Hard	2100	0.1	32.6	26.8	28
		2100	0.2	32.6	26.1	30
		2400	0.7	15.9	62.1	42
		2400	0.3	16.5	56.1	41
		2700	0.06	11.0	30.6	47
		2700	0.2	11.5	25.4	55
	1/4 Hard	2100	2.0	18.5	(b)	3
		2100	0.3	22.0	333.9(c)	
				24.2	357.2(c)	
				26.4	378.8	36
		2400	0.5	17.0	78.3	43
		2400	0.6	18.5	26.2	38
		2700	0.1	12.5	17.5	57
		2700	0.1	12.0	24.0	55
	Stress Relieved	2100	1.0	34.0	22.7	26
		2100	0.5	34.0	25.6	28
		2400	0.5	22.0	6.0	39
		2400	0.5	20.5	6.5	45
		2700	0.7	10.0	29.1	44
		2700	1.0	10.5	12.9	62
	Annealed	2100	0.5	18.5	64.9(c)	
				20.3	88.2(c)	
				22.2	114.8(c)	
				24.0	130.7	52
		2100	3.0	22.5	39.6	50
		2400	4.0	18.5	16.6	56
		2400	6.0	18.5	6.3	58
		2700	4.0	16.0	2.0	59
		2700	5.0	14.5	2.4	56

TABLE 22 (Continued)

Elevated Temperature Stress Rupture Data
Ta-10W Alloy

Size	Condition	Test Temp °F	Vacuum(a) torr x 10^{-5}	Stress (ksi)	Rupture Life (hrs)	Elong. (%)
3/8" OD x .062" W	Hard	2100	0.6	36.2	7.5	20
		2100	0.2	34.0	21.2	20
		2400	2.0	17.8	10.7	42
		2400	0.1	16.0	54.6	45
		2700	0.9	12.0	14.5	50
		2700	0.7	11.0	24.9	47
	1/2 Hard	2100	0.6	32.0	34.7	35
		2100	0.1	33.0	22.4	34
		2400	0.3	16.0	55.7	60
		2700	0.7	13.0	5.4	72
		2700	0.2	13.0	10.0	71
	1/4 Hard	2100	2.0	20.5	(c)	4
		2100	3.0	33.0	1.4	28
		2400	0.6	18.5	45.5	43
		2400	2.1	24.0	3.3	36
		2700	0.3	12.0	19.4	53
		2700	0.6	12.0	21.1	47
	Stress Relieved	2100	0.5	37.0	9.4	24
		2100	0.7	36.0	12.3	22
		2400	2.0	18.0	22.2	47
		2400	0.6	18.0	14.5	52
		2700	0.5	10.5	20.9	78
		2700	0.9	10.5	24.7	49
	Annealed	2100	2.0	19.5	(c)	
				11.4	63.2(c)	
				13.4	89.9(c)	
				15.3	133.4	47
		2100	0.5	23.0	24.1	36
		2400	5.0	18.0	8.8	47
		2400	0.5	17.0	20.8	49
		2700	0.3	12.0	10.7	61
		2700	0.7	11.0	15.1	59

TABLE 22 (Continued)

Elevated Temperature Stress Rupture Data
Ta-10W Alloy

Size	Condition	Test Temp °F	Vacuum[(a)] torr x 10^{-5}	Stress (ksi)	Rupture Life (hrs)	Elong. (%)
1/2" OD x .062" W	Hard	2100	0.4	34.0	39.3	27
		2100	2.0	35.0	34.0	27
		2400	3.0	18.0	20.2	64
		2400	2.0	18.0	19.1	61
		2700	0.6	11.0	17.0	75
		2700	2.0	10.5	20.4	73
	1/2 Hard	2100	0.3	33.0	16.8	26
		2100	0.3	33.0	15.7	27
		2400	0.5	17.5	45.8	47
		2400	0.7	19.0	31.0	45
		2700	3.0	12.0	11.4	60
		2700	2.0	11.0	21.1	56
	1/4 Hard	2100	0.3	30.0	3.1	38
		2400	0.2	20.0	9.0	41
		2700	3.0	12.0	14.9	64
	Stress Relieved	2100	8.0	35.0	16.8	22
		2400	0.4	18.0	16.8	51
		2700	5.0	11.5	10.5	67
	Annealed	2100	0.7	28.2	2.3	44
		2400	3.0	17.0	29.0	51
		2700	3.0	12.0	9.5	64

(a) Vacuum measured at test temperature.

(b) Removed after 230.3 hours.

(c) Step loaded to failure as indicated. Hours are cumulative.

(d) Removed after 283 hours.

TABLE 23

High Temperature Tensile Data
T-222 Alloy

Size	Condition	Test Temp (°F)	Vacuum[(a)] torr x 10^{-5}	UTS (ksi)	.2% YS (ksi)	Elong. (%)
1/4" OD x .020 W	Hard	2100	0.2	94.5(b)		
		2400	0.8	65.0	46.3	30
		2400	3.0	71.0	43.6	25
		2700	4.0	39.0	24.2	48
		2700	4.0	37.4	23.8	56
	1/2 Hard	2100	1.0	86.3	51.0	16
		2400	2.0	57.5	44.4	24
		2400	0.9	58.4	43.8	26
		2700	3.0	34.6	22.8	40
	1/4 Hard	2100	0.1	89.4	84.1	3
		2400	2.0	61.0	46.0	18
		2400	1.0	54.5	42.6	25
		2700	2.0	32.8	22.2	38
	Stress Relieved	2100	0.5	93.7	88.9	9
		2400	1.0	62.4	41.8	79
		2400	1.0	60.0	45.2	32
		2700	1.0	33.8	23.2	59
	Annealed	2100	0.8	71.0	43.4	17
		2400	5.0	50.4	33.8	34
		2400	0.6	45.1	32.0	37
		2700	2.0	34.2	27.7	57
3/8" OD x .062" W	Hard	2100	3.0	98.0	57.4	7
		2100	6.0	102.0	78.2	6
		2400	4.0	73.5	57.7	14
		2400	4.0	71.8	53.8	14
		2700	3.0	37.7	25.9	41
	1/2 Hard	2100	1.0	99.7	(c)	8
		2100	0.8	94.9	50.7	6
		2400	1.0	74.0	(c)	10
		2400	3.0	67.7	51.0	14
		2700	3.0	38.7	(c)	54
		2700	3.0	37.8	25.0	45
	1/4 Hard	2100	1.0	95.0	(c)	9
		2100	2.0	81.6	73.5	13
		2400	1.0	66.5	(c)	19
		2400	1.0	55.7	47.6	29
		2700	2.0	44.0	(c)	62
		2700		34.6	27.6	63

TABLE 23 (Continued)

High Temperature Tensile Data
T-222 Alloy

Size	Condition	Test Temp (°F)	Vacuum[(a)] torr x 10^{-5}	UTS (ksi)	.2% YS (ksi)	Elong. (%)
3/8" OD x .062" W	Stress Relieved	2100	1.0	92.6	70.0	12
		2100	1.0	91.6	85.4	9
		2400	1.0	63.1	50.3	24
		2400	2.0	53.7	47.8	39
		2700	4.0	34.7	27.6	61
	Annealed	2100	2.0	70.4	53.3	19
		2100	3.0	67.0	30.1	19
		2400	4.0	50.8	37.8	26
		2400	2.0	48.4	36.7	31
		2700	3.0	34.2	27.4	62
		2700	4.0	52.1	29.6	48
1/2" OD x .062" W	Hard	2100	4.0	92.5	66.6	12
		2400	2.0	63.0	43.0	28
		2400	3.0	57.9	45.5	31
		2700	3.0	33.8	23.2	17
	1/2 Hard	2100	3.0	98.0	65.9	9
		2400	2.0	61.7	43.2	25
		2400	2.0	36.4	30.2	26
		2700	3.0	39.0	24.9	54
	1/4 Hard	2100	2.0	85.0	63.4	9
		2400	3.0	54.0	44.6	22
		2400	0.9	53.2	43.8	26
		2700	2.0	34.0	27.4	54
	Stress Relieved	2100	2.0	93.0	64.0	11
		2400	2.0	56.0	43.8	30
		2400	2.0	56.6	47.4	31
		2700	3.0	37.2	23.1	57
	Annealed	2100	2.0	74.0	50.0	17
		2400	2.0	53.0	41.6	28
		2700	2.0	36.6	27.8	51

(a) Vacuum measured at test temperature.

(b) Pin hole failure.

(c) .2% yield unattainable from stress/strain curve.

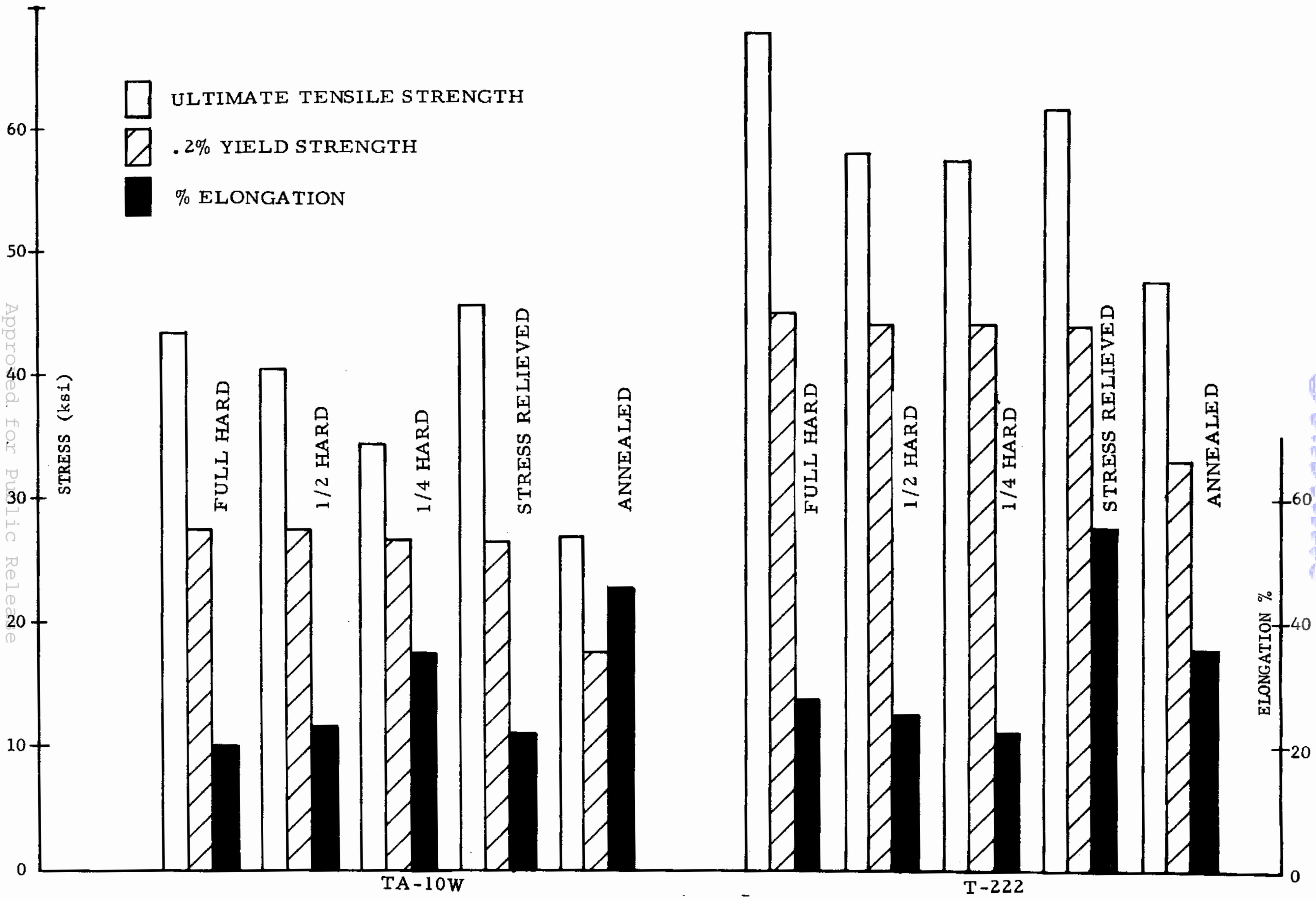


Figure 35

TENSILE PROPERTIES 1/4-INCH OD TA-10W AND T-222 TUBING AT 2400F

The stress rupture data on the T-222 tubes are presented in Table 24. The stress calculated to yield 20-hour rupture lives in the stress relieved T-222 alloy at 2400F were in the range of 25 to 27 ksi. A direct comparison of the rupture properties of the tubing in the various temper conditions cannot be made because of the limited data available.

Based on the test results obtained on the test lot sequence tubes, it was recommended that the production lot tubing in both alloys be processed to finished size and supplied for testing in the stress relieved condition.

TABLE 24 (Continued)

Elevated Temperature Stress Rupture Data
T-222 Alloy

Size	Condition	Test Temp (°F)	Vacuum(a) torr x 10^{-5}	Stress (ksi)	Rupture Life (hrs)	Elong. (%)
3/8" OD x .062" W	Stress Relieved	2100	0.05	50.0	20.0	28
		2400	5.0	30.0	7.5	50
		2400	1.0	27.0	8.3	48
		2700	7.0	13.5	14.7	89
		2700	1.0	12.5	11.6	78
	Annealed	2100	0.6	42.0	27.6	26
		2100	0.6	43.0	11.0	24
		2400	0.5	25.0	14.2	37
		2700	0.4	13.0	11.8	62
		2700	0.7	12.0	16.9	67
1/2" OD x .062" W	Hard	2100	0.9	50.0	20.8	25
		2100	0.3	48.0	43.7	32
		2400	1.0	30.0	6.0	45
		2400	0.6	25.0	22.9	57
		2700	0.5	12.5	12.0	79
		2700	1.0	11.5	20.1	77
	1/2 Hard	2100	1.0	55.0	7.1	32
		2400	0.6	30.0	9.0	48
		2400	0.04	25.0	31.3	56
		2700	2.0	16.0	4.0	83
		2700	3.0	14.5	6.1	79
	1/4 Hard	2100	0.2	48.0	10.2	26
		2400	1.0	27.0	15.3	46
		2700	4.0	13.0	11.9	79
		2700	0.4	11.5	38.8	91
	Stress Relieved	2100	0.09	50.0	28.2	26
		2400	0.7	26.0	16.6	59
		2400	0.4	24.0	37.4	60
		2700	0.5	13.5	11.0	87
		2700	0.4	12.5	18.6	97
	Annealed	2100	1.0	42.0	39.8	29
		2400	5.0	23.0	42.3	60
		2400	0.6	26.0	24.2	55
		2700	0.7	14.0	12.3	94
		2700	0.3	12.0	24.1	84

(a) Vacuum measured at test temperature.

(b) Step loaded to failure. Hours are cumulative.

SECTION V

Phase IV - Production of Tubing

A. Introduction

Under Phase IV of the program the balance of the tubing in both alloys was processed to each of the three finished sizes and finished, in accordance to the recommendations of the Phase III work, in the stress relieved condition. As required by the contract the tubing of both alloys was divided for processing to the three finished sizes in accordance to the following schedule:

40% by weight to 0.500-inch OD by .062-inch wall
40% by weight to 0.375-inch OD by .062-inch wall
20% by weight to 0.250-inch OD by .020-inch wall

In order to demonstrate the reproducibility of the process, the tubing produced during this phase was checked for dimensional tolerances and spot checked for elevated temperature tensile and stress rupture properties.

B. Cold Drawing of Phase IV Tubing

The remaining tubes from both alloys were processed to finished size in accordance to the processing schedule shown in Figure 36. This is the same schedule used to produce the tubing in the stress relieved condition in Phase III. Area reductions of approximately 30 percent were accomplished and, in each case, over-all reductions up to 60 percent were accomplished between anneals. All tubes were stress relieved for 1 hour at 1850F. The in process vacuum anneal for the Ta-10W alloy tubes was 1/4 hour at 2900F, while the T-222 tubes were vacuum annealed for 1/4 hour at 3000F. The tubes were divided for processing as shown in Table 25. The yield data shown in the table are based on weights at the 7/8-inch diameter. Some severe ID defects were observed in the tubes at 7/8-inch diameter, but these were not cut from the tubes, in order to obtain the greatest possible amount of finished tubing. Figure 37 illustrates these defects as they appeared in the finished tubing. While most of the yields were somewhat low, all but the 1/2-inch diameter Ta-10W were considered to be acceptable.

C. Testing of the Phase IV Tubing

While the maximum flare and flattening results obtained on the tubing produced in Phase III indicated that both alloys exhibited good ductility, even with some degree of cold work, inconsistencies existed which were difficult to explain. It was not known whether these inconsistencies were the result of defects in the tubing or non-uniformity of the alloy. For this reason some additional flare and flattening tests were conducted on

TUBE REDUCING AND DRAWING SEQUENCE FOR MATERIAL IN STRESS RELIEVED CONDITION

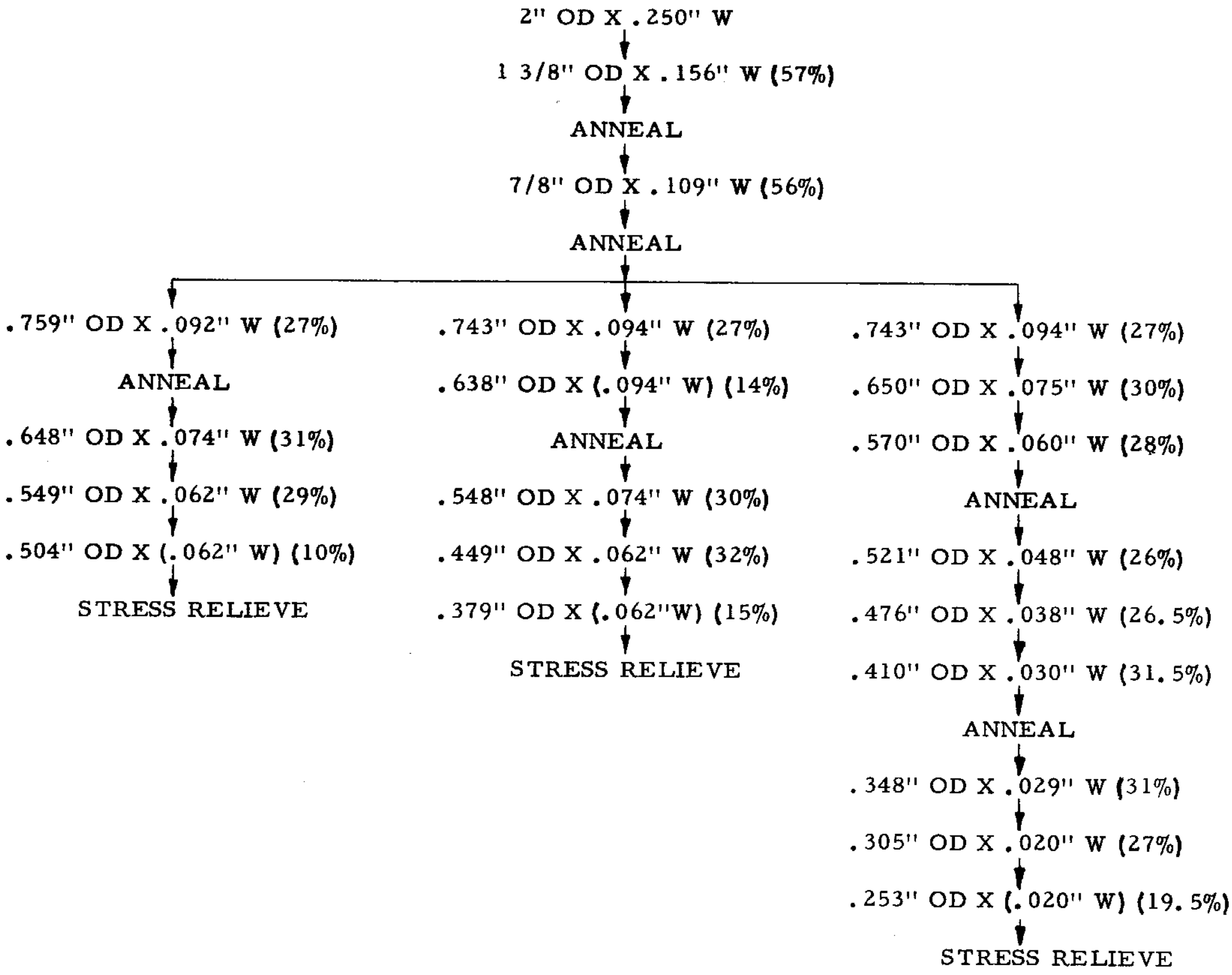


Figure 36

TABLE 25

MATERIAL ACCOUNTABILITY

Alloy	Size	STC Order No.	Tube Nos.	Tube Reduced Weight	Finished Tube Weight	Weight of Acceptable Tubing(1)	Yield From Tube Reduced Size
Ta-10W	.500" x .062"	5-8044	4B,7	48.7#	44.7#	14.3 (22 ft.)	29.4%
Ta-10W	.375" x .062"	5-8043	2, 6, 4A, 7	49.8#	43.4#	39.4 (87 ft.)	79.0%
Ta-10W	.250" x .020"	5-8045	7	25.1#	18.6#	15.8 (157 ft.)	63.0%
T-222	.500" x .062"	5-8046	9, 3A, 3B, 5B	45.5#	39.7#	24.6 (39 ft.)	54.2%
T-222	.375" x .062"	5-8047	9, 10, 11	45.2#	31.9#	22.3 (58 ft.)	49.4%
T-222	.250" x .020"	5-8048	10, 11	23.1#	16.7#	13.4 (121 ft.)	58.0%

(1) Acceptable tubing refers to all material which passed ultrasonic inspection.

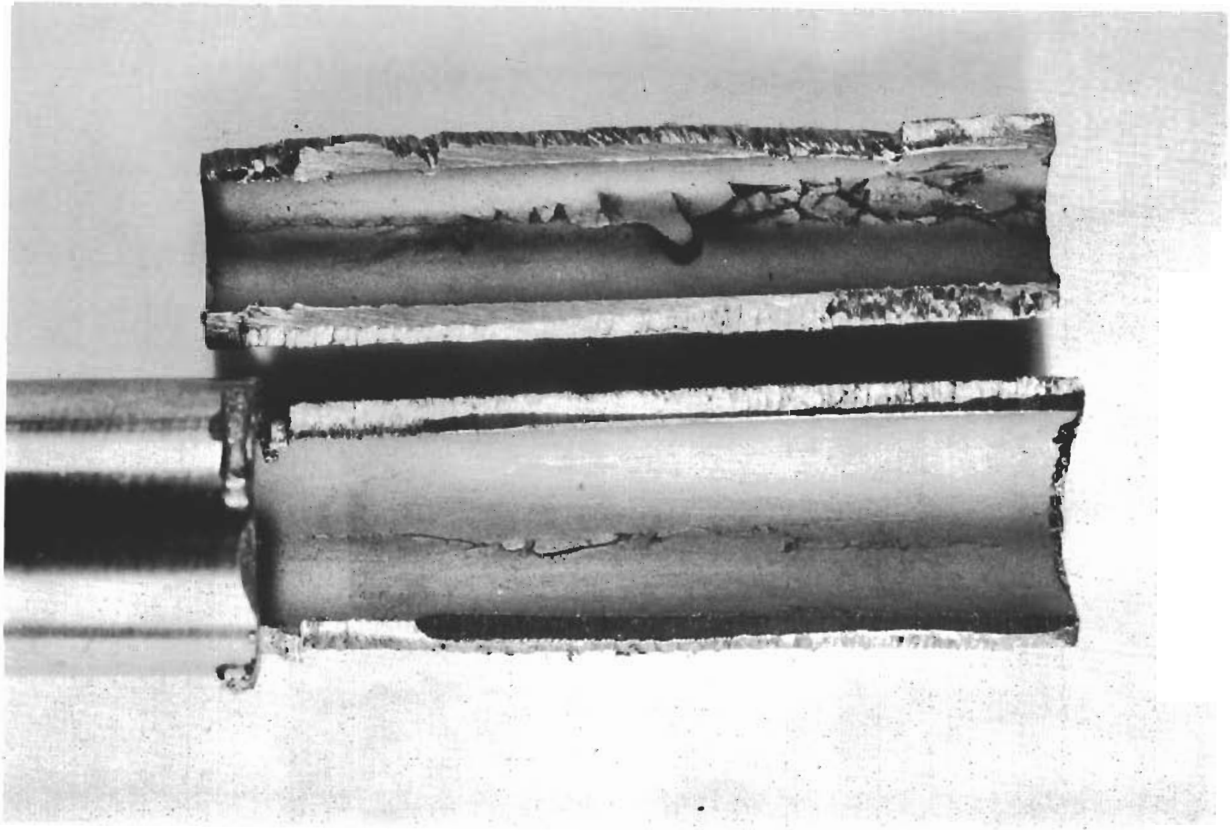


Figure 37

Defects in Ta-10W Tubing

Mag: 30X

Top Tube Shows Typical Defects Observed on ID of .500-inch OD by .062-inch W tube. Tube No. 7 Defect Depth was .006-inch Maximum. Bottom Tube Shows Defects Observed on ID of .375-inch OD by .062-inch W tube, Tube No. 7. Defect Depth was .016-inch maximum.

the tubing produced in Phase IV. The results of the flaring and flattening tests conducted on Phase IV material are reported in Table 26. Little correlation was observed between these results and the results obtained from the Phase III material. The Ta-10W, at 1/4-inch diameter, flared 24 percent as compared with 8 percent for the previous material; however, the T-222 could not be flared, whereas it was flared 10 percent previously. The Ta-10W could be flattened "wall to wall" (3X) as compared with 11 times the wall thickness for the Phase III material. The flattening results for T-222 were consistent at approximately ten times the wall thickness in both cases. It was concluded that the discrepancies were probably the result of isolated ID defects.

The hydrostatic burst test results on the Phase IV tubing, also reported in Table 26, were consistent with those realized on Phase III of the program.

Final dimensional inspection was performed using ultrasonic methods and the variation in wall thickness reported in Table 27 is within commercial limits.

Specimens were selected at random from all three sizes of both alloys for tensile and stress rupture testing at 2400F. These tests were conducted in accordance to the procedures described in Section IV, Page 22. The results of the tensile and stress rupture tests conducted on the Ta-10W tubes are reported in Tables 28 and 29, respectively. The results of the tests conducted on the T-222 alloy are reported in Tables 30 and 31. There is excellent correlation with the data obtained on the Phase III tubing processed in a like manner. There is also excellent correlation in the data on the tests conducted on specimens from all three tube sizes of each alloy and excellent correlation on the tests conducted on specimens from various pieces of the same tube size.

TABLE 26

Flare, Flatten, Bend and Hydrostatic Test Results

Alloy	Size	% Flare to Failure	Flatten to Failure	Bend Test	Hydrostatic Burst Pressure
Ta-10W	.500" x .062"	43	Not Tested	Not Tested	Not Tested
Ta-10W	.375" x .062"	41	Not Tested	Not Tested	Not Tested
Ta-10W	.250" x .020"	24	3 x W Thickness (No Failure)	180° Around 3/4" dia. (No Failure)	23,500 psi
T-222	.500" x .062"	0 (Could Not Flare)	Not Tested	Not Tested	Not Tested
T-222	.375" x .062"	15	Not Tested	Not Tested	Not Tested
T-222	.250" x .020"	0 (Could Not Flare)	10 x W Thickness	180° Around 3/4" dia. (No Failure)	30,000 psi

TABLE 27

Results of Vidigage Inspection[1]

Alloy	Size	Min. Wall Thickness	Max. Wall Thickness	Total Variation
Ta-10W	.500" x .062"	.0590"	.0625"	.0035"
Ta-10W	.375" x .062"	.0600"	.063"	.0030"
Ta-10W	.250" x .020"	.0190"	.0215"	.0025"
T-222	.500" x .062"	.0580"	.0630"	.0050"
T-222	.375" x .062"	.0590"	.0635"	.0045"
T-222	.250" x .020"	.0190"	.0215"	.0025"

---

[1] Vidigage readings taken at four points on circumference of tube at 2-foot intervals.

TABLE 28

VACUUM TENSILE TESTING OF PRODUCTION LOT Ta-10W TUBE SPECIMENS

Test Temperature - 2400F
Test Condition - Full Hard + Stress Relieved

Specimen Number	Tube Size	Vacuum (a) Torr x 10^{-6}	U.T.S. (ksi)	.2% Y.S. (ksi)	Elong. (%)
2L1X	1/4" OD x .020" W	1.0	44.5	28.3	20
2L1Y		1.0	44.8	28.0	22
2L1Z		6.7	45.2	27.5	20
2LJA		1.0	46.2	26.9	23
2LJB		8.6	45.9	26.9	20
2LJC	3/8" OD x .062" W	8.2	39.9	25.7	21
2LJD		9.4	43.8	33.1	19
2LJE		4.4	42.3	32.9	18
2LJF		9.4	39.9	29.4	20
2LJG		9.6	45.2	34.7	20
2LJH	1/2" OD x .062" W	9.7	48.8	28.6	20
2LJI		9.8	45.3	30.5	22
2LJJ		5.4	41.8	32.0	20
2LJK		7.1	46.2	33.0	15

(a) Vacuum measured at test temperature.

TABLE 29

VACUUM STRESS RUPTURE TESTING OF PRODUCTION LOT Ta-10W TUBE SPECIMENS

Test Temperature - 2400F
Test Condition - Full Hard + Stress Relieved

Specimen Number	Tube Size	Vacuum (a) Torr x 10^{-6}	Stress (ksi)	Rupture Life (hours)	Elong. (%)
2LJL	1/4" OD x .020" W	3.6	18.0	18.0	52
2LJM		4.0	18.5	18.8	52
2LJN		5.4	18.0	26.1	46
2LJO		1.0	17.5	27.2	48
2LJP		1.1	17.5	20.7	53
2LJQ	3/8" OD x .062" W	1.1	17.0	30.4	54
2LJR		1.0	18.0	13.9	49
2LJS		1.2	17.5	24.1	45
2LJT		1.0	17.5	8.6	53
2LJU	1/2" OD x .062" W	1.3	18.0	21.7	58
2LJV		1.2	17.5	15.7	46
2LJW		1.0	18.0	19.9	38
2LJX		8.0	18.0	12.7	53

(a) Vacuum measured at test temperature.

TABLE 30

VACUUM TENSILE TESTING OF PRODUCTION LOT T-222 TUBE SPECIMENS

Test Temperature - 2400F
Test Condition - Full Hard + Stress Relieved

Specimen Number	Tube Size	Vacuum (a) Torr x 10^{-6}	U.T.S. (ksi)	.2% Y.S. (ksi)	Elong. (%)
2LJY	1/4" OD x .020" W	1.0	63.1	39.5	25
2LJZ		3.8	65.5	42.2	28
2LKA		4.8	64.4	43.5	26
2LKB		7.0	66.5	47.0	26
2LKC		8.2	61.3	38.2	30
2LKD		7.4	66.2	42.6	26
2LKE		7.1	61.4	38.1	30
2LKF		1.0	62.2	42.1	26
2LKG	3/8" OD x .062" W	5.4	64.8	48.2	24
2LKH		1.0	67.4	52.4	20
2LKI		1.2	69.5	56.1	23
2LLB		9.8	65.6	49.8	21
2LKK		8.4	63.6	52.4	26
2LKL		1.0	66.5	47.6	25
2LKM	1/2" OD x .062" W	1.4	62.9	43.6	24
2LKN		5.1	63.5	44.5	25
2LKO		8.6	63.7	45.6	24
2LKP		1.0	64.3	47.4	11

(a) Vacuum measured at test temperature.

TABLE 31

VACUUM STRESS RUPTURE TESTING OF PRODUCTION LOT T-222 TUBE SPECIMENS

Test Temperature - 2400F
Test Condition - Full Hard + Stress Relieved

Specimen Number	Tube Size	Vacuum (a) Torr x 10^{-6}	Stress (ksi)	Rupture Life (hours)	Elong. (%)
2LKQ	1/4" OD x .020" W	1.0	23.0	26.2	53
2LKR		1.0	23.0	24.6	49
2LKS		7.8	23.5	17.8	48
2LKT		1.3	24.0	16.6	45
2LKU		1.0	25.0	12.7	50
2LKV		1.0	23.0	21.6	56
2LKW		0.5	23.0	26.2	51
2LKX		1.0	25.0	19.0	46
2LKY	3/8" OD x .062" W	1.2	24.0	26.4	56
2LKZ		1.3	24.5	18.8	58
2LLA		1.0	25.5	9.3	49
2LKJ		10.0	24.0	26.8	51
2LLC		0.4	24.5	21.7	58
2LLD	1/2" OD x .062" W	4.1	25.0	20.7	54
2LLE		2.6	25.5	21.6	54
2LLF		1.0	25.5	21.0	56
2LLG		8.3	25.5	31.4	54

(a) Vacuum measured at test temperature.

SECTION VI

Summary and Conclusions

A. Phase I - State-of-the-Art Analysis

1. The greatest potential usage is in the nuclear aerospace applications, especially in the nuclear auxiliary power generation systems involving liquid metals.
2. The properties required are corrosion resistance to liquid alkali metals at temperatures to 2400F, weldability and metallurgical stability over long periods of time at temperatures to 2400F.
3. The main difficulty in the production of tantalum alloy tubing is obtaining quality tube hollows. It is concluded that the extrusion approach offers the most economical and practical means of obtaining quality tube hollows.

B. Phase II - Development of Tube Hollows

It has been demonstrated in Phase II of this program that:

1. Billets of both the Ta-10W and T-222 alloys could be successfully extruded into 2.060-inch OD by 1.440-inch ID tube hollows from a 5-inch diameter tooling system on the 2200-ton press. This is an extrusion ratio of 10.6:1. Short billets of the Ta-10W alloy were successfully extruded at temperatures of 2800F requiring pressures considerably lower than those required to extrude the T-222 alloy under similar conditions at 3000F. It was necessary to utilize an extrusion temperature of 3100F for the longer billets of Ta-10W because of the additional frictional forces present. In order to utilize lower extrusion temperatures the longer billets of the T-222 alloy were extruded using a molybdenum cladding technique. Excellent extrusions were made in this manner. In view of the superiority of the clad extrusions, it would be recommended that this technique also be applied to the Ta-10W alloy in the future.
2. Since relatively high extrusion temperatures were required it was necessary to coat the dies, mandrels and corner cones with zirconia. Die wash was encountered on all of the unclad extrusions except the Ta-10W extruded from 2800F. This extrusion also required less pressure than the other extrusion. The die wash can be attributed to a combination of the extremely high pressures encountered and the presence of a highly abrasive tantalum oxide layer on the billets. Die wash was not encountered on the molybdenum clad T-222 extrusions, since cladding reduced the pressures considerably and molybdenum oxide has lubricating rather than abrasive characteristics.

3. The main problem is in post extrusion conditioning. While both alloys could be roll straightened at ambient temperature, it is recommended that the material be annealed or at least stress relieved between extrusion and the various conditioning operations to avoid stress cracking. The T-222 alloy, in particular, appears to be extremely sensitive to thermal stress cracking. The molybdenum cladding can be successfully removed by pickling in a 25 percent HNO_3 solution. Conditioning of the inside diameters is a major problem area in the production of long tantalum alloy tube hollows. ID boring was found to be the most satisfactory method currently available. However, if the "as extruded" ID surfaces could be improved, other means such as abrasive polishing belts or honing would be satisfactory. Extreme care should be taken in selecting coolants or lubricants to be used during conditioning operations. While tantalum alloys are inert to most reagents, they are attacked by HF, free SO_3 and highly alkaline solutions. Many coolants contain inert sulfur compounds which can breakdown into free SO_3 during use.

C. Phase III - Development of Tube Production Process

1. Superior Tube Company reported that both the Ta-10W and the T-222 alloys displayed good drawability. In addition, despite the defects in the starting material, the material was superior to that normally available from the standpoint of the formation and propagation of defects during drawing. Tantalum alloy material previously processed by Superior Tube was prone to the formation of OD defects during drawing. This was not the case with this material and the defects on the starting tube hollow did not propagate further during drawing. Area reductions up to 62 percent were successfully accomplished without intermediate annealing. It has also been demonstrated that the T-222 can be subjected to the same processing schedules as the Ta-10W alloy and provide comparable results in the finished tubing.

2. Yield results were comparable to those normally attained for the size and quantities processed. Metallographic examination revealed that the defects located by the ultrasonic and eddy current tests were in most cases ID laps.

3. The results of the room temperature tensile, flaring, flattening and bend tests indicated that both alloys exhibit good ductility for their respective strength levels, even with some degree of cold work.

4. Elevated temperature tensile tests revealed that the best combination of strength and ductility was obtained by the material in the stress relieved condition in both alloys. However, depending upon the application and specific properties desired, any of the other temper conditions could produce tubing possessing very desirable properties.

In general, the strengths obtained on the T-222 alloy were about 20,000 psi higher than those obtained on the Ta-10W alloy, while the ductilities, as measured by percent elongation, were comparable in the two alloys.

5. Tubing from both the Ta-10W and T-222 alloys can be successfully welded. However, the ductility of the welded material was considerably less than that of the base metal. The strengths were essentially comparable to that of the base metal.

D. Phase IV - Production of Tubing

1. Processing for the Ta-10W alloy has been optimized and the alloy is considered by Superior Tube Company to be commercially available.

2. While a great degree of experience was gained with the T-222 alloy, Superior Tube feels that it cannot be considered as being commercially available at this time. In order to advance this alloy to the "commercially available" status the following must be accomplished:

    (a) It must be demonstrated that high quality raw material can consistently be made available. Superior felt that the T-222 material processed on the program has displayed the best performance of any T-222 fabricated at Superior.

    (b) It must be demonstrated that the alloy is ductile throughout its range of chemical composition; i.e., high carbon and high tungsten content material may perform differently than low carbon and low tungsten material.

3. The results of the physical tests conducted on the production lot tubes compare very favorably with the data obtained on the test lot sequence tubing, indicating that the process developed is completely reproducible. However, it should be recognized that the starting material used for both the test lot sequence and the production lot sequence tubes in both alloys were from the same heat.

REFERENCES

1. "Tantalum Alloy Ingot Reliability and Sheet Rolling Development Program" First Quarterly Report, Contract AF 33(657)-7015, November, 1961.

2. "Tantalum Extrusion Program" First Interim Technical Report, Contract AF 33(600)-42396.

3. Ault, Marvin P., "Requirements for High Temperature Materials for Space Vehicles" prepared for AIME Conference on High Temperature Materials, Cleveland, Ohio, April, 1961.

APPENDIX A

COMPANY________________________ DATE________________________

Q U E S T I O N N A I R E

STATE-OF-THE-ART-SURVEY

Tantalum Alloy Tubing Development Program

Contract AF33(657)-11261

SECTION I

Interests, Applications, and Requirements for Tantalum Alloy Tubing

A. Do you have any specific needs for tantalum alloy tubing?

	Yes	No
1. At present		
2. In the near future		
3. In the distant future (five years or more)		

B. If a user or potential user of tantalum alloy tubing, please provide the following information:

1.

Type of Application	Alloy Composition	Tubing Size OD	Wall	Length	Supplier

2.

Minimum Properties Required	Temperature, °F 75	2000	2400	2700	3000	3500
Ultimate Strength						
Yield Strength						
% Elongation						
% Reduction in Area						
Minimum Bend Radius						
Creep Strength						
Rupture Strength						
Service Environment						
Hardness						

3. Other properties or characteristics required____________________________________

____

4. Comments on presently available tantalum alloy tubing___________________________

____

C. If a producer of tantalum alloy tubing please supply the following information pertaining to your product:

Alloy Composition	Tubing Sizes Available

Note: If available, will you please attach any literature or brochures describing these products.

COMPANY________________________ DATE________________________

QUESTIONNAIRE

STATE-OF-THE-ART-SURVEY

Tantalum Alloy Tubing Development Program

Contract AF33(657)-11261

SECTION II

Raw Materials

1. Are you a supplier of tantalum powder?________________________

2. Please list your available types and grades of tantalum powder

Grade Designation	Starting Material	% Purity (incl. Gases)	Average Particle Size

3. Please specify impurity levels in the tantalum powders used for alloying

Impurity	Maximum %
C	
N	
O	
H	
Cb	
Fe	
Ni	
Si	
Ti	
W	
Others	

4. Please specifiy impurity levels in the alloying powders used__________________

____

____

5. Please supply pricing and availability information on the tantalum powders and alloying powders produced by your company____________________________________

____

____

COMPANY________________________________DATE________________________________

QUESTIONNAIRE

STATE-OF-THE-ART-SURVEY

Tantalum Alloy Tubing Development Program

Contract AF33(657)-11261

SECTION III

Consolidation of Raw Materials

1. Do you supply pressed and sintered tantalum alloy electrodes for arc-casting? __________________ Pressed and sintered tantalum alloy billets for direct forging? __________________ Pressed and sintered tantalum alloy billets for direct extrusion? ____

2. What tantalum alloy compositions do you supply in pressed and sintered ingots?

____

3. Please indicate the form of the raw materials (particle size, etc.) used for compacting pressed and sintered billets for use as:

   a. Arc casting electrodes ______________

   b. Direct forging billets ______________

   c. Direct extrusion billets ______________

4. How are the powders blended and what are the distribution characteristics of the alloy and tantalum powders? ____

____

5. What general compacting procedures (mechanical pressing, hydrostatic pressing, etc.) are used? ____

____

6. Compacting details:

Alloy	Method	Pressure	Billet Shape	Dimensions of Largest Shape Produced	As Pressed Density

7. Please give details such as type of heating, maximum temperature, billet size capability and atmospheres for the sintering furnaces available at your installation____

____

8. Sintering details:

					Density	
Alloy	Maximum Billet Size	Time	Temperature	Atmosphere	Initial	Final

9. Have you encountered any reactions with furnace hearth materials during sintering?______________If so, please give details______________________

____

____

10. Are claddings or coatings used to prevent contamination during sintering?

______________If so, please give details______________________

____

____

11. From your experience, what are the approximate diffusion rates of the alloy additions in tantalum________________________________

____

____

12. What dimensional tolerances can you hold on pressed and sintered billets

Cross Sectional________________________________

Length________________________________

Straightness________________________________

13. What inspection methods are used to insure soundness and homogeneity of pressed and sintered billets

Penetrant Methods

Ultrasonic

Magnetic Susceptibility

Magnetic Particles

Other

COMPANY____________________ DATE____________________

QUESTIONNAIRE

STATE-OF-THE-ART-SURVEY

Tantalum Alloy Tubing Development Program

Contract AF33(657)-11261

SECTION IV

Melting Techniques

A. General

1. Are you a supplier of tantalum alloy melted ingots?____________________

2. If a supplier please list below the maximum ingot sizes available in the various alloys

Composition	Ingot Diameter	Weight

3. What general melting methods are employed at your facility?

Consumable Electrode________________

Electron Beam____________________

Other_________________________

4. What impurity levels do you specify for tantalum melting stock?

C

N

O

H

Cb

Fe

Ni

Si

Ti

W

Other

5. Is tantalum scrap utilized in your melting practice?__________If so, to what extent?______________________________

6. Is multiple melting utilized to insure ingot homogeneity?__________ Please specify______________________________

B. Consumable Electrode Melting

1. What method is employed to join electrode sections?

Threaded Nipple______________

Welding______________

Other______________

(If welding, specify practice used)

2. What electrode configurations are used?______________________________

____

3. What crucible construction is used?__________Material__________

Wall Thickness______________Taper______________

Liners (if used)______________________________

4. What is the size of the electrode in relation to the size of the crucible?

____

5. According to your experience, what are the optimum melting conditions for the various tantalum alloys?

Alloy Composition	Electrode Diameter	Ingot Dia.	Voltage	Amps	Polarity	AC or DC	Furnace Atmosphere	Melt Rate (lbs/min)

6. Is stirring used during melting?__________If so, please specify__________

____

7. Is hot topping practiced?______________If so, please specify procedures?______________________________

8. Are deoxidizing agents used?________________If so, please specify______

____

9. Are grain refining additions used?______________If so, please specify____

____

10. What is your normal yield from electrode to conditioned ingot?____________

C. Electron Beam Melting

1. According to your experience, what are the optimum melting conditions for the various tantalum alloys?

Alloy Composition	Ingot Dia.(in)	Ingot Length(in.)	Power Required	Vacuum Pressure	Melting Rate(lbs/min)	Average Hardness

2. Are grain refining additives used?__________If so, please specify________

____

3. Are deoxidizing additions used?_____________If so, please specify_______

____

4. What is your normal yield from electrode to conditioned ingot?____________

COMPANY ______________________ DATE ______________________

QUESTIONNAIRE

STATE-OF-THE-ART-SURVEY

Tantalum Alloy Tubing Development Program

Contract AF33(657)-11261

SECTION V

Ingot or Pressed and Sintered Billet Evaluation

A. Inspection for Homogeneity

1. What inspection methods are used to insure ingot soundness? ______________________

   ____

   Please indicate effectiveness of each method ______________________

   ____

2. What surface conditions are normally obtained on arc-melted ingots and pressed and sintered billets? ______________________

   ____

3. If available, please submit photographs of typical macro and microstructures obtained on the as-cast ingots and the pressed and sintered billets ______________________

   ____

4. Have homogenizing heat treatments been effectively used to obtain a more homogeneous structure? ______________________ Please specify the effect and the treatment used for the various tantalum alloys ______________________

   ____

   ____

5. Have consumable arc, electron beam, and powder metallurgy techniques been compared on one tantalum alloy? If so, what alloys? ______________________

   ______________________ What method or combination of methods proved best?

   ____

   ____

B. Ingot Conditioning

1. What method of cropping is normally used in conditioning ingots?____________

____

2. What method of surface conditioning is most effective?____________________

________________________What is the finest surface obtainable by this

method?____

3. Ingot conditioning losses:

Average shrinkage,%___________________Surface conditioning plus clean-up

loss,%______________________________Hot top loss, %________________________

Butt loss,%______________________________Average over-all recovery___________

____

3. Please describe the inspection techniques used to determine straightness of the finished extrusion

C. Detwisting

1. Please describe the equipment and techniques, including temperatures used to detwist the extrusions

2. What conditioning, if any, is required following the detwisting operation?

3. What inspection techniques are used to determine degree of twist in final product?

D. Heat Treatment

		Stress Relief		Recrystallization			
Alloy	Temp. °F	Time	Atmosphere	Temp. °F	Time	Atmosphere	% Recrystallization

COMPANY_______________________________ DATE_____________________________________

STRESS RUPTURE DATA

Temp (°F)	Atmosphere	Stress (psi)	Life (hrs)	%EL	%RA	Comments

HARDNESS

Room Temp	°F	°F	°F	°F	°F

OXIDATION DATA

Temp(°F)	Time	Atmosphere	Weight Gain	Penetration (mils/side)	Metal Loss (mils/side)

IMPACT DATA

	RT	°F	°F	°F	°F
Charpy					
Izod					

BEND TRANSITION

Temp________°F Speed_________in/min Angle_________°

COMPANY____________________ DATE____________________

METALLOGRAPHIC

ASTM GRAIN SIZE__________ INCLUSIONS__________

NOTE: Please list pertinent references and attach data which supplements the information requested above____________________

COMPANY____________________ DATE____________________

TANTALUM ALLOY DATA SHEET

Alloy Composition____________________ Melting Point____________________

Consolidation and Fabrication History____________________

____

____

Condition (as-extruded, stress relieved, recrystallized, % cold worked, etc.)__________

____

____

Heat Treatment____

____

TENSILE DATA

Test	Strain Rate__________				Strain Rate__________			
Temp °F	0.2% Y.S.(psi)	T.S.(psi)	%EL	%RA	0.2% Y.S.(psi)	T.S.(psi)	%EL	%RA

CREEP DATA

	Stress To Produce % Strain								
	Hours			Hours			Hours		
Temp °F	%	%	%	%	%	%	%	%	%

COMPANY________________________ DATE____________________________

STRESS RUPTURE DATA

Temp (°F)	Atmosphere	Stress (psi)	Life (hrs)	%EL	%RA	Comments

HARDNESS

Room Temp	____°F	____°F	____°F	____°F	____°F

OXIDATION DATA

Temp(°F)	Time	Atmosphere	Weight Gain	Penetration (mils/side)	Metal Loss (mils/side)

IMPACT DATA

	RT	____°F	____°F	____°F	____°F
Charpy					
Izod					

BEND TRANSITION

Temp________°F Speed_________in/min Angle_________°

COMPANY______________________________DATE______________________________

METALLOGRAPHIC

ASTM GRAIN SIZE______________INCLUSIONS______________

NOTE: Please list pertinent references and attach data which supplements the information requested above______________________________

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13. ABSTRACT

A process has been developed to produce tantalum alloy tubing in two alloys, Ta-10W and T-222 (Ta-9.6W-2.4HF-.01C) to each of three sizes; .500-inch OD by .062-inch wall, .375-inch OD by .062-inch wall, and .250-inch OD by .020-inch wall. The extrusion method was selected as the most economical and practical approach to producing tube hollows for subsequent tube reducing and drawing. Both the Ta-10W and T-222 alloys were extruded to 2.062-inch OD by 1.440-inch ID tube hollows from a 5-inch liner system on a 2200-ton extrusion press. Short billets of the Ta-10W alloy were successfully extruded unclad from a temperature of 2800F. Because of the additional frictional forces present, a temperature of 3100F was required for longer billets. Because initial extrusions of the T-222 alloy indicated that this alloy required considerably more pressure to extrude, a molybdenum cladding technique was utilized to reduce pressures and enable us to extrude from lower temperatures. Excellent extrusions of the T-222 alloy were made using this technique from a temperature of 3000F. The main problem is in post extrusion conditioning. The tubes were roll straightened cold and conditioned by OD centerless grinding and ID boring. Many stress corrosion type cracks developed during this stage of processing. The material should be annealed or at least stress relieved between extrusion and the various conditioning operations to avoid stress cracking and extreme care should be taken in selecting coolants and lubricants.

The conditioned tube hollows were then tube reduced to 7/8-inch OD by .109-inch

(continued)

14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
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Cold Drawing						
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wall in two steps and then cold drawn to each of the finished sizes in each of five temper conditions; 1/4 hard, 1/2 hard, full hard, full hard + stress relieved, and fully annealed. Both alloys displayed good drawability with area reductions up to 62 percent without intermediate annealing. It was demonstrated that the T-222 alloy can be processed through the same schedule as Ta-10W and provide comparable results in the finished tubing. The tubing was evaluated in terms of all the routine non-destructive and destructive tests. Flaring, flattening, bend and tensile tests indicated that both alloys exhibit good ductility for their respective strength levels even with some degree of cold work. Extensive tensile and stress rupture tests were conducted at temperatures of 2100F, 2400F and 2700F. In both alloys the best combination of strength and ductility at 2400F was obtained by material in the stress relieved condition. The properties obtained for stress relieved Ta-10W were ultimate tensile strength 45 to 49 ksi, .2% yield strength 27 to 37 ksi, % elongation 20 to 25. The properties obtained for the stress relieved T-222 alloy were ultimate tensile strength 56 to 63, .2% yield strength 44 to 50 ksi, % elongation 30 to 79. In general, the strengths obtained with the T-222 alloy were 15 to 20 ksi higher than the Ta-10W, while elongations are comparable. Tubing from both alloys was successfully welded in an inert atmosphere using a tungsten electrode. While strengths were comparable to those obtained on the base metal, ductility of the welds was considerably lower than that of the base metal.

To demonstrate reproducibility of the process, a second lot of tubing in each alloy was processed to each of the three finished sizes and finished in the stress relieved condition. The tubing produced during this phase was checked for dimensional tolerances and spot checked for elevated temperature tensile and stress rupture properties at 2400F. Excellent correlation was obtained with the properties obtained on the test lot sequence tubes.

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