FINAL REPORT

DEVELOPMENT OF FABRICATION PROCESS FOR METALLIC FIBERS OF REFRACTORY METAL ALLOYS

by

L. H. Amra, L. F. Chamberlain, F. R. Adams, J. G. Tavernelli and G. J. Polanka

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FOREWARD AND ABSTRACT

The work described herein was performed by the Lamp Metals and Components Department of the General Electric Company, under NASA Contract NAS 3-7906. The objectives of this program were to develop process techniques for production of small diameter refractory wires; to design and build a multi-station, high vacuum, creep-rupture, stress-rupture test apparatus; and to evaluate these wires at elevated temperatures in the test apparatus.

A molybdenum, two columbium, and a tantalum base alloy were drawn to 5 mils diameter wire. Various problems with the high alloy content influenced the termination of a powder metallurgy approach to making billets from tungsten-rhenium-thoria and tungsten-hafnium-carbon for two additional wire types.

A six station creep, stress-rupture high vacuum test unit was designed and built, for tests on the above wires, at temperatures from 1800°F to 2800°F.

Only the molybdenum alloy, TZC, approached the goal of a stress-to-density ratio of 125,000 inches for a rupture life of 3000 hours in the temperature range of 2000° to 2200°F.

The contractor's Project Manager was George J. Polanka; Project Engineers were Leon F. Chamberlain and Joe F. Tavernelli.

SUMMARY AND RECOMMENDATIONS

The work required to be done under this contract falls essentially into two categories. The first deals with the preparation of materials and their conversion to wire, and the related discussion and data is compiled in Section I of the report. The second category involves the designing and building of the test apparatus, and the testing of the wire, and this is grouped in Section II of the report.

SECTION I SUMMARY

Wire Production

The plan for the original contract was to produce the two tungsten base alloys by powder metallurgy techniques. The columbium base AS-30, and the molybdenum base TZC alloy were planned to be melted in-house, extruded, and then rolled or swaged to sizes suitable for wire drawing. Because of other demands for melting time on the vacuum arc furnace, approval was obtained to purchase the FS-85 and the T-222 as 1/4 inch diameter rod, annealed and ready for wire drawing. Despite the fact that the NASA engineers tried to restrict the alloy selection to commercially available material, an unusually long delay in the delivery of the T-222 rod from one vendor resulted in reordering from another vendor. Likewise, the tungsten alloys, at the alloy levels specified, proved to be unavailable at the time from any vendor.

Neither of the two tungsten alloys reached the point of wire drawing. The TZC molybdenum alloys were warm drawn at red heat, part of the AS-30 columbium alloy was drawn by cold-drawing and part by slightly warm-drawing as well. Both the FS-85 and the T-222 alloys were drawn at room temperature. Various of these alloys demonstrated the presence of surface contamination during drawing and suitable cleaning techniques were used to clean the wire or rod. Opportunities to exploit processing variables are discussed in creep-rupture summary category.

SECTION II SUMMARY

Test Equipment Design

The ultra high vacuum multi-station creep and stress rupture tester designed for the testing program of this contract had a capability for testing 6 wire samples at one time. Creep and stress rupture tests in this tester were down at pressures of 10^{-9} Torr or less at temperatures from 2000°F to 2400°F for times as long as 500 hours. The creep measurements were made using a linear variable differential transformer (LVDT) technique.

Tensile Properties

Duplicate tensile tests were made on each of the alloy wires drawn to the final diameter size of .005". For the Mo-TZC and FS-85 alloy wires, a

Tensile Properties (continued)

second set of tensile tests was run on the material drawn by a particular process that yielded greater 70°F tensile properties. The 0.2% yield strength (calculated from an assume 1" uniform gage section), the ultimate tensile strength, and the percent reduction in area were the tensile mechanical properties calculated from the tensile test data. All the tensile tests were run using the alloy wire at a diameter of .005". In order to compare the properties of the various alloy wires on the basis of a strength/density ratio, the density of each alloy was calculated.

The average ultimate tensile strength/density ratios for the four alloy wires at 2000°F, 2100°F, and 2200°F show that the Mo-TZC Process B wire is about twice as strong as the next strongest alloy wire (T-222A) at all these temperatures. The T-222A and AS-30 Process A wire are similar at 2000°F, but as the temperature is increased, the T-222A alloy wire weakens less rapidly. The FS-85 New Process A wire is significantly weaker than the others at all temperatures.

Creep and Stress-Rupture Properties

Creep-rupture testing of the alloy wires was performed at temperatures of 2000°F and 2300°F in a vacuum atmosphere, $<5 \times 10^{-8}$ Torr pressure, to define the 70 hour rupture life. All the samples tested for this part of the contract were prepared by an etching technique to have a gage section with a diametral reduction. It was assumed that all the creep deformation that took place during a test was confined to this etched gage length. Those stress-rupture tests that were run in the multi-station creep-rupture tester were done in either a vacuum atmosphere, $<5 \times 10^{-8}$ Torr pressure, or in inert atmosphere of 99.999% pure argon. These tests were made on samples having the "as-received" wire diameter.

The creep and stress rupture properties at 2000°F and 2300°F for each alloy wire were investigated with a limited number of tests. The results from some of these tests revealed some conflicting data, and an interpretation of the rupture behavior of these materials was difficult to make. It was not possible to determine the effect of factors such as reducing the cross-sectional area by etching away the outer surface, testing in a vacuum atmosphere (pressure $<5 \times 10^{-8}$ Torr) or compositional changes under vacuum-temperature condition. Therefore, the creep and stress rupture properties are not presented in this report, but are to be presented in a future report together with other data.

Chemical Analysis for Interstitial Elements

One gram samples of each alloy wire in the "as-received" and vacuum heat treated condition (2300°F for 50 hours) were analyzed for the interstitial elements, oxygen, nitrogen, hydrogen, and carbon. A limited amount of work was done with 218 tungsten in an effort to obtain a correlation with tests already performed elsewhere under different atmospheres. The results showed that, for all the alloy wires, the nitrogen and hydrogen levels were reduced by the vacuum heat treatment. The oxygen level for the 218 tungsten, Mo-TZC,

Chemical Analysis for Interstitial Elements (continued)

and AS-30 alloy wires was decreased, while that for FS-85 and T-222 remained the same. 218W and FS-85 were the only alloy wires which showed a definite decrease in carbon level after the heat treatment. The analyses showing the high carbon level for the 218W initially and after the heat treatment is contrary to previous experience with this material.

RECOMMENDATIONS

After reviewing the results of this program such as this, it should not be unexpected that along with some finite values, one also is aware of several unanswered questions. Some of these questions have generated the following recommendations:

1. Determine whether optimization of the wire drawing process to obtain higher tensile strengths also produces gains in high temperature rupture strength.

2. Perform a statistically significant number of tests to overcome the inconsistencies noted in creep rupture properties obtained at elevated temperatures and vacuum atmospheres of $<5 \times 10^{-8}$ Torr for these alloy wires.

3. Determine more fully the effect of etching away the outer layer of wire on the strength properties.

4. Determine the relationship between microstructure and properties with consideration for the eventual application of the wire.

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INTRODUCTION

In recent years there has been considerable activity in the materials development field to exploit the beneficial reinforcement influence of a pattern of strong fibers imbedded in a matrix which possesses additional desirable engineering properties. Some composites, like fiber glass-reinforced-plastic, are well entrenched as useful materials; but investigations such as those spear-headed by investigations at the NASA Lewis Research Center, have indicated that composite metal-metal systems may offer the means of attaining physical and mechanical properties not available from any single metal system.

To explore the possibilities of developing a composite material for high temperature applications, NASA Lewis Research Center submitted an RFQ for the "Development of Fabrication Process for Metallic Fibers of Refractory Metal Alloys". The Lamp Metals and Components Department of the General Electric Company responded to the RFQ because of its long experience and position as one of the world's largest producers of tungsten and molybdenum wires and eventually was awarded contract NAS 3-7906.

The initial contract was amended several times to take advantage of information generated during the course of the work. The essential requirements of the final contract form are reviewed in the following paragraphs and pages and range from condensed versions of voluminous detail to direct quotation of the contract stipulations.

I. Program Objective

The objective of this work is to develop process techniques for the production of small diameter refractory metal alloy wires. The wires will be incorporated into superalloy matrices, with the ultimate objectives of providing a fiber reinforced superalloy composite, for applications in advanced turbojet engines. The goal for the drawn wire is a stress-to-density ratio of 125,000 inches for a rupture life of 3,000 hours in a temperature range of 2000°F to 2200°F.

II. Statement of Work

The contractor shall furnish the necessary personnel, facilities, materials, and services to develop processes for the production of wire smaller than ten (10) mils in diameter, from six (6) refractory alloys. The contractor shall also design, fabricate,and test a multi-station, creep and stress-rupture apparatus for the testing of these alloys.

A. Material Specifications

The basic alloy material may be purchased in rod or large wire sizes if commercially available; it may be vacuum arc melted, cast, and extruded or rolled to a suitable size for drawing; or it may be consolidated by powder metallurgy methods and processed in accordance with established industry practice.

A. Material Specifications (Cont'd.)

The six (6) candidate alloys are:

(1) Mo-TZC (Mo-1.25Ti-0.25Zr-0.12C)

(2) AS-30 (Cb-20W-1.1Zr-0.09C)

(3) W+Re, ThO_2 (5 ± 1 Re and 3 ± 1 ThO_2)

(4) W+Hf, C (2 ± 1 Hf and 0.005 - 0.02C)

(5) FS-85 (Cb-28Ta-10W-0.9Zr)

(6) T-222 (Ta-9.6W-2.4Hf)

B. Test Apparatus Specifications

The test apparatus shall consist of an ultrahigh vacuum chamber, bakeable to at least 500°F, and be of sufficient size to contain the six (6) creep, stress rupture stations. The vacuum pumping system should have an ion pump capable of at least 500 l/s pumping speed. Roughing shall be accompanied by at least three (3) sorption type pumps, each having a liquid nitrogen chamber and a bake-out heater. The system shall also include a titanium sublimation pump and baffle. The vacuum system shall reach a pressure of 5×10^{-11} Torr within 15 hours after being open to atmospheric pressure. It shall maintain a pressure level of 1×10^{-7} Torr. or less with furnaces installed and operating while the furnaces are being heated from room temperature to 2800°F. Suitable controls for the vacuum system shall be housed in a separate cabinet.

The stress-rupture, creep testing apparatus shall be designed to include resistance heated furnace elements with refractory metal radiation shielding. The temperature control and measurement system and the creep sensing and recording system shall also be housed in suitable cabinets.

C. Wire Fabrication

The contractor shall provide, with exceptions approved by the NASA Project Manager, a quantity of material in the billet, rod, or sintered bar form sufficient to produce 6,000 to 100,000 feet of wire of each alloy, if fabricable.

The wire from fabricable alloys shall be drawn to a size below 10 mils in diameter, with 5 mils being the target diameter. The wire should be in reasonable lengths, preferably over 50 feet per piece, and should be cleaned and spooled onto a suitable container.

C. Wire Fabrication (Cont'd.)

The Contractor shall draw these materials into wire by standard or modified techniques, and may explore property optimization by processing. The NASA Project Manager may give technical direction for the production of additional 6,000 feet quantities of an optimized process wire for the test program.

The Contractor shall supply the following quantity of each alloy for the wire drawing process.

TZC Alloy - 110 pounds $\pm$ 10 pounds, AVC ingots

AS-30 Alloy - 100 pounds $\pm$ 10 pounds, AVC ingots

FS-85 Alloy - 80 pounds, 1/4 inch diameter rod, annealed, purchased from an approved vendor

T-222 Alloy - 10-20 pounds, 1/4 inch diameter rod, annealed, purchased from an approved vendor.

W-5Re-3ThO_2 Alloy - "X" number of 500 to 3,000 grams sintered bars (to explore overall processing and fabricability), from a supply of 100 pounds of W+2.75% ThO_2 powder.

W-2Hf-0.02C Alloy - "Y" number of 500 to 3,000 grams sintered bars (to explore overall processing and fabricability), from a supply of 100 pounds of tungsten powder.

All input material; ingot, rod, or sintered bar shall be analyzed and submitted for approval by the NASA Project Manager before beginning processing towards wire. Normal techniques utilized in development work of this type shall be followed by the Contractor to assure collection of information needed for control purposes.

D. Wire Evaluation Tests

Tensile tests shall be made on wire from the alloys fabricated into wire below 10 mils in diameter. Two tensile tests shall be made at room temperature and 1800°F, 2000°F, 2200°F, 2400°F and 2600°F in a vacuum of 10^{-6} Torr. or better. The NASA Project Manager shall select materials for the initial tensile property evaluation, as well as any materials produced by an optimized process, for further tensile testing.

Creep tests shall be made at 2000°F and at 2300°F to define a 70 hour rupture life curve with a minimum of two data points. Wire samples to be so tested are to be selected by the NASA Project Manager, with the tests being run in the previously described apparatus, preferably in a vacuum to limit contamination. The extent of increase or decrease in the levels of oxygen, nitrogen, and hydrogen in the alloy wires tested in the high temperature creep, stress-rupture apparatus, after extended exposure to the high temperatures and atmosphere, shall be determined.

D. Wire Evaluation Tests (Cont'd.)

The Contractor shall also perform two stress-rupture runs in the apparatus at 2000°F to determine a 100 to 200 hour maximum rupture life, each run to consist of six specimens. The first run is to consist of three specimens of a 218 tungsten alloy reference wire and three specimens of the FS-85 wire tested at three stress levels. A second run will be performed with conditions based on the results of the first run, and likewise a third run will be based on information generated in the previous two test runs.

III. Technical Data Requirements

The Contractor shall follow recognized development procedures in collection and maintenance of technical data and equipment logs, and will observe the need to calibrate apparatus and equipment at scheduled intervals.

Section I - Materials and Fabrication Processes

A. Molybdenum Base Alloy (TZC)
Mo+(1.2-1.5)Ti+(.15-.21)Zr+(.11-.14)C

Electrode Preparation

Molybdenum powder containing 1734 ppm carbon was used in preparing the four electrodes for arc melting. The electrodes were hydropressed at 35,400 psi to 2.27 inches O.D. x 25 inches long, having a .74 inch concentric hole running through the entire length. The electrodes were sintered at 1850°C for six hours in a hydrogen atmosphere. Table IAI is a description of the sintered electrodes and analysis of molybdenum powder. The overall average density of the sintered material was 96% of theoretical, having a carbon content of about 1100 ppm. Physically, the electrodes were fairly uniform in size without noticeable distortion lengthwise. The electrodes were then threaded and joined to form a single electrode, 88.25 inches long, weighing 111.0 pounds. Metallic alloying additions were made by inserting, in the center hole, the proper amounts of Ti and Zr rods. Two sizes of Zr (.1" and .085") from the same lot of material plus a .370" Ti rod were inserted to make the required composition. Table IAII is a complete vendor analysis of the Zr and Ti additions.

Arc Melting

Two ingots, TZC-1 and TZC-2, were melted into a four inch diameter copper crucible. The diameter of the cast ingot was 3.908 inches. Melting conditions are shown in Table IAIII. It should be noted that the melting parameters are almost identical for both castings with TZC-2 having only a somewhat slower melting rate.

The two ingots were then machined to 3.620 inches diameter to obtain a smooth surface and inspected by ultrasonic, die penetrant, and microstructural techniques for piping, microcracks and other defects. From Table IAIV, it is evident that 1-3/4 inches were discarded from the top due to piping. Two to three inches were cropped because of hairline cracking, indicated by ultrasonic testing, and corroborated by subsequent microstructure examination. The total ingot conditioning losses in melting TZC-1 and TZC-2 were about 45 and 40%, respectively. The two billets then were turned down to 3.490 inches diameter for extrusion, with a 45° nose camfer and a 1.5 inch nose diameter. Figure IA1 shows the two machined billets.

Metallographic Examination

Prior to further processing, a slice 1/2 inch thick was sawed from the top of each casting. This was done after cropping defective material as shown previously in Table IAIV. Each slice was then bisected: The first section was used for hardness, macrostructure and chemical analysis; the second half was cut as shown in Figure IA2,

Metallographic Examination (Cont'd.)

in order to examine the transverse and longitudinal microstructure and to obtain grain size measurements. As shown in Figure IA2, Sections 1, 2, and 3 represent longitudinal sections progressing from the outside to the center of the casting, while Sections 4, 5, and 6 represent transverse sections progressing from the center to the outside. Using the ASTM Heyn Intercept method, the grain size of all sections is shown in Table IAV.

Average hardness of five readings on the cast ingots, measured in the radial direction using a 1 kg load, was 233 DPH. Variations of hardness readings were about -6 + 4 DPH. This small variation is significant since it is an indication of the extent of alloying and homogenization obtained in melting.

Metallographic preparation was tedious and accomplished by mechanical polishing then swab etching the specimen in a solution of 2 gr. $K_3Fe(CN)_6$+ 6 gr. NaOH + 600 cc H_2O. The macrostructure as revealed in Figure IA3 shows the extent of grain refinement due to the alloying additions. Typical unalloyed molybdenum has a grain size at least twice as large as that observed for TZC. Examination of the microstructure as shown in Figures IA4 and IA5 reveals a heavy carbide network around each grain and abundant distribution of fine carbide particles within the grains. Identification of the dispersion phases in TZC-type alloys has been made by Chang (1). It is most probable that in both TZC-1 and TZC-2 the coarse particles are Mo_2C and the fine particles are TiC and ZrC.

Chemical Analysis

Complete analysis for metallics as well as Ti and Zr additions and interstitials are shown in Table IAVI. Sections taken from the same slice used in macrostructural studies were consumed in these analyses. It is worth mentioning that the precision in carbon determination is $\pm$10%. Also, the carbon in the castings seems to be 50 to 100 ppm higher than that of the sintered electrodes.

Primary Breakdown

Extrusion of castings TZC-1 and TZC-2 was done at the Research Laboratory of the General Electric Company under the supervision of Mr. John Hughes. The extrusion press used was a 1250-ton Loewy. The billets were heated in a hydrogen atmosphere furnace at 1750 $\pm$5°C for one hour. Dies with a 130° die angle were plasma sprayed with ZrO_2 and lubricated with No. 7052 glass powder plus graphite. The billet lubricant was No.7052 glass.

Under these conditions, an extrusion constant of 84,500 psi was obtained for TZC-2 using the theoretical relationship - $P = K \ln A_o/A$ - where P is the breakthrough pressure, A_o/A is the extrusion ratio and K is the extrusion constant. This value agrees well with that obtained by D. R. Carnahan and V. DePierre (2) of approximately 83,000 to 95,000

Primary Breakdown (Cont'd.)

psi. No K factor could be obtained from extrusion of TZC-1 because of a failure in instrumentation.

Extrusion at a ratio of 6.2:1 was fairly successful in spite of several tears in the extruded bar. The complexities of high extrusion ratios at high temperature with attendant problems of lubricant breakdown and contamination are all at least partly responsible for the surface tears. Figure IA6 shows TZC-1 and TZC-2 extrusions after machining to 1.315 inches and 1.370 inches respectively.

After eliminating about two inches from each extruded end, sections from TZC-1 were examined for microstructure and hardness. Figure IA7 reveals a structure which is mostly wrought with the exception of a few small areas which appear to be partially recrystallized, again probably due to the high extrusion temperature and high reduction ratio. The average hardness over the cross-section was 274 $\pm$10 DPH using a 1 kg. load.

Rod Breakdown and Wire Drawing

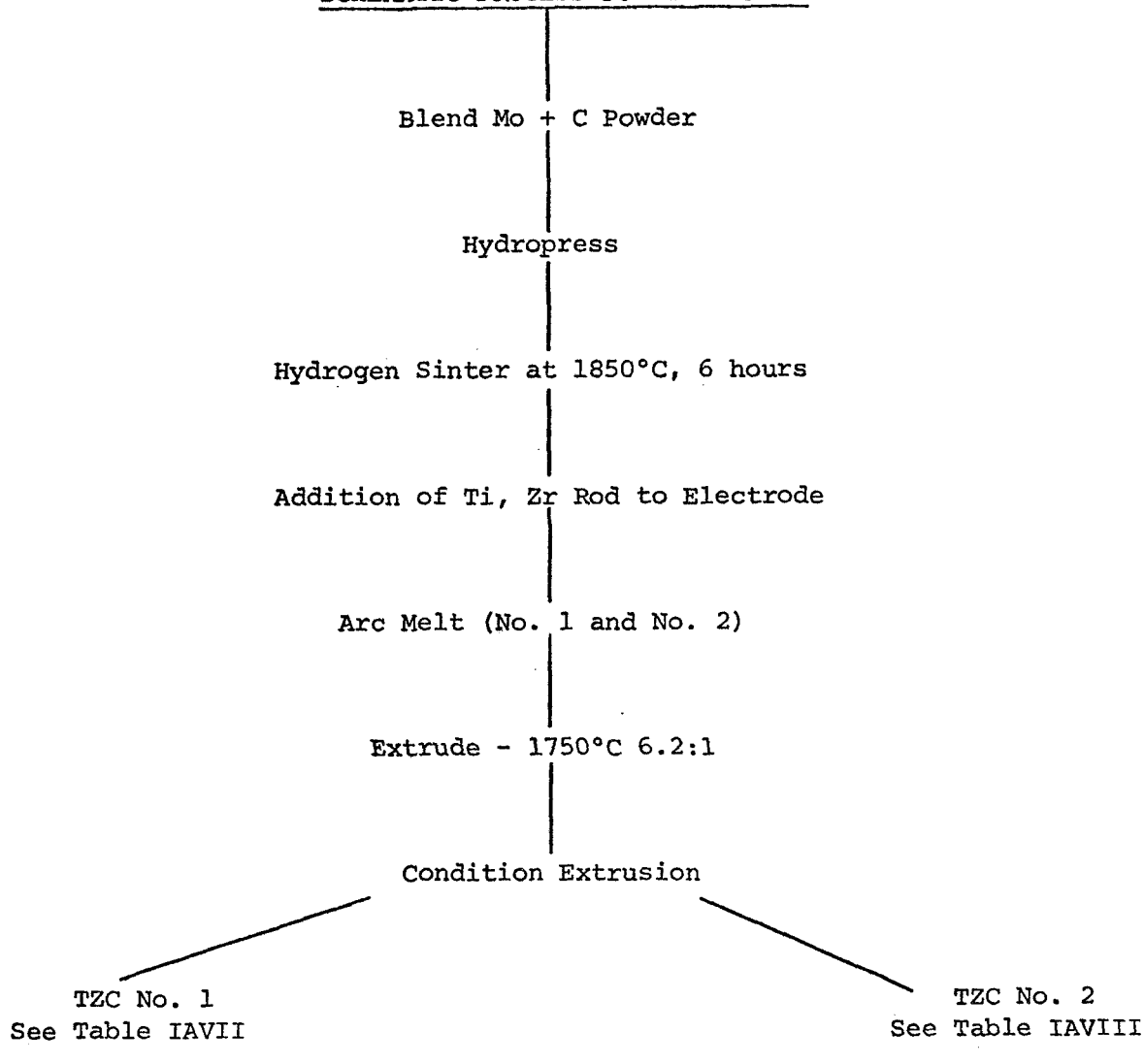
The problem of developing a fabrication technique for producing .005" diameter TZC wire with optimum creep properties at 2000°F - 2200°F was analyzed in parts. The approach was to first develop a workable process to fabricate TZC from 1-1/2" rod to .005" diameter wire with minimum loss of chemistry (decarburization) and free of contamination and physical defects such as splits, slivers, etc. Once a routine was established, modifications in annealing points and temperatures, coupled with processing variables, were investigated to develop the most optimum wire for creep properties at 2000°F to 2200°F.

Two extruded bars, one from TZC-1 and one from TZC-2, were conditioned by local grinding to remove tears and seams prior to rolling. In addition, two remaining bars, one from each ingot, were set aside to be processed pending the results of the initial trials.

Both bars were rolled on a 3-high rod mill using 10 diamond shaped passes, one oval and a final round pass at .650" diameter. The bars were first soaked in a hydrogen furnace at 1475°C for 30 $\pm$5 minutes, a temperature thought high enough for fabrication and low enough to prevent uncontrolled recrystallization prior to or during rolling. Two rolling passes were made between reheats which were of sufficient duration to return the bar temperature to 1475°C. Exit rolling temperatures ranged from 1375°C to 1275°C. Rolling and swaging details are shown in Table IAVII for TZC-1 and Table IAVIII for TZC-2.

"Alligatoring" (end splitting) occurred on the sixth and ninth pass on TZC-1 and on the ninth pass on TZC-2. After the ninth pass, TZC-2 was cut in half to permit full entry into the furnace for reheating. This is shown as A and B in Table IAVIII. Whenever alligatoring occurred, the rods were cooled to room temperature and the split end

SCHEMATIC PROCESS FOR TZC ALLOY



Rod Breakdown and Wire Drawing (Cont'd.)

cut off at a point where splitting had disappeared. The rods would then be reheated to the 1475°C rolling temperature and processing continued.

At .650" diameter, all three rods were annealed at 1600°C for two hours in hydrogen in an attempt to recrystallize the structure prior to swaging. The degree of strain in the rolled rod was not as high as anticipated because microstructural studies showed the material to be approximately 60% recrystallized with general fiber widening in the non-recrystallized areas. Figure IA8 shows representative areas of the center, mid-radius, and edge at .650" diameter after heat treatment at 1600°C for two hours.

TZC-1 was too short to swage on the regular production lines so it was set aside to be hand swaged. TZC-2 was swaged through 0.560", 0.460", 0.390", and 0.345" diameters without incident. The temperature during swaging ranged from 1350°C to 1250°C in an effort to retain TiC in solution and redistribute what Mo_2C might be present at the grain boundaries. At 0.345" diameter, TZC-2 was recrystallized at 1750-1800°C for 60 minutes in argon. Metallographic samples were taken at this size and Figure IA9 shows representative microstructures before and after recrystallization. The recrystallized grain count was 1200 to 1600 grains per square millimeter for both TZC-2 rods. The structure is relatively clean and free of large carbide particles, so it is evident that most of the TiC was put into solution while the massive Mo_2C particles presumed to be at the grain boundaries of the ingot and extruded bar have been redistributed or solutioned at this stage of processing. Recrystallized hardness was 178 DPH using a 1 kg. load.

The recrystallized rods were further swaged at 1200°C to .250" diameter and were then centerless ground to 0.236" diameter to assure removal of the decarburized surface that measured approximately 0.004" in depth. The rod was prepared (oxidized) and hot drawn (850°C) through carbide dies to 0.186" diameter in two passes. The material was found to be badly split at this size. Figure IA10 shows a typical worked structure at 0.186" diameter.

Sections of unsplit material from TZC-2 at .186" were swaged at 1050°C to .122" diameter in an effort to attain more "work" in the material in order to overcome the brittle behavior. After three passes on the draw bench, the material was still brittle at .100" diameter. Examination of the surface at 30X revealed fine hairline transverse cracks estimated to be about .002" in depth. The material was etched in molten caustic-Nitrite salts to remove approximately .005" from the diameter and was then found to be ductile. The microstructure did not reveal contamination, Figure IA11; therefore, it is assumed that the brittle behavior was a result of the "stress raiser" effect of the fine transverse cracks.

By using the surface removal techniques described above and following the process shown in Table IAVIII, 1350 feet of 0.005" diameter wire was produced from part of TZC-2.

Rod Breakdown and Wire Drawing (Cont'd.)

The work hardening characteristics of the alloy were measured by tensile tests from the .048" anneal point to .010" diameter where the material required another anneal to enable further processing to .005" diameter. The data is shown graphically in Figure IA12.

TZC-1 was hand-swaged with 20% reduction per pass from 0.650" diameter to 0.460" diameter where it attained enough length to fit the production equipment. From this point, through centerless grinding, (0.236") it was processed similarly to the TZC-2 rods. Swaging proceeded without incident from 0.236" diameter to 0.186" diameter. The rod was drawn from 0.186" to .111" in six passes using carbide dies, graphite lubricant and standard heating practice (800°C). At 0.111" diameter, the wire was still very brittle and failed by transverse cracking. The wire was cleaned, examined and found to be badly split. No further work was done on this section of TZC-1.

TZC-1 (tail) was rolled following the most successful rolling schedule shown in Table IAVIII. Rolling yield was good: 97.5%. The as rolled rod was electroetched to remove possible contamination from rolling, and swaging to .370" diameter, using reductions of 20% RA and processing temperatures of 1200°C and 1250°C. The .370" diameter rod was annealed at 1800°C for one hour in argon which resulted in a grain count of 3000 grains/sq. mm, double that which was found previously on TZC-2. This fine grained structure was accomplished by eliminating the 1600°C anneal at .650" diameter after rolling, for the purpose of enhancing the fabricability during further processing. The material was further processed following the schedule in Table IAIX.

Eleven spools, totaling 5,188 feet of 5 mil diameter wire, were produced from TZC-1 (tail). The individual breakdown was as follows:

Spool #1	410	
Spool #2	312	
Spool #3	991	
Spool #4	312	
Spool #5	207	
Spool #6	180	
Spool #7	1,214	
Spool #8	214	
Spool #9	270	
Spool #9A	128	(9A resulted from a break in cleaning)
Spool #10	950	
	5,188	feet or 204 grams

TZC-2 (tail) was processed through rolling and swaging to .367" diameter following the same schedule as TZC-1 (tail). At .367" diameter, the rod was electroetched to .356" diameter and cut in half. One-half (B) was held in reserve while the other half (A) was solution annealed

Rod Breakdown and Wire Drawing (Cont'd.)

one hour at 1825°C in argon. Microhardness readings before anneal averaged 262 DPH; after anneal, the hardness dropped to 173 DPH. The recrystallized grain count was 2,500 grains/sq./mm. The grain size and hardness measurements are comparable to the results reported for TZC-1 (tail).

Due to the poor material yields experienced during the previous processes, special swaging equipment was installed to enable swaging to below .100" diameter. The additional swaging equipment was utilized with good results and enabled the processing to proceed to .090" diameter without incident. Drafts were approximately 20% reduction in area per pass from the annealed rod at .356" diameter to the finished swaged rod at .090" diameter. Swaging temperatures decreased approximately 25°C per pass from starting temperatures of 1150°C to a final temperature of 1000°C which was maintained for the last eight passes. The .090" swaged rod was electroetched to remove decarburization and contamination which measured approximately .002" deep. The material then followed the process as outlined in Table IAX with the exception that 745 grams were set aside at .087" diameter for future disposition. The process yielded three spools as follows:

Spool #11	125 feet
Spool #12	66 feet
Spool #13	177 feet
	368 feet

This completed the wire fabrication portion of Task I with a total of sixteen spools totaling 6,906 feet of .005" diameter TZC wire.

Diameter measurements were taken on both ends of each spool of wire produced and are presented in Table IAXI.

Samples of ingot, rod, and finished wire were analyzed from TZC-1 and TZC-2 in order to determine what effect, if any, processing had on contamination or loss of chemistry. The data are presented in Table IAXII. There appears to be a slight loss in carbon and nitrogen. The increase in oxygen and nitrogen is thought to be attributed to the method of heating the rod and wire for processing, a mixture of natural gas and air.

Optimized Process Wire

Initial testing revealed the process represented by spool #4 (Table IAIX) to be the optimum process for stress rupture. Unfortunately, the "optimum process" for finished wire properties is one of the most difficult to process in wire drawing and would be uneconomical to incorporate into a production item because of excessive wire splitting and breakage. Approximately 513 grams of TZC-2 (tail "A") which had been held at .086" diameter was processed according to the spool #4 schedule and produced 14 spools of .005" diameter wire totaling 6,022 feet or 236 grams.

Discussion of Results

Fabrication through rolling and swaging was not difficult; temperatures were kept high, and from microstructure examination (after 0.345" recrystallization) the massive carbides evident in the "as extruded" wire bars were successfully broken up and distributed throughout. The decarburized layer that occurred during processing was removed by centerless grinding; however, it is recommended that grinding be eliminated from the conditioning process, based on the results in heavy wire drawing. Figure IA13 shows an area that was spot conditioned by grinding and then swaged two passes of 20% RA. The photograph shows the surface rupturing that occurred at the ground areas during processing. Surface removal should be accomplished through chemical or electrochemical etching to eliminate the possibility of localized surface stresses.

The higher rolling temperature (1600°C versus 1475°C) for the first six of the twelve pass rolling schedule was successful in eliminating end-splitting and subsequent material loss at that stage of processing. Eliminating the off mill anneal at .650" diameter did result in a much finer grain size at .370" diameter (2,500/3,000 grains/sq. mm versus 1,200/1,600 grains/sq. mm) and helped in further processing by reducing the propensity toward splitting and breakage. Swaging to .095" diameter also helped in reducing defects. Electroetching and removing the decarburized and contaminated outer surface before hydrogen annealing was a definite help in enhancing fabricability and should be included in any process for producing TZC wire.

The recommended process for producing .005" diameter TZC wire from extruded rod is presented in Table IAX. In addition, since all of the major difficulties in processing were associated with surface condition, it is highly recommended that the TZC extrusion billet be canned in a pure molybdenum jacket as was the AS-30 reported on in Section IB. The jacket could be retained until after the last process anneal and then electrochemically removed to present the uncontaminated TZC alloy.

Reference No. 1

Chang, W. H., "A Study of the Influence of Heat Treatment on Microstructure and Properties of Refractory Alloys", AF 33 (616)-7125, August 31, 1962

Reference No. 2

D. R. Carnahan and V. DePierre, "The Primary Working of Refractory Metals", Report No. AFML-TR-64-387, November 1964.

TABLE IAI

Description of Sintered TZC Electrodes

Prior to Melting

Electrode Number	Overall Density	Individual Carbon (ppm)				Avg. Carbon (ppm)	O	N	H_3
1	96.2%	829	1136	933	1022	980	9±5	3±3	1±1
2	96.2%	1310	1089	1155	1088	1160	8±5	5±5	1±1
3	96.4%	1272	1247	952	1252	1181	6±5	6±5	1±1
4	96.3%	1203	945	1259	900	1077	8±5	6±5	1±1

Size: 2.27 inches O.D. x 0.74 inches I.D. x 25 inches long

Chemical Analysis of Nickel Powder

Element	ppm	Element	ppm
Al	<8	Cu	4
Ca	<5	W	82
Si	<19	Mn	10
Fe	<35	Mg	<10
Cr	10	Sn	8
Ni	<5		

TABLE IAII

Analysis of Metal Additions to TZC Melting Electrodes

A. Zirconium Rod (0.100 and 0.085 inch diameters)

Vendor (Wah Chang Corporation) Analysis

	Top ppm	Bottom ppm
C	<30	40
H	3.2	3.2
N	17	29
O	60	80
Al	<25	<25
B	<0.2	<0.2
Cb	<100	<100
Cd	<0.3	<0.3
Co	<5	<5
Cr	40	32
Cu	<25	<25
Fe	120	150
Hf	125	1100
Mg	<10	<10
Mn	<10	<10
Mo	<10	<10
Ni	<10	<10
Pb	5	10
Si	<40	<40
Sn	<10	<10
Ta	<200	<200
Ti	<20	<20
V	<5	5
W	<25	<25
Zn	<50	<50
Ca	<10	10
Na	<10	<10
U	<0.5	

B. Titanium Rod (.375 in. dia.)

Vendor (Reactive Metals Inc.) Analysis

Carbon	0.03 wt. %
Nitrogen	.01 wt. %
Oxygen	.16 wt. %
Hydrogen	54/60 ppm

TABLE IAIII

Melting Data - TZC Alloy

Ingot No.	Current Amp.	Voltage Volts	Stirring Amp.	Predominant Vacuum Torr.	Casting inches	Casting lbs.	Melting Rate lb./min.
TZC-1	5000	37-38	.5	1.2×10^{-5}	11.14	49	3.3
TZC-2	5000	37-38	.5	1×10^{-5}	14	62	3.0

TABLE IAIV

Summary of Material Losses Incurred on Casting TZC

TZC-1 Length, inches	TZC-2 Length, inches	
11-1/8	14	As-cast length
1-3/4	1-3/4	Cut from top due to piping
2	3	Cut from top due to hairline cracks
1/2	1/2	Used for microstructure and NASA approval
3/4	7/16	Cut from bottom to remove starting pad
5	5-9/16	Total length lost from original as-cast ingot

TABLE IAV

Grain Size of Arc Cast Molybdenum Alloys (TZC)

grains per square millimeter

Section	TZC-1	TZC-2
1	5	12
2	11	14
3	16	8
4	23	25
5	19	22
6	14	18

TABLE IAVI

Analysis of Molybdenum Alloys (TZC)

Spectro Analysis ppm

Ingot No.	Al	Ca	Si	Fe	Cr	Ni	Cu	Mn	Mg	Sn	Ag	Pb
TZC-1	<8	<5	42	43	<8	6	6	12	<10	<7	<1	<10
TZC-2	<8	<5	29	29	<8	<5	5	<10	<10	<7	<1	<10

Gas Analysis ppm

Ingot No.	Carbon	Oxygen	Nitrogen	Hydrogen
TZC-1	1257	3±3	1±1	1±1
TZC-2	1326	3±3	1±1	1±1

Chemical Analysis (% by Wt.)

Ingot No.	Titanium	Zirconium
TZC-1	1.2±.1	.26±.04
TZC-2	1.2±.1	.25±.04

TABLE IAVII

Processing of Extrusion TZC-1

1.478" diameter extruded rod

Rod rolled 12 passes at 1475°C
Reheat every two passes
Finish size: 0.650" diameter
Total R.A. - 80.7%

↓

Annealed at 1600°C - 2 hrs. in H_2

↓

Swaged at 1250°C - 1300°C

↓

.580" - 20% R.A.
.520" - 20% R.A.
.460" - 20% R.A.
.390" - 30% R.A.
.345" - 21% R.A.

↓

Recrystallized 1750°C-1800°C - 60 Min. - Argon

↓

Swaged to

↓

.295" - 27% R.A. - 1175°C
.250" - 28% R.A. - 1050°C

↓

Centerless ground to .235"

↓

Swaged to

↓

.210" - 20% R.A. - 1120°C
.185" - 22% R.A. - 1120°C

↓

Draw Bench

↓

.170" - 15% R.A. - 800°C
.152" - 20% R.A. - 945°C
.132" - 23% R.A. - 900°C
.125" - 10% R.A. - 850°C
.119" - 10% R.A. - 850°C
.111" - 10% R.A. - 850°C

↓

Production on TZC-1 Curtailed
Material 100% Split

TABLE IAVIII

Processing of Extrusion TZC-2

1.466" diameter extruded rod
Rod rolled 12 passes at 1475°C
Reheat every two passes
Finish size: 0.650" diameter
Total R.A. - 80.4%
Material cut in half during rolling due to excessive length and designated TZC-2a and 2b

Annealed at 1600°C - 2 hours in H_2

Swaged at 1250°C-1300°C

.560" - 25% R.A.
.460" - 32% R.A.
.390" - 28% R.A.
.345" - 20% R.A.

Recrystallize 1750°C-1800°C - 60 Min. in Argon

.295" - 27% R.A. - 1200°C
.250" - 28% R.A. - 1200°C

Centerless ground to .235" diameter

Draw Bench

.205" - 23% R.A. - 800°C
.186" - 18% R.A. - 800°C

Both rods 2a and 2b were found to be 50% split. Split sections were cut out, and processing was continued with sound material.

← TZC-2b	TZC-2a →
Swaged at 1050°C-1100°C	Wire Draw
.170" - 15% R.A.	.178" - 8% R.A. - 800°C
.152" - 20% R.A.	.170" - 8% R.A. - 800°C
.136" - 20% R.A.	.165" - 6% R.A. - 800°C
.122" - 20% R.A.	Material broke up - 75% split with many transverse cracks
Wire Drawing at 800°C	
.110" - 14% R.A.	
.105" - 10% R.A.	
.100" - 10% R.A.	
Electroclean and etch to .094" diameter	

TABLE IAVIII (Cont'd.)

Finish Drawing TZC-2b

.085" dia.	18% R.A.	850°C
.077" dia.	18% R.A.	850°C
.0685" dia.	20% R.A.	850°C
.061" dia.	20% R.A.	850°C
.054" dia.	20% R.A.	850°C
.048" dia.	20% R.A.	850°C

Anneal 1475°C - 30 min. ± 5 min. in H_2

20% drafts to .010" diameter at 700°C

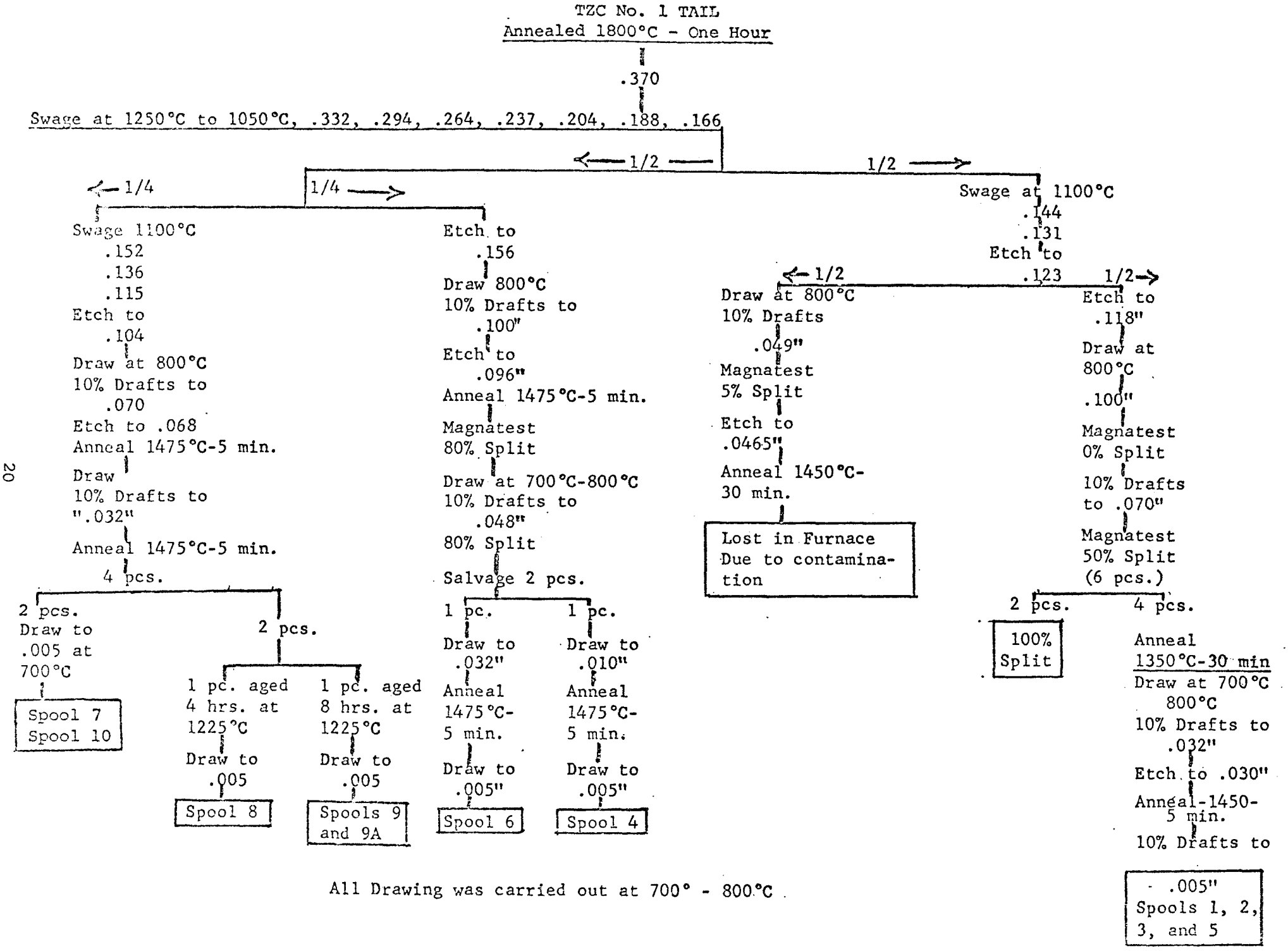
Strand anneal at 1250°C, 2 meters per min. through 10" heat zone in H_2

20% drafts to .005" diameter at 700°C

Electroclean

Yield:	3 spools -	65 meters	(8.3 gms)
		95 meters	(12.2 gms)
		251 meters	(32.2 gms)
	Total -	411 meters or 1,350 feet of .005" TZC	

TABLE IAIX



All Drawing was carried out at 700° - 800°C

TABLE IAX

Recommended Process for Fabrication of
.005" Diameter TZC Wire from
1.5" Diameter Extruded Rounds

1.5" diameter extruded round
Rod roll 6 passes at 1600°C
Rod roll 6 passes at 1475°C
Reheat every two passes - 10 minute soak
Finish rolled size .640" diameter

Total R.A. - 80.4%

Electroclean and etch approximately .010" off the diameter to remove possible contamination. Rotary swage from .630" diameter to .370" diameter using drafts of 20% R.A. and processing temperatures of 1225 to 1300°C.

Electroetch to remove contamination (approximately .010" off diameter).

Solution anneal 1800°C - 60 minutes

Rotary swage from .360" diameter as follows:

.322" at 1250°C - 20% R.A.
.288" at 1200°C - 20% R.A.
.258" at 1150°C - 20% R.A.
.231" at 1100°C - 20% R.A.
.206" at 1050°C - 20% R.A.
.185" at 1000°C - 20% R.A.
.165" at 1000°C - 20% R.A.
.147" at 1000°C - 20% R.A.
.132" at 1000°C - 20% R.A.
.118" at 950°C - 20% R.A.
.106" at 950°C - 20% R.A.
.095" at 950°C - 20% R.A.

Electroetch to .087" diameter
Anneal 1475°C - 30 minutes in H_2
Wire drawn at 700 to 800°C
10% drafts from .087" to .032" diameter
Electroetch to .028" diameter
Anneal 1475°C - 30 minutes in H_2
Wire drawn at 700 to 800°C
10% drafts from .028" to .005" diameter
Electroclean to remove graphite lubricant and surface oxide

TABLE IAXI

Diameter Measurement of Finished Wire

Spool Number	Lead End	Tail End
1	.00498", .00500" .00500", .00499"	.00500", .00500" .00498", .00498"
2	.00490", .00499" .00495", .00494"	.00490", .00490" .00494", .00492"
3	.00490", .00491" .00490", .00488"	.00495", .00495" .00492", .00494"
4	.00492", .00491" .00492", .00490"	.00494", .00491" .00495", .00492"
5	.00488", .00485" .00485", .00486"	.00496", .00496" .00496", .00497"
6	.00492", .00490" .00490", .00490"	.00497", .00496" .00495", .00496"
7	.00495", .00495" .00495", .00494"	.00498", .00498" .00498", .00498"
8	.00492", .00492" .00494", .00492"	.00493", .00497" .00492", .00492"
9	.00498", .00498" .00498", .00498"	.00493", .00497" .00494", .00500"
9A	.00496", .00495" .00494", .00490"	.00498", .00497" .00498", .00498"
10	.00498", .00499" .00500", .00496"	.00490", .00492" .00492", .00492"
11	.00492", .00493" .00492", .00494"	.00492", .00492" .00490", .00493"
12	.00488", .00487" .00490", .00487"	.00494", .00494" .00494", .00498"
13	.00490", .00490" .00491", .00491"	.00488", .00490" .00490", .00488"

TABLE IAXII

Analysis of Molybdenum Alloy (TZC)
Change in Chemistry through Processing

TZC-1 Spectro Analysis ppm

	Al	Ca	Si	Fe	Cr	Ni	Cu	Mn	Mg	Sn	Ag	Pb
Ingot	<8	<5	42	43	<8	6	6	12	<10	<7	<1	<10
.005" wire	<8	<5	25	36	<8	6	11	13	<10	11	<1	<10

TZC-1 Carbon and Gas Analysis ppm

	Carbon	Oxygen	Nitrogen	Hydrogen
Ingot	1257	3 ± 3	1 ± 1	1 ± 1
.005" Diameter Wire	967/1060	81 ± 8	54 ± 5	2 ± 2

TZC-1 Chemical Analysis (% by weight)

	Titanium	Zirconium
Ingot	1.2 ± 0.1%	.26 ± 0.04%
.005" wire	1.1 ± 0.1%	.27 ± 0.04%

TZC-2 Spectro Analysis ppm

	Al	Ca	Si	Fe	Cr	Ni	Cu	Mn	Mg	Sn	Ag	Pb
Ingot	<8	<5	29	29	<8	<5	5	<10	<10	<7	<1	<10
.185" wire	<8	<5	32	21	<8	5	<4	<10	<10	<7	<1	<10
.005" wire	<8	<5	23	42	<8	6	<4	11	<10	<7	<1	<10

TZC-2 Carbon and Gas Analysis ppm

	Carbon	Oxygen	Nitrogen	Hydrogen
Ingot	1326	3 ± 3	1 ± 1	1 ± 1
.185" wire	1160	11 ± 5	9 ± 5	1 ± 1
.005" wire	961/1010	41 ± 5	64 ± 6	2 ± 2

TZC-2 Chemical Analysis (% by weight)

	Titanium	Zirconium
Ingot	1.2 ± 0.1%	.25 ± 0.04%
.185" wire	1.2 ± 0.1%	.28 ± 0.04%
.005" wire	1.1 ± 0.1%	.27 ± 0.04%

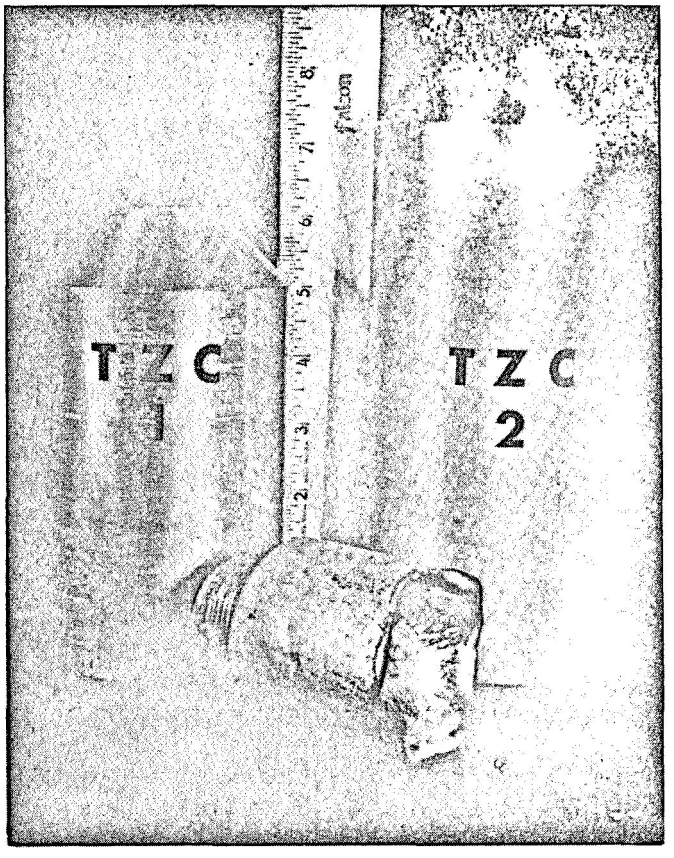


FIGURE IA1 - Machined arc-cast ingots prior to extrusion and stub of melting electrode

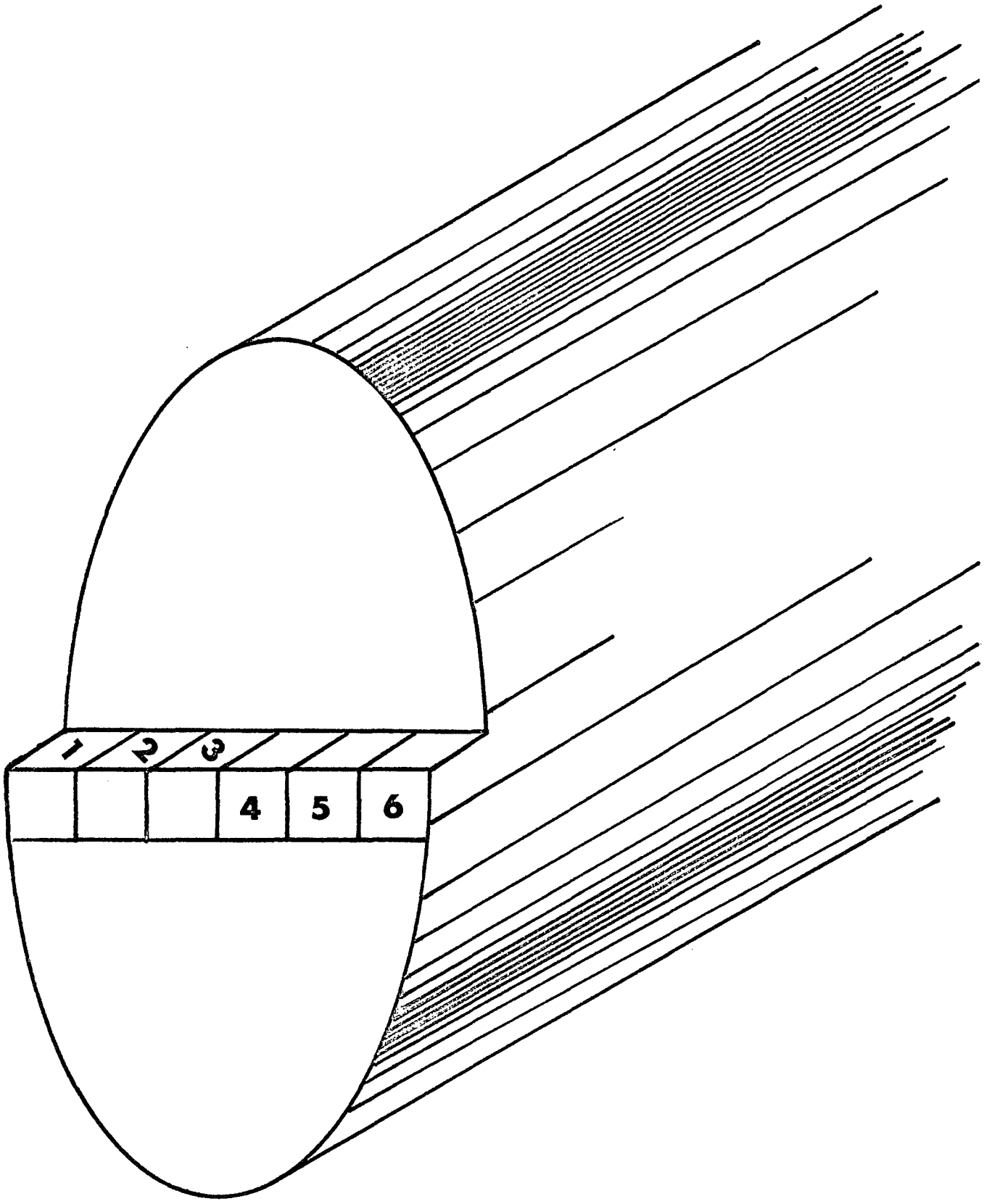


FIGURE IA2 - Metallographic Sections in TZC Cast Billets

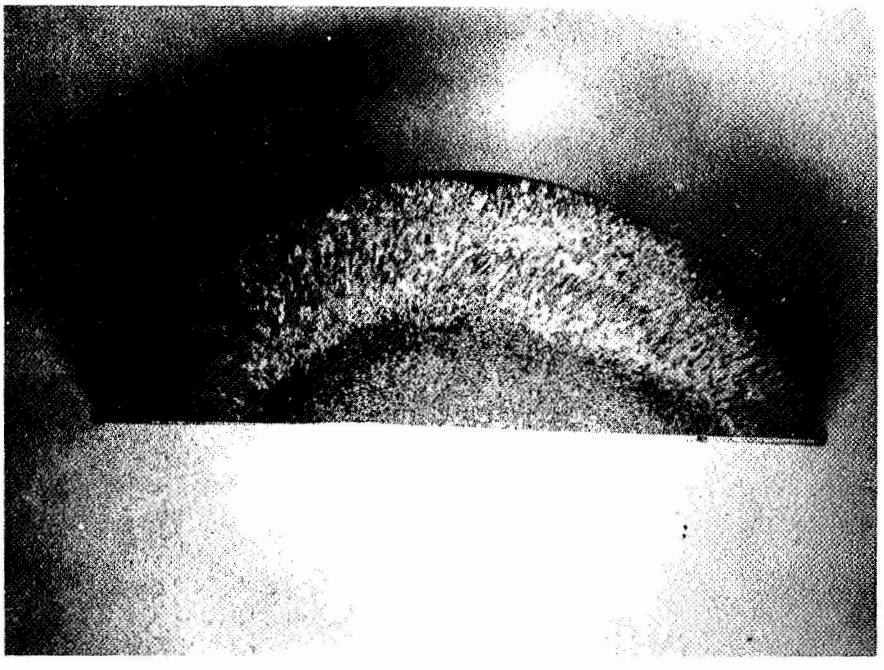
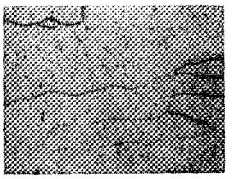
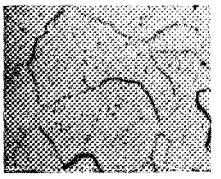


FIGURE IA3 - Macrostructure of cast TZC ingot

TZC-1



7768 100X



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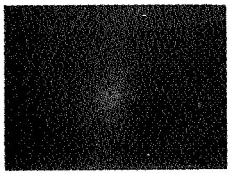

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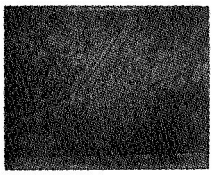
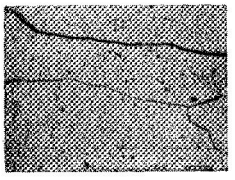
Longitudinal - Section 1

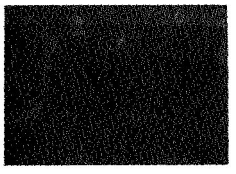


7783 1000X
Transverse - Section 4



7769 100X

7772 100X



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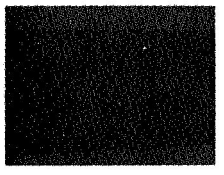
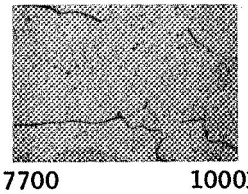
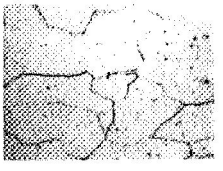
Longitudinal - Section 2



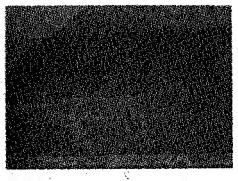
7784 1000X
Transverse - Section 5



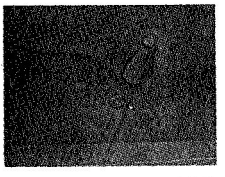
7700 1000X
Longitudinal - Section 1



7773 100X

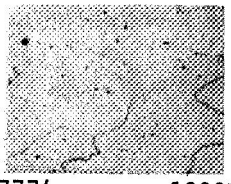


7782 1000X
Longitudinal - Section 3

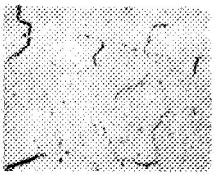


7785 1000X
Transverse - Section 6

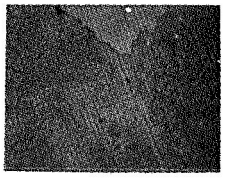
FIGURE IA4 - Microstructure of Ingot TZC #1 as indicated in Figure IA2



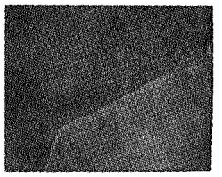
7774 1000X



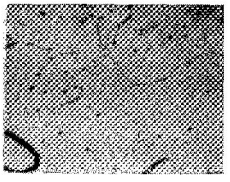
7777 100X

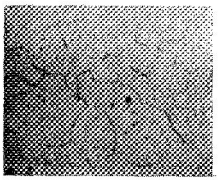


7756 1000X
Longitudinal - Section 1

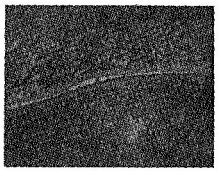


7789 1000X
Transverse - Section 4

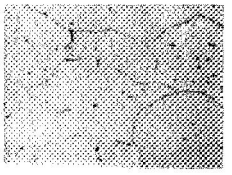




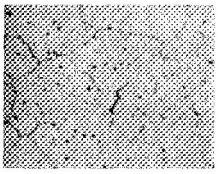
7787 1000X
Longitudinal - Section 2



7790 1000X
Transverse - Section 5



7776 100X



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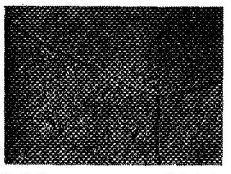
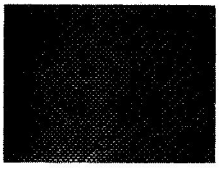


7788 1000X
Longitudinal -Section 3



7791 1000X
Transverse - Section 6

FIGURE IA5 - Microstructure of Ingot TZC #2 as indicated in Figure IA2

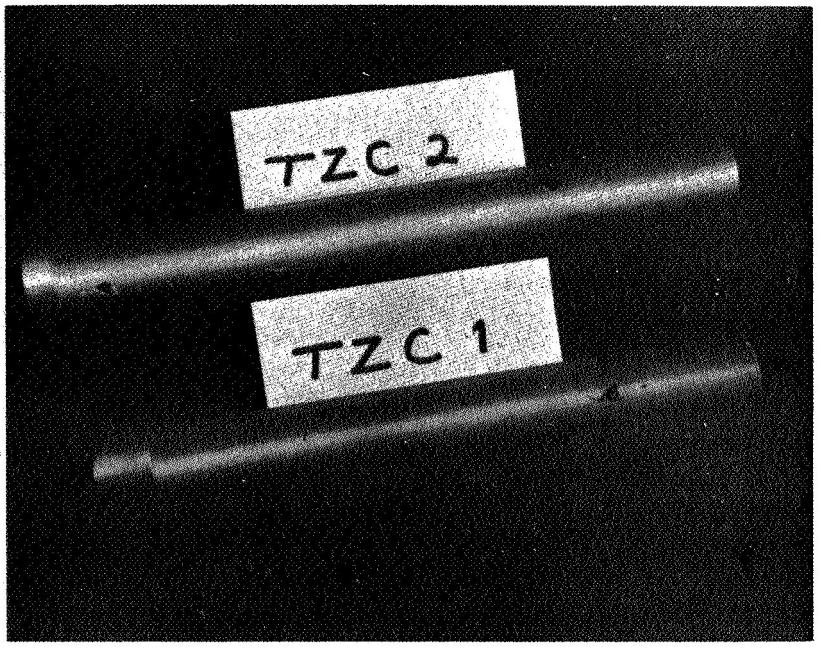
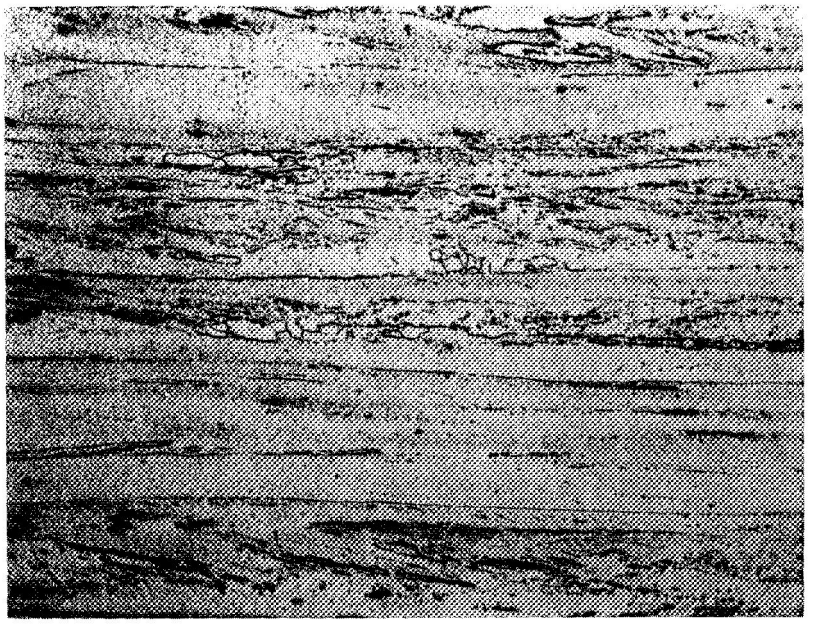
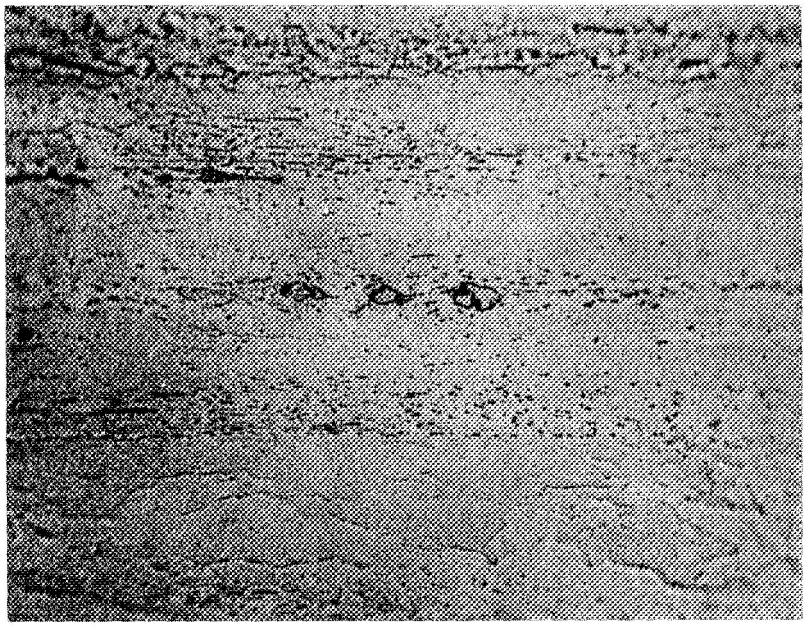


FIGURE IA6 - Machined extrusion of TZC showing localized defects

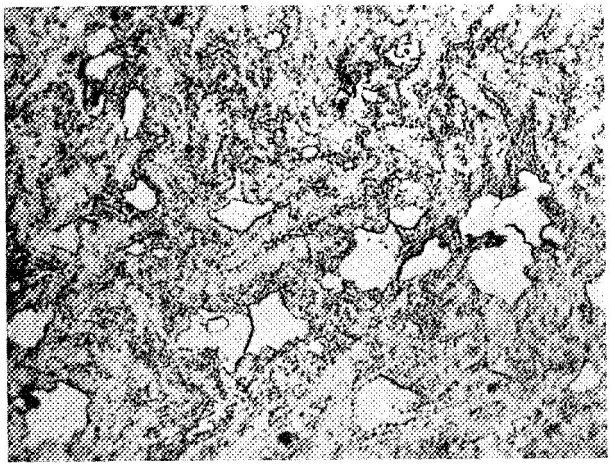


100X

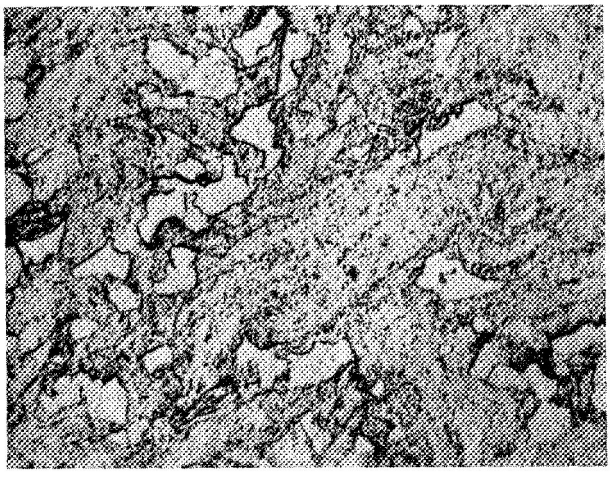


1000X

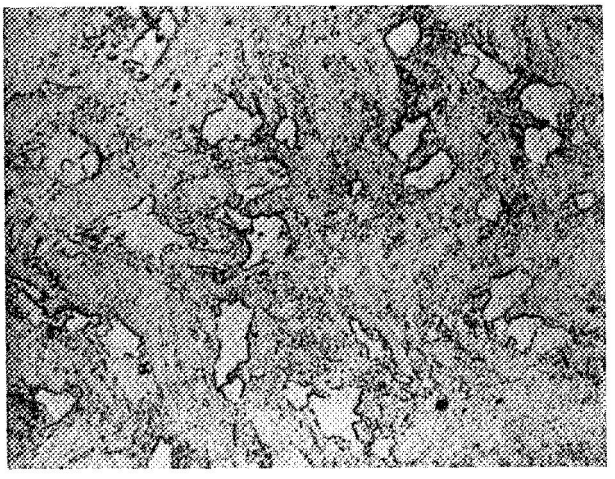
FIGURE IA7 - Microstructure of TZC extrusions showing predominantly cold worked material with small areas of recrystallized, equiaxed grains



314C Transverse Center 250X

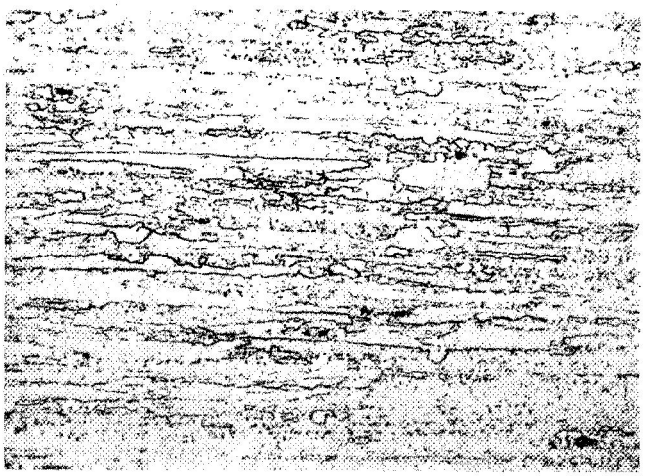


314M Transverse Mid-Radius 250X



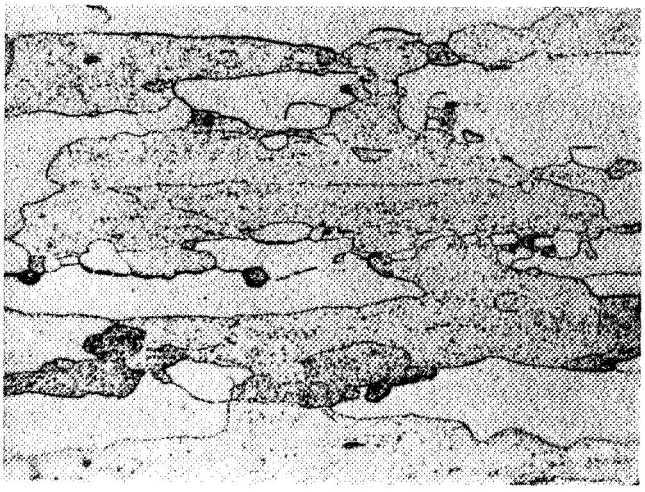
314E Transverse Edge 250X

FIGURE IA8 - Typical microstructures of TZC at 0.650" diameter after annealing at 1600°C for two hours in hydrogen

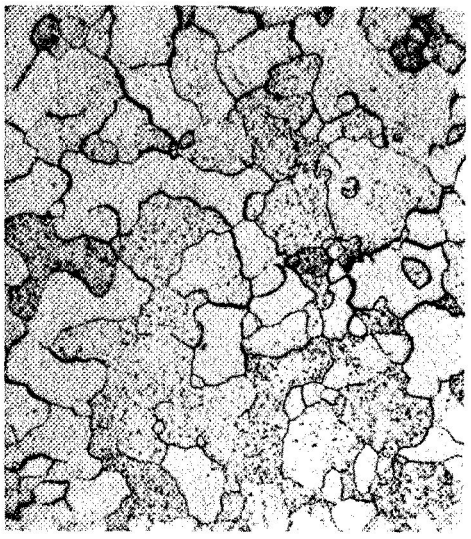


320L Longitudinal 250X

320T Transverse 250X



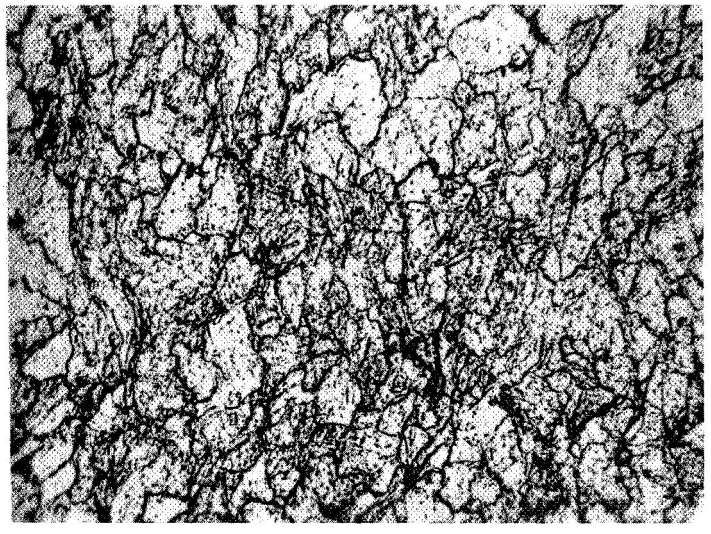
321L Longitudinal 250X



321T Transverse 250X

FIGURE IA9 - Typical microstructures of TZC before and after recrystallization (1800°C - one hour) at 0.345" diameter

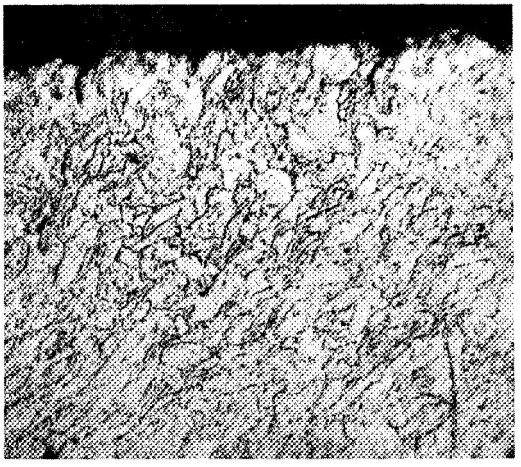
331L Longitudinal 250X



331T Transverse 250X

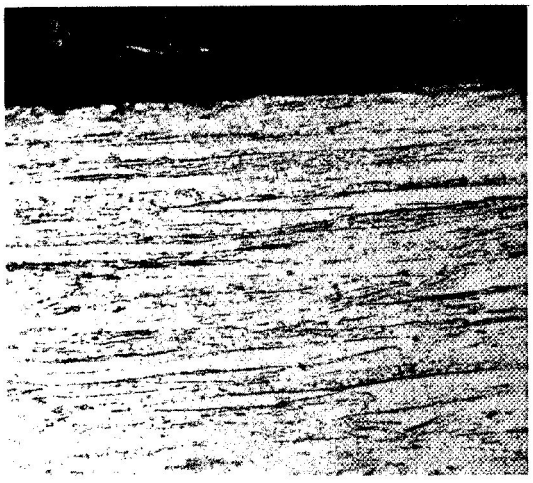
FIGURE IA10 - Typical as worked microstructure of TZC at 0.186" diameter

361L 250X

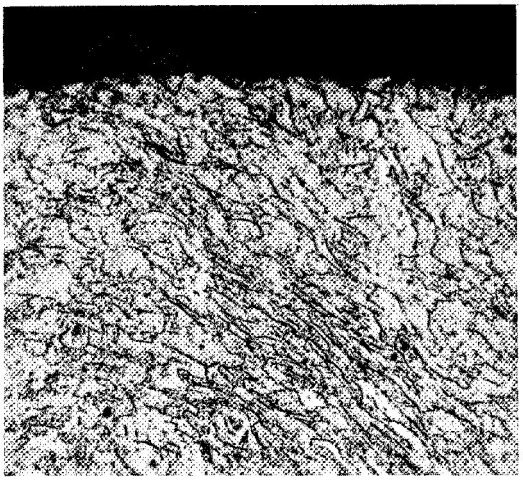


361T 250X

Unetched Surfaces



361L 250X



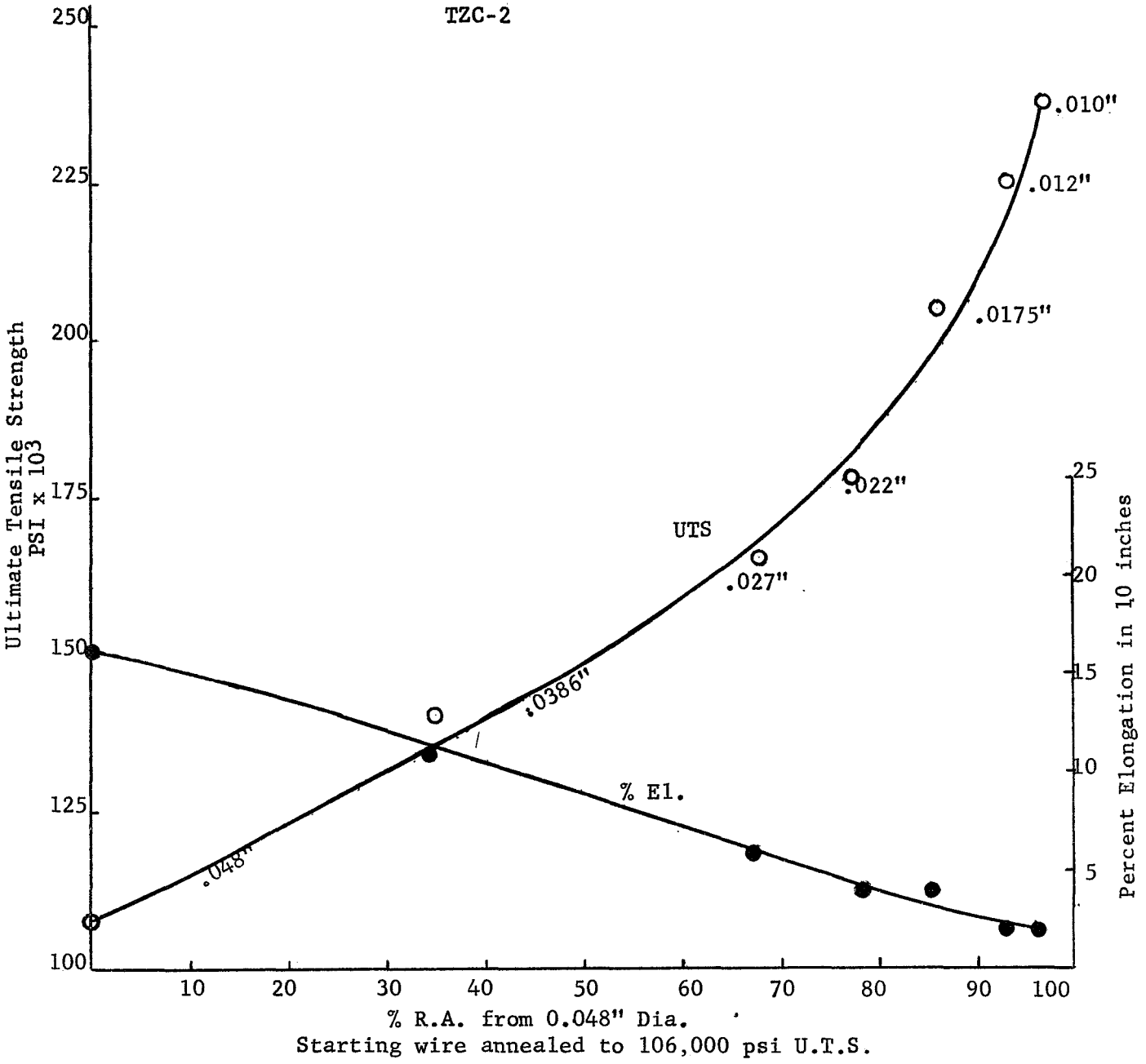
361T 250X

Etched Surfaces

FIGURE IA11 - Surfaces of TZC rod (0.100" diameter) indicating "cracks" possibly responsible for brittle behavior (top) and surface after etching. Lack of microstructural evidence of surface contamination is also apparent.

FIGURE-IA12

Change in Room Temperature Tensile Strength
and Percent Elongation Versus Percent
Reduction in Area
TZC-2



Starting wire annealed to 106,000 psi U.T.S.

Notes:

1. Yield strength data was not recorded because chart speed was too slow to accurately determine intersection at 0.1% offset.
2. Starting material was .048" diameter TZC-2b annealed 1475°C for 30 min., process temperature during wire drawing was 700°C-800°C
3. Strain rate was 0.2 in./in./min.

485 30X

FIGURE IA13 - Section of TZC rod spot conditioned by grinding and swaged showing evidence of surface rupturing.

B. Cb-Base Alloy, AS-30
Cb+(19-21)W+(0.8-1.0)Zr+(0.08-0.1)C

Electrode Preparation

Columbium granules were purchased from duPont deNemours and Company because of their low interstitial content, as presented in the vendor's analysis in Table IBI. The zirconium chips, obtained from Wah Chang Corporation, were attrition-milled to round shapes (-20 +40 mesh) which were more suitable for blending than the chips. Table IBII is a complete analysis of the zirconium. Tungsten "granules" in the -20 +40 mesh size were used as the tungsten addition. Carbon was added as "G" powder obtained from Fisher Scientific.

The blending of the components was performed in a V-cone blender, using a mixing time of 15 minutes. Prior experience had indicated that longer blending times lead to segregation of the widely variant density metals. After blending, the powder was packed into a columbium foil lined rubber mold to prevent the powder from sticking to the rubber. The mold was then hydrostatically pressed at 35,400 psi. Six logs, measuring approximately two inches in diameter were pressed. Figure IB1 shows an as-pressed AS-30 log contained in Cb-foil. The as-pressed electrodes were strong enough to be handled as long as the Cb-foil was intact.

Sintering

The electrodes were individually sintered in a vacuum self-resistance furnace. This furnace provides for heating compressed powder ingots by means of their own electrical resistance. 720 KW of powder is available between a stationary top electrode and a movable lower electrode, both of which are made of copper and are water cooled. A vacuum of about 5×10^{-6} Torr. pressure is maintained by means of a 10 inch diffusion pump (using liquid nitrogen) backed by a six inch oil booster and a 140 $feet^3$/minute mechanical pump. Figure IB2 shows the described vacuum furnace.

Sintering increased the mechanical strength and electrical conductivity of the green compacts. A summary of the sintering parameters follows:

Log No.	Length Pounds	Length Inches	Sintering Temp. °C	Sintering Hours	Vacuum X10^{-5} Torr.	Leak Rate Micron/Hour
A	20.7	27	1750	1	8	1
B	22.2	26.25	1550	1	5	1
C	18.7	22	1500	2	2	.4
D	14.1	16	1500	2	2.8	.9
E	12.8	15	1600	2	1.4	.6
F	11.0	12.75	1500	2	4	1
Total	99.5	119				

Sintering (Cont'd.)

Examination of the above data shows that the leak rate was about <1 micron/hour, and therefore, sintering was done predominantly at 10^{-5} Torr. pressure.

Sintering Log A at 1750°C for one hour caused considerable buckling of the electrode. Therefore, the rest of the electrodes were sintered at a lower temperature. Sintering the remaining electrodes at about 1550 ±50°C for two hours resulted in fairly straight, continuous lengths. A detailed description of the sintering schedule is shown in Table IBIII.

Arc Melting

Two melting electrodes were prepared by joining the sintered bars using tungsten-inert-gas welding methods. Electrodes B, C, and D were joined together to form a 57 inch length weighing 48.5 pounds. Electrodes A, E, and F were welded together to form another melting electrode, 52 inches long weighing 42.5 pounds. A "starting pad" for the bottom of the melting crucible, weighing 16 pounds (of AS-30 composition), was hydropressed and then sintered by radiation heating at 1500°C for two hours. The final pad size was 3.908 inches in diameter x 6.5 inches long.

Two ingots, AS-30 No. 1 and AS-30 No. 2 were conventionally consumably melted from the above electrodes into a four inch copper crucible. Melting was done at the Refractory Metals Plant of the General Electric Company, Euclid, Ohio. The as-cast diameter of the ingot was 3.9 inches. Melting parameters are shown in Table IBIV.

Melting and Chemistry Approval

After machining the two ingots to 3.820 inches diameter to obtain a smooth surface and cropping about 1/2 inch from the top of the ingot, a slice 1/4 inch thick was taken for structure and chemistry analyses. Figure IB4a reveals the microstructure of AS-30 No. 1. It clearly shows that incomplete fusion of tungsten granules occurred during arc melting. The microstructure, at higher magnification, revealed the presence of a fine second phase(s) as shown in Figure IB4b.

Interstitial analysis of ingot AS-30 No. 2 was as follows:

C	789±80 ppm
O	23± 5 ppm
N	10± 5 ppm
H	1± 1 ppm

Since remelting to gain complete fusion of the tungsten was necessary, it was felt that it might be possible to add carbon to assure retention of at least 900 ppm after remelting. A carbon check was run on both ingots with the following results:

AS-30, No. 1 - 828 ppm carbon
AS-30, No. 2 - 849 ppm carbon

Melting and Chemistry Approval (Cont'd.)

In view of the fact that a total of 900 ppm of carbon was in the charge for the first melt, an extra 50 ppm was provided for the second melt to bring the level back near the center of the desired range. The two castings had been quartered diametrically, and then each quarter was welded end-to-end to others to make two electrodes weighing 60 pounds total. A filler rod of pure Cb was used.

During the remelting, Ingot No. 1 was prematurely ended because the operator thought unusual meter readings indicated trouble. Ingot No. 2 was successfully cast into an ingot 10 inches long. After cropping the top ends to remove piping, the total alloy weight available at this juncture was 46 pounds.

The billets or ingots were turned to 3.8 inches O.D. and examined for cracks using Sonoray 50 equipment, but no defects were found. Chemical analyses were run at the Lamp Metals and Components Laboratory where standards were available. Otherwise analyses were determined by Ledoux and Company, Teaneck, New Jersey. The second melt successfully alloyed all the tungsten as can be seen in comparing Figure IB5 with Figure IB4.

The complete analyses are listed in Table IBV. These data indicate the tungsten to be somewhat higher than the specified range of 19-21 percent, while the zirconium is presumably between .70 and 1.0 percent. The carbon depletion is more perplexing since the first melt-down showed only a small loss of carbon, from 900 ppm charged to roughly 850 ppm, while the second melt-down saw a loss from approximately 890 ppm charged to about 540 ppm retained. However, after review of all data, the NASA Project Manager approved the ingots for further processing.

Canning and Extrusion

AS-30 No. 1, the small (short) ingot weighing 12 pounds was encapulated in a thick molybdenum jacket and was successfully extruded after soaking at 1795°C in an argon atmosphere for one hour. A reduction ratio of 6.5:1 was achieved. This ingot preceded the larger ingot No. 2 by several months and was delivered to Mr. Chamberlain at the Dover, Ohio, plant with the molybdenum jacket retained to facilitate his rolling, preheat and breakdown by protecting the core material.

The second extrusion was patterned after the first, and a total weight of 25.9 kilograms (57 pounds) was delivered to the Dover plant of Lamp Metals and Components Department. Both ingots were extruded at the Research and Development Center of the General Electric Company.

Rolling, Swaging, and Wire Drawing

Our approach to processing was influenced by the work reported by W. H. Chang (ASD-TDR-62-211 and P66FPD102) on similar alloys and the similar response to heat treatment of the extruded AS-30, No. 1 shown in Figure IB6. The changes in microstructure through annealing are shown in Figure IB7. The extrusion was annealed prior to rolling and processed through rolling at the aging temperature followed by decreasing temperatures through swaging and wire drawing. The effect of lower processing temperatures was to lower the ductile-brittle transition temperature as an aid to processing as well as induce a high degree of strain hardening to aid in controlling precipitation.

AS-30, No. 1

AS-30, No. 1 extruded bar was recapped and T.I.G. welded to cover the end that was exposed after sampling. The extruded bar was zyglo inspected for defects in the cladding; none were found. The molybdenum jacket was judged adequate for protection during hydrogen firing and subsequent processing.

The extrusion was rod rolled on the same equipment described in Section IA (TZC). AS-30, No. 1 was processed according to Tables IBVI, IBVII, and IBVIII. No difficulties were encountered during rolling until the sixth pass, at which point the welded end plug separated and cracked at the AS-30, molybdenum interface. The rod was rolled through finish size (.650" diameter round), where the contaminated end, approximately 1", was cut off.

The rolled rod was cut in half and cleaned for inspection. No breaks were observed in the cladding by zyglo inspection as well as visual inspection at 10X. Hardness, as rolled, was 290 D.P.H. One rod (AS-30, No. 1B) was set aside for future disposition, and the second rod was processed through swaging to .344" diameter where difficulty was experienced. The original end plug from the extrusion ruptured at the AS-30, molybdenum interface, and a 52" long section of the clad AS-30 began to rupture. The defective section was cut from the rod and stripped in acid; the bare AS-30 was inspected and found to split-free. This section was labeled AS-30, No. 1A3 and was further processed according to Table IBVI and IBVIII.

The remaining .344" diameter rod was cut in half and labeled AS-30, No. 1A1 and AS-30, No. 1A2. AS-30, No. 1A1 and No. 1A2 were hydrogen annealed at 1380°C for one hour. Hardness dropped from 290 D.P.H. before annealing to 250 D.P.H. after. The rods were swaged to .233" diameter with 30% R.A. drafts, then wire drawn on a mechanical bench to .111" diameter using 15% R.A. drafts. At .111" diameter, the rods were vacuum annealed at 1370°C for one hour with a resultant hardness of 240 D.P.H.

AS-30, No. 1 (Cont'd.)

AS-30, No. 1A1 proceeded as shown in Table IBVII. At .044" diameter, the molybdenum jacket was chemically removed in nitric/sulfuric acid solution. The AS-30 surface was star-shaped and very irregular so further etching was attempted in nitric/sulfuric/hydrofluoric acid solution in an effort to smooth the surface prior to room temperature wire drawing. Our success was marginal due to the many breaks experienced caused from slivers associated with the irregular surface. An additional cleaning and etching step at .020" diameter enabled the processing to continue to .005" diameter.

Attempts at room temperature drawing from .018" diameter resulted in many drawing breaks and subsequent poor yield of 340 feet of .005" diameter wire. Drawing at a slightly elevated temperature (200-300°C) proceeded without a break and produced one piece 2,100 feet long.

AS-30, No. 1A2 was processed as shown in Table IBVI. Based on the results of No. 1A1, No. 1A2 was drawn at 200-300°C after stripping and produced 3,940 feet of finished wire. Diameter measurements are shown in Table IBX for 1A1 and 1A2.

AS-30, No. 2

AS-30, No. 2 was one extrusion weighing 57 pounds and too large for handling on our equipment. The extrusion was cut in four equal sections weighing approximately 14 pounds each. Molybdenum end plugs were T.I.G. welded on the exposed ends prior to rolling.

Because of the rather large cross section of the extrusion (2-1/6" diameter), it was decided to roll the material to an intermediate size that would fit into any future roll pattern design. All four pieces of AS-30, No. 2 were rolled 10 passes to a 3/4" square according to the schedule shown in Table IBVI. No difficulties were encountered.

Discussion of Results

Processing through rolling and swaging was not difficult using the temperatures and heating practices shown in Table IBVI. Retention of the molybdenum jacket is mandatory to successful processing through rolling, swaging, and heavy wire drawing. In addition, the molybdenum jacket made stress relieving at rod and heavy wire sizes possible in hydrogen furnaces. After enough ductility (work) had been accrued, the molybdenum jacket was readily etched off. The jacket should be stripped off at the largest possible size to minimize the problem of smoothing out the irregular star shaped cross section.

Wire drawing the bare AS-30 was only successful at slightly elevated temperatures of 200-300°C. It was felt that this temperature range was high enough to enhance processing ductility while being low enough not to seriously contaminate the alloy.

Discussion of Results (Cont'd.)

All finished wire was cleaned of drawing lubricants and chemically etched with .0001" to .0002" metal removal to final size.

Table IBIX presents the recommended process for producing relatively long lengths of .005" diameter AS-30 wire.

TABLE IBI

Analysis of Columbium Granules (ppm)

E. I. duPont deNemours and Company

O	77	Fe	<50
H	-	Cr	<30
N	5	Ni	38
C	12	Ti	<50
Ta	700	W	<50

TABLE IBII

Analysis of Zirconium Additions to AS-30 (ppm)

Wah Chang Corporation

Al	32	Mg	<10	Ti	36
B	0.4	Mn	10	V	<5
C	80	Mo	<10	W	<25
Cb	<100	N	31	Zn	<50
Cd	<0.3	Ni	15	P	<10
Co	<5	O	1142	U	<0.5
Cr	155	Pb	6		
Cu	<25	Si	<46		
Fe	928	Sn	10		
Hf	69	Ta	<200		

TABLE IBIII

Typical Sintering Schedule of AS-30 Electrodes

(Electrode C)

Leak Rate, .4 micron/hour

Time Min.	Pressure Torr.	Amperes Approx. amp	Voltage Approx. volt	Temp. °C ±25	Note
Start	6×10^{-6}				
9	8×10^{-5}		2.5		
15	2×10^{-5}	800	5		
17	2×10^{-4}	960	5	~600	
18	9×10^{-5}	400	2.5	~600	Lowering power
27	9×10^{-5}	800	3	~600	
62	5.6×10^{-5}	800	3	~700	First hold for 35 min.
82	5.2×10^{-5}	1200	3.25	900	
89	6×10^{-5}	1600	3.75	1000	
102	7×10^{-5}	2400	4.25	1200	
107	9×10^{-5}	3200	5	1300	
117	3.4×10^{-5}	3200	5	1300	
132	3×10^{-5}	3800	5.4	1350	
147	3.6×10^{-5}	4500	5.6	1450	
162	4.5×10^{-5}	4800	5.8	1500	Final hold one hour
172	3.6×10^{-5}	4800	5.8	1500	
179	4.2×10^{-5}	4800	6.0	1500	
182	3.0×10^{-5}	4800	6.0	1500	
192	2.4×10^{-5}	4800	6.0	1500	
200	2.2×10^{-5}	4800	6.0	1500	
212	2.0×10^{-5}	4800	6.0	1500	
217	1.8×10^{-5}	4800	6.0	1500	
222	1.8×10^{-5}	Power off			

TABLE IBIV

Melting Data for AS-30

Ingot No.	Current Amp	Electrodes Used	Predominant Vacuum Torr.	Casting Inches	Casting Pounds	Melting Rate Lb./Min.
AS-30 No. 1	4300	B,C,D	4×15^{-4}	11.86	48.5	2.5
AS-30 No. 2	3800	A,E,F	4×15^{-5}	10.5	42.5	2.7

TABLE IBV

Chemical Analysis of Remelted AS-30 Ingots
(Cb-20W-1Zr-.09C)

Spectrographic Analysis, Weight Percent

	Cr	Fe	Mo	Ni	Si	Ta	Ti
Ledoux	N.D. <.001	.002	.003	.004	.001	.07	N.D.< .001

(N.D.< - not detected, less than)

LMCD	W	Zr % by weight
AS-30 No. 1 (small ingot)		
Top Center	23.4	.74
Side Turnings	22.3	.58
AS-30 No. 2 (large ingot		
Top Center	21.8	.57
Side Turnings	21.8	.75

Ion Exchange, Gravimetric Precipitation (LMCD)

AS-30 No. 2	
Top	1.0
Side	1.1

Vacuum Fusion Analysis (LMCD) ppm and Carbon Analysis

	No. 1	No. 2
O_2	39	33
N_2	28	33
H_2	1	1
C	529 (1)	540 ave. 6 locations

TABLE IBVI

2" Dia. Extruded Round With Mo Can (20.2 Lbs. Including Mo Can)
Soak Anneal at 1510°C. - 1 Hour in H_2
Rod Rolled 12 Passes at 1510°C.
Reheat Every Two Passes, 10 Min. Soak
Finish Size 0.650" Dia. Round
Total Reduction In Area - 89.4% - As Rolled DPH 290

← ½ | ½ → 10.85 Lbs.

AS-30, No. 1A

Clean - Inspect Zyglo

Swage: .560" at 1225°C.
.460" at 1100°C.
.390" at 1100°C.
.344" at 1100°C.

Cut ½ →

AS-30, No. 1A1

Stress Relieve 1380°C.
1 Hour in H_2
DPH 294 Before
DPH 250 After

Swage .281", .233" at 900°C.

Draw Bench 15% Drafts
to .111" Dia.

Vacuum Anneal
1375°C. - 1 Hr. - DPH 240

25 Ft. | 25 Ft.

See Table 1BII

AS-30, No. 1B
Held for Future Disposition
10.8 Lbs. Including Mo Can

Cladding Ruptured

AS-30, No. 1A2, 84" Long

Same Processing as No. 1A1
to .111" Dia.

Vacuum Anneal 1375°C. - 1 Hr.

Oxidize and Lube
Wire Draw 600-700°C.
10-15% R.A. per Pass
to .015" Dia.

Molybdenum Can Etched Off

Wire Draw 8% R.A. per Pass
at 200-300°C. to .0052" Dia.

Etch to .005" Dia.

Yield: 3,940 Feet

AS-30, No. 1A3, 52" Long
Mo Stripped Off
.321" Dia. As Stripped
Centerless Ground to .280" Dia.

Vacuum Anneal 1370°C. - 1 Hr.

Clad in Ni Tube

Swage: .235" at 1000°C.
.185" at 1000°C.
.165" at 950°C.
.145" at 950°C.

Cladding Ruptured

See Table 1BIII

TABLE IBVII

.111" Dia. Moly Clad AS-30, No. 1A1 (Approx. 50 Feet)

Vacuum Anneal - 1375°C. - 1 Hr.

D.P.H. 240-243

3 Pieces - 3 Ft., 22 Ft., and 25 Ft.

Oxidize and Lube

Wire Draw

.104" - 800°C.
.100" - 800°C.
.095" - 800°C.
.089" - 800°C.

.086" - 700°C.
.079" - 700°C.
.073" - 700°C.
.067" - 700°C.
.061" - 700°C.
.056" - 700°C.
.044" - 700°C.

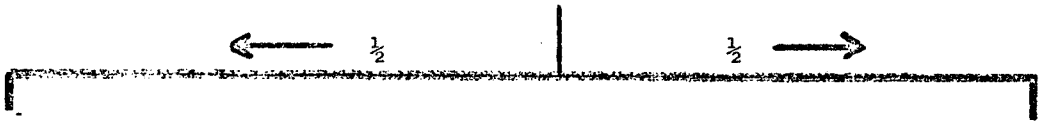
Molybdenum Can Etched Off

Wire Draw at Room Temperature

11% to 12% R.A. per Pass

.034" to .020"

Clean and Etch to .015"-.018"



Wire Draw at 200-300°C.

7% R.A. per Pass to

.005" Diameter.

Yield: 1 Piece - 2,100 Feet

Wire Draw at Room Temperature

7% R.A. per Pass to .010" Dia.

Clean and Etch to .009" Dia.

Wire Draw at Room Temperature

Yield: 2 Pieces - 40 Ft. and 300 Ft.

TABLE IBVIII

AS-30, No. 1A3

2 Pcs. - Ruptured Nickel Cladding .145" Dia. (Overall)

↓

Stripped Cladding

↓

Conditioned AS-30 - 1A3
Ground .010" from Surface

↓

Clad in New Nickel Tube
Shrink Pass through .170" Dia. (Overall)
Wire Draw 15% R.A. per Pass to .044" Dia., 600-700°C.

↓

Stripped Cladding

↓

Wire Draw at Room Temperature

↓

.032"

↓

.031"

↓

.030"

↓

Chemically Clean and Etch to .0296" Dia.

↓

Vacuum Anneal - 1375°C. - 1 Hr.

↓

Wire Draw 10% R.A. per Pass to .015" Dia. at Room Temperature

1A3A1	1A3A2
↓	↓
Wire Draw 7% R.A. per Pass to .005" Dia. at Room Temperature	Wire Draw 10% R.A. per Pass to .005" Dia. at 200-300°C.
Chemically Cleaned	Chemically Cleaned
Yield: 187 Feet	Yield: 560 Feet

TABLE IBIX

Recommended Process for Fabrication of
.005" Diameter AS-30 Wire from
2.00" Diameter Extruded Rounds

2.00" Diameter Extruded Round with Molybdenum Jacket
Soak Anneal at 1510°C. - 1 Hour in H_2

↓

Rod Roll at 1510°C.
Reheat at 1510°C. Every Two Passes, 10 Min. Soak
Finish Roll to Nominal .650" Round
Total R.A. 89%

↓

Clean and Inspect Cladding for Ruptures

↓

Swage 30% R.A. per Pass at Decreasing Temperatures
(1225°C. to 1050°C.) to .344" Diameter

↓

Stress Relieve Anneal 1380°C. - 1 Hour in H_2

↓

Draw on Draw Bench 15% R.A. per Pass at
600/700°C. to .100" Diameter

↓

Clean and Wrap in Self-contained Coil

↓

Vacuum Stress Relieve Anneal 1 Hr. at 1375°C.

↓

Wire Draw 15% R.A. per Pass at 600/700°C.
to .030" Diameter

↓

Strip Molybdenum Jacket

↓

Chemically Etch to Smooth AS-30 Surface

↓

Wire Draw 10% R.A. per Pass at 200/300°C.
to .0052" Diameter

↓

Chemically Etch to Finish Size of .0050"

TABLE IBX

Diameter Measurements of Finished Wire

Spool No.	Lead End	Tail End
12 (1A1)	.00498"/.00496"	.00464"/.00500"
13 (1A2)	.00490"/.00496"	.00479"/.00464"
14 (1A2)	.00496"/.00500"	.00485"/.00489"
15 (1A2)	.00519"/.00500"	.00483"/.00488"
16 (1A2)	.00479"/.00486"	.00491"/.00487"
17 (1A2)	.00490"/.00488"	.00500"/.00496"

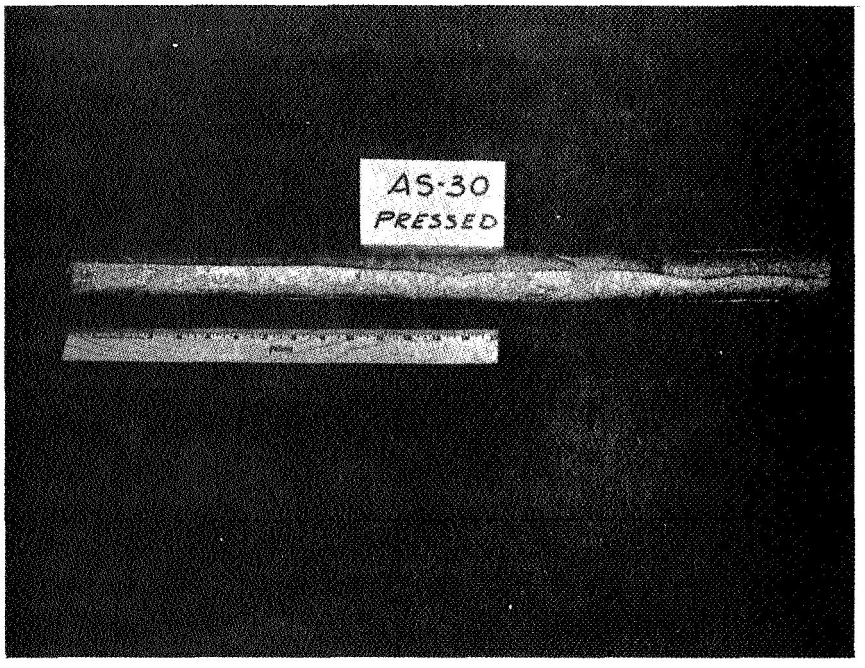


FIGURE IB1 - Cb-Base alloy, AS-30 electrode in the pressed condition

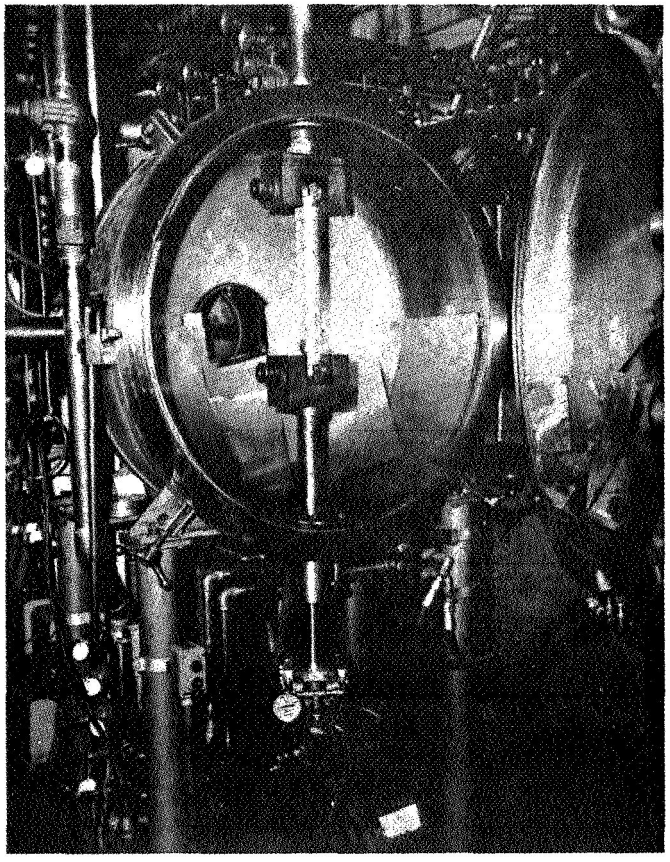
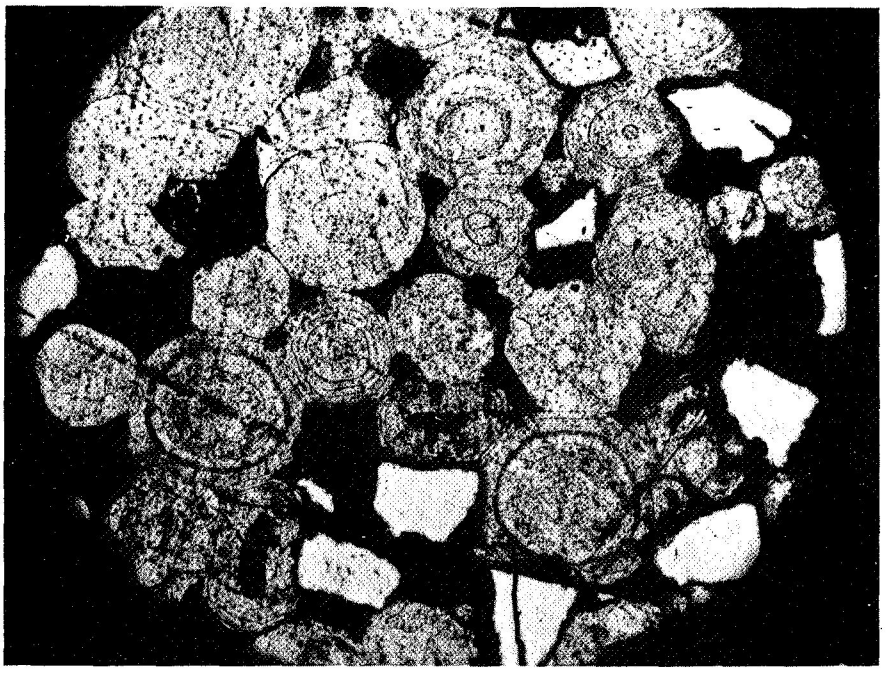
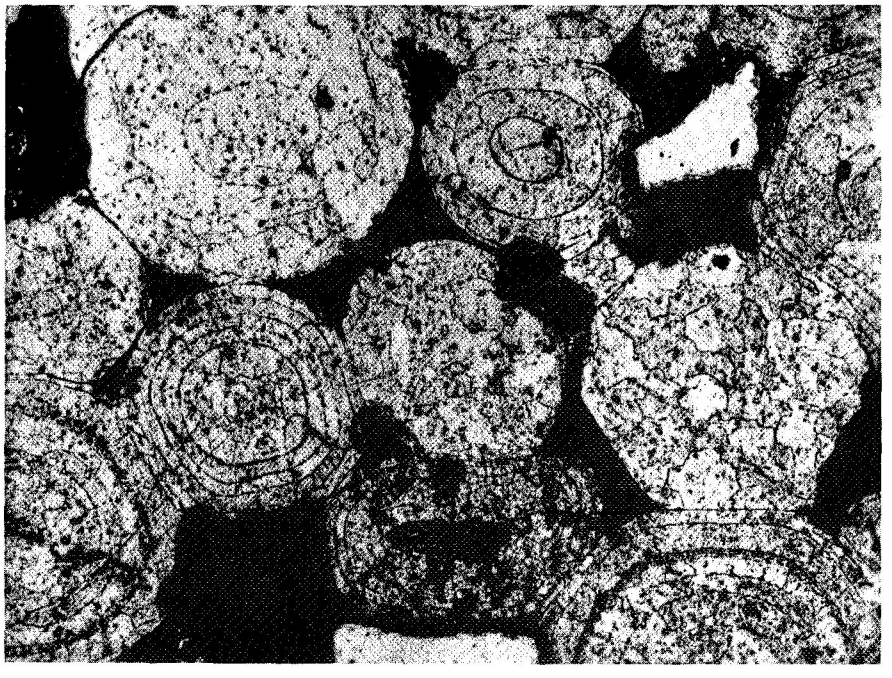


FIGURE IB2 - High vacuum resistance sintering furnace

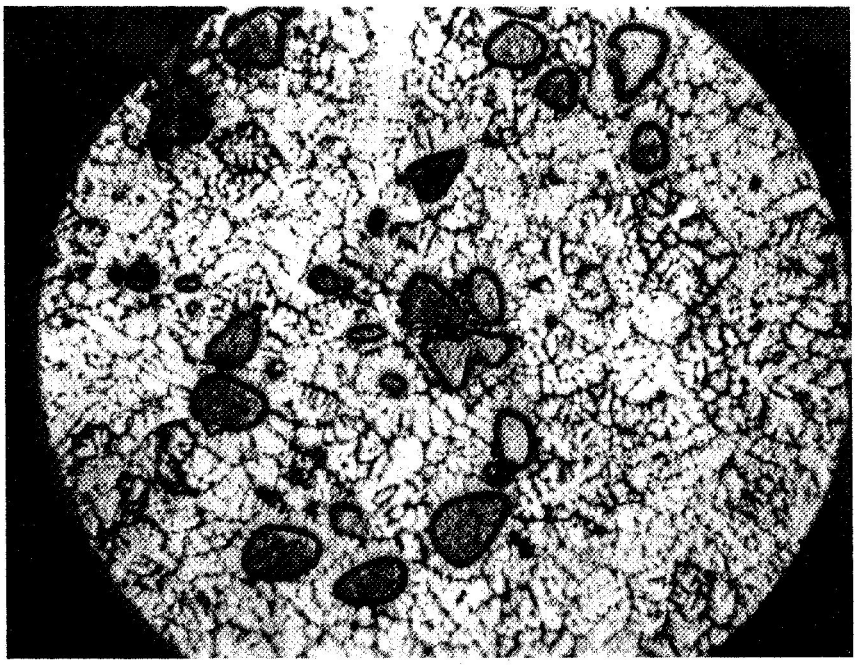


25X 7817



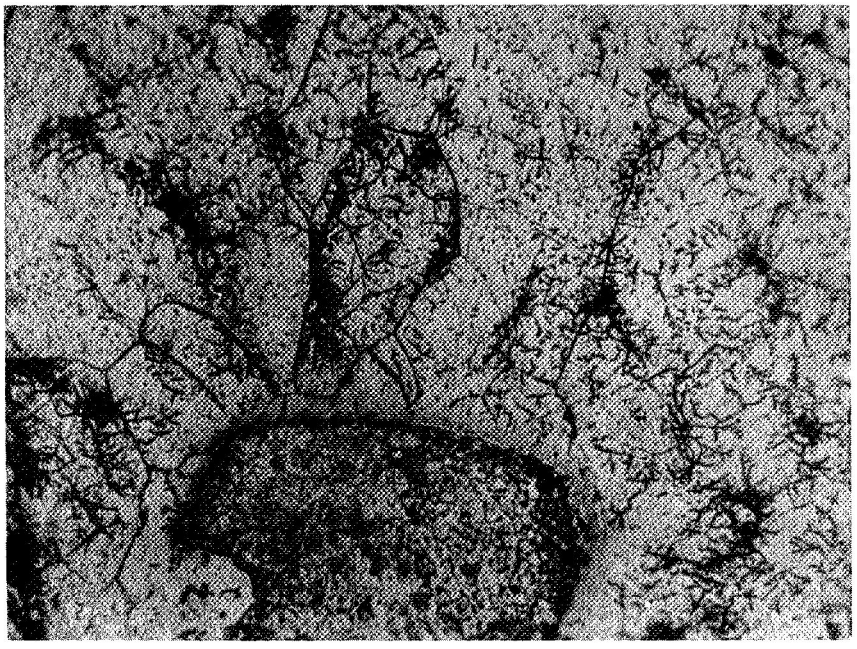
50X 7818

FIGURE IB3 - AS30 pressed and sintered "logs" made from columbium granules



25X 7835

FIGURE IB4a - Macrostructure of AS-30 No. 1



200X 7836

FIGURE IB4b - Microstructure of Cb-Base Alloy, AS-30 No. 1

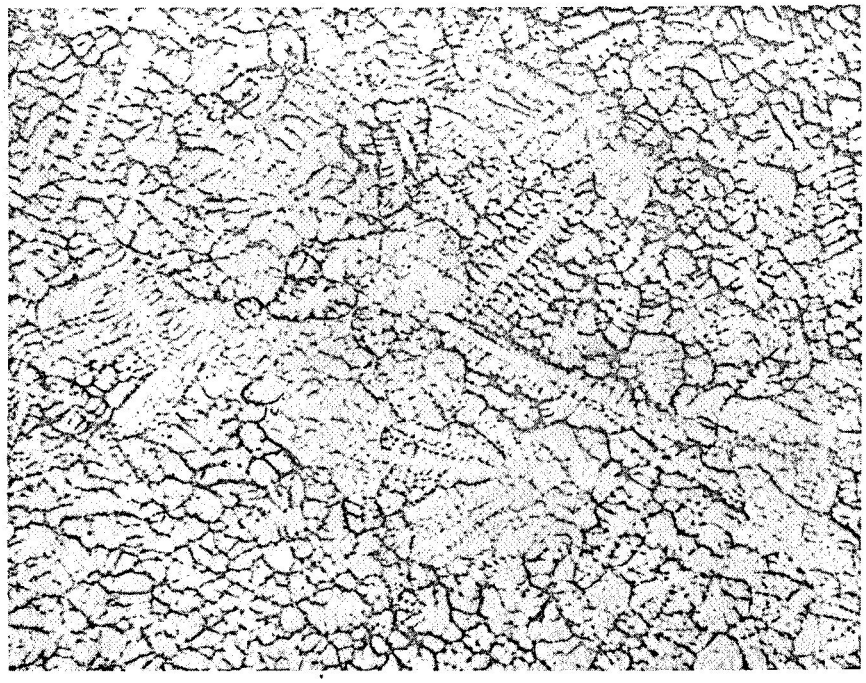


Figure IB5a - Macrostructure of remelted AS-30 No. 2. Tungsten completely alloyed in remelt as verified by improved metallographic technique. 25X

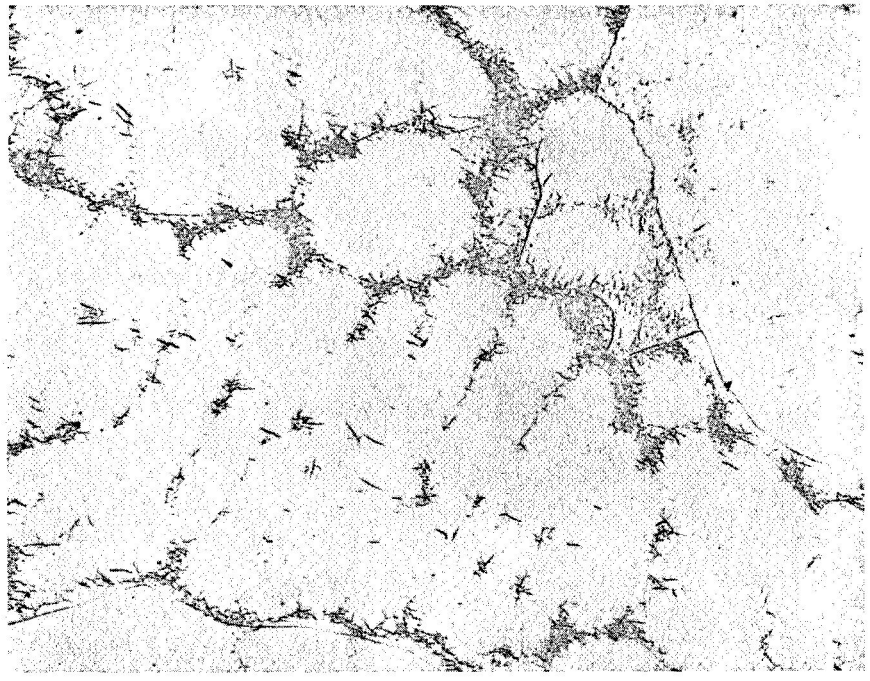


Figure IB5b - Microstructure of remelted AS-30, No. 2 200X

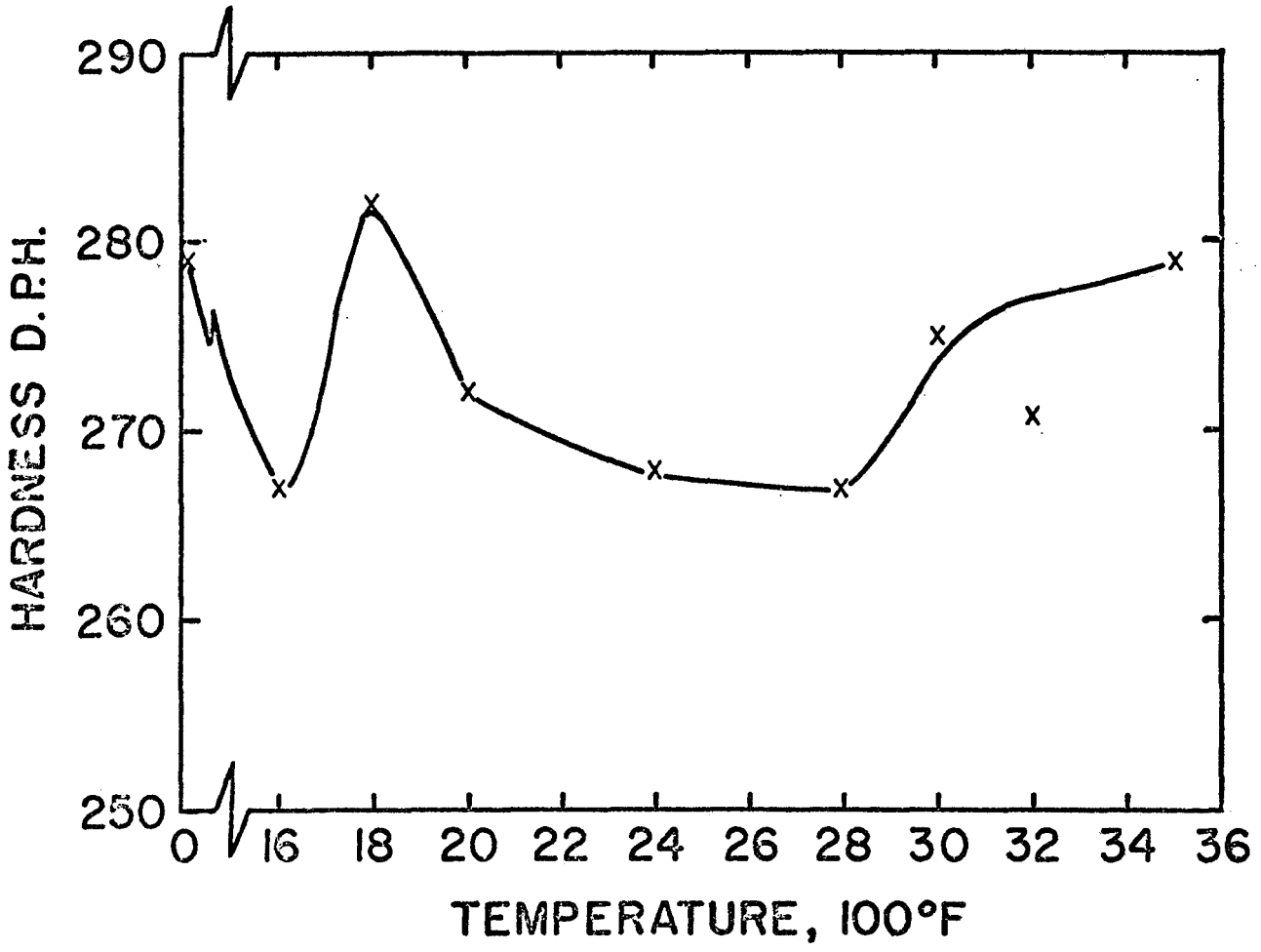
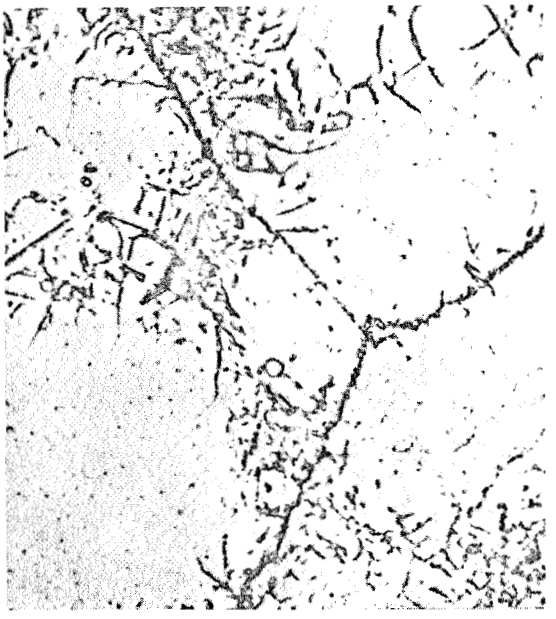
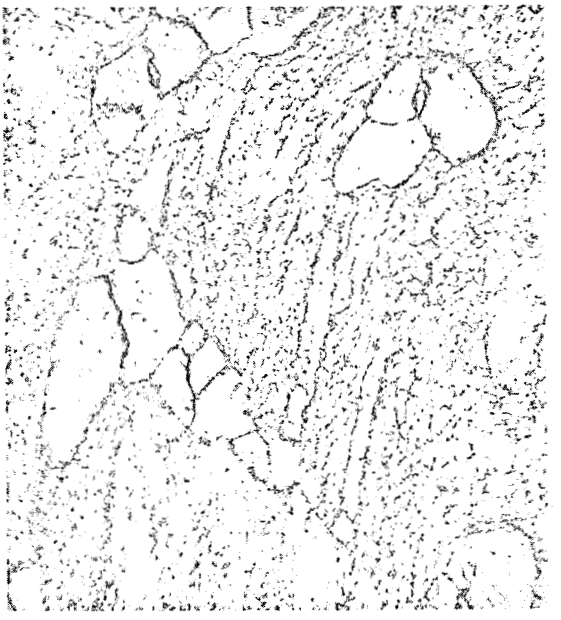


Figure IB6

Effect of One-Hour Annealing on Hardness of Cast and Extruded Cb-Base Alloy AS-30 No. 1

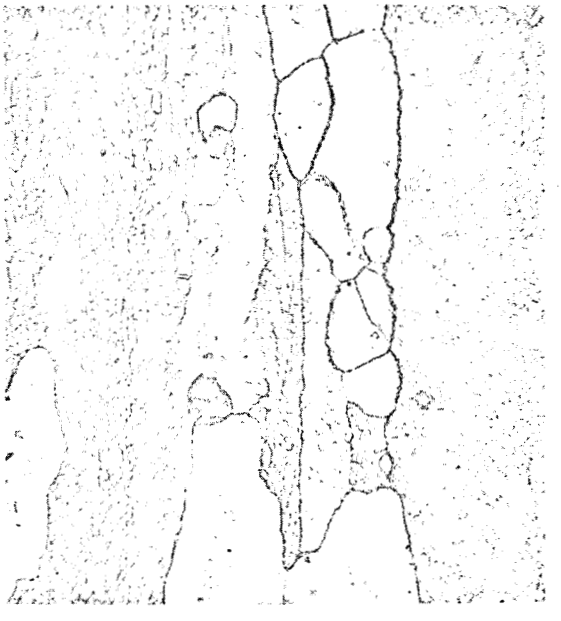


Cast 1000X 7931



Extruded 500X 7994

1600°F 500X 7995



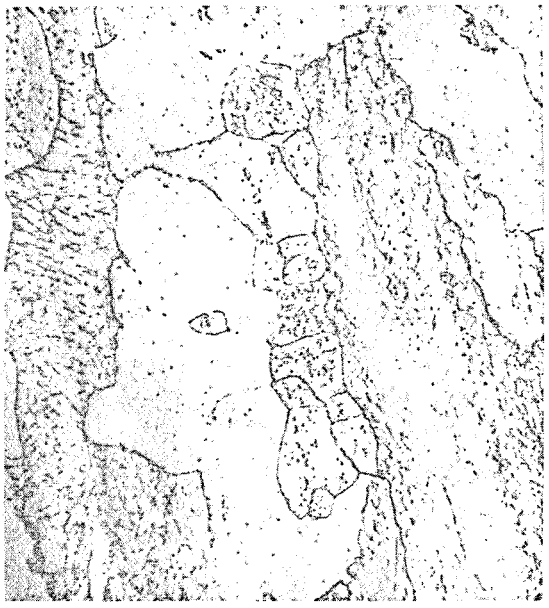
1800°F 500X 7996

Figure IB7a

Microstructural Changes in AS-30 Cast, Extruded,
and Annealed at Indicated Temperatures

2000°F 500X 7997

2400°F 500X 7998

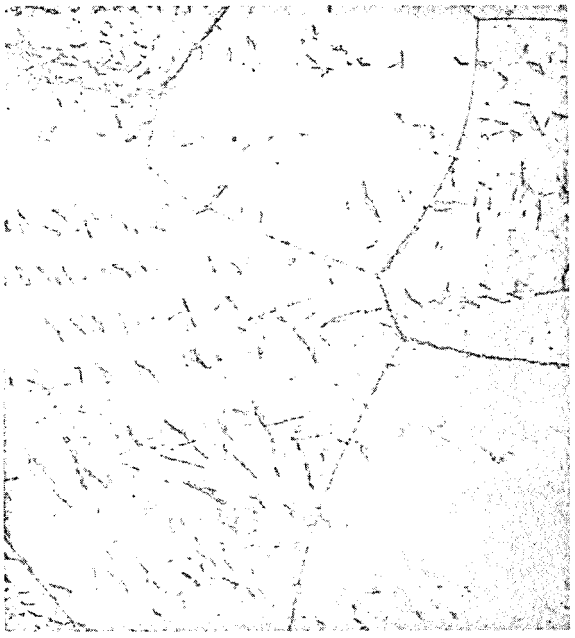


2800°F 500X 7999

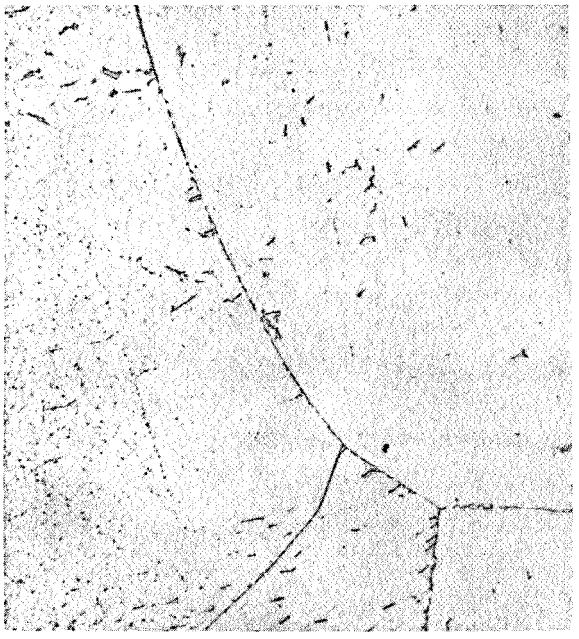
3000°F 500X 8000

Figure IB7b

Microstructural Changes in AS-30 Cast, Extruded,
and Annealed at Indicated Temperatures



3200°F 500X 8001



3500°F 500X 8002

Figure IB7c

Microstructural Changes in AS-30 Cast, Extruded
and Annealed at Indicated Temperatures

C. Tungsten-Rhenium-Thoria Alloy
W+(4-6)Re+(2-4)ThO_2

Powder and Ingot Preparation

A note of explanation at this point may prevent confusion on the part of those to whom "ingot" means an object cast from a molten metal. At the Lamp Metals and Components Department of the General Electric Company, a local jargon has become the accepted terminology during 60 years of tungsten manufacturing, and a bar shaped compact, pressed from tungsten powders is called an ingot. When electric current is sent through the bar in sufficient amount to heat the bar almost to the melting point this is called "treating", even by metallurgists who know that it really should be called sintering. Likewise, when a wrought rod is heated electrically to recrystallize it, local jargon calls it "retreating". Therefore if, in addition to the frequent use of "ingot" in the two tungsten sections of the report, one finds the word treating or retreating, it means editorial effort failed to change these to more recognizable terms.

A special blend of tungsten plus 2.75 percent, by weight, of ThO_2 was prepared using commercial procedures. An aqueous solution of thorium nitrate [$Th(NO_3)_4.H_2O$] was added to ammonium paratungstate crystal, dried, and then the mixture was fired at 1100°C to green oxide. The oxide was attrition milled, and then reduced to metal powder by firing in a hydrogen atmosphere at about 1000°C. Table ICI lists a complete analysis of the tungsten-thoria powder used in these experiments. It should be noted that the thoria analysis show 2.6±.1% present. Later references to 2.75% or 2.2% thoria levels really mean nominal or intended development of thoria and not actual.

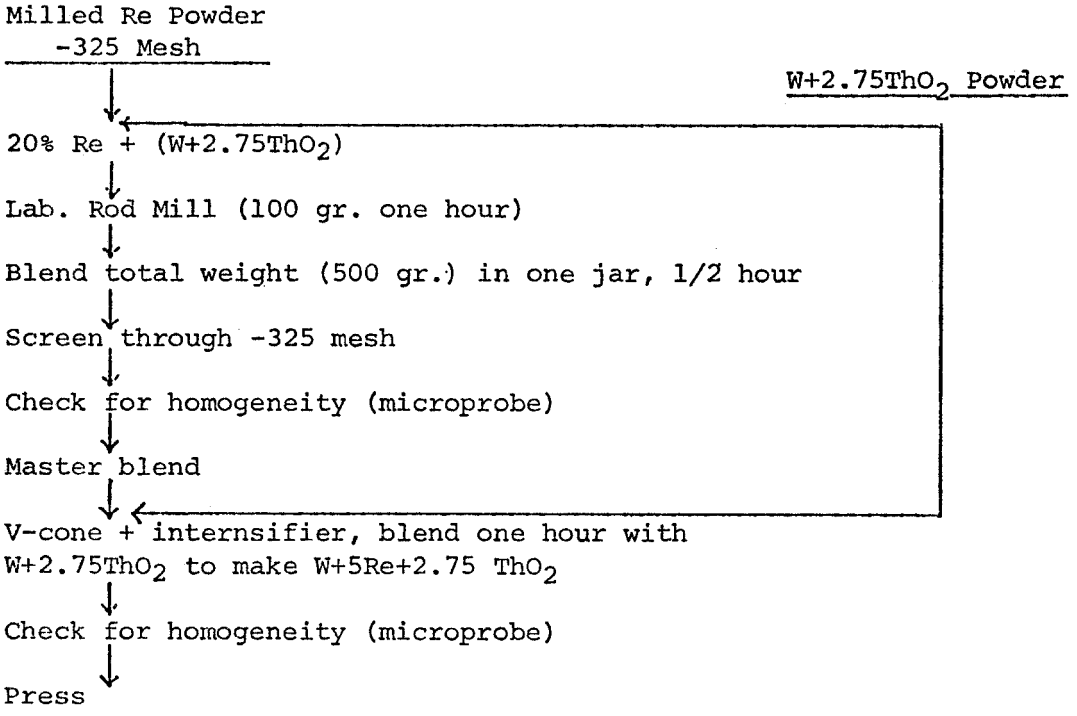
Table ICII shows the vendor analysis of rhenium powder purchased from the Chase Brass Company. The as-received rhenium powder was milled for one hour and then screened through -325 mesh. Five weight percent rhenium was added to the W-2.75 ThO_2 powder followed by blending for one half hour in a V-cone blender. Because rhenium is a costly metal, small powder batches and small ingots or bars were pressed during this learning stage. The ingot mold produced bars measuring 5/8" x 13/16" x 16" when a pressure of 22.5 tons per square inch was used to compress the powder. Each tungsten alloy bar was numbered consecutively, as it was pressed, to identify it through subsequent processes.

The first bar successfully removed from the ingot mold (WRT-1) was presintered for one hour at 1200°C, in hydrogen, to improve the green strength. It was then resistance sintered in a hydrogen "bottle" at 2830°C for one half hour. The sintered ingot showed segregation of rhenium and had a density of around 92%. Experience indicated that this density was not sufficient for fabrication.

Powder and Ingot Preparation (Cont'd.)

Slices cut from the ends of each sintered ingot were examined. The microstructure of Ingot WRT-1 as shown in Figure IC1, indicated localized rhenium melting which upon freezing resulted in shrinkage cavities. The reasons for this failure were a combination of inadequate and poor blending as well as insufficient sintering time for complete dissolution of the rhenium particles in the tungsten matrix.

To alleviate the problem of rhenium segregation, a new and more thorough blending technique was followed as summarized below:



The new powder mixture was pressed and then presintered for one hour at 1200°C in a muffle furnace using a hydrogen atmosphere, followed by an additional presintering treatment at 1850°C for six hours. The bars were then resistance-sintered in a hydrogen atmosphere according to a prescribed schedule as shown in Table ICIII. Initially, there were three high temperature holding periods with a 2700°C final hold. This original plan was later modified to include a fourth holding period at 2900°C for 1/2 hour to obtain the high density (over 95% theoretical) required. Table ICIV summarizes the various techniques and results that were obtained on all ingots.

The final method that was developed to produce uniform, homogeneous ingots of this alloy differs from that previously described in the literature, Ratliffe et al. (1). The differences are primarily in the methods of powder preparation and consolidation: mechanical pressing and self-resistance sintering in this investigation versus gas pressure bonding.

Powder and Ingot Preparation (Cont'd.)

Improvements in blending techniques allowed a more uniform rhenium distribution to be obtained in the W+ThO_2 matrix. Using a microprobe examination technique, it was shown (Figure IC2) that the rhenium can be fairly evenly distributed in a green compact, made from the same starting constituents, by varying only the blending procedure. Also, the microprobe method demonstrated that the as-sintered ingots were fairly homogeneous alloys. In Figure IC3 the microstructure of representative ingots (WRT-4, WRT-5, WRT-8, WRT-9) and the dispersion alloy WRT-12 are shown for comparison. The photomicrographs reveal that no gross rhenium segregation occurred. The thoria dispersion was uniform and no evidence of ThO_2 gross agglomeration was observed. Table ICIV shows that the grain size ranged from 32,000 to 53,000 grains/mm^2 for the W-Re-ThO_2 alloy. Unalloyed tungsten processed under similar conditions would be in the range of 20,000 grains/mm^2. This grain refinement is attributed mostly to the addition of the thorium oxide rather than the rhenium metal. It is obvious that grain refinement, as evidenced in the W-ThO_2 alloy (Ingot WRT-12) has been due to the ThO_2 dispersion.

The results of blending and sintering can be summarized as follows:

1. There was complete solid solution of rhenium in tungsten. No sigma phase was evident.

2. Elimination of internal defects, i.e., large holes, shrink cavities, etc., was accomplished.

3. High density; well over 95% of theoretical was obtained.

4. Thoria seemed to be well dispersed and no gross agglomeration was observed.

5. A high degree of grain refinement was obtained, as well as grain size uniformity across the cross section of the sintered ingot.

Ingot Breakdown History

Because considerable difficulty was encountered in the rolling and swaging of the tungsten-rhenium-thoria alloys, there was also considerable effort made to alter the working schedules to try to overcome these difficulties. As a result, the various ingots (sintered bars) were not necessarily processed in a chronological order but to reduce the confusion that would result from a chronological discussion, the following report will fully describe processing of each bar from start to finish. In general, whenever end splits were found after a rolling pass, that end was trimmed back to sound metal to prevent further propagation. The bar cross-section after sintering was nominally .710 x .530 inches.

Ingot Breakdown History (Cont'd.)

WRT-3 (5Re+2.75ThO_2) (See roll pass design page 65)

Roll Pass	Time, Minutes	Temp. °C	Remarks
1*	10	1800	Light pass to break corners.
2*	10	1800	Transverse cracks.

*Swaged

Discontinued

WRT-2 (5Re+2.75ThO_2)

1	10	1730	Split 1-1/2" long at nose.
	Recrystallize by resistance heating, 5000 amps.		
2	10	1710	No new cracks visible.
3	10	1710	Split full length.

Discontinued

WRT-4 (5Re+2.75ThO_2) Clad in Molybdenum

1	30	1700	Light pass to shape molybdenum cladding.
2	15	1700	Good appearance.
3	15	1700	Good appearance.
4	10	1700	Good appearance.
5	15	1450	Good appearance.

Pickled to remove molybdenum cladding, core about .500" diameter. Inspection found many transverse cracks and tears.

WRT-5 (5Re+2.75 ThO_2) Clad in Molybdenum

1	30	1700	Light pass to shape molybdenum cladding.
2	15	1700	Good appearance.
3	15	1700	Good appearance.
4	30	1450	Tears evident even through cladding.

Discontinued

To reduce the loss of valuable material, an effort was made to explore the cause for failure which was presumed to be the high additive levels. Prior experience with the three percent rhenium-tungsten commercial alloy which was patented by LMCD had not encountered such failures, nor do the standard 2% ThO_2-tungsten materials in daily production suffer such breakage. The combination of the 5 rhenium - 3 ThO_2 was being developed at NASA's request and no known commercial supply had been found. Therefore, two bars were pressed and processed without the rhenium addition, WRT-12 and 13, to isolate the main contributant to the failures.

Ingot Breakdown History (Cont'd.)

WRT-12 (2.75% ThO_2)

Roll Pass	Time, Minutes	Temp. °C	Remarks
1	20	1680	Fractured at tail. Transverse cracks, checks, and chipping appear to be related to ingot consolidation.

WRT-13 (2.75% ThO_2)

1	20	1680	No cracks visible.
2	20	1630	1-1/2" split at nose.
	Recrystallize by resistance heating, 3650 amps in H_2.		
3	20	1630	Slight nose and tail cracks.
4	20	1595	No cracks visible.
5	10	1530	No cracks visible - to swaging at approximately .385" diameter.
	Recrystallize by resistance heating at 2000 amps (2500°C) - 10 minutes, H_2		
Swaged .330" dia.	10	1600	Good appearance.
Swaged .280" dia.	10	1600	Good appearance.
Swaged .235" dia.	10	1600	Good appearance.
	Recrystallize by resistance heating at 950 amps (2100°C) - two minutes - H_2 - 1100 grains/mm^2 - hardness R_c 39.		
Swaged .203" dia.	10	1650	2" split on end cutoff.
	Discontinued, accomplished purpose, no rhenium		

The success in rolling could be attributed to the longer preheat times (20 minutes vs. 10 minutes), the increase in draft (20% elongation/pass instead of 10 to 15% elongation), to the relocation of the recrystallization anneal from after the first pass to after the second pass, and lastly to the absence of rhenium.

Based on the encouraging results with WRT-13, a few W+5 Re ingots (bars) were made with two different thoria levels, to determine the level that would be conducive to rolling.

WRT-9 (5Re+2.2ThO_2)

1	20	1710	Split 1-1/2" at nose.
2	20	1650	No cracks visible.
	Recrystallize by resistance heating at 4100 amps in H_2. Grain count 2480 - 7130 grains/mm^2. R_c 31 -33.		

Ingot Breakdown History (Cont'd.)

WRT-9 (5Re+2.2ThO_2) (Cont'd.)

Roll Pass	Time, Minutes	Temp. °C	Remarks
3	20	1650	After rolling, split full length while cooling.

Discontinued

WRT-10 (5Re+2.2ThO_2)

1	20	1710	Split 1-1/2" at nose.
2	20	1650	Split 1-1/2" at nose.

Recrystallized by resistance heating at 4100 amps, in H_2. Grain count 3100/mm^2 at edge, 11460/mm^2 in center. R_c 31-34. Only 6" long, at this point after trimming, not processed further.

WRT-6 (5Re+2.75ThO_2)

1	20	1720	No cracks visible.
2	20	1650	No cracks visible.

Recrystallized by resistance heating at 2090 amps (2500°C) 10 minutes - H_2, grain counts 3100/mm^2 edge, 10300/mm^2 center, R_c 33-35.

3	20	1650	Good appearance.
4	10	1600	Edge tears.

Discontinued

WRT-11 (5Re+2.2ThO_2)

1	20	1710	Cracked at tail.
2	20	1640	2" crack at nose.

Recrystallized by resistance heating at 3840 amps, in H_2 (10 minutes). Grain counts: 38,000/mm^2 edge; 42,600/mm^2 center.

3	10	1650	Good appearance.
4	10	1655	Good appearance.
5	10	1600	Good appearance.

Recrystallize by resistance heating at 1730 amps (2200°C) in H_2 (10 minutes). Grain counts: 12,300/mm^2 edge, 12,300/mm^2 center, R_c 41

Ingot Breakdown History (Cont'd.)

WRT-11 ($5Re+2.2ThO_2$) (Cont'd.)

Roll Pass	Time, Minutes	Temp. °C	Remarks
Swaged .330" dia.	10	1600	Good appearance
Swaged .280" dia.	10	1600	5" crack on end, several radial and longitudinal cracks due to excessive heat loss.
Swaged .235" dia.	10	1660	Cracks did not propagate.

Recrystallized by resistance heating at 950 amps (2100°C) in H_2 (two minutes). Grain count: 2350/mm^2 edge, 3975/mm^2 center, R_c 33.

Swaged .203" dia.	10	1650	Severely split and slivered

Discontinued

WRT-7 ($5Re+2.75ThO_2$)

This ingot split full length during first rolling pass.

Discontinued

At a joint meeting of NASA and General Electric engineers in April, 1966, it was agreed to terminate all efforts to produce either the sintered bars or the resultant wire.

Experimental Roll Pass Design for Tungsten (Box Pattern)

Groove No.	Depth ±.002"	Width at Bottom ±.002"	Side Wall Angle	Blend Radii
1	.245"	.796"	8°	3/32"
2	.240	.622	8°	3/32
3	.155	.657	8°	3/32
4	.180	.454	8°	1/16
5	.125	.537	8°	1/32
6	.385 Dia.	--	-	--

Nominal Gap .050", adjustable

Reference

Ratliffe, J. L., Abbott, W. H., Maykuth, D. J., Ogden, H. D., Blocher, J. M., Jr., Jaffe, R. I., "Further Development of a Ductile Tungsten Base Sheet Alloy", 2nd Quarterly Progress Report, December 19, 1964, Bur. Nav. Weaps. Contract N600 (14) 61982.

TABLE ICI

Analysis of W+2.75ThO_2 (Nominal)

Below are listed the analyses of the tungsten powder lot No. ID 65-8-56. This material was transferred on 8/23/65. Qty. 50.000 kgs.

Spectrographic Analysis

Element	ppm	Element	ppm
Al	<6	Ni	20
Ca	17	Cu	<3
Si	11	Mn	<6
Mo	52	Mg	7
Fe	18	Sn	10
Cr	5		

Flame Photometry Analysis

Sodium - 1 ppm
Potassium - 25 ppm

LECO Analysis

Carbon - 11 ppm

Average Particle Diameter by Fisher Sub-Sieve Sizer

	as supplied	Lab. milled
Number (microns)	2.57	2.49
Porosity	.659	.488

ThO_2 - 2.6±.1% (by x-ray fluorescence)

Apparent Density by Scott-Schaeffer-White Volumeter (ASTM B-329 58)

67.7 gms. per cu. in.

Wt. % Particle Size Distribution by Photelometer

Micron Range	Lab Milled
0- 1	2.4
1- 2	17.6
2- 3	38.3
3- 4	27.8
4- 5	10.4
5- 6	.7
6- 7	.8
7- 8	.9
8- 9	1.0
9-10	-
10-11	
11-12	
12-13	
13-14	
14-15	
15-20	
20-25	

TABLE ICII

Physical, Chemical Analysis of Rhenium Powder
Chase Brass Company

Batch 446

Description - Rhenium Grade 1 -200 mesh

Microns - 2.35

Scott Density gm/in^3 - 27.6

Spectrographic Analysis (ppm)

Element	ppm	Element	ppm
Al	<1	Mg	<1
Ca	<1	Mo	<1
Cr	<1	Ni	1
Cu	1	Si	<1
Fe	15		

Gas Analysis - performed by the General Electric Company, Oxygen = 2860 ppm

TABLE ICIII

Sintering Schedule of W-Re-ThO_2 Alloys (Typical)

Presintered one hour at 1200°C and six hours at 1850°C, followed by resistance sintering according to the following schedule:

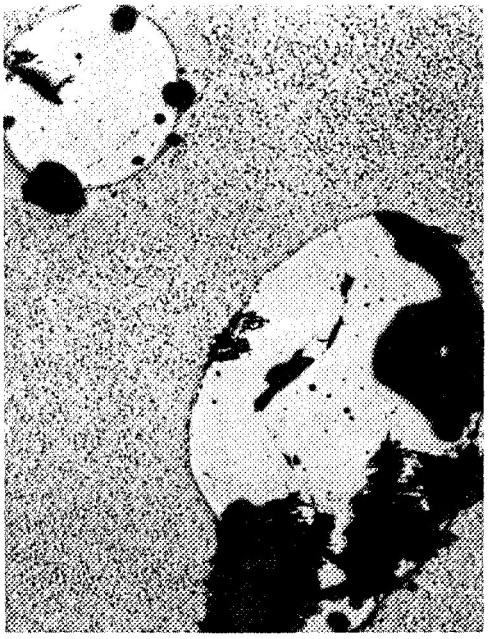
Time Min.	Current Amps	Temperature °C±25
1	1200	
2	1600	1750
3	2000	
4	2400	
5	2800	
5-1/2	3000	2270
6	3200	
7	3600	
8	4000	2500
18	1st hold for 10 min.	
18-1/2	4500	2570
48-1/2	2nd hold for 30 min.	
49	5000	2700
79	3rd hold for 30 min.	
80	5300	2900
110	4th hold for 30 min.	

TABLE ICIV

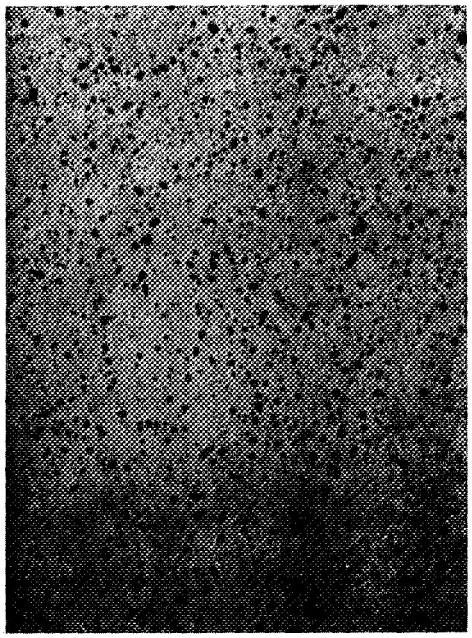
Summary of Sintering Conditions and Properties of
Tungsten + Rhenium + Thoria Alloys

Ingot No.	Additions Wt.%	H_2 Sintering, 1/2 Hr. 1st Temp. °C ±25	H_2 Sintering, 1/2 Hr. 2nd Temp.	Grain Size Grains/mm² X1000	*Theoretical g/cm³	Density, ± .15 g/cm³ 1st density g/cm³	Density, ± .15 g/cm³ 2nd density g/cm³	Remarks
WRT1	5Re+2.75ThO_2	2830	-	-	18.9	17.30	-	Poor blending, low density
WRT2	5Re+2.75ThO_2	2830	-	-	-	17.60	-	Good blending, low density
WRT3	5Re+2.75ThO_2	2830	-	-	-	17.75	-	" "
WRT4	5Re+2.75ThO_2	2830	-	-	-	17.65	-	" "
WRT5	5Re+2.75ThO_2	2830	-	-	-	17.75	-	" "
WRT6	5Re+2.75ThO_2	2800	2900	53	-	17.27	17.92	Good blending, increased density
WRT7	5Re+2.75ThO_2	2800	2900	53	-	17.35	18.01	" "
WRT8	5Re+2.75ThO_2	2800	2900	46	-	17.38	18.01	" "
WRT9	5Re+2.20ThO_2	2830	2900	34	19.0	17.40	18.01	" "
WRT10	5Re+2.20ThO_2	2830	2900	49	-	17.40	18.11	" "
WRT11	5Re+2.20ThO_2	2830	2900	32	-	17.40	18.10	" "
WRT12	2.75ThO_2	2800	2900	40	18.8	17.00	17.96	" "
WRT13	2.75ThO_2	2800	2900	-	-	17.00	17.96	" "

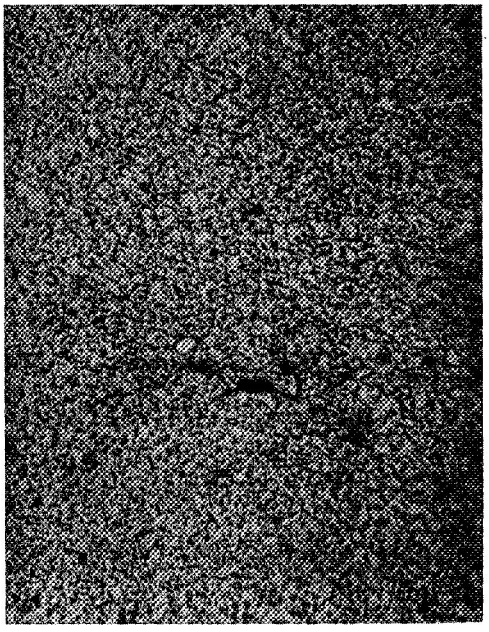
*Calculated using formula $\frac{1}{d_T} = \frac{\text{Wt. \% A}}{d_A} + \frac{\text{Wt. \% B}}{d_B} + \ldots$, where densities of Re, W and ThO_2 are 21.04, 19.30 and 10.03 g/cm³ respectively.



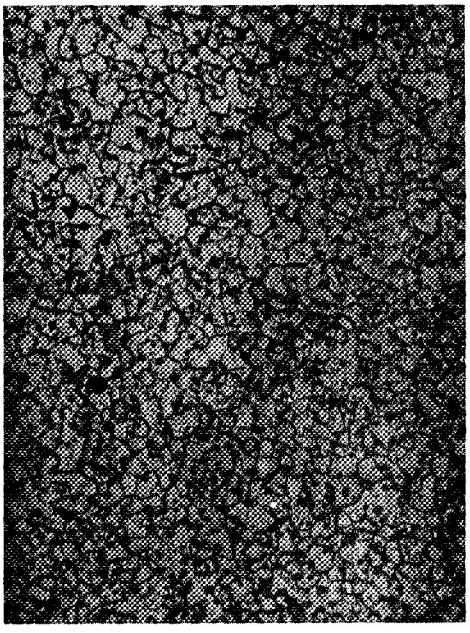
Polished showing localized Re melting



Polished showing ThO_2 phase

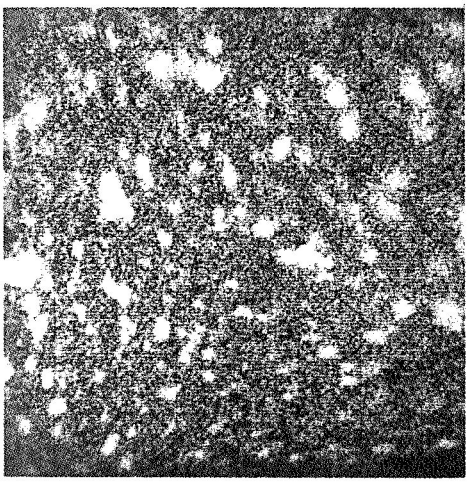


Large porosity-polished.

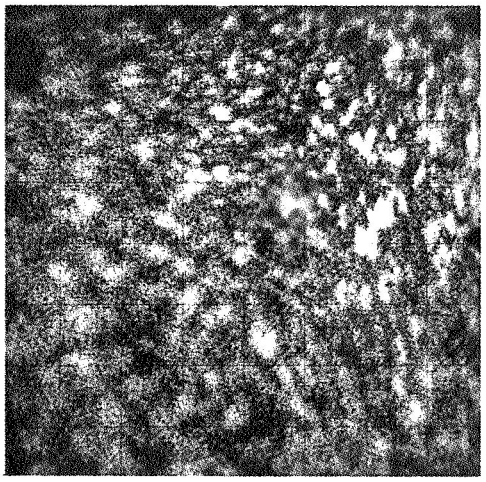


Grain refinement-Murakami etch

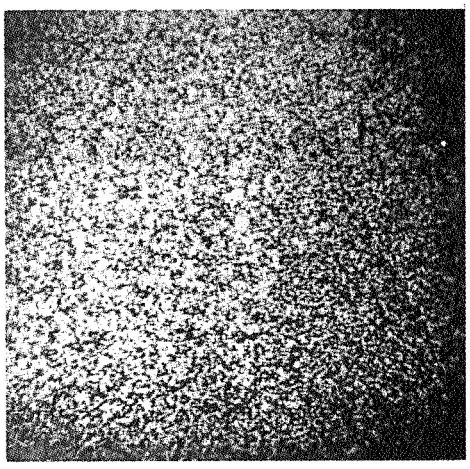
FIGURE IC1 - Typical microstructures which may occur in W - Re - ThO_2 alloy (WRT-1). 400X



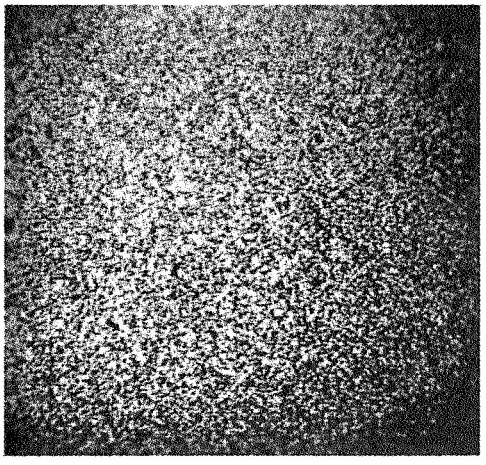
A
WRT-1 Poor Blend



B
WRT-3 Good Blend



C
Sintered - Good Ingot



D
Sintered - Good Ingot

FIGURE IC2 - W + 5 Re + 2.75 ThO_2 Alloys. Electron microprobe examination. White areas show presence of rhenium. 222X

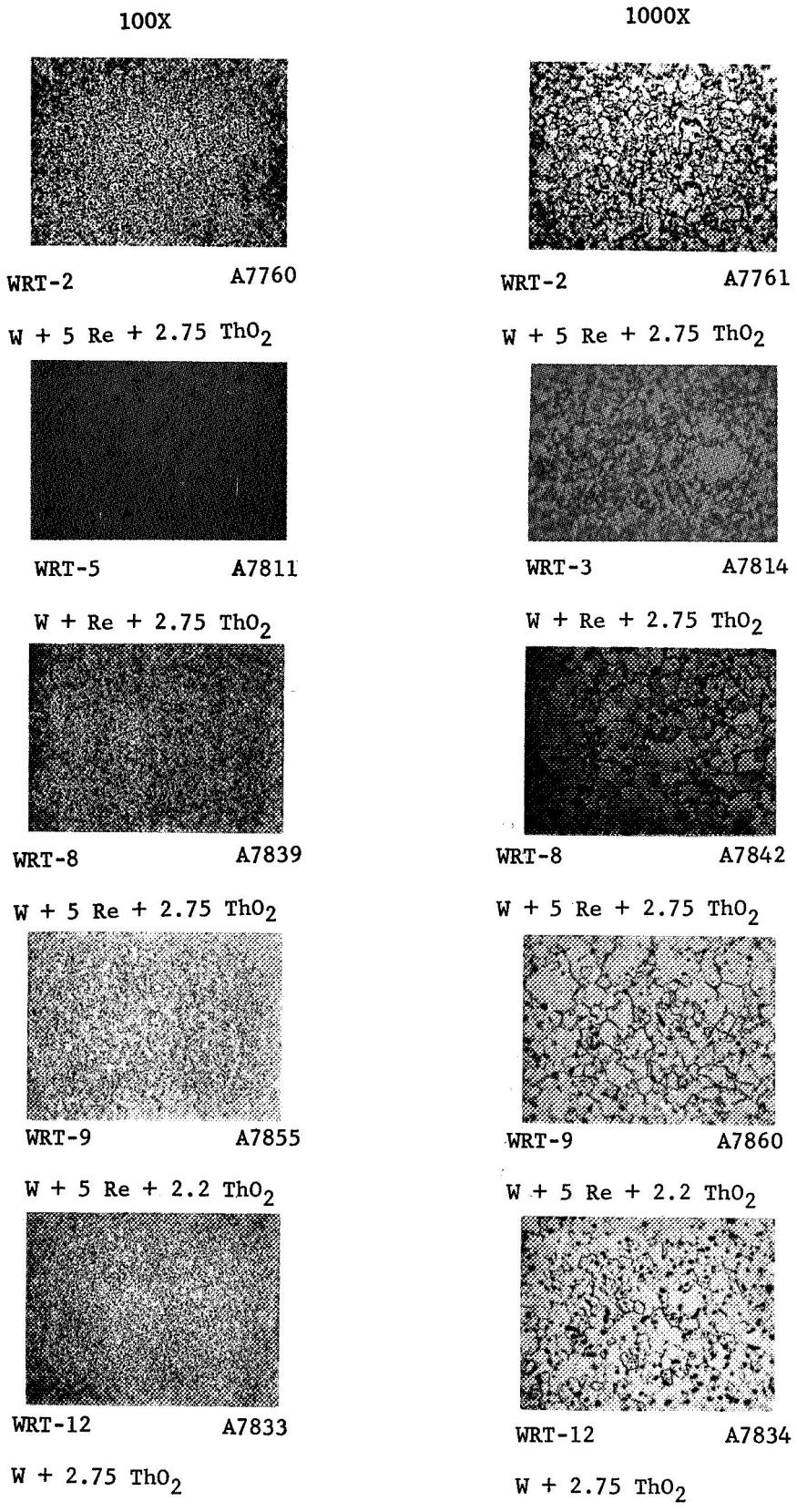


FIGURE IC3 - Typical microstructure of as-sintered W-Re-ThO_2 ingots - all etched in Murakami except WRT-5 which was buff etched

D. Tungsten-Hafnium-Carbon Alloy
W+(1-3)Hf+(0.005-0.02)C

Like the tungsten-rhenium-thoria system, the report shows problems had been encountered in the powder metallurgy approach, with oxide particle formation, blending, and in trying to mechanically work the sintered bars. Lower alloy levels were also formulated to relieve the problem. Dynapak extrusion was resorted to as a means of expanding the state of knowledge of refractory metal systems.

Powder and Ingot Preparation

The first selection of tungsten metal powder had a Fisher sub-sieve size of 4.0. This type of powder normally results in adequate as-pressed green strength to allow handling without breakage. When mixed with hafnium hydride and carbon, however, it was found that the green strength of the pressed ingot was very low. A 2.1 Fisher sub-sieve size powder was selected next and resulted in adequate green strength. The complete analysis of this powder is shown in Table IDI. Hafnium was added to the tungsten base metal as hafnium hydride obtained from Metals Hydrides Incorporated, Beverly, Massachusetts. Table IDII is the vendor's analysis of the hafnium hydride. Lamp black was used in all the experiments as the carbon additive.

Compacts of the blended powders were prepared by mechanical pressing into 5/8" x 13/16" x 16" long bars. A pressure of 44.6 tons/sq. in. was used to assure sufficient green strength for handling. The pressed ingots were then transferred to the vacuum furnace described in Section IB for sintering.

Upon initial heating, the hafnium hydride dissociated at approximately 5000°C. After the hydride dissociation, the pressure was maintained in the 10^{-5} Torr. range throughout the sintering cycle. Table IDIII shows a typical sintering schedule. The table indicates that during sintering there were five holding periods. The first hold at 1200°C was done primarily to strengthen the compact since little sintering was accomplished at the 500°C hydride dissociation temperature. The other holding periods were for the purpose of homogeneously alloying the material.

Ingots WHC-1, WHC-2 and WHC-3 were sintered at a final holding temperature in the range of 2800-2830°C $\pm$ 25°C. Metallographic examination indicated that possible localized melting was occurring during sintering. It should be noted that hafnium oxide has a melting point of about 2810°C. All subsequent final sintering temperatures were, therefore, limited to a maximum of 2800°C.

Efforts to develop a tungsten-hafnium-carbon system by the powder metallurgy method within the approved concepts encountered problems similar to those reported for the tungsten-rhenium-thoria system. These problems include oxide particle formation, blending, and mechanical working. Lower alloy levels were also formulated to relieve the problem. Dynapak extrusion was attempted as a means of expanding the state of knowledge of this refractory metal system. Eventually, in June 1966, work was terminated on the system.

Powder and Ingot Preparation (Cont'd.)

These first three ingots in the W-Hf-C system, prepared as described above, were not homogeneously alloyed and exhibited coarse second phase particles identified as hafnium oxide by microprobe examination. A different method of preparing the compacts was therefore developed for all subsequent ingots.

As in the case of the W-Re-ThO_2 alloys, improved blending techniques were employed. Small batches of a master blend were prepared from the tungsten powder, hafnium hydride and carbon. The master mix was then blended back to the desired composition with unalloyed tungsten. In order to minimize oxygen pickup during blending, re-reduced tungsten powder was obtained (from the original powder lot) and kept in argon-filled polyethylene bags and stored in metal cans. Weighing of the powders was done in an inert (argon) atmosphere by using a dry-box, shown in Figure IDl. Mixing was also done in an argon atmosphere using a twin-shell blender with an intensifier. Pressing and sintering conditions were not substantially changed from the original experiments. Table IDIV summarizes all the sintering experiments that have been performed.

Chemistry

Analysis of WCH-1 and WHC-2 for hafnium retention indicated that very small losses occurred. Hafnium was determined by both wet chemical and x-ray spectrographic methods. The wet chemical method indicated that hafnium was present as hafnium oxide to the extent of 0.36% and 0.24% in WHC-1 and WHC-2 respectively. The remaining hafnium (2.04% and 2.18%) should therefore be in solid solution in the tungsten matrix or present as hafnium carbide. All percentages are on a weight basis.

Interstitial content of the sintered ingots was not acceptable, especially with regard to oxygen content. Despite precautions that were taken in storing, weighing, blending, and sintering there appears to be no easy way, at the present, to reduce the oxygen content of the sintered ingot. Similar difficulties were encountered in other investigations related to this alloy system (1). Ingot WHC-4 was sintered in a dry hydrogen atmosphere to try and reduce the oxygen level; however, this was not successful. Ingot WHC-7 was sintered by radiation heating rather than self-resistance, but this also was unsuccessful. Table IDV shows the chemistry results of the various ingots prepared. A complete spectrographic analysis for metallic impurities in ingots WHC-1 and WHC-] is also given in Table IDV. Initially, it was difficult to adjust the carbon content to the level desired, 50 to 200 ppm. There were two reasons for this problem; carbon pickup was occurring due to back diffusion of oil and secondly, carbon that was added in hopes of "deoxidizing" the ingot was not effective, probably because of the affinity of the hafnium for both oxygen and carbon. In order to obtain the desired carbon level, additions in the range of 100 to 125 ppm were added as in Ingots WHC-11, 12 and 13.

Metallographic Examination

In order to evaluate the structure and characterize the sintered ingots, sections from each sintered ingot were conventionally polished

Metallographic Examination (Cont'd.)

and then etched in:

50 parts 1N NaOH
50 parts 30% $K_3Fe(CN_6)$

All the W-Hf-C alloy ingots prepared have exhibited large second phase particles within the matrix. Figure ID2 shows the structure of representative ingots at 100 and 1000 magnifications. Microprobe examination to date has revealed only hafnium oxide. There is a good possibility that the smaller particles may be hafnium carbide; however, this has not been determined analytically. Microstructure examination has also shown that a modest degree of grain refinement occurs in the W-Hf-C system; however, it is not as pronounced as is the W-Re-ThO_2 alloys. A variation of grain size from edge to center was also observed in many of the sections examined, the edge grains being about twice as large as grains near the center of the ingot. This grain size variation may indicate a chemistry variation from edge to center.

The average hardness of the various ingots was about R_C 33 regardless of composition or sintering schedule.

Microprobe Examination

Microprobe techniques were employed to study the extent of uniformity in blending the powders and also to check the degree of alloying after sintering. Figure ID3-c indicates that a relatively uniform mixture of hafnium hydride in tungsten was obtained using the improved blending technique. After sintering microprobe examination indicated that agglomeration occurred. Figure ID3-a and b show local concentration of hafnium (as hafnium oxide). The intense white area in Figure ID3-b is suspected to be an area where localized melting occurred during sintering.

Summary of Sintering W-Hf-C Alloys

Most of the ingots have been unsatisfactory for one or more of the following reasons:

1. Dispersion of a large second phase.
2. Localized melting due to too high of a sintering temperature.
3. Agglomeration of the second phase, hafnium oxide.

Nevertheless, the ingots listed in Table IDIV have all been worked, except the first three.

Fabrication, Ingot Breakdown

Rolling Trials

Ingot WHC-1 (2.4Hf+0.02C)

Pass	Preheat Time, Minutes	Preheat Temp.°C	Remarks
1	20	1705	Good appearance, but cracks at both ends. Split full length while cropping ends.

Discontinued

WHC-4 (2.4Hf+.045C)

1	20	1705	Good appearance, small nose crack.
2	20	1700	Good appearance.

Recrystallized by resistance heating at 3575 amps in H_2.
Grain count: 4160/mm^2 edge, 8980/mm^2 center - R_c 40-41.

3	10	1650	Good appearance.
4	10	1655	Good appearance.
5	10	1600	Good appearance.

Recrystallized at 1730 amps (at 2200°C), 10 minutes, H_2.

.352" dia. swage	10	1600	Cracked and slivered

Discontinued

WHC-5 (2.4Hf+.045C)

1	20	1705	Transverse cracks, broke in two.

WHC-6 (2.4Hf+.045C)

1	20	1705	Splits both ends, split full length while cropping.

Discontinued

WHC-8 (1.5Hf+.045C)

1	20	1700	Good appearance.
2	20	1640	Badly cracked and broken.

Discontinued

Fabrication, Ingot Breakdown (Cont'd.)

WHC-10 (1.5Hf+.03C)

Pass	Preheat Time, Minutes	Preheat Temp. °C	Remarks
1	20	1705	2-3/4" crack at nose.
2	20	1640	1-1/2" crack at nose.
	Recrystallized by resistance heating, 3730 amps, H_2.		
3	10	1650	Jammed in guide, broke.
		Discontinued	

WHC-13 (1.5Hf+.012C)

1	20	1700	Split at tail, transverse cracks along length.
		Discontinued	

WHC-9 (1.5Hf+.03C) Clad with Molybdenum

1	20	1700	Good appearance.
2	20	1640	Broke in guide, half rolled, cracked when etched to remove cladding.
		Discontinued	

WHC-14 (1.5Hf+.012C)

1	20	1705	Broke into three pieces.
		Discontinued	

Dynapak Extrusion Trial

Interspersed with the rolling trials, a number of attempts were made to extrude sections of the sintered ingots as a way to assess fabricability of these compositions, and later as means of producing rods nearer to the drawing input size. Sections of 5/8" square ingots were cut about three inches long and the corners were rounded to gain a tight fit into one inch diameter cans as shown in Figure ID1. The cans were TIG welded with a 1/8 inch thick disc closing the open end. Extrusion trials on the Model 620-C Dynapak were run as shown:

	Extrusion Condition				
	Ratio	Temp.°C	Pressure	Lengths,In.	Dia.,In.
WHC-7 (1.5Hf+.045C)	10.5:1	1800	1500	16	.338
WHC-12 (1.5Hf+.012C)	10.5:1	1800	1400	14	.338
WHC-15 (1.5Hf+.012C)*	10.5:1	1800	1500	15-1/2	.340
WHC-16 (1.5Hf+.012C)*	10.5:1	1800	1500	15-1/2	.340

*A total of 8 sections from two ingots, canned and extruded, four months after WHC-12.

After the poor results encountered in rod rolling, it was thought that though impractical for commercial production, the Dynapak extrusions might yet yield wire for evaluation purposes. The WHC-12 extrusion was selected and the molybdenum jacket was removed by pickling. Zyglo inspection of the surface revealed small transverse cracks or checks along each original corner, with a few small transverse cracks on the flats near the tail. The WHC-12 core measured about .200 inches square by almost 14 inches long.

A swaging preheat of 1750°C for 15 minutes was used along with preheated swaging dies in the 600 to 800°C range. A .200" diameter swaging pass served to straighten the rod as well as round it. A second preheat (1750°C) and swage sequence to .183" diameter followed. Only 3" was swaged, slow cooled, and then was inspected. Almost the entire length was found to be cracked and split longitudinally.

One extrusion each from WHC-15 and WHC-16 was resistance annealed, in vacuum, for three to five minutes at 2000°C. Swaging at 20 percent reduction in area was attempted but discontinued after the first pass when extensive cracking and rupture of the molybdenum jacket was noted. This was the last effort to process any tungsten-hafnium-carbon material during the course of this contract, by mutual agreement between NASA and the contractor.

References

1. Stephen Foldes, "The Effect of Alloying Additions on the Low and High Temperature Properties of a Tungsten-Molybdenum Base Alloy", Presented at the Fourth Symposium on Refractory Metals, Sponsored by the AIME, October, 1965.

2. Peter L. Raffo and William D. Klopp, "Solid Solution and Carbide Strengthened Arc Melted Tungsten Alloys", Presented at the Fourth Symposium on Refractory Metals, Sponsored by the AIME, October, 1965.

TABLE IDI

Tungsten Powder
U2.1-5071

Spectro Analysis of Powder (ppm)

Al	-6	Mg	5
Ca	14	Sn	8
Si	-7	Co	-3
Fe	6	Ti	-6
Cr	-3	Ag	-3
Ni	10	Pb	-6
Cu	-3	Nb	-20
W/Mo	68	Zr	-3
Mn	-6		

Chemical Analysis of Powder (ppm)

O_2	380
C	18

Physical Measurements

Cubic Inch	38.7
Tap Test	
Fisher No.	2.10
Porosity	.717
Milled Fisher	1.98
Milled Porosity	.540

Photelometer Distribution

Micron Size	% by Weight (as is)	(milled)
1.		8.7
2.		42.4
3.		29.0
4.		12.6
5.		6.2
6.		.9
7.		
8.		
9.		
10.		

TABLE IDII

Hafnium Hydride
(Prepared from Hafnium Crystal Bar)
Metal Hydrides Inc.

Chemical Analyses (Wt. %)

H	-	1.06
N	-	<.0010
O	-	<.0500
Ca	-	<.0050
Cu	-	<.0050
Al	-	<.0050
Ni	-	<.0050
Fe	-	.0012
Mg	-	.0016
Si	-	.0012
Mn	-	.0001
Ti	-	.0125
Zr	-	1.86

TABLE IDIII

A Summary of a Typical Sintering Schedule of W-Hf-C Alloys

Ingot WHC12

Time Min.	Pressure Torr.	Volts	Amp	Temp. °C ±25	
0	Leak rate 1 micron/hour				
25	1×10^{-4}	2-1/2			H_2 evolution
45	1×10^{-4}	2-1/2		(Red Color)	H_2 evolution
52	8×10^{-5}	4	800	935	
60	5×10^{-5}	4	1200	1200	
Hold for one hour					(1)
130	2×10^{-5}	4-1/2	1680	1450	
135	1.5×10^{-5}	5	1840	1555	
140	3×10^{-5}	6-1/2	2200	1800	
Hold for one hour					(2)
205	7×10^{-5}	7	3040	2150	
215	9×10^{-5}	7-1/2	3200	2250	
Hold for 1/3 hour					(3)
240	9×10^{-5}	8-1/4	3600	2450	
Hold for 1/4 hour					(4)
249	8×10^{-5}	8-1/2	3840	2600	
252	4×10^{-5}	10-1/2	4240	2780	
Hold for one hour					(5)
212	3×10^{-5}	10-1/2	4240	2780	Power off

TABLE IDIV

Summary of Sintering Conditions and Properties of Tungsten + Hafnium + Carbon Alloys

Ingot No.	Additions Wt. %	Sintering Atm.	Sintering Temp. °C ±25	Sintering Time, Hr.	Grain Size Grains/mm^2 1000X	Density grm/cm^3
WHC-1	2.44 HfH_2 + .02C	Vac.	2830	1/6		18.16
WHC-2	2.44 HfH_2 + .02C	Vac.	2830	3		18.50
WHC-3	2.44 HfH_2 + .05C	Vac.	2800	1/2		18.10
WHC-4	2.44 HfH_2 + .045C	H_2	2750	1/2		18.41
WHC-5	2.44 HfH_2 + .045C	Vac.	2750	1.0		18.31
WHC-6	2.44 HfH_2 + .045C	Vac.	2750	1.0		18.07
*WHC-7	1.5 HfH_2 + .045C	Vac.	2750	1.0		18.30
WHC-8	1.5 HfH_2 + .045C	Vac.	2750	1.0		18.31
WHC-9	1.5 HfH_2 + .03C	Vac.	2460	1/3		17.47
WHC-10	1.5 HfH_2 + .03C	Vac.	2780	1/2		18.50
WHC-11	1.5 HfH_2 + .03C	Vac.	2000	1/3	Discontinued sintering because of high carbon in WHC-10	
WHC-12	1.5 HfH_2 + .012C	Vac.	2780	1/2	8600	18.40
WHC-13	1.5 HfH_2 + .012C	Vac.	2760	1/2	3700	18.29
WHC-14	1.5 HfH_2 + .012C	Vac.	2760	1/2		
WHC-15	1.5 HfH_2 + .012C	Vac.	2750	1.0	3050	18.35
WHC-16	1.5 HfH_2 + .012C	Vac.	2750	1.0	4650	18.35

*Sintered in a Brew type furnace by radiation heating.

Nominal cross-section .625 x .520 inches, (higher density than for WRT system).

TABLE IDV

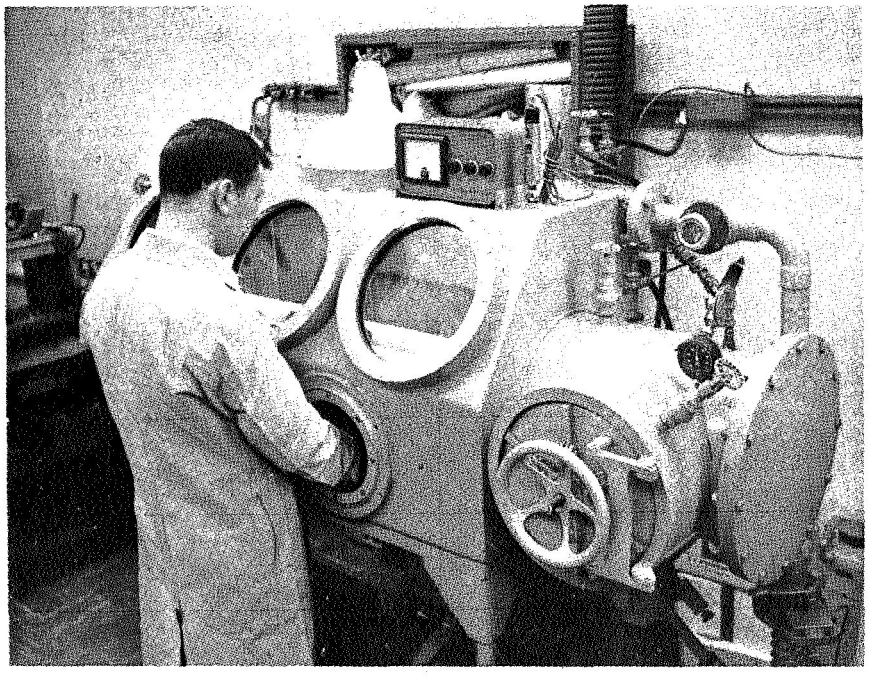
Chemical Analysis of Tungsten + Hafnium + Carbon Alloys

A. Chemical and Gas Analysis

Ingot No.	Nominal Additions Wt. %	Analysis Wt. % Hf	ppm C	O	H	N
WHC1	2.44 HfH_2 + .02C	2.40	260	518	3	5
WHC2	2.44 HfH_2 + .02C	2.42	300	791	1	38
				806	1	30
WHC3	2.44 HfH_2 + .05C		174			
WHC4	2.44 HfH_2 + .045C					
WHC5	2.44 HfH_2 + .045C		700	774	1	34
WHC6	2.44 HfH_2 + .045C		439	663	1	20
WHC7	1.5 HfH_2 + .045C		291			
WHC8	1.5 HfH_2 + .045C		378			
WHC9	1.5 HfH_2 + .03C	1.5				
WHC10	1.5 HfH_2 + .03C		311			
WHC11	1.5 HfH_2 + .03C					
WHC12	1.5 HfH_2 + .012C		160			
WHC13	1.5 HfH_2 + .012C		161			
WHC14	1.5 HfH_2 + .012C					

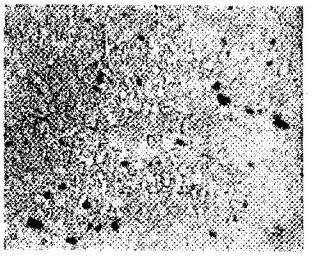
B. Spectro Analysis of Ingots WHC1 and WHC2

Element	W-1 (ppm)	W-2 (ppm)	Element	W-1 (ppm)	W-2 (ppm)
Al	15	14	Mn	6	<6
Ca	19	19	Mg	7	13
Si	36	46	Sn	<6	<6
Mo	125	230	Co	8	9
Fe	17	17	Ti	<6	<6
Cr	6	5	Ag	<3	<3
Ni	6	8	Pb	<6	<6
Cu	<3	<3	Zr	64	63
Cb	<20	<20			

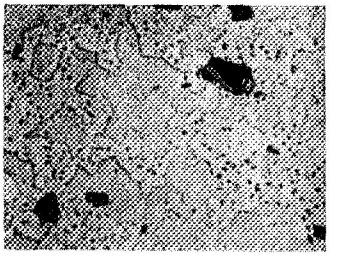


No. 115P

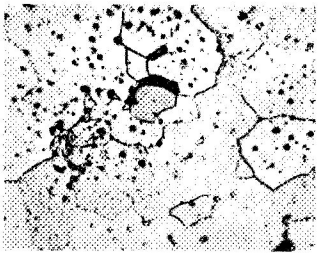
FIGURE ID1 - Dry box used to screen, blend, and prepare W-Hf-C powders prior to pressing



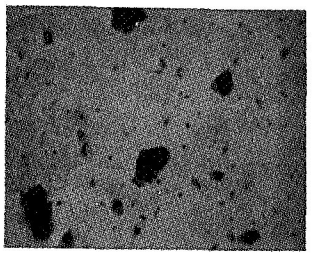
WHC-1 A7735
100X
W + 2.44 Hf + .02 C



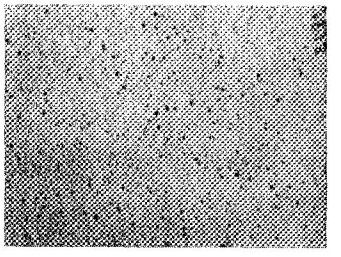
WHC-1 A7736
1000X



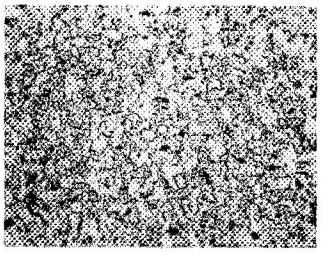
WHC-3 A7762
1000X
W + 2.44 HfH_2 + .05C



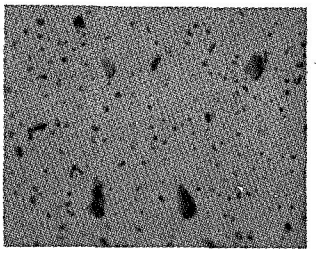
WHC-5 A7812
1000X
W + 2.44 HfH_2 + .045 C



WHC-5 A7813
100X

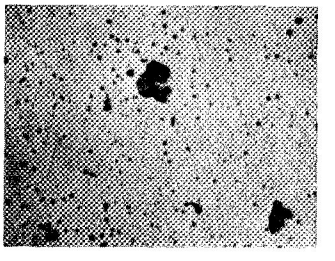


WHC-3 A7759
100X

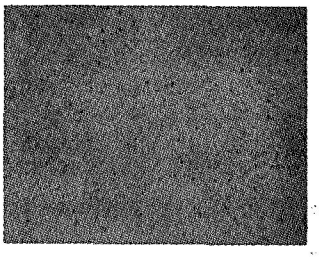


WHC-10 A7843
1000X
W + 1.5 HfH_2 + .03 C

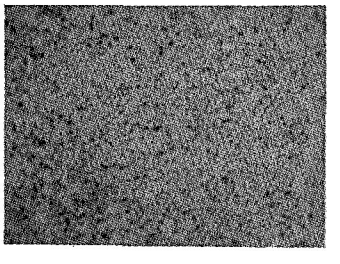
WHC-12 A7849
100X
W + 1.5 HfH_2 + .012 C



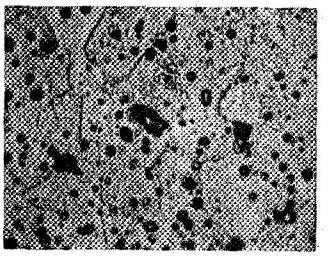
WHC-12 A7850
1000X



WHC-10 A7844
100X



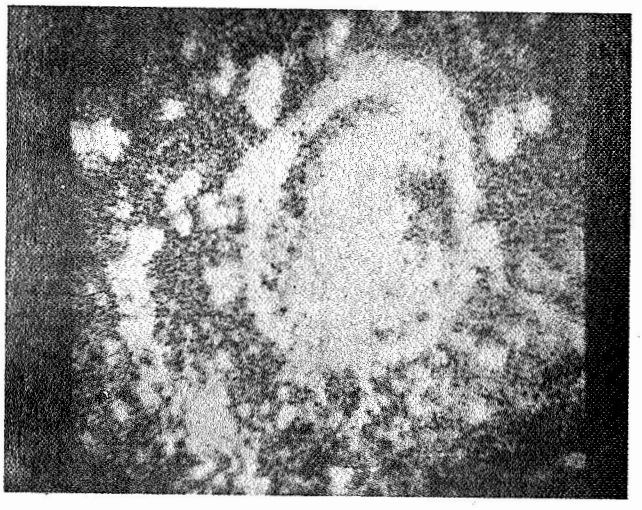
WHC-13 A7852
100X
W + 1.5 HfH_2 + .012 C



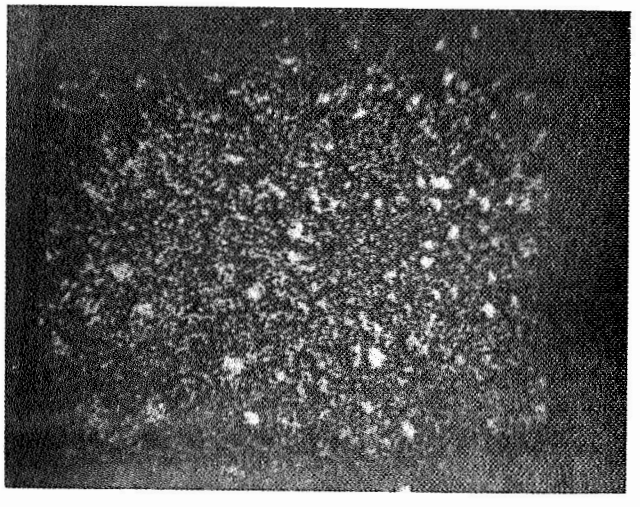
WHC-13 A7853
1000X

FIGURE ID2 - Microstructure of Sintered W-Hf-C Alloys
Illustrating Second Phase

A. WHC-1, As-Sintered
W + 2.44 HfH_2 + .02C



B. WHC-3, As-Sintered
W + 2.44 HfH_2 + .05C



C. WHC-5, Powder Compact
W + 2.44 HfH_2 + .05C

FIGURE ID3 - Microprobe Examination of W-Hf-C. White Areas Indicate Hafnium 222X

E. Columbium Alloy FS-85
Cb+(27-29)Ta+(9.5-10.5)W+(0.7-1.1)Zr

The columbium base, FS-85 alloy was one of the few materials in the contract that had been truly commercially available for some time. Therefore, to expedite production of wire, it was suggested to, and approved by, the NASA Program Manager that FS-85 be purchased at one quarter inch diameter in the stress-relieved condition which was suitable for wire drawing. The negotiated ordering specification, the vendor analysis, and the check analyses are shown in Table IEI.

In Table IEII will be found the vendor processing history for the one quarter inch FS-85 rod, along with room temperature and elevated temperature tensile tests performed at LMCD. The chemistry, processing details and physical properties should completely describe this input material.

Rod and Wire Drawing

Since FS-85 is a solid-solution-strengthened alloy, finished wire creep properties can only surpass the bulk properties above the recrystallization temperature via grain size differences. Therefore, our approach was to process the rod via two different schedules to present a finished wire with two different degrees of strain: (cold work) high and intermediate.

FS-85, No. 1A and 1B were processed according to the routines shown in Table IEIII. Processing to the first anneal point (.090" diameter) proceeded without incident using 10% R.A. per pass. Prior to vacuum annealing, the wire was chemically cleaned in $HF/HNO_3/H_2SO_4$ solution and wrapped in tantalum foil. FS-85, No. 1A was processed from .090" diameter at room temperature with no intermediate anneals. Drafts were held constant at 10% to 8% R.A. per pass. Drawing became increasingly difficult below .008" diameter and many short breaks were experienced before the target size of .005" diameter was reached. The NASA preferred process wire (based on initial testing) also followed this routine.

FS-85, No. 1B had an intermediate anneal at .013" diameter which produced a finished .005" diameter wire representing 85% R.A. total cold work. The anneal at .013" diameter enabled the processing to continue trouble-free to finish size.

In an effort to add to the data base on FS-85, one piece of annealed .090" diameter wire was drawn to produce samples representing increasing degree of strain. The samples were examined after recrystallization (2400°F - 30 minutes) for room temperature tensile properties as well as grain size. The results are presented in Table IEIV.

Discussion of Results

Processing .250" diameter FS-85 to .005" diameter presented relatively few problems. All wire drawing operations were carried out at room temperature. In-process anneals at approxiamtely 85% to 95% reduction in area should be incorporated if long lengths are to be obtained.

The recommended process for producing .005" diameter wire from .250" diameter rod is presented in Table IEV. Diameter measurements of the finished wire are tabulated in Table IEVI. All finished wire was cleaned of residual lubricants and possible processing contamination via degreasing followed by an $HF/HNO_3/H_2SO_4$ acid etch to remove approximately .0005" from the diameter.

TABLE IEI

FS-85 Alloy Analyses

Element	Ordering Spec.	Lot 85D1525 Vendor Analysis	Check Analysis - Check Source	
C	50 ppm max.	10 ppm		
O	150 ppm max.	28 ppm	99 ppm	GE - LMCD
N	150 ppm max.	47 ppm	32 ppm	GE - LMCD
H	10 ppm max.	6 ppm	3 ppm	GE - LMCD
Zr	0.9 ± 0.2%	0.94%	0.93%	GE - LMCD
Ta	28.0 ±1%	27.80%	27.4%	GE - LMCD
Ti		50 ppm		
Fe	100 ppm max.	70 ppm	26 ppm	Ledoux
Ni		10 ppm		
W	10 ± 0.5%	10.5%	10.4%	GE - LMCD
Si	100 ppm max.	100 ppm	10 ppm	Ledoux
Mo		200 ppm	30 ppm	Ledoux
Mn		10 ppm		
Al		10 ppm		
Cu		10 ppm		
Cr		10 ppm		
Co		10 ppm		
Cb	Balance	Balance		

TABLE IEII

FS-85 Alloy Rod

Processing Details and Physical Properties of 1/4" Diameter Rod

Vendor Processing - Lot 85D1525

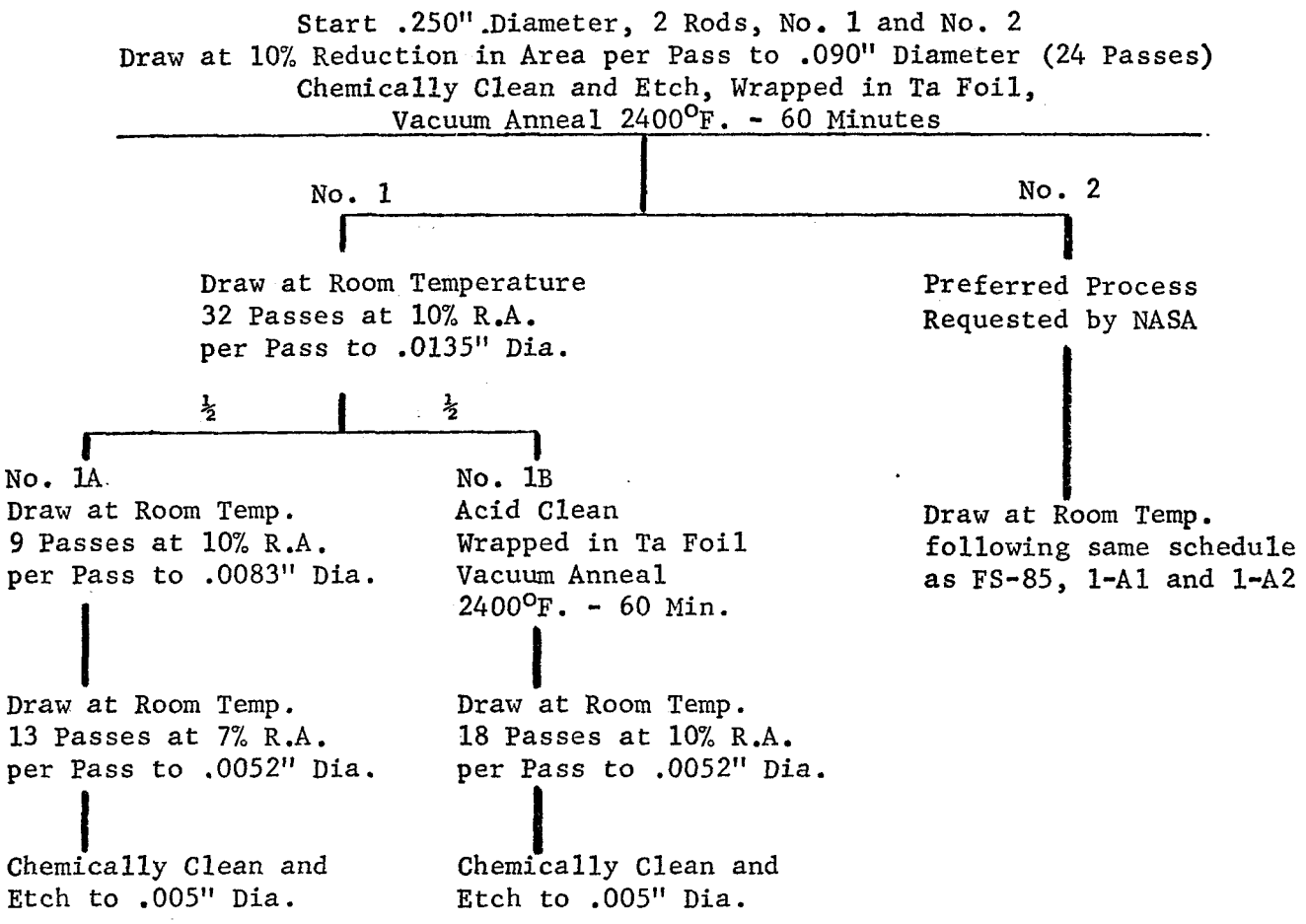
1. Billet hot forged at 2300°F to 1" square bar.
2. One inch square bar rolled to .440" square bar at 800°F.
3. Acid cleaned.
4. Annealed 1250°C one hour.
5. Swaged to 0.258" diameter at room temperature.
6. Acid cleaned and inspected.
7. Annealed at 1250°C, 1 hour.

Short Time Tensile Tests - specimens from as-received rod.

Test	Temp. °F	Ultimate Strength, KSI	.2% Yield Strength, KSI	% Elongation
1	Room	105	91	23
2	Room	107	92	24
3	Room	106	82	25
	Average	106	88	24
4	2200	34.8	25.0	39
5	2200	28.8	20.3	45
	Average	31.8	22.7	42

Strain rate .005 in./in./min. to yielding, .05 in./in./min. to fracture.

TABLE IEIII



YIELD: 2,171 Feet	YIELD: 4,790 Feet	YIELD: 6,570 Feet
2 Pieces	2 Pieces	1 Spool
FS-85, 1-A1 FS-85, 1-A2	FS-85, 1-B1 FS-85, 1-B2	FS-85, No. 2

TABLE IEIV

Properties of FS-85 Wire After Various Amounts of Work Followed by a Recrystallization Anneal

Size	% R.A.	Ultimate	Yield at .1%	% Elong. in 10"	DPH	Grain Count
88.5	3	84,000	61,000	23.2	195	15,100
85.0	10	85,200	64,500	25.0	195	2,400
79.0	23	81,500	59,000	23.0	180	1,330
75.0	30	80,300	57,700	25.0	173	2,240
71.0	38	81,600	58,600	23.0	187	3,200
63.0	51	83,600	57,600	23.0	178	4,700
56.5	60	83,600	58,400	25.0	183	8,000
51.0	68	83,700	59,800	21.6	187	8,950
41.0	79	81,700	57,500	29.7	183	8,950
34.0	86	85,000	64,400	23.0	176	11,100
28.7	90	86,500	63,200	28.0	183	13,450

Notes

1. Original wire sample at .090" diameter annealed at 2400°F. one hour.
2. All reported samples were from original piece to minimize effect of possible differences in chemistry.
3. All samples were recrystallized in the same furnace charge at 2400°F., 30 minutes in vacuum.
4. % R.A. calculated from starting diameter of .090".
5. Strain rate 0.2"/in./min. through fracture. Yield strength determined at .1% offset.
6. DPH using 100 gr. load.
7. Grain count in grains per square mm at 200X.

TABLE IEV

Recommended Process for
the Fabrication of .005" Diameter
FS-85 from .250" Diameter Rod

Conventional wire drawn at room temperature.

10% R.A. per pass from .250" diameter to .090" diameter (approximately 24 passes).

Degrease and acid clean to remove all traces of lubricant.

Vacuum anneal 2400°F. - 60 minutes.

Conventional wire drawn at room temperature 10% R.A. per pass from .090" diameter to .013" diameter (approximately 35 passes).

Degrease and acid clean to remove all traces of lubricant.

Vacuum anneal 2400°F. - 60 minutes.

Conventional wire drawn at room temperature 7% R.A. per pass from .013" diameter to .005" diameter (approximately 24 passes).

Degrease and acid clean.

TABLE IEVI

Diameter Measurements FS-85

Spool Number	Code	Lead End	Tail End
1	1-A1	.00495", .00495" .00497", .00498"	.00502", .00500" .00500", .00499"
2	1-A2	.00500", .00500" .00501", .00500"	.00499", .00496" .00500", .00500"
3	1-B1	.00498", .00496" .00496", .00497"	.00500", .00496" .00497", .00497"
4	1-B2	.00496", .00495" .00497", .00496"	.00493", .00492" .00488", .00497"
5	Preferred Process No. 2	.00499", .00496" .00496", .00495"	.00492", .00491" .00486", .00490"

F. Tantalum Alloy, T-222
Ta+9.6±0.5%W+2.4±0.3%Hf

Like the FS-85 material, the T-222 alloy was approved to be purchased as stress-relieved, 1/4 inch diameter rod suitable for development of the drawing process to the take-out size of 5 mils. Like FS-85, T-222 was considered to be a commercially available material, although it may not have been in commercial production as long as FS-85 had been.

Apparently not all the process details for making T-222 material are known or recognized by people in the refractory metal business. Just as we had trouble in making the high additive tungsten alloys with rhenium and thoria or with hafnium and carbon, the vendor also had troubles in making 1/4" diameter T-222 rod. Finally, after several unsuccessful efforts including casting, extruding, and rolling sequences over an 18 month span, the original order was cancelled and a smaller quantity was ordered from another qualified vendor.

A total of 17 pounds of rod was received in August 1967. Details of the vendor analysis and a selective check analysis are shown in Table IFI. Table IFII shows the mechanical property tests on specimens machined from these rods and tested at room temperature and elevated temperature.

After sample inspection, the rods were sent to the Dover Wire Plant for processing to 5 mil wire,

Rod Breakdown and Wire Drawing

The T-222 was processed at room temperature from the as received, stress relieved .250" diameter rod through finish .005" diameter wire. No heat treatments were incorporated to enhance finish wire properties because it was judged that the biggest gains would be through a high degree of strain hardening; however, stress relief anneals were used at points in the process when the wire became too high in tensile strength to enable further drawing without breakage and/or galling. Stress relief parameters of 2000°F for 30 minutes in vacuum were used as recommended by the vendor's published data sheet.

Table IFIII outlines the process followed to produce the total reuqirement of 6,000 fett of .005" diameter wire. Drafts of 15% reduction in area were used to .060" diameter, 10% R.A. per pass to .020", and 8% R.A. per pass to finish size for T-222B. The wire was chemically cleaned before each stress relief anneal as well as at finish size in $H_2SO_4/HNO_3/HF$ solution. T-222A was generated from a small break at .061" diameter. It was decided to attempt to draw T-222A from the .060" anneal point to finish size without an additional stress relief. The process followed for T-222A is shown in Table IFIII. Many difficulties were encountered in wire and die breakage so at .0137" diameter warm drawing was employed (200-300°C) along with reduced drafts. A small piece 200 feet long was eventually produced at

Rod Breakdown and Wire Drawing (Cont'd.)

.005" diameter with a room temperature tensile strength of approximately 350,000 psi.

Room temperature tensile test results are presented in Table IFIV for T-222B at representative process sizes as well as pre and post anneals.

Diameter measurements of the finished wire are presented in Table IFV.

Discussion of Results

Fabrication of .005" diameter T-222 wire from stress relieved .250" diameter rod was straightforward. Annealing points were dictated by work hardening and associated processing difficulties. The recommended process is the same as shown for T-222B in Table IFIII.

Table IFI

Analysis of T-222 Alloy

Heat No. 65006-T-222

	Vendor Check Analysis		General Electric Check Analysis - Rod 1/4" Dia.
	Top	Bottom	
Hf	2.60%	2.25%	2.7
W	9.30%	9.40%	9.3
Ta	Balance	Balance	
	Impurities, ppm		
C	90	100	
N	15	20	
O	40	30	
H	4.0	3.3	
Al	20	20	
Cb	420	350	
Cd	5	5	
Co	10	10	
Cr	20	20	
Cu	40	40	
Fe	50	50	
Mg	20	20	Vendor process details unavailable.
Mn	20	20	
Mo	35	35	
Ni	20	20	
Pb	20	20	
Si	40	40	
Sn	20	20	
Ti	50	50	
V	20	20	
BHN	248-285, 265 average		

Table IFII

Tensile Tests - T-222 Alloys

Specimens Machined from 1/4" Dia. Rod

Test Temp. °F	Ult. Str., Ksi	.2% Yield Str., Ksi	Elongation,%
75	169.1	155.2	17.4
75	169.8	160.4	20.0
2200	76.3	33.8	41.0
2200	79.5	39.4	37.8
2600	33.4	18.0	83.8
3500	14.8	8.1	118.3

Strain rate .005 ipm to yield, .05 ipm to fracture.

TABLE IFIII

Start .250" diameter, room temperature draw 17 passes at 15% R.A. to .061" diameter, one wire break, chemically clean and etch to .060" diameter

T-222-A (Small break)	T-222-B
Draw at room temperature 20 passes 10% R.A. to .0205" diameter.	Vacuum anneal 2000°F., 30 minutes.
11 passes 8% R.A. to .0137" diameter. (Broke 4 diamond dies)	Room temperature draw 20 passes 10% R.A. per pass to .0205" diameter.
Reduce draft to 5% R.A. per pass. Warm draw 200-300°C.	Chemically clean and etch to .0198" diameter.
.008" diameter.	Vacuum anneal 2000°F., 30 minutes.
Reduce drafts to 4% R.A. Reduce drawing speed to 75 feet/minute.	Room temperature draw 33 passes 8% R.A. per pass to .0051" mil.
To .0051" diameter.	Chemically clean and etch to .005" diameter.
Chemically clean and etch to .0050" diameter.	
YIELD: 200 Feet	YIELD: 3 Bands: 2,085 Feet
1 Band	2,020 Feet
T-222-A	1,970 Feet
	6,075 Feet
	T-222B

TABLE IFIV

Room Temperature Tensile Tests* - Process Wire T-222-B

Size	Condition	U.T.S.	% Elongation
.100"	As Drawn	240,000 psi	2% in 10"
.0775"	As Drawn	245,000 psi	2% in 10"
.0605"	As Drawn	265,000 psi	1% in 10"
.0605"	Annealed	168,000 psi	20% in 10"
.050"	As Drawn	193,000 psi	10% in 10"
.040"	As Drawn	204,000 psi	3% in 10"
.030"	As Drawn	225,000 psi	1.5% in 10"
.0205	As Drawn	250,000 psi	< 1% in 10"
.0198"	Annealed	156,000 psi	7% in 10"
.017"	As Drawn	190,000 psi	3% in 10"
.015"	As Drawn	192,000 psi	2% in 10"
.012"	As Drawn	195,000 psi	< 1% in 10"
.010"	As Drawn	208,000 psi	< 1% in 10"
.008"	As Drawn	226,000 psi	< 1% in 10"
.0051"	As Drawn	274,000 psi	< 1% in 10"

Room Temperature Tensile Tests* - Process Wire T-222-A

Size	Condition	U.T.S.	% Elongation
.017"	As Drawn	272,000 psi	< 1% in 10"
.012"	As Drawn	289,000 psi	< 1% in 10"
.010"	As Drawn	310,000 psi	< 1% in 10"
.008"	As Drawn	330,000 psi	< 1% in 10"
.006"	As Drawn	350,000 psi	< 1% in 10"
.005"	As Drawn	352,000 psi	< 1% in 10"

*Strain rate of 0.2 inches/inch/minute.

TABLE IFV

Diameter Measurements of Finished Wire

Process	Lead End	Tail End
T-222-B	.00487"/.00480"	.00498"/.00493"
T-222-B	.00500"/.00494"	.00495"/.00499"
T-222-B	.00495"/.00493"	.00500"/.00498"
T-222-A	.00493"/.00495"	.00493"/.00496"

Section II - The Evaluation of Materials

A. The Multi-Station Creep and Stress-Rupture Apparatus

Introduction

The design of a multi-station stress-rupture and creep apparatus for testing wire of less than 10 mils diameter was undertaken after receipt of the contract. Some of the basic features of this apparatus which needed to be incorporated in the design were:

1. Testing capability to a maximum temperature of 2800°F.
2. Vacuum capability to pressures of 10^{-9} to 5×10^{-11} Torr.
3. Continuous operation for periods up to 1500 hours.
4. Measurement of creep deformation to a precision of 50×10^{-6} inches.

The basic design was completed within the first 90 calendar days of the contract and was subsequently approved by NASA for fabrication. The tester was built, assembled and debugged during the next 15 month period (October 1965 to January 1967) and then calibrated prior to its use for creep-rupture testing. A photograph of the tester is shown in Figure IIA3.

Summary and Overall Description of the Equipment

The Basic Components of the Apparatus

1. An ultrahigh vacuum chamber for housing six individual creep-rupture test stations is shown in Figure IIA1. The chamber is constructed of stainless steel and uses metal gasket seals. Therefore, it is capable of being baked out to a temperature of at least 500°F. Strip-type heaters, attached to the outer chamber wall, are used to heat the chamber for the bakeout cycle, and the entire chamber is insulated with an aluminum outer shroud. Water cooling channels are welded to the chamber and are used together with a blower for cooling after the bakeout cycle.

   The stainless steel shell of the chamber is flanged so as to be bolted to the Main Base Plate and a large metal gasket serves as the vacuum seal (Figure IIA2). In addition, the Main Base Plate is so constructed that each of the six creep-rupture test stations are individually mounted on the Base Plate by means of flanges and metal gasket seals.

2. Individual Test Stations (See Figure IIA2)

   Each test station is fabricated, assembled and aligned onto a separate flange which in turn may be independently bolted to the Main Base Plate. Therefore, each one can be replaced by a blank flange. This has the advantage of allowing the replacement of one or more stations with a blank flange if there is a need for vacuum

2. Individual Test Stations (See Figure IIA2) (Cont'd.)

checkout of individual stations, or for the use of less than six test stations.

3. The Vacuum Pumping System (See Figure IIA1)

For rough pumping the test chamber to a pressure of approximately 10^{-2} Torr., the system is provided with three sorption-type pumps attached to a roughing manifold through three nonbakeable valves and one bakeable valve. To attain pressures of the order of 10^{-9} to 5×10^{-11} Torr., the system has a bakeable 500 liter/sec. triode ion pump and a titanium sublimation pump. The titanium sublimation pump has a removable liquid nitrogen or water-cooled cryopanel of 500 square inches surface area for receiving the titanium film from the pump.

4. Equipment Controls

Controls for the vacuum system are encased in one panel cabinet and include the following:

a. Ion pump control
b. Titanium sublimation pump control
c. Ionization gage control
d. Thermocouple gage control
e. Ion pump pressure relay
f. Automatic temperature controls and timer for the bakeout cycle.

The temperature recorders and controllers and the electrics for the six test stations are encased in three panel cabinets. The system for each test station is the same and was devised and supplied by the General Electric Instrument Department, Lynn, Massachusetts.

Each test station system consists of the following component parts: Step-down transformer; SCR Power Pack; Type 550 MV/I transmitter for use with two W-5% Re/W-26% Re thermocouples in a range of 1000 to 3000°F; a Type 521-1/2% - 2-pen recorder; and a Type 560 dual alarm for automatically switching the control system from the temperature controlling thermocouple to the temperature recording thermocouple. In addition, a Minneapolis-Honeywell manual switch allows the switching of thermocouples at the operator's discretion.

This extra emphasis on the thermocouple switching arrangement was deemed appropriate in order to decrease the risk of loss of data from thermocouple failure during a run.

5. The Hoist System (See Figure IIA1)

An electrically driven hydraulic hoist system, with safety latches and control valves, lifts the outer stainless steel shell exposing the individual test stations mounted on the Main Base Plate.

Elements of a Creep-Rupture Test Station

The basic elements of a creep-rupture test station are shown in Figure IIA2 and are as follows:

1. Invar Posts

These serve as the stationary support for the specimen train. They are constructed of low coefficient of thermal expansion material to minimize errors in the creep measurement.

A zero mark or fiducial reference point is positioned on the invar post. Provision for sighting on this reference point with a cathetometer through a window is made for alignment adjustments at set-up time and for measuring or corroborating measurements of extensions during test.

2. Water-Cooled Copper Jacket, Radiation Shields and Heating Elements

The central part of a test station is located between the two invar posts and is delineated by a water-cooled copper jacket. Within this jacket are positioned a series of radiation shields (five in number) and a heating element fabricated from .005 inches thick Ta-10% W sheet. Two tungsten-rhenium thermocouples (W-5% Re/W-26% Re) are positioned inside the heating element, in close proximity to the wire sample, for recording and controlling the test temperature.

3. Wire Samples

Wire of approximately five mils diameter is electrolytically etched to a slightly smaller diameter for a length of .75 inches in order to define the gage length. The sample is clamped to the upper invar post and then attached to the desired load (weights). It is positioned within the heating element so that the etched gage length is located at the uniform temperature zone of the furnace. The weights are suspended by a pivoted lever arrangement and are released only when the test conditions are achieved.

4. Creep Sensing System

For measuring the extension of the wire sample under stress an LVDT (Linear Variable Differential Transformer) method, designed and built by Schaevitz Engineering Corporation, is employed. This consists of a nickel-iron core rod attached to the weights and is a part of the total load on the specimen. This core rod hangs within a stainless steel tube welded to the test station flange. The LVDT coils are positioned externally around the tube and core. The movement of the core rod within the field produces an electrical signal which can, by calibration, be related to creep deformation (see below). In addition to the usual LVDT coils, an additional coil has been designed into the system for use as a "Specimen Break"

4. Creep Sensing System (Cont'd.)

detector signal. When the sample breaks, the "Specimen Break" detector signal is used to shut down that particular test station's temperature recorder and controller.

The Creep Measuring System

The extension of the loaded specimen with time requires the measurement of deformation perhaps as great as 1.0", to a precision as high as 50 microinches for expected testing times as long as 1500 hours.

The LVDT system of measurement (see above) is identical for each of the six test stations. Movement of the Ni-Fe core suspended from the specimen is monitored by the signal induced in the surrounding LVDT coils. Calibration of the LVDT system is effected by measuring the change in output of the LVDT when the inside cores are moved a known distance. A sensitive mechanical adjustment of the position of the external coils is also available for repositioning during a test.

The primaries of the LVDT's are excited by a 2.5 KC carrier at 3 VAC. The output voltage from each differential winding of the LVDT's leads into a separate carrier-amplifier-demodulator where it is converted to an analog DC voltage for presentation on a multi-channel recorder. In addition, this output may be switched to a single channel recorder for continuous recording of the rapid changes during the initial stages of creep.

A range selector switch at the output of the amplifier-demodulator will select full scale measurement ranges for this displacement equivalent to 0.005", 0.050", 0.100", 0.250", and 0.500".

Each LVDT output is fed into a six-channel Bristol recorder with color dot printout. The input to the recorder is 0 to 50 millivolts. Chart speed is one inch/hour and sampling rate is one print/minute for a total cycle time of six minutes.

The LVDT system contains an additional coil which will produce a signal when specimen rupture occurs. The signal through the control relays to the multi-channel recorder will give a record of rupture within six minutes of actual rupture time.

The second recorder, a single channel recorder, is used to accept any one of the six station outputs for recording continuously when desired. This recorder has a 0 to 50 millivolt range and chart speeds of one inch/hour and one inch/minute.

Temperature Profile Determinations

Experiments to determine the temperature profile of the furnaces in the multi-station tester were made so as to be able to estimate the temperature gradient seen in a test wire. Because of the complexity of the equipment and procedure, and the time involved for these experiments, only one test station's temperature profile was determined. Each test station is small in size and was constructed with very fine dimensional tolernaces. Therefore, it has been assumed that the results from the temperature profile of one test station could adequately describe the temperature profile of the others.

Experimental Procedure - Test Station 5

These experiments were performed using multiple tungsten-5% rhenium/tungsten-26% rhenium thermocouples (hereafter called the thermocouple package). The .015" diameter tungsten-5% rhenium and tungsten-26% rhenium wires were purchased from Hoskins Manufacturing Company and certified to have particular temperature/emf characteristics. The thermocouple package was made from three separate tungsten-26% rhenium wires and one common tungsten-5% rhenium wire so that the junctions were spaced at 1/2" intervals. Each junction was separated from the next one by a two-hole, high-purity alumina insulator, and the pairs of thermocouple wires exiting at the top and bottom of the test station were each contained within a single length of two-hole, high-purity alumina insulator.

There were two separate experimental runs made to determine the temperature profile of Test Station 5. In the first run, the center thermocouple, T_2 (see Figure II-A4) of the package was positioned axially at 5/16" above the center or midpoint of the furnace. In the second run, the center thermocouple, T_5, was positioned 7/16" below the midpoint of the furnace. In both experimental runs, two additional tungsten-5% rhenium/tungsten-26% rhenium thermocouples were used for controlling and measuring the test temperature. These were firmly positioned axially so that they could not move and were located at 5/16" above the midpoint of the furnace, but displaced to the side by .28". A diagram of the axial arrangement of the thermocouples for these runs is shown in Figure II-A4.

Prior to performing these measurements, the six GE Type 540-01 basic temperature controllers and six Type 521 two-pen temperature recorders were checked for calibration using the procedures as specified in the Instruction Manual from the Instrument Department of General Electric, Lynn, Massachusetts.

A typical profile test run procedure involved the positioning of the thermocouple package. The chamber was closed and evacuated to a pressure of 10^{-9} torr with a bakeout. Station Number 5 furnace was heated to a specific temperature in the range 1800°F to 2800°F and allowed to attain equilibrium conditions at the test

temperature (accomplished in 15 to 20 minutes, as determined by monitoring the emf of the measuring thermocouple on a precision potentiometer). The emf values, for each of the three thermocouples in the package, were measured by means of the precision potentiometer when no temperature drift was apparent. These emf readings were converted to temperatures using the temperature - emf characteristics as calibrated and supplied by Hoskins Manufacturing Company. After collecting data in one run, the tester was disassembled, the thermocouple package repositioned, and the test procedure, described above, was followed again.

The temperature data from the two test runs are graphically represented in Figure II-A5 as the temperature measured at the six axial positions versus the measured test temperature (dashed straight line). Each of the curves drawn in this figure represent, what is felt to be, the best possible curve for those six data points. Examination of the data allows for the following observations to be made:

1. If the data from both runs are examined collectively, and the best curve fit is made, the apparent temperature variation from the mean temperature in the designated gage length at 1/16" above to 11/16" below the physical center of the furnace is from ± 7 to ± 10 °F. This holds for test temperatures from 2000 to 2400 °F, the temperatures used in the wire testing program.

2. Each of the two runs shows the maximum temperature at each test temperature to occur at the same furnace position. This is located within the designated gage length at .250" below the physical center of the furnace.

The experimental technique used for these measurements involved some considerations necessary for interpretation of the data. First, the mass of the thermocouple wires and insulators was large compared to that of a wire sample. Probably, the "measured" temperatures were influenced by conduction losses. Second, the thermocouple package was moved from one run to the other and there would be different radiation effects on the thermocouples. Third, it must be assumed that the test temperatures of the second run exactly reproduced those of the first run.

Under actual test conditions, the wire sample is undisturbed and axially located in the center of the furnace with its reduced diameter length at 1/16" above to 11/16" below the center of the furnace. It is felt that the real temperature profile of a test wire sample is more uniform than that ± 7 to ± 10 °F determined experimentally.

Temperature Calibration Using Gold as the Standard

Absolute temperature calibrations for each of the six test stations were made using the melting point of pure gold as the standard. Two separate setups were required to complete the six calibrations. The first setup was used for calibration of Test Stations 1, 2, and 4 and the second for 3, 5, and 6.

The experimental setup and procedure followed for each test station's calibration was the same. A Pt/Pt - 10% rhodium thermocouple, fabricated from .015" diameter wire and insulated with a two-hole, high-purity alumina insulator, was suspended in the furnace simulating the test wire sample position. The junction bead was positioned axially at exactly 3/16" below the center of the furnace. A one-hole zirconia insulating tube (1/16" diameter x 3" long weighing approximately .5 grams) was suspended downward from the thermocouple junction by means of a small loop of .010" diameter gold wire. The gold wire was purchased from Sigmund Cohen Company and was certified to be 99.999% pure. Two tungsten-5% rhenium/tungsten-26% rhenium thermocouples in each test station (one for control and the other for temperature measurement) were positioned axially at 3/16" below the center of the furnace, but displaced to the side by .28". These thermocouples were fixed rigidly to their position by the top assembly of the furnace.

After the thermocouples were positioned, the test chamber was closed and evacuated to a pressure of 10^{-9} torr with a bakeout. Each furnace was slowly heated to within 50°F of the melting point of gold. When equilibrium conditions were obtained, as determined by monitoring the emf of the measuring thermocouple with a precision potentiometer, the emf of the Pt/Pt - 10% rhodium thermocouple was measured with another precision potentiometer. The temperature of the furnace was then increased slowly in increments of 10° until the measured temperature was 20° from the melting point of gold, and then was increased slowly in 5° increments until the gold melted, as indicated by the visual dropping of the ceramic tube. Before going from one temperature increment to the next, conditions of thermal equilibrium were always obtained and held at the lower temperature and the emf's of the tungsten-5% rhenium/tungsten-26% rhenium and Pt/Pt - 10% rhodium thermocouples were measured.

The results of these calibration runs are compiled and given in Table IIA1 (attached). The temperatures as measured with the Pt/Pt - 10% rhodium thermocouples are indicative of the accuracy with which the technique can be used to measure the melting point of gold and, therefore, the temperature at that position in the furnace. It can be seen that by this technique, it is possible to measure the absolute temperature to less than ± 8°F.

Each temperature, as shown in the column for the measured temperature (tungsten-5% rhenium/tungsten-26% rhenium recording thermocouple) is a value attained at a specific location of this thermocouple when a piece of gold at the position of a test wire melts. It can be seen that, in all cases, this temperature is greater than the temperature at which gold melts. In addition, the deviation from the melting point of gold varies from station to station, even through four of the six stations show a deviation within 5°F of each other (i.e., from 30°F to 35°F).

For the purpose of describing the actual creep and stress rupture test temperatures, the assumption is made that the test temperatures are near the melting point of gold, therefore, the temperature corrections from the calibration study still apply. It is also assumed that the tungsten-5% rhenium/tungsten-26% rhenium measuring (recording) thermocouples remained in fixed position and thermoelectrically homogeneous with time.

Table IIAI

Temperature Calibration Results Using Melting Point of Gold

Test Station Number	At the Melting Point of Gold - 1945.4 °F		Correction Recording Temp-M.P. Au(1945 °F)
	Measured Temperature W-5%Re/W-26%Re (Recording Thermocouple)	Measured Temperature Pt/Pt-10%Rh	
1	2005 °F	1948 °F	-60 °F
2	1979 °F	1937 °F	-34 °F
3	1980 °F	1942 °F	-35 °F
4	1962 °F	1948 °F	-17 °F
5	1980 °F	1950 °F	-35 °F
6	1975 °F	1950 °F	-30 °F

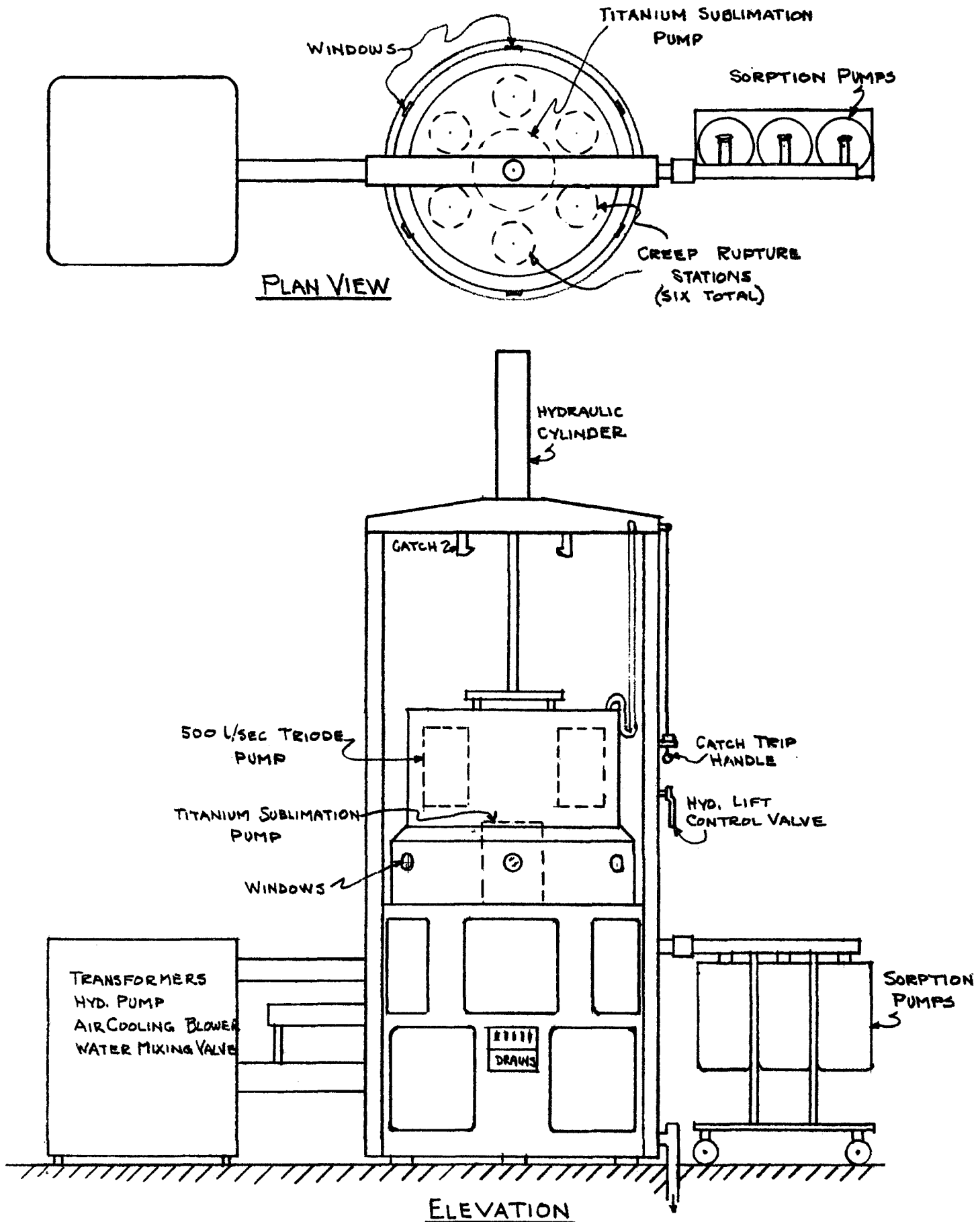


FIG. II A 1 - CREEP RUPTURE APPARATUS

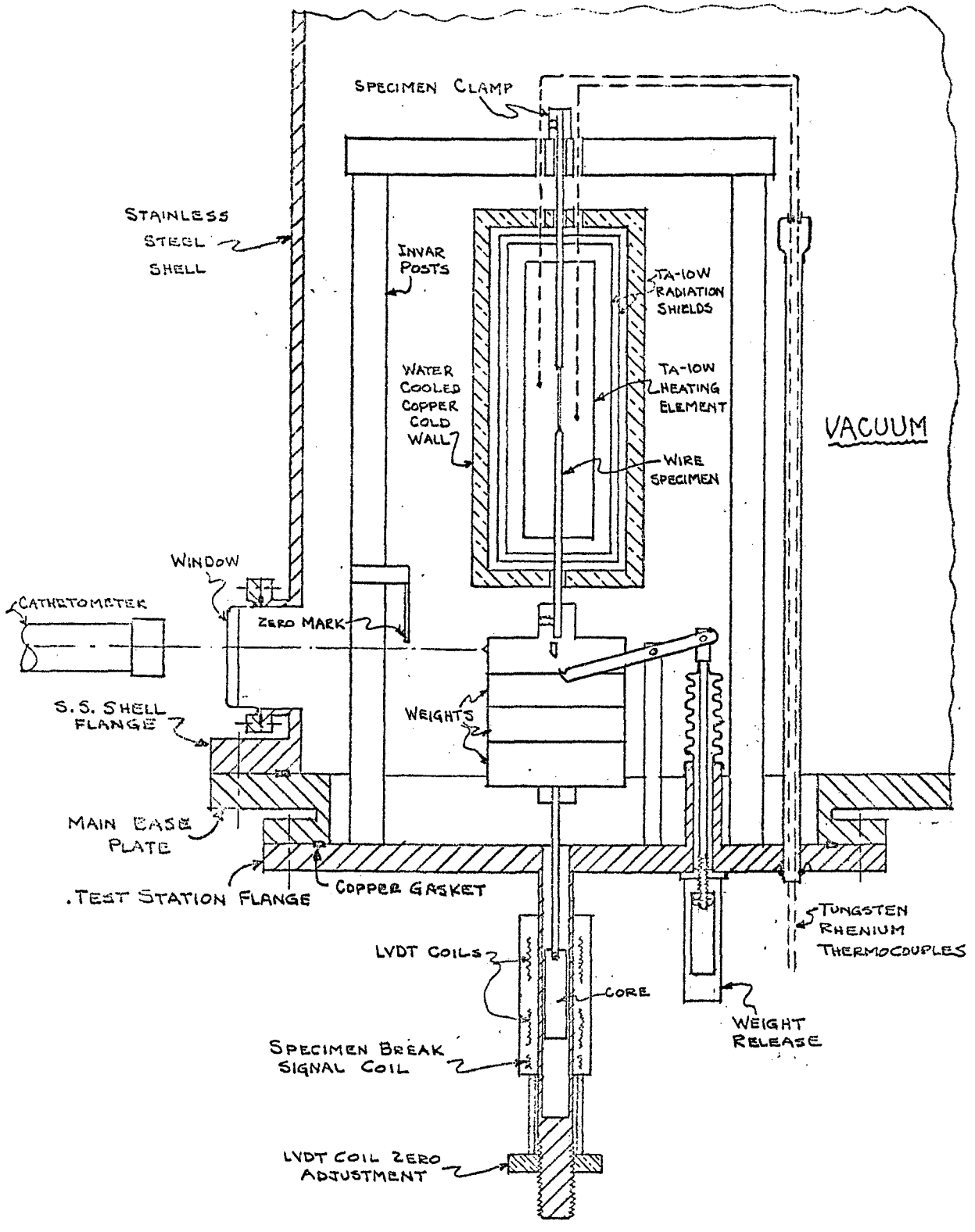


FIG. II A 2 - CREEP RUPTURE STATION

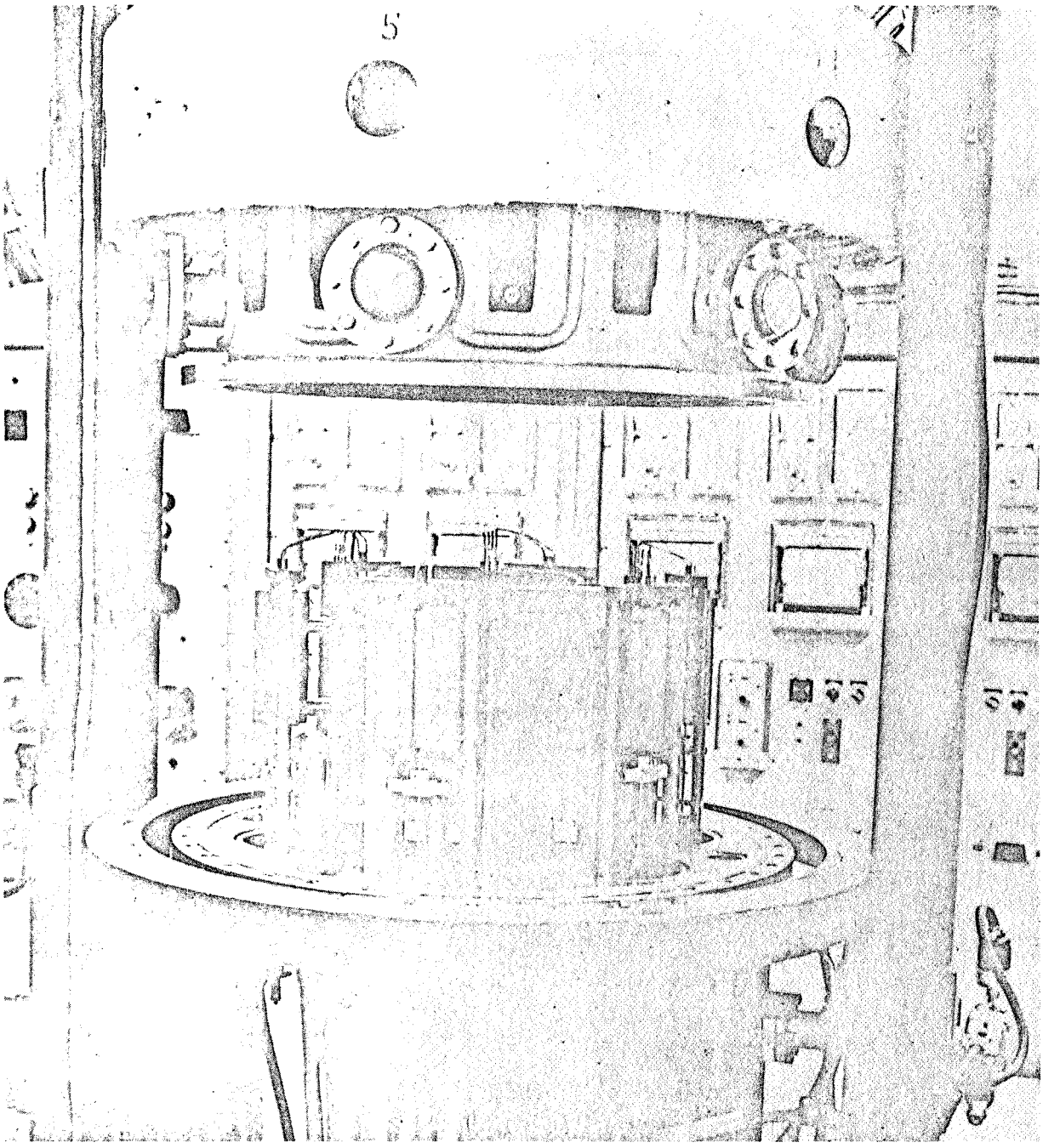


Figure IIA3

Photograph of Multi-Station Creep Rupture Tester

THERMOCOUPLE POSITIONS FOR TEMPERATURE CALIBRATION RUNS

MULTISTATION TESTER

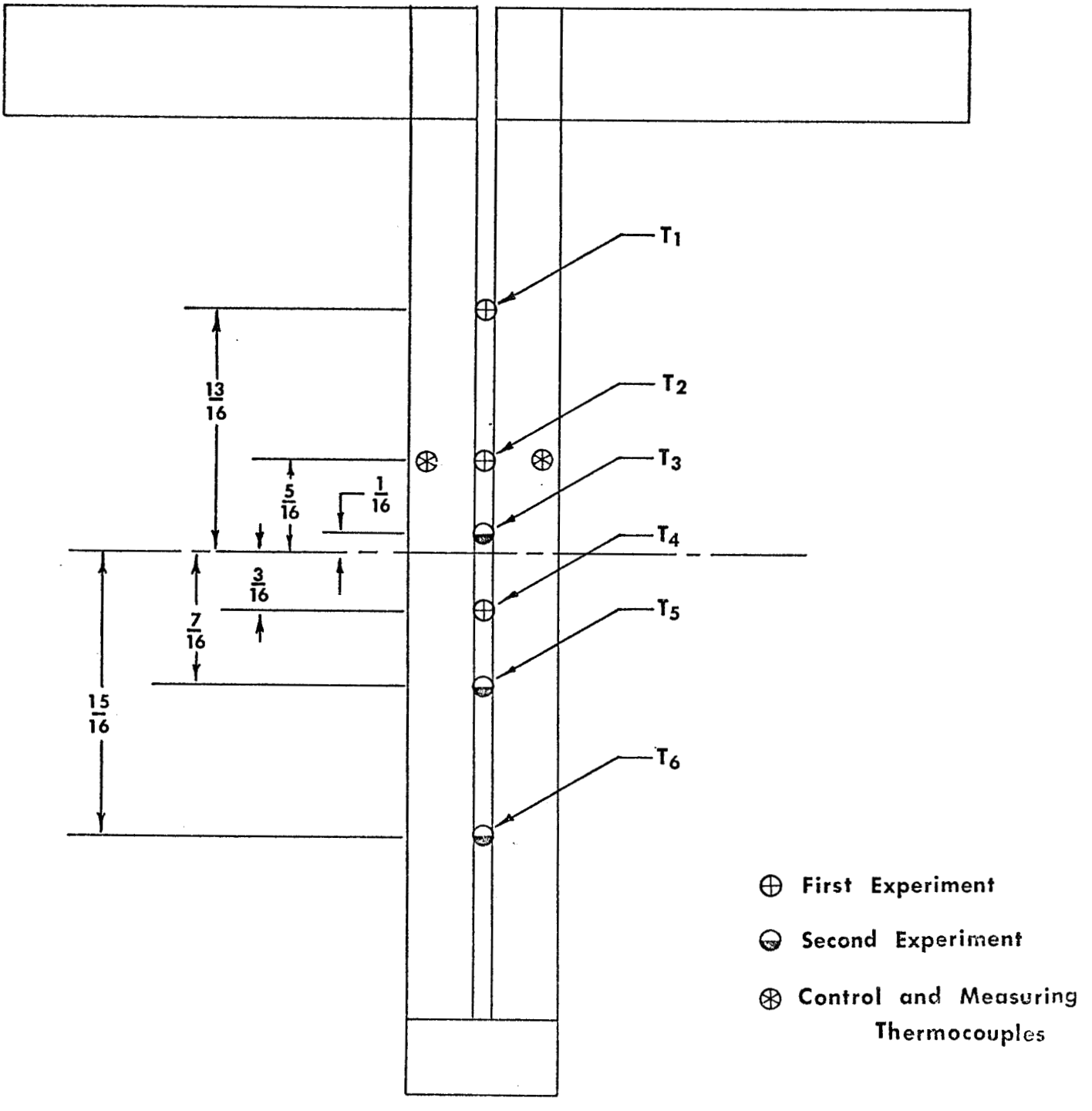
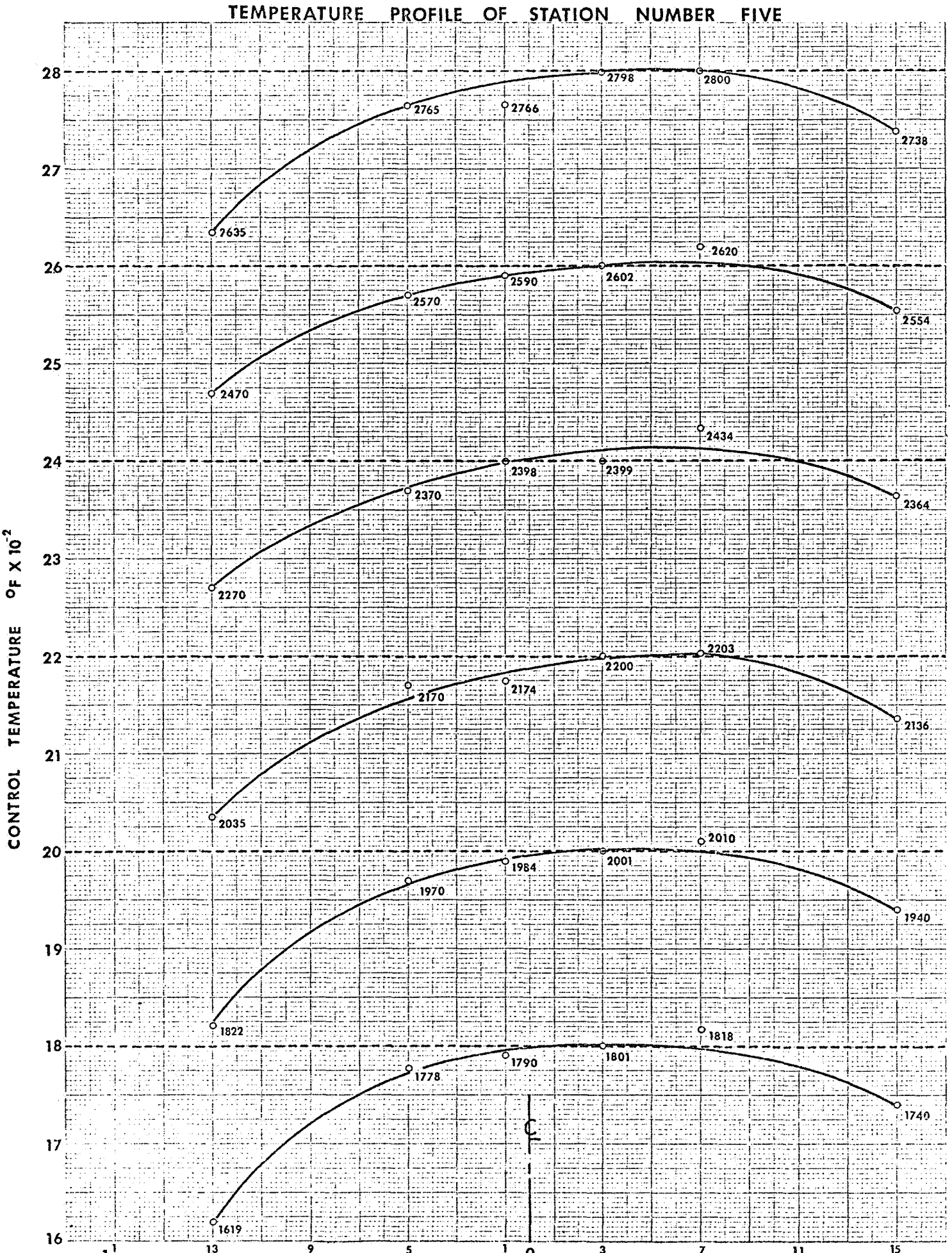


FIGURE II A 4 113

FIGURE II A 5



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B. The Ultrahigh Vacuum and Temperature (UHVT) Wire Tensile Tester

Introduction

The UHVT wire tensile tester is a tester, designed and constructed by General Electric, for the purpose of evaluating the tensile mechanical properties of small diameter wires at elevated temperatures. These tests can be made at temperatures as high as 2500°C in a vacuum atmosphere of 10^{-8} to 10^{-9} Torr. pressure. The equipment was designed for testing wire diameters ranging from .010 inches to .001 inches. A schematic drawing of this tester is shown in Figure IIB1.

Summary and Overall Description of the Equipment

The Basic Components of the Apparatus

1. Ultrahigh Vacuum Test Chamber

An ultrahigh vacuum chamber for containing the tensile test chamber is basically the General Electric Model 22TP275 250 liters/second triode ion pump. The tensile test chamber, which is made up of a furnace, radiation shields and a water-cooled jacket, is positioned vertically in the center of the ion pump chamber. The ten inch diameter top cover flange was modified to support the test chamber and its associated parts and is lifted from the ion pump chamber by a hoist to allow for easy access to the test chamber for loading the wire sample. The standard triode ion pump chamber was also modified by placing a sight glass port on the side wall, in line with a horizontal hole, for making optical pyrometer temperature measurements; and a flanged opening in the bottom for coupling the specimen-to-crosshead drive system.

2. Tensile Test Chamber

The tensile test chamber is suspended from the top flange so as to be positioned vertically in the center of the ion pump chamber. The test chamber consists of a 2-1/4" diameter x 5" long water-cooled cylinder (called the radiation shield housing) within which is positioned a radiation shield package made up of a series of .005" thick tantalum sheets. A radiation shield package consists of a set of top, middle and bottom shields, with each set made up of seven individual shields. To minimize heat loss by conduction, each individual shield is separated from the adjacent one by a set of spacers made from tantalum wire coils.

Also suspended from the top flange are two water-cooled electrodes to which is attached the furnace element. The furnace element, fabricated from .005" thick tantalum sheet, is a 1/2" diameter by 3" long tube. When it is fixed to the two electrodes, it is centrally

2. Tensile Test Chamber (Cont'd.)

located within the radiation shield housing. Inside the furnace element, centrally positioned, a 1/4" O.D. x 1/8" I.D. x 5" long tungsten tube is used as a susceptor to obtain a more uniform temperature over the length of the furnace.

3. The Load Sensor

For measuring the load during a tensile test a special load cell, designed and built by Instron Corporation, is located inside the vacuum chamber within a load cell container on the top flange. Penetration to the outside from the load cell is made through the top of the load cell cover with insulated feed throughs (in this case, Advac seals). The load cell was constructed to be usable at pressures of 10^{-8} to 10^{-9} Torr. with a minimum of outgassing. Its principle of operation, strain gages in Wheatstone bridge arrangement on a cantilever bar, is similar to other Instron strain-gage load cells. The normal range of loads sensed by this load cell is from one to fifty pounds for full-scale recorder deflection. However, by selecting the high sensitivity option of the Instron recorder, a minimum load of 0.2 pounds (full-scale deflection) can be sensed. The Instron recorder used to obtain an autographic record of the load versus elongation during a tensile test, is a potentiometric-type recorder which receives its signal from the load cell through a load cell amplifier.

4. The Load Application System

For applying the tensile load to the wire sample, the bottom sample grip is connected to an Armco iron cylinder, the internal member of an electromagnetic drive system. This iron cylinder serves as the magnetic core which is moved downward within a stainless steel tube when the external electromagnet couples with it and is also moved downward. The electromagnet was designed and fabricated like a hollow cylinder and surrounds the stainless steel tube in which the core moves. The external electromagnet is moved up or down on a pair of crosshead screws which are coupled to the drive system of a table model Instron tensile tester by a roller chain. When the electromagnet is activated, a positive coupling is obtained with the internal iron core and a vertical force is transmitted to the wire specimen as the core moves downward simultaneously with the driven electromagnet. A Sorensen D.C. power supply is used to activate the electromagnet and can supply a maximum of eighty volts D.C. at a current of five amperes. The maximum vertical pull that can be exerted under vacuum conditions on a wire specimen is approximately seven pounds.

The rate of loading a specimen is governed by the speed at which the electromagnet is moved downward and, therefore, by the rate at which it is driven by the Instron drive system. The crosshead rates can be varied from .020 inches to 50 inches per minute, but for testing

4. The Load Application System (Cont'd.)

purposes, the limits of crosshead rates are from .020 inches to 2.0 inches per minute.

5. The Vacuum Pumping System

The vacuum pumping system is made up of a sorption pump and a triode ion pump with appropriate vacuum valves for isolating parts of the system.

The sorption pump is a molecular-sieve type pump which evacuates the test chamber by the process of physical adsorption of gas molecules when the sieve material is cooled by liquid nitrogen. It is used to attain a pressure of 10^{-4} Torr. prior to high vacuum pumping with the triode ion pump.

The 250 liter per second triode ion pump which is used to attain pressures in the 10^{-9} Torr. range evacuates the test chamber by the formation of chemically stable compounds from active gases and by the ion burial of the inert gases. The ion pump has four pumping elements, each is a multi-celled anode structure mounted on the chamber wall between two titanium cathode plates and two collector surfaces surrounded by permanent magnets. The ion pump is started by applying a high negative voltage to the element cathodes and electrons are driven from the cathodes. These electrons are forced into spiral paths by the magnetic field as they attempt to reach the anodes and collide with gas molecules to produce ions. Positive ions, drawn out of the volume defined by the anode cells, are accelerated toward the sputter cathode and sputter titanium onto the collector surface. Since the cathode is an open structure, many ions also pass through but are reflected by the potential of the collector and return to the cathode. The ions, attracted to the cathodes, sputter titanium atoms which form stable compounds with the active gas atoms. The inert gas ions are accelerated toward the cathodes and are buried by the accumulating titanium compound deposits.

An ion pump control (Model 22TC275) is used to supply the high negative voltage needed to activate the triode ion pump and to measure the resulting current between the pump anode and cathode. The ion pump is protected from high pressure operation while unattended by means of a relay which is adjusted to trip at an output current of 50 milliamperes (equal to 5×10^{-4} Torr. pressure.)

6. Vacuum Pressure Sensors

a. Thermocouple Gage Tube

A General Electric thermocouple gage tube has been mounted on the rough pumping manifold line (between the vacuum chamber and the sorption pump) to monitor the initial pumpdown pressure. The millivolt output of the thermocouple is calibrated on a meter for indicating chamber pressures from 1000 microns to one micron.

b. Trigger Gage

A General Electric trigger gage (Model 22GT210), mounted on the load cell cover, is used to measure the chamber pressure in the range of 10^{-4} to 10^{-10} Torr.

c. Ion Pump Current

A measurement of the current between the anode and cathode of the triode ion pump has been calibrated for relationship to the chamber pressure on an appropriate meter. This provides for a continuous monitoring of the chamber pressure while the ion pump is operating.

7. Temperature Recorder and Controller

The electrical power to heat the furnace element is supplied by a saturable core reactor, General Electric Reactrol Model "M",utilizing low voltage-high amperage output. The Reactrol can be operated in either a manual or an automatic position. In the manual position, the amount of current is regulated and held constant by a particular setting of the power input and the temperature attained is dependent upon the equilibrium between heat input and heat loss of the system. In the automatic position the current is governed by a temperature sensor, a W-5% Re/W-26% Re thermocouple, for transmitting a control signal through a temperature controller-recorder system back to the Reactrol. The wire tester is usually operated in the automatic mode position.

The temperature controller for the wire tester is a three-mode Bristol Series 820 electronic controller with selection for manual or automatic temperature control. The temperature recorder is a Bristol Dynamaster 0-50 millivolt potentiometric recorder which monitors the millivolt output from the W-5% Re/W-26% Re thermocouple and operates in conjunction with the three-mode controller for regulating and controlling the furnace temperature.

8. Temperature Measurement

A W-5% Re/W-26% Re thermocouple, insulated with high purity zirconia tubing is used for potentiometric measurement of the furnace temperature, as well as the signal for automatically controlling the temperature. As many as four thermocouples can be used, but in the normal operation of the tester, only one is used inside the tungsten susceptor. The thermocouple wires are internally coupled to "Varian" high vacuum connectors which in turn are brought outside the vacuum chamber with OFHC copper wires soldered to the "Advac" ceramic feed-through seals on the load cell cover.

9. Temperature Profile Calibrations

Calibration experiments to determine the temperature profile of the test furnace were performed at temperatures from 1800°F to 3600°F.

9. Temperature Profile Calibrations (Cont'd.)

For these experiments a package of three thermocouples was fabricated from one common W-5% Re wire and three separate wires of W-26% Re so that the thermocouple junctions were one-half inch from each other. A diagram showing the arrangement of the thermocouple package for these calibration experiments is shown in Figure IIB2.

The calibration results are shown in Figure IIB3 for temperatures from 1800°F to 2600°F, the tensile test temperatures for this contract. In this graph are plotted the temperatures as measured with the top and bottom thermocouples versus the temperature as measured with the middle thermocouple. The results show that the most uniform temperature zone is at the position of the furnace located from the center to one-half inch below the center. The temperatures at these two positions are within 1% of each other. The results, here, are quire similar to those obtained in the temperature calibration runs of the multi-station creep-rupture tester where the uniform temperature zone was located below the center of the furnace.

ULTRAHIGH VACUUM AND TEMPERATURE WIRE TENSILE TESTER

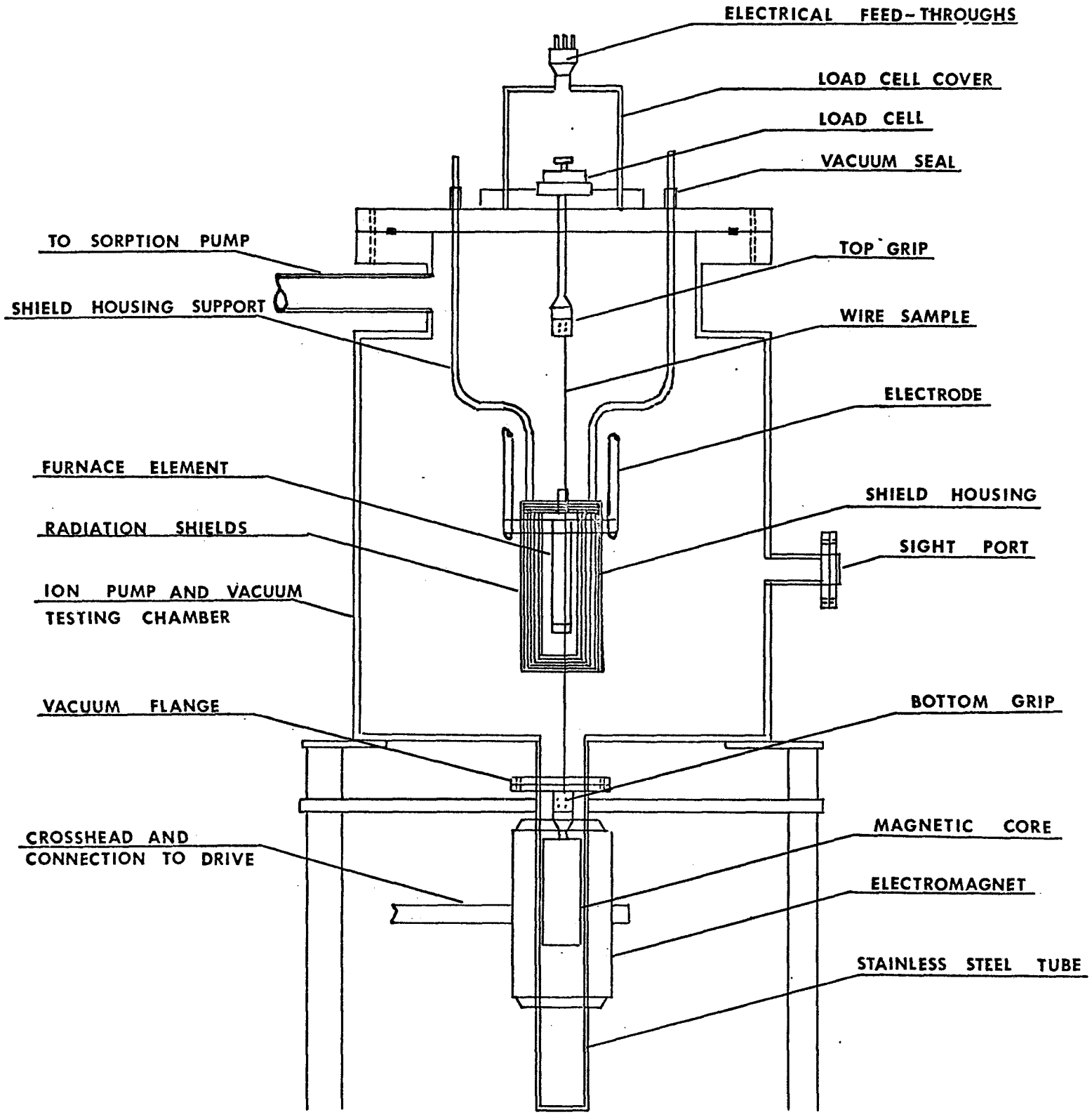


FIGURE II B 1

THERMOCOUPLE POSITIONS FOR TEMPERATURE CALIBRATION RUNS

UHVT TENSILE TESTER

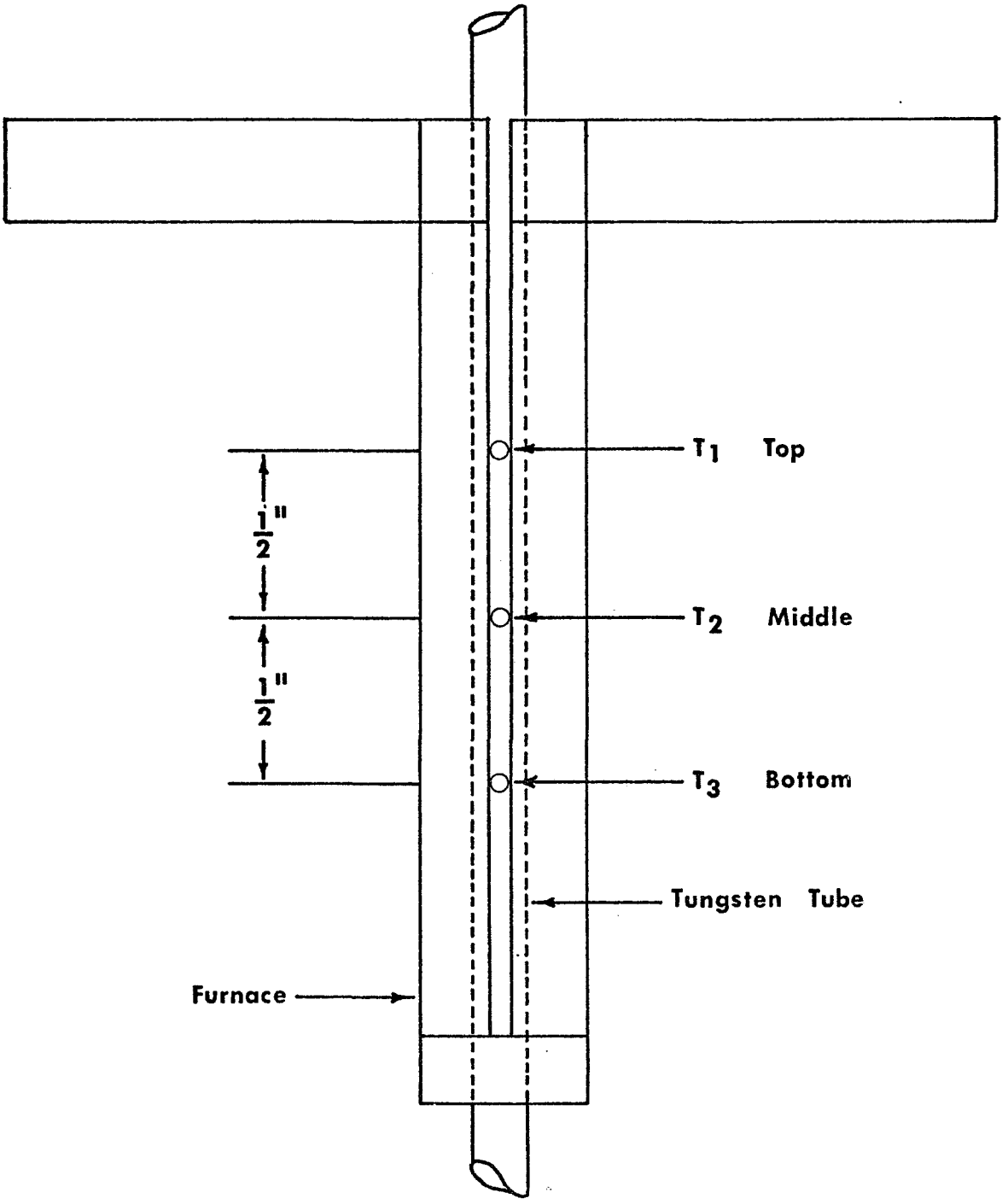
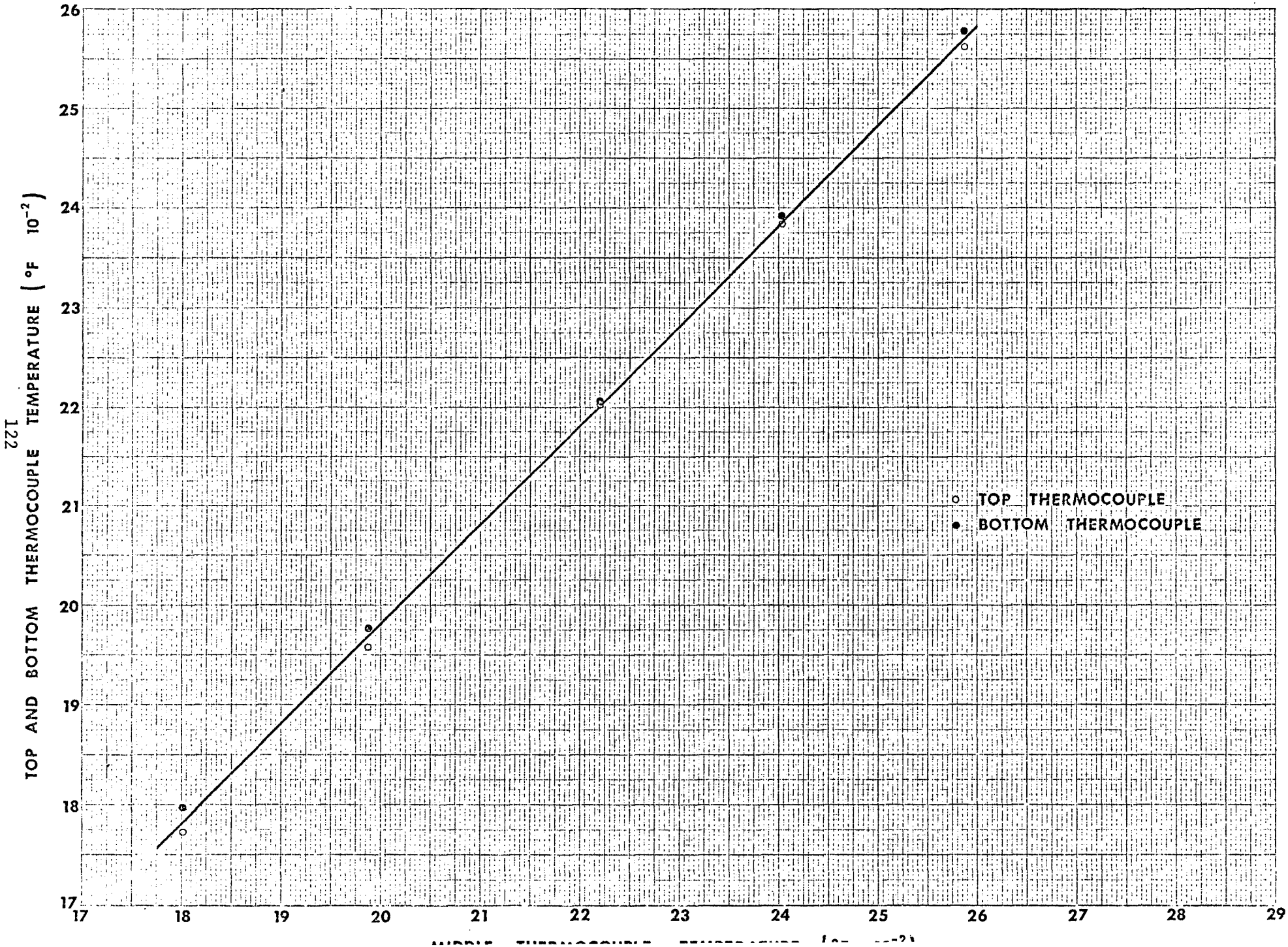


Figure II B 2 121

FIGURE II B 3

TEMPERATURE PROFILE OF UHVT TENSILE TESTER



C. Supplementary Investigations

Specimen Gage Length

Introduction

In order to define the length of wire specimen being deformed during a creep-rupture test, it was necessary to find a method to reduce uniformly the diameter of a section of wire exactly one inch long. Preliminary experiments using electrochemical etching revealed that a more uniform diameter gage length could be obtained by use of alternating current rather than direct current. In addition, it was found that rotation of the wire on its axis during electrochemical etching produced a preferred and better specimen. Specifically, this method yields smoother and more gradual diameter changes at the ends of the gage length.

Etching Apparatus (See Figure IIC1)

The apparatus designed for etching the 1.00 or .75 inch gage sections is shown in Figure IIC1. The basic features of this apparatus are:

1. Plexiglass etching container
2. Braided tungsten wire coil
3. Capillary glass tubing
4. Variable speed motor
5. A.C. voltage variable transformers and voltmeter

The plexiglass container is fabricated so that its internal diameter is exactly equal to the dimension of the desired gage length. Through the walls of the container are two diametrically opposed .015 inch diameter holes. A tungsten coil, 5/16 inch I.D., of .065" diameter braided wire is inserted into the ten normal NaOH solution and serves as the anode. This coil is positioned such that the wire specimen passes through its center when the specimen is threaded through capillary glass tubing and plexiglass container. One end of the wire is gripped by a pin vise chuck which is rotated by a variable speed motor.

Procedure

The wire specimen was inserted through the plexiglass container, tungsten coil anode and capillary glass tubing and attached to the variable speed motor by the pin vise chuck. The variable speed motor for these experiments was a Dremel Model-2 Moto-Tool, the speed being controlled by controlling the input voltage with a variable AC transformer. No attempt was made to measure the actual rotational speed as the voltage was varied. The speed was varied to determine the effect of rotation on uniformity of etching. The AC voltage for etching was controlled and varied by using a Powerstat variable transformer in the circuit. The etching voltage used was measured by a voltmeter. Preliminary experiments for the determination of feasibility of this method were tried on .007" diameter 218 tungsten wire under the following experimental conditions.

Procedure (Cont'd.)

Experimental Conditions

Experiment No.	Moto-Tool Rotation Speed (AC Volts)	Etching Voltage (AC Volts)	Etching Time (Seconds)
1	25	9	5
2	25	9	10
3	25	9	20
4	40	9	20
5	60	9	20
6	25	4.5	40
7	25	2	90

After each wire specimen was etched as described above, dimensional measurements were made on the etched section using an optical microscope with a 10X objective lens and the Bausch and Lomb filar micrometer eyepiece from a Tukon Microhardness Machine. The filar micrometer was calibrated directly by means of a stage micrometer. The sensitivity of this method is calculated to be 20×10^{-6} inches. Diameter measurements were then made by traversing the gage length and making measurements at 1/4 inch intervals. Measurements to show the transition from the etched section to the original diameter were not made. However, photographs of typical junctions were taken.

Results from these experiments indicated that electrochemical etching method could be used to obtain a reasonably uniform gage length with a diameter less than that of original wire. The final gage length diameter is dependent on the etching parameters, such as time, etching voltage and rotational speed.

The next step was to determine whether or not the etching process caused a significant change in mechanical properties. Tensile tests at room temperature and at 1670°C were made on unetched wire and wire etched approximately .0005" and .002". The .2% offset yield stress, ultimate tensile strength and percent elongation were determined from the load-deflection test data record of an Instron tensile testing machine.

From the experience gained with 218 W wire, TZC .005" diameter wire, drawn for property evaluation in this contract, was etched using the same technique and the same electrolyte. The etching parameters for the TZC were as follows:

Procedure (Cont'd.)

Experiment No.	Experimental Conditions: Moto-Tool Rotation Speed (AC Voltage)	Etching Voltage (AC Voltage)	Etching Time (Seconds)
1	25	4.5	15
2	25	4.5	20
3	25	5	15
4	25	2.5	40

Room temperature tensile tests were made on these materials to again see whether the mechanical properties were affected by the etching process.

Results of Etching Experiments

218 Tungsten .007" Diameter Wire

Figure IIC2 shows a graph of the variations in diameter of the gage length profile. In addition, the influence of variations in rotational speed, etching voltage and time are shown. It can be seen that the variations in diameter from one end of the gage length to the other are quite small (approximately .0001") for the poorest case and are significantly better if the proper etching conditions are selected (such as 25-9-20).

Photographs of the gage section and the area of transition from the original to the etched diameters are shown in Figures IIC3, 4, 5, and 6. Figures IIC3 and IIC5 show the gradual reduction of diameter at 100X magnification for the 25-9-5 (diameter decreased by .0005) and the 25-9-20 (diameter reduced .002") respectively. Figures IIC4 and IIC6 show the entire gage length at 10X magnification for these same etching parameters.

Table IICI is a compilation of the room temperature and 1670°C tensile properties of etched and unetched 218 tungsten wire. Although the data presently available should be considered as quite limited, some tentative observations and conclusions are suggested. At both room temperature and 1670°C the ultimate tensile strength of 218 tungsten wire does not appear to be influenced by the etching process. For example, at room temperature five samples of unetched wire gave an average ultimate tensile value of 425,500 psi (range of values 423,600 psi to 426,200 psi). The etched samples gave room temperature ultimate tensile strengths averaging 413,300 psi (range 408,900 psi to 417,800 psi). Thus, agreement is indicated within approximately three percent. At 1670°C the ultimate tensile strengths of an unetched sample 93,100 psi compares very favorably with three values listed for etched samples (average = 92,400 psi).

Procedure (Cont'd.)

For the .2% yield strength, at room temperature, the average value for etched samples is 361,000 psi compared to the average 363,800 psi for unetched wire. At 1670°C the average value of 90,400 psi is within 4% of the single value now available for an unetched sample. The room temperature elongation data (of which there is admittedly a paucity) indicate that with a change in diameter of .0015" or greater from etching, the elongation is approximately 1/3 that of the unetched material. However, with a diameter change of only .0004", the elongation of the etched material is about 3/4 that of the unetched. At 1670°C, the elongation of etched samples is 1/2 that of the unetched. The amount of material removed by etching does not appear to be as important at this elevated temperature.

TZC .005" Diameter Wire

Figure IIC7 shows the variation of diameter with gage length profile. In this case, a constant speed of rotation was chosen and the etching voltages and times varied. Of significance here is the fact that the etching voltage needed to be decreased to roughly 1/2 that used for the 218 W .007" diameter wire. At this decreased etching voltage measurements of the resulting etched diameter revealed extremely good uniformity in the gage section.

Photographs at 100X magnification in Figures IIC8 and IIC9 show that portion of the etched wire at the junction of the original and gage length diameters. Figure IIC8 is the TZC sample decreased approximately .0005" in diameter by etching conditions of 25-4.5-15 and Figure IIC9 is the TZC sample representative of a .001" decrease in diameter by 25-5.0-15 etching conditions.

Table IICII is a compilation of the room temperature tensile property data for TZC samples etched by the technique discussed above. Comparison of the strength properties reveals no significant influence of the ultimate tensile strength of the material by the etching process. The average ultimate tensile strength for etched wire (215,600 psi) is almost identical to the average value of 215,700 psi for unetched wire. On the other hand, the data for .2% yield strength show the unetched wire to have an average value (189,800 psi) lower than the average (199,600 psi) for etched samples. This difference is approximately five percent of the unetched value. All percent elongation values for etched wire are lower than for the unetched. It is not now apparent that this can be correlated with the degree of etching.

Conclusions

From the results of the etching experiments presented here, it was apparent that this technique could be used to establish a uniform gage length section in a wire specimen in order to define the deformation zone during mechanical property tests. The data seem to indicate that the properties of the etched material approximate those of the unetched

Conclusions (Cont'd.)

material. For the ultimate strengths and .2% yield strength, correspondence has been demonstrated to within 5% for 218 tungsten wire at room temperature and 1670°C and for TZC at room temperature. Apparent significant differences in percent elongation have been observed both in tungsten and TZC. In all cases, percent elongation is lower for etched specimens.

Etching Conditions for Materials Creep-Rupture Tested

Of the four refractory metal alloy wires tested, only three of them, FS-85, TZC and AS-30, were electrochemically etched in the 10 N NaOH electrolyte solution. For all of these wire materials the etching conditions of 25 volts A.C. rotational voltage and 5 volts A.C. etching voltage were maintained to reduce the gage length diameter to approximately .004 inches. The etching times, however, were varied to obtain the required diametral reduction. The etching times for FS-85, TZC and AS-30 wire specimens were 240 seconds, 20 seconds and 20 seconds respectively.

The tantalum alloy wire, T-222, was chemically etched in a solution of five parts concentrated sulfuric acid, one part concentrated hydrofluoric acid, one part concentrated nitric acid and two parts of distilled water for five minutes to reduce the diameter to .0045 inches. For the preparation of these specimens only, the wire was inserted in the plexiglass container and was not rotated while being etched.

Control Thermocouple Switching Experiments

To establish the feasibility of an automatic temperature control switching arrangement on each of the test stations, it was necessary to estimate the rate of change of temperature with time at power interruption. Experiments in a Brew furnace (vacuum pressure 5 x 10^{-6} Torr. and temperature 2400°F) were made where the change in temperature at the center of the furnace, as measured by a thermocouple, was recorded on an X-Y recorder as a function of time when the power to the furnace was instantaneously shut off.

The average results of five such experiments are graphed in Figure IIC10. The data are shown for the actual temperature vs. cooling time and also as the change in temperature vs. the cooling time. From these experiments, it can be seen that in five seconds after complete power shut off (a condition which approximates the automatic switching from the control thermocouple to the recording thermocouple for control) the furnace cooled 2.5°F. For time intervals of seven, eight, and 10 seconds, the furnace cooled 12°F, 22°F, and 62°F respectively.

It was assumed that this experiment adequately approximated the test furnaces to be constructed for the multi-station tester and that it was desirable for the temperature control switching system to be designed into the equipment. The transfer of temperature control from one thermocouple to the other within a 0 to 7 second period was, therefore, incorporated.

TABLE IICI

218 Tungsten .007" Diameter Wire

Etching Procedure	Test Temperature	Average G.L. Diameter	Ultimate Tensile Strength in psi	.2% Yield Strength in psi	% Elongation
25-9-5A	1670°C	.00644	95,400	93,400	0.4
25-9-5B	25°C	.00659	417,800	325,400	3.22
25-9-20A	1670°C	.00520	95,500	93,500	0.4
25-9-20B	25°C	.00522	408,900	369,200	1.47
25-4.5-40A	1670°C	.00546	86,200	84,500	0.4
25-4.5-40B	25°C	.00534	415,200	388,400	1.35
Unetched	1670°C	.00700	93,100	87,100	0.8
Unetched	25°C	.00700	425,500*	363,800*	4.33*

*Average results of five tensile test on unetched wire

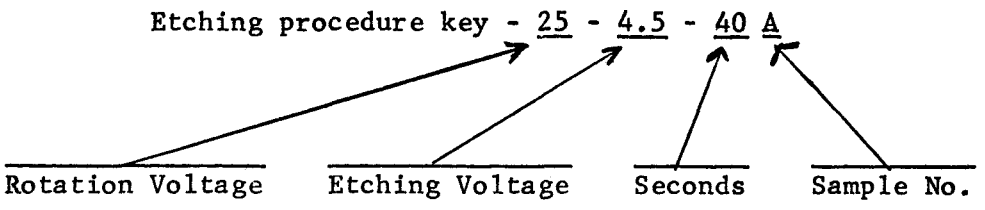
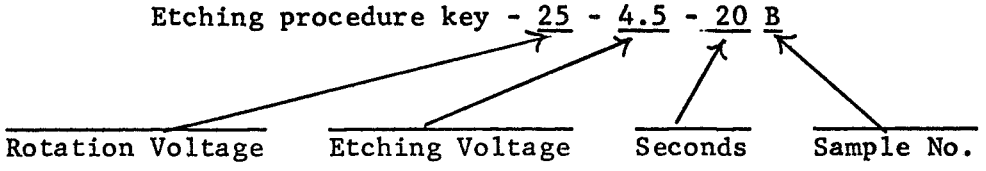


TABLE IICII

TZC .005" Diameter Wire

Etching Procedure	Test Temperature	Average G.L. Diameter	Ultimate Tensile Strength in psi	.2% Yield Strength in psi	% Elongation
25-4.5-20B	Room Temp.	.00366	218,200	200,200	1.88
25-4.5-20C	Room Temp.	.00363	217,600	196,300	1.47
25-9-5A	Room Temp.	.00446	214,900	202,100	1.44
25-4.5-15A	Room Temp.	.00415	217,700	201,500	2.24
25-4.5-15B	Room Temp.	.00421	218,800	201,600	2.06
25-5-15B	Room Temp.	.00402	223,500	206,300	2.54
25-5-15C	Room Temp.	.00398	212,900	194,400	2.26
25-2.5-40C	Room Temp.	.00398	206,100	190,800	2.68
25-2.5-40B	Room Temp.	.00396	210,900	194,700	2.37
Unetched*	Room Temp.	.00500	215,700*	189,800*	3.93*

*Average results of six tensile test on unetched wire



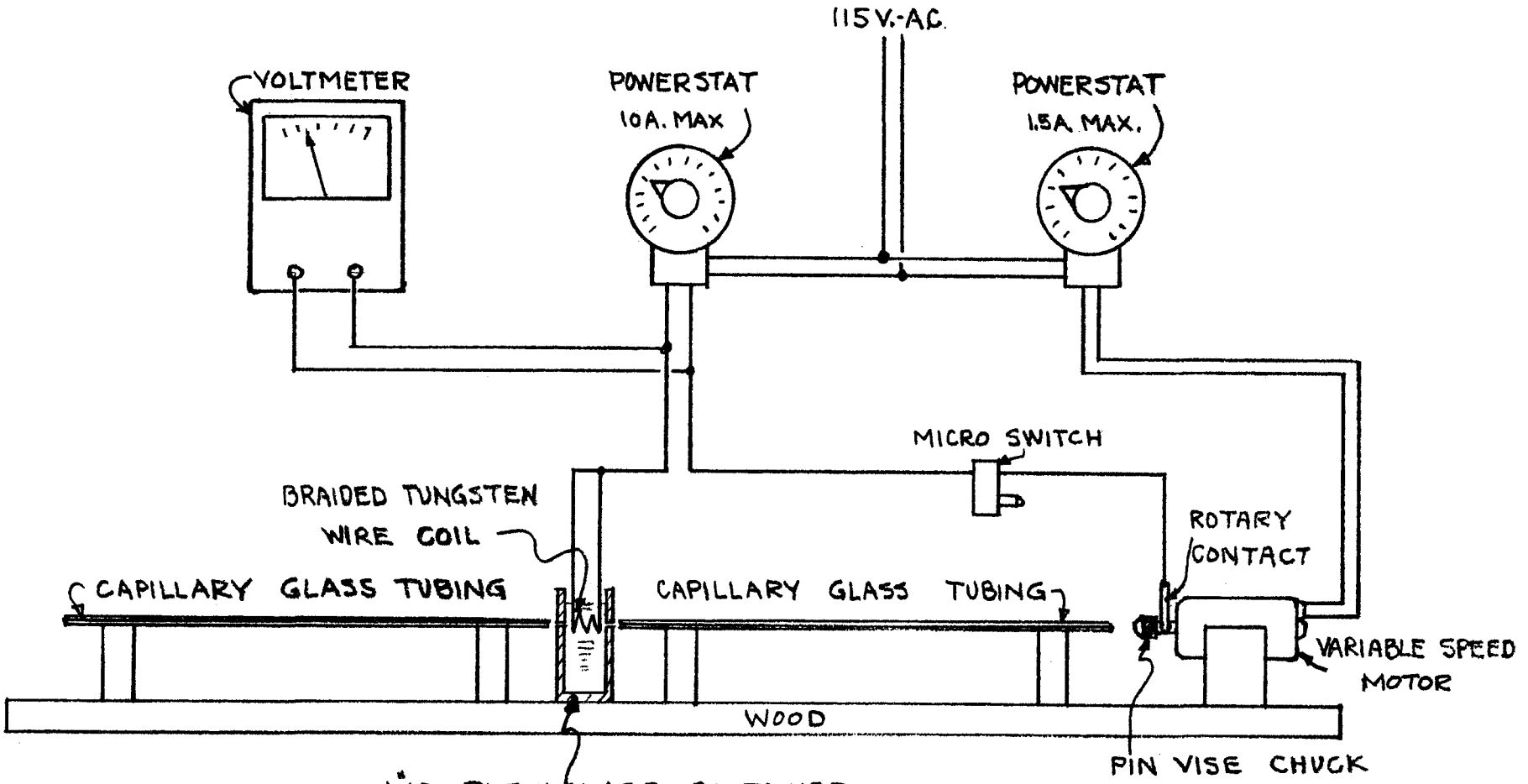


FIG. II C1 - WIRE ETCHING DEVICE

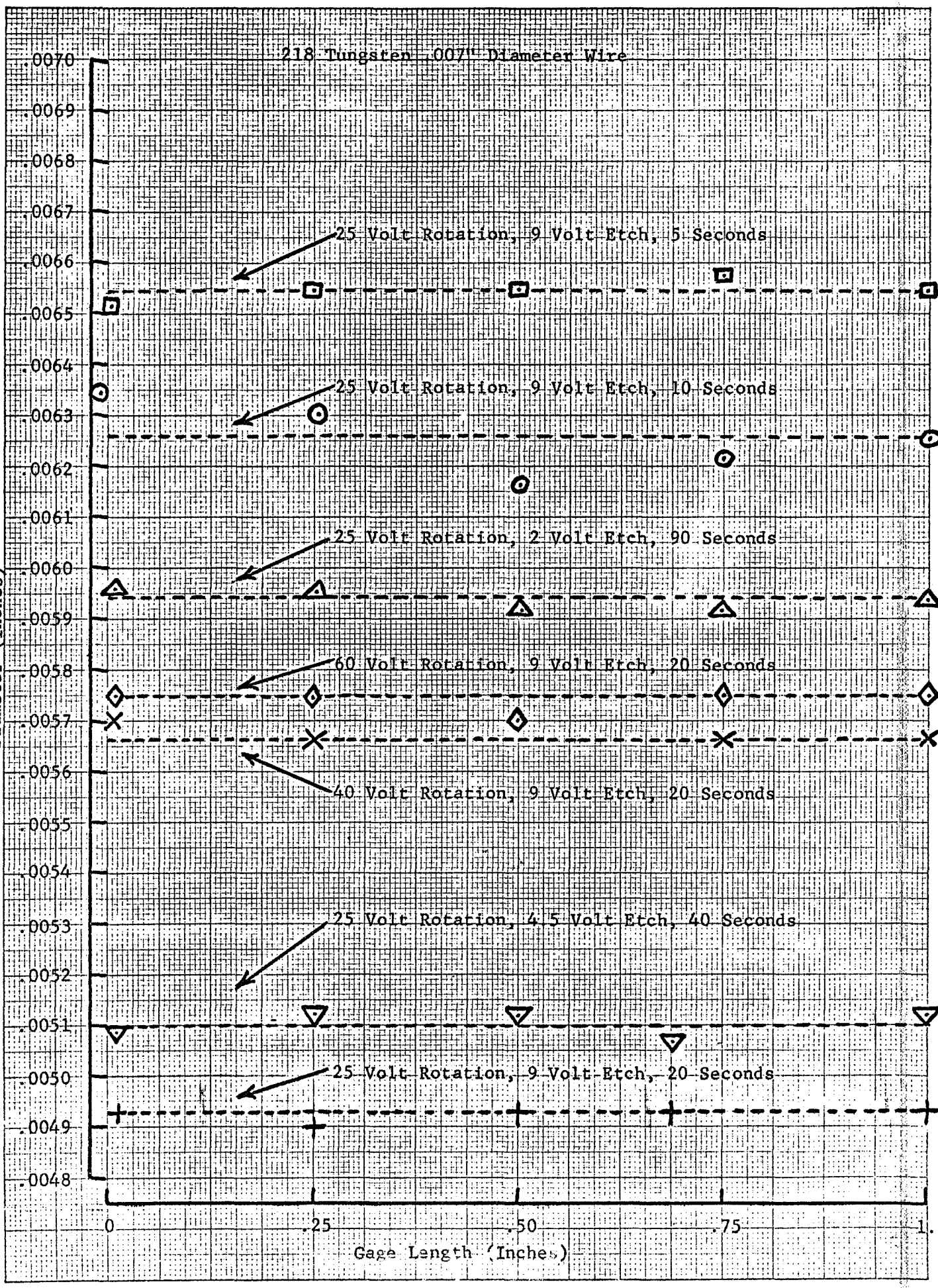

218 Tungsten .007" Diameter Wire
25 Volt Rotation, 9 Volt Etch, 5 Seconds
25 Volt Rotation, 9 Volt Etch, 10 Seconds
25 Volt Rotation, 2 Volt Etch, 90 Seconds
60 Volt Rotation, 9 Volt Etch, 20 Seconds
40 Volt Rotation, 9 Volt Etch, 20 Seconds
25 Volt Rotation, 4.5 Volt Etch, 40 Seconds
25 Volt Rotation, 9 Volt Etch, 20 Seconds
Diameter (Inches)
.0070
.0069
.0068
.0067
.0066
.0065
.0064
.0063
.0062
.0061
.0060
.0059
.0058
.0057
.0056
.0055
.0054
.0053
.0052
.0051
.0050
.0049
.0048
0
.25
.50
.75
1.0
Gage Length (Inches)

FIGURE - IIC2

218 Tungsten Wire

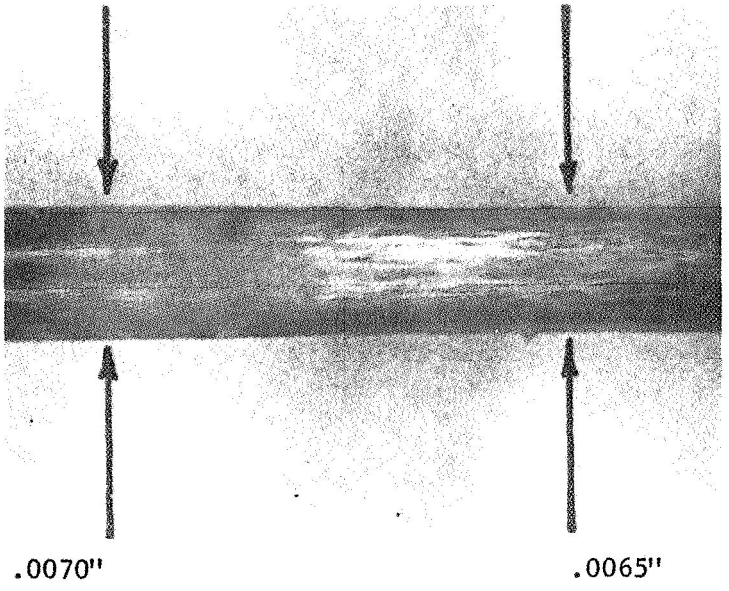


FIGURE IIC3 - 25 Volt Rotation, 9 Volt Etch
5 Seconds, 100X

218 Tungsten Wire

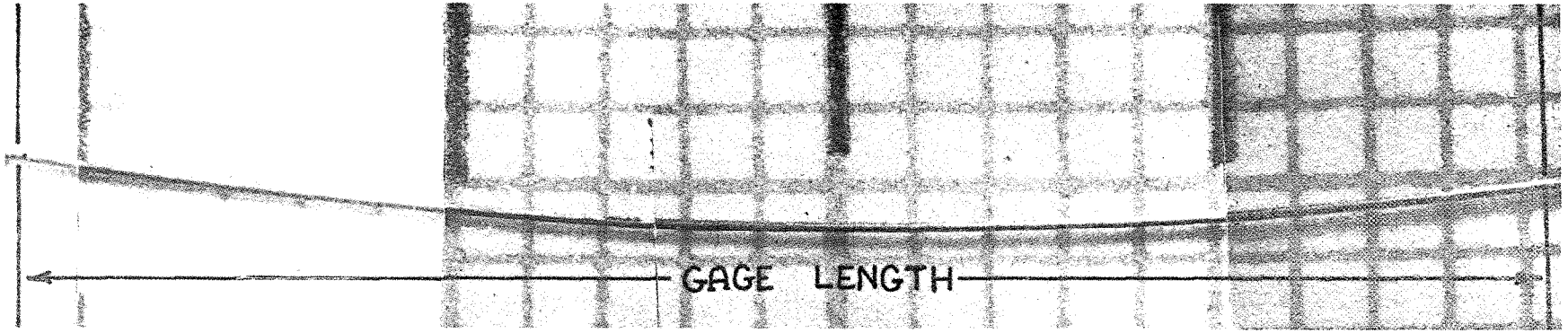
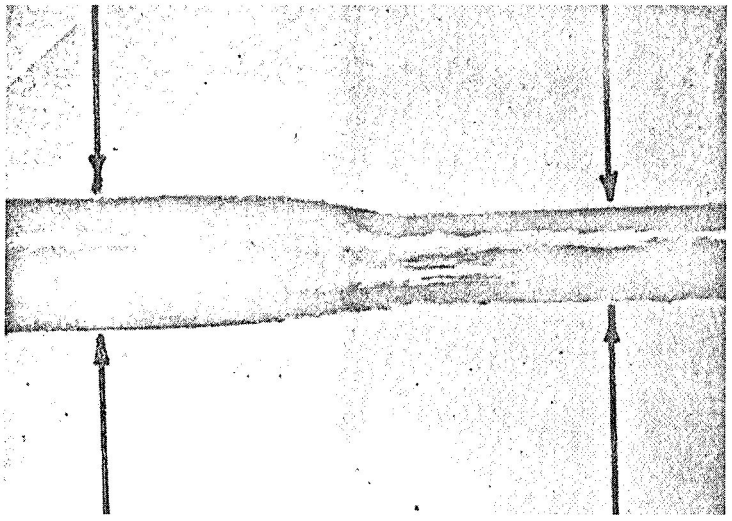


FIGURE IIC4 - 25 Volt Rotation, 9 Volt Etch, 5 Seconds, 10 X

218 Tungsten Wire



.00700" .0049"

FIGURE IIC5 - 25 Volt Rotation, 9 Volt Etch, 20 Seconds, 100X

218 Tungsten Wire

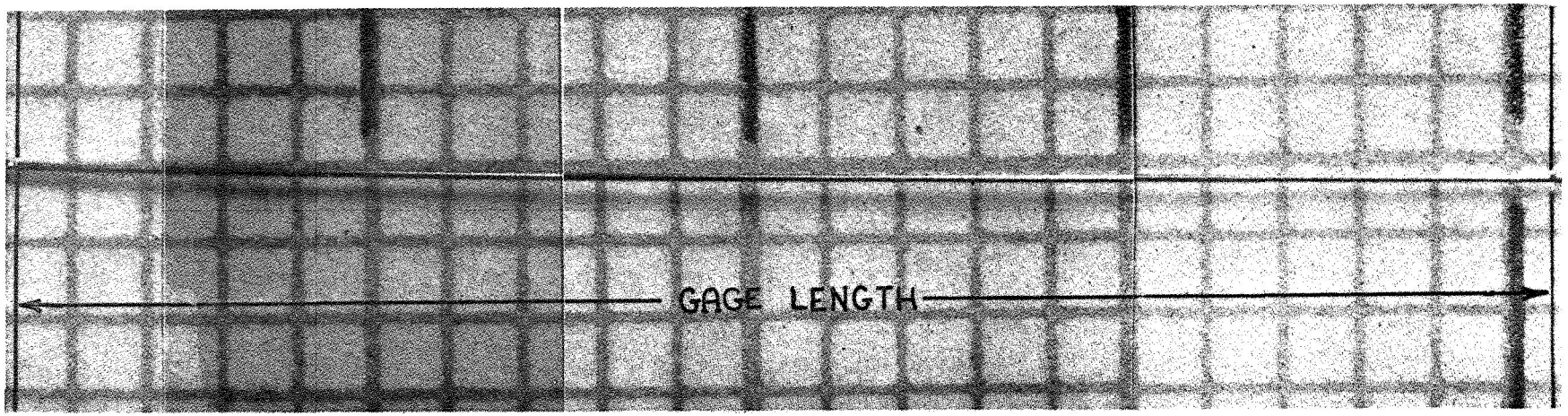


FIGURE IIC6 - 25 Volt Rotation, 9 Volt Etch, 20 Seconds, 10 X

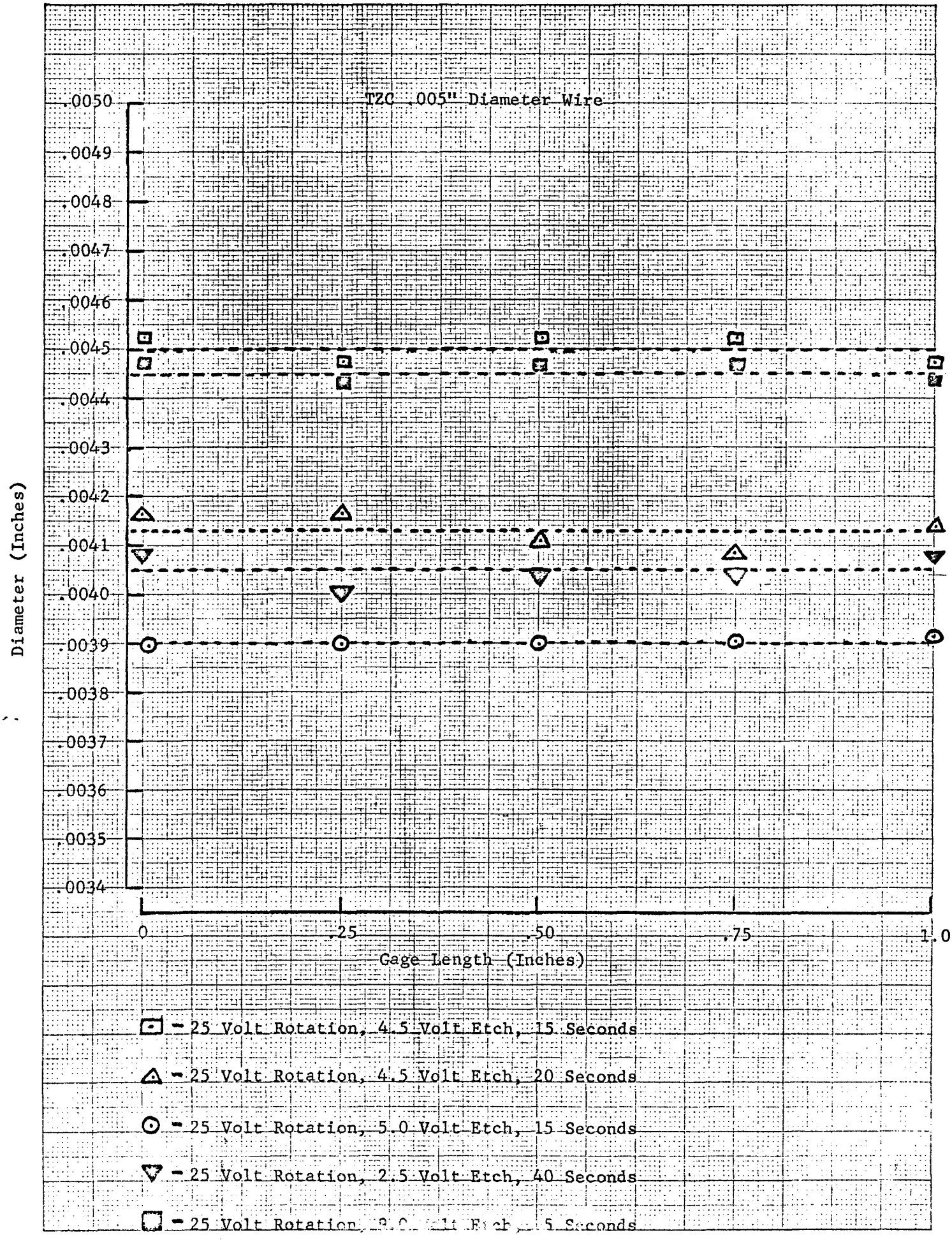
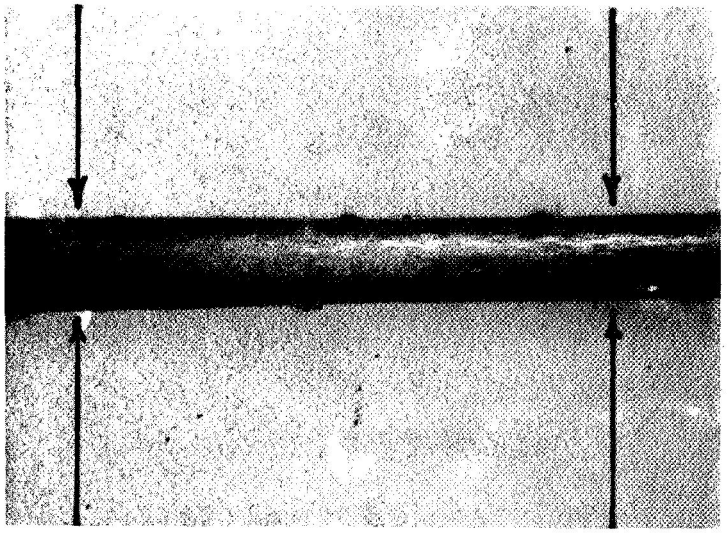

TZC .005" Diameter Wire
.0050
.0049
.0048
.0047
.0046
.0045
.0044
.0043
.0042
.0041
.0040
.0039
.0038
.0037
.0036
.0035
.0034
Diameter (Inches)
0
.25
.50
.75
1.0
Gage Length (Inches)
- 25 Volt Rotation, 4.5 Volt Etch, 15 Seconds
- 25 Volt Rotation, 4.5 Volt Etch, 20 Seconds
- 25 Volt Rotation, 5.0 Volt Etch, 15 Seconds
- 25 Volt Rotation, 2.5 Volt Etch, 40 Seconds
- 25 Volt Rotation, 3.0 Volt Etch, 25 Seconds

FIGURE - IIC7

TZC Wire



.0050" .0045"

FIGURE IIC8 - 25 Volt Rotation, 4.5 Volt Etch
15 Seconds, 100X

TZC Wire

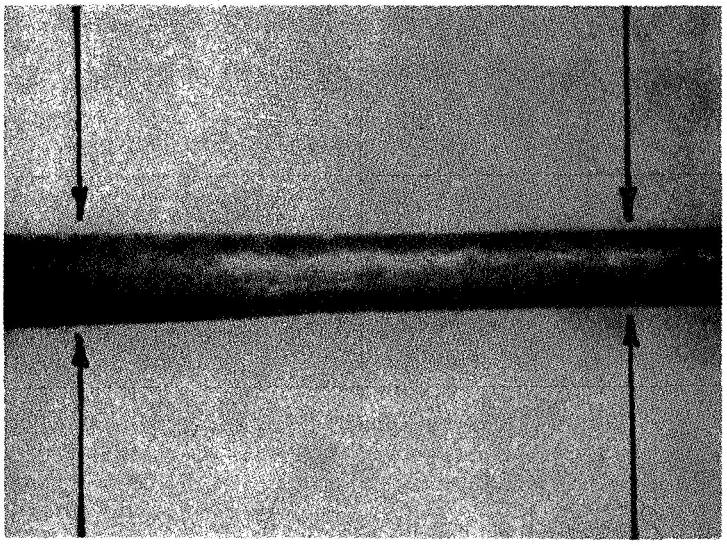


FIGURE IIC9 - 25 Volt Rotation, 5 Volt Etch
15 Seconds, 100X

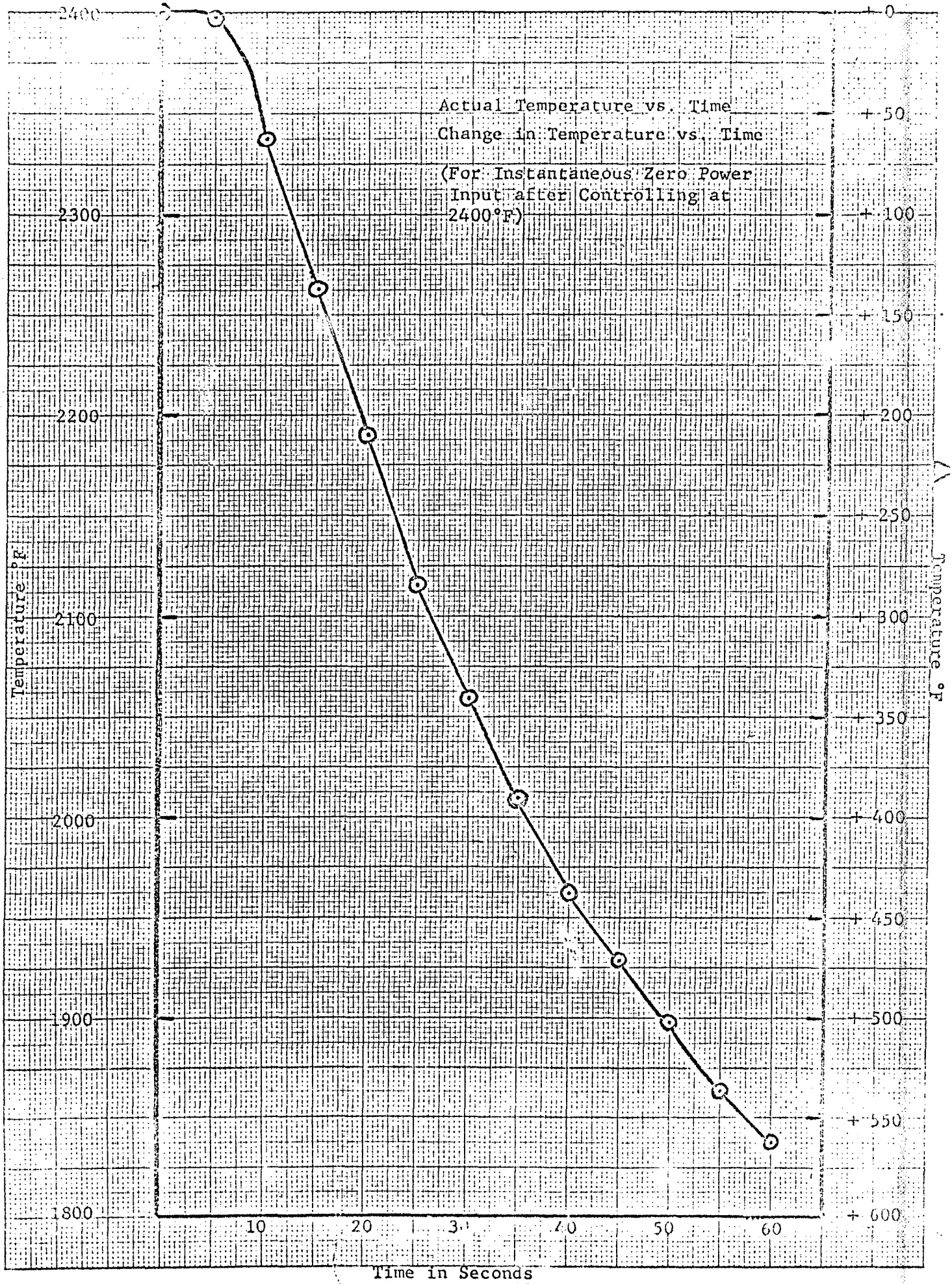


FIGURE - IIC10

D. Testing and Results

Tensile Testing Procedure

The tensile tests were all performed in the UHVT wire tensile tester on the wire materials in the "as-received" condition. The diameters of the wire samples were those specified as the final wire size by the contract and no attempt was made to test samples with a reduced gage section. For each test a 15 inch length of wire was required. The three inch section of the wire sample which would be located in the furnace zone was measured optically to determine its diameter for subsequent calculations of the mechanical properties. The optical device used for all diameter measurements (a microscope with 10X objective lens and a calibrated filar eyepiece) was capable of determining the measurements to 20 x 10^{-6} inches. The sample was attached to the top and bottom grips and coupling was made to the internal Instron load cell and the Armco iron cylinder. The chamber was closed and then evacuated to a pressure of 5 x 10^{-9} Torr. prior to heating the sample to the test temperature. The Armco iron core was held suspended with the field force of the electromagnet as heating took place so that there would be no tensile force applied to the wire specimen until the test was ready to be started. When all the testing conditions were met the tensile test was run and the tensile properties were calculated from the load-extension record.

Creep-Rupture Testing Procedure

All the wire samples used for the creep-rupture tests were prepared with a definitive gage section by the etching processes as described in II-C. Those which did not have this gage section were part of an additional investigation for this contract and were tested for rupture data only.

The diameter of the specimen, with or without the gage section, was measured optically as described above. If there was a variation in diameter for any one specimen, the minimum diameter was always used for the calculation of the stress. For those specimens with the gage section, a measurement of the length of the reduced section was made with a traversing microscope. The load train for a particular test (made up of the bottom specimen grip, chuck holder, weight pan and rod, weights, LVDT core rod and core) was measured to the nearest .05 gram on a beam balance.

Before placing the samples in the test stations, each of the linear variable differential transformers (LVDT) was calibrated if creep measurements were to be made. The calibrations were made with the core in its normal test position, so that it was separated from the transformer coils by the stainless steel tube. The "wire break" circuit for each test station was checked for every test, creep or stress rupture, to insure

Creep-Rupture Testing Procedure (Cont'd.)

that all station control power would shut off upon specimen fracture. In addition, each test station was visually examined for anything faulty such as shorted thermocouples, shorted radiation shields, etc. After the specimen was inserted through the furnace zone and attached to the grips and load train, it was aligned with reference to the vertical centerline of the furnace. The chuck holder of the load train system was then placed on the pivot bar so that the load could be removed to begin the creep test.

The chamber was then closed and evacuated to a pressure of $<5 \times 10^{-9}$ Torr. before each test station was heated to the desired temperature. During the heat-up period, the pressure was not allowed to rise above a pressure level of 1×10^{-7} Torr. After the test temperature was attained and being controlled automatically the pivot arm was lowered to release the weight (load train) onto the specimen. At the same time as this was being done, the LVDT was repositioned, if needed, and the single point recorder was monitoring the creep deformation.

Stress-Rupture Testing Procedure

The procedure for stress-rupture testing was essentially the same as that used for the creep-rupture tests except that the LVDT creep sensing system was not calibrated prior to the test and was used only for monitoring the fracture time. For the tests which required an inert atomosphere of argon, the test chamber was evacuated, without a bakeout cycle, using the sorption pumps and triode ion pump until a pressure of $<5 \times 10^{-7}$ Torr. was obtained. The argon gas was then put into the test chamber to a pressure of one atmosphere. The argon used was cylinder argon, analyzed to have a purity of 99.999%. The analysis of this cylinder argon is listed below:

H_2O vapor	<1 ppm	(Dew point <-105°F)
O_2	<1 ppm	
N_2	<5 ppm	
Carbonaceous Material	<1 ppm	

Heat Treatment and Chemical Analysis Procedure

One-gram samples of the alloy wires produced for this contract were chemically analyzed in the "as-received" and "heat-treated" conditions for the interstitial elements: oxygen, hydrogen, nitrogen and carbon. Each one gram sample was made up by compacting the required length of .005" diameter wire into a bundle approximately one inch long. The "as-received" samples were degreased with C.P. acetone prior to their analyses. The "heat-treated" samples were degreased with C.P. acetone, placed in a test station of the multi-station creep-rupture tester and then heat treated at a temperature of 2300°F for 50 hours at a pressure of $<5 \times 10^{-8}$ Torr. After the heat treatment was completed, the samples were removed and chemically analyzed without any further treatment.

Heat Treatment and Chemical Analysis Procedure (Cont'd.)

The chemical analysis method for determining the interstitial elements, oxygen, hydrogen and nitrogen, was the vacuum fusion gas technique (manometric fractional freezing method). The carbon analyses were made on a Leco instrument using a thermal conductivity technique for analysis of carbon dioxide on samples weighing approximately .2 gram.

Mo-TZC Results

Tensile Tests

Two sets of tensile tests on Mo-TZC .005 inch diameter wire were made at 70°F, 1800°F, 2000°F, 2100°F, 2200°F, 2400°F, and 2600°F. At each one of the test temperatures greater than 70°F two specimens were tested. The first set of tensile tests was performed on wire specimens made by the drawing method, Process A; the second set on wire specimens by the drawing method, Process B. Figures IID1 and IID2 and Tables IIDI and IIDII give the results of these tensile evaluations. The 0.2% yield stress for all tensile tests was calculated on the assumption of a one inch specimen gage length and the reduction in area was calculated from the final diameter measurements, made optically as described previously, at the fracture point. The results of these tests show that the Process B wire has a significant higher yield and tensile strength than the Process A wire at the elevated test temperatures, even though its room temperature yield and tensile strengths are less. For example, at 70°F, the ultimate tensile strength of Process A wire is about 10% higher than that for Process B wire; but at 2400°F, the ultimate tensile strength of Process B wire is about 40% greater than that of Process A wire. The ductility of the wire from the two processes, as measured by the reduction in area, is slightly less for the Process B wire at the elevated temperatures. Because of its greater tensile strength properties at the elevated test temperatures, Process B wire was selected for the creep-rupture evaluations.

Creep and Stress-Rupture Tests

Creep-rupture tests to define the 20-hour rupture life of Mo-TZC were performed on etched gage-length samples at 2000°F and 2300°F. In addition, two stress-rupture tests at 2000°F on unetched samples were run in an argon atmosphere. The results of the tests on Mo-TZC, as well as those on AS-30, FS-85 and T-222, showed some inconsistencies in the data which might be attributed to the effect of the ultrahigh vacuum atmosphere or the etching of the diameter to get a uniform gage length. Since all the creep-rupture information was obtained from a minimum testing program, these data are not reported here, but will be presented in a future report.

Chemical Analysis

The chemical analysis results for determining the increase or decrease in the levels of oxygen, nitrogen, hydrogen and carbon for a one-gram sample subjected to a heat treatment at 2300°F for 50 hours in vacuum and a similar sample not exposed to these conditions are given below:

Sample Material	Element (ppm)			
	Oxygen	Nitrogen	Hydrogen	Carbon
"As Received"	119±12	29±5	11±5	1263
Vacuum Heat Treated	24±5	14±5	1±1	1115

From these results, it is apparent that a vacuum heat treatment of Mo-TZC decreases the interstitial content of the wire. The decrease in oxygen level, however, is the most conspicuous.

Table IIDI

Tensile Test Data

Mo-TZC Process A

Sample No.	Test Temp.	Ultimate Tensile Strength, psi	.2% Yield Strength, psi	% Reduction in Area
A	70°F	242,000	209,400	54.2
B	70°F	242,500	208,400	55.2
C	70°F	241,500	205,300	53.2
1	1800°F	135,000	113,600	83.8
2	1800°F	65,500	*	85.4
5	2000°F	85,000	84,100	95.1
6	2000°F	87,800	87,300	93.4
9	2100°F	76,900	71,300	92.1
10	2100°F	76,900	74,900	93.0
3	2200°F	55,000	54,500	97.6
4	2200°F	58,050	47,400	97.3
7	2400°F	36,200	31,600	96.8
8	2400°F	37,700	35,650	96.6
11	2600°F	26,500	23,900	95.7
12	2600°F	21,900	17,100	96.0

*This test showed a premature break and no yield data could be calculated from the record.

Test Conditions:
Cross Head Rate .05"/minute
Chart Speed 5"/minute
Atmosphere $<5 \times 10^{-8}$ Torr.

Table IIDII

Tensile Test Data

Mo-TZC Process B

Sample No.	Test Temp.	Ultimate Tensile Strength, psi	.2% Yield Strength, psi	% Reduction in Area
A	70°F	217,000	184,000	52.9
B	70°F	209,400	179,800	51.9
C	70°F	210,400	182,400	52.9
D	70°F	218,000	183,900	51.9
1	1800°F	152,300	137,500	79.5
2	1800°F	122,800	114,600	80.8
3	2000°F	128,900	112,600	87.7
4	2000°F	108,000	96,800	85.6
5	2100°F	113,600	106,500	89.6
6	2100°F	116,100	112,100	87.7
7	2200°F	85,600	80,500	91.0
8	2200°F	80,500	73,400	90.6
9	2400°F	50,900	40,200	91.5
10	2400°F	52,000	39,000	91.9
11	2600°F	26,500	22,000	93.8
12	2600°F	27,550	22,900	93.5

Test Conditions:
Cross Head Rate .05"/minute
Chart Speed 5"/minute
Atmosphere $< 5 \times 10^{-8}$ Torr.

FIGURE II D 1

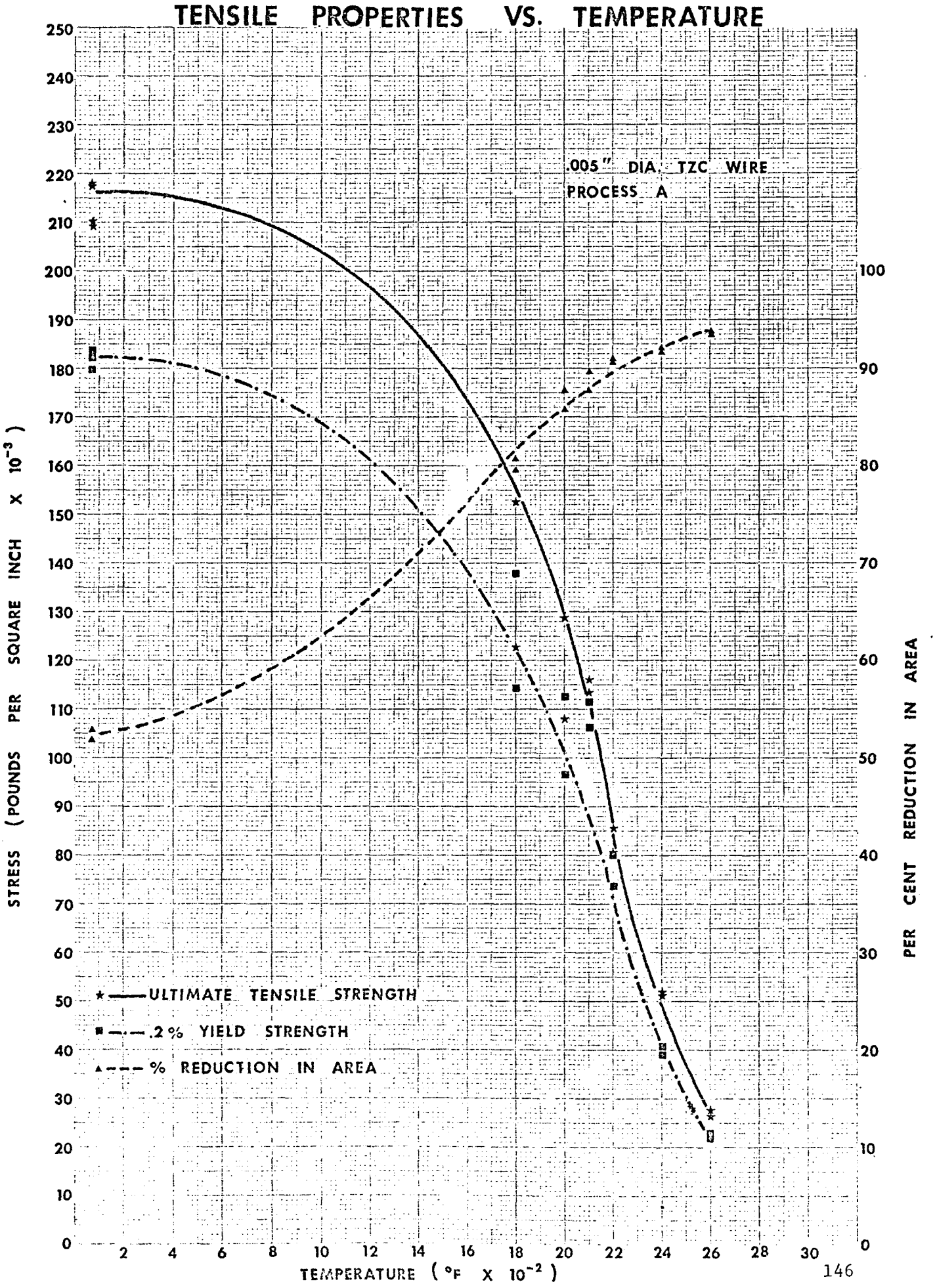


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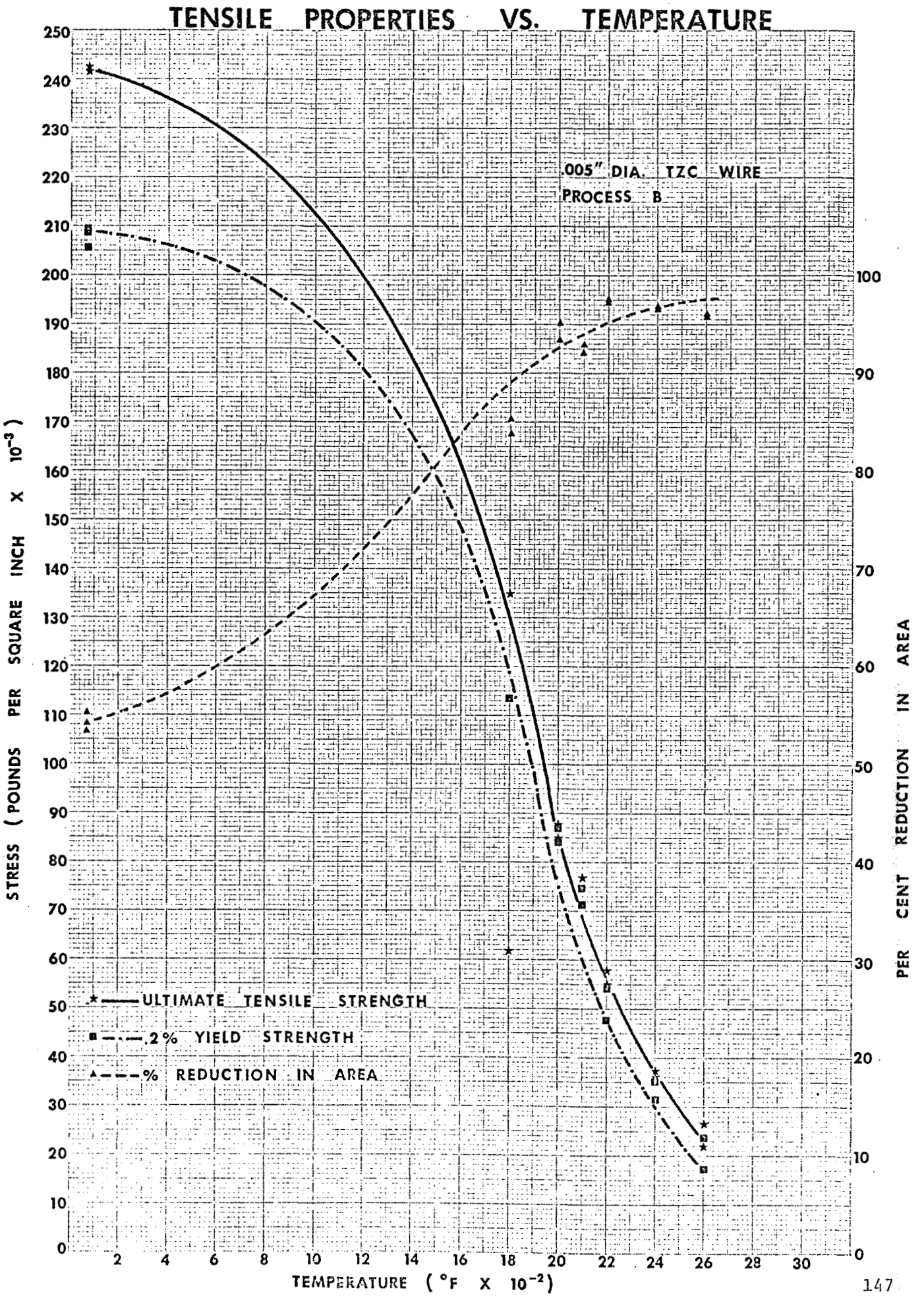
FIGURE II D 2



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AS-30 Results

Tensile Tests

Duplicate tensile tests on .005" diameter AS-30 Process A wire were performed at temperatures of 70°F, 1800°F, 2000°F, 2100°F, 2200°F, 2400°F, and 2600°F in the ultrahigh vacuum and temperature wire tensile tester. The mechanical properties, yield and ultimate tensile strengths and percent reduction in area, are shown in Figure IID3. All the tensile test data are compiled and shown in Table IIDIII.

Chemical Analysis

A one-gram sample of AS-30, .005" diameter wire, was heat treated at 2300°F for 50 hours in a vacuum atmosphere, pressure $<5 \times 10^{-8}$ Torr., and then was chemically analyzed for the interstitial elements. The results of the analysis, together with that for the "as received" sample are given below.

	Element (ppm)			
Sample Material	Oxygen	Nitrogen	Hydrogen	Carbon
"As Received"	593±59	135±14	34±5	897
Vacuum Heat Treated	320±32	67±7	1±1	806, 835

These analyses show that the oxygen and nitrogen content are decreased by about 50% after a vacuum heat treatment. The vacuum heat treatment reduces the hydrogen level to a negligible amount while the carbon content is only slightly decreased.

Table IIDIII

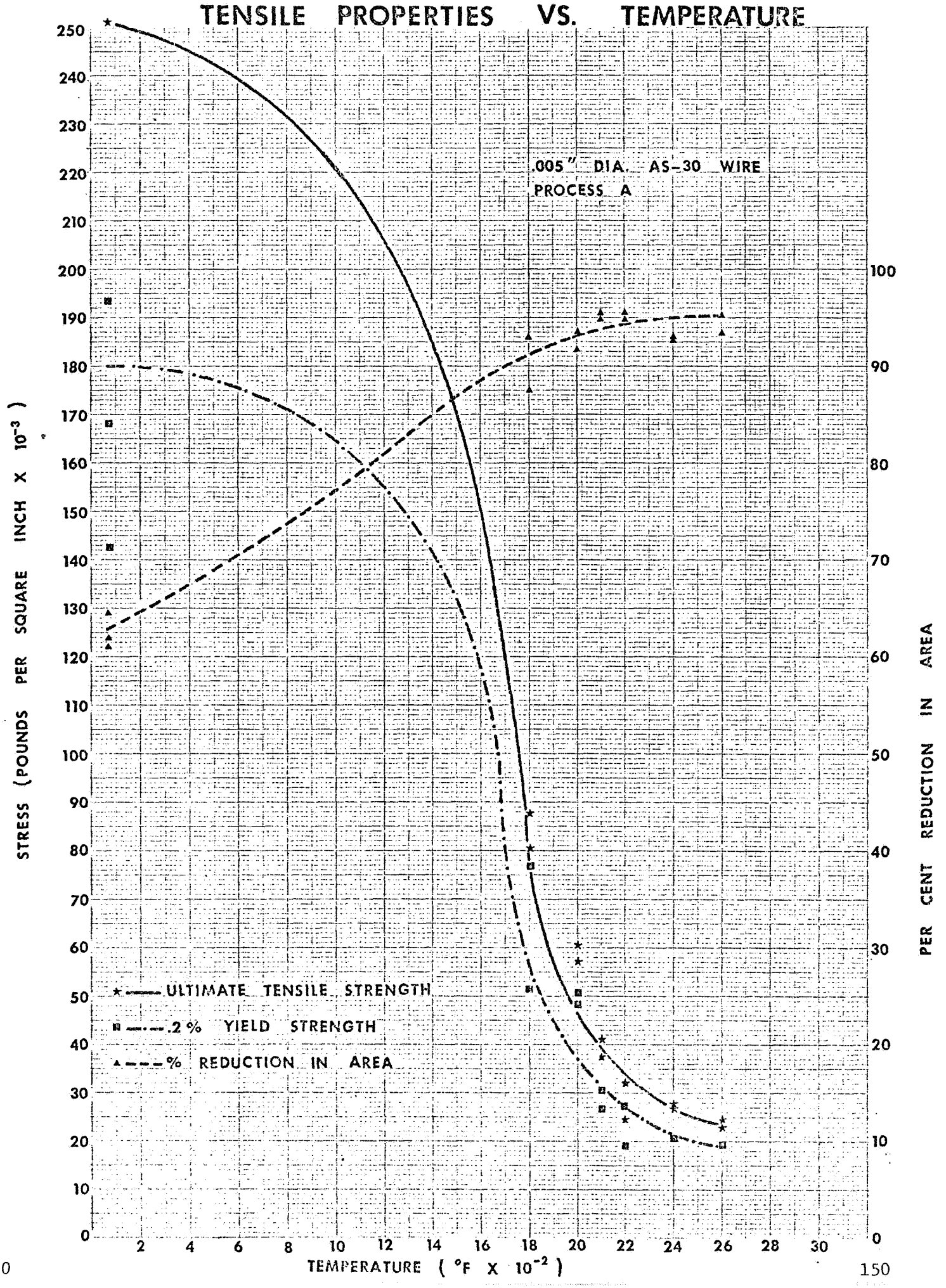
Tensile Test Data

AS-30 Process A

Sample No.	Test Temp.	Ultimate Tensile Strength, psi	.2% Yield Strength, psi	% Reduction in Area
A	70°F	251,650	142,650	64.5
B	70°F	252,150	168,100	60.9
C	70°F	252,250	193,600	61.8
1	1800°F	80,500	76,400	87.5
2	1800°F	88,650	51,450	93.0
3	2000°F	60,100	50,950	91.7
4	2000°F	57,050	48,900	93.7
5	2100°F	41,000	30,800	95.7
6	2100°F	37,450	26,750	94.8
7	2200°F	32,350	27,500	94.8
8	2200°F	24,700	18,850	95.5
9	2400°F	27,750	20,400	92.6
10	2400°F	25,900	20,900	93.0
11	2600°F	24,800	19,950	93.4
12	2600°F	22,900	19,350	95.1

Test Conditions:
Cross Head Rate .05"/minute
Chart Speed 5"/minute
Atmosphere $\leq 5 \times 10^{-8}$ Torr.

FIGURE II D 3



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FS-85 Results

Tensile Tests

Two sets of duplicate tensile tests on FS-85 .005 inch diameter wire were made at 1800°F, 2000°F, 2100°F, 2200°F, 2400°F and 2600°F. The first set of tensile tests were made on FS-85 wire fabricated by Process A and the second set on that identified as being fabricated into wire by New Process A. Room temperature (70°F) tensile evaluations were made only on Process A wire. The tensile test data for both sets of tests are given in Tables IIDIV and IIDV and Figures IID4 and IID5. Examination of the data shows that the FS-85 wire fabricated by New Process A possesses higher yield and ultimate tensile strengths at temperatures of 2100°F or higher. The ductility of New Process A wire as measured by the percent reduction in area shows more uniformity at the elevated temperatures.

Chemical Analysis

A one-gram sample of FS-85, New Process A, .005" diameter wire, was heat treated at 2300°F for 50 hours in a vacuum atmosphere, pressure $<5 \times 10^{-8}$ Torr., and then was chemically analyzed for oxygen, nitrogen, hydrogen and carbon. The results of the analyses, together with that for the "as received" sample are given below.

	Element (ppm)			
Sample Material	Oxygen	Nitrogen	Hydrogen	Carbon
"As Received"	408±41	70±7	16±5	371
Vacuum Heat Treated	461±46	32±5	1±1	67

The results show that there is a decrease of nitrogen, hydrogen and carbon after the vacuum heat treatment, with the decrease in carbon, the most significant. The oxygen content remains essentially the same, within the variance of the analysis technique.

Table IIDIV

Tensile Test Data

FS-85 Process A

Sample No.	Test Temp.	Ultimate Tensile Strength, psi	.2% Yield Strength, psi	% Reduction in Area
A	70°F	222,500	171,500	77.1
B	70°F	222,500	173,200	81.2
C	70°F	223,000	175,000	77.9
1	1800°F	58,100	55,500	93.4
2	1800°F	64,200	45,300	92.3
3	2000°F	34,100	27,000	97.1
4	2000°F	34,600	29,000	96.9
5	2100°F	23,175	15,300	92.2
6	2100°F	24,700	16,050	91.9
7	2200°F	21,750	14,250	94.7
8	2200°F	22,650	16,300	92.9
9	2400°F	18,100	13,250	97.4
10	2400°F	20,300	19,500	95.2
11	2600°F	15,300	13,250	98.8
12	2600°F	14,700	13,150	98.1

Test Conditions:
Cross Head Rate .05"/minute
Chart Speed 5"/minute
Atmosphere $< 5 \times 10^{-8}$ Torr.

Table IIDV

Tensile Test Data

FS-85 New Process A

Sample No.	Test Temp.	Ultimate Tensile Strength, psi	.2% Yield Strength, psi	% Reduction in Area
A	70°F	222,500*	171,500*	77.1*
B	70°F	222,500*	173,200*	81.2*
C	70°F	223,000*	175,000*	77.9*
1	1800°F	60,100	46,900	94.5
2	1800°F	57,600	56,100	94.9
3	2000°F	36,700	30,600	96.4
4	2000°F	31,000	27,000	95.8
5	2100°F	29,250	23,800	96.7
6	2100°F	28,900	23,400	96.7
7	2200°F	25,500	21,700	97.9
8	2200°F	27,900	21,900	97.4
9	2400°F	24,500	19,200	97.4
10	2400°F	26,400	25,300	96.9
11	2600°F	20,900	18,950	97.7
12	2600°F	20,500	17,400	98.1

Test Conditions:
Cross Head Rate .05"/minute
Chart Speed 5"/minute
Atmosphere $<5 \times 10^{-8}$ Torr.

* These data are taken from the 70°F test for FS-85 Process A.

FIGURE II D 4

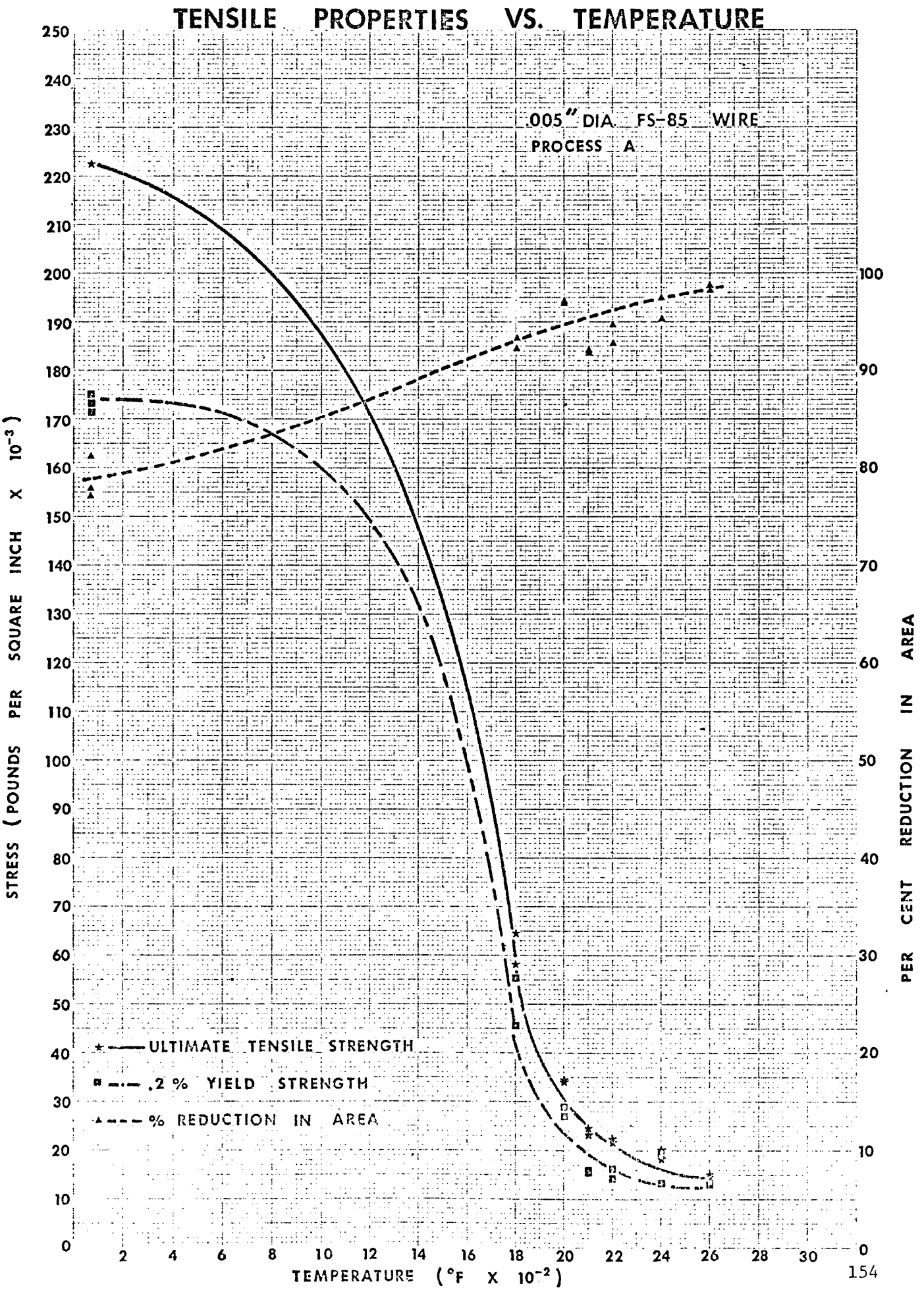

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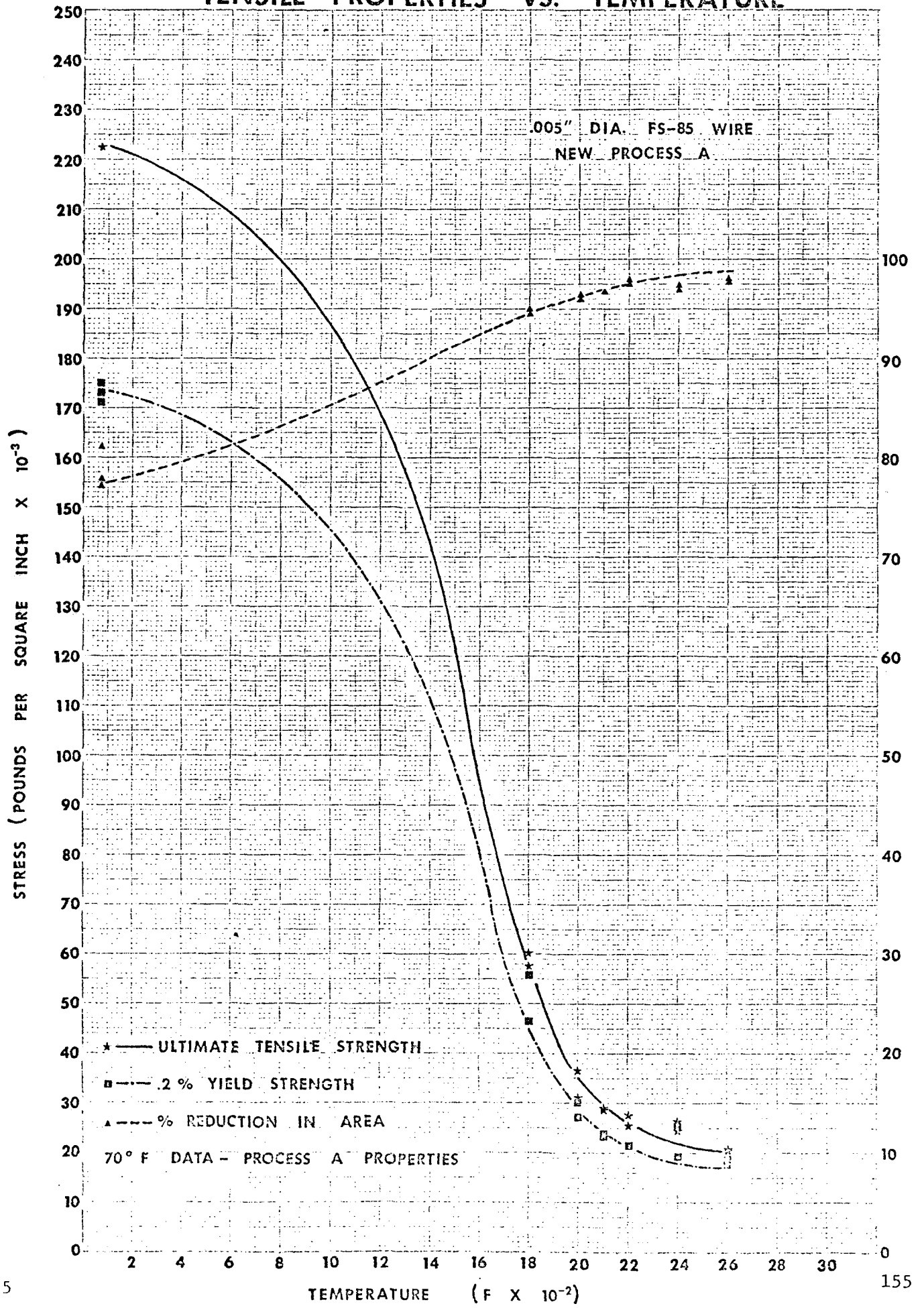
FIGURE II D 5

TENSILE PROPERTIES VS. TEMPERATURE



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Tantalum Base Alloy - T-222A Results

Tensile Tests

Tensile tests on .005" diameter wire, identified as T-222A, were made at 70°F, 1800°F, 2000°F, 2100°F, 2200°F, 2400°F, and 2600°F. At 70°F (room temperature) three tensile tests were run. At all other temperatures, two tensile tests were run. All the tensile test data are given in Table IIDVI and the mechanical properties as a function of the test temperature are graphically represented in Figure IID6.

Chemical Analysis

A one-gram sample of T-222A, .005" diameter wire, was heat treated at 2300°F for 50 hours in a vacuum atmosphere, pressure $<5 \times 10^{-8}$ Torr., and then was chemically analyzed for the interstitial elements. A one-gram sample of T-222A in the "as received" condition was also analyzed. The results of the two analyses are given below.

	Element ppm				
				Carbon	
Sample Material	Oxygen	Nitrogen	Hydrogen	1st	2nd
"As Received"	256±26	27±5	49±5	171	165
					185
Vacuum Heat Treated	220±22	18±5	1±1	189	204
				206	219

The results of the analyses show no increase or decrease in oxygen and nitrogen, a decrease in hydrogen and a pick-up of approximately 30 ppm carbon. Only about half of the carbon pick-up (15 ppm) can be attributed to the inaccuracies of the measurement technique.

The first carbon analysis of the "as received" wire was analyzed before the vacuum heat treatment of a wire sample was done. The results of the first carbon analysis of the vacuum heat treated wire indicated that it might be beneficial to re-examine the carbon content of two additional wire samples. The second determination for carbon on an "as received" and a vacuum heat treated sample was done on the same day. These results confirmed those of the first analyses. A possible explanation for the increase in carbon after a vacuum heat treatment is that the vacuum heat treated sample of T-222 experienced two evacuations and bake-out cycles before the final one where the heat treatment at 2300°F was achieved.

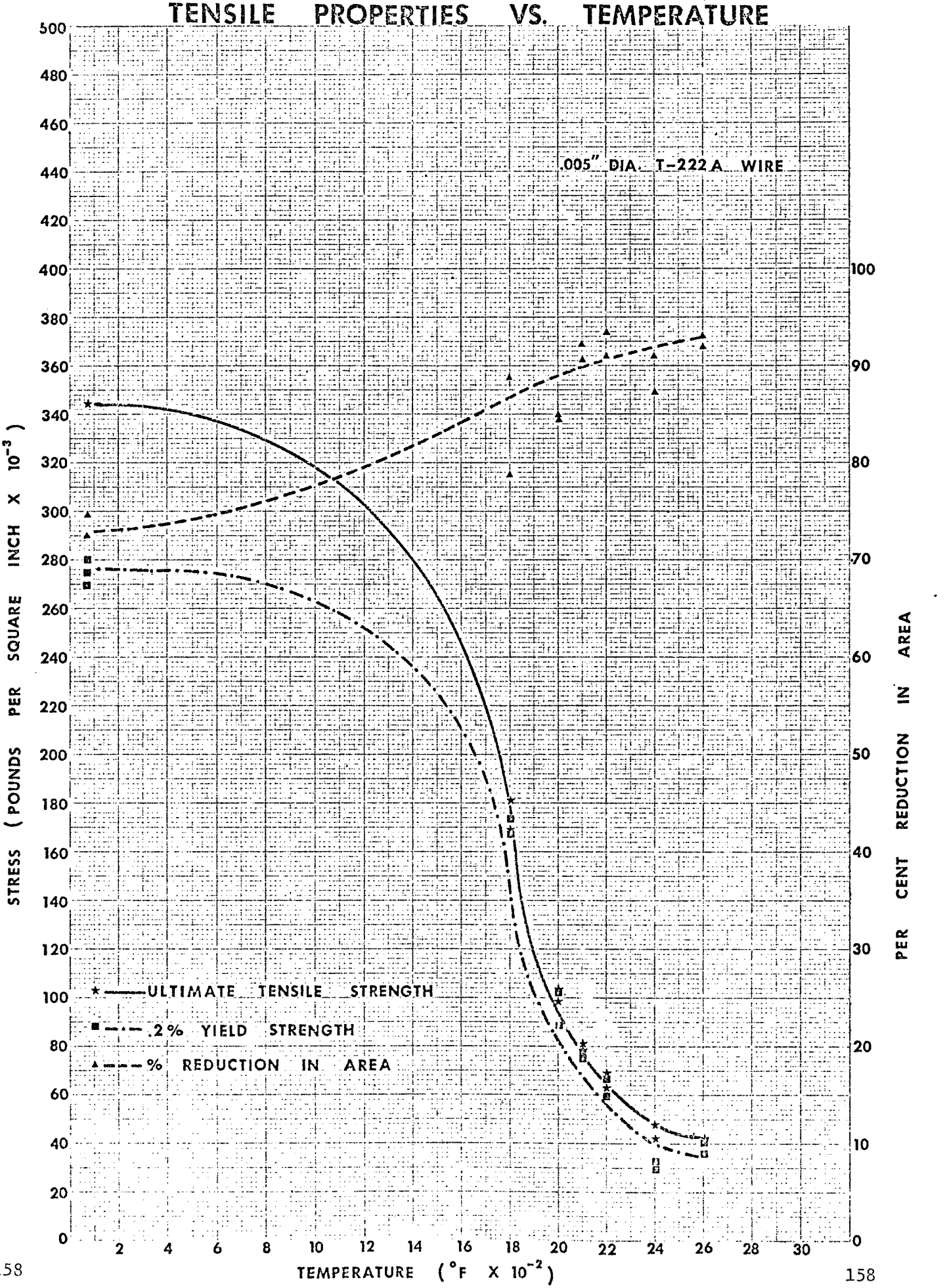
Table IIDVI

Tensile Test Data
T-222A

Sample No.	Test Temp.	Ultimate Tensile Strength, psi	.2% Yield Strength, psi	% Reduction in Area
A	70°F	343,900	280,200	74.6
B	70°F	343,900	270,000	72.4
C	70°F	343,900	275,100	74.6
1	1800°F	180,850	173,200	88.7
2	1800°F	169,600	167,100	78.9
3	2000°F	104,400	103,400	84.5
4	2000°F	98,800	88,600	85.0
5	2100°F	81,000	74,900	92.3
6	2100°F	78,500	75,900	90.6
7	2200°F	69,800	67,200	93.5
8	2200°F	63,200	60,100	91.0
9	2400°F	48,100	33,660	91.0
10	2400°F	48,100	29,800	87.2
11	2600°F	42,500	42,300	93.1
12	2600°F	41,100	36,170	91.9

Test Conditions:
Cross Head Rate .05"/minute
Chart Speed 5"/minute
Atmosphere $< 5 \times 10^{-8}$ Torr.

FIGURE II D 6



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218 Tungsten Results

The testing of tungsten wire was not originally planned for in the contract but was performed (per an amendment to the contract) in an attempt to resolve some questions concerning the creep resistance of the alloy wires when tested in a vacuum atmosphere of $<5 \times 10^{-8}$ Torr. pressure.

Tensile Tests

No elevated tensile tests were performed on this wire material.

Stress-Rupture Tests

Stress-rupture tests on four 218 tungsten wire samples, unetched, were performed with two samples tested in a vacuum atmosphere of $<5 \times 10^{-8}$ Torr. pressure and two in an argon atmosphere. The results of these tests were somewhat conflicting and will also be presented in a future report.

Chemical Analysis

A one-gram sample of 218 tungsten, .004" diameter wire, was heat treated at 2300°F for 50 hours in a vacuum atmosphere, pressure $<5 \times 10^{-8}$ Torr., and then was chemically analyzed for the interstitial elements. A similar sample, in the "as-received" condition, was also analyzed. The results of these analyses are given below.

	Element (ppm)			
Sample Material	Oxygen	Nitrogen	Hydrogen	Carbon
"As Received"	114±11	16±5	9±5	147
Vacuum Heat Treated	13±5	1±1	1±1	52

The interstitial analyses show a decrease in all elements as a result of the vacuum heat treatment. The carbon content of 147 ppm for the 218 tungsten wire in the "as-received" condition is abnormally high, since traditionally and historically it is well below 50 ppm and the normal range is 4-15 ppm. In similar instances where a high carbon content was reported for 218 tungsten wire, we have learned that an HCl rinse drastically reduced the level, indicating there was an adsorbed carbonate on the surface. This carbonate film is the result of poorly rinsed wire which was cleaned in a caustic solution. This plus the fact that the wire tested had been stored for a long period of time and could have adsorbed a carbonate film on its surface probably account for the unusually high initial carbon content. The fact that 52 ppm remain after the vacuum heat treatment is interesting, but further investigation of this was not possible.

Table IIEI

Density of Alloy Wires

Material	Density (lbs./in.^{3})(calculated)
Mo-TZC Process B	.356
AS-30 Process A	.347
FS-85 New Process A	.383
T-222A	.604

Table IIEII

Average Ultimate Tensile Strength/Density Ratio (Inches)

Material	Ratio at Temperature (°F) 2000	2100	2200
Mo-TZC Process B	287,450	278,700	201,550
AS-30 Process A	143,600	96,200	69,950
FS-85 New Process A	78,300	66,300	60,800
T-222A	148,400	116,450	97,100

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Many people were active participants in, or made significant contributions to, the tasks involved in this contract. We wish to acknowledge their help and identify the areas benefited by their efforts.

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Finally the authors wish to thank Dr. C. A. Brown whose contributions throughout the contract were especially appreciated.

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