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STAINLESS STEEL CLADDING DEVELOPMENT
QUARTERLY PROGRESS REPORT
FOR THE PERIOD ENDING JUNE 30, 1970

Approved:

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Contract AT(30-1)-4135
U. S. Atomic Energy Commission
Submitted to AEC-NY00 in August 1970

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SECTION 1 INTRODUCTION

1. GENERAL

This is the eighth quarterly progress report and covers the work performed during the fourth quarter of FY-1970.

2. SUMMARY OF OBJECTIVES

This project is focused on the short term, and is concerned with steps to improve commercially available Type 316 austenitic stainless steels for use as cladding in LMFBR fuel pins. The overall project objective is to determine the influence of structure on mechanical properties of Type 316 austenitic stainless steel so that optimum specifications, which will assure maximum performance under LMFBR service conditions, can be selected.

The importance of structure on mechanical properties has been well demonstrated in the published literature. For example, in one series of experiments,^[1] prestraining of Type 316 austenitic stainless steel, followed by a specific heat treatment to produce carbide precipitation, decreased the minimum creep rate at temperatures of interest to LMFBR by as much as a factor of 250, and increased creep rupture life by as much as a factor of 25.

Initially in the cladding development program, a survey will be made to determine tubing material property and microstructural variability based on procurement of tubing under an existing FFTF specification. This specification will be used as a starting point. Materials with various fabrication histories will be procured and evaluated in both the as-fabricated condition and after post-fabrication heat treatment. The relations between mechanical properties and structure will be investigated.

It is anticipated that this first phase of the cladding program will culminate at the end of FY-1972 with a definition of the most desirable mechanical and microstructural properties in austenitic stainless steel, to maximize the strength and ductility of the material under the most limiting LMFBR service conditions.

3. SUMMARY OF PRIOR WORK

All work related to the present investigations and performed prior to the present period has been fully reported in:

WARD-3791-26, "Stainless Steel Cladding Development Quarterly Progress Report for the Period Ending September 30, 1968."

- WARD-3791-29, "Stainless Steel Cladding Development Quarterly Progress Report for the Period Ending December 31, 1968."
- WARD-3791-35, "Stainless Steel Cladding Development Quarterly Progress Report for the Period Ending March 31, 1969."
- WARD-3791-41, "Stainless Steel Cladding Development Quarterly Progress Report for the Period Ending June 30, 1969."
- WARD-4135-2, "Stainless Steel Cladding Development Quarterly Progress Report for the Period Ending September 30, 1969."
- WARD-4135-5, "Stainless Steel Cladding Development Quarterly Progress Report for the Period Ending December 31, 1969."
- WARD-4135-9, "Stainless Steel Cladding Development Quarterly Progress Report for the Period Ending March 31, 1970."

4. SUMMARY OF CURRENT PROGRESS

Under Task SSBA-110, project administration and day-to-day liaison with the AEC/RDT continued.

Under Task SSBA-210, microcharacterization and mechanical property testing, including biaxial tensile, biaxial stress rupture, and both uniaxial and biaxial creep were continued on ARD vacuum melted materials.

Comprehensive characterization, including texture evaluation, was completed for two lots of planetary swaged tubing and the results were compared to plug drawn material.

Biaxial tensile testing at room temperature and five elevated temperatures was completed on four tubing lots, completing the biaxial tensile testing planned for the vacuum melted steels program. Results were compared for the evaluation of the effects of the independent variables on the properties of tubing.

Thirty-eight stress rupture tests were completed, and longer term tests are continuing. The results from seven lots of material representing various combinations of the independent variables were compared.

Classification of the mode of rupture of post-test biaxial stress rupture specimens continued. Strain measurements of fifteen specimens were completed and results compared. Metallographic examination of selected specimens from this group is planned.

A shipment of 500 feet of Battelle-Northwest (BNW), double vacuum melted, Type 316 stainless steel tubing has been received for use in the Oxide Fuel Element Development Program.

SECTION 2

SSBA-110 PROJECT ADMINISTRATION

F. A. Brassart, W. B. Heubel, and P. Murray

1. OBJECTIVES

The objectives of this task are to assure that the project is successfully completed, on schedule, within budget, and to the satisfaction of the Atomic Energy Commission (AEC); to assure compliance with contracted obligations; and to coordinate this project with other AEC-sponsored and Westinghouse-sponsored LMFBR development projects.

Overall project direction and day-to-day administration will be provided under this task. Plans and controls will be established and maintained, periodic reviews will be held with the AEC, correspondences and reports will be coordinated, and day-to-day technical and administrative liaison with the AEC will be provided.

2. PRIOR WORK

Work programs for FY-1970 were submitted to the Commission in June 1969. Technical work has continued on the basis of those work programs. The project is divided into the following tasks for FY-1970 administrative control.

SSBA-110 Project Administration

SSBA-210 Evaluation of Austenitic Stainless Steel Cladding

SSBA-211 Effect of Cladding Properties and Interactions

A revised work program to cover SSBA-211 was submitted in December 1969. SSBA-211 was scheduled for completion in February 1970; however, at RDT's request, the scope of this task was increased. A final report for the task will be published in July 1970; no separate section of current progress, for that task, is included in this report.

3. CURRENT PROGRESS

The quarterly progress report, WARD-4135-9, for the period ending March 31, 1970, was prepared and distributed.

Schedule 189a/Work Plan for FY-1971 was prepared and submitted to the New York Operations Office.

SECTION 3

SSBA-210 EVALUATION OF AUSTENITIC STAINLESS STEEL CLADDING

M. G. Cowgill, T. R. Padden, W. E. Ray,
H. J. Snyder, E. J. Tarby, and K. C. Thomas

1. OBJECTIVES

The objective of this task is to characterize in detail, by microstructural analysis, chemical analysis, and mechanical property testing, thin-wall stainless steel tubing fabricated under carefully controlled commercial practice. This information will be used to:

1. Enhance the understanding of the effect of fabrication processes and resulting structure on the properties of thin-wall tubing.
2. Provide out-of-reactor data required to predict and improve swelling behavior and strain limits of cladding in LMFBR environments.

The objective of a supplementary weldability investigation is to determine the effect of the degree of cold working on the weldability of Type 316 stainless steel tubing.

2. PRIOR WORK

A detailed test program was prepared. Air melted, annealed Type 316 stainless steel tubing, 0.300-inch OD at three wall thicknesses (0.010, 0.012, and 0.014-inch), and 0.250-inch OD x 0.016-inch was procured. Extensive biaxial mechanical property testing at 850, 1000, 1200, and 1300°F, and chemical and microstructural evaluation of the tubing were performed.

Cold worked (15 to 20 percent), air melted tubing and micropeened air melted tubing were also procured. A study to determine the effects of micropeening on mechanical properties was performed, and an investigation of the effects of cold work was initiated.

All twelve lots of ARD vacuum melted tubing representing three melting practices, two grain sizes, and two conditions (20 percent cold worked and solution annealed) were received and accepted. Microcharacterization and mechanical property testing, including biaxial tensile, biaxial stress rupture, and both uniaxial and biaxial creep were initiated.

Chemical analysis and general metallographic characterization of the ARD vacuum melted tubing were completed.

Comprehensive structural characterization of two ARD double vacuum melted, plug drawn tubing lots, including electron microscopy, and probe analysis as well as electron and X-ray diffraction analysis was completed.

Comprehensive structure characterization of the planetary swaged materials, including transmission and scanning electron microscopy, electron and X-ray diffraction, and texture analysis by pole figures, was 75 percent completed.

Post test stress rupture specimens have been classified according to rupture type, and microstructural evaluation of selected specimens has been initiated.

Biaxial stress rupture data scatter was evaluated, and a temperature correction procedure was proposed for the minor temperature variation ($\pm 15^{\circ}\text{F}$) inherent in the test.

Additional low cycle, high strain fatigue tests were conducted. The new specimen design, testing procedure, and results from the recent tests were evaluated.

A thermal aging study of several lots of Type 316 stainless steel was completed for times up to 5000 hours.

The experimental phase of an investigation on the effects of degree of cold work on weldability was completed; a summary is being prepared. A low cycle high strain fatigue study was initiated.

All ten lots of BNW, double vacuum melted tubing for the Oxide Fuel Element Development Program (19-pin encapsulated assembly) were received and accepted. A partial shipment of 250 feet of 20 percent cold worked, BNW double vacuum melted, Type 316 stainless steel tubing was received and accepted for use in the Oxide Fuel Element Development Program (37-pin assembly). A shipment of 200 feet of 20 percent cold worked, double vacuum melted, Type 316 stainless steel tubing, manufactured by planetary swaging was received for use in the characterization studies, and mechanical property testing was initiated.

3. CURRENT PROGRESS

TEST PROGRAM

Testing continued as outlined and defined in the seven previous quarterly reports (Section 1).

PROCUREMENT AND MATERIAL HANDLING

Vacuum Melted Type 316 Stainless Steel Tubing for the Oxide Fuel Element Development Program (37-Pin Assembly)

Two additional lots of BNW, VM(IVCEV) Type 316 stainless steel tubing were received. One lot contained 300 feet of tubing in the annealed condition

and the other 200 feet in the 20 percent cold worked condition. This shipment, plus the 500 feet of 20 percent cold worked tubing received previously completes the order; the non-destructive test records for the last shipment have not yet been received.

To date, 200 feet of the 20 percent cold worked, BNW, VM(IVCEV)* Type 316 stainless steel tubing have been shipped to the Westinghouse ARD Cheswick site for use in fabrication of the 37 pin assembly. In addition, three lots of Westinghouse, VM(IVCEV), Type 316 tubing were shipped to the Cheswick site for use in producing fuel pins for flow and fretting tests. A total of 236 feet of cold worked tubing, and 106 feet of annealed tubing were included in these shipments.

STRUCTURAL CHARACTERIZATION**

Comprehensive Analysis of Planetary Swaged Tubing

Comprehensive characterization has been completed for both the cold worked and the solution annealed, planetary swaged, Type 316 stainless steel tubing lots. The results are summarized in Table 1; results for comparable plug drawn lots were reported in Reference 2.

General Microstructure Planetary Swaged Tubing

Scanning electron microscopy examination of the as-received surfaces of both tube lots (49193, 20% CW; and 49194, annealed) revealed some grinding marks and 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 electro-polished outer surfaces of both tube lots indicated an inclusion particle density of about 1×10^6 particles/cm² in both lots, and a grain size of ASTM 6 after etching.

The grains in the solution annealed sample were equiaxed and contained annealing twins, whereas the cold worked sample consisted of slightly elongated grains containing twins and a very fine, irregular, needle-like substructure. The frequency of the needles was considerably higher in the cold worked, planetary swaged tubing than in the 20 percent cold worked, plug drawn lot (42402) described previously.[2]

Transmission electron micrographs of the solution annealed, planetary swaged tubing revealed a dislocation density of $6(+3) \times 10^8$ dislocations/cm², and a general structure similar to that observed for the annealed,

* Double vacuum melted, induction vacuum plus consumable electrode

** B. Weiss and R. Stickler (WRL), and T. R. Padden (ARD)

Table 1. Summary of Results		
ARD, VM(IVCEV) Type 316 SS - Planetary Swaged Tubing		
	Tube Lot 49194 (Annealed)	Tube Lot 49194 (20% Cold Worked)
1. <u>Microstructure</u>		
Grain Size according ASTM No.	6	6
Grain Morphology	equiaxed and twins	slightly elongated, twins and needle-like structure
Dislocation Arrangement	Some arranged in rows	Heavy dislocated structure with numerous complex faults and micro-twins
Dislocation Density (dislocations/cm ²)	6 (+3) x 10 ⁸	$\sim 10^{11} - 10^{12}$
Stacking Fault Energy (erg/cm ²)	~ 25	No measurable from TEM's
2. <u>Second Phase Particles</u>		
Arrangement	Aligned into stringers parallel to tube axis	Same
Amount	Approx. 10 ⁶ particles/cm ²	
Identification by ED	Not Possible	Same
by XD	a - SiO ₂ (~ 0.13 wt%)	
3. <u>Residual Stress</u>		
Outer Surface	Strain Free	Cold Worked
Inside Surface	Strain Free	Cold Worked

plug drawn lot (42403). Dislocation nodes were observed but their width was close to the limit of resolution indicating a stacking fault energy of 25 to 30 erg/cm².

Transmission electron micrographs of the cold worked tubing revealed a high dislocation density estimated to be 10¹¹ to 10¹² dislocations/cm², similar to that reported for the cold worked, plug drawn lot, and numerous bands of irresolvable fine substructure which exhibited weak fringe contrast.

A detailed analysis of the diffraction contrast of similar bands observed in well characterized bulk Type 316 stainless steel, having known amounts of cold work achieved by rolling, was reported by Weiss and Stickler.^[3] It was shown that the extinction conditions were similar to those reported for stacking faults. The bands were interpreted as clusters of stacking faults produced by slip on closely spaced slip planes. It was also shown that cold work exceeding 20 percent increased the number of micro-twins considerably.

Based on the results in Reference 3, the higher frequency of micro-twins observed in the cold worked planetary swaged tubing, relative to the plug drawn tubing, was interpreted as a higher degree of effective cold work. This apparent higher degree of cold work is in agreement with the higher hardnesses reported previously for this tubing lot.

A review of the fabrication histories of the cold worked, plug drawn, and cold worked, planetary swaged lots revealed that both lots had received the specified 20 percent cold work, based on percent reduction of the cross sectional area; the uncertainty was less than 2 percent.

Identification of Second Phase Particles

Electron micrographs of carbon-extraction replicas, revealed some second phase particles; however, they were too fine in size to be identified by electron diffraction. In some instances, impressions on the replicas indicated that some larger particles present in the sample had not been extracted. The amount of bulk extracted residues was 0.13 percent of the specimen weights for both the annealed and the cold worked, planetary swaged tubing lots. X-ray diffraction analysis showed the presence of α -quartz (ASTM Card No. 5-0490) in both materials.

Analysis of Precipitates After Heat Treatment

Metallography and analysis of extracted precipitates after "Garofalo" heat treatment (900°F/24 hours plus 1300°F/216 hours) showed a higher frequency of precipitates and a greater tendency for precipitation on deformation bands and twins in the planetary swaged tubing; this is indicative of a higher effective cold work in the planetary swaged tubing. The concentrations of the precipitates based on chemical bulk extractions were 1.7 w/o for the planetary swaged material and 1.3 w/o for the plug drawn lot. The extracts from both heat treated lots contained approximately equal amounts of M₂₃C₆ and Laves phase (Fe₂Mo); however the planetary swaged lot also contained some sigma phase.

Residual Stress

Laue back reflection X-ray diffraction patterns were made of the inside and outside surfaces of specimens from both planetary swaged tubing lots. Both outside and inside surfaces of the annealed tube sample yielded a spotty ring pattern, characteristic of strain-free material. The X-ray diffraction pattern from the cold worked tube sample consisted of smooth rings with an indication of a weak texture.

Textures of Plug Drawn and Planetary Swaged Tubing*

Crystallographic textures of Type 316 stainless steel plug drawn tubing, lots 42402 and 42403, were determined as part of a microstructural study. Textures of planetary swaged tubing of the same double vacuum melted heat were later determined so that a correlation between texture and the tube fabrication process can be made. The texture of the tube samples was determined by a conventional X-ray goniometer technique (Siemens Kristalloflex-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 1-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 1-inch by 1-inch in size.

The (200) and (111) Pole Figures for the drawn tubing in the cold worked and the annealed conditions are shown in Figures 1 and 2, and for the planetary swaged tubing, also in the cold worked and in the annealed conditions in Figures 3 and 4.

The Pole Figures in Figures 1 and 2 for the plug drawn lots and their interpretation supersedes those previously reported as Figures 10a and 10b of Reference 2. The pole figure reported previously was mis-identified in the print out and consequently the interpretation was incorrect. The texture of the drawn tubing (lot 42402, 20% cold worked), based on the (200) and (111) Pole Figures, consists of two components:

COMPONENT 1

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

* Work performed by B. Weiss and W. T. Reynolds at Westinghouse Research and Development Laboratories.

Figure 1. Plug Drawn Type 316 SS Tubing-(200) Pole Figures

Figure 2. Plug Drawn Type 316 SS Tubing-(111) Pole Figures

Figure 3. Planetary Swaged Type 316 SS Tubing-(200) Pole Figures

Figure 4. Planetary Swaged Type 316 SS Tubing-(111) Pole Figures

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

COMPONENT 2

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

or $\{011\}_{TS} \langle 100 \rangle_{AD}$

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.^[4,5] The $\{112\} \langle 111 \rangle$ and $\{110\} \langle 100 \rangle$ components are found in rolling textures of Fe-Ni alloys.^[4] Comparison of the Pole Figures in Figures 1 and 2 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 x 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 of the drawn tubing.

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

COMPONENT 1

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

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

$\{011\}_{TS} \langle 111 \rangle_{AD}$

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 in 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 broadened, low intensity texture, essentially the same as that of the cold worked tubing ($\{110\}_{TS} <111>_{AD}$). These textures are compared in Table 2 with the results of texture analysis of 0.2% Ti Type 304L stainless steel tubing reported by Nilsson.^[6] Equivalent notation is used. Note that the $[112]_{RD}[111]_{AD}$ component was not observed in the drawn Type 304 stainless steel.^[6] 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 304 L tubing).

Textures of the planetary swaged Type 316 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 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.

MECHANICAL PROPERTIES

Biaxial Tensile Testing

Four lots of ARD, VM Type 316 stainless steel tubing were biaxial tensile tested at room temperature and at five elevated temperatures in the range of 850 to 1300°F. This essentially completes the biaxial tensile testing planned for the vacuum melted steels program except for a few individual test points. The ultimate tensile strengths, the 0.2 percent offset yield strengths, and the uniform diametral strains for the four lots are listed in Table 3. These results are compared in Figures 5 to 13, with results reported previously.^[2,7]

A comparison of three vacuum melting practices, for the fine grained, 20 percent cold worked condition is given in Figures 5, 6, and 7. The double vacuum melted lot has slightly higher ultimate and yield strengths, and lower ductility than the single vacuum melted lots at 1300°F, but considering the whole temperature range, there is little difference in biaxial tensile properties attributable to vacuum melting practice. However, the 1200°F strain for the induction vacuum melted heat is inconsistent with the rest of the data. The records of this test will be checked to determine the source of the discrepancy.

Figures 8, 9, and 10 compare the results from fine-grained and coarse-grained, plug drawn tubing with the results from coarse-grained planetary swaged tubing. All three tubing lots were in the 20 percent cold worked condition. 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 from the coarse-grained,

Table 2. Textures of Stainless Steel Tubing

Deformation Process	Type 316 Tubing		0.2% Ti Type 304L Tubing (Ref.6)	
	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 }	$[011]_{RD}[1\bar{1}\bar{1}]_{AD}$	—	—
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	

Table 3. Biaxial Tensile Results from Various Heats of ARD, VM Type 316 Stainless Steel Tubing

Lot	Melting Practice	Certified Grain Size	Condition	Hardness DPH (1)	Mechanical Properties						
					Property	RT	850°F	1000°F	1100°F	1200°F	1300°F
42394 Item 1	CEV	9/9.5	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	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
42405 Item 12	IVECV	5/5.5	Anneal.	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	--	--
49194 Item 20	IVCEV	6	Plan. Swage. Anneal.	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
(1) Average of Seven Measurements						IV - Induction Vacuum Melt					
500 gram load - 20% CW						CEV - Consumable Electrode Vacuum Melt					
300 gram load - Annealed						IVCEV - Induction Vacuum plus Consumable Electrode Vacuum Melt					
<u>Mechanical Properties</u>						FG - Fine Grain ASTM 8-9					
a Ultimate Tensile Strength (ksi)						CG - Coarse Grain ASTM 5-6					
b 0.2% Yield Strength (ksi)											
c Uniform Diametral Strain (%)

Figure 5. Cold Worked Plug Drawn Tubing Type 316 Stainless Steel Made by Three Vacuum Melting Practices - Biaxial Ultimate Tensile Strength vs Temperature

3789-1

Figure 6. Cold Worked Plug Drawn Tubing Type 316 Stainless Steel Made by Three Vacuum Melting Practices - Biaxial 0.2% Offset Yield Strength vs Temperature

3789-2

Figure 7. Cold Worked Plug Drawn Tubing Type 316 Stainless Steel Made by Three Vacuum Melting Practices - Biaxial Tensile Test Uniform Diametral Strain vs Temperature

3789-3

Figure 8. Cold Worked Type 316 Stainless Steel Tubing Fabricated by Plug Drawing and by Planetary Swaging - Biaxial Ultimate Tensile Strength vs Temperature

Figure 9. Cold Worked Type 316 Stainless Steel Tubing Fabricated by Plug Drawing and by Planetary Swaging - Biaxial 0.2% Offset Yield Strength vs Temperature

3789-5

Figure 10. Cold Worked Type 316 Stainless Steel Tubing Fabricated by Plug Drawing and by Planetary Swaging - Biaxial Tensile Test Uniform Diametrical Strain vs Temperature

Figure 11. Annealed Type 316 Stainless Steel Tubing Fabricated by Plug Drawing and by Planetary Swaging - Biaxial Ultimate Tensile Strength vs Temperature

3789-7

Figure 12. Annealed Type 316 Stainless Steel Tubing Fabricated by Plug Drawing and by Planetary Swaging - Biaxial 0.2% Offset Yield Strength vs Temperature

3789-8

Figure 13. Annealed Type 316 Stainless Steel Tubing Fabricated by Plug Drawing and by Planetary Swaging - Biaxial Tensile Uniform Diametral Strain vs Temperature

3789-9

planetary 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).

It would appear from these results that planetary swaging has a real effect, and that it tends to be opposite to the effect of increasing grain size. Two possible reasons are suggested by the results of the characterization part of the program: First, planetary swaging introduces a different crystallographic texture than plug drawing at least for the fabrication schedules used for these materials, and second, 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. A comparison of the stress rupture results from the same three lots of material is given in a later section of this report.

The results from the same combination of variables of plug drawing, planetary swaging, and grain size, except in this case for the annealed condition, are given in Figures 11, 12, and 13. Again, the fine-grained, plug drawn lot has higher ultimate and yield strength than the coarse-grained, plug drawn lots, and lower ductility, at least at the intermediate temperatures 1100°F and 1200°F. The coarse-grained, planetary swaged lot has approximately the same ultimate strengths as the coarse-grained, plug-drawn lot, but the yield strengths are higher at all temperatures and the ductilities are about the same for the three temperatures for which data have been obtained (850, 100 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 increased yield strength is related to some real difference associated with planetary swaging, other than grain size. The most likely factor in this case is the crystallographic texture discussed earlier.

Biaxial Stress Rupture Tests

Table 4 gives the results of eight biaxial stress rupture tests that were completed during the report period. Figures 14 through 17 are graphs of the biaxial stress rupture results from seven tubing lots representing partial comparisons of some of the variables being tested, namely three vacuum melting practices, plug drawing, planetary swaging, fine and coarse grain size, as well as annealed and 20 percent cold worked conditions. The data obtained at $1300 \pm 15^\circ\text{F}$ were normalized to 1300°F using the procedure described previously. [8] Figure 14 is a comparison of the three vacuum melting practices for the plug drawn, fine-grain sized, 20 percent cold worked condition. The double vacuum melted lot of tubing has very slightly better stress rupture properties than the induction vacuum melted lot and significantly better properties than consumable electrode vacuum melted lot for rupture times up to about 900 hours. The double vacuum melted material, for which there is longer term data, shows a change in the slope of the log stress versus log rupture time curve at about 900 hours at 1300°F. Additional data extending to at least 2000 hours are planned.

Table 4. Biaxial Stress Rupture Data for Type 316 Stainless Steel Tubing

Lot	Material Condition	Tubing Dimension (in.)	Nom. Temp. (°F)	Test Temp. (°F)	Stress (ksi)	Rupture Time (hr)	Rupture Type
42394	CEV-20%CW-FG	0.015	1300	1294	18	588.0	Pinhole
		0.015	1300	1315	15.5	585.8	Pinhole
42398	IV-20%CW-FG	0.015	1300	1296	19	746.2	Pinhole
		0.015	1300	1317	17.7	336.0	Pinhole
42402	IVCEV-20%CW-FG	0.015	1200	1198	33	731.0	Split
		0.015	1200	1213	29	938.9	Split
		0.015	1200	1213	26	1821.7	Pinhole
		0.015	1200	1214	24	2124.2	Pinhole
		0.015	1300	1318	17	592.2	Pinhole
		0.015	1300	1314	15	805.4	Pinhole
42403	IVCEV-ANN.-FG	0.015	1300	1316	25	7.6	Pieces
		0.015	1200	1213	25	113.0	Split
		0.015	1200	1197	23	718.5	Split
		0.015	1300	1295	21	138.1	Split
		0.015	1200	1199	21	1513.1	Split
		0.015	1300	1303	17.7	203.9	Split
		0.015	1300	1292	15	702.5	Split
		0.015	1300	1313	13	867.0	Pinhole
42404	IVCEV-20%CW-CG	0.015	1200	1204	41	123.9	Split
		0.015	1200	1205	37	415.2	Pinhole
		0.015	1200	1187	32.6	1480.0	Pinhole
		0.015	1300	1295	29	69.5	Split
		0.015	1300	1305	27	106.8	Split
		0.015	1300	1318	24	169.9	Pinhole
		0.015	1300	1310	22.2	193.4	Pinhole
		0.015	1300	1286	21	807.5	Split
		0.015	1300	1315	19.2	554.1	Pinhole
49193	IVCEV-20%-PS-CG	0.015	1200	1189	37	307.7	Pinhole
		0.015	1300	1292	31	129.5	Split
		0.015	1200	1186	28	1142.0	Pinhole
		0.015	1300	1316	20	186.4	Pinhole
		0.015	1300	1315	19.7	244.5	Pinhole
		0.015	1300	1293	16	685.9	Pinhole
		0.015	1300	1315	13	507.9	Pinhole
		0.015	1300	1300	10.5	1493.4	Pinhole
49193	IVCEV-20%CW-PS+GHT	0.015	1300	1294	21	95.1	Pinhole
49194	IVCEV-ANN-PS-CG	0.015	1300	1300	15.4	501.6	Pinhole
		0.015	1300	1296	13.5	984.9	Pinhole

CW - Cold Worked

ANN - Annealed

FG - Fine Grain

CG - Coarse Grain

PS - Planetary Swaged

GHT - Garofalo Heat Treat

CEV - Consumable Elec.

IV - Induction Vac.

IVCEV - Consumable Elec. Plus Induction Vac.

Figure 14. Biaxial Stress Rupture Results - 1300°F, Plug Drawn Tubing Type 316 Stainless Steel Made by Three Vacuum Melting Practices. Fine Grain Size, 20% Cold Worked

3789-10

Figure 15. Biaxial Stress Rupture Results - 1300°F, Cold Worked Type 316 Stainless Steel Tubing Fabricated by Plug Drawing and by Planetary Swaging

3789-11

Figure 16. Biaxial Stress Rupture Results - 1300°F, Annealed Type 316 Stainless Steel, Fabricated by Plug Drawing and Planetary Swaging

3789-12

Figure 17. Biaxial Stress Rupture Results - 1300°F, Planetary Swaged Type 316 Stainless Steel Tubing, Grain Size 6, Annealed and 20% Cold Worked Conditions

3789-13

These data should permit additional comparison of the various melting practices, after the expected break in the stress rupture curves occurs.

The long term, double vacuum melted tubing specimens have not yet been examined metallographically but presumably the break in the curves is the result of recovery and recrystallization. Selected specimens will be examined during the next report period.

Figure 15 shows a comparison of results from plug drawn material, both fine and coarse-grain size with results from coarse-grained, planetary swaged material (all double vacuum melted Type 316 stainless steel in the 20 percent cold worked condition). There is no significant difference attributable to grain size in the plug drawn lots at least out to about 900 hours at 1300°F. There is a marked difference however, between results from the plug drawn lots and results from the planetary swaged lot. The fine-grained, plug drawn lot, for which there are data points out to 1250 hours, shows the break in the stress rupture curve at about 900 hours whereas the planetary swaged lot shows a break at about 300 hours.

Figure 16 shows a comparison of results from plug drawn and planetary swaged material in the annealed condition. The difference between these lots is, at most, very slight and may not be significant. A few more data points are needed to complete this comparison.

By combining the stress rupture observations for the annealed condition with the cold worked condition and assuming that the lack of a grain-size effect in the plug drawn material also applies to the planetary swaged material (no interaction of variables); it appears that the poorer stress rupture properties of the planetary swaged material relative to the plug drawn material in the cold worked condition is the result of the higher degree of effective cold work in the planetary swaged lot, and that this level of cold work is above optimum. The lack of a difference in annealed condition also indicates that if there is a beneficial effect on stress rupture properties to be gained by planetary swaging through structural effects such as texture, then future experimental tubing lots should be fabricated with a greater fraction of the reduction accomplished by planetary swaging to accentuate the structural differences. This approach is planned in the next phase of the program.

Figure 17 is a comparison of results from annealed and from cold worked, planetary swaged materials of the same grain size (grain size 6). These curves show that although the cold worked lot starts out at higher stress levels for equivalent rupture times, a break in the line occurs at about 300 hours and as a result of the steeper slope of the new line segment, the stress rupture curves for the cold worked and the annealed material intersect at about 700 hours at 1300°F, and a hoop stress of 14,000 psi. Additional data from longer time tests in progress are expected to show whether the annealed material will have better long-term, stress rupture properties than the cold worked material at 1300°F for this level of cold work.

Post test strain measurements have been completed for selected specimens from this series of tests and metallographic examination is planned.

CHARACTERIZATION OF POST TEST BIAXIAL STRESS RUPTURE SPECIMENS

General

Thus far 83 post test biaxial stress rupture specimens have been classified according to rupture type, material, stress, and temperature. The general observations reported previously^[8] still appear to be valid.

1. The cold worked, planetary swaged material tends to rupture with a Type 1 fracture (Pin hole).
2. The annealed materials tend to fail with a high fraction of Type 2 or Type 3 rupture (Splits or multiple pieces).
3. The cold worked, plug drawn materials are intermediate between (1) and (2).
4. In general the tendency for Type 1 rupture is greater at lower stresses.

Post Test Strain Measurements

Detailed strain measurements were obtained from 15 post test biaxial stress rupture specimens selected to give partial comparisons of the variables being tested (stress, temperature, grain size, annealed and cold worked conditions, and plug drawn and planetary swaged fabrication methods). Diametral strain along two perpendicular diameters was measured with a comparitor at various positions along the length of the specimens and the average value plotted as a function of position.

The strain at the rupture was measured in two ways. For pin hole type ruptures, the average circumference obtained from comparitor measurements was corrected for the width of the pin hole rupture and converted to equivalent diameter. For split and tear type ruptures, the equivalent circumference and thus the equivalent diameter was obtained from impressions made by rolling the specimen against the edge of a lead strip and measuring the length of the imprint with a calibrated microscope. In presenting the results (Figures 19, 20, and 21), the points representing comparitor strain measurements were arbitrarily connected by straight lines. The length of the ruptures parallel to the tube axis in split and multiple piece fractures (Type 2 and Type 3) are indicated by vertical bars. The strain measurements at the fractures, measured by the impression method, are shown as X's. The location of the pin hole ruptures is indicated by the arrows.

Figure 18 illustrates the sequence of increasing severity of rupture that is observed in annealed materials with increasing applied stress. The diametral strain measurements for five of these specimens are shown in Figure 19. This set of specimens is from the double vacuum melted, plug drawn, fine-grained lot (42403). (Note that the order of presentation has been changed from that of Figure 18). The first four plots in Figure 19 are for test temperature of $1300 \pm 15^\circ\text{F}$, and the last is for $1200 \pm 15^\circ\text{F}$.

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

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Figure 19. Diametral Strain Along Length of Type 316 Stainless Steel, Post Test, Biaxial Stress Rupture Specimens, Plug Drawn, Solution Annealed, Grain Size 8, Lot 42403

Figure 20. Diametral Strain Along Length of Type 316 Stainless Steel Post Test, 1300°F Biaxial Stress Rupture Specimens, Plug Drawn 20% Cold Work

Figure 21. Diametral Strain Along Length of Type 316 Stainless Steel, Post Test, 1300°F Biaxial Stress Rupture Specimens Planetary Swaged-
 3789-17 ASTM GS 6

The strains ($\Delta D/D\%$) at the rupture location in this series of samples vary from 20 to 39.5 percent, and, except for the pin hole specimen, there is an increase in $\Delta D/D\%$ with increasing stress. The average strain away from the rupture is approximately 10 to 20 percent and, in general, the strain at the rupture is about twice that of the adjacent positions in the specimen. It is significant to note that despite the difference in the mode of rupture, the particular specimens shown in Figure 19 A and D, the pin hole specimen and the multiple rupture specimen, have approximately the same strain at the failure location, (35.4 percent and 39.5 percent respectively). Although the pin hole specimen appeared to bulge more on one side of the tube than the other, measurements for ovality at the rupture showed that the specimen was close to circular at this cross section, but the center of this circular cross section was displaced 0.018-inch with respect to the tube axis. Metallographic examination of this cross section is planned.

There appeared to be little effect of test temperature on the mode of rupture of the annealed material but there was a marked effect of applied stress.

Strain measurements for a set of plug drawn, 20 percent cold worked specimens, all tested at $1300 \pm 15^\circ\text{F}$, are shown in Figure 20. The first four plots in this figure are for fine-grained specimens and the last is for a coarse-grained specimen. Because of the generally lower strains, the ordinate scale of these plots has been changed relative to the previous figure. Two of the specimens ruptured with pin holes and three with split and tear Type 2 ruptures. For the fine-grained lot the strains at the rupture increased with applied stress from 5.5 percent to 14.7 percent, and the average strains away from the rupture were in the range of 4 to 8 percent. The coarse-grained specimen had lower strains at both the rupture area and away from the rupture than the fine-grained specimens. In addition to the generally lower strains, the ratio of the strain at the rupture to the average strain away from the rupture was generally lower for the cold worked specimens than for the annealed specimens.

Strain measurements for a set of planetary swaged specimens, all tested at $1300 \pm 15^\circ\text{F}$, are shown in Figure 21. The first four plots are for 20 percent cold worked specimens and the last is for an annealed specimen; all were coarse grain size. The annealed specimen in this set was the data point which lay near the intersection of the stress rupture curves for the annealed and the cold work, planetary swaged lots shown in Figure 17, and Figures 21A and B are from the cold worked specimens that bracket it. The scale of this figure is the same as that of Figure 20.

For the cold worked, planetary swaged specimens the strains of the rupture were in the range of 5 to 8.5 percent and the average strains away from the rupture were about 4 to 8 percent. There was very little increase in the strain at the rupture relative to the adjacent areas. In general, the strains in this set were lower than the fine-grained, plug drawn, cold worked lot but higher than the coarse-grained lot.

There are great differences in the post-test strains between the annealed, planetary swaged specimen (Figure 21E) and the two cold worked, planetary swaged specimens which bracketed it on the stress rupture curves. The annealed specimen had twice the ductility in areas away from the rupture

and about 5-1/2 to 6 times the ductility at the rupture area than the cold worked specimens.

Metallographic examination of selected areas of these 15 specimens is planned to further elucidate the relation between mode of rupture and the other variables being investigated.

SECTION 4
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