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TOPICAL REPORTS

- I. STRUCTURAL CHARACTERIZATION AND BIAXIAL MECHANICAL PROPERTIES OF VACUUM-MELTED TYPE 316 STAINLESS STEEL TUBING
- II. CLADDING WELDABILITY STUDY

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REPORT I
STRUCTURAL CHARACTERIZATION AND
BIAXIAL MECHANICAL PROPERTIES OF
VACUUM-MELTED TYPE 316 STAINLESS
STEEL TUBING

T. R. Padden, W. E. Ray, E. J. Tarby, K. C. Thomas

SECTION 1 INTRODUCTION

1.1 GENERAL

A comprehensive test program is being conducted on Type 316 stainless steel. Early selection of this alloy for FFTF use permits in-depth studies of fundamental problems associated with Type 316 stainless steel as an oxide fuel pin cladding for LMFBR applications. The study described herein is concerned solely with unirradiated properties, but is focused on those properties identified as basically influencing inpile behavior, such as: grain size; cold work; melting practice; texture; dimensional variations; and surface defects.

Early work in this program was performed on air-melted, stainless steel tubing to set a base line for a later systematic investigation of vacuum-melted, Type 316 stainless steel tubing. Reported here are the results from that vacuum-melted steels program, plus a summary of a weldability study conducted on air-melted tubing.[1-10]

Results from a separate study of the thermal precipitation behavior of air-melted, Type 316 stainless steel tubing have already been reported in Reference 3. Additional precipitation studies, conducted as Westinghouse research programs, are covered in Westinghouse reports by Weiss, Stickler, and co-workers.[11-13]

The scope of the vacuum-melted steels program, which comprises the bulk of this report, is outlined in Table 1-1. Fourteen lots of tubing were procured for the systematic investigation. The prime variables were vacuum-melting practice, cold work, grain size, and fabrication procedure. The melting practices included: 1) consumable electrode vacuum; 2) induction vacuum; and 3) induction vacuum followed by consumable vacuum. All vacuum-remelt heats were produced from the same air-melt ingot. Twelve of the tubing lots were fabricated by drawing, and included the systematic combination of the three melting practices, two grain sizes (ASTM 6 and 9, nominal), plus the annealed and cold worked conditions. Two lots of tubing, fabricated from the double vacuum melted heat, and using planetary swaging for the final passes, were also procured. One lot was in the annealed condition and one lot in the 20 percent cold worked condition; both had a coarse grain size of (ASTM 6).

As-received tubing was characterized by chemistry, grain size and shape, inclusion evaluation, surface morphology, and hardness. In addition, four of the double vacuum-melted lots were comprehensively characterized by light microscopy, transmission electron microscopy, and electron diffraction of thin sections and of extraction replicas, X-ray diffraction analysis of bulk extractions, electron micro-probe analysis of inclusion particles in situ, Laue back reflection analysis for residual stress, and X-ray pole figure texture studies.

Table 1-1. Program Scope				
Fabrication	Plug Drawn (12 Lots)		Planetary Swaged (2 Lots)	
Three Melting Practices	a. Consumable Vacuum Melt		---	
	b. Induction Vacuum Melt		---	
	c. Double Vacuum Melt a + b		c. Double Vacuum Melt a + b	
Condition	Solution Treated	20% Cold Worked	Solution Treated	20% Cold Worked
Grain Size	5-6 8-9	5-6 8-9	5-6	5-6
Observation and Test	Chemistry Grain Size and Shape Hardness Inclusions Transmission Microstructure Dislocation Density Electron and X-Ray Diffraction Pole Figures Electron Probe Analysis		Biaxial Tensile Biaxial Stress Rupture Post-Test Specimen Characterization: . Rupture Morphology . Strain Profile . Rupture & Shoulder Ductility . Local Wall Strain . Metallography	

The short-term mechanical properties of 8 of the 14 tubing lots and several heat treated specimens were evaluated by biaxial tensile testing (internal pressurization with gas) at room temperature and five elevated temperatures up to 1300°F. An extensive biaxial stress rupture program was also conducted at elevated temperatures to evaluate the longer-term mechanical properties. Because of limited facilities, a complete test program of all 14 lots could not be run; however, sufficient tests were conducted to evaluate the major material variables.

Post-test stress rupture specimens were characterized and evaluated using a variety of observations and measurements. The post-test specimens were classified according to rupture morphology, and evaluated by this characteristic with respect to the major material variables. Diametral strain profile measurements were made of 53 of the specimens to show the general shape of the strain profile along the length of the specimens, similar to the approach used by Conway and Flagella.^[14] The rupture ductilities and the shoulder ductilities were measured, and used to compare material variables and stress levels based on ductility. Local wall thickness measurements of specimens were made with a calibrated microscope at the rupture areas and other locations of interest to evaluate variations in local wall strain with respect to the processing variables. In addition, 28 post-test rupture specimens from the double vacuum melted lots were examined metallographically and evaluated to determine the origin of rupture, the controlling strain mechanism, and the general microstructure.

Work in progress on these tubing lots, but not reported here, includes uniaxial creep testing at very low stress levels.

Throughout the investigation, the 20 percent cold worked, fine-grained, double vacuum-melted tubing lot was considered as the reference material, and it received the most comprehensive characterization and testing.

The presentation of the weldability investigation is in Report II of this book.

1.2 TUBING HISTORY

Fourteen lots of tubing were fabricated to the Westinghouse ARD-AMMA-001-2 tubing specification, [15] modified to include the material variables of the test program. The general fabrication schedule is shown in Figure 1-1. Lot identification, description, and certified properties for each lot are given in Table 1-2, along with the average hardness.

The tubing specification permitted flash pickling and surface polishing of the OD. Tubing was accepted to an ultrasonic test standard containing both longitudinal notches and radially drilled holes. The longitudinal OD notch had a depth 10 percent of the wall (0.0015 inch), a width of 0.004 inch, and a maximum length of 1/4 inch. The hole had a 0.006-inch diameter and a depth of 0.0128 inch, approximately 85 percent of the wall.

Figure 1-1. Material History

1.3 CHEMISTRY

The 14 lots of vacuum-melted, Type 316 stainless steel tubing were procured to Reference 15. The chemistry specification was similar to the reference FFTF fuel element cladding composition in effect at the time, [16] which was less restrictive than the current FFTF specification. [17]

The chemical analysis after various processing steps in the manufacture of the tubing are given in Table 1-3. Included are: 1) the ladle analysis for the original air-melt heat and the three vacuum-remelt heats; 2) the ARD check analysis of the bar stock from the three vacuum-remelt heats; and 3) the ARD check analysis of tubing from each remelt heat. Two tubing lots from each heat were analyzed and the results averaged; also included for comparison is the current FFTF fuel cladding chemistry specification.

Table 1-2. Certified Mechanical Properties and Description of ARD, VM, Type 316 Stainless Steel Tubing

Heat	Superior Lot Number	Item	Condition	Superior Certified ASTM Gr. Size	Fabrication	Superior Certified Uniaxial Mechanical Properties at Room Temperature			Superior Certified Dimensions			ARD Measured Hardness DPH
						Yield (ksi)	Ultimate (ksi)	Elong. - 2 In. (%)	OD	ID	Ovality ID	
30160 Consumable Electrode Vacuum Remelt	42394	1	20% CW	9/9.5	PD	108.9 111.2	125.1 127.0	18.0 22.0	0.2304 / 0.2310	0.1997 / 0.2001	0.0001 / 0.0002	276
	42395	2	Annealed	8	PD	43.7 44.6	90.2 90.4	53.0 54.0	0.2302 / 0.2308	0.1998 / 0.2000	0.0001 / 0.0003	142
	42396	3	20% CW	5.5/6	PD	104.0 106.4	118.0 118.8	18.0 22.0	0.2305 / 0.2310	0.1999 / 0.2003	0.0001 / 0.0002	266
	42397	4	Annealed	5.5	PD	38.8 40.8	84.6 85.0	57.0 64.0	0.2300 / 0.2306	0.1997 / 0.2000	0.0001 / 0.0003	150
91694 Vacuum Induction Remelt	42398	5	20% CW	9	PD	111.2 111.2	124.0 125.0	17.0 20.0	0.2302 / 0.2310	0.1999 / 0.2003	0.0001 / 0.0003	278
	42399	6	Annealed	8	PD	50.5 51.0	90.7 90.7	52.0 55.0	0.2300 / 0.2307	0.1996 / 0.2001	0.0001 / 0.0003	145
	42400	7	20% CW	5.5/6	PD	101.5 104.0	115.8 116.5	20.0 22.0	0.2302 / 0.2309	0.1998 / 0.2000	0.0001 / 0.0003	269
	42401	8	Annealed	5/5.5	PD	46.5 47.1	83.6 84.3	58.0 64.0	0.2298 / 0.2304	0.1995 / 0.1999	0.0001 / 0.0003	144
91695 Vacuum Induction Plus Consumable Electrode Remelt	42402	9	20% CW	9	PD	108.9 115.0	123.3 123.3	18.0 22.0	0.2304 / 0.2310	0.2000 / 0.2303	0.0001 / 0.0002	273
	42403	10	Annealed	8	PD	47.6 51.0	89.9 90.5	52.0 55.0	0.2300 / 0.2307	0.1995 / 0.1999	0.0001 / 0.0003	148
	42404	11	20% CW	5.5/6	PD	101.6 104.0	117.0 117.5	21.0 22.0	0.2304 / 0.2309	0.1997 / 0.2001	0.0001 / 0.0004	261
	42405	12	Annealed	5/5.5	PD	35.2 36.8	82.4 82.4	60.0 67.0	0.2300 / 0.2307	0.1996 / 0.2001	0.0001 / 0.0003	132
	49193	19	20% CW	6	PS	119.9 122.0	134.0 136.4	9.0 10.0	0.2302 / 0.2308	0.0150 / 0.0157*	0.0003 / 0.0004**	325
	49194	20	Annealed	6	PS	32.2 33.6	83.1 84.1	62.0 64.0	0.2302 / 0.2309	0.0151 / 0.0161*	0.0001 / 0.0006	131
CW - Cold Work PD - Plug Drawn				PS - Planetary Swage DPH - Diamond Pyramid Hardness (Avg. 7 indentations) Coldworked 500-gram load Annealed 300-gram load					* Wall ** OD			

The bar stock and five of the air tubing lots met the ARD chemistry specification. For the sixth tubing lot, the carbon analysis showed 0.035 weight percent (w/o) (Heat No. 91695-1, lot 42405) compared to specified range of 0.04 to 0.06 w/o, and one nitrogen analysis 0.032 w/o (lot 42405) compared to 0.030 w/o maximum. These values were close to the specified limits, and both the bar stock and the other tubing lot, analyzed from the same heat, were within specifications. All other values were within specification.

Comparison of the chemistry data at various fabrication steps indicates loss of some Mn during single vacuum-remelting, and additional loss during the second vacuum-remelting. The Mn dropped from 1.8 w/o in the air-melted ingot to 1.3 w/o in the double vacuum-melted material (heat 91695).

The supplier's nitrogen data from the air-melted heat and from the vacuum-remelt heats indicate that a slight increase in nitrogen occurred during vacuum remelting. The ARD data from the bar stock and from the fabricated tubing indicate that an additional slight increase in nitrogen content occurred during tubing fabrication. The maximum nitrogen increase reported was from 0.019 w/o in the air melted ingot to 0.032 w/o in the (CEV) tubing (lot 42397).

Relative to the current FFTF specification, the ARD tubing was within range for Ni and Mo, slightly low in Cr for all heats, and slightly low in Mn for the double vacuum-melted heat only. The carbon analysis (average of two lots from each remelt heat) were within the FFTF specification, but the nitrogen levels were high. The current FFTF specification is generally more restrictive in minor elements than the ARD specifications. However, the tubing was within the current FFTF specification for a number of these elements (Si, Co, Nb, P, and B). The tubing was slightly high in Cu and S; Al, As, Ta, and V were not analyzed.

SECTION 2

PRE-TEST CHARACTERIZATION

2.1 GENERAL METALLOGRAPHY

Transverse and longitudinal sections of all 14 lots of tubing were examined in the as-polished, lightly etched, and etched (to show grain size) conditions. Figure 1-2 illustrates the etched microstructures of longitudinal sections from the fine-grained, annealed and the fine-grained, cold worked, plug drawn tubing lots from each of the three vacuum-melting practices.

2.2 GRAIN SIZE AND SHAPE

Check grain sizes were determined for all 14 lots of tubing by comparison with ASTM charts; the results generally confirmed the supplier certified grain sizes listed in Table 1-2. There was a slight laboratory bias favoring higher ASTM grain size numbers in some of the check analyses. The difference was about 1/2 grain size number, which was judged to be in satisfactory agreement. The certified grain sizes listed in Table 1-2 were accepted as the correct values.

The grain shape in the annealed lots was equiaxed in both the longitudinal and transverse sections, whereas the grain shape in the cold worked lots was elongated slightly in the longitudinal direction.

2.3 INCLUSIONS

In general, the inclusions were frequent in number and very small in size. Tubing from all three vacuum-melted heats appeared to be significantly cleaner than air-melted, Type 316 stainless steel tubing examined previously. A high fraction of the inclusions in the vacuum-melted material was in the form of very narrow continuous and discontinuous stringers. There were also small isolated particles, and in the single vacuum-melted lots there were occasional comet shaped inclusions larger in size than the general stringers.

The inclusions were not readily observed in the as-polished condition because of their narrow width. When the specimens were lightly etched, the inclusions could be observed, but the narrow width precluded quantitative rating according to ASTM charts. In many cases, the width of the inclusions was less than 1/5 that of the thin classification in ASTM designation E45, Chart III. As a result, characterization of the inclusion content by comparison with this chart at the specified 100 power magnification would be misleading.

In the absence of a meaningful quantitative evaluation method, the difference in inclusion content associated with the three vacuum-melting practices was evaluated on a relative basis by direct comparison of lightly etched surfaces from each of the four tubing lots from each heat.

(a) CONSUMABLE ELECTRODE VACUUM HEAT 30160. 20% COLD WORK. LOT 42394

(b) CONSUMABLE ELECTRODE VACUUM HEAT 30160, ANNEALED. LOT 42395

(c) INDUCTION VACUUM HEAT 91694. 20% COLD WORK. LOT 42398

(d) INDUCTION VACUUM HEAT 91694. ANNEALED. LOT 42399

(e) INDUCTION VACUUM PLUS CONSUMABLE ELECTRODE VACUUM HEAT 91695. 20% COLD WORK LOT 42402

(f) INDUCTION VACUUM PLUS CONSUMABLE ELECTRODE VACUUM HEAT 91695. ANNEALED. LOT 42403

20 μ m
TUBE AXIS

Figure 1-2. Micrographs of Six Tubing Lots, Midwall Locations in Longitudinal Sections

The tubing from the double vacuum-melted heat appeared to be significantly cleaner than that from either of the single vacuum-melted heats. In comparing the tubing from the two single vacuum-melted heats, it appeared that the inclusions were broken-down to a greater degree in the consumable electrode heat than in the induction vacuum heat, but it did not appear that there was less inclusion content on a volume basis.

Several of the tubing specimens that were examined showed grain boundary precipitates, having the appearance of carbides, near the ID of the tube. This structure was observed only in sections near the tube ends, and only in fine-grained lots; this observation was made at least once for each melting practice but was not a general occurrence.

To evaluate the inclusion content, the source bar stock was also examined. Micrographs at 100 power magnification from longitudinal sections are shown in Figure 1-3. The induction vacuum-remelted stock, heat number 91694, had the highest general inclusion count and contained local areas (not shown), of high inclusion density. The consumable electrode vacuum-remelted stock, heat number 30160, exhibited the second highest general inclusion content and a few local areas of high inclusion density. The double vacuum-remelted stock, heat number 91695, had a slightly lower general inclusion content than heat number 30160 and showed no segregated areas of a high inclusion density in the section examined.

The chemical composition of the inclusions in the double vacuum-melted material was investigated as part of the comprehensive studies, and is treated in a later subsection.

2.4 HARDNESS

Diamond pyramid hardness was measured for the 12 plug drawn tubing lots and the two planetary swaged lots. Seven indentations were placed in the approximate mid-wall position of polished transverse sections of each lot. The loads were 500 grams for the cold worked lots and 300 grams for the annealed lots. The average measured hardness value for each lot is included in Table 1-2 along with the certified properties. For the 12 plug drawn lots, the fabrication variables include three melting practices, two grain sizes, and two fabrication conditions (20 percent cold worked and solution annealed). The data from the as-fabricated lots show that:

1. The six cold worked lots have much higher hardness than the corresponding annealed lots
2. The fine-grained, cold worked lots have slightly higher hardness than the coarse-grained, cold worked lots
3. There is no correlation between hardness and grain size in the annealed lots
4. There is no consistent difference in hardness attributable to type of vacuum-melting practice

a

HEAT 91695 VM (IVCEV) 100X

b

HEAT 30160 VM (CEV) 100X

c

HEAT 91694 VM (IV) 100X

TUBE AXIS

Figure 1-3. ARD VM Bar Stock As-Polished Longitudinal Sections

5. The hardness of the annealed, planetary swaged lot is not significantly different from the hardness of the annealed, plug drawn lots.
6. The cold worked, planetary swaged lot has significantly higher hardness than the comparable plug drawn lots, suggesting a higher level of effective cold work in the planetary swaged material for the same nominal level of cold work, based on percent reduction in area after the last anneal.

2.5 CHARACTERIZATION OF FOUR DOUBLE VACUUM-MELTED TUBING LOTS^(a)

With this general background of characterization information, the four tubing lots listed below were characterized in detail by light microscopy, extraction replica electron microscopy, transmission electron microscopy, X-ray and electron diffraction analysis of extracted precipitates, Laue back reflection analysis, X-ray and pole figure analysis. In addition, the inclusions in tubing lot 42402 and the source bar stock were analyzed in situ by electron micro-probe analysis. All four lots investigated were fabricated from the same double vacuum melted (IVCEV) heat (91695).

1. Plug Drawn Lot 42402, 20 percent Cold Worked Grain Size ASTM 9
2. Plug Drawn Lot 42403, Annealed, Grain Size ASTM 8
3. Planetary Swaged Lot 49193, 20 percent Cold Worked Grain Size ASTM 6
4. Planetary Swaged Lot 49194, Annealed, Grain Size ASTM 6

2.5.1 Metallography^(b)

2.5.1.1 Internal and External Surface Morphology

The internal and external surfaces of the four tubing lots were examined metallographically by slitting and flattening the tubing sufficiently for viewing with vertical reflected illumination. An area about 0.375 inch in diameter was examined from each surface of each sample. Representative micrographs of the plug drawn lots are shown in Figure 1-4. The external surfaces of both the annealed tubing (lot 42403) and the cold worked tubing (lot 42402) had circumferential grooves, probably resulting from surface polishing, and some shallow longitudinal scratches. The circumferential grooves in the cold worked tubing were generally 3 to 5 microns deep with an occasional groove 9 microns deep. Those in the annealed tubing were sharper and somewhat deeper, frequently more than 5 microns and occasionally

^(a) A major part of this subtask was conducted in cooperation with B. Weiss, R. Stickler, and co-workers of the Westinghouse Research Laboratory.

^(b) Westinghouse Advanced Reactors Division Data and Interpretations

INTERNAL SURFACE 500X
ARD VM (IVCEV) TUBING, LOT 42403, ITEM 10, ANNEALED, GRAIN SIZE 8

INTERNAL SURFACE 500X
ARD VM (IVCEV) TUBING, LOT 42402, ITEM 9, 20% CW, GRAIN SIZE 9

TUBE AXIS →

Figure 1-4. Internal and External Surface Structure of As-Received Tubing. Pickling and Surface Polishing were Permitted in the Tubing Specifications

10 microns deep. Both surface polishing and pickling were permitted in the tubing specifications. Review of the suppliers records showed that these lots were surface polished prior to the last draw, but not after.

The internal surfaces of both plug drawn lots appeared to be etched (due to pickling or heat treatment) and both showed longitudinal grooves; the deepest longitudinal grooves in both lots were about 5 microns. The annealed tubing had equiaxed grains, the cold worked tubing elongated grains but not so well delineated and the grain boundary grooves and the general surface roughness were shallower. One cavity-type defect was observed in the internal surface of the annealed tubing. This defect was 450 microns long, 90 microns wide, and 25 microns deep. The longitudinal scratches in both specimens were generally the smear-type and less than 3 microns deep.

The inside and outside surfaces of the as-fabricated, planetary swaged tubing lots showed some differences from the corresponding drawn materials. The outside surfaces of both planetary swaged lots had higher luster than the drawn materials, and the observation of well delineated grain structure on the outside surface of the annealed lot, indicated very little if any surface polishing of the lot. Both lots had shallow circumferential grooves generally less than 3 microns deep which were both narrower and fewer in number than in the drawn materials. Each lot also had a few shallow longitudinal scratches and some very shallow helical scratches.

Both lots also contained angular equiaxed surface cavities up to 10 microns deep on the OD surface, which were not observed in the drawn materials. These cavities were probably the result of spalling, resulting from a non-optimum swaging procedure.

The inside surfaces showed readily observable grain structure in the annealed tubing and indications of a distorted grain structure in the cold worked tubing. The inside surfaces of both lots contained short, shallow longitudinal grooves less than 5 microns deep. The groove frequency was higher in the annealed lot than in the cold worked lot. Scanning electron microscopy examination^(a) of the as-received surfaces of tube lots 49193 and 49194 revealed, in addition to the polishing marks, a fine "crack-like" structure oriented perpendicular to the axial direction of the tube. These cracks probably resulted from too great a deformation for a given ball size in the swaging head.

High magnification light micrographs of electropolished surfaces near the OD, from all four lots, showed small inclusion particles, arranged in rows and stringers. The particle density was similar in the four lots (approximately 1×10^6 particles/cm²).

Except for the one internal surface cavity (lot 42403), the surface defects in the specimens examined were smaller than the smallest notch used in the current FFTF non-destructive test standard.

^(a)At Westinghouse Research Laboratory

2.5.1.2 Inclusion and Second Phase Particles - Extraction Replicas and Bulk Extractions^(a)

Electron micrographs of carbon-extraction replicas (Figure 1-5, a and b) revealed that the extracted particles from the plug drawn lot are polyhedra with an average diameter of less than 0.25 μm . No differences could be detected between the particles extracted from the annealed and cold worked specimens. Occasionally, impressions on the replicas indicated that some larger particles present in the sample had not been extracted. The fine extracted particles were identified by electron diffraction as MC-type carbides.

The weights of the bulk extracted residues were less than 0.5 percent of the specimen weights for both plug drawn tubing lots (42402 and 42403).

X-ray diffraction analyses showed the presence of α -quartz in each material. The MC carbides present in the residues were not observed by X-ray diffraction analysis because of the small particle size.

The planetary swaged lots (49193 and 49194) were also bulk extracted but using more precise quantitative procedures. The extraction weights were 0.13 percent of the tube-weight for both lots. X-ray diffraction analysis of the bulk extraction showed the presence of α -quartz, similar to the plug drawn lots.

Analysis of carbon-extraction replicas by electron diffraction identified some of the adhering particles as MC type carbides. Impressions in the replicas indicated that some of the larger particles present in the specimens had not been extracted.

2.5.1.3 Electron Probe Microanalysis of Inclusions^(b)

Two vacuum melted Type 316 stainless steel source bar stock specimens [heat number 91695 (IVCEV) and heat number 30160 (CEV)], and three specimens from tubing lot number 42402 (IVCEV) (fabricated from heat number 91695) were examined by electron probe microanalysis to determine the composition of the inclusions for elements with atomic number greater than 10. (Because of the difference in experimental procedure between in situ analysis with an electron micro-probe and the diffraction analysis of extracted particles, the two methods may or may not analyze the same inclusions and precipitates depending on the particle size and the chemical resistance to the extracting solution.)

The electron micro-probe analyses were conducted in situ on the inclusions of the type resolvable with a light microscope. Count rate data from the various fluorescent characteristic X-ray lines, relative to the matrix, were the bases of the analysis. The inclusions in the bar stock material were generally one to three microns wide, which was sufficient for obser-

(a) At Westinghouse Research Laboratory

(b) At Westinghouse Advanced Reactors Division

ELECTRON MICROGRAPHS OF CARBON-EXTRACTION REPLICAS NEAR OUTER SURFACE OF TUBE

(a) LOT 42402, PLUG DRAWN, 20% COLD WORKED
TUBE AXIS

(b) LOT 42402, PLUG DRAWN, 20% COLD WORKED

(c) LOT 42402, PLUG DRAWN, 20% COLD WORKED, BACK SCATTERED ELECTRON SCAN, ELECTROLYTICALLY PREPARED SURFACE

ELECTRON
PROBE
ANALYSIS

(d) LOT 42402, PLUG DRAWN, 20% COLD WORKED, S K α X-RAY SCAN OF AREA SIMILAR TO (c), LIGHT CATHODIC VACUUM ETCH

(e) LOT 42402, PLUG DRAWN, 20% COLD WORKED, BEFORE AND AFTER ELECTROPOLISHING, TYPICAL PATTERN FOR INSIDE AND OUTSIDE SURFACE

LAUE BACK-
X-RAY
REFLECTION
PATTERN

(f) LOT 42402, PLUG DRAWN, ANNEALED, AFTER REMOVAL OF 0.5 MILS. FROM OUTSIDE SURFACE OF ANNEALED TUBING LOT 42403

Figure 1-5. Illustrations of Results From Various Characterization Procedures

vation and location in the electron probe in the as-polished condition. The inclusions in the fabricated tubing were much narrower, making electron probe analysis of particles, in situ, more difficult. For tubing specimens, a lightly cathodic etched surface preparation proved to be the best for electron probe analysis, but an electrolytically polished and etched surface was better for microstructural identification and evaluation.

In general, the inclusions in both bar stock and tubing were high in Mn and S and low in Fe, Cr, Ni, and Mo relative to the matrix, indicating that these inclusions are MnS. Several of the most prevalent types of inclusions in each bar stock specimen (viz., gray stringers) were analyzed in detail for elements other than Mn and S in concentrations higher than the matrix. After extensive searching a few inclusions were found which had high Al areas within the Mn and S-rich inclusions. A few inclusions were also found in the single melted bar stock material, heat number 30160 (CEV), which were high in Si and low in Fe, Cr, Ni, Mo, Mn, and S, relative to the matrix.

Similar analysis was also performed on the tubing specimens. Figure 1-5c is a back scattered electron microprobe scan of the electrolytically prepared surface, showing both continuous and discontinuous type stringers (tubing lot number 42402).

Twelve of the continuous type stringers in a similar specimen, but lightly cathodic etched, were conclusively analyzed to be high in Mn and S and low in Fe, Cr, Ni, and Mo relative to the matrix. Figure 1-5d is a SK_Q X-ray scan showing the high S concentration in one of these stringers. Several of these inclusions were further analyzed and showed no elements with atomic numbers greater than 10, other than Mn and S, in concentrations higher than the matrix. Some of the discontinuous stringers in this specimen also analyzed high in Mn and S, whereas others analyzed no different than the matrix.

Combining the information from the various analytical methods, it appears that at least for tubing lot 42402: 1) the continuous stringers are predominately Mn and S, probably in the form of MnS; 2) the small particles (< 0.25 μ m) arranged in rows are predominately MC-type carbides; 3) the α -quartz particles could not be identified in the microstructure with the procedure used; but 4) these α -quartz particles make up the major part of the bulk extractions. The MnS inclusions are probably totally dissolved in the extractive procedure.

2.5.1.4 Residual Stress^(a)

Laue back reflection patterns of the inside and outside surfaces of specimens from all four lots yield two classes of patterns. A spotty ring pattern similar to that shown in Figure 1-5f, characteristic of unstrained material, was obtained from the inside surface of both the annealed, plug drawn lots and the annealed, planetary swaged lot. This type of pattern was also

^(a) At Westinghouse Research Laboratory

obtained from the as-fabricated outside surface of the annealed, planetary swaged lot, indicating little residual surface stress, and for the annealed, plug drawn lot after approximately 0.5 mils were removed from the outside surface by electropolishing. The residual surface stress on the OD in these materials probably resulted from the straightening operation.

The second type of pattern illustrated by Figure 1-5e consists of smooth rings, plus some indication of weak texture. This type of pattern was obtained from the inside and outside surfaces of both cold worked lots and from the as-received outside surface of the annealed, plug drawn lot. This type pattern was also obtained from the cold worked lots after the surface layers were removed by electropolishing.

No distinction could be made between the level of residual stress on the tube surfaces resulting from cold work during tube reduction and from straightening, but the difference between the annealed and the cold worked tubing condition in the bulk of the tube wall was clearly evident.

2.5.1.5 Transmission Electron Microscopy^(a)

Plug Drawn Lots 42402 and 42403

Transmission electron micrographs of the annealed tube sample (lot 42403) revealed a dislocation density of $6 \pm 3 \times 10^8$ dislocation/cm² with the dislocations arranged in rows illustrated by Figure 1-6a. The relatively high dislocation density indicates that the tubes were slightly deformed after the annealing treatment, e.g., during a final straightening operation. In some grains, rows of small (approximately 1000 Å dia.) equiaxed particles were visible.

The width of the dislocation nodes was close to the limit of resolution. Since no widely extended dislocation nodes could be observed,^[18] the stacking fault energy was estimated to be about 20 to 30 erg/cm².

A transmission electron micrograph of the cold worked tube sample (lot 42402) is shown in Figure 1-6c. The high dislocation density made counting impossible but it was assumed^[19] to be of the order of 10^{11} to 10^{12} dislocation/cm². The needle-shaped features observed under the light microscope appeared in the transmission electron micrographs as thin bands of unresolvably fine substructure. From a comparison of observations made on deformed, bulk Type 316 stainless steel samples^[18] it can be inferred that these deformation bands correspond neither to twins nor to the hexagonal epsilon phase, which was reported in Reference 20, 21, and 22 to occur in cold worked stainless steel as a transition phase between austenite and martensite. A detailed analysis of the diffraction contrast of these deformations revealed extinction conditions similar to those reported for stacking faults.^[23] Thus, the deformation bands may be rationalized as clusters of stacking faults produced by slip on closely spaced slip planes.

^(a)At Westinghouse Research Laboratory

(a) LOT NO. 42403,
PLUG DRAWN

(b) LOT NO. 49194,
PLANETARY SWAGED

ANNEALED

(c) LOT NO. 42402,
PLUG DRAWN

(d) LOT NO. 49193,
PLANETARY SWAGED

20% COLD WORKED

Figure 1-6. Transmission Electron Micrographs of Thinned Tube Near Midwall Location

Planetary Swaged Lots 49194 and 49193

The annealed and cold worked planetary swaged lots 49194 and 49193 were similar to the corresponding plug drawn lots described above with respect to dislocation density, inclusion particles, dislocation nodes, and deformation bands. The transmission microstructures are illustrated in Figures 1-6b and 1-6d. There was a very significant difference between the cold worked lots however. In addition to the deformation bands, the cold worked planetary swaged lot also contained micro-twins. In a separate investigation of rolled strip Type 316 stainless steel with various levels of cold work,[13] a few narrowly spaced micro-twins could be detected in the 20 percent cold worked material. Greater amounts of cold work, up to at least 40 percent, increased the number of micro-twins, which greatly facilitated their identification.

Re-evaluation of the transmission microstructures from the tubing specimens led to the following observations and interpretation. The plug, drawn, cold worked lot (42402) contained a few micro-twins and the cold worked, planetary swaged lot contained a higher frequency. Comparing the planetary swaged tubing structure with the rolled strip revealed that with respect to micro-twins, the structure is similar to that exhibited by the 25 to 40 percent cold worked strip.

Since both lots had the same nominal cold work based on reduction of area, these observations were interpreted as further evidence that a higher level of effective cold work results from planetary swaging, relative to plug drawing.

2.5.1.6 Crystallographic Textures of Plug Drawn Tubing^(a)

Crystallographic textures of Type 316 stainless steel, plug drawn tubing lots 42402 and 42403 and the planetary swaged lots 49193 and 49194 were determined by pole figure analyses as part of the comprehensive characterizations. The texture of the tube samples was determined by a conventional X-ray goniometer technique (Siemens Kristalloglex-4 with Siemens texture goniometer), and computer plotting of the measured intensities in the form of (200) and (111) pole figures. Since flat samples are required for this technique, the tube samples were prepared as follows: Approximately one-inch-long pieces from the tubes were electropolished with a perchloric-acetic acid mixture to a remaining wall thickness of approximately 0.005-inch. To achieve a uniform thinning, the tube sample was mounted in the center of a cylindrical stainless steel cathode. The thin-walled tube sample was then longitudinally sectioned by electro-discharge machining (to avoid gross plastic deformation) into four strips which were carefully flattened. The flattened strips were mounted onto a specimen holder to form a composite sheet sample approximately one-inch by one-inch in size.

(a) Work performed by B. Weiss and W. T. Reynolds at Westinghouse Research Laboratory.

The (200) and (111) pole figures for the drawn tubing in the cold worked and the annealed conditions are shown in Figure 1-7, and for the planetary swaged tubing, also in the cold worked and in the annealed conditions, in Figure 1-8. The texture of the drawn tubing (lot 42402, 20 percent cold worked), based on the (200) and (111) pole figures, consists of two components:

COMPONENT 1

$$(\bar{1}\bar{1}2)_{TS}[\bar{1}\bar{1}\bar{1}]_{AD} + (\bar{1}12)_{TS}[\bar{1}1\bar{1}]_{AD} + (1\bar{1}2)_{TS}[1\bar{1}\bar{1}]_{AD} + (112)_{TS}[11\bar{1}]_{AD}$$

$$\text{or } \{112\}_{TS}\langle 111 \rangle_{AD}$$

COMPONENT 2

$$(0\bar{1}1)_{TS}[\bar{1}00]_{AD} + (011)_{TS}[100]_{AD}$$

$$\text{or } \{001\}_{TS}\langle 100 \rangle_{AD}$$

Where TS = Tube Surface
AD = Axial Direction

The $\langle 111 \rangle$ and $\langle 100 \rangle$ directions of this texture correspond to the usual double fiber texture of fcc metals. [24, 25] The $\{112\} \langle 111 \rangle$ and $\{110\} \langle 100 \rangle$ components are found in rolling textures of Fe-Ni alloys. [24] Comparison of the pole figures in Figures 7a and 7b reveals that the textures are qualitatively the same in the cold worked and in the annealed tubing. In the histograms accompanying each pole figure, the fraction of total X-ray count which is reflected from annular regions of 5 degrees by 360 degrees is plotted against alpha, the angle between the reflecting plane and the specimen surface (tube surface). Contours shown in the pole figures represent multiples of reflected intensity from a standard Type 316 stainless steel randomly oriented sample. It is apparent both from the histograms and the contours that the intensity of textural components diminished upon annealing cold worked tubing compared to the cold worked condition.

The texture of planetary swaged tubing (lot 49193, 20 percent cold worked) has two texture components which are similar:

COMPONENT 1

$$(\bar{1}01)_{TS}[\bar{1}\bar{1}\bar{1}]_{AD} + (101)_{TS}[\bar{1}\bar{1}1]_{AD}$$

Figure 1-7. Plug Drawn Type 316 Stainless Steel Tubing

(200) POLE FIGURES

(111) POLE FIGURES

Figure 1-8. Planetary Swaged, Type 316 Stainless Steel Tubing

COMPONENT 2

$$(011)_{TS}[\bar{1}\bar{1}1]_{AD} + (0\bar{1}1)_{TS}[1\bar{1}\bar{1}]$$

These can be expressed simply as

$$\{010\}_{TS} \langle 111 \rangle_{AD}$$

This texture differs from the $\{110\}_{TS} \langle 100 \rangle_{AD}$ component of the drawn tubing by a ± 45 -degree rotation of $\langle 010 \rangle$ from the axial direction. Such rotation probably results from the helical path of tensile deformation in planetary swaged tubing in contrast to the axial tensile deformation of drawn tubing. The $\{112\}_{TS} \langle 111 \rangle_{AD}$ component, seen in the drawn tubing, is not apparent in the swaged tubing texture.

Planetary swaged tubing in the annealed conditions (lot 49194) has a broad, low intensity texture, essentially the same as that of the cold worked tubing ($\{110\}_{TS} \langle 111 \rangle_{AD}$). These textures are compared in Table 1-4 with the results of texture analysis of 0.2 percent Ti Type 304L stainless steel tubing reported by Nilsson; [26] equivalent notation is used. Note that the $[112]_{RD} [111]_{AD}$ component was not observed in the drawn Type 304 stainless steel. [26] The $[011]_{RD} [100]_{AD}$ component occurred in both Type 316 and Type 304L drawn tubing (this texture is equivalent to $[110]_{RD} [001]_{AD}$ used in the description of the 304L tubing).

Textures of the planetary swaged, Type 316 stainless steel tubing differed significantly from that of the planetary swaged Type 304L. In addition to the differences in chemistry, there were also differences in the fabrication schedules which probably influenced the textures obtained. The Type 316 stainless steel had only the last 20 percent reduction by planetary swaging. The "Q" value (percent reduction in wall thickness/percent reduction in tube diameter) was 0.6. This specific schedule was selected to obtain as direct a comparison as practicable with the Westinghouse plug drawn materials being investigated. The Ti modified Type 304 stainless steel ORNL tubing, on the other hand, had about 40 percent reduction by planetary swaging, achieved in four passes, with an overall Q value of 0.35, which corresponds to relatively less tube wall reduction and more tube sinking.

2.5.1.7 Analysis of Precipitates After Heat Treatment

As part of the characterization study of the cold worked lots, and to provide specimens to study the effects on mechanical properties of the heat treatment described by Garofalo, [27] specimens of plug drawn lot 42402 and planetary swaged lot 49193 were heat treated 900°F for 24 hours plus 1300°F for 216 hours in vacuum. Metallography and analysis of extracted precipitates after heat treatment showed a higher frequency of precipitates and a greater tendency for precipitation on deformation bands and twins in the planetary swaged tubing than in the plug drawn tubing. This again indicates

Table 1-4. Textures of Stainless Steel Tubing				
Deformation Process	ARD Type 316 Tubing		0.2% Ti Type 304L Tubing	
	Condition	Textures	Condition	Texture
drawing	CW 20% annealed	$[112]_{RD}[11\bar{1}]_{AD}$ $[011]_{RD}[100]_{AD}$	_____	_____
drawing (ironing)	_____	_____	CW 29% annealed	$[110]_{RD}[001]_{AD}$
drawing (sinking)	_____	_____	CW 27% annealed	$[110]_{RD}[001]_{AD}$ $[111]_{RD}[\bar{1}2\bar{1}]_{AD}$ $[111]_{RD}[1\bar{2}1]_{AD}$
planetary swaging	CW 20% annealed	$[011]_{RD}[11\bar{1}]_{AD}$	_____	_____
planetary swaging	_____	_____	CW 23% annealed	$[001]_{RD}[110]_{AD}$ $[111]_{RD}[\bar{1}2\bar{1}]_{AD}$ $[111]_{RD}[1\bar{2}1]_{AD}$
RD = Radial Direction			AD = Axial Direction	

a higher level of effective cold work in the planetary swaged tubing. Representative scanning electron micrographs are shown in Figure 1-9a and b, and electron micrographs of carbon extraction replicas are shown in Figure 1-9c and d.

To determine the nature of the phases present, bulk extractions were made and the residues examined by X-ray diffraction. The weights of the bulk extracted residues were about 1.69 percent for planetary swaged lot (49193-G) and 1.29 percent for plug drawn lot (42402-G). The phases identified were approximately equal quantities of $M_{23}C_6$ and Fe_2Mo (Laves). In addition to these, a small amount of σ -phase was found in the planetary swaged, heat treated material (49193-G).

(a) AGED, LOT 42402

10 μm

(b) AGED, LOT 49193

10 μm

SCANNING ELECTRON MICROGRAPHS

(c) $M_{23}C_6$ + Fe_2Mo
abundant abundant
AGED, LOT 42402

1 μm

(d) $M_{23}C_6$ + Fe_2Mo + σ
abundant abundant weak
AGED, LOT 49193

1 μm

PHASES IDENTIFIED BY X-RAY DIFFRACTION OF BULK EXTRACTED RESIDUE

TRANSMISSION ELECTRON MICROGRAPHS OF EXTRACTION REPLICAS

Figure 1-9. Microstructures of Thermally Aged (900°F/24 hr Plus 1300°F/216 hr) 20% Cold Worked Double Vacuum Melted Stainless Steel Tubing Fabricated by Plug Drawing (Lot 42402) and by Planetary Swaging (Lot 49193)

2.5.1.8 Summary of Comprehensive Characterization

The results of the comprehensive characterizations of the two plug drawn lots and the two planetary swaged lots are summarized in Table 1-5. In comparing the plug drawn lots with the planetary swaged lots there are differences in grain sizes and crystallographic texture. There were also several differences between the cold worked lots which indicate that planetary swaging produces a higher level of effective cold work than plug drawing for the same percent reduction in cross sectional area. The cold worked, planetary swaged lot:

Table 1-5. Summary of Characterization Results

Characteristic	Plug Drawn Tubing		Planetary Swaged Tubing	
	20% Cold Worked Lot 42402	Annealed Lot 42403	20% Cold Worked Lot 49193	Annealed Lot 49194
1. MICROSTRUCTURE				
Grain Size ASTM No.	9	8	6	6
Grain Morphology	Slightly elongated, twins and needle-like features	Equiaxed and twins	Slightly elongated, twins and needle-like structure	Equiaxed and twins
Hardness Diamond Pyramid			325	131
Dislocation Arrangement	Heavy dislocated microstructure with deformation bands	Majority arranges in rows	Heavy dislocated structure with numerous complex faults and microtwins	Some arranged in rows
Dislocation (dislocations/cm ²)	$\sim 10^{11} - 10^{12}$	$6 \pm 3 \times 10^8$	$\sim 10^{11} - 10^{12}$	$6 \pm 3 \times 10^8$
Stacking Fault Energy (erg/cm ²)	Not measurable from TEM's	~ 25	Not measurable from TEM's	~ 25
2. HARDNESS	273 DPH (500 g)	148 DPH (300 g)	325 DPH (500 gm)	131 DPH (300 gm)
3. SECOND PHASE PARTICLES				
Arrangement	Same	Aligned into continuous and discontinuous stringers parallel to tube axis	Same	Aligned into stringers parallel to tube axis
Amount	Same	$1 \pm 0.5 \times 10^6$ particles/cm ²	—	Approx. 10^6 particles/cm ²
Identification bv ED(Extraction replica) bv XD(Bulk extraction) bv EPA (In Situ inclusions)	Same Same Continuous and some discontinuous inclusions of size observable with light microscope, high in Mn and high in S	MC $\alpha\text{-SiO}_2$ (<0.5 w/o)	Same —	$\alpha\text{-SiO}_2$ (<0.13 w/o)
4. RESIDUAL STRESS				
Outer Surface	Cold worked	Cold worked	Cold worked	Strain free
Inside Surface	Cold worked	Strain free	Cold worked	Strain free
After removal of 0.0005 in. from OD	Cold worked	Strain free		
5. TEXTURE ^(a)	$\{112\}_{TS} \langle 111 \rangle_{AD}$ $\{011\}_{TS} \langle 100 \rangle_{AD}$	Same as cold worked but weaker	$\{011\}_{TS} \langle 111 \rangle_{AD}$	Same as cold worked but weaker
6. PRECIPITATES AFTER HEAT TREATMENT 900°F/24 HR + 1300°F/216 HR	1.29 w/o $M_{23}C_6 + Fe_2Mo$	—	1.69 w/o $M_{23} + \text{Laves } Fe_2Mo + \text{weak } \sigma$	—
TS = Tube Surface AD = Axial Direction			(a) {plane tangential to OD} <direction parallel to tube axis>	

1. Had higher hardness
2. Contained a higher frequency of micro-twins than the plug drawn tubing, which was shown to be a characteristic of a higher level of cold work
3. Showed both higher frequency and higher weight fraction of precipitates for comparable heat treatment at elevated temperature.

Other than the differences in grain size and texture there was little difference between the annealed tubing lots, except that the planetary swaged material did not appear to be surface polished on the OD to the same extent on the plug drawn tubing.

SECTION 3

MECHANICAL PROPERTY TESTING

3.1 BIAXIAL TENSILE TESTING

Various tubing lots were tensile tested at room temperature and five elevated temperatures under biaxial stress conditions to evaluate short-term mechanical properties.

3.1.1 Experimental Procedure

The tests were conducted in an air atmosphere, using argon for the internal gas pressure; the argon was supplied from a gas compressor, gas receiver, and appropriate controls. The diametral strain was measured, using an LVDT and recorded along with the load using an X-Y recorder capable of adjustment for high and low magnification. The specimen heating rate was adjusted to reach the nominal test temperature within 20 minutes, at which time the test was commenced. Temperature was controlled to $\pm 3^{\circ}\text{F}$ throughout the test, and the load rate was adjusted between limits of 1900 lb/min and 3400 lb/min so that the test was completed in less than 30 minutes total elapsed time. The specimens were 6 inches long, and sealed into the system with Swage-lock or Gyro-lock fittings.

The reported mechanical property values were obtained as follows: The yield strength was determined by the 0.2 percent offset method using the load versus strain diagram (high magnification) and the thin walled hoop stress formula:

$$\sigma_h = \frac{P D}{2t}$$

where

P = internal pressure

D = initial mid wall diameter

t = initial wall thickness

The ultimate tensile strength was calculated from the maximum load attained in testing, the initial tubing dimensions, and the thin-walled hoop stress formula.

The uniform diametral strain was obtained from the low magnification load-strain curve. The difference between the diameter at offset yield and the diameter at the drop off of load divided by the original outside diameter was taken for the uniform diametral strain $\Delta D/D$ in percent.

3.1.2 Results

The results of the biaxial tensile tests for eight lots of tubing, tested at room temperature and five elevated temperatures, are given in Table 1-6. These eight lots were selected to show the effects of the major material variables in the program, namely: melting practice, grain size, annealed and cold worked condition, plug drawing, and planetary swaging.

The effects of the major material variables on yield strength and uniform elongation are shown in Figures 1-10 for various combinations of variables. The relative effects of the material variables on the ultimate strengths were very similar to the effects in the yield strength, except for the planetary swaged material which will be discussed later.

A comparison of the three vacuum melting practices for the fine-grained, 20 percent cold worked condition is given for lots 42393, 42398, and 42402 in Table 1-6, and shown graphically in Figure 1-10a and b. The double vacuum-melted lot has slightly higher ultimate and yield strengths, and lower ductility than the single vacuum-melted lots at 1300°F. However, considering the whole temperature range, there is little difference in biaxial tensile properties attributable to vacuum-melting practice. The 1200°F ductility for the induction vacuum-melted heat appears to be inconsistent with the rest of the data; however, no reason was found to explain this apparent discrepancy.

Comparison of results from fine-grained and coarse-grained, plug drawn tubing with the results from coarse-grained planetary swaged tubing, all in the 20 percent cold worked condition, is given for lots 42402, 42404, and 49193 in Table 1-6, and shown graphically in Figure 1-10 c and d. The fine-grained, plug drawn lot has higher ultimate strength, higher yield strength, and lower ductility than the coarse-grained, plug drawn material at all temperatures. The results for the coarse-grained, planetary swaged lot are generally similar to the fine-grained, plug drawn lot at the lower temperatures, and intermediate between the coarse and fine-grained, plug drawn lots at the highest temperature (1300°F).

These results indicate that planetary swaging has a significant effect in raising the yield strength compared to plug drawing at the same grain size. Two possible reasons are suggested from the results of the characterization program: 1) planetary swaging introduces a different crystallographic texture than plug drawing, and 2) for 20 percent nominal cold work, the planetary swaged lot had a higher degree of effective cold work than 20 percent cold worked, plug drawn lot, based on hardness, microstructure, and precipitation characteristics. In view of the results given below for the annealed condition, the more likely cause is a higher level of effective cold work.

Table 1-6. Biaxial Tensile Results from Various Heats of ARD, VM Type 316 SS Tubing											
Lot	Melting Practice	Certified ASTM Grain Size	Condition	Pretest Hardness DPH	Mechanical Properties						
					Property	RT	850°F	1000°F	1100°F	1200°F	1300°F
42394 Item 1	CEV	9/9.5	PD 20% CW	276	a	148.3	113.9	106.0	99.6	85.9	72.3
					b	138.6	109.6	101.7	--	--	59.8
					c	1.2	0.6	0.8	0.78	0.8	3.2
42398 Item 5	IV	9	PD 20% CW	278	a	148.3	115.3	108.9	99.7	85.9	74.4
					b	139.3	108.9	106.0	94.9	77.7	62.0
					c	1.5	0.8	0.7	--	1.61	2.91
42402 Item 9	IVCEV	9	PD 20% CW	273	a	141.9	114.0	106.4	100.5	85.9	79.0
					b	136.2	106.8	103.9	96.4	81.3	70.2
					c	1.2	0.7	0.9	1.0	0.9	1.8
42403 Item 10	IVCEV	8	PD Annealed	148	a	79.4	59.8	55.2	51.6	51.1	42.8
					b	53.4	36.6	34.0	31.5	30.4	26.5
					c	--	16.1	--	9.1	10.0	18.3
42404 Item 11	IVCEV	5.5/6	PD 20% CW	261	a	132.0	103.6	96.8	90.6	76.0	69.3
					b	126.5	98.0	93.2	85.6	71.0	62.2
					c	2.7	1.5	1.5	2.4	1.7	3.7
42405 Item 12	IVCEV	5/5.5	PD Annealed	138	a	76.5	53.7	51.6	49.9	46.9	40.4
					b	49.8	29.7	27.7	26.3	24.2	23.8
					c	56.0	15.2	17.4	14.7	--	--
49193 Item 19	IVCEV	6	Planetary Swaged 20% CW	325	a	--	112.7	106.2	103.8	84.6	73.6
					b	--	107.1	100.7	97.6	75.4	63.8
					c	--	1.0	0.83	1.1	2.2	2.4
49194 Item 20	IVCEV	6	Planetary Swaged Annealed	131	a	79.5	54.6	51.6	49.8	45.9	43.3
					b	45.8	21.6	20.7	18.9	18.9	17.1
					c	--	18.0	16.5	15.1	15.6	16.1
DPH Diamond Pyramid Hardness			Mechanical Properties				IV - Induction Vacuum Melt				
Average of Seven Measurements			a Ultimate Tensile Strength (ksi)				CEV - Consumable Electrode Vacuum Melt				
500 gram load - 20% CW			b 0.2% Yield Strength (ksi)				IVCEV - Induction Vacuum plus Consumable				
300 gram load - Annealed			c Uniform Diametral Strain (%)				Electrode Vacuum Melt				
							-- - Mechanical Difficulties Nullified Test

Figure 1-10. Biaxial Tensile Results, Gas Pressurized Burst Tests

The results from the same combination of variables of plug drawing, planetary swaging, and grain size are given for the annealed condition lots 42403, 42405, and 49194, in Table 1-6 and Figure 1-10 d and e. The fine-grained, plug drawn lot has higher ultimate and yield strength at all temperatures than the coarse-grained, plug drawn lot and lower ductility at the intermediate temperatures 1100 and 1200°F. The coarse-grained, planetary swaged lot has approximately the same ultimate strengths as the coarse-grained, plug drawn lot, but lower yield strengths at all temperatures. The ductilities are about the same for the three temperatures for which data have been obtained (850, 1000, and 1100°F).

Since these three tubing lots are in the annealed condition, have approximately the same hardness, and were fabricated from the same double vacuum-melted heat, it appears that the lower yield strength is associated with the planetary swaging, rather than grain size. The most likely factor in this case is the crystallographic texture, discussed earlier, which is developed by planetary swaging.

In comparing the results from annealed and cold worked, planetary swaged lots relative to the plug drawn lots with the same grain size and fabricated from the same double vacuum-melted heat, it appears that planetary swaging produces a crystallographic texture conducive to lower yield strength in the annealed condition. However, since planetary swaging produces a higher level of effective cold work for the same amount of nominal cold work, this overshadows the texture effect and results in higher yield strengths than for comparable plug drawn material in the cold worked condition.

3.2 Biaxial Tensile Tests Under LMFBR Conditions

The temperature range of interest in LMFBR application is within the precipitation range for Type 316 stainless steel. Based on results obtained by Stickler, Weiss and co-workers^[6,11] significant precipitation occurs in a very short time at 1200 and 1300°F (illustrated in Figure 1-11) for air-melted Type 316 stainless steel, annealed tubing. After a short time at these temperatures the as-fabricated tensile properties could be markedly changed, consequently, the as-fabricated short-term tensile properties must necessarily be used with judgement for service application.

In addition to the inherent precipitation effects, the tensile properties measured may also be sensitive to the test conditions, especially at elevated temperatures. Time is required both to reach the test temperature and to conduct the test, during which time the properties could be changing. The possibility of this effect is also illustrated by Figure 1-11, in which the time-temperature combinations of the biaxial tensile tests are superimposed on the time-temperature-precipitation curves for two lots of annealed Type 316 stainless steel. It can be seen that at 1300°F carbide precipitation has started before the tensile tests are completed and at 1200°F precipitation may be just initiating.

TIME-TEMPERATURE COMBINATIONS OF THE BIAXIAL TENSILE TESTS INDICATED BY CIRCLES

Figure 1-11. Time-Temperature-Precipitation Diagrams for Two Solution Annealed ARD Air-Melted Tubing Lots

To provide information to aid in making engineering decisions relative to the application of short-term mechanical properties, two series of biaxial tensile tests were conducted, one on various heat-treated materials for comparison with as-received material, and the second series to evaluate the effect of the test time in the standard elevated biaxial tensile testing procedures. These tests also permitted partial evaluation of the heat treatment schedule reported by Garofalo^[27] to improve stress rupture properties, and are described in the following paragraphs.

3.2.1 Heat Treated Materials

The heat treatments, listed below, were given to the double vacuum-melted, fine grained, 20 percent cold worked tubing lot (42402) previously tested in the as-received condition.

1. 24 hours at 900°F vacuum
2. 24 hours at 1300°F vacuum
3. 216 hours at 1300°F vacuum
4. 24 hours at 900°F + 216 hours at 1300°F vacuum (Garofalo treatment)

Tensile, offset yield, and elongation results for these materials are given in Table 1-7. The yield strengths and the uniform diametral strains are shown in Figure 1-12 a and b, along with results reported previously for the as-received cold worked lot and the comparable annealed lot.

Table 1-7. Biaxial Tensile Results for Heat Treated Type 316 SS Tubing

Lot	Melting Practice	Pretreatment Condition	Heat Treatment	Pretest Hardness DPH	Mechanical Properties						
					Property	RT	850°F	1000°F	1100°F	1200°F	1300°F
42402 Item 9 H.T.	IVCEV	20% CW G.S. 9	24 Hr @ 900°F	272	a	134.7	105.0	99.6	94.5	88.2	76.7
					b	114.1	96.4	93.2	90.3	82.4	69.5
					c	5.5-11	2.9	2.6	1.4	1.1	--
42402 Item 9 H.T.	IVCEV	20% CW G.S. 9	216 Hr @ 1300°F	230	a	108.9	81.0	76.7	73.2	68.6	64.3
					b	91.7	69.4	66.4	64.5	61.0	56.9
					c	--	5.0	4.3	4.0	3	3
42402 Item 9 H.T.	IVCEV	20% CW G.S. 9	24 Hr @ 900°F +216 Hr @ 1300°F	232	a	110.2	82.1	77.4	74.3	69.4	66.1
					b	94.2	69.4	66.6	65.7	62.8	57.5
					c	6.9	4.1	4.3	3.1	3.7	3.3
DPH Diamond Pyramid Hardness			Mechanical Properties			IV - Induction Vacuum Melt					
Average of Seven Measurements			a Ultimate Tensile Strength (ksi)			CEV - Consumable Electrode Vacuum Melt					
500 gram load - 20% CW			b 0.2% Yield Strength (ksi)			IVCEV - Induction Vacuum plus Consumable					
300 gram load - Annealed			c Uniform Diametral Strain (%)			Electrode Vacuum Melt					
						-- - Mechanical Difficulties Nullified Test

Figure 1-12. Biaxial Tensile Results for Pre-Test Heat Treated, Type 316 Stainless Steel

strengths and increases in elongations relative to the as-received, cold worked material. The effects were more pronounced at the lower test temperature. The differences in biaxial tensile properties between these two heat-treated lots were small. The Garofalo treated material had slightly higher yield and ultimate strengths at all test temperatures and higher elongations at the 1200 and 1300°F. The greatest difference in properties was in the elongation at test temperatures below 1100°F.

It can be seen from these results that treatment as short as 216 hours at 1300°F has a marked effect on the short-term biaxial tensile properties of cold worked material, and that direct use of as-fabricated properties is not valid for any but the shortest-term applications at this temperature.

The data show that in general the heat treated, cold worked lots have properties intermediate between the as-received cold worked material and the solution annealed material. The specific effect of the post-fabrication heat treatments on the fine-grained, 20 percent cold worked, vacuum-melted material, was to decrease the yield and the ultimate strengths, especially at the lower temperatures, and to increase the elongations relative to as-received, cold worked material.

The 24 hour heat treatment at 900°F reduced both the ultimate strength and yield strength significantly at temperatures up to 1100°F. At the higher test temperatures of 1200 and 1300°F, the heat-treated tubing had approximately the same ultimate and yield strengths as the non-heat-treated tubing.

A single test point obtained at 1200°F, for the specimen heat treated 20 hours at 1300°F prior to test, was intermediate between the 24 hour treatment at 900°F and the 210 hour treatment at 1300°F.

Both the 216-hour treatment at 1300°F and the Garofalo type treatment of 24 hours at 900°F, plus 216 hours at 1300°F resulted in marked reductions in yield and ultimate

3.2.2 Variation in Hold Time at Temperature

In addition to the standard tests, a limited number of biaxial tensile tests were conducted at 1200°F in which hold-time prior to test was varied between 10 and 240 minutes to determine the effect of this variable on the measured results. The hold-time at temperatures before and during loading in the customary procedure for this laboratory is between 10 and 30 minutes.

The tensile, offset yield and elongation results for solution annealed tubing (lot 42403), for 20 percent cold worked tubing (lot 42402), and for specimens from each lot heat treated 24 hours at 1300°F prior to test are given in Table 1-8. Also included in the table are the pre-test room temperature hardness values and the tensile results previously obtained at 1200°F from the two as-fabricated lots, using the customary test procedure.

The data in Table 1-8 for the as-received, annealed tubing and the heat-treated, annealed tubing show that variation in pre-test hold time from 10 to 240 minutes had essentially no effect on measured biaxial tensile properties. In addition, pre-test heat treatment of this lot for 24 hours at 1300°F caused only a small reduction in strength relative to the non-treated lot as described previously, and the measured difference in ductility 12.1 to 11.7 percent (3.3 percent change) may not be significant.

The cold worked tubing, however, behaved differently. For hold times between 10 and 30 minutes there was little change in measured properties, at 30 minutes there was a slight change, but beyond 30 minutes there was reduction in both the ultimate and the yield strengths and slight change in diametral strain. Pre-test heat treatment of the cold worked tubing at 1300°F for 24 hours caused a significant drop in both ultimate and yield strengths and a slight increase in ductility.

Previously described elevated temperature biaxial tensile data indicated that annealed materials has a ductility minimum at about 1100 to 1200°F (Figure 1-10). Initially, it was believed that this may be due to a structure change occurring during the test. If the reduction in elongation is, in fact, due to a structure change occurring during test, then this change must occur in less than 10 minutes hold time at 1200°F and not change much thereafter (up to 4 hours at 1200°F).

3.3 Biaxial Tensile Tests Summary

The results from the biaxial tensile tests may be summarized as follows:

1. The biaxial tensile testing procedure appears to be satisfactory in that hold time at temperature and the difference in hold time allowed by the procedure are sufficiently short causing little change in material properties within the time limits of the test (at least at 1200°F).

Table 1-8. Effect of Hold Time at Test Temperature on Biaxial Tensile Results at 1200°F

Lot	Practice	Pretreatment Condition	Heat Treatment	Pretest Hardness DPH	Property	Hold Time Before Test at 1200°F			
						10 Min.	30 Min.	120 Min.	240 Min.
42403 Item 10	IVCEV	Annealed G.S. 8	As-Fabricated	148	a b c	49.8 29.5 12.1	49.2 29.2 11.9	49.4 30.1 --	49.2 28.7 12.1
42403 Item 10	IVCEV	Annealed G.S. 8	24 Hr @ 1300°F	158	a b c	47.4 26.1 12.1	-- -- --	46.4 25.4 11.7	-- -- --
42402 Item 9	IVCEV	20% CW G.S. 9	As-Fabricated	273	a b c	84.1 80.2 1.7	83.9 79.6 1.8	79.2 71.7 2.2	78.2 69.9 1.7
42402 Item 9	IVCEV	20% CW G.S. 9	24 Hr @ 1300°F	235	a b c	71.8 66.3 2.3	-- -- --	70.4 64.6 2.3	-- -- --
						Standard Test Procedure			
						Property		10-30 Min.	
42403 Item 10	IVCEV	Annealed G.S. 8	As-Fabricated	148		a b c		51.1 30.4 10.0	
42402 Item 9	IVCEV	20% CW	As-Fabricated	273		a b c		85.9 81.3 0.9	
DPH Diamond Pyramid Hardness Average of Seven Measurements 500 gram load - 20% CW 300 gram load - Annealed			Mechanical Properties a Ultimate Tensile Strength (ksi) b 0.2% Yield Strength (ksi) c Uniform Diametral Strain (%)		IV - Induction Vacuum Melt CEV - Consumable Electrode Vacuum Melt IVCEV - Induction Vacuum plus Consumable Electrode Vacuum Melt -- - Mechanical Difficulties Nullified Test				

2. The temperature range of interest in LMFBR application is within the precipitation range for Type 316 stainless steel, and as-fabricated short-term properties should be used with judgement.
3. There is little difference in short-term biaxial tensile properties attributable to melting practice.
4. For plug drawn, 20 percent cold worked, double vacuum-melted Type 316 stainless steel, fine-grained tubing (ASTM9) has higher ultimate strength, higher yield strength, and lower ductility than coarse-grained tubing (ASTM 5.5/6) at all temperature tested.
5. Cold worked tubing has higher ultimate and yield strengths, and lower ductilities than annealed tubing for all combinations tested.
6. Cold worked, planetary swaged tubing has higher ultimate and yield strengths and generally lower ductility than cold worked, plug drawn tubing with the same grain size, fabricated to the same nominal cold work based on percent reduction in area.
7. The coarse-grained, annealed planetary swaged tubing has about the same ultimate strength as the coarse-grained, plug drawn lot, but the yield strengths are lower at all temperatures. The ductilities were about the same for the three temperatures for which data were obtained (850, 1000, and 1100°F).
8. Comparing results for annealed and cold worked, planetary swaged lots to the plug drawn lots with the same grain size, all fabricated from the same double vacuum melted heat, it appears that planetary swaging produces a crystallographic texture conducive to lower yield strength in the annealed condition. However, planetary swaging also produces a higher level of effective cold work for the same amount of nominal cold work, and this overshadows the texture effect and results in higher yields than comparable plug drawn material in the cold worked condition.
9. The effect of heat treatment of cold worked, plug drawn tubing in the precipitation range is to reduce the ultimate and the yield strengths and to increase ductility.
10. The effect on short-term tensile properties of heat treatment of cold worked tubing, first at low temperature (900°F/24 hour), followed by higher temperature (1300°F/216 hours) is about the same as the high temperature treatment alone.

3.4 BIAxIAL STRESS RUPTURE TESTING

Various lots of the vacuum-melted tubing were also stress rupture tested under biaxial conditions to evaluate the effects of the major variables; namely, vacuum-melting practice, grain size, cold worked and annealed conditions, and plug drawing and planetary swaging fabrication procedures. As with other tests, the 20 percent cold worked, fine grained, plug drawn tubing was considered as the reference tubing and was used as a baseline.

3.4.1 Experimental Procedure

The tests were conducted in air atmosphere. The specimens were internally pressurized with argon supplied from a high pressure gas cylinder pressures of 5000 psi or less, and from a gas compressor and receiver for pressures exceeding 5000 psi. Appropriate regulators and gages were included in the system. Internal gas pressure was maintained at ± 5 psig of the nominal test pressure during test. A timer was started when the specimen reached temperature equilibrium; thereafter, the test was timed automatically and shut off at burst or when a pressure drop greater than 100 psig occurred.

Temperature was controlled to within 20°F of the nominal test temperature and measured throughout test with a thermocouple attached to the specimen; the temperature variation from the control temperature was about $\pm 3^\circ\text{F}$.

The nominal temperatures were 1200 and 1300°F. The thin wall hoop stress formula, given on page 1-29 was used with the original tube dimensions to calculate the engineering hoop stress of the test.

Test specimens were nine inches long, contained internal mandrels to minimize stored energy, and were fitted with a solid end plug, welded onto one end, and a hollow end plug on the other. Specimens were sealed into the system with high pressure. Swage-lock fittings, and data were reported in the form of standard plots of logarithmic engineering hoop stress versus logarithmic rupture time.

The data were normalized to the nominal test temperature using the "m" factor of the Chitty-Duval parameter^[28] (there is a linear relationship between m and stress). The m-values that were used were taken from a standardized Chitty-Duval plot of stress versus m for Type 316 stainless steel. The Chitty-Duval parameter "d" is defined as:

$$d = T - m \log t$$

where:

T = temperature (°C)

$$m = \frac{dT}{d \log t}$$

t = time (hr.)

The analysis of the data using this technique has been described previously in detail.^[7] It was shown as part of this investigation, that the m-factor, relating temperature and rupture time, could be used to normalize the data obtained at measured temperatures, (within 20°F of the nominal 1300°F test temperature) to equivalent 1300°F rupture times. This approach was used to reduce the scatter in the raw data due to the variation from the nominal temperature resulting from position in the multiple specimen stress-rupture furnaces.

The m-factor was also used to estimate rupture times at 1200°F from 1300°F data in order to select test conditions for testing at 1200°F. Reasonably good agreement was obtained with this estimate over the 100°F range, providing comparable segments of the stress-rupture lines were compared.

3.4.2 Results

The stress rupture data obtained from 112 tests at 1200 and 1300°F nominal test temperature are given in Table 1-9. These data were obtained from 10 lots of tubing selected to compare the major variables, and in addition from several heat treated specimens and from several post fabrication micro-peened specimens.

Table 1-9. Stress Rupture Data								
Item	Lot	Material Condition	Nom. Temp. (°F)	Test Temp. (°F)	Stress (ksi)	Rupture Time (hr)	Normalized Rupture Time (hr)	Rupture Type
1	42394	CEV-20% CW-FG	1300	1291	26	104.7	74.2	pinhole
				1295	24	129.9	108.0	pinhole
				1294	18	588.0	482.2	pinhole
				1315	15.5	585.8	935.1	pinhole
				1313	14	852.0	1259	pinhole
				1295	13.5	1300.3	1120	pinhole
				1291	12	2313.2	1796.5	pinhole
				1303	10	2734.4	2957.0	pinhole
1	42394	CEV-20% CW-FG	1200	1193	30	1683.9	1268.6	pinhole
				1211	29	1651.7	2559.6	pinhole
				1213	27	1382.3	2287.4	split
2	42395	CEV-ANN-FG	1300	1295	14	696.1	601.0	pinhole
3	42396	CEV-20% CW-FG	1300	1305	30	33.7	41.2	split
				1293	28	89.7	68.1	pinhole
				1299	24	185.2	178.5	pinhole
				1303	22	392.2	436.6	pinhole
				1290	20	514.2	364.3	pinhole
5	42398	IV-20% CW-FG	1300	1286	30	34.6	19.6	pinhole
				1286	24	266.1	158.5	split
				1290	20	508.9	361.1	pinhole
				1296	19	746.2	652.3	pinhole
				1317	17.7	336.0	589.0	pinhole
				1297	14	1369.1	1253.5	pinhole
				1315	12	1109.4	1690.9	pinhole
				1301	10	2445.9	2510.3	pinhole

Table 1-9. Stress Rupture Data (continued)

Item	Lot	Material Condition	Nom. Temp. (°F)	Test Temp. (°F)	Stress (ksi)	Rupture Time (hr)	Normalized Rupture Time (hr)	Rupture Type
5	42398	IV-20% CW-FG	1200	1214	30	676.5	1192.0	pinhole
				1187	29	1866.7	1112.3	pinhole
				1194	27	2082.2	1650.2	pinhole
7	42400	IV-20% CW-CG	1300	1285	30	42.3	23.0	pinhole
				1300	28	49.0	49.0	pinhole
				1295	26	80.5	66.5	split
9	42402	IVCEV-20% CW FG	1300	1313	30	26.6	45.0	split
				1294	27	140.2	111	pinhole
				1286	24	340.2	202	multiple pieces
				1294	24	395.6	317	split
				1293	22	588.8	459	split
				1295	22	365.0	305	split
				1308	20	685.3	901	pinhole
				1312	20	341.8	516	split
				1314	18	557.3	883.1	pinhole
				1318	17	592.2	1057.3	pinhole
				1314	15	805.4	1239.5	pinhole
				1303	12	2367.6	2575.7	pinhole
				1301	10	3446.5	3537.3	pinhole
9	42402	IVCEV-20% CW FG	1200	1201	40	51.1	53.4	multiple pieces
				1207	37	68.9	93.7	multiple pieces
				1185	33.5	1801.3	956.4	pinhole
				1198	33	731.0	672.0	split
				1213	29	938.9	1575.6	split
				1213	26	1821.7	2992.0	pinhole
				1214	24	2124.2	2563.1	pinhole
10	42403	IVCEV-ANN FG	1300	1316	25	7.6	8.1	multiple pieces
				1295	21	138.1	115.9	split
				1290	20	113.3	80.2	split
				1303	17.7	203.9	216.2	split
				1295	16	328.0	280.1	pinhole
				1292	15	702.5	664.7	split
				1313	13	867.0	1264.9	pinhole
				1301	11.5	2423.9	2425.0	pinhole
				1316	10	3533.0	5362.8	pinhole

Table 1-9. Stress Rupture Data (continued)								
Item	Lot	Material Condition	Nom. Temp. (°F)	Test Temp. (°F)	Stress (ksi)	Rupture Time (hr)	Normalized Rupture Time (hr)	Rupture Type
10	43403	IVCEV-ANN FG	1200	1203	32	7.7	8.7	multiple pieces
				1203	30	20.0	22.5	multiple pieces
				1211	26	59.5	90.5	split
				1213	25	133.0	216.8	split
				1197	23	718.5	672.8	split
				1199	21	1513.1	1461.0	split
11	42404	IVCEV-20% CW CG	1300	1295	29	69.5	56.9	split
				1305	27	106.8	126.3	split
				1318	24	169.9	330.3	pinhole
				1310	22.2	193.4	276.7	pinhole
				1286	21	807.5	758.4	split
				1315	19.2	554.1	921.5	pinhole
				1318	17	1092.5	1947.8	pinhole
				1317	15	1890.2	3191.1	pinhole
11	42404	IVCEV-20% CW CG	1200	1204	41	123.9	134.5	split
				1205	37	415.2	517.1	pinhole
				1187	32.6	1480.0	1372.6	pinhole
				1215	32	354.5	381.9	pinhole
				1203	30	1081.3	1162.8	multiple pieces
				1195	28	4321.9	3550.5	pinhole
19	49193	IVCEV-20% CW Plan. Swage CG	1300	1284	32	124.6	64.1	pinhole
				1292	31	129.5	93.3	split
				1292	26	100.9	74.2	pinhole
				1292	24	131.0	97.4	pinhole
				1316	20	186.4	323.0	pinhole
				1315	19.7	244.5	407.7	pinhole
				1293	16	685.9	549.9	pinhole
				1315	13	507.9	785.2	pinhole
				1300	10.5	1493.4	1493.4	pinhole
				1305	7.6	2256.5	2536.4	pinhole
19	49193	IVCEV-20% CW Plan. Swage CG	1200	1200	42.5	58.0	58.0	split
				1189	37	307.7	189.7	pinhole
				1186	28	1142.0	658.4	pinhole
				1189	23	2376.9	1590.5	pinhole

Table 1-9. Stress Rupture Data (continued)								
Item	Lot	Material Condition	Nom. Temp. (°F)	Test Temp. (°F)	Stress (ksi)	Rupture Time (hr)	Normalized Rupture Time (hr)	Rupture Type
20	49194	IVCEV-ANN Plan. Swage CG	1300	1316	29	1.4	2.6	multiple pieces
				1316	26	4.4	8.1	split
				1301	23	19.8	20.5	multiple pieces
				1297	21	45.8	43.0	split
				1300	19	113.7	113.7	split
				1297	18	234.6	212.5	split
				1298	16.4	346.8	325.3	split
				1300	15.4	501.6	501.6	pinhole
				1296	13.5	984.9	875.8	pinhole
				1300	11.8	2493.9	2493.9	pinhole
9-M	42402	IVCEV-20% CW FG Micro-peen	1300	1318	23	132.2	255.1	pinhole
				1291	13	1661.3	1279.0	pinhole
10-M	42463	IVCEV-ANN FG Micro-peen	1300	1324	16	272.1	580.4	split
19 GHT	49193	IVCEV-20% CW Plan. Swage CG Garofalo Heat Treat.	1300	1290	29	16.1	10.8	split
				1294	26	42.8	34.0	pinhole
				1294	21	95.1	77.0	pinhole
				1318	18	161.9	293.4	pinhole
19 GHT	49193	IVCEV-20% CW Plan. Swage CG Garofalo Heat Treat.	1200	1198	20	1899.3	1772.9	split

Figures 1-13 through 1-19 show the results of biaxial stress rupture tests in air for seven of the tubing lots, representing partial comparisons of some of the variables being tested. The variables include three vacuum-melting practices, plug drawing, planetary swaging, fine and coarse grain size, and annealed and 20 percent cold worked conditions. The data were obtained at 1200 and 1300°F ($\pm 20^\circ\text{F}$), and normalized to the nominal temperatures by means of the Chitty-Duval m-factor.^[28,7] Also included are data from tests in static sodium,^[29] and several points for micro-peened specimens and pre-test heat-treated specimens.

The upper two curves in Figure 1-13 are for ARD data, obtained in air at 1200°F for 20 percent cold worked, plug drawn tubing and the AI data obtained at 1200°F in static sodium for 10 to 15 percent cold worked material. The ARD tubing tested in air compares very favorably with the AI tubing tested

Figure 1-13. Biaxial Stress Rupture Results for Plug Drawn Tubing, Type 316 Stainless Steel, Made by Three Vacuum-Melting Practices, Fine Grain Size, 20 Percent Cold Worked
4100-16

in sodium. The differences which are observed in the stress rupture results for these two materials (the higher stress rupture strength for the ARD tubing and the break in its rupture curve) are the expected differences resulting from a higher level of cold work.

The lower three curves in Figure 1-13 are for 1300°F ARD data. These three curves compare three vacuum-melting practices for one set of fabrication conditions, namely fine grain size, plug drawn tubing in the 20 percent cold worked condition. The double vacuum-melted lot of tubing has slightly better stress rupture properties than the induction vacuum-melted lot, and significantly better properties than consumable electrode vacuum-melted material. All three materials show a change in the slope of the log stress versus log rupture time curve at approximately the same rupture time (700 to 900 hours at 1300°F).

The double vacuum-melted material (lot 42402) tested at 1200 and 1300°F shows an interesting comparison. The breaks in the rupture curve at 1200 and 1300°F occur at approximately the same rupture time; however, the stress levels are much higher for the 1200°F data. It is apparent that the mechanism which controls the break in the rupture curve is either independent of both temperature and stress, or is dependent on both. The latter is much more likely, and in this case it appears that the increased stress just offsets the lower temperature. For the material and test condition described here, the difference in temperature between 1200 and 1300°F corresponds to a stress difference of about 19,000 to 33,000 psi.

Figure 1-14 shows a comparison of 1300°F test results from both fine and coarse-grained, plug drawn tubing with the test results from coarse-grained, planetary swaged material; all double vacuum-melted Type 316 stainless steel is in the 20 percent cold worked condition.

Figure 1-14. Biaxial Stress Rupture Results at 1300°F for Cold Worked, Type 316 Stainless Steel Tubing, Fabricated by Plug Drawing and by Planetary Swaging

There is only a slight difference attributable to grain size in the two plug drawn lots out to about 800 to 900 hours, where there is a break in each curve. Thereafter, the coarse-grained, plug drawn material has significantly better stress rupture strength. However, as shown in Section 4, the rupture ductility of the coarse-grained tubing is less than the fine-grained material.

The coarse-grained, planetary swaged lot shows a similar break in the curve at about the same stress as the plug drawn lots, but it occurs at a much shorter time (about 300 to 400 hours). This difference is not attributable to grain size, since the two coarse-grained lots tested have the same grain size.

Figure 1-15 shows a comparison for the annealed condition between fine-grained, plug drawn tubing and coarse-grained, planetary swaged tubing. Contrary

Figure 1-15. Biaxial Stress Rupture Results at 1300°F for Annealed, Type 316 Stainless Steel, Fabricated by Plug Drawing and by Planetary Swaging

Figure 1-16. Biaxial Stress Rupture Results for Plug Drawn 20 Percent Cold Worked and Annealed, Type 316 Stainless Steel Tubing

to results from tubing in the cold worked condition, there is no significant difference between the plug drawn and the planetary swaged tubing in the annealed condition.

Figure 1-16 is a comparison of the annealed and the cold worked conditions for plug drawn material with fine-grain size. Whereas the cold worked material starts out with much higher stress rupture strength than the annealed material, there is a break in its stress rupture curve, occurring at about 800 to 900 hours and 19,000 to 20,000 psi, causing a change in slope and intersection with the curve for the annealed material at about 2700 hours and 11,000 psi. Beyond the intersection, the annealed material has better stress rupture life and, as shown in Section 4, also has greater rupture ductility.

Figure 1-17 is a similar comparison of the annealed and cold worked conditions for planetary swaged tubing with coarse-grain size.

Figure 1-17. Biaxial Stress Rupture Results for Planetary Swaged, Annealed Type 316 Stainless Steel Tubing

Qualitatively the curve shapes in Figure 1-17 are similar to the plug drawn materials in that the break in the curve occurs at about the same stress levels (21,000 to 22,000 psi), but the break occurs at much shorter time (300 to 400 hours), and the intersection occurs at about 700 hours and 14,000 psi.

Previous characterization results indicated that the cold worked, planetary swaged lot had a higher level of effective cold work than the plug drawn lots in spite of the fact that both types of material received the same nominal cold work, based on percent reduction in cross-sectional area after the last anneal. The cold worked, planetary swaged lot had higher hardness, faster precipitation kinetics, and a microstructure characteristic of much higher cold work than the plug drawn lot. The plug drawn and the planetary swaged, annealed materials, on the other hand, were similar in hardness and microstructure, and the only difference found in the material properties attributable to the difference in fabrication history was a slight difference in crystallographic texture.

Considering these pre-test characterization results, together with the lack of differences in the stress rupture curves for the annealed condition and the significant difference in the cold worked condition even at the same grain size, it appears that the differences observed in the stress rupture characteristics between the cold worked, plug drawn and the cold worked, planetary swaged tubing are the result of the higher level of effective cold work in the planetary swaged material.

Screening tests were also conducted at 1200 and 1300°F to determine the effects of melting practice for different sets of fabrication conditions, and to test the effects of micro-peening and pretest heat treatments. Results are shown in Figures 1-18 and 1-19, along with the general lot descriptions; specific descriptions are given in Table 1-2.

Figure 1-18 compares the results from the 1300°F screening tests (plotted as data points) to those for tubing lots 42402, 42403, and 49193 (solid

Figure 1-18. Biaxial Stress Rupture Results for Type 316 Stainless Steel Tubing

lines) described in previously. The one point obtained at 1300°F for the consumable electrode-melted, fine grained, annealed tubing lot (42395) fell slightly below the line for the comparable double vacuum-melted lot. This was also generally true for the four points from the coarse grained, cold worked lot (42396) from the same heat, tending to confirm the previous observation that the stress rupture life of consumable electrode-melted tubing is slightly less than for comparable double vacuum-melted material.

The three short-term points for cold worked, coarse grained, induction vacuum-melted lot (42400) also tend to confirm the previous observation made for the fine-grain condition that this single vacuum-melting practice also results in slightly shorter stress rupture life than the double vacuum-melting practice.

The 1200°F data points, shown in Figure 1-19, compared to the solid line for lot 42402, generally show the same effects observed at 1300°F for these variables, except that the difference in rupture life, resulting from melting practice which was slight at 1300°F, is not evident at 1200°F.

Based on the four data points in Figure 1-18 for the heat-treated lot 49193G compared to the non-treated lot, pre-test treatment at 900°F for 24 hours plus 1300°F for 216 hours does not improve the stress rupture life, to at least 300 hours at 1300°F. For the test conditions described, the effects of this heat treatment schedule on cold worked tubing is to impart properties intermediate between the as-cold worked tubing and the annealed tubing. This same observation was made in the biaxial tensile tests.

Based on the two data points indicated in Figure 1-18, micro-peening caused no increase in 1300°F rupture life of cold worked lot 42402. The one data point obtained at this temperature for micro-peened, annealed tubing did show an increase in the rupture life for the stress tested, but this increase could readily be explained by the cold work added to the annealed tubing surface.

Figure 1-19. Biaxial Stress Rupture Results for Type 316 Stainless Steel Tubing

3.5 STRESS RUPTURE TESTS SUMMARY

The stress rupture test results are summarized as follows:

1. ARD results conducted in air compare very favorably with AI results conducted in static sodium at 1200°F. The differences which are observed are due to the difference in level of cold work; i.e., 20 percent for the ARD data and 10/15 percent for the AI data.
2. Melting practice had only a slight effect on stress rupture behavior for the 20 percent cold worked condition. The double vacuum-melted material had slightly better stress rupture life than the single vacuum-melted lots at 1300°F, but this difference was not apparent at 1200°F.
3. For the cold worked condition and long-term tests at 1300°F, coarser grain size increased stress rupture life. For short- and intermediate-term tests there was no significant difference in rupture life for the fine-grained materials.
4. In the annealed condition, fine-grained, plug drawn tubing and coarse-grained, planetary swaged tubing gave almost identical stress rupture results.
5. Both the plug drawn and planetary swaged, cold worked lots showed breaks in their stress rupture curves which resulted in steeper slopes, and intersection with the curves for the annealed materials.
6. The difference in stress rupture behavior between the planetary swaged and the plug drawn, cold worked lots are due to different levels of effective cold work.
7. The indicated higher level of effective cold work in the planetary swaged tubing, relative to the plug drawn tubing, suggests again that definition of cold work as percent reduction in area is inadequate, particularly when different fabrication procedures are involved.
8. The superior stress rupture life for annealed materials at low stress indicates that, at least from a stress rupture viewpoint, 20 percent cold work may be above optimum.
9. No beneficial effect was obtained from micro-peening.
10. The pre-test heat treatment of cold worked tubing, first at low temperature 900°F/24 hours, followed by higher temperature 1300°F/216 hours produced stress rupture characteristics intermediate between the cold work and the annealed condition, for the range of conditions tested.

SECTION 4

POST-TEST STRESS RUPTURE SPECIMEN CHARACTERIZATION

4.1 INTRODUCTION

Selected post-test stress rupture specimens were further evaluated to investigate the effects of the four primary variables of melting practice, grain size, cold work, and fabrication practice on rupture morphology, strain at rupture, and structural changes occurring during test.

The post-test characterizations included the following:

1. Rupture morphology
2. Strain profile along the specimen length
3. Rupture ductility and shoulder ductility
4. Local circumferential strain based on wall thickness changes
5. Metallographic Evaluation
 - a. Rupture initiation site
 - b. Grain size and shape
 - c. Creep void and grain boundary cavity formation
 - d. Distribution of precipitates.

In addition to relating the primary variables to stress rupture behavior, these post-test characterizations also indicate promising directions for future studies.

4.2 RUPTURE MORPHOLOGY

The various types of rupture morphology observed in the stress rupture study are illustrated in Figure 1-20. This series is arranged in the order of increasing rupture severity, and illustrates all of the rupture types observed for the ten lots of tubing tested. However, not every rupture type was obtained in every lot. Increase in rupture severity proceeded from very small pinhole ruptures (not visually detectable), to splits first without and then with secondary circumferential tears, and finally to multiple-piece ruptures where a length of tubing (usually about 3/4 to 1-inch-long) was split away from the remaining two pieces. No rupture exceeding 1-1/2-inch of tubing length was observed in any of the tests.

(a) 13,000 psi, 867.0 hr, 1313°F, 0.008 in. PIN HOLE, MAXIMUM ϵ 35.4%, AVERAGE ϵ 12-14%

(b) 15,000 psi, 702.5 hr, 1292°F, MAXIMUM ϵ 30.0%

(c) 17,700 psi, 203.9 hr, 1303°F, MAXIMUM ϵ 20.8%, Average ϵ 10-12%

(d) 21,000 psi, 138.1 hr, 1295°F, MAXIMUM ϵ 29.4%, AVERAGE ϵ 10%

(e) 23,000 psi, 718.5 hr, 1197°F, MAXIMUM ϵ 20.0%, AVERAGE ϵ 9-10%

(f) 25,000 psi, 7.6 hr, 1316°F, MAXIMUM ϵ 39.5%, AVERAGE ϵ 18-20%

Figure 1-20. Various Modes of Ruptures Observed in Annealed, Plug Drawn Type 316 Stainless Steel Post-Test Stress Rupture Specimens

Preliminary results indicated that rupture morphology was related to both the hoop stress of the test and to the material variables. In order to illustrate these relations and rank the variables in the order of importance, specimens were classified according to rupture morphology using the following definitions:

Type 1 - Pinholes including the narrow longitudinal separations up to 1/8-inch total length. Illustrated by Figure 1-20a.

Type 2 - Splits greater than 1/8-inch long, with or without secondary tears. Illustrated by Figure 1-20b through e.

Type 3 - Multiple piece-type ruptures, including the two or three piece cases. Illustrated by Figure 1-20f.

The classified specimens included those from the $1300 \pm 20^\circ\text{F}$ tests listed in Table 1-9 plus additional tests conducted at both higher and lower temperatures. The higher temperature range was from 1320 to 1355°F , and the lower range from 1250 to 1280°F . Table 1-10 shows rupture Types 1, 2, and 3 as function of stress for each lot of material. The various lots are ranked in the order of increasing tendency to form pinhole ruptures. Temperature ranges are indicated by the letter subscript (H for high and L for low). The diagonal line separates the stress region of mixed-type rupture from the stress region of pinhole rupture only.

4.2.1 Rupture Morphology Results

Table 1-10 illustrates the following:

1. The tendency to form pinhole ruptures increases with decreasing stress.
2. Temperature in the range investigated has little or no effect on rupture morphology.
3. Cold worked materials have a greater tendency to form pinhole ruptures than the annealed materials.
4. The planetary swaged, cold worked lot, which was shown previously to have a higher level of effective cold work than the plug drawn lots, also has the greatest tendency to form pinhole ruptures.
5. For the cold worked condition, coarse-grain size increased the tendency to form pinhole ruptures relative to fine-grain size.
6. The effect of melting practice is not so apparent; if there is a real difference, it is probably that the consumable electrode vacuum-melted tubing has a slightly greater tendency to form pinhole ruptures than either induction vacuum or the double vacuum-melted tubing.

Table 1-10. Stainless Steel Biaxial Stress Rupture Fracture Classification - 1300°F Nominal Temperature (+ 20°F) Air Atmosphere

Lot	Condition	Melting Practice	Fabrication Practice	ASTM Grain Size	Stress (10 ³ psi)																											
					32	31	30	29	28	27	26	25	24	23	22	21	20	19	18	17	16	15	14	13	12	11	10	9	8	7		
49193	CW	IVCEV	PS	6	1	2	1		1		1		1		H ¹		H ¹			1			1		1			1				
	CW	CEV	PD	5.5/6			2		1		H ¹		1		1					1												
49193G	CW GHT	IVCEV	PS	6				2			1					1			1													
	CW	IV	PD	5.5/6			1		1		2		H ¹		H ¹		H ¹															
42402G	CW GHT	IVCEV	PD	9			L ³		L ³		L ³		L ³		H ¹		1															
42404	CW	IVCEV	PD	5.5/6			L ³	2	L ²	2	L ¹		H ^{2,1}	1	H ^{2,1}	2	H ¹	1		1		1										
42402	CW	IV	PD	9			1		2		H ²		2		H ²		1	1	1			1		1		1						
	CW	IVCEV	PD	9			2		1	1	1		3		2		2,1	1	1		1		1		1		1					
	CW	CEV	PD	9/9.5			1		H ²		1		1		H ¹		H ²		1		1		1		1		1					
	ANN	CEV	PD	8									2		1		2		2		1		1									
49194	ANN	IVCEV	PS	6				3			2			3		2		2	2		2	1	1		1							
42403	ANN	IVCEV	PD	8								3	H ²		L ²	2	H ^{3,2}		H ²		1	2			1	1	1	1				

1. Pinhole Rupture

2. Split

3. Pieces

PD - Plug Drawn

PS - Planetary Swaged

CW - 20% Cold Worked

ANN - Annealed

H - 1320-1355°F

L - 1250-1280°F

CEV - Consumable Elec.

IV - Induction Vacuum

IVCEV - Consumable Elec. Plus Induction Vacuum

GHT - Garofalo Heat Treat

7. The effect of pre-test heat treatment cannot be evaluated from these data.

Considering the table as a whole, it appears there is a dividing hoop stress which could be assigned to each material lot below which only pinhole ruptures would be expected. At stress levels below 13,000 psi only pinhole ruptures were observed for the 12 lots of Type 316 stainless steel tested, including the two heat treated and the three annealed lots. When the classification is limited to cold worked materials, the dividing stress is 18,000 psi at 1300°F.

4.3 STRAIN PROFILE MEASUREMENTS

Diametral strain profile measurements, similar to those described by Conway and Flagella,^[14] were made for 53 specimens selected from the stress rupture test program. The diametral strain profiles were obtained as follows:

1. Two perpendicular diameters were measured with a comparator at various positions along the length of the specimen
2. The average diameter together with the pre-test certified tube dimensions were used to calculate the $\Delta D/D$ strain value at each location.
3. The strain at the rupture was measured in two ways:
 - a. For pinhole ruptures, the average circumference obtained from comparator measurements was corrected for the width of the pinhole rupture and converted to an equivalent diameter and then to a strain $\Delta D/D$.
 - b. For split and multiple piece ruptures, a special technique was developed. The equivalent circumference of tubing at the center of the rupture (excluding the gap) was obtained from an impression made by rolling the specimen against the straight, cut edge of 1/16-inch-thick strip of lead, and measuring the length of the imprint with a calibrated microscope.

4.3.1 Strain Profile Results

Figures 1-21 and 1-22 illustrate various types of strain profile curves observed; additional results have been reported previously.^[8,9,10] In general:

1. There was non-uniform average strain ($\Delta D/D$) along the length of the specimens.
2. The rupture area had the greatest strain in almost every specimen.
3. The annealed materials had much greater $\Delta D/D$'s than the cold worked materials.

Figure 1-21. Strain Profile Measurements Post-Test Stress Rupture Specimens

4100-22

^ LOCATION OF TRANSVERSE SECTION USED
 FOR WALL THINNING MEASUREMENTS
 ↓ LOCATION OF PIN HOLE RUPTURE
 × LOCATION AND MAGNITUDE OF SPLIT RUPTURES

Figure 1-22. Strain Profile Measurements Post-Test Stress Rupture Specimens

4100-23

4. In general, the specimens that had the highest average $\Delta D/D$'s also had the highest diameter non-uniformity along the length (i.e., localized bulging).
5. In some specimens there was more than one local bulge along the length; this observation was more frequent for the low stress, long-term specimens.
6. For cold worked, plug drawn material, both increased grain size and lower test temperature (1200°F nominal) resulted in lower $\Delta D/D$'s.
7. For the annealed conditions, the fine-grained, plug drawn lot and the coarse-grained, planetary swaged lot had generally similar curves.

4.4 RUPTURE DUCTILITY AND SHOULDER DUCTILITY

The measured strains at rupture, together with the average of the strains one inch on each side of the ruptures (defined here as shoulder strains) were used to evaluate ductility with respect to the material variables and test conditions.

4.4.1 Rupture Ductility and Shoulder Ductility Results

Figures 1-23 and 1-24 illustrate the ductility results for the cold worked materials. In general:

1. Both rupture and shoulder ductility decreased with hoop stress and the associated increase in rupture time.
2. Shoulder ductility followed the same general curve shape as rupture ductility but at a lower level.
3. There appears to be no discontinuous change in ductility associated with change in rupture morphology from Type 1 (pinhole) to Type 2 (split) ruptures.

Ductility as a function of melting practice for the plug drawn, cold worked, fine-grained condition is illustrated in Figure 1-23.

1. There is slightly greater ductility associated with double vacuum-melting practice (Lot 42402-IVCEV).
2. There is little if any difference between the two single vacuum-melting practices (Lot 42394-CEV and Lot 42398-IV).

Figure 1-23. Single and Double Vacuum-Melted, Type 316 Stainless Steel Tubing Post-Test Air Atmosphere Stress Rupture - Fine Grain Size, 20 Percent Cold Worked Condition

Figure 1-24. Double Vacuum-Melted, Type 316 Stainless Steel Post-Test Air Atmosphere Stress Rupture - 20 Percent Cold Worked Condition

Ductility as a function of grain size, test temperature, and fabrication procedure for the cold worked, double vacuum-melted material, is shown in Figure 1-24.

1. At 1300°F the coarse-grained, plug drawn lot has significantly lower ductility than the fine-grained, plug drawn lot over the whole stress range investigated (29,000 to 15,000 psi).
2. Comparison of the 1300°F results for each of these two lots with the 1200°F results shows significantly lower ductility at the lower temperature. The relative reduction is about the same for the fine-grained lot as for the coarse-grained lot.
3. The coarse-grained, planetary swaged tubing had generally higher ductility than the coarse-grained, plug drawn tubing, but was not as high as the fine-grained, plug drawn material.

4. The one point for micro-peened tubing fell on the line for the same material without this surface treatment (Lot 42402), indicating little or no effect of micro-peening on the ductility for the cold worked condition.
5. Heat treatment of the cold worked, planetary swaged tubing at 900°F for 24 hours, plus 1300°F for 216 hours significantly increased ductility over the stress range investigated (26,000 to 18,000 psi).

Comparison of ductilities for annealed, plug drawn, fine-grained tubing with annealed, planetary swaged, coarse-grained tubing is shown in Figure 1-25. The observations are as follows:

1. The annealed materials have higher ductilities than the comparable cold worked lots (Figure 1-24).
2. The shoulder ductilities for these two annealed lots indicate a downward trend in ductility with reduced stress as was observed for the cold worked materials.
3. There were too few data points for the amount of scatter in the rupture ductility data to show the effect of stress on rupture ductility.

Figure 1-25. Double Vacuum-Melted, Type 316 Stainless Steel Tubing, Post-Test Air Atmosphere Stress Rupture - Annealed Condition

4. There appears to be no significant difference in ductility between these two annealed lots in spite of the difference in grain size. However, significant difference was found in the comparable cold worked lots (Figure 1-24).
5. The one point measured for a 1200°F plug drawn specimen indicates a drop in ductility associated with the lower test temperature.
6. An important observation made in the stress rupture testing program was that, for the low stresses, both of the annealed lots had greater stress rupture life than the comparable cold worked lots. Comparison of Figure 1-25 with 1-24 shows that they also retained much greater ductility under these conditions.

4.5 RATIO OF RUPTURE TO SHOULDER DUCTILITY

Using the ratio of rupture to shoulder ductility as the measure, an attempt was made to evaluate differences in localized bulging associated with the various material lots tested and the hoop stress of the test.

The ratio of rupture to shoulder ductility was calculated for each of the 53 specimens measured. The data were grouped by lot and ordered by stress within lots; average lot ratios were also calculated.

4.5.1 Ratio of Rupture to Shoulder Ductility Results

The results for this evaluation were as follows:

1. No marked effect of stress was observed within lots.
2. There was little difference in the average ratios between the cold worked lots, and also little difference between the average ratios for the annealed lots.
3. However, there was a marked difference between the annealed and cold worked lots. The annealed materials had a greater tendency for local bulging as illustrated by the higher ratios in the following tabulation.

20% Cold Worked		Annealed	
Lot	Avg. Ratio	Lot	Avg. Ratio
42402 IVCEV PD FG	1.37	42403 IVCEV PD FG	2.31
42404 IVCEV PD CG	1.38	49194 IVCEV PS CG	2.16
42394 PD FG	1.21		
42398 PD FG	1.26		
49193 IVCEV PS CG	1.29		
49193 Plus heat treat	1.09	Ratio = Rupture D/D / Shoulder D/D	

The factors contributing to the observed differences in the ratios probably include the strain hardening potential, the total ductility at rupture and perhaps mechanical differences in the tubing such as pre-test wall thickness variations.

4.6 LOCAL WALL STRAIN VARIATIONS AROUND THE TUBE CIRCUMFERENCE

4.6.1 General

Average diametral strain ($\Delta D/D$) has been used extensively both in this program and elsewhere as a measure of ductility for tubes experiencing hoop stress resulting from internal gas pressurization. It is a convenient statistic but it measures the average effect over the whole tube circumference and may obscure important localized variations. Also, the actual $\Delta D/D$ observed is the result of at least three contributing factors: the material properties; the mechanical configuration; and the conditions under which the specimen was strained. In some applications, for example, in a comparison of material properties at high total strains, the materials contribution may be confounded with the other two factors.

In order to 1) explore the potential information to be obtained from local wall strain measurements in separating the factors contributing to the average $\Delta D/D$, and 2) to illustrate the results from various materials, local wall strain measurements were made for a limited number of specimens from this test program. The results are discussed along with the inherent characteristics of the testing procedure, and some inferences made which can be tested experimentally in future work.

4.6.2 Measurement Procedure

The 14 specimens, listed in Table 1-11, were measured for local wall strain variation around the circumference of transverse sections of the tubes. One section was taken through the fracture area, and at least one additional section at some location of interest along the strain profile curve for each specimen examined.

The sections were mounted and metallographically polished. Wall thicknesses were measured using a light microscope equipped with a Filar eye-piece, calibrated against a standard stage micrometer. For the split-type rupture specimens, where the section could not be mounted perpendicular to the plane of polish, the observed wall thicknesses were corrected for the tilt angle. Wall thickness measurements (t_1) were made at five locations, based on polar angle around the circumference of the ruptured specimen sections, and at four positions around the non-ruptured sections.

The rupture location was arbitrarily designated as the zero degree position, and wall thickness measurements were made at approximately +10, -10, +90, and 180 degrees. The non-ruptured specimens were measured at 0, +90, -90, and 180 degrees, maintaining the angular orientation of the non-ruptured section, relative to the ruptured section from the same specimen.

Table 1-11. Local Wall Strain Measurements

Lot	Condition	Hoop Stress (psi)	Temp. (°F)	Rupture Time (hr)	Rupture Type	Avg. $\Delta t/t_0$ $\pm 10^\circ$ (%)	Min. $\Delta t/t_0$ Rupture Section	Figure	Rupture $\Delta D/D$ (%)
42402	20% CW GS ASTM 9 Plug Drawn	30,000	1313	26.6	split	22.2	7.8	-	13.8
		15,000	1314	805	pinhole	10.1	0.2	1-27	5.5
		12,000	1303	2368	pinhole	6.8	7.4	1-27	8.1
		24,000	1214	2124	pinhole	10.6	4.4	1-27	4.8
42403	Annealed GS ASTM 8 Plug Drawn	25,000	1316	7.6	pieces	32.3	19.2	-	39.5
		13,000	1313	867	pinhole	42.2	15.5	1-28	35.2
		11,500	1301	2424	pinhole	34.5	18.7	-	40.0
42404	20% CW GS ASTM 6 Plug Drawn	21,000	1286	808	split	6.3	0.4	1-27	4.0
49193	20% CW GS ASTM 6 Planetary Swaged	32,000	1284	125	pinhole	7.8	2.9	-	7.2
		20,000	1316	186	pinhole	5.8	2.5	-	6.2
		16,000	1293	686	pinhole	5.0	2.8	1-28	4.9
		10,500	1300	1493	pinhole	8.2	1.8	1-28	6.2
49194	Annealed GS ASTM 6 Planetary Swaged	13,500	1296	985	pinhole	34.8	19.0	1-28	32.5
		11,800	1300	2494	pinhole	34.4	26.9	-	36.0

From these thickness measurements, $\Delta t/t_0$ values were calculated, where Δt is the change in wall thickness and t_0 is the average initial wall thickness calculated from the certified maximum and minimum OD's and ID's for the particular tubing lot. In presenting the results, $\Delta t/t_0$ values were plotted as a function of circumferential angle for each transverse section.

The greatest source of error originated with the use of the certified lot dimensions for the t_0 . This source was estimated to induce a maximum error of about ± 1.5 percent absolute in the $\Delta t/t_0$ data.

Figure 1-26 graphically illustrates the relation between $\Delta t/t_0$ and $\Delta D/D_0$. This curve was calculated for tubing having initial dimensions of 0.230-inch OD by 0.200-inch ID, and was based on area considerations assuming uniform strain around the tube wall and negligible strain in the longitudinal direction. For the real case, where the wall does not strain uniformly, this curve can be used to sum the local wall strains and calculate an equivalent $\Delta D/D$ for the section. This calculated $\Delta D/D$ can then be compared with the $\Delta D/D$ directly measured before sectioning.

Figure 1-26. Relations Between Various Ratios Used to Measure Ductility

4.6.3 Results

Typical results are shown in Figures 1-27 and 1-28. The data points for each section were arbitrarily connected by straight lines, except for the rupture areas, which were left open. In plotting the results, the orientations of some sections were rotated, as indicated in the figures, to bring positions of maximum strain into coincidence. The percent wall thickness reduction ($\Delta t/t_0$) was used here as the direct measure of the percent local strain.

The following observations were made from the 14 stress rupture specimens examined.

1. All of the specimen sections containing the rupture area and most of the other sections showed non-uniform wall thickness reduction during test, indicating some type of circumferential heterogeneity in the pre-test tubing.

Figure 1-27. Wall Thickness Variation Around Circumference of Transverse Sections Indicated in Figure 1-22

4100-25

Figure 1-28. Wall Thickness Variation Around Circumference of Transverse Sections Indicated in Figure 1-21

4100-26

2. The annealed materials had the higher $\Delta t/t_0$ values, as would be expected, since they had the higher average ductilities as measured by $\Delta D/D$.
3. In all of the specimens examined the maximum $\Delta t/t_0$ was observed in the section containing the rupture, and in 12 of the 14 specimens the maximum $\Delta t/t_0$ was located near the rupture. An exception was the plug drawn, annealed specimen which had a low stress and long rupture time; in this specimen there were two thin areas in the same transverse plane. The other exception was in planetary swaged, cold worked specimen which had a slightly thinner area 90 degrees from the rupture than at 10 degrees.
4. In 9 of the 14 specimens the $\Delta t/t_0$ maxima in the non-ruptured sections were not aligned with the maxima of the ruptured specimens, indicating that the source of the pre-test nonuniformity was not longitudinally aligned.
5. No positive statement could be made regarding the relative degree of variation in wall thickness in the ruptured section (i.e., maximum to minimum $\Delta t/t_0$), with respect to either rupture stress or material. This approach might show significant differences if more specimens were considered and if the pre-test wall thickness variations were known more accurately.

Table 1-11 tabulates the maximum and minimum $\Delta t/t_0$ values observed in the transverse section containing the rupture and the $\Delta D/D$ value for each of the section measured. It is apparent that great variations in local wall strain existed. This variation in local ductility was not observed in the usual $\Delta D/D$ measurement of tube ductility, which is an average measurement for the whole circumference.

4.6.4 Discussion of $\Delta t/t_0$ Measurements

The general observation of non-uniform wall straining is perhaps the most significant result from the $\Delta t/t_0$ measurement of these 14 specimens, representing five fabrication practices, grain size, and cold work combinations.

Two sources which could cause the observed non-uniform wall straining during test are circumferential temperature variations or deviations from a homogeneous, uniform walled, circular cross-section in the starting material. Circumferential temperature variation is highly unlikely in the furnace configuration used for these tests; the more probable cause is some type of circumferential heterogeneity in the pre-test tubing, and wall thickness variation is the most likely source.

4.6.5 Ductility Limitation

The effect of nonuniform straining on limiting potential ductility is illustrated as follows.

The particular specimen shown in Figure 28c, showed a maximum strain, measured by $\Delta t/t_0$, of about 42 percent at the 10 degree locations. The minimum was about 15.5 percent $\Delta t/t_0$ at the 180 degree position. The ± 90 degree positions were intermediate. If the straining were uniform around the wall to the potential level attained at the ± 10 degree locations, taken to be a good measure of the potential ductility of the material, the $\Delta D/D$ results would have been much different. Converting the $\Delta t/t_0$ value of 42 percent to an equivalent $\Delta D/D_0$ value, assuming uniform wall thickness reduction to this level, a ductility value of 65 percent $\Delta D/D_0$ would be obtained compared to the 35.3 percent $\Delta D/D_0$ observed. In other words, as a result of the non-uniform wall thickness reduction, this specimen actually reached only a little more than half the potential ductility based on the potential ductility of the material.

As a check on the $\Delta t/t_0$ measurements used in this discussion, the $\Delta t/t_0$ measurements from the 180, +90, -90 degree positions, and the average value from the ± 10 degree positions for this section were converted to equivalent $\Delta D/D_0$ values using a calculated equivalence curve in Figure 1-26. In Figure 1-26, the estimated $\Delta D/D_0$ calculated from the mean of these four $\Delta t/t_0$ measurements was 33.4 percent $\Delta D/D_0$ compared to 35.3 percent $\Delta D/D_0$ independently measured by a comparator. This agreement is quite good, especially considering the number of independent measurements involved.

The specimen in this example is, perhaps, a more extreme case than usually would be encountered, but it does illustrate the principle that, in order to obtain maximum hoop ductility ($\Delta D/D$) for a given material those factors which lead to non-uniform wall straining should be minimized.

4.6.6 Rupture Morphology

Non-uniform straining, coupled with the inherent characteristics of internally pressurized tubing tests, leads to an explanation for the explosive type rupture.

In biaxial stress rupture tests, the tubing is internally pressurized with gas to give a specific initial hoop stress and held at a constant pressure and temperature until rupture occurs. The rupture time is recorded and the results are presented in the form of standard stress rupture plots of log engineering stress versus log rupture time. The engineering stress plotted for the test is the initial hoop stress defined as follows for thin walled tubing:

$$\sigma_h = \frac{PD}{2t}$$

Where:

σ_h = hoop stress

P = pressure

D = pre-test diameter at the mid wall

t = pre-test wall thickness

Figure 1-29. Ratio of Actual Hoop Stress to Engineering Hoop Stress Versus $\Delta D/D_0$

As the test proceeds the actual hoop stress increases as a result of both the increase in the tube diameter and the decrease in wall thickness. This point is illustrated by Figure 1-29, which is a plot of the ratio of actual stress to engineering stress (initial hoop stress) as a function of $\Delta D/D$. This plot was calculated for 0.230-inch OD by 0.200-inch ID tubing, using the same equation for the stress given previously, but substituting the actual mid-wall diameters and wall thickness for the various $\Delta D/D_0$ values, and assuming uniform hoop strain and negligible longitudinal strain. It can be seen that the actual stress experienced by the material in the tube wall increases rapidly with $\Delta D/D$.

Assuming no strain hardening or any other process which would tend to offset the effects of non-uniform straining and increased stress, an accelerated strain rate would be expected as the test proceeds, and in the extreme case this instability could lead to explosive type ruptures, blowing-out the thin side.

4.6.7 Eccentricity Effects

If it is assumed that initial wall thickness variation is the primary cause for the observed non-uniform wall thickness reduction during tests, then based on the initial differential stress and the differential increase in stress as the test proceeds, the tube will fail at its thinnest area. Also it is this area which will determine to a great extent the rupture life of the specimen; however, it is the whole tube circumference not just the weakest area which determines the diametral ductility.

It follows that a higher diametral ductility would be expected from a tube having a uniform 0.014-inch wall than from one having a 0.014 inch wall in part of the circumference and a thicker wall in the remainder, but that the hoop strength would be determined primarily by the 0.014 inch thickness in both cases. This leads to the conclusion that eccentricity is an important factor in tubing specifications, and that the quantitative evaluation of its importance could be determined by this approach.

It is recognized that real materials include other factors, such as strain hardening, which tend to offset the effects of initial wall thickness variation, and recovery and recrystallization which change the stress-strain rate

relation during test. Surface defects, if large enough, could also be an important variable. Differences in inherent ductility, such as the difference between annealed and cold worked tubing, also influence the relative results between the materials. Evaluation of these factors was beyond the scope of this investigation.

4.6.8 Summary

It appears from these results that local wall thickness measurements around the ruptured tube circumference, combined with accurate pre-test wall thickness measurements, would aid in separating mechanical effects and materials effects during test. This distinction is not apparent when only $\Delta D/D_0$ measurements are made. The $\Delta t/t_0$ type of measurement could also aid in evaluating the effects of natural and intentional defects on mechanical properties. Combined with a well planned test program, $\Delta t/t_0$ measurements could also be used to evaluate the effect of irradiation on strain hardening potential under long-term test conditions.

4.7 METALLOGRAPHIC EXAMINATIONS

Fourteen specimens from the stress rupture program were metallographically examined, in detail, along with untested control samples. The selection was based on stress rupture results, strain profile curves, $\Delta t/t_0$ measurements, and preliminary metallographic evaluations of about an equal number of specimens. The metallographic evaluation reported here is limited to the double vacuum-melted tubing lots.

At least two sections were examined from each of the 14 post-test specimens, including a transverse section through the rupture and a transverse section from some other location of interest along the strain profile curves.

The cut sections were mounted in epoxy resin, rough ground with SiC abrasives, using automet equipment and finished with diamond abrasives on rotating laps, followed by final polishing with alumina in a vibrating polisher. The polished sections were chemically etched in modified glycergia containing three parts glycerine, three parts HCl, and two parts HNO_3 . Etching time was varied to achieve the desired results.

In addition to these 14 preparations, transverse sections from seven of the specimens were prepared in special mounts and cathodic vacuum etched to show grain size and cavity distribution. The location of the cathodic etched sections was generally 1/2 to 1 inch from the rupture.

The structural information obtained from both etching procedures was combined in the evaluations.

4.7.1 Results

4.7.1.1 Cold Worked, Plug Drawn Lot 42402

Figures 1-30 and 1-31 illustrate the various structures observed in this

(a) SPLIT TYPE RUPTURE 120X

OD

$T_R = 26.6$ hr
30,000 psi
1313°F

ID

(b) RUPTURE AREA 400X

(c) PINHOLE RUPTURE 120X

OD

$T_R = 805$ hr
15,000 psi
1314°F

ID

(d) 90 DEGREES 400X
ANGULAR FROM RUPTURE

(e) PINHOLE RUPTURE 150X

OD

$T_R = 2368$ hr
12,000 psi
1303°F

ID

(f) 20 DEGREES 150X
ANGULAR FROM RUPTURE

Figure 1-30. Microstructures from Short, Intermediate, and Long-Term Stress Rupture Specimens for the Plug Drawn, Fine Grained, 20% Cold Worked Lot (42402)

- (a) CATHODIC VACUUM ETCHED, AS-RECEIVED, 20% COLD WORKED, ASTM GRAIN SIZE 9
- (b) CATHODIC VACUUM ETCHED POST-TEST RUPTURE SPECIMEN, $T_r = 2368$ hr, HOOP STRESS = 12,000 psi, 1303°F AIR ATMOSPHERE
SECTION: 1 in. FROM RUPTURE PLANE
- (c) CHEMICAL ETCH POST-TEST RUPTURE SPECIMEN, $T_r = 2368$ hr, HOOP STRESS = 1303°F AIR ATMOSPHERE
SECTION: IN RUPTURE PLANE, 20 DEGREES FROM RUPTURE

Figure 1-31. Composite Micrographs
Across the Tube Wall -
Plug Drawn Tubing, Lot
42402

cold worked, plug drawn lot after short, intermediate, and long-term stress rupture tests at 1300°F (nominal). Included for comparison are results from one long-term test at 1200°F (Figure 1-32a and b).

All of the pinhole ruptures examined for this lot of material from short, intermediate, and long-term tests initiated at the OD surface of the tube wall (Figures 1-30c and e, and 1-32a). The origin of split and multiple piece-type ruptures was not always conclusively determined because of the difficulty in distinguishing the primary break through the wall from the subsequent tear (Figure 20B through e). The structure shown in Figure 1-30a, for the very short-term test, is an example of a cross-section through the tear of a split-type rupture. It does not include the primary break through the wall. Figure 1-32a and b illustrate the structure observed for long-term lower temperature test specimens.

There are a number of changes in the microstructure which can be correlated with increased rupture time and the associated decreased hoop stress of the 1300°F tests.

In the short-term tests with high strain rates there is grain distortion in the vicinity of the rupture and a single size class of precipitate; this precipitate has the appearance of a carbide and is located in grain boundaries, slip planes, and twin interfaces. In the intermediate and the long-term tests no grain distortion is observed, but there is an additional class of precipitate. This precipitate is larger in size than the carbide-type, is preferentially located on the grain boundaries, and has the appearance of an intermetallic; it is probably sigma phase and its size and frequency increases with rupture time. In both the intermediate and long-term tests specimens this type of precipitate occurs in greater concentration near the OD of the wall than near the ID.

Detailed examination of the cathodic etched specimens from the whole test range showed that the microstructure contains grain boundary cavities for all test conditions, both before and after the break in the stress rupture curve, and that both the frequency and size of the cavities increase with rupture time. The highest concentration of cavities in each specimen from this lot is located near the OD surface. In the short-term test, the cavities are small wedge and "r-type" creep voids located respectively at triple points and along grain boundaries. In the intermediate and long-term-test, the cavity morphology changes to large grain boundary separations showing radial orientation.

The structures shown in Figure 1-31a and b illustrate another important observation relative to the stress rupture behavior of cold worked, plug drawn material. Comparison of the cathodic etched structures of the as-received material with the long-term test specimen shows that grain size and shape is uniform across the wall in both specimens. Also, there is very little difference between the specimens in either grain size or shape, indicating that the long-term specimen did not recrystallize during test.

The cathodic etched structure from the short and intermediate-term test specimens also show the same uniform grain size and shape as the starting material except for the distorted grain shape in the immediate vicinity of the split ruptures in the short-term test specimens described previously.

(a) PINHOLE RUPTURE

190X

OD

(b)

1000X

ID

LOT 42402, $T_R = 2124$ hr, 24,000 psi, 1214°F, 20 DEGREES ANGULAR FROM RUPTURE

(c) LIGHT CHEMICAL ETCH

220X

OD

(d) HEAVY CHEMICAL ETCH

220X

OD

(e) CATHODIC VACUUM ETCH

220X

ID

ID

LOT 42402, $T_R = 805$ hr, 15,000 psi, 1314°F, 90 DEGREES ANGULAR FROM RUPTURE

Figure 1-32. Variation in Apparent Microstructure with Etching Procedure for Stress Rupture Specimen

The long-term lower temperature (1200°F) test specimen (Figure 1-32a and b) ruptured with a pinhole initiating at the OD, contained grain boundary cavities, and both size classes of precipitates. Generally, the structure is similar to the intermediate-term specimen tested at higher temperature except it has a higher relative frequency of the small-size class of precipitates.

Figure 1-31 (b and c) illustrates some of the relative advantages of the two etching procedures, and together with Figure 1-32 (c, d, and e) demonstrates why both were required. The cathodic vacuum etched structure (Figure 1-31b) for the long-term specimen shows the grain structure and the relation of the cavities to the grain boundaries but does not show the precipitates distinctively. On the other hand, the chemical etch (Figure 1-31c) outlines the precipitates quite well, but does not show the grain boundaries directly. The apparent grain boundaries in this case are delineated by the locations of precipitates. In this long-term specimen the cavities are large enough to be observed by both procedures.

Figure 1-32c illustrates the structure developed by light chemical etching of the intermediate-term specimen, and Figure 1-32d by heavy etching at approximately the same location. In the heavy-etched structure the cavities are apparent which were not revealed by light etching. From this pair of structures alone it would not be obvious whether the black areas are cavities or etched-out precipitates. However, because of the non-specific nature of cathodic vacuum etching and the absence of galvanic attack, the structures shown in Figure 1-32c can be conclusively interpreted as containing cavities.

4.7.1.2 Cold Worked, Planetary Swaged Lot 49193

Figures 1-33 and 1-34 illustrate the various structure observed in the cold worked, planetary swaged lot after short, medium, and long-term tests.

There are some characteristics similar to the plug drawn lot. The short-term planetary swaged specimens have a few small wedge and r-type creep voids located at triple points and along grain boundaries, respectively. The highest concentration of the voids occurs near the OD. The grain structure is uniform across the wall and has the same grain size and shape as the starting material. There is one size-class of precipitate, having the appearance of a carbide, located on grain boundaries, slip planes, and twin interfaces.

The intermediate and long-term test specimens contain the two size-classes of precipitates described previously. Both have a morphology similar to those of the plug drawn lot and the intermetallic type increases in both size and frequency with increased rupture time. The highest concentration of the intermetallic occurs in an annulus which increases in thickness with increased rupture time and is located adjacent to the OD of the tube (Figure 1-33).

There are some characteristics of the planetary swaged material which are markedly different from the plug drawn material. The cathodic etched structure of a long term specimen (Figure 1-33f) shows that the OD half of the wall had recrystallized to finer grain size during test. In addition, chem-

100X
(a) $T_R = 186$ hr, 20,000 psi, 1316°F

OD

100X
(b) $T_R = 1493$ hr, 10,500 psi, 1300°F

ID

100X
(c) $T_R = 186$ hr, 20,000 psi, 1316°F

OD

100X
(d) $T_R = 686$ hr, 16,000 psi, 1293°F

ID

100X
(e) $T_R = 1493$ hr, 10,500 psi, 1300°F,
CHEMICAL ETCH

OD

100X
(f) $T_R = 1493$ hr, 10,500
psi, 1300°F, CATHODIC
VACUUM ETCH

ID

- a - PINHOLE RUPTURE INITIATE AT OD IN SHORT-TERM TESTS
- b - PINHOLE RUPTURE INITIATE AT ID IN LONG-TERM TESTS
- c,d,e - OD ANNULUS CONTAINING INTERMETALLIC PRECIPITATES INCREASES IN WIDTH WITH INCREASE RUPTURE TIME
- f - GRAIN BOUNDARY VOIDS ARE CONCENTRATED NEAR THE ID IN LONG-TERM TESTS

Figure 1-33. Microstructures from Short, Intermediate, and Long-Term Stress Rupture Specimens for the Planetary Swaged, Coarse Grained, 20% Cold Worked Lot (49193)

OD

ID

500X

(a) AS-RECEIVED SPECIMEN, 20% COLD
WORKED, ASTM GRAIN SIZE 6

OD

ID

500X

(b) POST-TEST STRESS RUPTURE SPECIMEN ONE
INCH FROM RUPTURE PLANE, T_r 1493 hr,
10,500 psi, 1300°F

Figure 1-34. Composite Micrographs Across the Tube Wall - Planetary Swaged
Tubing, Lot 49193, Cathodic Vacuum Etch

ically etched structures from a series of tests with increasing rupture time (Figure 1-33) indicate that the extent of recrystallization increases with time of test and progresses from the OD toward the ID of the tube. The location and width of the precipitate annulus is similar to the location and size of the recrystallized annulus.

There are, however, differences in location of the rupture initiation site between the planetary swaged and the plug drawn materials. Whereas rupture of the plug drawn tubing initiates at the OD over the whole range of test conditions, there is a shift in the location of rupture initiation in the planetary swaged material. In short-term tests rupture initiates at the OD surface and shifts to the ID surface in the intermediate and long-term tests.

This shift in rupture initiation site is correlated with a similar shift in the location of the cavity concentration in the planetary swaged specimens. It is significant to note that the rupture initiation site is not correlated with the highest concentration of intermetallic precipitates over the whole range of test conditions. In the intermediate and long-term specimens, rupture initiates at the ID and the location of highest concentration of intermetallics remains near the OD.

4.7.1.3 Annealed, Plug Drawn Lot 42403 and Annealed, Planetary Swaged Lot 49194

Since there is little difference in the stress rupture behavior of these two annealed lots, their metallographic results are treated together. The stress rupture curves are almost exactly super-imposed and show no breaks in the stress rupture line. They have approximately the same ductilities and for low stress conditions, both lots have greater stress rupture life and greater ductility than the corresponding cold worked lots.

Preliminary metallographic results indicated that where the rupture initiation site could be determined it was located at the OD of the specimen. Both lots of annealed tubing show grain distortion in the hoop direction both in the vicinity of the ruptures and in the general microstructure over the whole test range. The extent of the grain elongation was in general correlated with the amount of local wall strain. In short-term tests only the carbide precipitate is observed and in the long tests both types of precipitates are present. A few creep voids were found in the general micro-structure but only in the long-term specimens.

Figures 1-35 and 1-36 illustrate various structures observed in the annealed specimens from the long-term tests at 1300°F. The annealed, plug drawn specimen (Figure 1-35) had been tested at 1301°F and low stress (11,500 psi). It ruptured after 2424 hours with a pinhole initiating at the OD. It had both greater stress rupture life and greater ductility than the corresponding cold worked specimens tested at similar stresses and temperature.

In the rupture area of this specimen and in an additional thin area found in the same plane as the rupture there was pronounced grain elongation in the hoop direction, and there are a few large cavities and some grain separations. In the general microstructure, however, there are very few creep-

Figure 1-35. Microstructures for a Long-Term Stress Rupture Test Specimen from Plug Drawn, Annealed, Fine Grained Lot (42403) - $T_r = 2424$ hr, Hoop Stress = 11,500 psi, Test Temperature = 1301°F

(a) PINHOLE RUPTURE
ID 150X

(b) 180 DEGREES ANGULAR FROM RUPTURE
ID 1000X

(c) CATHODIC VACUUM ETCH ONE INCH FROM THE RUPTURE PLANE
ID 250X

Figure 1-36. Microstructures for a Long-Term Stress Rupture Test Specimen from the Planetary Swaged, Annealed, Coarse Grained Lot (49194)
 $T_r = 2492$ hr, Hoop Stress = 11,800 psi, Test Temperature = 1300°F

type voids. Comparison of the cathodic etched section, which was located about one inch from the rupture plane, with similar sections from the cold worked lot showed that the general microstructure of this long-term annealed specimen (2424 hours) has about the same frequency of creep voids as the very short-term, cold worked specimen, tested for 26.6 hours at the same nominal temperature.

The chemical etched microstructure of this specimen (Figure 1-35) shows the presence of the two classes of precipitates described previously. The smaller carbide type has a general distribution with no strong preference for grain boundaries. The larger intermetallic type tends to be located on grain boundaries but has a lower concentration than in the cold worked lots, and is not preferentially located near the OD.

The annealed, coarse-grained, planetary swaged specimen (Figure 1-36) was tested at almost identical test conditions as the annealed, fine-grained, plug drawn specimen. The microstructure of this specimen differs from the annealed, plug drawn specimens in that it has fewer cavities, fewer precipitates of both size classes, and a greater tendency for precipitates to be located in grain boundaries.

Both long-term annealed specimens exhibited severe oxidation on the OD resulting from the testing in air. It is significant that the oxide thickness was correlated with the local wall thickness reduction. This effect is probably the result of repeated breakdown of the protective oxide layer during straining.

4.7.2 Discussion of Metallographic Results

The general post-stress rupture metallographic results, showing the contrasts and comparisons between the various lots of tubing examined, are summarized in Table 1-12.

Examination of this table shows that the major differences in structural characteristics are between the annealed and the cold worked materials, and there is a secondary contrast between the two cold worked materials.

The major differences between the annealed and cold worked materials appears to relate to the relative strength of the internal grains and the grain boundaries which results in two different controlling mechanisms of creep deformation.

Rachinger^[30] has used an index for the change in shape of grains during deformation to evaluate the relative contribution of grain boundary shear and internal grain deformation. He reasoned that if there is no grain boundary sliding and all of the deformation occurs by slip inside the grain, then the average grain should suffer the same relative elongation and contraction as the specimen itself. On the other hand if all of the deformation occurs by shear and rotation of the grains relative to one another, then the average grain should maintain its original shape. It has also been well established that wedge^[31, 32, 33] and r-type^[34, 35] voids are the result of grain boundary shear.

Table 1-12. Summary of Metallographic Results

Characteristic	Lot 42402 Plug Drawn-ASTM 9	Lot 49193 Planetary Swaged-ASTM 6	Lot 42403 Plug Drawn-ASTM 8	Lot 49194 Planetary Swaged-ASTM 6
Rupture morphology dividing stress below which only pinhole ruptures were observed	20,000 psi	30,000 psi	13,000 psi	15,000 psi
Rupture initiation site	Where the rupture site could be deter- mined, rupture in- itiated at the OD	Ruptures initiated at the OD in short- term high stress tests but shifted to the ID in intermedi- ate and long-term low stress tests	Same as lot 42402	Same as lot 42403
Grain boundary cavities	Present in all specimens examined both in vicinity of rupture and in gen- eral microstructure	Same as lot 42402	Present near rup- tures but not in general microstructure except in long-term, low stress test where a few cavities were present in the general microstructure	Same as lot 42403
Grain boundary cavity distribution	a. Cavity size and fre- quency increased with reduced stress and increased rupture time b. The location or the highest concentration remained near the OD over the whole test range	a. Same as lot 42402 b. The location of the highest concentration shifted from the OD in short-term high stress tests to the ID in intermediate and long-term low stress tests	a. Cavities present were very few in number b. No localization effect was observed	a. Same as lot 42403 b. Same as lot 42403

Table 1-12. Summary of Metallographic Results (Continued)

Characteristic	Lot 42402 Plug Drawn-ASTM 9	Lot 49193 Planetary Swaged-ASTM 6	Lot 42403 Plug Drawn-ASTM 8	Lot 49194 Planetary Swaged-ASTM 6
Precipitates	<p>a. Fine carbide type precipitate present over whole test range</p> <p>b. Coarser intermetallic type grain boundary precipitate (probably sigma) present in intermediate and long-term, low stress tests.</p> <p>c. Location of coarse type in higher concentration near OD</p>	<p>a. Same as lot 42402</p> <p>b. Same as lot 42402</p> <p>c. Same as lot 42402</p>	<p>a. Same as lot 42402</p> <p>b. Same as lot 42402 but lower concentration</p> <p>c. No localization effect was observed</p>	<p>a. Same as lot 42403</p> <p>b. Same as lot 42403</p> <p>c. Same as lot 42403</p>
Grain size and shape	Grain size and shape appeared to remain unchanged in the specimens cathodic etched for grain size	Recrystallized to smaller grain size near OD in intermediate and long-term low stress tests	Grain elongation in hoop direction. The amount was correlated with the local strain	Same as lot 42403

The contrasts in the metallographic results from this investigation suggest that the two different deformation mechanisms are dominating in the annealed and the cold worked materials at the same temperature (1300°F) and for the same alloy chemistry.

The annealed materials show pronounced grain distortion in the hoop direction in the general microstructure and very few creep voids except in the very long-term specimens. Even in the long-term specimen, the creep void frequency is very low. These results suggest that deformation within the grains by slip and climb is the dominant mechanism.

In contrast, the cold worked materials show the presence of creep voids resulting from grain boundary shear over the whole test range investigated and the frequency and size of the voids increases with reduced stress. No apparent grain distortion was observed except in the vicinity of tears in short term tests. These results suggest that for the cold worked materials at 1300°F, grain boundary shear is at least an important contributing mechanism to deformation at high stresses, and that this mechanism becomes more dominant with reduced stress.

The major difference between the two cold worked lots can be attributed to the occurrence of recrystallization in one case and not in the other. Based on the microstructure, it appears that for the plug drawn lot the change in slope of the rupture curve is simply the result of increased frequency and size of the grain boundary voids, resulting from lower strain rates and the linking-up of these voids, causing reduced strength. No abrupt change in the microstructure was found associated with the change in slope of the rupture curve for this material.

The structural changes in the planetary swaged lot are more complex. The creep voids were also found over the whole range of test conditions, but, at stress levels and strain rates corresponding to the break in the curve, the structure of the tube wall begins to recrystallize at the OD. In tests with lower stresses, the extent of recrystallization increases with reduced test stress and progresses from the OD toward the ID. Simultaneously, with the start of recrystallization the site of rupture initiation and the concentration of creep voids also shifts from the OD to the ID. Since the recrystallized metal has higher ductility and lower strength, the expected net effect is to shift the load to the ID annulus of the tube and thus cause the reduced strength, as well as the observed shifts in the void location and the rupture initiation site.

The difference between the cold worked lots is most likely the result of the initially higher level of effective cold work in the planetary swaged tubing since this would tend to lower the temperature and other test conditions necessary to cause recrystallization. In addition, the metallographic results suggest that lower stress and longer rupture times also tend to promote recrystallization. Additional changes in the slope of the stress rupture curves may also occur at conditions beyond those tested here. A change in slope would be expected if the plug drawn material would start to recrystallize, and most probably a change in slope will occur when conditions are reached where recrystallization is complete in the planetary swaged material.

SECTION 5

SUMMARY AND RECOMMENDATION

5.1 SUMMARY OF THE VACUUM-MELTED STEELS PROGRAM

The most significant results from this investigation are:

1. There is little difference in tubing characteristics or mechanical properties between the single vacuum-melted and the double vacuum-melted heats of Type 136 stainless steel.
2. Increasing grain size from ASTM 9 to ASTM 6 has only a slight effect on biaxial stress rupture life of cold worked tubing, at least out to about 1000 hours at 1300°F, but does reduce diametral strain at rupture; thereafter the coarse-grained material has the greater rupture life. In short-term biaxial tests the increased grain size reduces strength by about 10 percent and slightly increases diametral strain.
3. At 1300°F test temperature, 20 percent cold worked materials show breaks in the stress rupture curves, resulting in steeper slopes and intersection with annealed curves, indicating that from a long-term stress rupture view-point, 20 percent cold work is above optimum.
4. For the low-stress conditions the annealed materials have longer stress rupture life and greater ductility than cold worked materials.
5. Defining degree of cold work in terms of percent reduction in cross-sectional area may not be adequate for characterizing the effective level of cold work, particularly when different fabrication procedures are involved.
6. The major differences in properties between plug drawn and planetary swaged tubing are explainable on the basis of a higher level of effective cold work in the planetary swaged tubing.
7. The tendency to form pinhole ruptures, in contrast to splits or multiple piece ruptures, increases with level of cold work, grain size, and decreasing hoop stress.
8. There is apparently a hoop stress level below which only pinhole ruptures are observed. That level is dependent on the material characteristics.
9. At stress levels below 13,000 psi hoop stress for tests at 1300°F, only pinhole ruptures were observed for the 12 lots of Type 316 stainless steel tested, including seven cold worked lots, two heat treated, cold worked lots and three annealed lots. When the classification was limited to the cold worked materials, the dividing stress was 18,000 psi at 1300°F.

10. For the cold worked materials, both rupture ductility and shoulder ductility ($\Delta D/D$) decreased with reduced hoop stress, increased grain size and lower test temperature.
11. There is too much scatter in the rupture ductility data for the annealed materials to determine the effect of hoop stress and rupture ductility. The shoulder ductilities however do initiate a downward trend with reduced stress.
12. The inclusions in the double vacuum-melted, Type 316 stainless steel in the as-received condition include MnS, alpha quartz, and a small quantity of MC-type carbide. After heat treatment at 900°F for 24 hours plus 1300°F for 216 hours, the major precipitates are $M_{23}C_6$ and Fe_2Mo Laves phase, in about equal proportion. Planetary swaged tubing from another lot, similarly heat treated, contained a small amount of sigma phase in addition to $M_{23}C_6$ and Fe_2Mo .
13. The effect of pre-test heat treatment of 20 percent cold worked tubing, at 900°F for 24 hours plus 1300°F for 216 hours, on the short-term, biaxial tensile ductility is to give properties intermediate between annealed and cold worked tubing. The effect on longer-term stress rupture properties is similar, but closer to the annealed material properties, at least out to about 300 hours at 1300°F and 1800 hours at 1200°F.
14. Wall thickness variations were observed in nearly every post-test, stress rupture specimen examined, indicating non-uniform circumferential strain during test.
15. From the local wall thickness measurements the following can be inferred:
 - a. Non-uniform circumferential straining is probably the result of non-uniform wall thickness in the starting tubing.
 - b. Significant eccentricity in the starting tubing reduces the potential $\Delta D/D$ ductility at rupture which could be obtained based on the material properties.
 - c. This type of measurement can be used to help separate the effects of material properties from mechanical variations in the tubing. It could also be used to help evaluate the effects of surface defects on both the ductility and rupture life in biaxial tests. The effects of these variables are confounded in the standard $\Delta D/D$ measurements.
16. The post-stress rupture test specimens of cold worked materials contain grain boundary creep voids over the whole range of test conditions. This, together with other observations indicates that one of the major controlling deformation mechanisms in the cold worked materials is grain boundary sliding.

17. The pronounced grain distortion in the hoop direction of the annealed materials, the general absence of grain boundary creep voids, and the insensitivity of stress rupture behavior to grain boundary creep size (corresponding to a factor of 4 inch grain area) indicate that the major controlling deformation mechanism in the annealed materials is deformation within the grains by slip and climb.
18. The planetary swaged tubing, interpreted as having a higher level of effective cold work than the plug drawn tubing based on hardness, transmission microstructure, and precipitation kinetics, showed some marked difference in stress rupture behavior from the plug drawn material. At reduced stress levels and long rupture times the structure near the OD of the tube recrystallized to finer grain size and the recrystallized annulus increased in size with reduced stress. Correlated with the start of recrystallization, the site of rupture initiation and the location of the area of highest concentration of creep voids shifted from the OD to ID. The cold worked, plug drawn tubing also showed creep voids but there was no indication of recrystallization based on grain size. The location of the creep voids and the initiation of rupture remained associated with the OD over the whole range of test conditions.
19. Considering all measurements, tests, and observations together, the most important materials variable in this program is the level of cold work; this is followed in rank by grain size and then, at a much lower level of importance, melting practice and fabrication procedure.

5.2 RECOMMENDATION

Since some of the effects of irradiation are similar to adding cold work to the structure, and since it was shown in this investigation of unirradiated mechanical properties that annealed material has greater stress rupture life and greater ductility than the cold work material at low stresses, it is recommended that the annealed condition of Type 316 stainless steel be given further consideration for LMFBR cladding and for other applications involving low stresses and low strain rates. The microstructures presented here and the interpretations indicate that if the grain structure becomes sufficiently hardened by irradiation there may be a shift in the controlling strain mechanism from grain deformation to grain boundary shear. Even if this does occur, the annealed material may still have some advantage for low strain rate applications because the formation of creep voids, which appear to control rupture behavior of the cold worked materials, would begin to form at a later stage in irradiation.

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