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WARD-3791-29
Category UC-25

STAINLESS STEEL CLADDING DEVELOPMENT

QUARTERLY PROGRESS REPORT

FOR THE PERIOD ENDING DECEMBER 31, 1968

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Under AEC Contract AT(30-1)-3791

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INTRODUCTION

This is the second quarterly progress report and covers the work performed during the second quarter of the fiscal year (FY)-1969.

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

A summary of current progress is shown below. Detailed progress is reported in the individual task sections of this document.

Under Task SSAA-110, authorization for the FY-1969 scope of work and full FY-1969 funding were obtained. Formal approval of our FY-1969 work program and formal concurrence with our October request for Government furnished materials and services were also received. A thorough review and updating of program status and plans were conducted for the AEC's mid-year review.

Under Task SSAA-210, an order was placed for procurement of vacuum-melted tubing with delivery scheduled for June, 1969. Air-melted Type 316 stainless steel tubing was received from PNL, and characterization studies were started.

Tensile test and biaxial stress rupture tests were conducted at temperatures of 850, 1000, and 1200°F. The biaxial stress rupture data were compared with other available data. Data obtained on micro-peened tubing revealed improvements in short-time mechanical properties. A program involving the characterization of tubing processed by planetary swaging was planned.

Under Task SSAA-310, preliminary examination of a stress-rupture specimen revealed intergranular as well as secondary transgranular cracks. Sensitization studies were started over the temperature range 750 to 1650°F for times up to 5000 hours.

SSAA-110 - PROJECT ADMINISTRATION

A. E. Duval, W. B. Heubel, P. Murray, P. L. Wyzenbeek

OBJECTIVES

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

PRIOR ACCOMPLISHMENTS

Authorization for work in FY-1969 was received from the Commission based on the work program submitted in June, and contract modifications 9 through 11, which funded work through September, were received and executed. Internal work authorizations and budgets were established based on RDT's directions of July 24. The FY-1969 work program, WARD-3791-22, was revised and issued.

CURRENT PROGRESS

Contract modifications 12 through 14 were received and executed. Modification 14 authorized the FY-1969 scope of work, and provided full FY-1969 funding. Formal approval of our FY-1969 work program and formal concurrence with our October request for Government-furnished materials and services were also received. At the end of the period, program status and plans were thoroughly reviewed and updated in preparation for the AEC's mid-year review.

SSAA-210 - EVALUATION OF AUSTENITIC STAINLESS STEEL CLADDING

E. C. Bishop, M. G. Cowgill, W. E. Ray, H. J. Snyder, 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:

- a. enhance the understanding of the effect of fabrication processes and resulting structure on the properties of thin-wall tubing, and
- b. provide out-of-reactor data required to predict and improve swelling behavior and strain limits of cladding in LMFBR environments.

PRIOR ACCOMPLISHMENTS

A detailed test program was prepared. Air-melted, annealed Type 316 stainless steel tubing, 0.30-inch O.D. at three wall thicknesses (0.010, 0.012, and 0.014-inch) was procured. Biaxial mechanical property testing of this material at 850, 1000, and 1200°F was initiated. Chemical and microstructural evaluation of the tubing was started.

Procurement of Type 316 stainless steel tubing (0.235-inch O.D. x 0.015-inch wall) produced from material melted as follows, was initiated:

- a. vacuum induction
- b. consumable electrode vacuum
- c. vacuum induction and consumable electrode vacuum.

Preliminary electron beam welding of axial fuel pin spacers was initiated. These tests indicate that fixed axial fuel spacers can be welded inside a fuel tube by electron beam welding.

CURRENT PROGRESS

Test Program

The detailed test program outlined in the previous quarterly report (WARD-3791-26) continued. Although not specified in the work program, mechanical property tests will be conducted at 1300°F, because the fuel element may be subjected to this temperature during service.

A preliminary evaluation of the effect of planetary swaging on the properties of austenitic stainless steel was carried out. The limited data available show a significant increase in ductility in internally pressurized tubes at high temperatures. A program to study the effect of planetary swaging in greater detail is outlined on page 14.

Procurement

Air-Melted Tubing - PNL

Thirty feet of air-melted, Type 316 stainless steel tubing (Heat #20276), 0.25-inch O.D. x 0.016-inch wall, was received from PNL. Fifteen feet of this tubing was reserved for the fabrication of mixed-oxide fuel pins for irradiation in EBR-II in mid-calendar year (CY)-1969. The remaining 15 feet are now being fully characterized at WARD. An additional 30 feet of this tubing have been requested from PNL.

Vacuum-Melted Tubing - Westinghouse

On November 22, an order was placed with Carpenter Steel Company (vacuum melting and fabrication to bar stock) and with Superior Tube Company (tubing processing) for Type 316 stainless steel tubing, 0.235-inch O.D. x 0.014-inch wall. Table 1 shows the delivery schedule for the tubing; anticipated delivery at WARD is in June 1969. Table 2 shows the various types of tubing to be procured, and the amounts of bar stock to be held in reserve for subsequent processing.

Vacuum-Melted Tubing - PNL

The following tubing was requested from PNL:

Type 316 stainless steel, double-vacuum melted, 0.230-inch O.D. x 0.014-inch wall	} annealed, 1000 ft - delivery May, 1969
	} 5% cold worked, 100 ft - delivery May, 1969
	} 10% cold worked, 100 ft - delivery May, 1969
	} 20% cold worked, 1000 ft - delivery May, 1969
Preproduction run of above tubing }	20% cold worked, 500 ft - delivery Feb., 1969

The above tubing will be fully characterized by WARD, and the results will be compared to those obtained on Westinghouse vacuum-melted and air-melted tubing.

To provide vacuum-melted tubing for irradiation test pins for the Oxide Fuel Element Development portion of this project, negotiations are proceeding with PNL and with Carpenter Steel Company to obtain sufficient bar material of the double-vacuum-melted, PNL heat (Heat #87210) to make the following tubing:

Table 1

Delivery Schedule for Westinghouse Vacuum-Melted Tubing

WARD Vacuum Melted Type 316 Stainless Steel Tubing Procurement Schedule

ACTION	ELAPSED TIME	DATE
1. Place order		11/22/69
2. Melt and process to bar stock (Carpenter Steel Company)	13 to 16 weeks	3/1/69 to 3/21/69
3. Ship to Superior Tube Company	1 week	3/8/69 to 3/28/69
4. Process to tubing (Superior Tube Company)	13 weeks	6/1/69 to 6/21/69
5. Ship tubing to <u>WARD</u>	1 week	6/8/69 to 6/28/69

Table 2

Processing Schedule for Westinghouse Vacuum-Melted Tubing

	To Be Processed		Bar Stock Held* in Reserve
	Grain Size I	Grain Size II	
<u>Vacuum Induction</u>			
Annealed	200 ft (48 lb used)	200 ft (48 lb used)	} 680 lb ~2800 ft tubing
25% CW	200 ft (48 lb used)	200 ft (48 lb used)	
25% CW + aged**	200 ft (48 lb used)	200 ft (48 lb used)	
<u>Vacuum Consumable Electrode</u>			
Annealed	200 ft (48 lb used)	200 ft (48 lb used)	} 680 lb ~2800 ft tubing
25% CW	200 ft (48 lb used)	200 ft (48 lb used)	
25% CW + aged**	200 ft (48 lb used)	200 ft (48 lb used)	
<u>Vacuum Induction + Vacuum Consumable Electrode</u>			
Annealed	1000 ft (240 lb used)	200 ft (48 lb used)	} 488 lb ~2000 ft tubing
25% CW	200 ft (48 lb used)	200 ft (48 lb used)	
25% CW + aged**	200 ft (48 lb used)	200 ft (48 lb used)	

*The bar stock reserve can be used for later procurement in order to assess process variability from one time period to another.

**Aging to be carried out at WARD

<u>Type of Processing</u>	<u>Tubing Manufacturer</u>	<u>End-Use</u>	(Preliminary Estimate) <u>Quantity</u>
Conventional	Superior	EBR-II Pins and Characterization Studies under this Task	500 ft
Conventional	Carpenter	Characterization under this Task	500 ft
Planetary Swage	ORNL, Superior Tube, and Carpenter Steel	Characterization under this Task	500 ft

As mentioned previously, a detailed study of the effect of planetary swaging on the mechanical properties of tubing is planned. Details of the test plan are discussed on page 14. The assistance of the USAEC in arranging for planetary swaging at ORNL has been requested. The planetary swaging subcontractor will be selected after schedule and cost information is reviewed.

Mechanical Properties

Annealed Tubing

The results of the mechanical property testing of annealed Type 316 stainless steel tubing (Westinghouse air melt and PNL air melt) under biaxial conditions at 850, 1000, and 1200°F are shown in Tables 3 and 4.

The tensile test results (Table 3) show that the 0.2% offset yield strength increases with a decrease in wall thickness at all temperatures. The yield strength of the 0.010-inch wall tubing is 18% higher than that of the 0.014-inch wall tubing at 850°F, and 15% higher at 1200°F. The ultimate tensile strengths and elongation values are not significantly different for the various wall thicknesses.

The biaxial stress rupture results are plotted in Figure 1 along with biaxial stress rupture data reported by the UKAEA^[1] and AI^[2]. The consistency of the data at 1200°F is noted notwithstanding differences in melting practice, chemical composition (Ni, C, and N), wall thickness, and test environment as described in Table 5.

Micro-peened Tubing

The outside surface of several lengths of 0.30-inch O.D. x 0.012-inch wall tubing (Tube Lot #7-2489) was micro-peened with a slurry of glass beads between 0.0025 and 0.0035-inch diameter by the Micropeen Corporation of New Jersey. Two peening conditions were obtained: 100 % coverage and 150% coverage, which are defined below:

Figure 2 shows a schematic diagram of the micro-peening unit. Test lengths are peened at a given air pressure, bead diameter, tube rotation, entry speed, and the time estimated to achieve complete coverage of the surface. The extent of surface coverage is determined at a magnification of x10. Complete surface coverage is then defined as 100% coverage. To obtain 150% coverage, the rotation and entry speeds of the tube into the machine are adjusted so that it takes 50% more time for the tube to pass through the peening unit.

Table 3

Biaxial Tensile Test Results on Westinghouse Air-Melted
Type 316 Stainless Steel Tubing

	Cladding Thickness (in.)	Test Temp. (°F)	0.2% Yield Strength (psi)	Ultimate Stress (psi)	Elongation (%)
Westinghouse Air Melt	0.010	850	31,200	61,000	19.3
	0.012	850	28,500	61,500	17.7
	0.014	850	26,500	59,000	18.6
	0.010	1000	30,400	59,800	20.0
	0.012	1000	25,200	59,000	16.3
	0.014	1000	25,000	58,000	20.0
	0.010	1200	26,000	53,200	15.0
	0.012	1200	24,500	52,300	19.7
	0.014	1200	22,500	51,400	17.0
	0.012	850		65,600B	18.3
	0.012	1000	35,000	62,400B	16.7
	0.012	1000	32,000	61,000B	18.3
	0.012	1000	28,500	62,500A	18.3
	0.012	1200	27,800	56,300A	18.3
	0.012	1200	33,900	56,400B	15.0

Annealed

Micropeened

Table 4

Biaxial Stress Rupture Test Results on Westinghouse and PNL
Air-Melted Type 316 Stainless Steel Tubing

Cladding Thickness (in.)		Test Temp. (°F)	Stress Level (psi)	Hours to Rupture
Westinghouse Air Melt	0.010 0.012 0.014	1200	29,000	99.3
		1200	36,000	1.6
		1200	40,800	5.9
	0.010 0.012 0.012	1000	50,800	194.0
		1000	48,000	308.0
		1000	40,000	100*
	0.014 0.016 0.016	1000	49,000	497.8
		1000	36,500	100*
		1000	35,000	100*
	0.016 0.016 0.016	1000	30,000	100*
		1000	25,000	100*
		1000	25,000	100*
Westinghouse Air Melt	0.012 0.012 0.012	1000	45,000	100*A
		1000	40,000	100*B
		1000	35,000	100*A
	0.010 0.012 0.014	850	58,000	300*
		850	54,000	1225*
		850	51,000	1630*

*Test in Progress

A = 100% Coverage

B = 150% Coverage

Figure 1 Stress Rupture Properties of Type 316 Stainless Steel under Biaxial Conditions

Table 5

Chemical Composition and Dimensional Characteristics of Type 316
Stainless Steel Tubing used in Biaxial Test Programs

	Westinghouse Air Melt Ht. #2303 0.30-in. O.D. x 0.010, 0.012, 0.014-in. wall (wt%)	PNL Air Melt Ht. #20276 0.250-in. O.D. x 0.016-in. wall (wt%)	AI ^[1] Air Melt Ht. #65808 0.0295-in. O.D. x 0.010-in. wall (wt%)	UKAEA ^[2] Vacuum Melt Ht. #VA444/03 0.230-in. O.D. x 0.015-in. wall (wt%)
Ni	11.66	13.31	13.4	13.8
Cr	18.20	17.70	17.43	17.3
Mo	2.48	2.45	2.5	2.43
C	0.054	0.05	0.012	0.05
N	0.0726	0.051	0.070	0.05
S	0.012	0.015	0.002	0.005
P	0.022	0.016	0.004	0.01
O	0.0087	-	-	-
B	0.0003	0.0007	0.0005	0.0001
Mn	1.39	1.56	1.83	1.78
Cu	0.06	0.11	0.09	-
Nb	0.014	-	-	-
Ti	0.033	-	-	-
Si	0.64	0.55	0.45	0.53
Co	-	0.096	0.04	0.009
As	-	-	-	0.005
Sn	-	-	-	0.01
V	-	-	-	0.027
Fe	balance	balance	balance	balance

Figure 2 Schematic Diagram of Micro-Peening Unit

Peening affects the cladding to a depth of approximately 0.001 inch and increases the outside diameter by between 0.0001 and 0.0003 inch.

The short-time mechanical properties of the micro-peened tubing are shown in Table 3. The ultimate tensile strength is increased between 3.5 and 6%, while the yield strength is increased between 13 and 39%. The yield strength values as a function of test temperature are shown in Figure 3. The highest yield strengths are obtained from longer peening time. In view of the significant improvement in yield strength due to micro-peening, a more detailed evaluation of this type of processing is planned the next quarter.

Planetary Swaging

A preliminary literature review revealed that thin-walled austenitic stainless steel tubes, cold worked by a process called planetary swaging, developed at AERE, Harwell[3,4,5] and also under development in the U.S.[6,7,8], have exhibited hoop ductilities and hoop strengths greater than drawn tubes.

Internal pressure burst tests of 20 wt% Cr - 25 wt% Ni - 1 wt% Nb steel tubes having a 0.5-inch O.D. x 0.030-inch wall, were carried out at temperatures up to 1460°F. At 1100°F, cold-drawn tubes solution heat treated at 1920°F, possessed hoop ductilities of 10 to 25%. Tubes processed by planetary swaging followed by solution heat treatment at 1920°F showed hoop ductilities of 40 to 65%.

This improvement in high-temperature burst properties of the tubing after planetary swaging could be due to:

- a. an extremely good surface finish (4 to 8 micro-inch range) resulting in a reduction of notch effects;
- b. accurate and uniform dimensional tolerances (+0.0005-inch on diameter and +0.0003-inch on wall) resulting in a reduction of stress variation due to changes in section; and
- c. very fine grains at the inside and outside surfaces of the tube (1/4 of the wall thickness) and larger grains in the center of the wall, resulting in a change of failure mode.

It has been suggested that the bands of very fine grains at the inside and outside surfaces of the tubes are responsible for the greater ductility by inhibiting the initiation of failures in these regions.

In view of the improvement in ductility after planetary swaging, this effect will be studied in greater detail; therefore, facilities for planetary swaging are now being investigated.

The planetary swaging studies will be carried out on vacuum-melted bar stock from Carpenter Steel Company. This will be processed in the following manner:

Figure 3 Effect of Wall Thickness, Micro-Peening, and Temperature on the Yield Strength of 0.30-in. O.D., Type 316 Stainless Steel Tubing

Table 6

Metallurgical Characteristics of Type 316 Stainless Steel Fuel Element Cladding

LOT #	Wall Thickness (in.)	Grain Size ASTM	Linear Intercept (in.)	Average Inclusion Count: D TYPE (ASTM)	Microhardness Values Average	(DPN) Range	Annealing Twin-Frequency Count, (twins/grain)
816727	0.010	7/8	0.00082	1.6 Thin	168	150-192	0.22
816726	0.012	7/8	0.00084	1.1 Thin	176	153-224	0.29
7-2489	0.012	7/8	0.00082	1.5 Thin	163	153-171	0.28
816728	0.014	7/8	0.00012	1.2 Thin	165	154-176	0.31

It has not yet been established at this stage whether the bulk of the testing will be carried out on annealed or cold-worked tubing. A decision will be made in January, 1969. Using the above tubing, the effects of conventional processing versus planetary swaging, and the fabrication practices of Superior Tube Company versus those of Carpenter Steel Company will be established.

Optical Metallography

Detailed optical metallographic examination of specimens from each lot of Westinghouse air-melted tubing has been completed, and the results are given in Table 6.

The grain sizes were determined initially by visual comparison with ASTM grain size standards. Later, during the twin-frequency counting by the linear-intercept method, estimates were obtained of the average grain diameter. The two methods gave comparable results, as an ASTM rating of eight indicates an average grain diameter of 0.001 inch.

The inclusion counts were determined in accordance with ASTM 45 to 63 Method A, modified for thin-wall tubing. The inclusions were mostly D type (thin), which are globular oxide type. There were also some angular yellow-pink colored inclusions associated with the D-type inclusions. These were presumed to be titanium carbo-nitride inclusions, and this was later confirmed by electron-diffraction determinations.

The microhardness determinations were made with a Tukon Model MO microhardness tester, with a 100 gram load. The average DPN values are quite comparable; but, the range of values recorded varies with the tube lot. Tube lots #7-2489 (0.012-inch wall) and 316728 (0.014-inch wall) have a narrow range of DPN values about the mean value; lot #316727 (0.010-inch wall) has a slightly wider range, and lot #316726 (0.012-inch wall) shows a considerable variation in microhardness values. The distribution of the DPN values about the mean value for each tube lot is given in histogram form in Figure 4.

The annealing twin-frequency counts were determined by the linear-intercept method over a minimum of 150 grains and were expressed in the form of twins per grain. The counts recorded, of the order of 0.2 to 0.3 twins per grain, reflect the dependency of twin frequency on grain size, among other factors.

Figure 4 Microhardness Variations of Austenitic Stainless Steel Fuel Element Cladding

This has been discussed by Silcock et al^[9] (on austenitic stainless steels) and Charnock and Nutting^[10] (on α - brass and plain carbon steels). The latter conclude that there is a critical grain size below which there is a grain-size dependence of annealing twin frequency; above it, there is little or no dependence on grain size. For undeformed plain carbon steels, this critical size is about 0.004 inch (ASTM grain size 4). Results obtained by Silcock et al^[9] on various austenitic stainless steels tend to support such an interpretation, with critical grain size at about 0.006 inch. Unfortunately, none of these steels had compositions similar to those in the present study, making comparison with the present results not fully justified, since chemical composition affects the annealing twin frequency.^[11,12] However, extrapolation of the available data indicates general agreement with the twin frequencies observed in the present study.

Optical metallographic examination of the fracture regions of tubing that had undergone stress-rupture testing at 1200°F has been initiated. Preliminary observations indicate that the cracking is all intergranular. Air-melted Type 316 stainless steel tubing (Heat #20276, Lot #H216), 0.250-inch O.D. x 0.016-inch wall, was obtained from PNL and is being subjected to metallurgical characterization and sensitization studies.

SSAA-310 - ELECTRON MICROSCOPIC ANALYSIS OF AUSTENITIC STAINLESS STEEL CLADDING

W. K. Appleby, K. C. Thomas, R. Stickler*, and B. Weiss*

OBJECTIVES

The objectives of this task are to augment the level of understanding of microstructure on deformation mechanisms of Type 316 stainless steel cladding over that obtained in Task SSAA-210. This program will provide information on sub-structural features such as dislocation network density, dislocation distribution, and subgrain size. Also, the cladding will be examined by Laue back-reflection techniques to determine whether any preferred orientation effects, such as preferential precipitation of second phases, occur. These features will be correlated with the test variables described in Task SSAA-210 and will provide a more fundamental basis for examining the influence of microstructure on the mechanical properties of Type 316 stainless steel cladding.

PRIOR ACCOMPLISHMENTS

Substructural characterization of commercial grade, air-melted, solution-annealed Type 316 stainless steel tubing was described. This characterization included grain size, precipitation and inclusion distribution, x-ray diffraction studies of the precipitates, transmission electron microscopic evaluation of the dislocation structure, and electron beam microanalysis of the precipitated particles. The results indicated that the tubing has a higher dislocation density than expected for solution-annealed material, indicating that the tubes were subjected to slight deformation after the annealing treatment. Also, the outer surfaces of these tubes are heavily cold worked up to a depth of 0.001 inch due to a final surface grinding operation.

CURRENT PROGRESS

Examination of Stress-Rupture Specimen (Westinghouse Air Melt - Tube Lot #7-2489)

The tube failed by explosive rupture. After the first crack penetrated the tube wall, other cracks formed and produced catastrophic failure. The original crack is believed to be intergranular since metallographic examination of regions near the fracture show intergranular cracks. The secondary cracks are probably ductile and transgranular.

*Westinghouse R & D Center

Electron microscope examinations of the replicas have revealed only dimpled fracture surfaces, features which indicate failure by ductile tearing.

Electron micrographs of the extraction replicas showed fine precipitates associated with grain boundaries; occasionally bulky particles could be observed in random distribution. The fine precipitate could be identified by electron diffraction as $M_{23}C_6$, the bulky particles very probably consist of $Ti(N,C)$.

Determination of Thickness of Deformed Surface Layers in Shot-Peened Tubing (Tubes A and B)

The surface roughness of the microshot-peened tube samples was determined by a focusing method with the light microscope. Tubes A and B were identical in appearance. The surface roughness of both tubes was 3 to 4 μm .

To determine the thickness of the deformed surface layer, the tube samples were examined by a method described in the previous quarterly report (WARD-3791-26). The outer surfaces of the tubes were consecutively electropolished to remove approximately 5 μm per polishing step. X-ray Laue back-reflection patterns were recorded after each polishing step, and the procedure continued until the x-ray pattern indicated that all strained material had been removed. The thickness of material that must be removed to expose strain-free material, determined by micrometer measurements, corresponds approximately to the thickness of the deformed surface layer. Approximately 25 μm had to be removed from Sample A, while 33 μm had to be removed from Sample B. These values should be compared with a value of less than 25 μm for the thickness of deformed material in the ground tube sample reported in Progress Report No. 1.

Sensitization Studies (Westinghouse and PNL Air Melt - Tube Lots #316727, 7-2489, and H-216)

To avoid any oxidation effects during long-time, high-temperature aging of the thin-walled tubes, 2-inch long specimens were cut from the as-received tubes and sealed in quartz vials (in vacuum greater than 10^{-5} Torr). The vials are now being heat treated at temperatures ranging from 750 to 1650°F for times up to 5000 hours. Only specimens of Tube Lot #316727 will be aged over the entire temperature-time interval, while for comparison, initially only a few specimens of the other tube lots will be aged at critical T-t values. Some of the short-time heat-treated specimens are expected to be available for electron microscope, electron diffraction, and light microscope examination in late January.

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