50 FAS REACTOR No Stock
2-10-70
H. A. Ackerly
Formal

MASTER

THIS DOCUMENT CONFIRMED AS
UNCLASSIFIED
DIVISION OF CLASSIFICATION
BY J. H. Kohn
DATE 2/18/70

Westinghouse Advanced Reactors Division

P4161

Release Given

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

STAINLESS STEEL CLADDING DEVELOPMENT
QUARTERLY PROGRESS REPORT
FOR PERIOD ENDING SEPTEMBER 30, 1969

Approved by:

P. Murray

Prepared for the New York Operations Office
U. S. Atomic Energy Commission
Under Contract AT(30-1)-4135

Submitted to AEC-NY00 in November 1969

Westinghouse Electric Corporation
Advanced Reactors Division
P. O. Box 158
Madison, Pennsylvania 15663

LEGAL NOTICE

This report was prepared as an account of Government sponsored work. Neither the United States, nor the Commission, nor any person acting on behalf of the Commission:

A. Makes any warranty or representation, expressed or implied, with respect to the accuracy, completeness, or usefulness of the information contained in this report, or that the use of any information, apparatus, method, or process disclosed in this report may not infringe privately owned rights; or

B. Assumes any liabilities with respect to the use of, or for damages resulting from the use of any information, apparatus, method, or process disclosed in this report.

As used in the above, "person acting on behalf of the Commission" includes any employee or contractor of the Commission, or employee of such contractor, to the extent that such employee or contractor of the Commission, or employee of such contractor prepares, disseminates, or provides access to, any information pursuant to his employment or contract with the Commission, or his employment with such contractor.

AVAILABILITY NOTICE

Printed in USA. Price \$3.00. Available from the Clearinghouse for Federal Scientific and Technical Information, National Bureau of Standards, U. S. Department of Commerce, Springfield, Virginia 22151.

TABLE OF CONTENTS

Section	Page
INTRODUCTION.....	1
General.....	1
Summary of Objectives.....	1
Summary of Current Progress.....	1
SSBA-110 PROJECT ADMINISTRATION.....	3
General.....	3
Current Progress.....	3
SSBA-210 EVALUATION OF AUSTENITIC STAINLESS STEEL CLADDING.	4
Objective.....	4
Prior Work.....	4
Current Progress.....	5
Test Program.....	5
Procurement.....	5
Characterization.....	7
Mechanical Property Testing.....	17
REFERENCES.....	31

LEGAL NOTICE

This report was prepared as an account of Government sponsored work. Neither the United States, nor the Commission, nor any person acting on behalf of the Commission

A. Makes any warranty or representation, expressed or implied, with respect to the accuracy, completeness, or usefulness of the information contained in this report, or that the use of any information, apparatus, method, or process disclosed in this report may not infringe privately owned rights, or

B. Assumes any liabilities with respect to the use of, or for damages resulting from the use of any information, apparatus, method, or process disclosed in this report.

As used in the above, "person acting on behalf of the Commission" includes any employee or contractor of the Commission, or employee of such contractor, to the extent that such employee or contractor of the Commission, or employee of such contractor prepares, disseminates, or provides access to, any information pursuant to his employment or contract with the Commission, or his employment with such contractor.

NOTICE

This report was received under the provisions of the *Atomic Energy Act of 1954* and is subject to the terms thereof.

LIST OF FIGURES

Figure

1	WARD VM (IVCEV) Tubing Lot 42403, Item 10, Annealed, Grain Size 8.....	11
2	WARD VM (IVCEV) Tubing Lot 42402, Item 9, 20% CW, Grain Size 9.	12
3	WARD VM (IVCEV) Tubing Lot 42402, Item 9, 20% CW, Grain Size 9..	14
4	WARD VM Bar Stock As-Polished Longitudinal Sections.....	15
5	Biaxial Ultimate Tensile Strength of WARD Vacuum and Air Melted 316 SS vs Temperature.....	18
6	Biaxial 0.2% Offset Yield Strength of WARD Vacuum & Melted Type 316 SS vs Temperature.....	19
7	Percent Uniform Elongation of WARD Vacuum and Air Melted 316 SS vs Temperature.....	20
8	Time-Temperature-Precipitation Diagrams for Two Solution Annealed WARD Air Melted Tubing Lots with the Time--Temperature Combinations of the Biaxial Tensile Tests Indicated by Circles.....	21
9	Time to Rupture as a Function of Stress-WARD Air Melted Type 316 Stainless Steel Tubing.....	24
10	Larson-Miller Plot of Biaxial Stress Rupture Data - WARD Air Melted Type 316 Stainless Steel Tubing.....	25
11	Larson-Miller Plot of Biaxial Stress Rupture Data.....	26
12	Cross-Section of Welds Between Type 316 Stainless Steel Tubing and Test Fixture.....	28
13	Typical End Closure in Cladding Weldability Study (75X)....	30

LIST OF TABLES

Table		Page
1	Vacuum Melted Tubing for the Oxide Fuel Element Development Program WARD VM(IVCEV)-BNW Heat V87210.....	6
2	Chemistry WARD VM Type 316 Stainless Steel Tubing.....	8
3	Comparison of Carpenter and WARD Bar Stock Analyses.....	9
4	Hardness and Preliminary Biaxial Tensile Results WARD VM Type 316 Stainless Steel Tubing.....	16
5	Biaxial Stress Rupture Test Results on Air Melted Type 316 Stainless Steel Tubing.....	23

INTRODUCTION

GENERAL

This is the fifth quarterly progress report and covers the work performed during the first quarter of the fiscal year (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 that will assure the maximum performance under LMFBR service conditions.

Initially, a survey will be made to determine tubing material property and microstructural variability based upon procurement of tubing under an existing FFTF specification. This specification will be used as a basic starting point. The unirradiated mechanical properties of the material are related to the tubing microstructure. This has been shown experimentally in unirradiated Type 316 austenitic stainless steel, where prestraining and cold working 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.

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 CURRENT PROGRESS

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

Under Task SSBA-210, stress rupture, and uniaxial and biaxial creep testing of air melted material continued. Additional low cycle high strain fatigue tests were delayed pending proof testing of a new specimen design.

All twelve lots of WARD vacuum melted tubing representing three melting practices, two grain sizes, and two conditions, e.g., 20% cold worked and solution annealed, were received and accepted. Microcharacterization and mechanical property testing were initiated on these materials. An order was placed for vacuum melted planetary swaged tubing to be used in the characterization studies.

All ten lots of Battelle-Northwest Laboratories (BNW) double vacuum remelted 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.

The weldability subtask continued. The first series of weldability studies has been completed on annealed tubing. The second series of weldability studies will be conducted on 15-20% cold worked tubing.

SSBA-110
PROJECT ADMINISTRATION

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

GENERAL

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

The quarterly progress report (WARD-3791-41) for the period ending June 30, 1969, was prepared and distributed. Contract Modification No. 1 was received and executed to cover funding for the month of September 1969.

SSBA-210
EVALUATION OF AUSTENITIC STAINLESS STEEL CLADDING

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

OBJECTIVE

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:

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

The objective of a subtask on weldability 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 O D at three wall thicknesses (0.010, 0.012, and 0.014-inch) and 0.250-inch O D x 0.016-inch wall, 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 a study to determine the effects of cold work was initiated.

Procurement was completed for Type 316 stainless tubing, 0.230-inch O D x 0.015-inch wall, produced from material melted by:

- Vacuum induction
- Consumable electrode vacuum
- Vacuum induction and consumable electrode vacuum

Procurement was initiated for double vacuum remelted Type 316 stainless steel tubing for the Oxide Fuel Element Development Program. A subtask to determine the effects of degree of cold working on weldability was initiated. Equipment requirements were identified and the facility prepared for the test program.

CURRENT PROGRESS

Test Program

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

Procurement

Identification

In this and future reports the following notations will be used to identify the various Type 316 stainless steel tubing procured for this program: the organization setting the specifications on the tubing will be considered the source, such as WARD or BNW. The melting practice will be included in abbreviated form:

● AIR MELTED	AM
● VACUUM MELTED
(General Term)	VM
● VACUUM MELTED
(Induction Vacuum)	VM(IV)
● VACUUM MELTED
(Consumable Electrode Vacuum)	VM(CEV)
● VACUUM MELTED
(Induction Vacuum plus
Consumable Electrode Vacuum)	VM(IVCEV)

The manufacturers lot number will be used for specific identification.

Vacuum Melted Tubing-Westinghouse

All twelve lots of WARD VM tubing were received on July 7, 1969, completing the order from Superior Tube Co.

Vacuum Melted Tubing for the Oxide Fuel Element Development Program (19 pin and 37 pin assemblies)

All ten WARD VM(IVCEV) tubing lots for the Oxide Development Program Irradiation Test (19 pin encapsulated assembly) were received from

Carpenter Technology. The lot numbers and tubing sizes are given in Table 1. Eight of the ten lots met specifications and were accepted. The mechanical test data from the two lots (#9020702 and #9020703) showed that they are slightly out of specification. Since the deviations will have negligible effect on the test program, the two lots have now been accepted.

Table 1. Vacuum Melted Tubing for the Oxide Fuel Element Development Program WARD VM(IVCEV) - BNW Heat V87210				
Carpenter Lot Number	Item Number	Type	Dimensions (in.)	Number of Tubes (3-foot length)
9020702	1	20% Cold Work	0.200 ID 0.010 Wall	18
9020703	2	20% Cold Work	0.200 ID 0.0125 Wall	18
9020704	3	Annealed	0.200 ID 0.010 Wall	24
9020705	4	Annealed	0.200 ID 0.0125 Wall	30
9020713	5	Annealed	0.200 ID 0.015 Wall	12
9020707	6	20% Cold Work	0.223 ID 0.010 Wall	18
9020708	7	20% Cold Work	0.223 ID 0.0125 Wall	30
9020709	8	Annealed	0.223 ID 0.010 Wall	48
9020710	9	Annealed	0.223 ID 0.0125 Wall	24
9020711	10	Annealed	0.223 ID 0.015 Wall	6

A purchase order was placed on September 22, 1969 with Battelle-Northwest for 1000 feet of BNW VM(IVCEV) Type 316 stainless steel tubing to be used in the Oxide Fuel Element Development Program experiments, (37 pin assembly). A partial shipment of 250 feet of 20% cold worked tubing is available and will be shipped as soon as a clearance is received from the Richland AEC office. The remaining 450 feet of cold worked tubing and the 300 feet of annealed tubing will be available for shipment by mid November.

Planetary Swaged Tubing

The confirmed shipping date is October 23, 1969 for the order placed with Superior Tube Company for 200 feet of cold worked and 100 feet of annealed WARD VM(IVCEV) Type 316 tubing to be manufactured by planetary swaging. A similar order will be placed with Carpenter Technology when they confirm their capability to planetary swage.

Fabrication History of Tubing Lots

Superior Tube Company was requested to provide the specific annealing conditions for the twelve WARD VM tubing lots now under investigation and three air melted tubing lots investigated during FY-1969. Characterization of the latter three lots indicated there were some factors not covered by present specifications which caused differences in aging characteristics between the lots. The information received from Superior Tube Company covered partial fabrication histories and annealing conditions for the three air melted annealed tubing lots investigated during FY-1969. No additional information was received relevant to the twelve vacuum melted lots currently under investigation.

It appears that variation in final annealing conditions was not the cause, as was previously suspected, of the observed differences in aging characteristics of the air melted materials. The histories did show differences in the percent reduction per draw, however, which could have caused differences in the texture. There could also have been differences in the amount of residual cold work resulting from post anneal straightening. Both of these could have caused differences in aging characteristics. These factors will be considered in the characterization of the vacuum melted tubing.

Structural Analysis

The aging studies started in FY-1969 on air melted Type 316 stainless steel tubing are now being completed. Samples aged in the temperature range 750°-1800°F for 1500 hours and 5000 hours are being examined. These results will be correlated with those obtained previously from the same lots aged for shorter times.

A support program for structural characterization of WARD VM tubing by light microscopy, electron microscopy, electron probe analysis, and x-ray diffraction has been defined and specimens from two (IVCEV) lots prepared (#42402 - 20% cold worked, grain size 9, and #42403 - annealed, grain size 8). Work is expected to begin by mid October.

Characterization

Chemical Analysis

Six lots of WARD VM tubing were chemically analyzed and the results are shown in Table 2. Data from the source bar stock and the original air melt heat analysis are given in Table 3. The six lots of tubing represent three vacuum melting practices, two grain sizes and two fabrication conditions,

Table 2. Chemistry WARD VM Type 316 Stainless Steel Tubing											
Lot Number - Sample Number											
42394 01-01-02		42397 04-01-02		42399 06-01-02		42400 07-01-02		42402 09-01-03		42405 12-01-02	
Heat Number											
30160-1 VM(CEV)		30160-1 VM(CEV)		91694 VM(IV)		91694 VM(IV)		91695 VM(IVCEV)		91695 VM(IVCEV)	
Ele	w/o	Ele	w/o	Ele	w/o	Ele	w/o	Ele	w/o	Ele	w/o
Fe*	64.8	Fe*	64.7	Fe*	64.5	Fe*	64.7	Fe*	65.0	Fe*	64.9
Cr*	16.2	Cr*	16.3	Cr*	16.1	Cr*	16.0	Cr*	16.3	Cr*	16.4
Ni*	13.6	Ni*	13.8	Ni*	13.8	Ni*	13.5	Ni*	13.6	Ni*	13.8
Mo*	2.6	Mo*	2.6	Mo*	2.5	Mo*	2.6	Mo*	2.4	Mo*	2.4
Mn*	1.5	Mn*	1.5	Mn*	1.5	Mn*	1.6	Mn*	1.4	Mn*	1.3
Si	0.45	Si	0.53	Si	0.47	Si	0.50	Si	0.54	Si	0.52
Co*	0.082	Co*	0.086	Co*	0.087	Co*	0.084	Co*	0.086	Co*	0.088
Cu*	0.22	Cu*	0.22	Cu*	0.21	Cu*	0.21	Cu*	0.21	Cu*	0.21
Nb*	0.017	Nb*	0.016	Nb*	0.016	Nb*	0.016	Nb*	0.018	Nb*	0.018
Ti*	< 0.05	Ti*	< 0.05	Ti*	< 0.05	Ti*	< 0.05	Ti*	< 0.05	Ti*	< 0.05
C	0.041	C	0.050	C	0.044	C	0.045	C	0.050	C	0.035
N	0.025	N	0.032	N	0.021	N	0.026	N	0.027	N	0.022
P	0.012	P	0.011	P	0.007	P	0.011	P	0.009	P	0.011
S	0.011	S	0.010	S	0.010	S	0.017	S	0.013	S	0.012
B** ppm	2.1	B** ppm	0.8	B** ppm	3.1	B** ppm	1.6	B** ppm	4.4	B** ppm	0.9
W*	0.13	W*	0.13	W*	0.11	W*	0.13	W*	0.13	W*	0.13

w/o = weight percent

* Values adjusted to 99.70% of all elements present

** ppm = parts per million

± < 5% Relative high elements

± 10% Relative medium elements

± 30-50% Relative on W

Table 3. Comparison of Carpenter and WARD Bar Stock Analyses													
Source of Analysis													
Carpenter								WARD					
Heat								Heat					
30173 AM		30160-1 VM(CEV)Bar		91694 VM(IV)Bar		91695 VM(IVCEV)Bar		30160-1 VM(CEV)Bar		91694 VM(IV)Bar		91695 VM(IVCEV)Bar	
Ele	w/o	Ele	w/o	Ele	w/o	Ele	w/o	Ele	w/o	Ele	w/o	Ele	w/o
Cr	16.2	Cr	16.5	Cr	16.5	Cr	16.4	Cr	16.9	Cr	17.1	Cr	16.9
Ni	13.8	Ni	13.7	Ni	13.7	Ni	13.8	Ni	14.1	Ni	14.1	Ni	14.0
Mo	2.2	Mo	2.2	Mo	2.2	Mo	2.2	Mo	2.2	Mo	2.2	Mo	2.2
Mn	1.8	Mn	1.6	Mn	1.6	Mn	1.5	Mn	1.5	Mn	1.6	Mn	1.3
Si	.050	Si	0.51	Si	0.47	Si	0.52	Si	0.58	Si	0.58	Si	0.58
Co	0.19	Co	0.19	Co	-	Co	0.19	Co	0.15	Co	0.14	Co	0.13
Cu	0.21	Cu	0.31	Cu	0.20	Cu	0.29	Cu	0.22	Cu	0.21	Cu	0.19
Nb	-	Nb	-	Nb	-	Nb	-	Nb	0.012	Nb	0.010	Nb	0.007
Ti	-	Ti	-	Ti	-	Ti	-	Ti	0.009	Ti	< .001	Ti	0.005
C	0.057	C	0.044	C	0.044	C	0.044	C	0.047	C	0.046	C	0.044
N	0.019	N	0.023	N	0.022	N	0.020	N	0.020	N	0.020	N	0.0175
P	0.016	P	0.016	P	0.017	P	0.017	P	0.014	P	0.014	P	0.014
S	0.017	S	0.014	S	0.016	S	0.013	S	0.020	S	0.020	S	0.019
B	-	B	< .001	B	-	B	< .001	B	.00037	B	.00045	B	0.00065
W	0.15	W	-	W	-	W	-	W	-	W	-	W	-
w/o = weight percent													

annealed and 20% cold worked. All values, except two, reported for the six tubing lots were within the chemical composition specified for the stock material, which was similar to the FFTF Fuel Cladding Specification A-0086 Rev. 1.

One carbon value, that reported for lot 42405, was slightly out of specification, 0.035 w/o compared to specification of 0.04 to 0.06 w/o. This value was probably in error since it was lower than both the supplier and WARD values for the source bar stock and the WARD values from another tubing lot from the same heat. A repeat carbon analysis will be made of lot 42405. One nitrogen value (lot 42397) was slightly over specification, 0.032 w/o compared to 0.030 w/o maximum. A similar interpretation of this

value is reasonable since it too, was out of line with other data from the same heat.

The tubing chemistry data confirmed the loss of some Mn during vacuum remelting and additional loss during the second vacuum remelting. The Mn dropped from 1.8 w/o in the air melted ingot to 1.3 w/o in the double vacuum melted (IVCEV) material.

The Carpenter nitrogen data from the air melted heat and from the bar stock indicate a slight increase in nitrogen occurred during vacuum remelting. The WARD data from the bar stock and from the fabricated tubing indicate that an additional slight increase in nitrogen content occurred during tubing fabrication. A cross comparison between the bar stock data from Carpenter and from WARD showed a slight laboratory bias in the results. The maximum nitrogen increase reported was from 0.019 w/o in the air melted ingot to 0.032 w/o in the VM(CEV) tubing (lot 42397).

Metallography

Internal and External Surface Morphology - The internal and external surfaces of two lots of WARD VM(IVCEV) tubing were examined metallographically by slitting and flattening the tubing sufficiently for viewing with vertical reflected illumination. An area about 0.375 inch in diameter was examined from each surface of each sample. Representative micrographs are shown in Figures 1 and 2.

The external surfaces of both the annealed tubing, lot 42403, and the cold worked tubing, lot 42402, had circumferential grooves, characteristic of surface grinding and some shallow longitudinal scratches. The circumferential grooves in the cold worked tubing were generally 3 to 5 microns deep with an occasional groove 9 microns deep. Those in the annealed tubing were sharper and somewhat deeper, frequently more than 5 microns and occasionally 10 microns deep.

The internal surfaces of both lots appeared to be thermally etched and showed longitudinal grooves. The annealed tubing had equiaxed grains and the cold worked tubing elongated grains but not so well delineated. The deepest longitudinal grooves in both lots were about 5 microns. The grain boundary grooves and the general surface roughness were less. One cavity type defect was observed in the internal surface of the annealed tubing. This defect shown in Figure 1b, was 450 microns long, 90 microns wide, and 25 microns deep. The longitudinal scratches in both specimens were generally the smear type and less than 3 microns deep. The scratch shown in Figure 1b measured 2 microns deep.

Inclusions - Both as-polished and lightly etched transverse and longitudinal sections of WARD VM(IVCEV) tubing lots 43403 and 42402 were examined for inclusion contents. Three inclusion shapes were observed: globular; continuous stringers; and discontinuous stringers. There were no large angular Ti(CN) inclusions as had been observed in air melted tubing. In general, the inclusions were gray in color and one micron or less in thickness.

a

INTERNAL SURFACE 500X

b

INTERNAL SURFACE 100X

c

EXTERNAL SURFACE 250X

TUBE AXIS →

Figure 1 WARD VM (IVCEV) Tubing Lot 42403 Item 10 Annealed, Grain Size 8

a

INTERNAL SURFACE 500X

b

INTERNAL SURFACE 250X

c

EXTERNAL SURFACE 250X

TUBE AXIS →

Figure 2 WARD VM (IVCEV) Tubing Lot 42402 Item 9 20% CW, Grain Size 9

The inclusions were frequent in number but difficult to discern in as-polished sections. Sufficient contrast was obtained by light etching, however. Figure 3 illustrates the difference in contrast between an as-polished surface and a lightly etched surface of a longitudinal section of WARD VM(IVCEV) 20% CW tubing, lot 42402, and a lightly etched surface of the transverse section, all at 500x magnification. The same inclusion morphology was observed with three different chemical etches and a vacuum cathodic etch.

The width of the inclusions is less than 1/5 to 1/8 that of the thin classification in ASTM designation E45 Chart III. As a result, characterization of the inclusion content by direct comparison with this chart at the specified 100x magnification could be misleading. An alternate quantitative or semi-quantitative method of characterizing inclusion content will be developed.

To clarify the apparent discrepancy between the inclusion contents in the WARD VM tubing shown here and the source bar stock described in WARD-3791-41, specimens of the bar stock from the three vacuum remelted heats were re-examined in the longitudinal direction and showed that the bar stock also contains stringer type as well as the globular "D" type inclusions observed previously. In general, the shapes of the inclusions in longitudinal section of the bar stock were consistent with the observations made to date on the tubing samples.

Representative micrographs, at 100x magnification, from the longitudinal sections, are shown in Figure 4. The induction vacuum remelted stock, heat number 91694, had the highest general inclusion count and contained local areas (not shown), of high inclusion density. The consumable electrode vacuum remelted stock, heat number 30160, had the second highest general inclusion content and a few local areas of high inclusion density. The double vacuum remelted stock, heat number 91695, had a slightly lower general inclusion content than heat number 30160 and had no segregated areas of a high inclusion density in the section examined. The width of the stringers in the bar stock should be sufficient for identification by electron probe analysis which is planned for the next report period.

Grain Size - Specimens of WARD VM(IVCEV) tubing lots 42403 and 42402 were etched with either chromic acid electrolytically, or HCL plus HNO_3 in glycerol. The grain shape in the annealed tubing is generally equiaxed in both the longitudinal and transverse sections, whereas the grains in the cold worked lot are elongated slightly in the longitudinal direction. Grain size measurements are not yet complete.

Hardness - Diamond pyramid hardness data from six WARD VM tubing lots are included in Table 4. They show a slight hardness difference between vacuum melting practices, a small increase in hardness with decreasing grain size, and a marked difference resulting from cold work.

Carbide Morphology - Several specimens selected from the aging studies, conducted during FY-1969, were vacuum cathodic etched for electron microscopy to determine if there are morphological differences between carbides precipitating from the matrix and carbides redissolving into the matrix. These specimens will also be used to evaluate the applicability of this etching

3404-3

14

Figure 3 WARD VM (IVCEV) Tubing Lot 42402 Item 9 20% CW, Grain Size 9

a

HEAT 91695 VM (IVCEV) 100X

b

HEAT 30160 VM (CEV) 100X

c

HEAT 91694 VM (IV) 100X

TUBE AXIS

Figure 4 WARD VM Bar Stock As-Polished Longitudinal Sections

Table 4. Hardness and Preliminary Biaxial Tensile Results
WARD VM Type 316 Stainless Steel Tubing

Lot	Melting Practice	Certified Grain Size	Condition	Hardness DPH (2)	Mechanical Properties						
					Property	RT	850°F	1000°F	1100°F	1200°F	1300°F
42402 Item 9	IVCEV ⁽⁵⁾	9	20% CW	273	a b c	141.9 136.2 1.2	114.0 106.8 0.7	106.4 103.9 0.9	100.5 96.4 1.0	85.9 81.3 0.9	79.0 70.2 1.8
42398 Item 5	IV ⁽³⁾	9	20% CW	278	-	(1)	-	-	-	-	-
42394 Item 1	CEV ⁽⁴⁾	9-9.5	20% CW	276	-	(1)	-	-	-	-	-
42404 Item 11	IVCEV	5.5-6	20% CW	261	a b c	132.0 126.5 2.7	103.6 98.0 1.5	96.8 93.2 1.5	90.6 85.6 2.4	76.0 71.0 1.7	69.3 62.2 3.7
42403 Item 10	IVCEV	8	Annealed	148	a b c	79.4 53.4 -	59.8 36.6 16.1	55.2 34.0 -	51.6 31.5 9.1	51.1 30.4 10.0	42.8 26.5 18.3
42395 Item 2	CEV	8	Annealed	142	-	(1)	-	-	-	-	-

(1) Data to be completed during next report period.

(3) IV - induction vacuum remelt

(2) Average of seven measurements
500 gram load 20% CW
300 gram load Annealed

(4) CEV - consumable electrode
vacuum remelt

(5) IVCEV - induction vacuum plus consumable electrode vacuum remelt

Mechanical Properties

- a. Ultimate Tensile Strength, ksi
- b. 0.2% Yield Strength, ksi
- c. Uniform Elongation, %

procedure for replica electron microscopy of thin walled stainless steel tubing. Evaluation of the specimens will be completed during the next report period.

Mechanical Property Testing

Specimen Preparation - Specimens from three lots of WARD VM(IVCEV) tubing, 42402, 42403, and 42404, were prepared for the following tests:

- Biaxial tensile
- Biaxial creep
- Biaxial stress rupture
- Uniaxial creep

Specimens from lot 42402 were also prepared for heat treating studies to be initiated during the next report period.

Equipment - Installation of the three new furnaces for biaxial stress rupture testing continued during this report period. The required modifications of the first of these have been completed. Installation of a second biaxial creep test facility was initiated and completed during this report period.

Biaxial Tensile Tests - Biaxial tensile data from three lots of WARD VM(IVCEV) tubing (lots 42402, 42403, and 42404) are given in Table 4 along with hardness data and grain sizes. The ultimate tensile strengths, yield strengths, and elongations are plotted in Figures 5, 6, and 7 for the vacuum melted lots and for two air melted lots tested previously, (AM lot 316728 - solution annealed 0.014-inch wall, and AM lot 321921 - 15 to 20% cold work 0.012-inch wall).

A comparison of results within the vacuum melted lots shows that the annealed fine grained tubing (lot 42403) has much lower hardness, yield and ultimate tensile strengths, and greater elongation than either the fine grained or coarse grained cold worked lots. The coarse grained cold worked tubing (lot 42404) does, however, have lower hardness, yield and ultimate tensile strengths, and greater elongation than the fine grained cold worked tubing (lot 42402).

A comparison of the elevated temperature tensile data, obtained to date, from the vacuum melted lots with those from the air melted lots reveals that cold work, as a variable, has a greater effect on biaxial tensile properties than either melting practice or grain size (Figures 5, 6, and 7). There are indications that the elongation of the annealed materials suffers a reduction at about 1200°F (Figure 5). This may be due to structural changes occurring during the test. In Figure 8 the time and temperature conditions of the test are superimposed on the time-temperature precipitation curves of annealed materials (reported in WARD-3791-35). It is apparent that, at temperatures of 1200°F and higher, precipitation can take place during the test but not at lower temperatures. To study this effect in

Figure 5 Biaxial Ultimate Tensile Strength of WARD Vacuum and Air Melted 316SS vs Temperature

Figure 6 Biaxial 0.2% Offset Yield Strength of WARD Vacuum & Air Melted Type 316SS vs Temperature

Figure 7 Percent Uniform Elongation of WARD Vacuum and Air Melted 316SS vs Temperature

Figure 8 Time-Temperature-Precipitation Diagrams for Two Solution Annealed WARD Air Melted Tubing Lots with the Time--Temperature Combinations of the Biaxial Tensile Tests Indicated By Circles

3404-8

greater detail, it is planned to conduct a series of biaxial tensile tests in which hold-time prior to test will be varied. The results of these tests will yield information on the effects of testing procedure, particularly soak time, on the measured mechanical properties and may result in a change of testing procedure.

Biaxial Stress Rupture - Six additional stress rupture tests were initiated during this report period. These specimens were air melted 15-20% cold worked tubing with fine grain size. Table 5 lists all of the WARD biaxial stress rupture results obtained to date. The five specimens which ruptured during the current report period are indicated. These and the other WARD data are plotted as log stress versus log rupture time in Figure 9 and as log stress versus the Larson-Miller parameter in Figure 10. The Larson-Miller Parameter is defined by

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

where: P = Larson-Miller Parameter

T = temperature, °R

C = Constant

t = time, hr

A value of 20 was used for the constant C, which has previously been reported for Type 316 stainless steel^(1,2).

The distributions of the WARD data points in Figures 9 and 10 indicate there may be a real effect of cold work on stress rupture properties, at least at the low values of the Larson-Miller Parameter which in this case corresponds to data obtained at 1000°F. There are insufficient data from cold worked material tested at 1200°F to indicate whether there is an increase in the Larson-Miller Parameter associated with cold work at the higher temperature. There appears to be no significant effect of micropeening on the stress rupture results.

Figure 11 is a Larson-Miller master plot of all available biaxial stress rupture data for Type 316 stainless steel. It includes data from BNW⁽³⁾, Atomic International (AI)⁽¹⁾, United Kingdom Atomic Energy Authority (UKAEA)⁽⁴⁾ and WARD. The testing conditions include stresses from 7,000 - 64,000 psi, temperatures from 1000 - 1400°F, times from 1.6 to 17,000 hours, and test environments of vacuum, helium, sodium, and air. Considerable scatter is evident in the results, which may, in part, be due to the choice of a value of 20 for the constant. During the next report period statistical methods will be used to evaluate the data and to determine the most applicable value of the constant. It should be noted that the data obtained in sodium and helium show excellent correlation (solid line in Figure 10). This may be the result of better temperature control and possibly cleaner surfaces in the presence of sodium and helium.

Table 5. 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	Larson-Miller Parameter ($P \times 10^{-3}$)
WARD 316727	Annealed	0.010	1000	50,800	194	32.6
			1200	29,000	99.3	36.8
WARD 7-2489	Annealed	0.012	1000	48,000	308	32.9
			1000	41,700	5598 ←	34.8
			1200	36,000	1.6	>35
			1200	27,000	75.5	36.5
			1200	24,000	27.1	35.7
			1200	24,000	31.5	35.8
			1200	19,800	1826	38.9
WARD 316728	Annealed	0.014	1000	49,000	498	33.4
			1200	40,800	5.9	>35
BNW 316870	Annealed	0.016	1000	36,500	7001 *	
			1000	35,000	7001 *	
			1000	30,000	7001 *	
WARD 7-2489 Micropeened	A	0.012	1000	46,800	1827	32.55
			1000	36,600	5662 ←	34.75
	B	0.012	1000	41,700	2234	34.3
WARD 321921	Cold Worked 15-20%	0.012	1000	60,000	1338	33.8
			1000	57,000	1194 ←	33.8
			1000	57,000	691	33.4
			1000	54,000	550	33.25
			1000	51,000	835	33.5
			1000	50,000	126 *	
			1000	48,000	2429 *	
			1000	45,000	3792 *	
			1000	41,600	5209 *	
			1000	40,200	1320 *	
			1200	24,000	448 *	
			1200	21,600	810 *	
			1200	19,800	1922 ←	38.9
			1200	18,000	137 *	

* Test in Progress

←Failed during current report period

$$P = T (20 + \log t)$$

where T = temperature, °R

t = time, hr

Micropeened (A) - 100% coverage with 0.0025"-0.0035" diameter glass beads.

Micropeened (B) - 150% coverage with 0.0025"-0.0035" diameter glass beads.

Figure 9 Time to Rupture as a Function of Stress-WARD Air Melted Type 316 Stainless Steel Tubing

Figure 10 Larson-Miller Plot of Biaxial Stress Rupture Data - WARD Air Melted Type 316 Stainless Steel Tubing

Figure 11 Larson-Miller Plot of Biaxial Stress Rupture Data

Uniaxial Creep - The 15-20% cold worked tubing, WARD (AM) lot 32192, was creep tested at 1200°F and 27,000 psi and ruptured in 1000 hours. The secondary creep rate was 3×10^{-4} percent per hour. Two creep tests were initiated on WARD VM(IVCEV) 20% cold worked tubing lot 42402. One of these was discontinued because of temperature excursions. Preliminary results from the second test after 650 hours at 1200°F and a stress of 27,000 psi gave a creep rate of 7.5×10^{-4} percent per hour, indicating a creep rate 2.5 times that of the air melted, cold worked material tested under comparable conditions. The test is still in progress.

Biaxial Creep - Preliminary results from a creep test, after 480 hours at 1200°F and 27,000 psi, on WARD VM(IVCEV) 20% cold worked tubing, lot 42402, indicates a secondary rate of 3×10^{-4} percent per hour. This value is comparable to the uniaxial creep rate of cold worked, air melted materials and is 40% that of the uniaxial creep rate of the same lot of cold worked vacuum melted tubing.

Low Cycle Strain Fatigue - Figures 12a and 12b are micrographs from longitudinal sections of the weld areas in two specimens from the low cycle strain fatigue tests described in WARD-3791-41. The weld in Figure 12a experienced no weld failure during 41 cycles at 1200°F over a strain range of approximately 1.46%, whereas the specimen containing the weld in Figure 12b experienced weld failure during 198 cycles at 1200°F over a strain range of approximately 0.69%. Improved welding procedure to ensure sufficient weld penetration has been incorporated into the fabrication of new specimens.

A new support fixture design has been developed which includes extended mandrels to provide support of the test specimen over approximately 90% of its length. This modified specimen design will be proof-tested during the next period by performing runs at the two strain ranges used previously. On successful completion of these tests, a full program of low cycle fatigue testing at 1200°F will be initiated. The program will include determining the strain range in which buckling is first noted and the variation of fatigue life with strain rate.

Cladding Weldability Subtask

Weldability-Tests on Annealed Type 316 Stainless Steel Cladding - The first series of weldability studies has been completed on annealed Type 316 stainless steel tubing (WARD (AM) Heat 20303, lot 316726). A total of 104 welds were made for this series, of which four were rejected from the weldability study because they were not made under the reference welding conditions. Three of these samples had not been completely dried after degreasing, resulting in acetone vaporizing from the joint and contaminating the arc. The other sample had not been properly tightened in the rotating chuck and the joint moved away from the electrode when the internal argon purge was started. The resultant weld was about an eighth of an inch from the joint line. Of the remaining 100 end-closure welds, only one exhibited a visible defect (a blow hole).

Seventy-five of these welds have been radiographed and leak tested using procedures similar to those used for EBR-II and GETR irradiation pins. Only

After 41 Cycles at 1200°F Over a Strain Range of Approx 1.46%;
Deep Weld Penetration; No Weld Failure

After 198 Cycles at 1200°F Over a Strain Range of Approx 0.69%;
Shallow Weld Penetration; Cracking Associated with Weld

Figure 12 Cross-Section of Welds Between Type 316 Stainless Steel Tubing and Test
Fixture

3404-12

the sample with the visible blow hole showed a defect indication during this examination. Metallographic examination has revealed satisfactory weld profiles in all samples examined. A typical longitudinal section is shown in Figure 13. Sixteen of these end-closure welds will be placed in WBTL -2* to characterize their behavior in a sodium environment simulating LMFBF conditions.

Weldability of Cold Worked Type 316 Stainless Steel Cladding - Samples of cold worked tubing (Lot 321921) from the same heat as the annealed tubing (Heat 20303) have been machined for the second series of the weldability study. Measurements of this tubing with air gauging equipment revealed that the average internal diameter was 0.001 inch larger than the annealed tubing. Consequently, another set of end plugs had to be machined so that plug-tubing interference fits, as specified for the annealed tubing, can be maintained.

Interference Fit Tests - The significance of fit-up variations is also being evaluated. Thirty test pieces each of the annealed and the cold worked cladding are being welded with a zero fit instead of the usual 0.0015 interference fit.

* Weld and Braze Test Loop

Figure 13 Typical End Closure in Cladding Weldability Study (75X)

3404-13

REFERENCES

1. W. T. Lee, "Biaxial Stress Rupture Properties of Austenitic Stainless Steels in Static Sodium," AI-AEC-12694.
2. J. B. Conway, "Stress-Rupture Parameters: Origin, Calculation and Use." GEMP-555, p. 47 (June 1967).
3. Staff, FFTF Fuels Department, "FFTF Fuels Development Current Preliminary Data Report for March 1969." BNWL-1054.
4. 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).

EXTERNAL DISTRIBUTION LIST

U. S. ATOMIC ENERGY COMMISSION

Division of Reactor Development and Technology
Washington, D. C. 20545

Assistant Director, Engineering Standards
Assistant Director, Nuclear Safety
Assistant Director, Plant Engineering
Assistant Director, Program Analysis
Assistant Director, Project Management (2)
Assistant Director, Reactor Engineering (2)
Assistant Director, Reactor Technology
Chief, Fuels and Materials Branch (3)
Chief, Fuel Engineering Branch
Chief, Reactor Vessels Branch

Division of Naval Reactors
Chief, Nuclear Materials Branch
United States Atomic Energy Commission
Washington, D. C. 20545

New York Operations Office
Manager (2)
U. S. Atomic Energy Commission
376 Hudson Street
New York, New York 10014

Division of Technical Information Extension (3)* (50)**
United States Atomic Energy Commission
P. O. Box 62
Oak Ridge, Tennessee 37831

Office of Assistant General Council for Patents
U. S. Atomic Energy Commission
Washington, D. C. 20545

Assistant Director Pacific Northwest Laboratory
U. S. Atomic Energy Commission
P. O. Box 550
Richland, Washington 99352

AEC-RDT Site Offices
Argonne National Laboratory
U. S. Atomic Energy Commission
Building 2
Argonne, Illinois 60439

* Submitted with transmittal form AEC-426

** Submitted for transmittal to recipient under UKAEA/USAEC and EURATOM/
USAEC Fast Breeder Reactor Information Exchange Arrangements

Argonne National Laboratory
U. S. Atomic Energy Commission
P. O. Box 2108
Idaho Falls, Idaho 83401

Atomics International
U. S. Atomic Energy Commission
P. O. Box 1446
Canoga Park, California 91304

General Electric Company, NSP
U. S. Atomic Energy Commission
P. O. Box 15132
Cincinnati, Ohio 45215

General Electric Company
U. S. Atomic Energy Commission
310 DeGuigne Drive
Sunnyvale, California 94086

Gulf - General Atomic
U. S. Atomic Energy Commission
P. O. Box 2325
San Diego, California 92112

Oak Ridge National Laboratory
U. S. Atomic Energy Commission
P. O. Box X
Oak Ridge, Tennessee 37830

Pacific Northwest Laboratory
U. S. Atomic Energy Commission
P. O. Box 550
Richland, Washington 99352

Westinghouse Electric Corporation (2)
U. S. Atomic Energy Commission
Advanced Reactors Division
P. O. Box 158
Madison, Pennsylvania 15663

LABORATORIES

Director, LMFBR Program Office (2)
Argonne National Laboratory
9700 South Cass Avenue
Argonne, Illinois 60439

Director, Metallurgy Division (2)
Argonne National Laboratory
9700 South Cass Avenue
Argonne, Illinois 60439

Manager, FFTF Project (2)
Pacific Northwest Laboratory
P. O. Box 999
Richland, Washington 99352

FTTF Fuels Department (2)
Pacific Northwest Laboratory
P. O. Box 999
Richland, Washington 99352

Manager, Chemistry and Metallurgy Division
Pacific Northwest Laboratory
P. O. Box 999
Richland, Washington 99352

Division Leader, Chemistry and Metallurgy Division (CMB)
Los Alamos Scientific Laboratory
P. O. Box 1663
Los Alamos, New Mexico 87544

Director, Metallurgy and Materials Science Division
Brookhaven National Laboratory
Upton, New York 11973

Director, Metals and Ceramics Division (2)
Oak Ridge National Laboratory
P. O. Box X
Oak Ridge, Tennessee 37830

Division Chief, M & S Division
NASA - Lewis Research Center
21000 Brookpark Road
Cleveland, Ohio 44135

Director, Atomics International
Liquid Metal Engineering Center
P. O. Box 309
Canoga Park, California 91304

Division Leader, Inorganic Materials
Chemistry Department
Lawrence Radiation Laboratory
P. O. Box 808
Livermore, California 94551

Manager, Advanced Development Activity
General Electric Company
Knolls Atomic Power Laboratory
P. O. Box 1072
Schenectady, New York 12301

General Manager, Westinghouse Electric Corporation
Bettis Atomic Power Laboratory
P. O. Box 79
West Mifflin, Pennsylvania 15122

CONTRACTORS

Director, LMFBR Technology Program
Atomics International
P. O. Box 309
Canoga Park, California 91304

Associate Manager, Materials Engineering Department
Battelle Memorial Institute
Columbus Laboratories
505 King Avenue
Columbus, Ohio 43201

Director, Nuclear Development Center
The Babcock and Wilcox Company
Atomic Energy Division
Lynchburg, Virginia 24501

Manager, Nuclear Laboratories
Combustion Engineering, Inc.
Nuclear Division
Prospect Hill Road
Windsor, Connecticut 06095

Laboratory Assistant Director
Gulf-General Atomic
P. O. Box 608
San Diego, California 92112

Manager, Plutonium Chemistry and Ceramics Fuels Development
Plutonium Laboratory
Nuclear Materials and Equipment Corporation
Leechburg, Pennsylvania 15656

Manager, Sodium Reactor Technology
General Electric Company
Breeder Reactor Development Operation
210 DeGuigne Drive
Sunnyvale, California 94086

Manager, Research, United Nuclear Corporation
Research and Engineering Center
Grasslands Road
Elmsford, New York 10523

Head, Fuels and Materials
Atomic Power Development Associates
1911 First Street
Detroit, Michigan 48226

K-2 Group Leader
Reactor Division
Los Alamos Scientific Laboratory
P. O. Box 1663
Los Alamos, New Mexico 87544

Irradiations Manager
EBR-II Project
Argonne National Laboratory
P. O. Box 1096
Idaho Falls, Idaho 83401

Director, Vallecitos Nuclear Center
General Electric Company
P. O. Box 846
Pleasanton, California 94566