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FABRICATION OF THE PRTR ZIRCALOY-2 HIGH PRESSURE PROCESS TUBES

R. L. KNECHT

JULY 31, 1959

HANFORD LABORATORIES

HANFORD ATOMIC PRODUCTS OPERATION
RICHLAND, WASHINGTON

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By

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TABLE OF CONTENTS

FABRICATION OF THE PRTR ZIRCALOY-2 HIGH PRESSURE PROCESS TUBES

I. INTRODUCTION

The Plutonium Recycle Test Reactor (PRTR) under construction at Hanford will have high pressure process tubes of Zircaloy-2 passing through the reactor core contained in a low pressure moderator tank. The process tubes are unique as a reactor component because of their shape and because they are designed to operate in-reactor at a high temperature and pressure (542 F, 1050 psig).

For the critical service in the reactor, it is necessary that the tubes be fabricated to close dimensional tolerances, be of sound structural integrity and possess good corrosion resistance. However, at the time the pressure tube was designed, no tubes of a comparable size and shape had been previously formed in Zircaloy.

Under the sponsorship of Hanford, commercial tube vendors undertook the development of a fabrication process to form the process tube. The successful development of a fabrication process and the production of 95 PRTR high pressure process tubes are described in this document.

II. SUMMARY AND CONCLUSIONS

Initial development efforts by commercial vendors to fabricate the PRTR process tubes were started in January of 1957. These efforts were concentrated on forming or attaching a flange on one end of each tube and tapering or pointing the other end into a smaller diameter with a thicker wall than the rest of the tube.

By August of 1958, four tubes had been successfully formed. However, the tubes did not possess the close dimensional tolerances needed nor the physical integrity required. At this point, the Tube Reducing Corporation proposed to fabricate the tubing by combining the best features from the separate development programs and by continuing development during production of the tubing. They were contracted for the production and guaranteed delivery of 95 to 105 tubes.

Ninety-five tubes out of a possible 138 were produced. Development work during the production order resulted in the elimination of one heat treating step and one fabrication step. The first large scale use of billet piercing to produce Zircaloy tubes was implemented to increase metal yield. A new method of attaching the flange was developed. Reflecting the development work and the refinement of processing techniques, 72 acceptable tubes were produced from the last 82 pieces processed compared to 23 good tubes from the first 56 processed.

Tubes were accepted from the vendor after passing exacting tests and inspections including borescoping of the inside surface and ultrasonic measurement of the wall thickness. Additional nondestructive tests including fluorescent penetrant and ultrasonic flaw detection are being performed at Hanford. The results of these tests will be discussed in a later report.

Through development of a fabrication process and the manufacture of 95 PRTR process tubes, it has been demonstrated that the commercial fabrication of high quality Zircaloy-2 pressure tubing is feasible.

III. NEW AREAS IN FABRICATING ZIRCALOY-2

The size and shape of the PRTR process tube is illustrated in Figure 1. A detailed tube drawing and specification are given in Appendix A.

At the time the tube design was completed, there was no commercially available and proven fabrication process for the manufacture of this tube in Zircaloy-2. The initial process steps, however, involved the use of conventional practices: producing ingots by double vacuum arc melting of consumable electrodes, reduction of the ingots by forging and, the preparation of hollow billets for extrusion by boring and machining. Refinements in extrusion were needed to minimize eccentricity and to provide smooth surfaces free of contamination from jacketing materials.

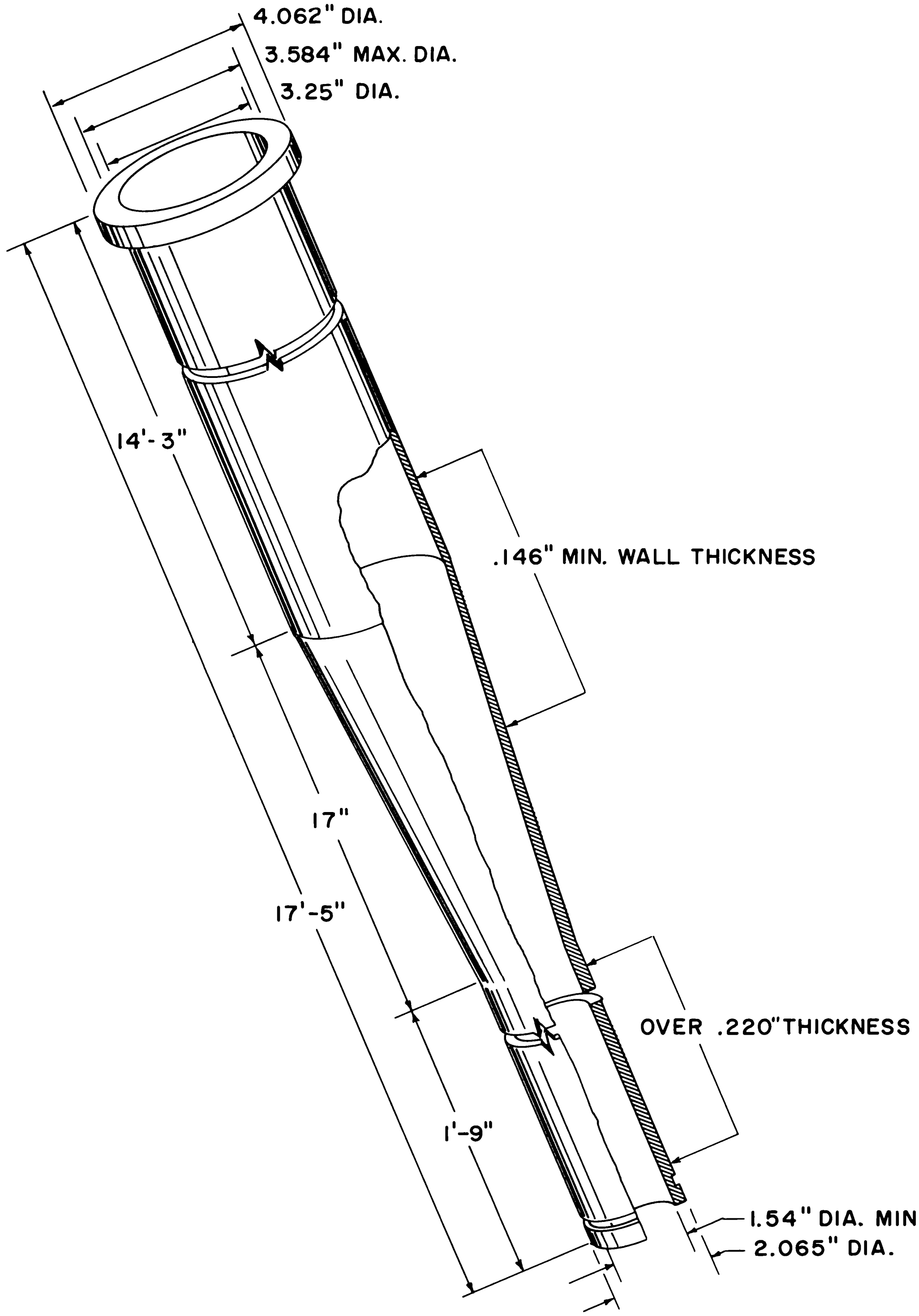

FIGURE 1

The PRTR Process Tube with Nominal and Approximate Dimensions

To form the hollow extrusion to the final shape of the PRTR process tube, two major metal forming problems had to be solved. First, one end of the tube had to be pointed or tapered into a smaller diameter with a thicker wall than the rest of the tube; and second, a flange had to be formed or attached on the other end of the tube.

The fabrication approaches were limited by the tight dimensional tolerances and the sound physical integrity required in the finished tube. Essentially no atmospheric contamination of the metal could be allowed because of the deleterious effect on the corrosion resistance of Zircaloy.

IV. INITIAL FABRICATION TRIALS

The initial attempts to fabricate the process tube were made under best effort contracts with the Chase Brass and Copper Company in Waterbury, Connecticut and the Allegheny Ludlum Steel Corporation in Watervliet, New York. Also, Le Fiell in Los Angeles was contracted for development work on tapering and flanging tube sections.

Hollow Zircaloy-2 billets jacketed in copper with a steel interliner were extruded at a temperature of about 1550 F by Chase. The OD of the extrusion was that required for the large diameter part of the tube and the wall thicknesses were approximately those needed for the finished tube. The thicker wall for the tapered portion of the tube was obtained by advancing a tapered mandrel through the die opening as the tube was extruded. The extrusions were only slightly eccentric and the surfaces were of good quality except for a slight wavy characteristic on the inner surface.

Tapering by swaging was attempted by Fenn Mfg. Company under a subcontract. The swaging operation was conducted on tubes at room temperature and without internal support. Results of this work indicated that tapering by this technique would result in metal failure and severe wrinkling of the inner surface. For flanging, Chase attempted to develop an inert atmosphere welding technique for depositing Zircaloy-2 filler wire on the ends of the tube sections; the deposited metal was subsequently machined to flange dimensions. Visual inspection of the welds, the machined flange and the tube sections, indicated there was little if any contamination of the Zircaloy by the air or other foreign material. However, the deposited metal did contain some porosity and there was local inward distortion of the tube sections. The distortion was caused by thermal expansion resulting from the application of the welding heat on the outside of the tube.

Allegheny Ludlum hot extruded unclad Zircaloy-2 billets using glass as a preheating and lubricating medium-a technique similar to their standard practice for stainless steel. With no copper jacketing or steel interlining, the possible formation of eutectic between the copper or iron and Zircaloy was avoided. Difficulty was encountered in minimizing eccentricity during extrusion and the glass lubricant left surface impressions on the extruded tubes.

Rockrite* or rocking reductions were to be used to form the extrusion to final size and shape. The initial rocking produced the large diameter, thin wall section of the tube with a reduction in diameter and wall thickness. The outer diameter of that part of the tube to be tapered was also reduced but the wall thickness was increased by free sinking. After vacuum annealing at Allegheny Ludlum, tapering or pointing was attempted with a single rocking operation. The tube cracked or broke up. However, tubes were successfully tapered in two rocking or tube reducing steps with an intermediate vacuum anneal. In both reductions, mandrels were used to provide limited support of the inner surface while the tube was sunk.

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* In the Rockrite process, semi-circular, tapered groove dies are rocked back and forth over the specially conditioned seamless tubing stock to compress the metal over the correspondingly polished, tapered mandrel which controls inner diameter. The outside diameter, the inside diameter, and the wall thicknesses are all reduced as the length of the tube is increased. Rockrite Cylinder-Finish Tube, Brochure, Tube Reducing Corporation, Wallington, New Jersey.

With flange attachment, Allegheny Ludlum finished the only completed tubes produced under the development contracts. Grooved rings were welded to the tube for flange attachment. The rings had two circumferential grooves located on the outer surface. These grooves were filled by depositing Zircaloy-2 filler wire by arc welding in an inert atmosphere; sufficient penetration was provided to secure the rings to the tubes. The final flange dimensions were obtained by machining through the filler welds and ring. Machining exposed porosity in the weld metal and there was local inward distortion of the tube as in the Chase tubes.

Le Fiell used hot swaging to form tapers on tube sections. Initial trials with the Zircaloy at room temperature resulted in metal failure. However, by preheating, the taper could be swaged but excessive nitrogen contamination occurred and there was severe wrinklng of the inner surface. Thompson Products subcontracted to flange tube sections by machining metal gathered from the tube wall by a combination of resistance heating and pressure. The metal gathering was successful but surface contamination with nitrogen occurred.

V. THE FABRICATION OF 95 PRTR PROCESS TUBES

A. Proposed Fabrication Process for the Production Tube Order

Four completed tubes were produced by development efforts. The tubes, however, did not meet the close dimensional tolerances or the physical integrity required. Improvement of tube quality was needed in the following areas:

1. Surface Finish. The outer surfaces contained defects resulting from die pickup in tube reducing and others which originated in extrusions; the inner surface also contained defects which originated in extrusion and the inner surface of the tapered portion exhibited some wrinkling and cracking.

2. Concentricity. The tube walls were eccentric.

3. Flanges. The weld metal of the flanges contained porosity and there was local tube distortion.

At this time, the Tube Reducing Corporation of Wallington, New Jersey was confident that enough information and experience had been gained by the development efforts to produce acceptable tubes. It was proposed to utilize the progress in extrusion quality achieved by Chase under the development contract. Eccentricity in the extrusions had been held to low levels and the copper cladding with steel interlining of the billets had provided extrusions of good surface quality. The development work on tube reducing indicated the tooling corrections necessary to avoid die pickup. Further, development work indicated that with the redesign of tooling to provide more internal support, cracking and wrinkling of the inner surface during tapering could be minimized or eliminated. Development work on flange attachment was to continue concurrently with fabrication of the tubes. Efforts were to be concentrated on flanging by slip fitting or threading a ring on the tube end and securing by welding in an inert atmosphere.

Based upon these proposals, a maximum price contract with the Tube Reducing Corporation for the guaranteed delivery of 95-105 PRTR Zircaloy-2 pressure process tubes was negotiated in the Fall of 1958. The process was to consist of the following major steps:

1. Ingot forging with subsequent boring or piercing to obtain the extrusion billets. (Ingots were provided byHanford under the contract.)
2. Hot extrusion of copper clad and steel interlined billets.
3. Tube reducing to produce the large diameter, thin wall section of the tube leaving a heavy wall thickness on one end.
4. Vacuum anneal.
5. Tube reducing to form part of the taper and to obtain a smaller diameter and reduce the wall thickness on the bottom of the tube.
6. Vacuum anneal.
7. Tube reducing to complete the taper and to obtain the finished diameter and wall thickness on the bottom of the tube.
8. Flange attachment.
9. Final inspection.

B. Billet Preparation

Ingots of 12-inch nominal diameter weighing approximately 1000 pounds were cast from AEC allocated reactor grade zirconium sponge at the Allegheny Ludlum plant in Watervliet, New York. Casting was by double vacuum arc melting of consumable electrodes. The ingots were machine conditioned and shipped to the Heppenstall Company, Bridgeport, Connecticut for forging on a 500-ton steam-hydraulic press. Seven ingots were reduced to an 8-inch diameter, cut machined and bored to provide 35 hollow extrusion blanks. The remaining 15 ingots were reduced to a 7-inch diameter, rough machined and cut for subsequent piercing and machining to extrusion blank size.

This was to be the first large scale use of upsetting and piercing in the fabrication of Zircaloy tubing. It provided a marked reduction in the metal required per tube; boring the hole in extrusion blanks required 27 per cent more starting ingot material than that required by upsetting and piercing.

Upsetting and piercing was performed by Chase on a horizontal hydraulic press of 2200 tons capacity on the main ram and 295 tons on the piercing ram. The billets were heated to 1600 - 1650 F in a gas-fired, car bottom type furnace with three separately controlled temperature zones. Dies were preheated to approximately 500 F. Lubrication on the mandrel and dies was by graphite and tallow. Figure 2 shows an over-all view of the press.

The piercing operation was successful. Difficulty was initially experienced in obtaining the correct ram alignment with the result that some of the as-pierced billets were slightly eccentric. However, this eccentricity was eliminated with clean-up of the surfaces during machining to the extrusion blank dimensions. After machining to the OD required for the extrusion blank, the surface of the hole was machined concentric with the outer surface. Clean-up of the surfaces was good except for traces of laps or other inner surface irregularities on approximately 12 pieces.

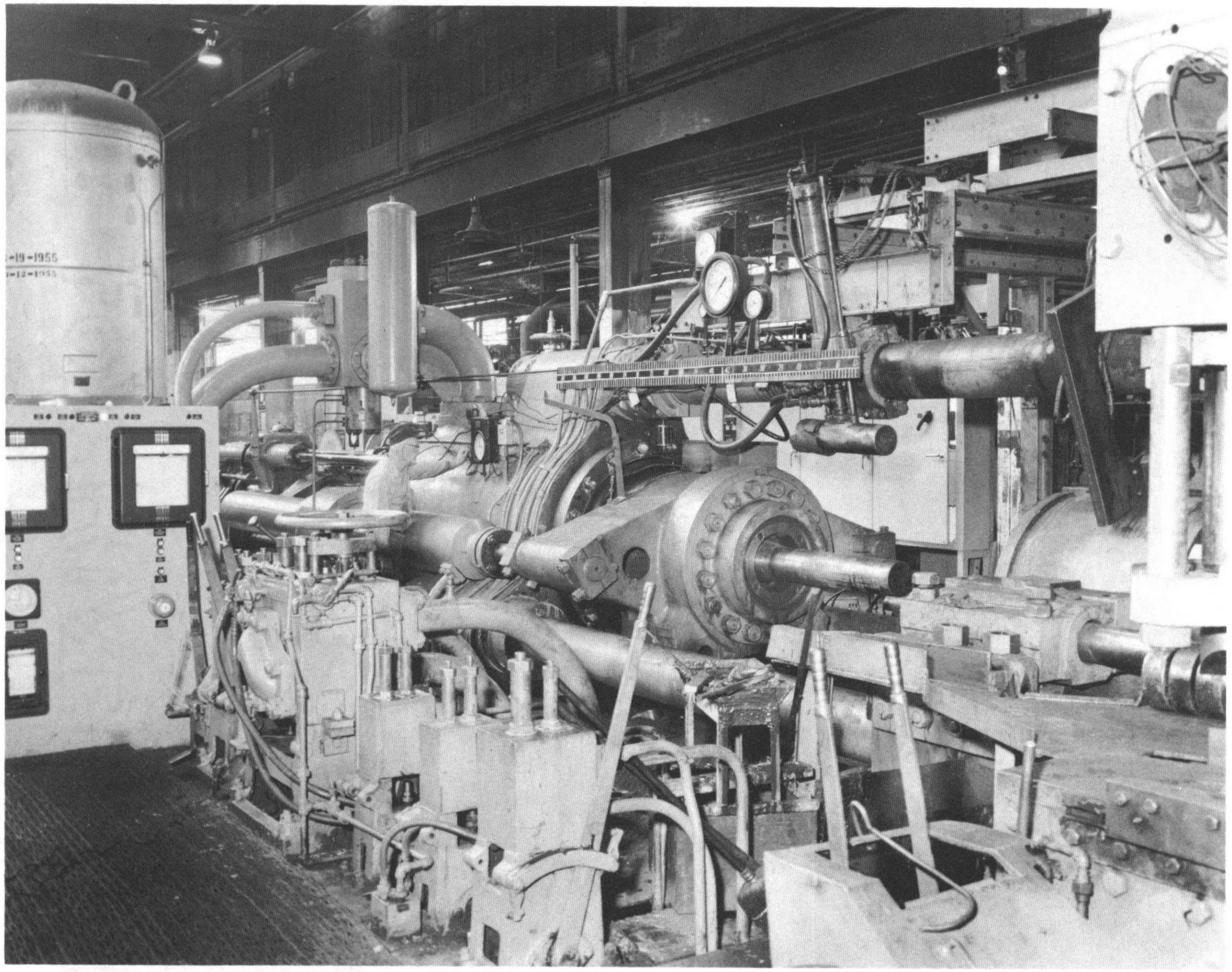

FIGURE 2

Extrusion Press Used for the PRTR Tubes
(Photo Courtesy of the Chase Brass and Copper Co.)

C. Hot Extrusion

The extrusions were made by the Chase Brass and Copper Company at their Waterbury, Connecticut plant. The 3-7/8 ID x 7-3/4 OD x 14-1/2-inch long billets were jacketed in copper with a steel interliner to prevent eutectic formation between the copper and Zircaloy. The jacketing provided protection from contamination by the air, surface lubrication for the extrusion, and a means of maintaining good surface quality during extrusion. The billets and graphite dummy blocks were normally preheated for 2-1/2 hours at 1500-1600 F in the same gas-fired furnace used in the piercing operation. The dies were of a conical entrance type with a 110 degree included angle and were preheated to 500 F. The dies and container were lubricated with graphite and tallow. Extrusion was on the same horizontal hydraulic press used for piercing. The extrusion ratio was approximately 14 to 1 with extrusion pressures around 2000 tons normally encountered. A total of 138 extrusions were made in groups of 35, 51 and 52. The first three extrusions were excessively eccentric. Alignment was corrected and the remaining 135 blanks were extruded with good concentricity. Three billets did fail to push in the first group of 35; however, these were successfully extruded with the second batch of billets. Figure 3 pictures a tube (not Zircaloy) emerging from the press.

Further processing consisted of: air cooling; cropping for stamping the tube number; removal of jacketing by pickling in a 40 - 50 per cent nitric and 1 - 2 per cent hydrofluoric acid solution; rinsing in hot and cold water; straightening; inspection for surface defects; cropping; clean-up of the outer surface by centerless belt grinding; borescoping; clean-up of inner surface with abrasive belts on wheels; vidigaging for wall thickness; degreasing in hot alkaline cleaner of trisodium phosphate base; and, a final pickle in nitric-hydrofluoric acid and rinsing in water before shipment.

The nominal size of the extrusions produced was 4-1/4 inch OD x 3-3/4 inch ID x 13 feet long. As measured by vidigage, the eccentricity of the first three billets pushed was greater than plus or minus 8 per cent. These were scrapped. The eccentricity of the other 135 extrusions was less than plus or minus 6 per cent with most of these being in the range of plus or minus

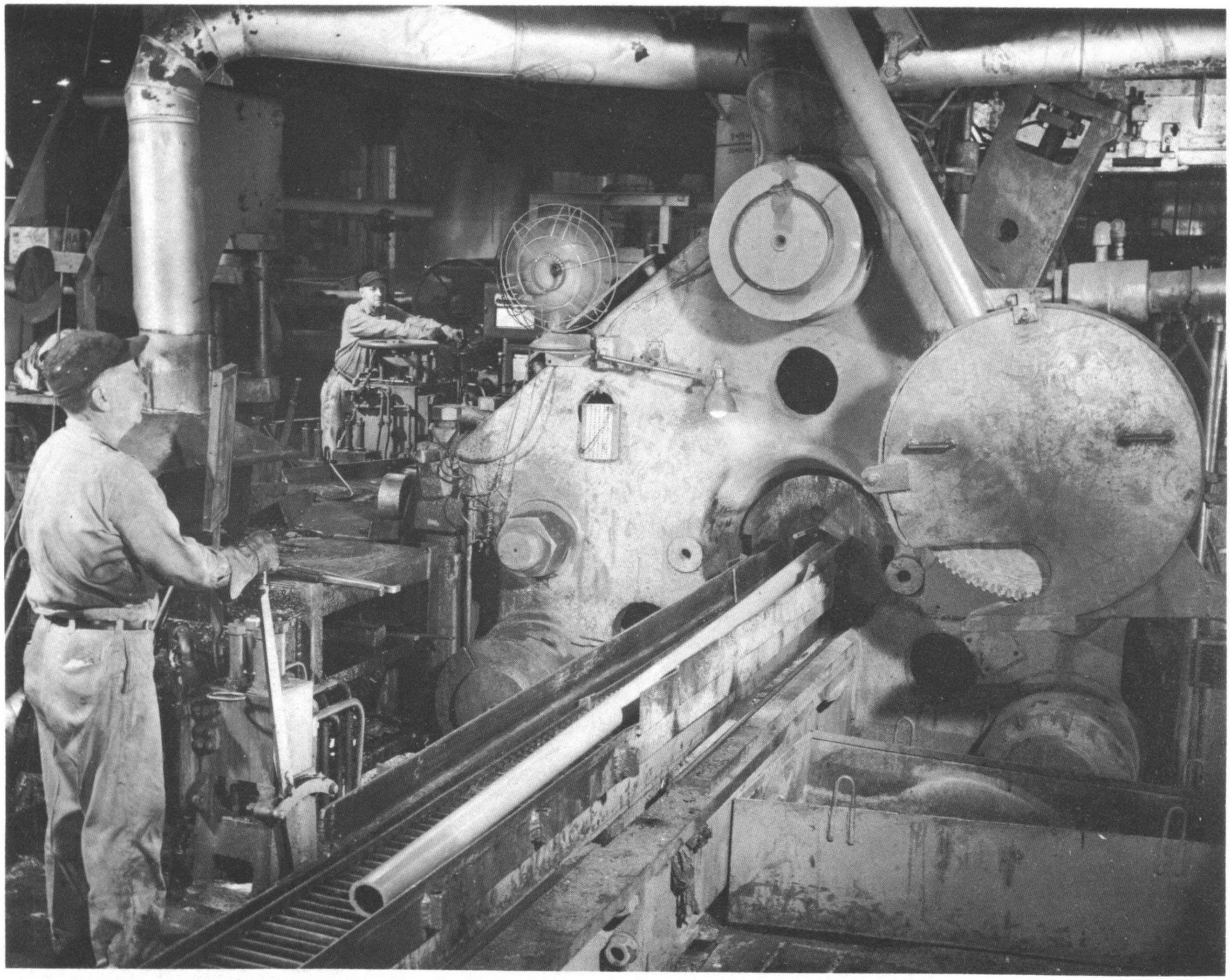

FIGURE 3

An Extrusion Emerging from the Press
(Photo Courtesy of the Chase Brass and Copper Co.)

2-1/2 to 4-1/2 per cent eccentric. Total ovalness was in the range of 0.003 to 0.012 inch. In general, the quality of the extruded surfaces was excellent. Laps were located on the inner surfaces of approximately 12 extrusions and were removed by grinding. Figure 4 shows the inner surface of an extrusion.

D. First Tube Reduction

To obtain the close dimensional tolerances and good surface finishes afforded by the Rockrite process, it is necessary that feed material of high quality be used. The extrusions made by the Chase Brass and Copper Company provided this for the PRTR tubes. To maintain tube quality and to successfully perform the tube reductions, it was necessary to inspect and condition - when required - before each rocking operation. Surface condition is of particular importance since small cracks or laps tend to propagate during the rocking reduction and cause metal failure.

The outer surfaces of the PRTR tubes were visually inspected and when necessary were wet belt ground, hand ground or filed to remove defects. The inner surfaces were inspected with a borescope and any defects found were removed by blasting with fine white sand.

Vidigage measurements of wall thickness were made to determine if there were any wall areas that might be difficult to form properly. The internal diameters were checked with a plug gage to insure correct mandrel fit. The outer diameters were measured for proper die fit. If present, ovality and bow were removed or minimized by rotary or press straightening. The tubes were cleaned before rocking in a hot alkali bath and rinsed in water. Commerical chlorinated oils were used as lubricants on the mandrels and dies during reduction. After rocking, the tubes were cleaned with naphtha on the outer surface, then submerged in an alkali bath, and subsequently rinsed in water.

The first tube reduction was performed in two steps. A reduction of 45 - 50 per cent formed the large diameter portion of the finished tube, i. e., the finished dimensions of that part of the tube were produced and no

FIGURE 4

Inner Surface of an Extrusion (3X)

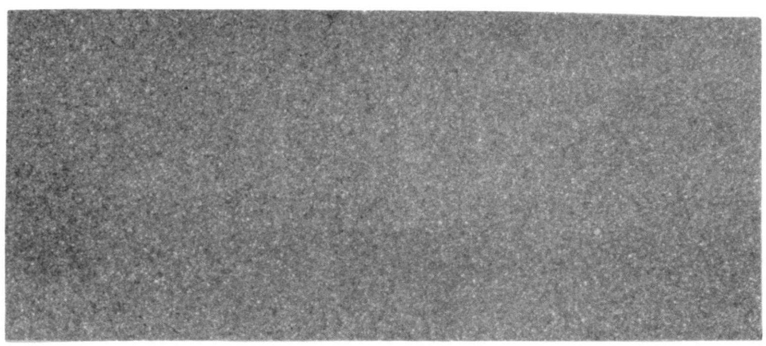

FIGURE 5

Inner Surface of the Tube after the First Tube Reduction and Sand Blasting (3X)

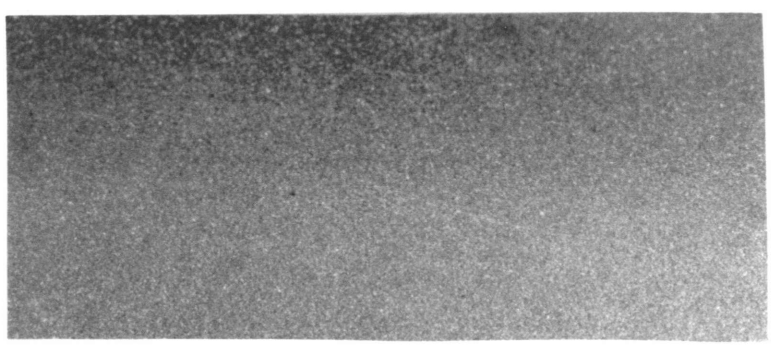

FIGURE 6

Inner Surface of the Tube after the Tapering Operation and Sand Blasting (3X)

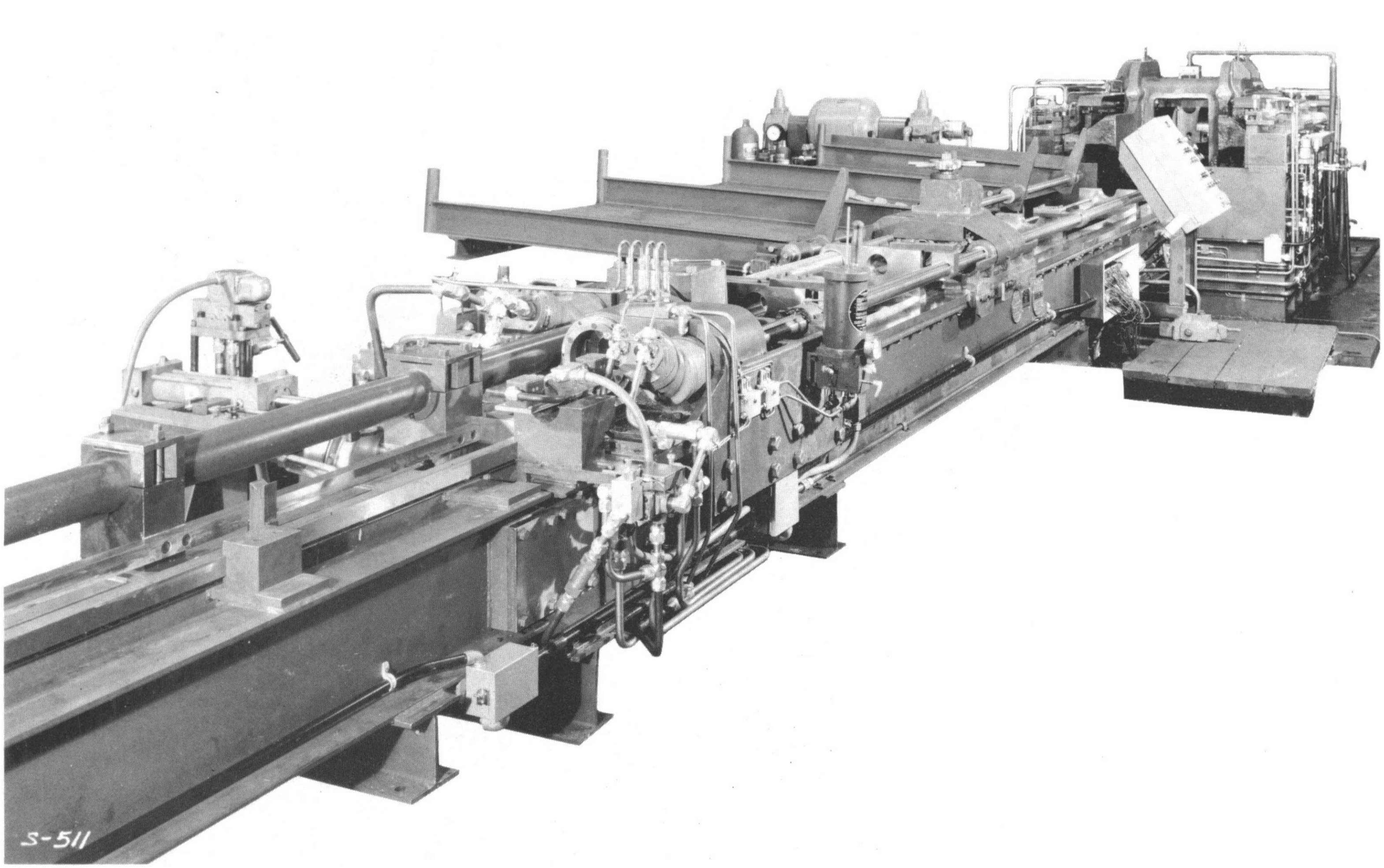

FIGURE 7

A Tube Reducing Machine
(Photo Courtesy of the Tube Reducing Corporation)

further working of the metal was required. The remaining portion of the tube was then reduced by 25 per cent using the same dies to produce the outside diameter but a different mandrel to maintain a thicker wall. Figure 7 pictures a tube reducing machine while Figure 8 illustrates the rocking dies working on a piece of tubing with a mandrel furnishing internal support.

Out of 135 extrusions, only two failed during the first tube reducing. The failures were caused by a binding mandrel on undersize ID tubes. The surfaces were of good quality and a gradual transition in wall thickness from the light to the heavy wall section was attained.

E. Vacuum Annealing

The tubes were vacuum annealed at the Allegheny Ludlum plant in Watervliet, New York. A 14-inch horizontal tube continuously gas-fired furnace with a 20-foot hot zone and a 20-foot cold zone was used. For the initial annealing runs, the tubes were put in stainless steel sleeves to minimize possible handling damage and to provide even support. These sleeves were loaded into a semi-circular stainless steel basket for insertion into the furnace.

Annealing was at 1450 F with an estimated time at temperature for the tubes of two hours. The vacuum typically was less than one micron at the start of heating, rose to seven microns during heating, and returned to less than one micron at approximately the time the tubes reached the annealing temperature.

Seven tubes were thus annealed. The tubes bowed as much as 6 inches and became oval up to 0. 060 inch. Several of the tubes were bound tight in the stainless steel sleeves and were marred when removed.

The distortion of the tubes evidently resulted from uneven heating or from stress relief. Several actions were taken to minimize or eliminate this distortion. To provide more even radiant heating, the stainless steel sleeves were eliminated and dimpled stainless steel sheets were placed over the top of the semi-circular basket. Only the end of the tube to be further

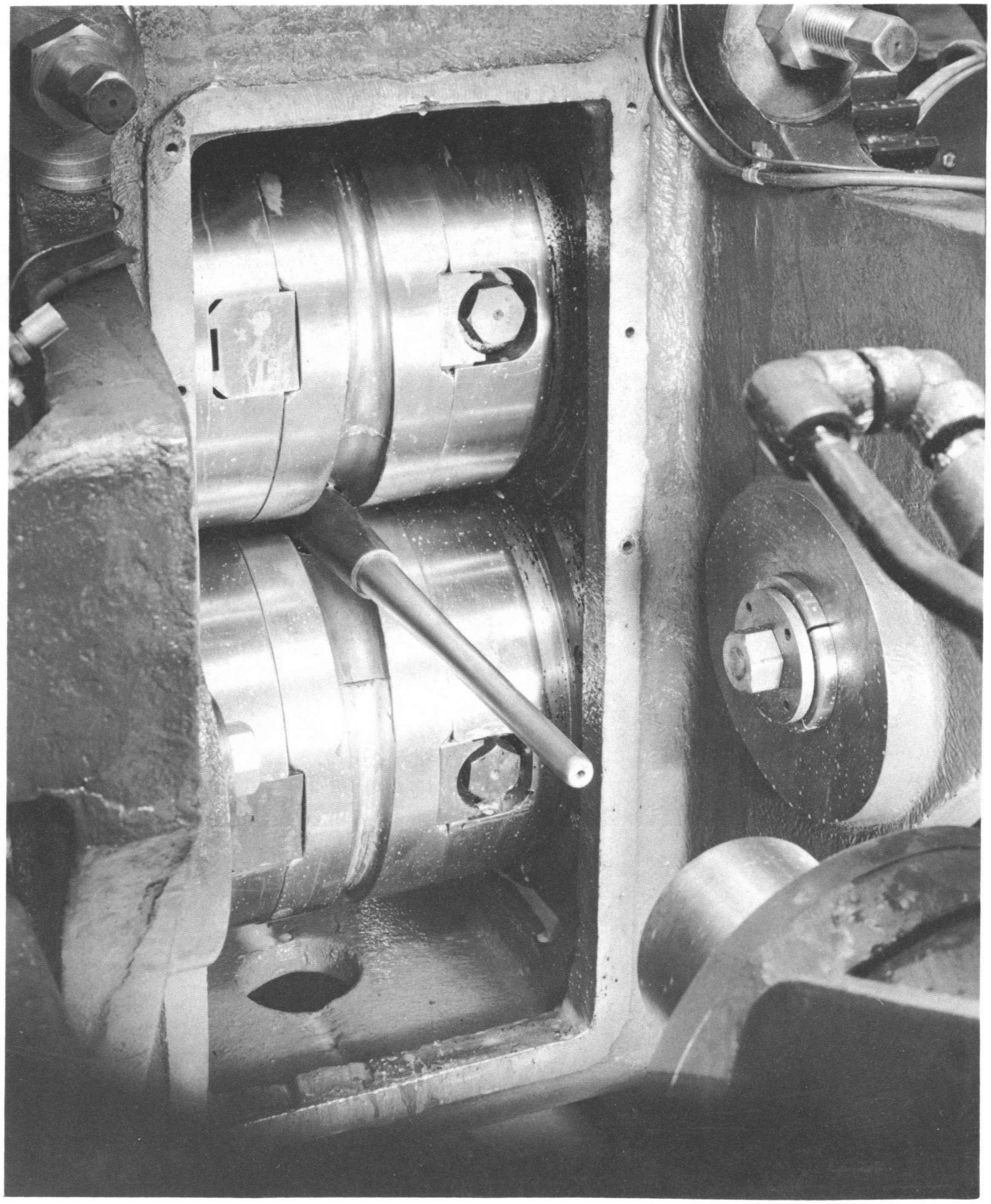

FIGURE 8

The Dies of a Tube Reducer Working a Tube with a Mandrel Providing Internal Support (Photo Courtesy of the Tube Reducing Corporation) cold worked in tapering was inserted into the hot zone of the furnace. The annealing temperature was lowered to 1350 F and the time at temperature was reduced to 3/4 hour.

Annealing under these conditions minimized the bowing to less than 1/2 inch and caused no measurable ovality. Metallographic samples indicated that the annealing time and temperature was sufficient for complete grain recrystallization for the heated portion of the tube. The surfaces of the part of the tube in the cold zone exhibited a light grey film deposit. Most of this deposit (thought to be from the condensation of metal vapors) could be wiped off and that remaining was apparently superficial as it could be easily scraped free.

F. Tapering or Pointing

Development efforts indicated that adequate internal support during tapering would avoid or minimize cracking or wrinkling of the inner surface. Therefore, during the first tube reduction in the production process, a thicker wall was maintained on that part of the tube to be tapered. Subsequently during tapering, considerable internal support would thus be provided by the mandrel used in reducing the wall thickness.

Initial plans for forming the taper and small diameter portion of the tube called for two rocking reductions with an intermediate vacuum anneal. Limited success in development work had been achieved with this approach while tapering in one pass had been unsuccessful. However, with provision for internal support of the tube during reduction, tooling for both a one and two pass tapering operation was designed and procured. The one pass approach, if successful, would eliminate one vacuum anneal as well as the second tapering operation.

The first group of seven tubes to be tapered were badly distorted by the first annealing runs and it had not been possible to remove all of the bowing and ovality. In addition, the surface had been marred by the rotary straightening. Tapering was performed on one pass. Die pickup further marred the outer surfaces and the tube reduction increased the bow. Three tubes were immediately scrapped because of severe cracking of the inner surface of the taper. The other four tubes were scrapped because of the poor surface condition and the cracking that subsequently resulted from further rotary straightening.

The cracking of the metal on the inner surfaces was partly attributed to the tube condition prior to rocking. In subsequent processing, this was to be alleviated by the better quality tubes available from the revised annealing conditions and from more thorough conditioning of any defect on the inner surfaces. Die pickup was reduced by tooling changes; rotary straightening was not required on the balance of the tubes.

Tapering in one tube reducing operation was successful for the remaining part of the order. Out of 125 tubes processed, excluding the first seven, 12 were rejected because of metal rupture or cracking on the inner surface of the taper or small diameter portion of the tube. Marred surfaces from die pickup caused no further rejections. Figure 9 illustrates the taper section of the tube.

G. Flange Attachment

At the time the tube contract was signed, neither the exact requirements of the flange or the means of attachment to the tubes was known. While fabrication of the tubes was in progress, Chase Brass and Copper Company was working under subcontract to develop a flanging technique. Two methods were investigated: slip fitting a ring on the tube and securing by welding at the front and back tube-to-ring joints with subsequent machining to final dimensions; and, screwing a threaded ring on the tube and securing by welding on the outer face only, with subsequent machining to final dimensions. Arc welding was performed with a tungsten electrode in a dry box evacuated and back filled twice with argon. Figure 10 pictures the welding setup.

The major problem which arose in flanging was welding. On the outer flange face, welding with too high an energy input caused rounding of over 1/32 of an inch of the inner corner of the tube, while too little input failed to give adequate penetration. By striking a balance of welding conditions, it was possible to consistently obtain penetrations over 0. 030 inch after

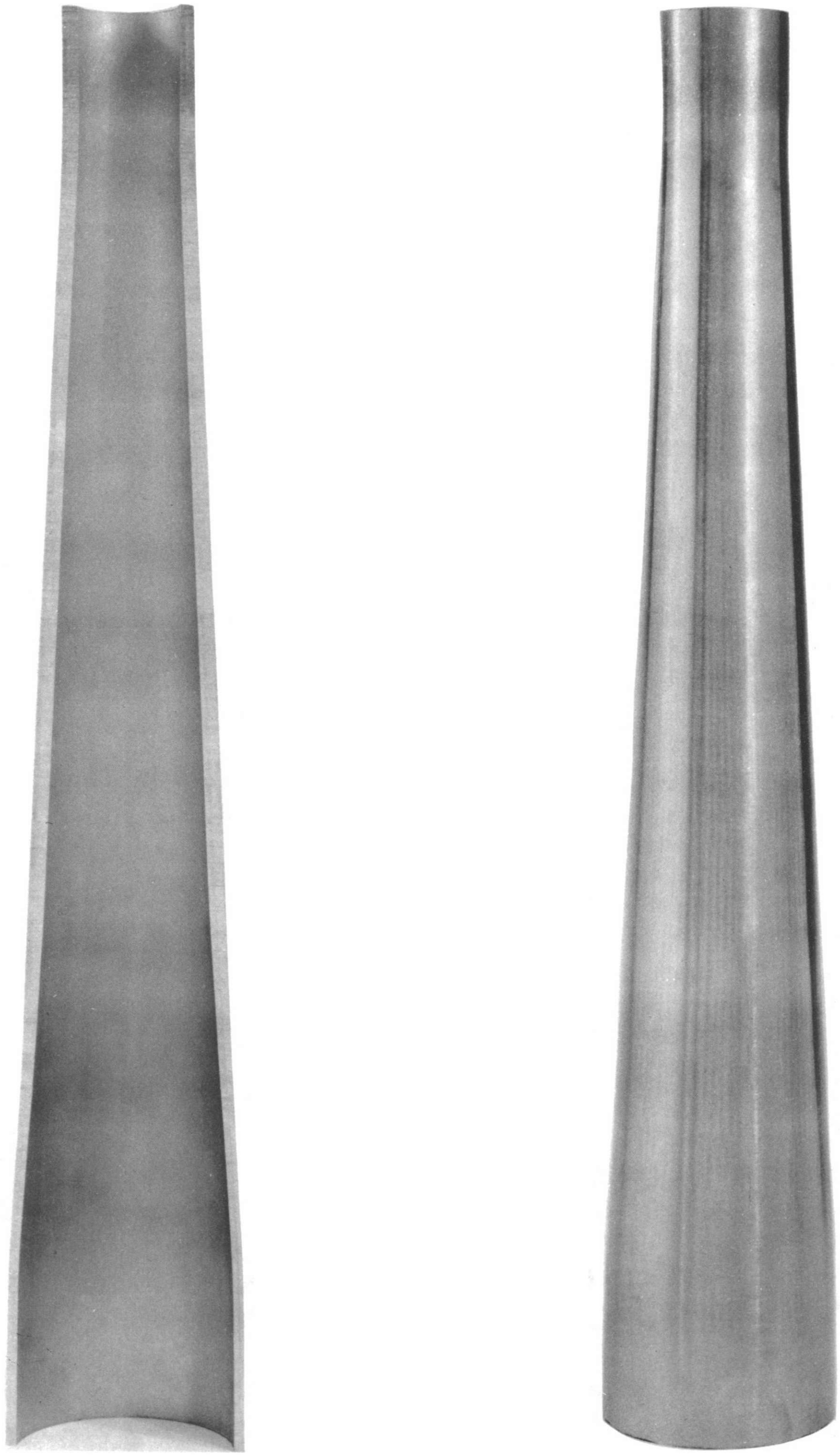

FIGURE 9

A Sectioned Taper from a PRTR Tube

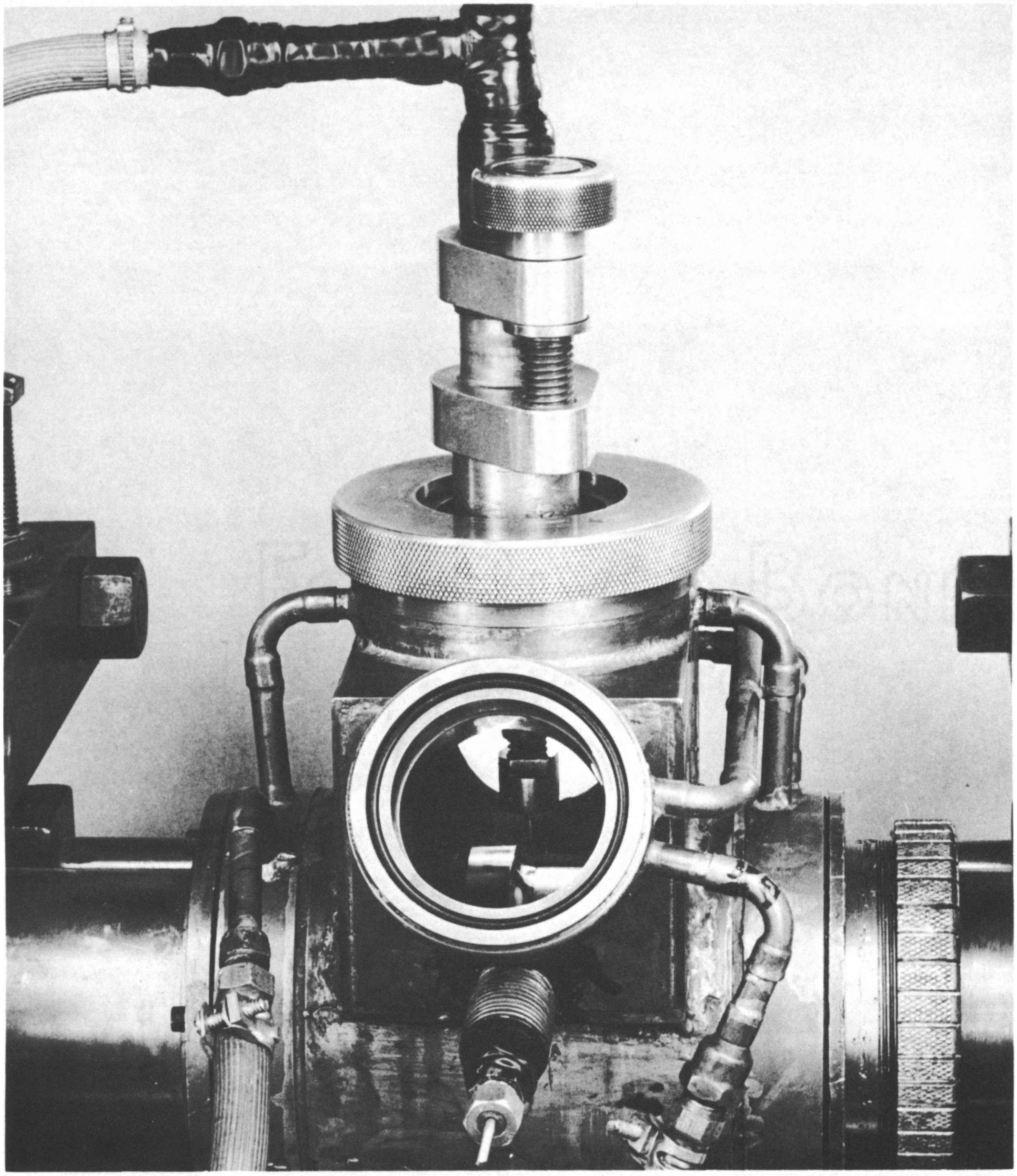

FIGURE 10

Flange Welding Setup

The flange and tungsten electrode can be seen through the porthole.
(Photo Courtesy of the Chase Brass and Copper Co.)

machining. On the inner face of the slip ring type, only a light penetration could be obtained without encountering undesirable side arcing to the back face of the flange.

Further development of the threaded ring flange was undertaken to provide a seal at the back face-to-tube joint. The seal was needed to assure exclusion of nitric-hydrofluoric acid from the threads during subsequent pickling operations at Hanford. To obtain the seal, a lip or extension was machined as an integral part of the back face of the flange. This in effect moved the weld area away from the back flange face and avoided the undesirable side arcing. A reliable sealing weld was thus obtained although it was necessary to strike a nice balance of welding conditions to provide sufficient penetration without burning through the thin lip. Blow holes initially encountered were avoided by welding the lip prior to the outer face. Figure 11 illustrates the flange assembly and Figure 12, a finished flange; dimensions and details are given in the drawing in Appendix A.

Hydrostatic burst tests at Hanford were run at 300 C on flanged tube sections to test for weakness of the flange or flange area. Tested were one slip ring flange, three threaded ring flanges with only the front face welded, and two flanges of the final design. In all cases, the tube burst away from the flange at pressures exceeding 4000 psi. To further test the strength of the threading and welds, an attempt was made to push a flange off the tube end. It held at the press capcity of 46 tons. An attempt was then made to telescope the threaded ring down over the tube and this occurred at a pressure of 42 tons.

H. Final Inspection and Acceptance

Ninety-five process tubes were produced that met all specifications. Appendix A gives the tube drawing and specifications used for this order. Following are descriptions of the inspections and tests used in determining the acceptability of the tubes.

1. Dimensional Inspection

The maximum allowable deviation from straightness was 0. 050 inch in any four-foot length of the tube and 0. 100 inch over the length of the large diameter. The outer diameter of the small end of the tube was to be

FIGURE 11

The PRTR Tube Flange Assembly
(Photo Courtesy of the Chase Brass and Copper Co.)

FIGURE 12

A Finished PRTR Tube Flange
(Photo Courtesy of the Chase Brass and Copper Co.)

concentric within 0.050 inch with the outer diameter of the larger portion of the tube immediately above the taper. The inside straightness was to be such that a 4-foot long by 3.200 plus or minus 0.005-inch plug could be inserted with a reasonable force applied by hand for a distance of 13 feet from the flange.

The minor amounts of straightening required on the finished tubes were performed on a beam straightener. Straightness of the tube and concentricity of the small diameter was measured along the length by a dial gauge while the tube was rotated on two roller supports. All tubes given a final inspection met the straightness specifications.

The minimum allowable wall thickness for the large diameter portion of the tube and the taper was 0.146 inches. The specified wall thicknesses of the small diameter part of the tube were 2.35 and 2.20 inches minimum depending on the location. Thicknesses not less than 0.180 inch were allowed for local conditioned areas if it was demonstrated by radiography that the defects were completely removed.

Wall thicknesses were measured at the ends by a micrometer and over-all with a vidigage instrument with a reliability of plus or minus 0.002 inch. Fifteen tubes were rejected during processing or at final inspection for local areas of low wall down to 0.138 inch caused by removal of surface defects.

The inside diameter of the large diameter portion of the tube was specified as 3.240 - 3.260 inches while that of the smaller diameter was 1.54 inches minimum. Inside diameters were measured by a micrometer at the ends of the tube. To check the inside diameter of the untapered portion of the tube a four-inch long plug gauge of 3.235 plus or minus 0.0005 inches was inserted for a distance of 13 feet 6 inches. No tubes were rejected at final inspection because of the inner diameter. It was necessary, however, for Chase to give six tubes a light sink to decrease the inner diameters to within specifications.

The specified outside diameter of the large OD was 3.584 inches maximum and for the small OD, 2.060 - 2.095, 2.060 - 2.090, and 2.060 - 2.065 inches depending on the location. Measurements were made at frequent random locations by micrometer and over-all on the large OD by an American Standard Ring Gage with 3.588 plus or minus 0.0005 inches ID. No tubes were rejected at final inspection because of outside diameter.

The over-all surface finish was specified as not greater than 125 microinches except for the machined faces of the flange and the bottom six inches of the tube which were to be not greater than 32 microinches. The surfaces were to be free of cracks and free of pits that were black in color, did not show a visible lustrous smoothly rounded bottom, were greater than 1/2 inch in their greatest dimension in the plane of the surface or greater than 0.005 inch in depth.

Visual inspection supplemented by profilometer measurements were made on the outer surfaces; inner surfaces were examined by use of a borescope. No rejections were made because of surface condition.

2. Hydrostatic Testing

Tubes were hydrostatically tested using water inside and air outside at room temperature for three minutes at a pressure of 1875 psi. There was no evidence of cracking, leakage, or permanent set and thus no rejections resulted from pressure testing.

3. Chemical Composition

The ingots were cast at Allegheny Ludlum from reactor grade zirconium under AEC allocation. The ingot composition was according to Hanford specification HWS-6327[1] part of which is given in Tables I and II.

(1) Oakes, H. P. and J. W. Riches, Ingot Procurement Zircaloy-2 and -3, HWS-6327, September 24, 1958.

TABLE I
ALLOY COMPOSITION

Alloying Element	Percent Composition
Tin	1.20 - 1.70
Iron	0.07 - 0.20
Chromium	0.05 - 0.15
Nickel	0.03 - 0.08
Impurities	See Table II
Zirconium	Balance

TABLE II
ALLOWABLE IMPURITIES

Element	PPM-Maximum	Element	PPM-Maximum
Aluminum	75	Magnesium	20
Boron	0.5	Manganese	50
Cadmium	0.5	Molybdenum	50
Carbon	500	Nitrogen	70
Cobalt	10	Oxygen	1400
Copper	50	Silicon	100
Hafnium	200	Titanium	50
Hydrogen	50	Tungsten	50
Lead	100	Vanadium	50

Oxygen determinations were made on only one ingot from each sponge blend. For the alloying elements and all other impurities, samples were taken from 1/4 to no more than 1/2 inch from the surface of the ingot at the top, middle and bottom. The chemical determinations of the samples made by Allegheny Ludlum showed the 22 ingots to be within specifications with the following exceptions: the nitrogen content of the top and middle sidewall samples from ingot 8YH015 was 76 and 84 ppm, and the bottom samples of ingot 8YH017 and 8YH029, 73 ppm; the tin content of the middle sidewall sample from ingot 8YH030 was 1.80 percent.

The tube specifications permitted an increase of nitrogen and hydrogen in the finished tubes to no more than 15 percent over the amount in the ingots or no more than 50 ppm hydrogen and 100 ppm nitrogen whichever was greater for each impurity. The chemical determinations were made on samples taken from the tapered end of the tube after annealing. It appears that only minor amounts, if any, of hydrogen and nitrogen were picked up by the tubes during processing. A comparison of the nitrogen and hydrogen contents of the ingots and the finished and accepted tubes is given in Appendix B. One tube was rejected because of high nitrogen content of 110-112 ppm; the content in samples from the ingot ranged from 63 to 75 ppm. No tubes were rejected for high hydrogen content.

4. Corrosion Characteristics

Corrosion samples were cropped from the tapered end of each tube and tested by Allegheny Ludlum. The specifications required that the samples be etched to remove 0.001 to 0.002 inch of surface and subjected to 400 C steam at 800 psi for 24 hours. For tube acceptance, the sample had to exhibit a smooth glossy black finish and have a weight gain of no more than 30 milligrams per square decimeter of surface. A deviation was granted for 28 samples subjected to 1500 psi steam for 72 hours but the acceptance criteria remained the same.

No tubes were rejected for high weight gain. However, reruns were made on samples from six tubes because of high weight gains. They were accepted on the basis of the retests. Tests at Hanford on the original and other samples from these particular tubes substantiated that the weight gains were within specifications. Test results on samples from these six tubes are given in Table III. The weight gains obtained at Hanford run consistently lower than those obtained by Allegheny. This may be the result of

TABLE III

CORROSION WEIGHT GAINS FOR TUBE SAMPLES

Tube No.	Tube Location	Weight Gain (mg/dm^2) — Original and Same Samples: Allegheny Test	Hanford Test	Allegheny Rerun	Additional Hanford Test
546	Top			22	15, 17
	Bottom	39*	21*, 17	30	17, 18
568	Top			23	16, 16
	Bottom	35*	22*, 18	25	17, 17
495	Bottom	over 30		23	14
503	Bottom	over 30		26	14
513	Bottom	over 30		18	14
530	Bottom	over 30		22	14

*Samples exposed to 750 steam at 1500 psi for 72 hours; all other samples exposed to 750 steam at 800 psi for 24 hours.

changing the steam during autoclaving at Hanford, while at Allegheny the same steam remains with the samples during testing. Further tests on tube samples are now under way at Hanford. The corrosion test results on all other accepted tubes are given in Appendix C.

I. Process and Piece Yields

A total of ninety-five process tubes that met all specifications set forth in the contract were produced. With a maximum number of 138 tubes possible, the piece yield was 69 percent. Many of the initial tubes into processing were rejected while some of the fabrication techniques were being developed and refined. A piece yield of 88 percent was obtained on the last 82 tubes processed compared to 41 percent for the first 56 tubes. An even higher piece yield would be expected on any future orders of this tube.

One hundred and thirty-eight extrusion blanks were made from 22 ingots. No piece rejections occurred during the preparation of the hollow blanks. Out of 138 extrusions made in three production runs, four were rejected. The first three of the first production run were scrapped because of excessive eccentricity of over 15 percent. The other bloom was cracked during straightening.

During the first tube reduction, two tubes failed because of a binding mandrel resulting from an undersize ID. Small cracks or defects generated on the inner surface during the first tube reduction were removed by sand-blasting. Fifteen tubes were rejected when removal reduced the wall thickness below the .146 inch minimum to as low as .138 inch.

The first seven tubes through the tapering operation were rejected because of metal rupturing and marred surfaces. The marred surfaces resulted from die pickup and the Medart straightening required by the distortion from vacuum annealing; metal failure resulted from poor surface condition and the tool design. With tooling corrections made and with good feed stock provided by the revised annealing cycle, only one tube subsequently failed during tapering; no further marred surfaces of a serious magnitude were encountered. However, the tapering operation did result in 12 reject tubes because of cracks or defects generated during reduction and the low wall thicknesses occasioned by their removal.

One tube was rejected for high nitrogen content of 110-112 ppm. One tube was rejected for short length.

Twenty-two ingots were furnished by Hanford under the contract. As noted earlier, the ingots were forged and the hollow extrusion blanks prepared by either boring or piercing. A nominal weight of 146 pounds of cast metal was needed for each billet that was pierced while the billets that were bored required about 184 pounds. Estimated typical losses for the purpose of describing the process are given in Table IV. The nominal finished tube weight is 85 pounds. The calculated process yield (percentage of the ingot metal weight remaining in the finished tube) for tubes produced from pierced billets is 58 per cent and for those from bored billets 46 per cent.

A total weight of 22,033 pounds of Zircaloy-2 in ingot form was used in producing the tubes. Of this total, 6,611 pounds were used for the 35 billets including excess weights of about 225 pounds in the billets; 15,237 pounds were used for 103 billets for piercing; and about 185 pounds were used as stock for flange rings.

The weight of the 95 tubes produced was 8,150 pounds. The overall weight yield from total ingot metal was 37 per cent. The weight yield of the 72 acceptable tubes from the last 82 pieces processed, which were all from pierced billets was approximately 51 per cent.

VI. CURRENT AND CONTEMPLATED PRE-IRRADIATION INSPECTION, TESTING AND TREATMENT AT HANFORD

A. Destructive Burst Testing[2]

Presently planned working pressures and temperatures for the PRTR tubes are 1060 psig at 478 F for the tube inlet and 1050 psig at 542 F for the tube outlet. Hydrostatic burst tests at approximately 572 F have been run on sections from the PRTR process tubes. Table V gives the results of these tests. The minimum burst pressures given in Table V were obtained by calculating the unit stress for the tube section tested and then calculating what the burst pressure would have been with a minimum wall thickness of 0.146 inch and the maximum specified inside diameter of 3.260 inches. Therefore, the burst pressures are representative for the weakest dimensional situation that would be encountered. Reference (2) discusses the results of burst testing of Zircaloy-2 pressure tubing. Burst testing of tube sections is continuing.

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(2) Kahle, V. E. Interim Report on the Hydrostatic Destructive Testing of Zircaloy-2 Pressure Tubing, HW-61272. August 16, 1959.

TABLE IV

ESTIMATED PIECE WEIGHT LOSSES DURING PROCESSING

Processing Description	Piece Weight (lb.)	Estimated Loss (lb.)
I. Billet Preparation		
A. Forge	184	
Cut to length	173.5	10.5
Bore	134	39.5
Turn OD and face ends	118	16
B. Forge	146	
Cut to length	144	2
Pierce	132	12
Bore, turn OD and face ends	118	14
II. Extrusion		
Crop	116.5	1.5
Dejacket and OD	108.5	8
Crop end and bright pickle	105	3.5
III. First Tube Reduction		
Surface preparation and chamfer	104	1
Cropping	100	4
IV. Pointing or Tapering		
Surface preparation and chamfer	99	1
Surface conditioning	98	1
Cropping to near length	87	11
V. Preparation for Inspection and Flanging		
Cut to length and groove	85	2
Average tube weight =	85	

TABLE V

BURSTING PRESSURES AT APPROXIMATELY 572 F FOR PRTR PROCESS TUBE SECTIONS

Metal History	Minimum Burst Pressures (psig)	Experimental Unit Stress (psi)
Forged, Extruded, Tube	3700	42,600
Reduced and Vacuum Annealed	4000	46,000
	4100	47,200

B. Nondestructive Testing and Inspection

The integrity of the tube is currently being examined at Hanford by the use of fluorescent dye penetrant, ultrasonic and radiographic inspection methods. Work thus far has been directed toward interpretation of test results and establishing standards. The contemplated tests are briefly described here.

The fluorescent dye penetrant test is used to locate surface cracks, pits, or other physical discontinuities. Visual inspection of the outer surface is conducted under a hood using ultra violet lighting; the inner surface is inspected with a borescope with a black light attachment.

Ultrasonic equipment known as the Immerscope is being used to detect physical reflecting surfaces such as cracks, holes, or mars at the surface or the interior of the wall.

Radiographs are being made of the taper section of the tube for assurance of metal integrity and soundness.

C. Autoclaving[3,4]

Preparation for autoclaving is by etching one to two mils of surface metal in a nitric hydrofluoric acid solution, stopping in an aluminum nitrate bath, rinsing in tap water, rinsing in deionized water and air drying.

To obtain a uniform black oxide film, tubes will be autoclaved in 400 C steam for 36 hours at 100 psi with the tubes submerged in deionized water at the start of heating. Proposed processing will be based on references (3) and (4).

D. Wall Thickness and Bow Measurement

The wall thickness of all tubes will be measured by vidigaging before delivery of the tubes to the contractor for insertion in the pile. Straightness of the tubes will be checked.

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(3) Shannon, D. W. The Etching and Autoclaving of Zircaloy-2, December 23, 1958, HW-58733, January 2, 1959.

(4) Shannon, D. W. and B. Griggs. Preparing Zircaloy-2 for Autoclave Testing, HW-60433, July 1, 1959.

VII. ACKNOWLEDGMENT

The author wishes to acknowledge the information and pictures furnished for this report by personnel at the Tube Reducing Corporation, Chase Brass and Copper Company and the Allegheny Ludlum Steel Corporation. He wishes to thank them for reviewing the original draft of the report and offering helpful suggestions.

APPENDIX A

SPECIFICATION H-3-11370 REV. 2

Specifications:

1. Tube Dimensions -
   Shall be in accordance with Drawing Number H-3-11370, Rev. 3.

2. Straightness -
   of tubes shall be such that the maximum deviation from a vertical straight line shall not exceed 0.100 inch in length of tube, large diameter only, measured with tube in vertical position hung from OD of the tube immediately below the flange. * The maximum deviation of the tube from a straight line shall not exceed 0.050 inch in any 4 foot length. The outer diameter of the small end of the tube shall be concentric with 0.050 inch with the outer diameter of the larger portion of the tube immediately above the taper. A drift pin fabricated by the seller 4 feet long x 3.200 ± 0.005 inch shall be inserted with a reasonable force applied by hand, for a distance of 13 feet from the flange.

3. Ovalness -
   Shall be included in the diameter tolerances.

4. Wall Thickness -
   Shall be measured by a micrometer at each end of the tube and over-all with a nondestructive test device provided by seller capable of reproducible results with an accuracy of plus or minus 0.002 inch. The wall thickness of the tubes between the small end of the taper and one inch from the end of the tube may be less than specified on Drawing H-3-11370 Rev. 3 but not less than 0.180 inch provided that it is demonstrated by radiography that no cracks remain in this area of the tubes.

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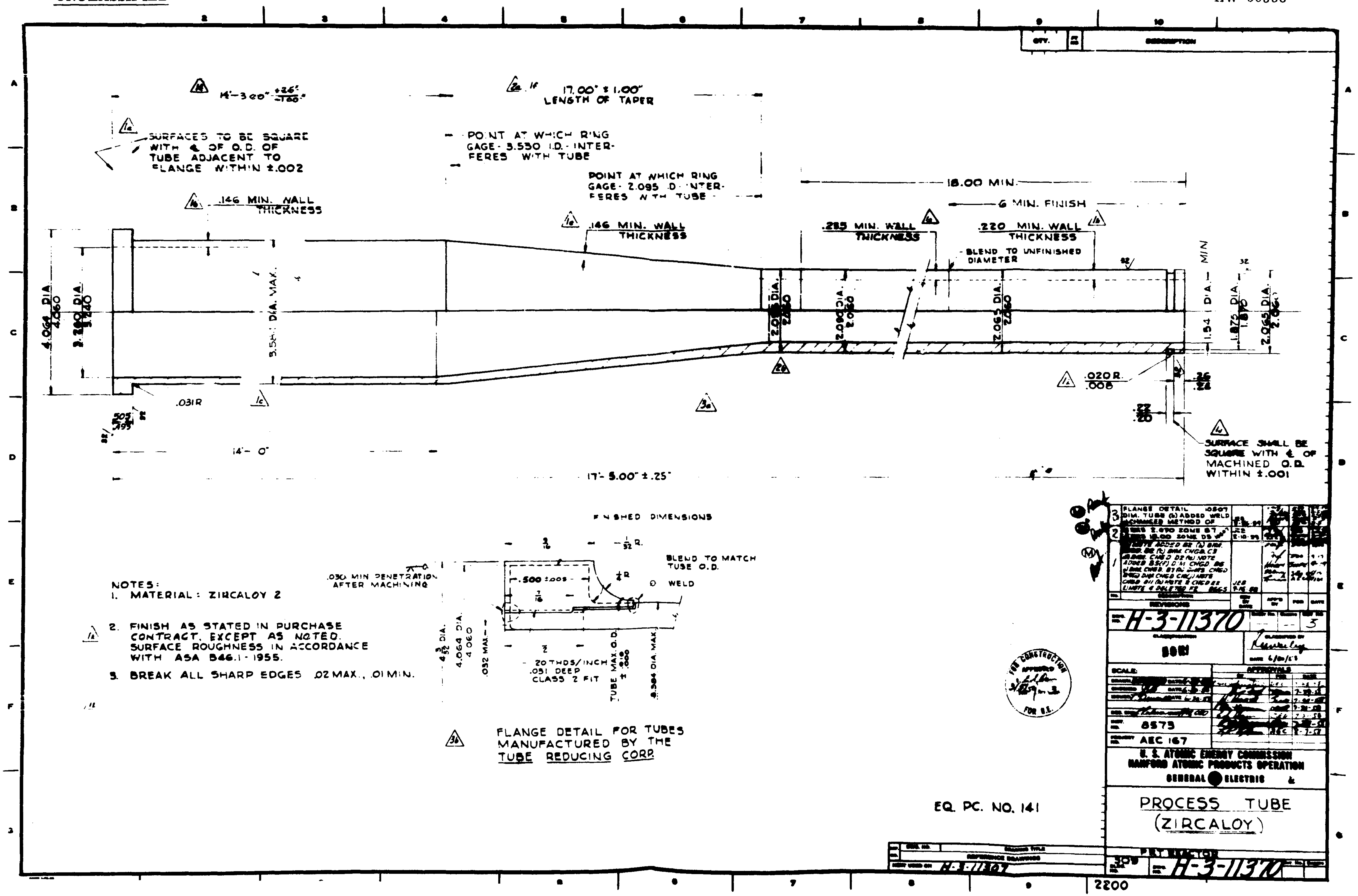
* A deviation was granted for measuring straightness with the tube in a horizontal position. Any sag encountered was considered part of the bow under the specification.


SURFACES TO BE SQUARE WITH ℄ OF O.D. OF TUBE ADJACENT TO FLANGE WITHIN ±.002
17.00" ± 1.00" LENGTH OF TAPER
POINT AT WHICH RING GAGE - 3.550 I.D. - INTERFERES WITH TUBE
POINT AT WHICH RING GAGE - 2.095 I.D. - INTERFERES WITH TUBE
.146 MIN. WALL THICKNESS
.146 MIN. WALL THICKNESS
.285 MIN. WALL THICKNESS
.220 MIN. WALL THICKNESS
18.00 MIN.
6 MIN. FINISH
BLEND TO UNFINISHED DIAMETER
4.064 DIA 4.060
3.554 DIA. MAX.
.031 R
14'- 0"
17'- 5.00" ± .25"
.020 R .008
SURFACE SHALL BE SQUARE WITH ℄ OF MACHINED O.D. WITHIN ±.001
FINISHED DIMENSIONS
.030 MIN PENETRATION AFTER MACHINING
BLEND TO MATCH TUBE O.D.
WELD
20 THDS/INCH .031 DEEP CLASS 2 FIT
NOTES:
1. MATERIAL: ZIRCALOY 2
2. FINISH AS STATED IN PURCHASE CONTRACT, EXCEPT AS NOTED. SURFACE ROUGHNESS IN ACCORDANCE WITH ASA B46.1-1955.
3. BREAK ALL SHARP EDGES .02 MAX., .01 MIN.
FLANGE DETAIL FOR TUBES MANUFACTURED BY THE TUBE REDUCING CORP.
EQ. PC. NO. 141
FOR CONSTRUCTION APPROVED FOR G.E.
H-3-11370
8575
AEC 167
U. S. ATOMIC ENERGY COMMISSION
HANFORD ATOMIC PRODUCTS OPERATION
GENERAL ELECTRIC
PROCESS TUBE
(ZIRCALOY)
H-3-11307
2200

5. Inside Diameter -
Shall be measured by a micrometer at each end and the large untapered portion of the tube shall be measured by a special plug gage. The special plug gage shall be fabricated by seller and shall be used to check the inside diameter of the untapered portion of each tube. The plug gage shall be 4 inches long and shall be 3. 235 inches plus or minus 0. 0005 inches in diameter. The inside diameter of the untapered portion of the tubes must be such that the plug gage may be pulled through the untapered portion of the tubes from the flanged end to a distance of 14 feet from the flanged end when a reasonable force is applied by hand.

6. Outside Diameter -
Shall be measured by an American Standard No. 10 Ring Gage with 3. 588 inches plus or minus 0. 0005 inches ID and a micrometer and shall not exceed the maximum specified on the applicable drawing.

7. Surface Roughness -
The inside and outside surfaces of the tubes shall exhibit a surface roughness not greater than 125 microinches. The surfaces shall be free of cracks and shall be free of pits that are black in color and/or do not show a visible lustrous smoothly rounded bottom or are greater than 1/2 inch in their greatest dimension in the plane of the surface or greater than 0. 005 inch (estimated) in depth, providing that at no point shall the wall thickness be below minimum wall.

8. Cleanliness -
Prior to submitting the tubes for inspection by the General Electric Company, the tubes must be cleaned, removing dirt and grease or oil from both the interior and exterior. All stages or steps of manufacture shall be clean to the extent that no contaminants are carried, pressed, or drawn-in to the surface which will be detrimental to the physical or chemical properties of the tube.

9. Hydrostatic Test -
Each length of tubing shall be hydrostatically tested using water in air or air in water at room temperature for three minutes at a pressure of 1875 psi. Evidence of cracking, leakage, or permanent set shall cause rejection of the tubing being tested.

10. Corrosion Test -
A corrosion sample shall be taken from the cropped end of each tube. Samples shall be etched to remove 0.001 and 0.002 inch of surface, subjected to 400 C steam at 800 psi for 24 hours and shall exhibit a smooth glossy black finish and have a weight gain of no more than 30 milligrams per square decimeter of surface.

11. Zirconium alloy ingots furnished under this contract will be Zircaloy-2 of nominal composition of 1.5% Sn, 0.15% Fe, 0.10% Cr, 0.5% Ni, and the balance zirconium. The seller will be furnished with the analyses of ingots and corrosion test results for comparison purposes. The ingots will be nominal 12-inch diameter machine conditioned ingots weighing 900 to 1000 pounds. The process used in the manufacturing of these tubes will be such that the increase of nitrogen and hydrogen shall not exceed 15 per cent of amount present in the ingot supplied or the final amount shall not exceed 50 ppm hydrogen and 100 ppm nitrogen whichever is greater for each impurity. The hydrogen and nitrogen content will be determined on a sample taken from the end cropping from each tube.

12. Certified Reports -
The seller shall furnish three (3) copies of notarized certification that the tubing complies with all specifications and a tabulation of the results of tests performed under items 10 and 11. Mail to General Electric Company, Purchasing and Stores, Richland, Washington, Attention: G. E. Lish.

13. Annealing -

Any annealing following initial hot forming shall be performed in vacuum unless otherwise authorized in writing by buyer.

14. Flanges -

Flanges on tubes manufactured by the Tube Reducing Corporation shall be fabricated in accordance with the detail shown on H-3-11370 Rev. 3. Depth of weld penetration remaining at the face of the flange after finish machining shall be determined by full radiography.

APPENDIX B

NITROGEN AND HYDROGEN CONTENT OF INGOTS AND TUBES

Ingot No.	Top Sidewall Middle Sidewall Bottom Sidewall Nitrogen (ppm)	Top Sidewall Middle Sidewall Bottom Sidewall Hydrogen (ppm)	Tube No. *	Nitrogen (ppm)	Hydrogen (ppm)
5Y-H-10	46	20	482	44	15
	41	20	484	35	19
	51	20			
5Y H-38	46	10	485	55	10
	51	10	489	72	21
	46	10			
8YHO-13	62	10	491	67	21
	64	10	493	80	15
	63	10			
8YHO-15	76	10	495	100	10
	84	10	497	86	13
	67	10	499	94	15
8YHO-17	66	10	501	83	15
	64	10	502	73	19
	73	10	503	76	10
			504	86	33
8YHO-29	63	3	505	74	15
	63	6	506	71	11
	73	6	507	73	11
			508	73	15
			509	69	19
8YHO-6	52	10	511	54	11
	56	10	512	69	15
	61	10	513	72	10
			514	88	15
8YHO-30	67	3	518	62	15
	70	6	519	72	19
	69	6	520	64	19
			521	68	27

* Location within the individual ingots of the metal of the particular tubes is not known.

APPENDIX B (contd.)

Ingot No.	Top Sidewall Middle Sidewall Bottom Sidewall Nitrogen (ppm)	Top Sidewall Middle Sidewall Bottom Sidewall Hydrogen (ppm)	Tube No. *	Nitrogen (ppm)	Hydrogen (ppm)
8YHO-20	61	10	522	62	31
	54	10	523	71	19
	47	10	525	67	15
			526	70	15
			527	78	15
			528	55	21
8YHO-19	56	10	529	69	26
	59	10	531	76	15
	53	10	532	68	21
			533	62	15
			535	83	15
8YHO-8	55	10	536	68	21
	59	10	537	69	15
	54	10	538	69	15
			540	91	15
			541	64	15
			542	72	27
8YHO-31	62	6	543	72	21
	58	6	544	65	21
	59	3	545	64	15
			548	60	10
			549	68	15
8YHO-28	69	6	551	73	15
	66	3	552	73	9
	64	3	553	66	10
			554	73	15
			555	68	10
8YHO-26	56	3	558	65	15
	56	3	560	58	10
	53	3	561	92	15
			562	69	10
			563	59	10

* Location within the individual ingots of the metal of the particular tubes is not known.

APPENDIX B (contd.)

Ingot No.	Top Sidewall / Middle Sidewall / Bottom Sidewall Nitrogen (ppm)	Top Sidewall / Middle Sidewall / Bottom Sidewall Hydrogen (ppm)	Tube No. *	Nitrogen (ppm)	Hydrogen (ppm)
8YHO-18	63	10	564	69	21
	68	10	565	70	15
	68	10	566	69	15
			567	69	15
			569	68	10
			570	77	15
8YHO-24	63	10	571	70	15
	59	10	572	68	10
	55	10	573	65	15
			574	65	15
			575	72	21
			577	60	21
8YHO-14	70	10	579	83	15
	68	10	582	84	15
	72	10	583	93	15
8YHO-25	60	3	585	64	10
	59	3	587	68	10
	68	3	588	67	15
			589	64	15
			591	71	15
8YHO-21	59	10	595	60	15
	60	10	596	94	10
	57	10	598	69	21
8YHO-16	63	10	599	77	21
	75	10	600	69	10
	63	10	601	73	21
8YHO-23	65	3	606	56	15
	59	3	607	61	15
	56	3	608	55	15
			609	54	15
			610	59	10
8YHO-27	60	6	614	58	13
	59	6	616	64	15
	54	6	618	72	10

* Location within the individual ingots of the metal of the particular tubes is not known.

APPENDIX C

CORROSION WEIGHT GAINS FOR TUBE SAMPLES

Tube No.	Wt Gain (mg/dm^2)	Tube No.	Wt Gain (mg/dm^2)	Tube No.	Wt Gain (mg/dm^2)
482	22	531	17	570	21*
484	18	532	13	571	18
485	24	533	22	572	23
489	22	535	23*	573	19
491	17	536	16	574	16
493	18	537	21	575	23*
497	18	538	17	577	13
499	16	540	20*	579	24*
501	22*	541	12	582	25*
502	11	542	18	583	24*
504	17	543	21*	585	23
505	26	544	16	587	20
506	13	545	15	588	13
507	15	548	25	589	19*
508	26	549	20*	591	19*
509	20	551	20	595	20
511	15	552	21	596	18
512	25	553	24	598	21
514	22*	554	23*	599	19*
518	22	555	25	600	14
519	17	558	12	601	14
520	12	560	22	606	15
521	29	561	21*	607	19
522	12	562	23	608	19
523	15	563	20	609	13
525	26	564	12	610	19
526	21	565	23	614	13
527	21*	566	12	616	19
528	21	567	14	618	25
529	21	569	16		

* Samples exposed to 750 F steam at 1500 psi for 72 hours; all other samples exposed to 750 F steam at 800 psi for 24 hours.

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