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STAINLESS STEEL CLADDING DEVELOPMENT
QUARTERLY PROGRESS REPORT
FOR THE PERIOD ENDING DECEMBER 31, 1969

Approved by:

P. MurrayContract AT(30-1) 4135
U. S. Atomic Energy Commission

Submitted to AEC-NYOO in February 1970

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Printed in the United States of America
Available from
Clearinghouse for Federal Scientific and Technical Information
National Bureau of Standards, U. S. Department of Commerce
Springfield, Virginia 22151
Price: Printed Copy \$3.00; Microfiche \$0.65

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SECTION 1

INTRODUCTION

GENERAL

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

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 can be selected which will assure maximum performance under LMFBR service conditions.

The importance of structure on mechanical properties has been well demonstrated in the published literature. For example, in one series of experiments^[1], prestraining 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. 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.

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."

SUMMARY OF CURRENT PROGRESS

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

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

Specimens of the WARD double vacuum melted 20% cold worked tubing were heat treated according to several schedules including one reported to give enhanced mechanical properties. These specimens have been tested and compared to the as-fabricated material.

A limited series of biaxial tensile tests were conducted in which hold time prior to test was varied to determine the effect this variable on the test data.

Comprehensive structural characterization of two WARD double vacuum melted tubing lots has been completed.

Additional low cycle high strain fatigue tests were delayed pending proof testing of a new specimen design.

An investigation of the effects of degree of cold work on weldability has also been completed. A summary is being prepared.

A shipment of 200 feet of 20% cold worked, double vacuum melted Type 316 tubing, manufactured by planetary swaging has been received for use in the characterization studies. Mechanical property testing was initiated.

A shipment of 250 feet of 20% cold worked Battelle-Northwest double vacuum melted Type 316 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

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.

CURRENT PROGRESS

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

SSBA-211 is a new task. A revised work program to cover that task was submitted in December 1969. Since SSBA-211 is scheduled for completion in February 1970, no separate section of current progress will be included in this report. A final report for that task will be published in March 1970.

The quarterly progress report, WARD-4135-2, for the period ending September 30, 1969, was prepared and distributed. Contract Modification No. 3 was received and executed to cover funding through June 30, 1970.

SECTION 3

SSBA-210 EVALUATION OF AUSTENITIC STAINLESS STEEL CLADDING

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

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.

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 (15-20%) 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 WARD vacuum melted tubing representing three melting practices, two grain sizes, and two conditions (20% cold worked and solution annealed) were received and accepted. Microcharacterization and mechanical property testing were initiated.

The weldability investigation continued. The first series of weldability studies on annealed tubing was completed. The second series on 15-20% cold worked tubing was initiated.

All ten lots of Battelle-Northwest Laboratories (BNW) double vacuum melted tubing for the Oxide Fuel Element Development Program (19-pin encapsulated assembly) were received and accepted. Double vacuum melted tubing for the 37-pin assembly was ordered.

An order was placed for double vacuum melted planetary swaged tubing to be used in the characterization studies.

CURRENT PROGRESS

Test Program

The detailed test program outlined in WARD-3791-26 and additional tasks defined in WARD-3791-29, WARD-3791-35, WARD-3791-41, and WARD-4135-2 continued.

Procurement

Planetary Swaged Vacuum Melted Tubing--Westinghouse - A shipment of 200 feet of 20% cold worked, double vacuum melted Type 316 tubing, WARD VM(IVCEV), manufactured by planetary swaging from heat #91695 has been received for use in the characterization studies. The certified physical properties are shown in Table 1.

Table 1 Certified Mechanical Properties, Westinghouse Vacuum Melted, Planetary Swaged, Type 316 Stainless Steel Tubing 0.230-Inch OD X 0.015-Inch Wall

Heat	Fabrication, Condition, And Lot Number	Superior Certified ASTM Grain Size	Total Length (feet)	Superior Certified Uniaxial R.T. Mechanical Properties		
				Yield (ksi)	Ultimate (ksi)	Elongation (%)
#91695 Planetary Swaged (IVCEV)	20% CW	6	188	119.9	134.0	9.0
				122.0	136.0	10.0
	Lot 49193					

Vacuum Melted Tubing for the Oxide Fuel Element Development Program - A shipment of 250 feet of 20% cold worked, Battelle-Northwest double vacuum melted Type 316 tubing, BNW VM(IVCEV), has been received for use in the Oxide Fuel Element Development Program (37-pin assembly).

Characterization

Hardness - Diamond pyramid hardness measurements have been completed for six additional lots of WARD VM tubing in the as-fabricated condition and for five post fabrication heat treated specimens. Table 2 includes these results plus the data from the six WARD VM lots measured previously.

Table 2. Diamond Pyramid Hardness^a
WARD Vacuum Melted Type 316 Stainless Steel Tubing

	VM(CEV) (Lot #) DPH	VM(IV) (Lot #) DPH	VM(IVCEV) (Lot #) DPH
AS FABRICATED			
20% CW Fine G.S.	(42394) 276	(42398) 278	(42402) 273
Solution Annealed Fine G.S.	(42395) 142	(42399) 145	(42403) 148
20% CW Coarse G.S.	(42396) 266	(42400) 269	(42404) 261
Solution Annealed Coarse G.S.	(42397) 150	(42401) 144	(42405) 132
HEAT TREATED			
VM (IVCEV) 20% CW Fine G.S. Lot 42402 Heat Treated			
		DPH	
900°F 24 hrs.		272	
1300°F 24 hrs.		235	
1300°F 216 hrs.		230	
b { 900°F 24 hrs.			
Plus 1300°F 216 hrs.		232	
VM (IVCEV) Solution Annealed Fine G.S. Lot 42403 Heat Treated			
		DPH	
1300°F 24 hrs.		158	

^a Average of Seven Measurements
500 Gram Load 20% CW
300 Gram Load Solution
Annealed

^b "Garofalo" Schedule

IV -Induction vacuum remelt
CEV -Consumable electrode vacuum
remelt
IVCEV -Induction vacuum plus consumable
electrode vacuum remelt

The fabrication variables include three melting practices, two grain size and two fabrication conditions; e.g., 20% cold worked and solution annealed. The data from the as-fabricated lots show (1) that the six cold worked lots have much higher hardness than the corresponding annealed lots, (2) that 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, and (4) there is no consistent difference in hardness attributable to type of vacuum melting practice.

The data from the heat treated lots show that (1) heat treatment of the 20% cold worked, fine grained double vacuum melted lot at 900°F resulted in no significant change in hardness, (2) heat treatment at 1300°F did cause a drop in hardness but there was no significant difference in hardness between the "Garofalo" treated material, 900°F for 24 hours plus 1300°F for 216 hours and the lot treated at 1300°F only, and (3) heat treatment of the solution annealed fine grained double vacuum melted lot at 1300°F for 24 hours resulted in a slight increase in hardness.

Grain Shape and Size - Longitudinal and transverse sections of the twelve WARD vacuum melted tubing lots were electrolytically polished with chromic-acetic acid and electroetched with chromic acid.

In the annealed tubing lots the grain shape was generally equiaxed in both the longitudinal and transverse sections. In the cold worked lots it was equiaxed in the transverse sections and slightly elongated in the longitudinal sections.

Check grain sizes were determined by comparison with ASTM charts. The results generally confirmed the supplier certified grain sizes reported in WARD-3791-41, except there was a slight laboratory bias favoring higher ASTM grain size numbers in the check evaluations and a little more internal variation.

The certified grain sizes reported previously will be accepted as the correct values.

Heat Treatment - Specimens of WARD VM(IVCEV) 20% cold work, fine grained tubing, lot #42402 were heat treated according to the four schedules listed below.

1. 900°F 24 hours
2. 1300°F 24 hours
3. 1300°F 216 hours
4. 900°F 24 hours plus 1300°F for 216 hours ("Garofalo" schedule)

The "Garofalo" schedule is reported^[1] to give marked enhancement of mechanical properties of pre-strained Type 316 stainless steel. The microstructure obtained from this treatment of tubing lot #42402 is shown in Figure 1 along with that of the as-cold worked material.

A specimen of WARD VM (IVCEV) solution annealed, fine grained tubing, lot #42403 was also heat treated 24 hours at 1300°F for use in biaxial tensile tests.

500X

A. BEFORE TREATMENT

500X

B. AFTER TREATMENT

Figure 1 WARD VM (IVCEV) Lot No. 42402, 20% Cold Worked, ASTM G.5.9, Before and After Heat Treatment at 900°F-24 Hours plus 1300°F-216 Hours. Transverse Sections.

3514-1

Plastic Replica, Electron Microscopy - Electron micrographs were made from plastic-carbon replicas of two heat treated air melted tubing specimens to survey for significant microstructural differences associated with carbide precipitation and dissolution. These specimens were also used to evaluate the applicability of cathodic vacuum etching to the characterization of Type 316 stainless steel.

The precipitation curves reported in WARD-3791-35 for those specimens show that carbides were precipitating from the matrix in the one specimen (Lot #7-2489, 1.5 hours at 1225°F) and were redissolving into the matrix in the other (Lot #7-2489, 1.5 hours at 1650°F).

Replicas were examined extensively with an electron microscope. A few pores were found in each specimen, but no generally porous areas were observed. Pores larger than 300Å would have readily been observed, if present, using this type of replica and etching procedure.

Both specimens contained a few large angular inclusions, probably Ti(C, N), two to five microns in size, and very few grain boundary precipitates. The specimen treated at 1650°F contained a relatively high frequency ($\sim 10^7$ particles/cm²) of small intragranular precipitates about 400Å (0.04 microns) to 4000Å (0.4 microns) in size. The 1225°F sample contained similar intragranular precipitates, but they were generally smaller in size and about half the frequency. There were no gross differences in precipitate morphology as observed by this approach.

Cathodic vacuum etching appears to be a good general etch for Type 316 stainless steel. The grain structure and the twin structure are well delineated by both height and surface texture differences related to orientation. Carbides and carbo-nitrides etch at slower rates than the matrix.

Comprehensive Characterization WARD Vacuum Melted Tubing Lots 42402 and 42403^a

Specimens from two lots of WARD double vacuum melted Type 316 stainless steel have been characterized in detail by light microscopy, extraction replica electron microscopy, transmission electron microscopy, X-ray and electron diffraction analysis of extracted precipitates, X-ray line broadening, and a limited number of X-ray pole figure studies. In addition, the inclusions in tubing lot #42402 and the source bar stock have been analyzed in situ by electron probe microanalysis^b. The specimens characterized were the following:

1. Lot 42403, Heat 91695 WARD VM(IVCEV)
Item 10, Tube Section #10-02-00
Solution Annealed; Grain Size: 8
2. Lot 42402, Heat 91695 WARD VM(IVCEV)
Item 9, Tube Section #09-02-32
20% Cold Worked; Grain Size: 9

^aB. Weiss, R. Stickler, Westinghouse Research Labs

^bElectron Probe Investigation, WARD

Experimental Techniques - Details of the techniques for light microscopy, electron microscopy, and X-ray diffraction investigations have been reported previously.^[2,3] A special bulk extraction method was developed for the quantitative and qualitative determination of inclusions and carbide precipitates. For this, a weighed amount of the tube sample was anodically dissolved in a 10% HCl-methanol solution. The electrolysis was carried out at approximately 3V and a current density of 150-250 mA/cm². After dissolution of most of the tube sample, the electrolyte was filtered. The filter residue and the remaining portion of the sample were weighed, and the amount of residue per dissolved tube portion calculated. The "bulk extracted" residue was then examined by electron and X-ray diffraction techniques.

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 (110) 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, as described in a previous report^[3], to a wall thickness of approximately 0.005 inches. 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 on a specimen holder to form a composite sheet sample approximately 1" x 1".

General Microstructure - Light micrographs of the electropolished outer surfaces of the cold worked and annealed tube samples are shown in Figure 2. Small particles arranged in rows and stringers can be seen. The total density of particles was similar in both tube samples, approximately 1×10^6 particles/cm². The microstructure of the two tube samples is shown in Figure 3. The grain size revealed by etching was measured as ASTM No. 9 for the cold worked sample and ASTM No. 8 for the annealed sample, agreeing with the information submitted by WARD. The grains in the annealed sample were equiaxed and contained numerous annealing twins, while the cold work sample consisted of slightly elongated grains containing twins and a needle-like substructure.

Transmission electron micrographs of the annealed sample, Figure 4a, revealed a dislocation density of $(6+3) \times 10^8$ dislocations/cm², with a large fraction of the dislocations arranged in rows. The relatively high dislocation density indicated that the tubes have been slightly deformed after the annealing treatment; e.g., during a final straightening operation. In some grains, rows of small ($\sim 1000\text{\AA}$ diam.) equiaxed particles were visible.

No widely extended dislocation nodes could be observed, which indicates that the stacking fault energy is at least in the order of 25-30 erg/cm².^[4] As shown in the transmission electron micrograph in Figure 5, the width of the dislocation nodes is close to the limit of resolution and no exact measurement of the stacking fault energy is possible under such conditions.

A transmission electron micrograph of the cold worked tube sample is shown in Figure 4b. The high dislocation density made counting impossible but it may be assumed^[5] to be of the order of $10^{11} - 10^{12}$ dislocations/cm².

A. LOT NO. 42403, ANNEALED

B. LOT NO. 42402, 20% COLD WORKED

Figure 2 Light Micrographs of Representative Areas of Electropolished Lots No. 42403 and 42402

A. LOT NO. 42403, ANNEALED

10 μ m

B. LOT NO. 42402, 20% COLD WORKED

10 μ m

Figure 3 Light Micrographs of Electropolished and Etched Outer Surface of Tube Lots No. 42403 and 42402

1 μm

A. LOT NO. 42403, ANNEALED

1 μm

B. LOT NO. 42402, 20% COLD WORKED

Figure 4 Transmission Electron Micrographs of Thinned Portions of Tube Lot No. 42403 and 42402

3514-4

PLANE OF FOIL (110), $g = \langle 111 \rangle$

0.1 μm

Figure 5 Transmission Electron Micrograph of a Thinned Portion of Tube Lot No. 42403; Showing Dislocation Modes

3514-5

The needle-shaped features observed under the light microscope appeared in the transmission electron micrographs as thin bands of unresolvable fine substructure. In comparison with observations made on deformed bulk Type 316 stainless steel samples^a, it can be inferred that these bands correspond neither to twins nor to the hexagonal epsilon phase, which has been reported^[6,7,8] to occur in cold worked stainless steel as a transition phase between austenite and martensite. Only minor orientation differences between the bands and the surrounding matrix could be found by electron diffraction of thin foils. Those observations indicate that the bands correspond to deformation bands as reported by several investigators.^[5,9,10] The deformation bands in the tubing are probably regions of high dislocation density resulting from dense slip on closely spaced planes. Further investigations would be required to determine the exact nature of these bands and their role during aging treatments.

Identification of Second Phase Particles - Electron micrographs of carbon-extraction replicas, Figure 6, reveal that the extracted particles are polyhedral with an average diameter of less than 0.25 μ m. No differences could be detected between the particles extracted from the annealed and cold worked tube samples. Occasionally, impressions on the replicas indicated that some larger particles present in the sample have not been extracted. The fine extracted particles were identified by electron diffraction as MC-type carbides, Table 3.

The weights of the bulk extracted residues were 0.5% of the specimen weights for both tubing lots 42402 and 42403. X-ray diffraction analysis showed the presence of α -quartz in each material (Table 4). The MC carbides present in the residues were not observed by X-ray diffraction analysis because of the small particle size.

Electron Probe Microanalysis of Inclusions^b - Two vacuum melted Type 316 stainless steel source bar stock specimens heat #10695 VM(IVCEV) and heat #30160 VM(CEV) and three specimens from tubing lot #42402 VM(IVCEV) fabricated from heat #91695 were examined by electron probe microanalysis to determine the elemental 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 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 probe analyses were conducted on the in situ inclusions of the type resolvable with a light microscope. In general, these inclusions were high in Mn and S and low in Fe, Cr, Ni, and Mo relative to the matrix. Several of the most prevalent type of inclusion in each specimen (e.g., gray stringers), were analyzed in detail for the elements Na and above in the periodic table. They showed no 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, S rich inclusions. A few inclusions were also found in the single melted bar stock material,

^a B. Weiss and R. Stickler, Unpublished Results

^b WARD

LONGITUDINAL
DIRECTION

A.

B.

Figure 6 Electron Micrographs of Carbon-Extraction Replicas of the Outer Surface of Tube Lot No. 42402

3514-6

Table 3. Evaluation of Electron Diffraction Patterns of Extraction Replicas of Tube Lot 42403 and Tube Lot 42402

Electron Diffraction of Extraction Replica	
$d_{\text{observed}}(\text{\AA})$	$d_{\text{TiC}}(\text{\AA})$ ASTM Card No. 6-0614
2.51	2.53
2.17	2.179
1.76	----
1.51	1.53
1.31	1.32
1.22	1.25

Table 4. Evaluation of X-ray Diffraction Pattern From Bulk Extracted Residue of Tube Lot 42403 and 42402

X-ray Diffraction of Residue	
$d_{\text{observed}}(\text{\AA})$	$d_{\alpha\text{-Quartz}}(\text{\AA})$ ASTM Card No. 5-0490
4.25	4.26
3.35	3.43
2.45	2.458
2.29	2.282
2.22	2.237
2.115	2.128
1.97	1.980
	1.817
	1.801
1.67	1.672
	1.659
	1.608
1.54	1.541
	1.453

heat #30160 VM(CEV), which were high in Si and low in Fe, Cr, Ni, Mo, Mn, and S relative to the matrix. Count rate data from the various characteristic X-ray lines relative to the matrix were the basis of those observations.

The inclusions were also investigated using the electron probe microanalyzer in the scanning mode as well as in the fixed probe mode. Figure 7a is a light micrograph at 1000X, and 7b is a back scattered electron scan at approximately the same magnification; of four of the gray stringer type inclusions found in the double melted bar-stock material, heat #91695. Figures 7c and 7d are Mn $K\alpha$ and S $K\alpha$ X-ray scans of the same four inclusions demonstrating the high concentration of Mn and S in these inclusions relative to the matrix.

The light micrograph and back scattered electron scan (made with electrons backscattered from a layer very near the surface of the specimen), shown in Figure 7, indicate that the two lower particles are not connected at the surface. However, the X-rays which originated from a volume extending at least one micron deep in the specimen show the presence of high Mn and high S between the two particles. The combined observations indicate that these two inclusions are connected at a level just below the surface.

The inclusions in the bar stock material were generally one to three microns wide which was sufficient for observation and location in the electron probe in the as-polished condition. The inclusions in the fabricated tubing were much narrower --less than one micron wide-- making electron probe analysis of in situ particles more difficult. A lightly cathodic etched surface preparation of the tubing specimen proved to be the best for electron probe analysis, but an electrolytically polished and etched surface was best for microstructural identification and evaluation.

Figure 8a is a back scattered electron scan of the electrolytically prepared surface showing both continuous and discontinuous type stringers in tubing lot #42402. Twelve of the continuous type inclusion in a similar specimen, except lightly cathodic etched, were conclusively analyzed as high in Mn and S and low in Fe, Cr, Ni, and Mo relative to the matrix. Several of these inclusions were analyzed further 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.

Figure 8b is a back scattered electron scan and Figure 8c is a S $K\alpha$ X-ray scan of the same area, demonstrating the high S concentration in part of the one discontinuous inclusion and very little S in the other, presumably due to etching. The X-ray scan also shows high S concentration just below the surface in part of the one inclusion.

None of the inclusions analyzed in the tubing specimen, WARD VM(IVCEV) lot 42402, showed Si concentrations significantly higher than the matrix but this does not rule out the additional presence of small SiO_2 particles observed for the same tubing lot as reported in the previous section.

The electron probe results from in situ inclusions are listed in Table 5 along with the diffraction results from extracted particles.

A. LIGHT MICROGRAPH

1000X

B. BACK SCATTERED ELECTRON
SCAN (BSE)

C. Mn $K\alpha$ X-RAY SCAN

D. S $K\alpha$ X-RAY SCAN

Figure 7 Type 316 Stainless Steel VM (IVCEV) Bar Stock Heat #91695, As-Polished
Longitudinal Section

3514-7

950X

A. BACK SCATTERED ELECTRON SCAN (BSE)

900X

B. BACK SCATTERED ELECTRON SCAN (BSE)

900X

C. S K α X-RAY SCAN

- A. BSE SCAN ELECTROLYTICALLY PREPARED SURFACE
- B. BSE SCAN LIGHT CATHODIC VACUUM ETCH
- C. S K α X-RAY SCAN SAME AREA AS B

Figure 8 Type 316 Stainless Steel VM (IVCEV) Tubing Lot No. 42402.
Longitudinal Section

3514-8

Table 5. Summary of Characterization Results

	Tube Lot 42403 Annealed	Tube Lot 42402 20% Cold Worked
1. Microstructure		
Grain Size ASTM No.	8	9
Grain Morphology	Equiaxed and twins	Slightly elongated, twins and needle-like features
Dislocation Arrangement	Majority arranges in rows	Heavy dislocated microstructure with deformation bands
Dislocation (dislocations/cm ²)	$(6 \pm 3) \times 10^8$	$\sim 10^{11} - 10^{12}$
Stacking Fault Energy (erg/cm ²)	~ 25	Not measurable from TEM's
2. Second Phase Particles and Inclusions		
Arrangement	Aligned into continuous and discontinuous stringers parallel to tube axis	Same
Amount	$1 \pm 0.5 \times 10^6$ particles/cm ²	Same
Identification		
by ED (Extraction replica)	MC	Same
by XD (Bulk extraction)	$\alpha\text{-SiO}_2$ (<0.5 wt%)	Same
by EPA (In situ inclusions)		Continuous and some discontinuous inclusions of size observable with light microscope, high in Mn and high in S.
3. Residual Stress		
Outer Surface	Cold worked	Cold worked
Inside Surface	Strain free	Cold worked
After removal of 0.0005" from OD	Strain free	Cold worked
4. Texture ^a	Very weak {112} < 113>	Weak {112} < 120 >
^a {plane tangential to OD} <direction parallel to tube axis>		

Residual Stress and Texture Analysis - Laue back-reflection X-ray diffraction patterns of the tube samples are shown in Figure 9. The outside surface of the annealed tube sample yielded a smooth ring pattern, Figure 9a, indicating a cold worked surface layer. After removal of approximately 0.0005 inches by electropolishing a spotty ring pattern was obtained, characteristic for strain-free material, Figure 9c. A similar spotty pattern was obtained from the inside tube surface, 9b. The X-ray diffraction pattern from both the outside and inside surfaces of the cold worked tube sample consisted of smooth rings with an indication of weak texture, Figure 9d. Removal of surface layers did not alter this diffraction pattern.

The (110) pole figures representing the tube textures are shown in Figure 10. The pole figure of the cold worked tube sample, Figure 10a, reveals a weak texture with $\langle 112 \rangle$ parallel to the radial direction and $\langle 120 \rangle$ almost parallel to the axial direction. The annealing treatment caused a texture which could be described by an approximate 20° rotation of the cold worked texture around the $\langle 112 \rangle$ pole, resulting in a texture with $\langle 112 \rangle$ parallel to the radial direction and $\langle 113 \rangle$ parallel to the axial direction. The texture of the annealed tube is even less pronounced than the weak texture of the cold worked tube, Figure 10b.

Conclusions - The results of this investigation are summarized in Table 5, from which the differences and similarities of the tube samples examined can be deduced.

1. The concentration of second phase particles (MC and non-metallic inclusions, $\alpha\text{-SiO}_2$) measured by bulk extraction is practically the same in both tube samples.
2. From the electron probe analysis of in situ inclusion, it is concluded that for lot #42402 the continuous stringers are high in Mn and S, low in Si and are probably MnS, and that some of the discontinuous stringers are also high in Mn and S.
3. As expected, the dislocation density in the 20% cold worked sample is too high to be determined quantitatively. The dislocation density in the annealed sample is higher than expected for well annealed material and must be a result of cold work after the final annealing treatment.
4. The outer surface of both tube samples is heavily deformed by a grinding or lapping process. The depth of this surface damage is less than 0.0005 inches in the annealed sample. The extent of such a damaged surface layer cannot be estimated for the cold worked tube sample since it cannot be distinguished from the general microstructure.
5. The stacking fault energy in both samples is too high to be determined precisely by transmission electron microscopy methods. Since no extended nodes nor wide stacking fault ribbons could be observed, a stacking fault energy of at least $25\text{-}30 \text{ erg/cm}^2$ is estimated.

A.

B.

C.

D.

- A. LOT NO. 42403, OUTSIDE SURFACE
- B. LOT NO. 42403, INSIDE SURFACE
- C. LOT NO. 42403, OUTSIDE SURFACE AFTER REMOVAL OF 9.5 MIL
- D. LOT NO. 42402, TYPICAL PATTERN FOR INSIDE AND OUTSIDE SURFACE
BEFORE AND AFTER ELECTROPOLISHING

Figure 9 Laue X-Ray Back-Reflection Pattern of Inside and Outside Surfaces of Tube Lot No. 42403 and 42402 Before and After Electropolishing

3514-9

3514-10

24

Figure 10a (110) Pole Figure of Cold Worked, Type 316 Stainless Steel Tubing: Contours are Multiples of Random Intensity. Specimen 42402 Chart 668.

3514-11

25

Figure 10b (110) Pole Figure of Annealed, Type 316 Stainless Steel Tubing: Contours are Multiples of Random Intensity.
Specimen 42403 Chart 667

6. Evaluation of the pole figures indicates a weak texture for the cold work tube sample $\{112\} \langle 120 \rangle$ and a still less pronounced $\{112\} \langle 113 \rangle$ texture in the annealed sample.

Precipitation Studies on Various Type 316 Air Melted Stainless Steel Tubing Lots^a

The precipitation studies initiated during FY-1969 on various lots of air melted Type 316 stainless steel tubing have been completed. The results from the long term tests, up to 5,000 hours at 750-1650°F are reported. Selected results from the short term tests, up to 150 hours, reported in WARD-3791-35 are included for comparison. The tubing lots investigated were the following:

1. Lot 7-2489, Heat 20303 WARD AM Solution Annealed, Dry H₂
2. Lot 316727, Heat 20303, WARD AM Solution Annealed, Dry H₂
3. Lot 316870(H), Heat 20276, BNW AM Solution Annealed, Dry H₂
4. Lot 321921, Heat 20303, WARD AM 15-20% Cold Worked

Tube Lot No. 7-2489 - Representative light micrographs of the heat-treated, polished, and etched specimens are shown in Figure 11. The specimens heat-treated for 5,000 hours at the lower aging temperature (900°F) exhibit an increase in the amount of grain boundary precipitate as well as of intragranular precipitate compared to the 1,500 hour treatment at the same temperature. Long-time aging treatments at the higher temperature (1500°F) did not significantly increase the amount of the intragranular precipitate.

Changes in precipitate morphology with aging time and temperature can be seen more clearly in electron micrographs of extraction replicas of these samples. Long-time annealing, especially at the higher temperatures, caused a coarsening of the grain boundary precipitate, identified as $M_{23}C_6$, Figure 12. The featherlike dendritic grain boundary particles formed during short-time aging treatments^[3] changed to large bulky particles, while the amount of the intragranular MC precipitate was not significantly altered by temperature and time of the aging heat treatment. The time-temperature precipitation (TTP) diagram for aging times up to 5,000 hours is shown in Figure 13. The diagrammatic representation of the relative amounts of the various precipitated phases, Figure 14, indicates that below 1350°F the amount of the $M_{23}C_6$ carbide was still increasing with aging time. The MC particles were almost completely dissolved after aging for 1,500 hours at 1650°F. After aging treatments above 1650°F and 150 hr. no $M_{23}C_6$ and MC particles could be found in the precipitate-free matrix. Transmission electron micrographs of the sample aged at 1500°F for 150 hours show MC particles and $M_{23}C_6$ sheets present on the grain boundaries, Figure 15a. After aging at 1500°F for 5000 hours only bulky $M_{23}C_6$ can be found on the grain boundaries and the MC particles were fewer in number (Figure 15b).

X-ray and electron diffraction analyses of the particles revealed that at temperatures below 1500°F even after aging for 5,000 hours only two carbide

a B. Weiss and R. Stickler, Westinghouse Research Laboratories

900°F/1500 HOURS 800X

1500°F/1500 HOURS 800X

900°F/5000 HOURS 800X

1500°F/5000 HOURS 800X

Figure 11 Light Micrographs of Representative Areas of Tube Lot No. 7-2489 After Long-Time Aging Treatments

3514-12

1200°F/5000 HOURS 1 μm

1500°F/5000 HOURS 1 μm

1200°F/1500 HOURS 1 μm

1500°F/5000 HOURS 1 μm

Figure 12 Electron Micrographs of C-Extraction Replicas of Tube Lot No. 7-2489 After Different Aging Treatments

3514-13

Figure 13 Time-Temperature-Precipitation Diagram for Tube Lot No 7-2489

Figure 14 Estimated Relative Amounts of MC and $M_{23}C_6$ Carbides Precipitated in Various Heat Treated Specimens of Tube Lot No. 7-2489.

3514-15

A. 1500°F/150 HOURS

1 μm

B. 1500°F/5000 HOURS

1 μm

Figure 15 Transmission Electron Micrographs of Specimens of Tube Lot No. 7-2489 Aged at 1500°F for 150 and 5000 Hours

3514-16

phases are present; i.e., MC and $M_{23}C_6$, and that no intermetallic phases as reported for similar steels (11) are formed in this tube lot.

Tube Lot No. 316727 - Due to the small amount of available sample material, only a few specimens could be heat treated in the time-temperature region of interest. Light micrographs of samples aged between 1200 and 1650°F illustrate the pronounced difference in precipitation behavior of this tube lot compared with tube lot 7-2489. A dense fine intragranular precipitate is present in specimen aged below 1350°F, Fig. 16a and b. Heat treatments at 1650°F resulted in the formation of bulky particles at grain boundaries and intragranularly. Electron micrographs of extraction replicas of these samples are shown in Figure 17. The fine intragranular needle-shaped precipitate coarsens with increasing aging temperatures, (Figure 17a and b), and only bulky particles can be seen after the 1650°F treatment, (Figure 17c).

The precipitates were identified by X-ray and electron diffraction techniques. The results are summarized in Table 6 and in the TTP diagram, Figure 18. In addition to $M_{23}C_6$, a chi-phase (X), a hexagonal Laves phase (Fe_2Mo) and a hexagonal M_6C carbide could be detected. The large amount of precipitates, the small size and narrow spacing of the individual particles made it impossible to correlate particle morphology with type of precipitate.

A transmission electron micrograph of the sample aged at 1200°F for 1,500 hours shows the plate-like particles to be oriented along several crystallographic directions, Figure 19.

Tube Lot No. 316870 (H) - Due to the short supply in sample material only one specimen could be heat treated for 1,500 hours at 1200°F. As previously reported^[3] the dislocation density in the as-received tube was slightly higher in this tube lot, resulting in an increased precipitation rate. However, no significant differences in the amount or types of the precipitated phases between this specimen and samples of tube lot 316737 could be detected after long-time aging.

Discussion - The long-time aging experiments confirmed the results obtained in the previous investigation.^[3] The tube lots 316727 and 316870 (H) reacted similarly during aging treatments, while tube lot 7-2489 responded entirely differently to the same aging treatments. The precipitation reactions in the first two tube lots are in good agreement with published results.^[11] The somewhat accelerated precipitation rate of $M_{23}C_6$ in present tube samples is explainable by the small amount of cold work introduced into the tubes prior to aging. After long-time aging, in addition to $M_{23}C_6$, a chi-phase (X), a hexagonal Laves phase (Fe_2Mo), and small amounts of a hexagonal M_6C carbide were found. The formation of Laves and chi-phases during long-time aging treatments is described in the literature,^[11] however, the presence of the M_6C carbide has never been reported. As pointed out by Goldschmidt,^[12] this M_6C carbide may constitute a transition phase between $M_{23}C_6$ and the Laves phase. No intermetallic sigma-phase could be detected in samples heat treated in the investigated time-temperature range. This too is in agreement with published information.^[11] Preliminary aging experiments on bulk 316 stainless steel samples^a revealed that sigma-phase is formed only after aging conditions exceeding 1,500 hours and 1350°F.

^a B. Weiss and R. Stickler, Unpublished Results

A. 1200°F/1500 HOURS 800X

B. 1350°F/1500 HOURS 800X

C. 1650°F/150 HOURS 800X

Figure 16 Light Micrographs of Representative Areas of Tube Lot No. 316727 after Various Aging Treatments 800X

3514-17

A. 1200°F/1500 HOURS

1 μ m

B. 1350°F/1500 HOURS

1 μ m

C. 1650°F/150 HOURS

1 μ m

Figure 17 Electron Micrographs of Carbon-Extraction Replicas of Tube Lot No. 316727 After Various Aging Treatments

3514-18

Table 6 d-values (in Å) Determined by X-ray Diffraction Patterns
of Extraction Replicas of Heat Treated Specimens

Tube Lot No. 316727

(1 Rad. - Debye-Scherrer Camera, Cr K_{α} Radiation)

1200°F/1500 hr	1350°F/1500 hr	1650°F/1500 hr	ASTM Card No. 14-407 for M ₂₃ C ₆	ASTM Card No. 6-0676 for Chi	ASTM Card No. 6-0622 for Fe ₂ Mo	ASTM Card No. 11-546 for M ₆ C
2.38	2.38	2.38	2.37	2.373		2.40
	2.36				2.36	2.31
2.22	2.23	2.22		2.219		2.22
2.18	2.18	2.18	2.17		2.18	2.16
2.09	2.09	2.09		2.09		2.09
2.05	2.05	2.05	2.05		2.05	2.05
	2.03				2.02	
1.98	1.98	1.98		1.98	1.98	1.99
1.93	1.93					1.95
1.88	1.88	1.89	1.88	1.89		1.92
1.80	1.80	1.18	1.80	1.81		1.91
1.775	1.775	1.78	1.78			1.86
	1.685	1.74		1.74		1.81
1.625	1.625		1.63			
1.605	1.605	1.61	1.61			1.52
	1.45	1.44	1.49	1.44		1.43
	1.37				1.37	1.413
1.33	1.33	1.335	1.33		1.33	1.34
1.29	1.292	1.295	1.29	1.28	1.29	1.32
1.256	1.256	1.256	1.256	1.256		1.293
1.236	1.236	1.232	1.231		1.23	1.28
	1.232			1.208		1.27
1.186	1.186	1.209	1.19		1.18	1.25
1.172	1.172	1.171	1.17			1.23

Figure 18 Time-Temperature-Precipitation Diagram for Tube Lot No. 316727

1 μm

Figure 19 Transmission Electron Micrograph of Tube Lot No. 316727 Aged at 1200°F for 1500 Hours

3514-20

The abnormal precipitation behavior of tube lot 7-2489 appears to be due to a low solution-treatment temperature in addition to a small amount of cold work introduced after the solution treatment prior to aging. It may be speculated that the low solution-treatment temperature resulted in the precipitation of the (Ti, Mo)C carbide, while the high dislocation density accelerated the formation of $(\text{Cr, Mo})_{23}\text{C}_6$ carbide. Thus, the austenitic matrix became depleted in sigma- or chi-forming elements and consequently the precipitation of these intermetallic phases was retarded. To prove this assumption, a remaining portion of tube lot 7-2489 was re-solution treated at 2000°F for 1 hour, water quenched, and subsequently aged at 1350°F for 150 hours. The resulting microstructure is compared in Figures 20 and 21 with the microstructure of the as-received and similarly aged sample. The 2000°F solution treatment resulted in an almost complete dissolution of the MC particles, and the aging treatment caused M_{23}C_6 precipitation on grain boundaries, twin boundaries, and intragranularly.

The effects of the re-solution treatment on the aging response can be seen more clearly in transmission electron micrographs, Figure 21. In the as-received and aged sample, equiaxed MC particles are randomly distributed while M_{23}C_6 particles are precipitated at only grain boundaries (Figure 21a). In the re-solution treated and aged sample, M_{23}C_6 carbides are precipitated on grain boundaries as well as on dislocations around massive MC particles (Figure 21b). In addition, M_{23}C_6 plates could be found on incoherent twin boundaries (Figure 21c), growing from the incoherent twin boundaries into the surrounding matrix similarly as reported for 304 type stainless steels.[13,14] In general, the microstructure of the re-solution treated and aged tube lot 7-2489 appears very similar to the "normal" microstructure of the comparably aged tube lot 316727. The results of these experiments illustrate the pronounced effect of solution treatment conditions on subsequent aging responses.

To obtain information about the effects of cold work on the precipitation kinetics, several samples of the cold-worked tube lot 321921 were heat treated at 1350°F for 9 minutes and 150 hours (the microstructure of this tube lot in the as-received condition has been described in a previous report).[3] The 9 minute aging treatment resulted in a pronounced precipitation of M_{23}C_6 on grain boundaries and dislocations in the cold worked sample, whereas aging treatments for more than 1 hour at the same temperature were required to form precipitates in the recrystallized material (e.g., compare with TTP-Diagram, Figure 18). The microstructure of the cold-worked and 150 hours aged sample can be seen in the electron micrographs in Figure 22. Massive stringers of grain boundary M_{23}C_6 , intragranular plates and equiaxed particles (identified by X-ray and electron diffraction as M_{23}C_6 , Laves, and chi-phases) are visible, Figure 22. The transmission electron micrograph of this sample shows a dense dislocation substructure containing numerous plate-like precipitates and a large number of deformation bands. The morphology of these bands has been previously described.[15] The present interpretation is that these deformation bands are probably the result of dense slip on closely spaced planes.

We may conclude from these experiments that the differences in aging behavior observed between the various tube lots are at least partly related to differences in the solution treatment procedure and in the amount of cold work

1 μm

A. GRAIN BOUNDARY $M_{23}C_6$ AND INTRAGRANULAR MC

1 μm

B. $M_{23}C_6$ GRAIN BOUNDARIES AND INTRAGRANULAR

A. AS-RECEIVED AND AGED

B. RE-SOLUTION TREATED (2000°F/1 HOUR, WATER QUENCHED) AND AGED
PARTICLES IDENTIFIED BY X-RAY AND ELECTRON DIFFRACTION ANALYSIS

Figure 20 Electron Micrographs of Carbon-Extraction Replicas of
Tube Lot No. 7-2489 Aged for 150 Hours at 1350°F in the
As-Received and Solution-Treated Condition

3514-21

1 μm
A. INTRAGRANULAR MC, GRAIN BOUNDARY $M_{23}C_6$

1 μm
B. FINE INTRAGRANULAR $M_{23}C_6$ AROUND BULKY MC

1 μm
C. $M_{23}C_6$ PLATES ON INCOHERENT TWIN BOUNDARIES

A. AS-RECEIVED AND AGED
B&C. RE-SOLUTION TREATED (2000°F/1 HOUR, WATER QUENCHED) AND AGED

Figure 21 Transmission Electron Micrographs of Tube Lot No. 7-2489 Aged for 150 Hours at 1350°F

3514-22

A. C-EXTRACTION REPLICA

1 μm

1 μm

B. TRANSMISSION ELECTRON MICROGRAPH

Figure 22 Electron Micrographs of Tube Lot No. 321921 (20% CW) Aged at 1350°F for 150 Hours. Particles were Identified by X-Ray and Electron Diffraction Analysis.

$M_{23}C_6$ (Strong), Fe_2Mo (Weak), and Chi(Weak)

3514-23

introduced after the solution treatment. However, the effects of variations in the concentrations of trace elements, such as Ti, Nb, and B, must not be neglected. For example, pronounced differences in mechanical properties of 304 stainless steels have recently been attributed to minor differences in the Nb content.^[16] The varying tendency to form intermetallic phases appears to be related to the rate of the precipitation of MC and $M_{23}C_6$ particles which deplete the matrix in elements favoring the formation of such intermetallic phases.

Summary - The results of the aging studies on three 316 stainless steel tube lots were as follows:

1. All three tube lots in the as-received condition had a fine equiaxed recrystallized grain structure. Rows of dislocations indicated a small amount of cold work (~5%) introduced after the annealing treatment, probably as a result of minor straightening operations.
2. Aging treatments between 750 and 1650°F up to 5,000 hours resulted in two of the tube lots (316727 and 316870) in precipitation reactions similar to those described in the literature. The precipitates consisted of $M_{23}C_6$ and M_6C carbides, intermetallic chi and Laves phases.
3. Tube lot 7-2489 exhibited a completely different aging response, aging treatments between 750 and 1650°F up to 5,000 hours resulted only in precipitation of MC and $M_{23}C_6$ carbides.
4. These differences in the precipitation behavior appear to be related to the solution treatment temperature and the degree of cold work introduced prior to the aging treatment. Low solution-treatment temperatures cause precipitation of MC carbides, while cold work prior to aging increases the precipitation rate for $M_{23}C_6$ carbides. Both these precipitates seem to deplete the matrix of elements which favor the formation of intermetallic phases such as sigma or chi. However, the effects of minor trace elements on the stability of the austenitic matrix and its tendency to precipitate intermetallic phases must not be neglected.
5. In view of possible adverse effects of the various precipitates on mechanical, physical, and chemical properties of the cladding, it is recommended that detailed information on trace element composition and thermomechanical processing history of the tubes are carefully recorded and evaluated.

Cladding Weldability

Present plans for this program are limited to producing and characterizing the following end closure welds:

<u>Quantity</u>	<u>End Plug</u>	<u>Cladding Grade</u>	<u>Plug-tube fit</u>
100	Annealed 316 SS	Annealed 316 SS WARD(AM) Heat 20303 Lot 316726	Interference fit of $0.0015'' \pm 0.0003''$
30	"	"	Close fit of $0.0000'' \pm 0.0003''$
100	"	15-20% Cold Worked 316 SS WARD(AM) Heat 20303 Lot 321921	Interference fit of $0.0015'' \pm 0.0003''$
30	"	"	Close fit of $0.0000 \pm 0.0003''$

The degree of fit is considered an important variable as it may have a greater effect on defect frequency rate than the amount of cold work in the cladding.

All of the above samples have been made and characterized. The following are defect frequency rates for the different series of weld tests, as established by radiographic examination.

Series I (110 welds), annealed cladding, 0.0015 inches plug-tube interference fit. Except for one weld with a visible blow hole, all the welds conformed to the inspection specification requirements for GETR fuel pin end closure welds. Some of the samples exhibited tube expansion adjacent to the weld, but no unacceptable wall thinning was associated with this expansion. Metallographic examination of ten samples has not revealed any wall thinning in excess of 7% of the wall thickness. Percentage defects 0.9%.

Series II (30 welds) annealed cladding, plug-tube fit 0.000 ± 0.0003 inch. Three joints exhibited tube wall thinning adjacent to the weld. Percentage defects 10%.

Series III (92 welds) cold worked cladding, 0.0015 inch plug-tube interference fit. One weld exhibited blowhole. One weld exhibited a tungsten inclusion. Four welds exhibited excessive tube wall thinning adjacent to the weld. Percentage defects 7.8%.

Series IV (30 welds) cold work cladding, plug-tube fit 0.000 ± 0.0003 inch. No defects were observed.

Quantitative metallographic examination of end closure welds, showing defect indications during radiography, has been completed. This examination confirmed and quantified the indications interpreted as defects during radiographic examination. No further tests are currently planned under this subtask and a summary is being prepared.

Mechanical Property Testing

Equipment - Installation of an additional furnace for biaxial stress rupture testing has been completed. Trial runs in this facility are in progress.

Biaxial Tensile Tests - Biaxial tensile testing of planetary swaged WARD VM (IVCEV) tubing Lot #49193 (20% cold worked) and of heat treated specimens from WARD VM (IVCEV) Lot #42402 (20% cold work--grain size 9) has been completed. The heat treatments of the cold worked lot (42402) were as follows:

- (i) 24 hours at 900°F
- (ii) 24 hours at 1300°F
- (iii) 216 hours at 1300°F
- (iv) 24 hours at 900°F + 216 hours @ 1300°F--("Garofalo" treatment).

Tensile, offset yield, and elongation results for these materials are given in Table 7A and B and shown in Figures 23, 24, and 25 along with relevant results reported previously.

The data show that heat treated lots in general have properties intermediate between the as-cold worked material and the solution annealed material and that the planetary swaged tubing has similar properties to the as-cold worked drawn tubing.

The single 900°F treatment has the least effect on properties, while the double treatment, "Garofalo," together with the 216 hour treatment had the greatest. The effect of all the post fabrication heat treatments on the fine grained 20% 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-cold worked material.

The 24 hour at 900°F heat treatment on fine grained cold worked material reduced both the ultimate strength and yield strength significantly at temperatures up to 1100°F. At 1200°F and 1300°F test temperatures the heat treated tubing had approximately the same ultimate and yield strengths as the non-heat treated tubing.

The "Garofalo" treatment, 900°F for 24 hours plus 1300°F for 216 hours of the fine grained cold worked tubing and the 1300°F for 216 hour treatment of the same lot resulted in marked reductions in yield and ultimate strengths and increases in elongations relative to the as cold worked material. The effects were more pronounced the lower the 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°F and 1300°F. The greatest difference in properties was in the elongations at the test temperature below 1100°F.

Figures 23, 24, and 25 show the comparison between the 20% cold worked, drawn tubing (lot 42402) and the planetary swaged tubing (lot 49193) fabricated from the same double vacuum melted heat to a nominal 20% cold work.

Table 7A. Biaxial Tensile Results From Heat #91695
WARD VM Type 316 Stainless Steel Tubing

Lot	Melting Practice	Pretreatment Condition	Heat Treatment	Before Test RT Hardness DPH	Mechanical Properties						
					Property	RT	850°F	1000°F	1100°F	1200°F	1300°F
42402 Item 9	IVCEV	20% CW G.S.-9	24 Hrs @ 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	IVCEV	20% CW G.S.-9	216 Hrs @ 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	IVCEV	20% CW G.S.-9	24 Hrs @ 900°F +216 Hrs @ 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
49193	IVCEV	Nominal 20% CW Planetary Swaged	As Fabricated	---	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
IVCEV - Induction Vacuum Plus Consumable Electrode Vacuum						<u>Mechanical Properties</u>					
* Had difficulty with Room Temperature Test						a. Ultimate Tensile Strength, ksi					
						b. 0.2% Yield Strength, ksi					
						c. Uniform Elongation, %					

Table 7B. Effect of Hold Time at Test Temperature on Biaxial Tensile Results at 1200°F

Lot	Practice	Pretreatment Condition	Heat Treatment	Before Test RT Hardness DPH	Hold Time Before Test At 1200°F								
					Property	10 Min.	30 Min.	120 Min.	240 Min.				
42403 Item 10	IVCEV	Annealed G.S. - 8	As Fabricated	148	a	49.8	49.2	49.4	49.2				
					b	29.5	29.2	30.1	28.7				
					c	12.1	11.9	----	12.1				
42403 Item 10	IVCEV	Annealed G.S. - 8	24 Hrs @ 1300°F	158	a	47.4	----	46.4	----				
					b	26.1	----	25.4	----				
					c	11.7	----	11.7	----				
42402 Item 9	IVCEV	20% CW G.S. - 9	As Fabricated	273	a	84.1	83.9	79.2	78.2				
					b	80.2	79.6	71.7	69.9				
					c	1.7	1.8	2.2	1.7				
42402 Item 9	IVCEV	20% CW G.S. - 9	24 Hrs @ 1300°F	235	a	71.8	----	70.4	----				
					b	66.3	----	64.6	----				
					c	2.3	----	2.3	----				
<table><tr><th colspan="2">Standard Test Procedure</th></tr><tr><th>Property</th><th>10-30 Min.</th></tr></table>										Standard Test Procedure		Property	10-30 Min.
Standard Test Procedure													
Property	10-30 Min.												
42403 Item 10	IVCEV	Annealed G.S. - 8	As Fabricated	148	a	51.1							
					b	30.4							
					c	10.0							
42402 Item 9	IVCEV	20% CW	As Fabricated	273	a	85.9							
					b	81.3							
					c	0.9							

IVCEV - Induction Vacuum Plus Consumable Electrode Vacuum	Mechanical Properties
	a. Ultimate Tensile Strength, ksi.
	b. 0.2% Yield Strength, ksi.
	c. Uniform Elongation, %

Figure 23 Biaxial Ultimate Tensile Strength of WARD Vacuum Melted and Heat Treated 316 SS vs. Temperature

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Figure 24 Biaxial 0.2% Offset Yield Strength of WARD Vacuum Melted and Heat Treated Type 316 SS vs. Temperature

3514-25

Figure 25 Uniform Elongation of WARD Vacuum Melted and Heat Treated 316 SS vs. Temperature

There are no significant differences in biaxial tensile properties between the drawn and the planetary swaged tubing properties at 850°F to 1100°F and only slight differences at the higher test temperatures. The yield and ultimate strengths of the drawn tubing are slightly higher and the elongations slightly lower at the 1200°F and 1300°F test temperatures. Comparison of room temperature properties can not be made at this time. The test of the planetary swaged tubing must be repeated because of mechanical difficulties.

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 minutes and 240 minutes to determine the effect of this variable on the measured results. The hold-time in the customary procedure for this laboratory is between 10 and 30 minutes.

The tensile, offset yield, and elongation results for solution annealed tubing WARD VM(IVCEV) Lot 42403, for 20% cold worked tubing, WARD VM(IVCEV) Lot 42402 and for specimens from each lot heat treated 24 hours at 1300°F are given in Table 7. Also included in the table are the pre-test room temperature hardness values and results previously obtained at 1200°F from the two as-fabricated lots using the customary test procedure.

The data in Table 7 for the annealed tubing show that variation in pre-test hold time from 10 minutes to 240 minutes had essentially no effect on measured biaxial tensile properties. Pre-test heat treatment of the annealed material for 24 hours at 1300°F caused at most only a small reduction in strength and the ductility.

The cold worked tubing behaved differently. For hold times between 10 and 30 minutes there was little change in measured properties but at 30 minutes and beyond there was reduction in both the ultimate and the yield strengths and a slight reduction in elongation. 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 an apparent slight increase in ductility.

Previously reported elevated temperature biaxial tensile data indicated that the elongation of the annealed material suffers a reduction at about 1200°F (Figure 25). Initially, it was believed that this may be due to a structural 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.

Biaxial Stress Rupture Data

Seventy biaxial stress rupture tests have been completed to date. The forty-nine tests completed during the current report period, together with those reported previously, are shown in Tables 8 and 9.

In the previous report (WARD-4135-2) the stress rupture data were shown as a function of the Larson-Miller parameter:

$$P = T (C + \log t)$$

Table 8. Biaxial Stress Rupture Test Results on Air Melted
Type 316 Stainless Steel Tubing

Lot	Material Condition	Wall Thickness (in.)	Test Temperature (°F)	Stress Level (psi)	Hours to Rupture	Chitty - Duval Parameter
WARD 316727	Annealed	0.010	1000	50,800	194	596.34
			1200	29,000	99.3	712.90
WARD 7-2489	Annealed	0.012	1000	48,000	308	603.2
			1000	41,700	5598	641.45
			1200	36,000	1.6	655.02
			1200	27,000	75.5	711.35
			1200	24,000	27.1	698.44
			1200	24,000	31.5	700.69
			1200	19,800	1826	771.31
			1200	18,000	3945	789.25
WARD 316728	Annealed	0.014	1000	49,000	498	608.13
			1200	40,800	5.9	670.59
WARD 7-2489 Micropeened	A	0.012	1000	46,800	1827	624.44
			1000	36,600	5662	648.71
	B		1000	41,700	2234	630.43
WARD 321921	Cold Worked 15-20%	0.012	1000	60,000	1338	613.04
			1000	57,000	1194	613.39
			1000	57,000	691	607.57
			1000	54,000	550	606.51
			1000	51,000	835	612.50
			1000	50,000	126	
			1000	48,000	4017.9	624.42
			1200	24,000	928	751.38
			1200	21,600	1794	766.14
			1200	19,800	1922	766.14

Table 9. Biaxial Stress Rupture Results
Vacuum Melted Type 316 Stainless Steel Tubing

Lot	Material Condition	Tubing Dimension	Test Temperature °F ^d	Stress psi	Hours to Rupture	Chitty-Deval Parameter	Rupture Strain %
WARD 42403 (Double-Vacuum Melt)	Annealed	0.015	1300	24,000	5.1	750.77	a
	Fine Grain	"	1300	22,000	74.5	781.04	a
	"	"	1300	20,000	29.9	790.92	a
	"	"	1300	18,000	58.5	795.40	a
	"	"	1300	16,000	328	805.13	25.3 ^b
	"	"	1300	14,000	272.6	839.73	a
WARD 42395 (Consumable Electrode Vacuum Melt)	Annealed	0.015	1300	24,000	10.4	717.29	a ^b
	Fine Grain	"	1300	22,000	21.5	729.97	22.6 ^b
	"	"	1300	20,000	35.5	743.76	a
	"	"	1300	18,000	26.7	772.63	a
WARD 42402 (Double Vacuum Melt)	Cold Worked	0.015	1300	30,000	6	744.67	17.0 ^c
	20%-Fine Grain	"	1300	28,000	12	755.07	10.3 ^b
	"	"	1300	26,000	24	766.38	10.9 ^b
	"	"	1300	24,000	340.2	784.85	a
	"	"	1300	22,000	588.8	800.72	10.6 ^b
	"	"	1300	20,000	685.3	815.77	10.7 ^b
WARD 42398 (Induction Vacuum Melt)	Cold Worked	0.015	1300	30,000	34.6	746.25	2.5 ^b
	20%-Fine Grain	"	1300	28,000	70.9	763.21	7100 ^c
	"	"	1300	26,000	32.3	770.56	a
	"	"	1300	24,000	266.1	780.66	47.4 ^c
	"	"	1300	22,000	72.1	787.88	78.6 ^c
	"	"	1300	20,000	508.9	799.96	5.9 ^b
WARD 42394 (Consumable Electrode Vacuum Melt)	Cold Worked	0.015	1300	30,000	6.58	760.48	9.9 ^b
	20%-Fine Grain	"	1300	28,000	10.6	768.19	a
	"	"	1300	26,000	104.7	767.0	4.6 ^b
	"	"	1300	24,000	129.9	774.92	9.8 ^b
	"	"	1300	22,000	42.9	788.77	8.5 ^b
	"	"	1300	20,000	60.4	801.43	92.9 ^c
WARD 42402 (Double Vacuum Melt)	Cold Worked	0.015 x	1300	30,000	189.7	757.2	a
	20%-Coarse	0.230 OD	1300	28,000	320.1	768.42	a
	Grain	"	1300	26,000	375.1	773.38	1.7 ^b
	"	"	1300	24,000	94.8	766.38	20.3 ^c
	"	"	1300	22,000	174.5	801.70	9.9 ^c
	"	"	1300	20,000	240.3	814.80	5.3 ^b
WARD 42402 (Double Vacuum Melt)	Cold Worked	0.015	1300	30,000	29.4	730.25	a
	20%-Fine Grain	"	1300	28,000	55.2	738.61	a
	Garofalo HT	"	1300	26,000	74.1	750.64	a
	Treat	"	1300	24,000	97.5	779.48	a
	"	"	1300	22,000	54.4	757.22	21.8 ^b
	"	"	1300	20,000	78.8	786.74	18.2 ^b
WARD 42402 (Double Vacuum Melt)	Cold Worked	0.015	1300	30,000	49.6	741.4	5.7 ^b
	20%-Fine Grain	"	1300	28,000	178.1	764.15	7.5 ^b
	Planetary	"	1300	26,000	100.9	767.13	8.7 ^b
	Swaged	"	1300	24,000	131	773.47	6.7 ^b
	"	"	1300	22,000	56.9	788.18	3.8 ^b
	"	"	1300	20,000	78.5	793.68	7.1 ^b

Notes:

^aMultiple Rupture

^bPin-hole Rupture

^cLongitudinal Split and Bulge

^dThe 1300°F temperatures are nominal. The exact temperatures were used to calculate parameters and these ranged 1286°F to 1355°F.

where

P = Larson-Miller parameter
T = temperature, °R
C = Constant
t = time, hours

Use of this parameter was an improvement over direct log stress-log time plots since data from different temperatures could be combined. However, a detailed examination of both the WARD and UKAEA^[17] data showed that the Larson-Miller parameter did not completely take into account the effect of temperature. In certain ranges of parameter values, data obtained at different temperatures from the same material did not follow the same curve.

Other parameters^[18,19,20,21] which have been developed to overcome the deficiencies in the Larson-Miller correlation are currently being examined. Although the evaluations of these other methods of data analysis are still in progress, preliminary indications are that the Chitty-Duval method provides a better correlation of the WARD data than the Larson-Miller does. The Dorn^[19] and Manson-Haferd^[20] methods are yet to be evaluated.

The Chitty-Duval parameter, d, is defined as:

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

where

T = Temperature, °C
 $m = \frac{dT}{d \log t}$ (There is a linear relationship between m and stress)
t = time, hours

Stress rupture data from the various WARD materials and data from other sources plotted as a function of the Chitty-Duval parameter are shown in Figures 26 through 32.

The preliminary stress rupture results indicate the following:

1. All the cold worked vacuum melted materials tested to date, have greater biaxial stress rupture strength than cold worked air melted material. (Figure 26)
2. At stress levels less than 24,000 psi, the cold worked double vacuum melted material VM (IVCEV) (both fine and coarse grain) has greater biaxial stress rupture strength than the other vacuum processed materials, while at a stress level of 30,000 psi, the fine grained double vacuum melted material is the weakest. The weakest vacuum processed material tested to date, in the 20,000 to 30,000 psi range, is the CEV fine grained annealed material. (Figure 27)
3. At stress levels higher than 24,000 psi coarse grained, cold worked double vacuum melted tubing has greater biaxial stress rupture strength than the comparable fine grained material. At stress levels less than 24,000 psi, they have approximately the same stress rupture strength. (Figure 28)

Figure 26 Chitty-Duval Biaxial Stress Rupture Plot of WARD Cold Worked Vacuum Melted and Air Melted 316 SS Tubing

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Figure 27 Chitty-Duval Biaxial Stress Rupture Plot of WARD Vacuum Method 316 SS Tubing 3514-28

Figure 28 Chitty-Duval Biaxial Stress Rupture Plot of WARD Fine Grain and Coarse Grain, Vacuum Melted-Cold Drawn 316 SS Tubing

3514-29

4. With the exception of the 20,000 and 30,000 psi stress levels, there is little difference in the stress rupture strengths of the fine grained cold worked tubing lots manufactured by the three different melting practices. At 20,000 psi, the double vacuum melted is superior, while at 30,000 psi the consumable remelt material is superior. (Figure 29)
5. At stress levels greater than 24,000 psi, there is little difference in the stress rupture strengths of the cold worked tubing lots manufactured by planetary swaging and by plug drawing. Below the 24,000 psi level plug drawn material is stronger. (Figure 30)
6. Whereas Garofalo^[1,22] increased the stress rupture property of his annealed material by prestrain and heat treatment. The WARD data indicate no such increase, Figure 31. However, the following differences in processing, prestrain, and test procedure should be noted:

	<u>Processing</u>	<u>Prestrain</u>	<u>Test</u>
WARD	Vacuum Melted	20%-Plug Drawn	Biaxial
Garofalo	Air Melted	25%-Tensile	Uniaxial

The WARD cold worked material is significantly stronger than the WARD cold worked and heat treated material.

7. Figure 32 compares the biaxial stress rupture data of cold worked type 316 stainless steel from three laboratories. The WARD double vacuum melted, coarse grained material has the highest stress rupture strength. However, the AI^[23] data refer to 15-20% cold work whereas the UK^[17] and WARD data are for 20% cold worked material.

The failures exhibited by the stress rupture specimens, based on visual examination, may be categorized into three types:

1. Pin hole type failure - Figure 33a
2. Longitudinal split and bulging - Figure 33b
3. Multiple failure - Figure 33c

The type of failure exhibited by each specimen is shown in Table 9 and Figure 33. Failure types (a) and (b) in 316 stainless steel were the pin-hole type as previously reported by UKAEA investigators^[17]. The failure was described as a slow leak whereas the longitudinal split and bulging type were described as an explosive type. In the present investigation the slow leak or explosive failure definition cannot be substantiated, since propagation rates were not measured.

A discussion of the reasons for the various types of fractures will be deferred until a detailed metallographic examination of the stress rupture specimens is completed.

Figure 29 Chitty-Duval Biaxial Stress Rupture Plot of WARD Vacuum Melted 316 SS Tubing
3514-30

Figure 30 Chitty-Duval Biaxial Stress Rupture Plot of Planetary Swaged and Cold Drawn
3514-31

Figure 31 Chitty-Duval Plot of WARD and Garofalo Stress Rupture Data

3514-82

Figure 32 Chitty-Duval Plot at WARD, A.I., UKAEA Data on Cold Worked 316 SS

3514-33

- A. PINHOLE
- B. SPLIT BULGE
- C. MULTIPLE PIECE

Figure 33 Rupture Modes in Stress Rupture Tests

3514-34

It was often difficult to detect the pinhole fracture site and leak checking of the specimens after test was used to locate the fracture. Strains at fracture of the pinhole type were determined after test with a precision micrometer, reading to 10^{-4} inches. The peak diametral strain at the fracture site was then determined and are shown in Table 9, and as a function of rupture life in Figure 34.

The fracture strains exhibited by the WARD cold worked tubing are significantly higher than those reported by the UKAEA, although it should be emphasized that the latter results are based on longer term tests. Only in one case, the coarse grained double vacuum melted material, are the rupture strain comparable to the UKAEA results.

The diametral strain for the 20% cold worked, fine grain (IVCEV) tubing was measured at regions removed from the fracture and is of the order of 8%. This indicates considerable ductility prior to fracture and confirms the high rupture strains ($\sim 10\%$) recorded for this material.

The following preliminary conclusions can be made from the present results:

1. The bulk of the cold worked material, particularly at the longer test times, failed by pin hole type fracture. This confirms the results of the UKAEA investigators.
2. All the planetary swaged tubing tested to date failed by a pin-hole type fracture.
3. The rupture strains exhibited by the WARD tubing is significantly higher than that exhibited by comparable tubing tested by the UKAEA. [(cf. WARD, 20% CW, (IVCEV) ($\sim 10\%$) with UKAEA (2-5%)]

Uniaxial and Biaxial Creep

Six uniaxial and five biaxial creep tests have been completed to date. The results are listed in Table 10. These data indicate that in both uniaxial and biaxial creep, annealed air melted material has a creep rate about 20 times that of the cold worked material, about $60 \times 10^{-4}\%/hr.$ compared to $3 \times 10^{-4}\%/hr.$ There are not sufficient data to make the same comparison for the vacuum melted material. In biaxial creep, the cold-worked, air melted and vacuum melted materials have about the same measured creep rate, but when tested uniaxially the vacuum melted material has a creep rate 2.5 times or greater than that of the air melted material. In uniaxial creep, the "Garofalo" treated material appears to have a greater creep rate than the 20% cold worked material.

Figure 34 Rupture Strain of Type 316 Stainless Steel at 1300°F Pin-Hole Failures

Table 10. Creep Results 1200°F 27,000 psi Stress

Type Test	Material	Creep Rate %/Hr	Comments
Biaxial	AM Annealed	60×10^{-4}	Tests Terminated at 1000 Hrs
Biaxial	AM Annealed	57×10^{-4}	"
Biaxial	AM Cold Worked	3×10^{-4}	"
Biaxial	VM Cold Worked	3×10^{-4}	"
Biaxial	VM Cold Worked	3.26×10^{-4}	Tests Terminated at 1600 Hrs
Uniaxial	AM Annealed	59×10^{-4}	Ruptured 968 Hrs ^a
Uniaxial	AM Annealed	61×10^{-4}	Ruptured 768 Hrs ^a
Uniaxial	AM Cold Worked	3×10^{-4}	Ruptured 1000 Hrs ^a
Uniaxial	VM Cold Worked	7.5×10^{-4}	Ruptured 672 Hrs ^a
Uniaxial	VM Cold Work + "Garofalo" HTr.		Ruptured 77 Hrs ^a
Uniaxial	VM Cold Work + "Garofalo" HTr.		Ruptured 128 Hrs ^a
^a Ruptures were outside gauge marks			

REFERENCES

1. F. Garofalo, F. Von Gemmingen, and W. F. Domis, Trans. ASM 54 (1961) 430.
2. WARD-3791-26, "Stainless Steel Cladding Development Quarterly Progress Report for the Period Ending September 30, 1968," Westinghouse Electric Corporation, Advanced Reactors Division.
3. WARD-3791-35, "Stainless Steel Cladding Development Quarterly Progress Report for the Period Ending March 31, 1969," Westinghouse Electric Corporation, Advanced Reactors Division.
4. D. Dulieu and J. Nutting, High Alloy Steels, Spec. Rep. 86 of the Iron and Steel Inst., 1964, pp. 140-142.
5. C. Barret and T. Massalski, Structure of Metals, McGraw-Hill Co., 1966, p. 389.
6. D. A. Blenkinsop and J. Nutting, J. Iron & Steel Inst. 205 (1967) 953.
7. P. Duhaï et al., J. of Iron and Steel Inst., 206 (1968) 1245.
8. J. Venables, Phil. Mag. 7 (1962) 35.
9. H. Hu et al., in Recrystallization, Grain Growth, and Textures, ASM (1966), p. 352.
10. H. Hu, in Recovery and Recrystallization of Metals, Edited by L. Himmel, Gordon and Breach, Science Publishers, Inc., 1963, p. 311.
11. H. Wiegand and M. Doruk, Arch. f. Eisenhuettenwesen, 33 (1962) 559.
12. H. J. Goldschmidt, Interstitial Alloys, Plenum Press, New York, 1967.
13. F. R. Beckit and B. R. Clark, Acta. Met., 15 (1967) 113.
14. R. Stickler and A. Vinckier, Mem. Scient. Rev. Met., 60 (1963) 2.
15. WARD-3791-29, "Stainless Steel Cladding Development Quarterly Progress Report for the Period Ending December 31, 1968," Westinghouse Electric Corporation, Advanced Reactors Division.
16. K. Kinoshita et al., Presented at the Fall AIME meeting, Philadelphia, 1969.
17. H. K. Richardson and R. McDowell, "Elevated Temperature Stress Rupture Properties of M316, FV548 and Nimonic PE16 P.F.R.-Type Tube." TRG Report 1482(c) FRPC-FEWP/P(67) 415.
18. J. B. Conway, Numerical Methods for Creep and Rupture Analyses, Gordon and Breach, Science Publishers, New York.

REFERENCES (Continued)

19. R. L. Orr, O. D. Sherby, and J. E. Dorn, "Correlations of Rupture Data for Metals at Elevated Temperatures," Trans. ASM, 46, 1954, p. 113.
20. S. S. Manson and A. M. Haferd, "A Linear Time-Temperature Relation for Extrapolation of Creep and Stress Rupture Data," NACA TN 2890, 1953.
21. H. Chitty and D. Duval, "The Creep Rupture Properties of Tubes for High Temperature Steam Power Plant," Joint International Conf. on Creep. ASME/ASTM/IME, London, 1963, pp. 4-1 to 4-10.
22. F. Garofalo et al., "Creep and Creep Rupture Relationship in an Austenitic Stainless Steel," Trans. AIME, Vol. 221, April 1961.
23. AI-AEC-12817, "Quarterly Technical Progress Report; Fuels, Materials, and Coolant Chemistry Programs; January - March 1969," Atomics International.

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