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Quarterly Report on the Strontium Heat Source Development Program, Advanced Systems and Materials Production Division for October-December 1978

H. T. Fullam

January 1979

Prepared for the U.S. Department of Energy
under Contract EY-76-C-06-1830

Pacific Northwest Laboratory
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QUARTERLY REPORT ON THE STRONTIUM HEAT SOURCE DEVELOPMENT PROGRAM, ADVANCED SYSTEMS AND MATERIALS PRODUCTION DIVISION FOR OCTOBER-DECEMBER 1978

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Pacific Northwest Laboratory
Richland, Washington 99352

SUMMARY

The 20,000-hr compatibility tests using WESF $^{90}SrF_2$ have been completed. The test capsules were shipped to Oak Ridge National Laboratory (ORNL) and metallographic examination of the test specimens is now underway.

Work continues on the final design of the prototype outer capsule. Efforts are underway to obtain the Hastelloy S and Hastelloy C-4 to be used in fabricating the prototype outer capsules. Puncture testing of the stand-in steel outer capsule was carried out without damage to the capsule. Effort continued on impact testing of the stand-in capsule.

Tests to measure the oxidation rates of Hastelloy S and Hastelloy C-4 in air at 600° to 800°C are continuing. Short-term test data for up to 2500-hr exposure indicated that oxidation of the two alloys adhered to parabolic rate relationships. Specimens oxidized for 5000 hr and 7500 hr exhibited erratic oxidation behavior, and the average oxidation rates were less than predicted by parabolic rate equations developed from the short-term rate data. Room-temperature tension testing of specimens oxidized in air at 600° to 800°C for up to 7500 hr indicated that oxidation had no significant effect upon the room-temperature tensile properties of the two alloys.

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STRONTIUM HEAT SOURCE DEVELOPMENT PROGRAM

H. H. Van Tuyl, Program Manager
H. T. Fullam, Principal Investigator
D. G. Atteridge
F. A. Simonen

At Hanford, strontium is separated from the high-level waste, converted to the fluoride, and doubly encapsulated in small, high-integrity containers for subsequent long-term storage. The fluoride conversion, encapsulation, and storage take place in the Waste Encapsulation and Storage Facilities (WESF). The encapsulated strontium fluoride represents an economical source of ^{90}Sr if the WESF capsule can be licensed for heat source applications under anticipated use conditions. The objectives of this program are to obtain the data needed to license $^{90}SrF_2$ heat sources, and specifically the WESF $^{90}SrF_2$ capsules. The information needed for licensing can be divided into three general task areas:

Task 1 - Chemical and Physical Properties of $^{90}SrF_2$

Task 2 - $^{90}SrF_2$ Compatibility Studies

Task 3 - Capsule Qualification and Licensing

Efforts are proceeding concurrently on all three tasks to obtain the required information.

TASK 1 - CHEMICAL AND PHYSICAL PROPERTIES OF $^{90}SrF_2$ (H. T. Fullam)

No activity this quarter.

TASK 2 - $^{90}SrF_2$ COMPATIBILITY STUDIES (H. T. Fullam)

All of the remaining compatibility tests are proceeding as scheduled. The capsules from the 20,000-hr tests with $^{90}SrF_2$ were shipped to the Oak Ridge National Laboratory (ORNL) for analysis. The capsules have been sectioned, and metallographic examination of the test specimens is underway.

Supplemental compatibility tests with nonradioactive SrF_2 are underway using test capsules having a metal surface-to-SrF_2 volume ratio (S/V) of 0.9 cm^{-1} (the WESF $^{90}SrF_2$ storage capsule has an S/V of 0.87 cm^{-1}). The SrF_2 used in the tests contains impurities similar to those found in WESF $^{90}SrF_2$. The 12,000-hr tests have been completed, and the metal specimens examined. The results obtained are presented in Table 1, and data from the 1000- and 6000-hr tests are also included for comparison purposes. Photomicrographs of the test specimens are shown in Figures 1, 2 and 3. Evaluation of the test results show that, overall, attack of the specimens did not increase significantly as the exposure time was increased from 6000 to 12,000 hours. This confirms the results obtained with nonradioactive SrF_2 test capsules having larger S/V values (i.e., 2.5, 4.5 cm^{-1}). The results provide additional evidence that metal attack is due primarily to impurities in the SrF_2 and that the impurities are consumed in a relatively short period of time.

TABLE 1. Estimated Attack of Metal Specimens Exposed to Nonradioactive SrF_2[(a)] - Capsule S/V = 0.9 cm^{-1}

Material	Temperature, °C	Depth of Metal Affected, mils[(b)]					
		Chemical Attack			Change in Microstructure		
		1,000 hr	6,000 hr	12,000 hr	1,000 hr	6,000 hr	12,000 hr
Hastelloy C-276	600	1	6	3	4	6	6
	800	10	10	7	15	15	8
	1000	4	8	7	6	12	12
Haynes Alloy 25	600	1	3	2	2	3	0
	800	1	5	4	2	17	5
	1000	3	12	14	2	15	16
TZM	600	1	2	2	0	0	0
	800	1	4	2	0	0	0
	1000	1	1	1	0	0	2

(a) SrF_2 contained impurities approximating those found in WESF $^{90}SrF_2$.
(b) Estimated from photomicrographs.

TASK 3 - CAPSULE QUALIFICATION AND LICENSING (D. G. Atteridge)

Capsule Design

Evaluation of the proposed outer capsule design continued during this quarter. Work was concentrated on impact and puncture testing of the stand-in AISI-1018 steel capsules. The puncture testing was completed this quarter, along with the evaluation of the drop test capsule alignment and release mechanism and the photographic drop event recording equipment.

The 0.5-in. wall thickness stand-in steel capsule used in the puncture testing was the same capsule that withstood the 1000-bar hydrostatic test reported on in the previous quarterly report. The capsule was puncture tested at the three locations shown in Figure 4: at the middle of the capsule, at the inner edge of the capsule lid, and at the inner edge of the weld. The three puncture drops left slight indentations on the capsule surface but did not puncture the capsules, as was evident from visual inspection and substantiated by subsequent helium leak checking.

Two sets of capsule drops were made from our 30-foot drop tower this quarter in order to evaluate different photographic equipment. We desired multiple pictures of the last eight feet of the drop with sufficient resolution capability to allow measurement of the off-vertical capsule axis angle at impact. A high-speed 35 mm camera was used to record one set of test drops, while a high-speed 16 mm movie camera was used to record the second set of tests. It was found that the 35 mm camera gave sharp photographs, as shown in Figure 5, but was not fast enough to assure capture of more than one photograph during the last eight feet of the drop. The movie camera was able to capture multiple photographs of the drop event but gave relatively poor capsule resolution when individual frames were blown up to 8-in. x 10-in. prints. Nevertheless, it is felt that the 16 mm film yields

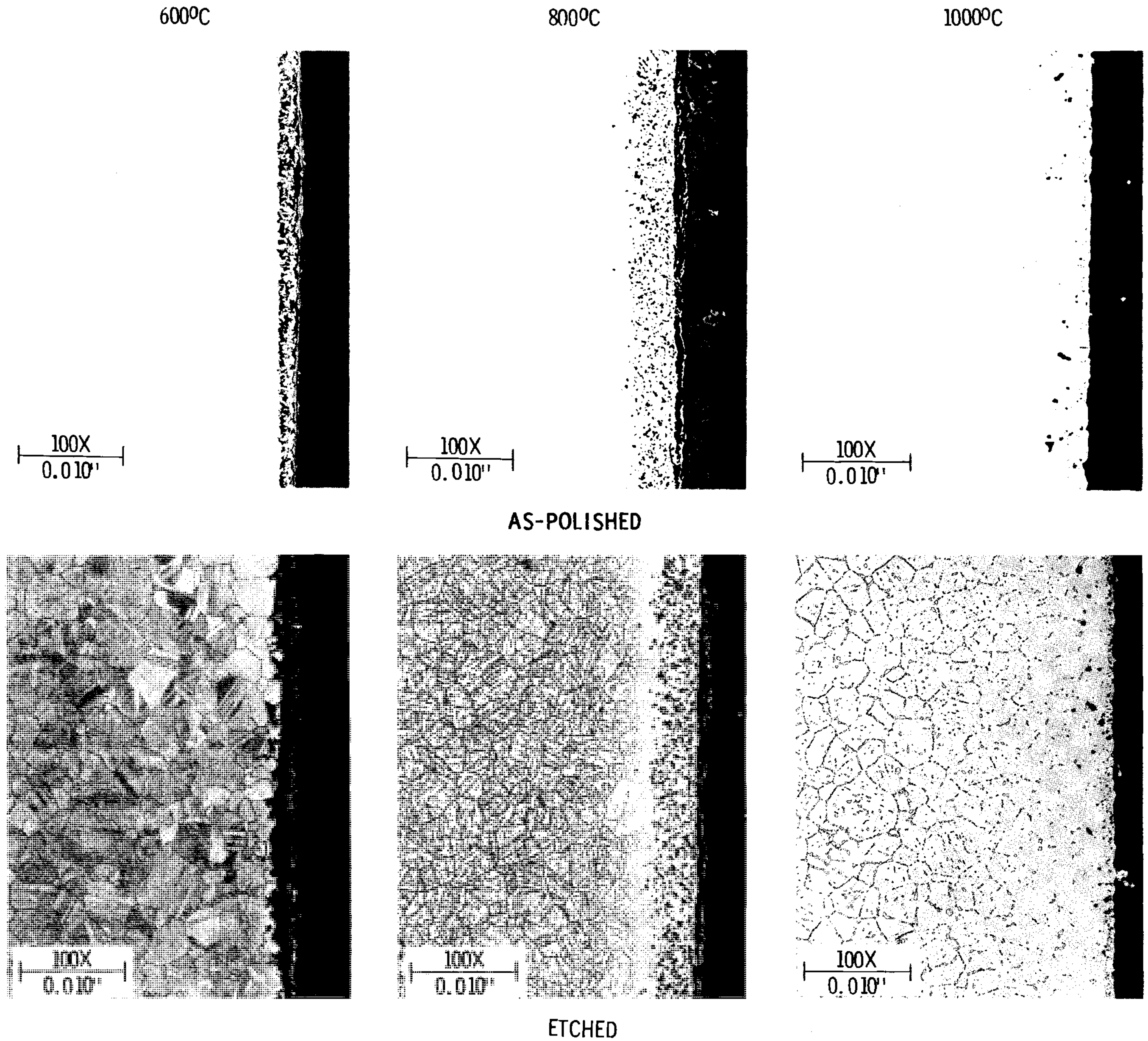

FIGURE 1. Hastelloy C-276 Specimens Exposed to Nonradioactive SrF_2 for 12,000 hr - Capsule S/V = 0.9 cm^{-1}

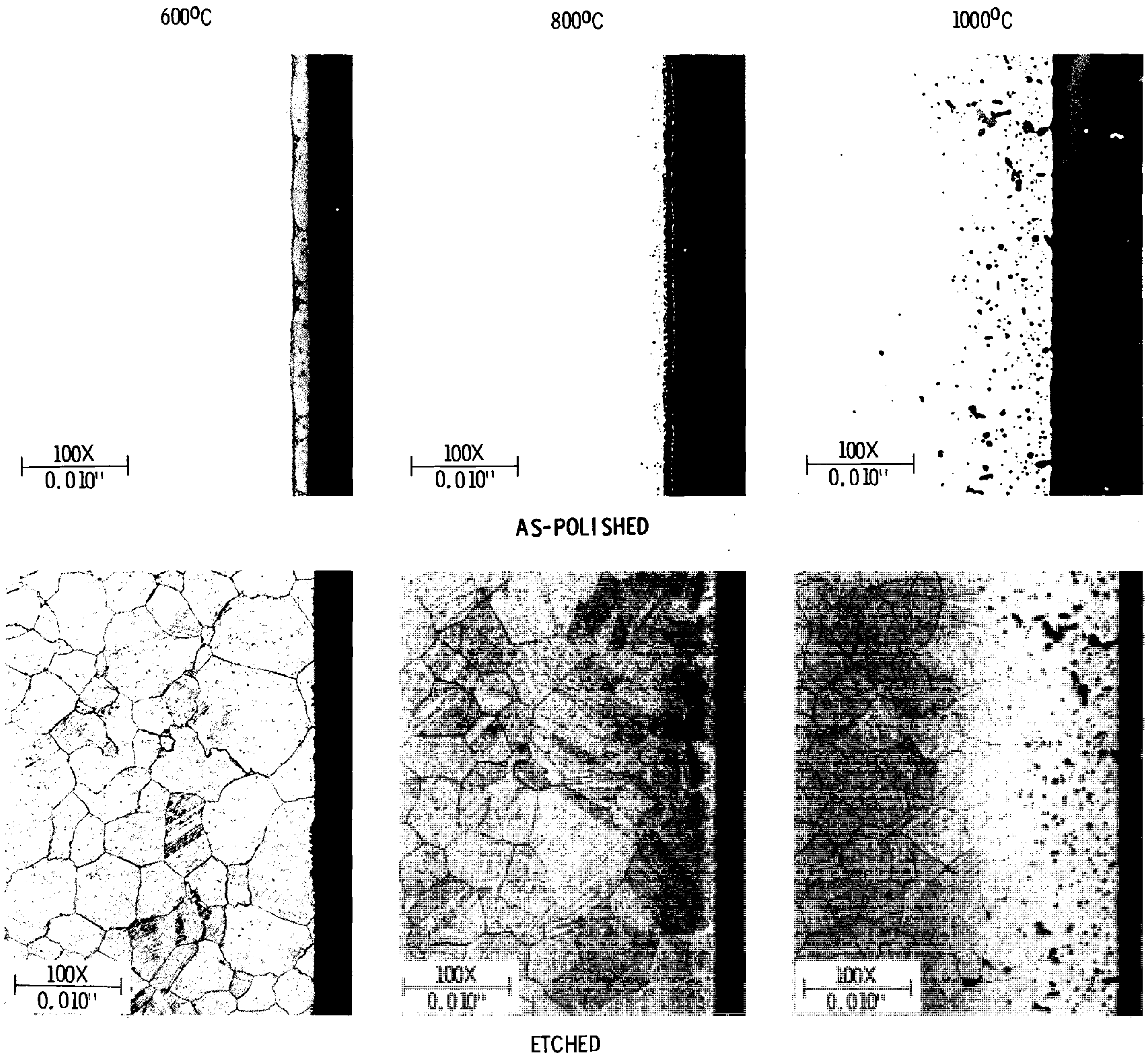

FIGURE 2. Haynes Alloy 25 Specimens Exposed to Nonradioactive SrF_2 for 12,000 hr - Capsule S/V = 0.9 cm^{-1}

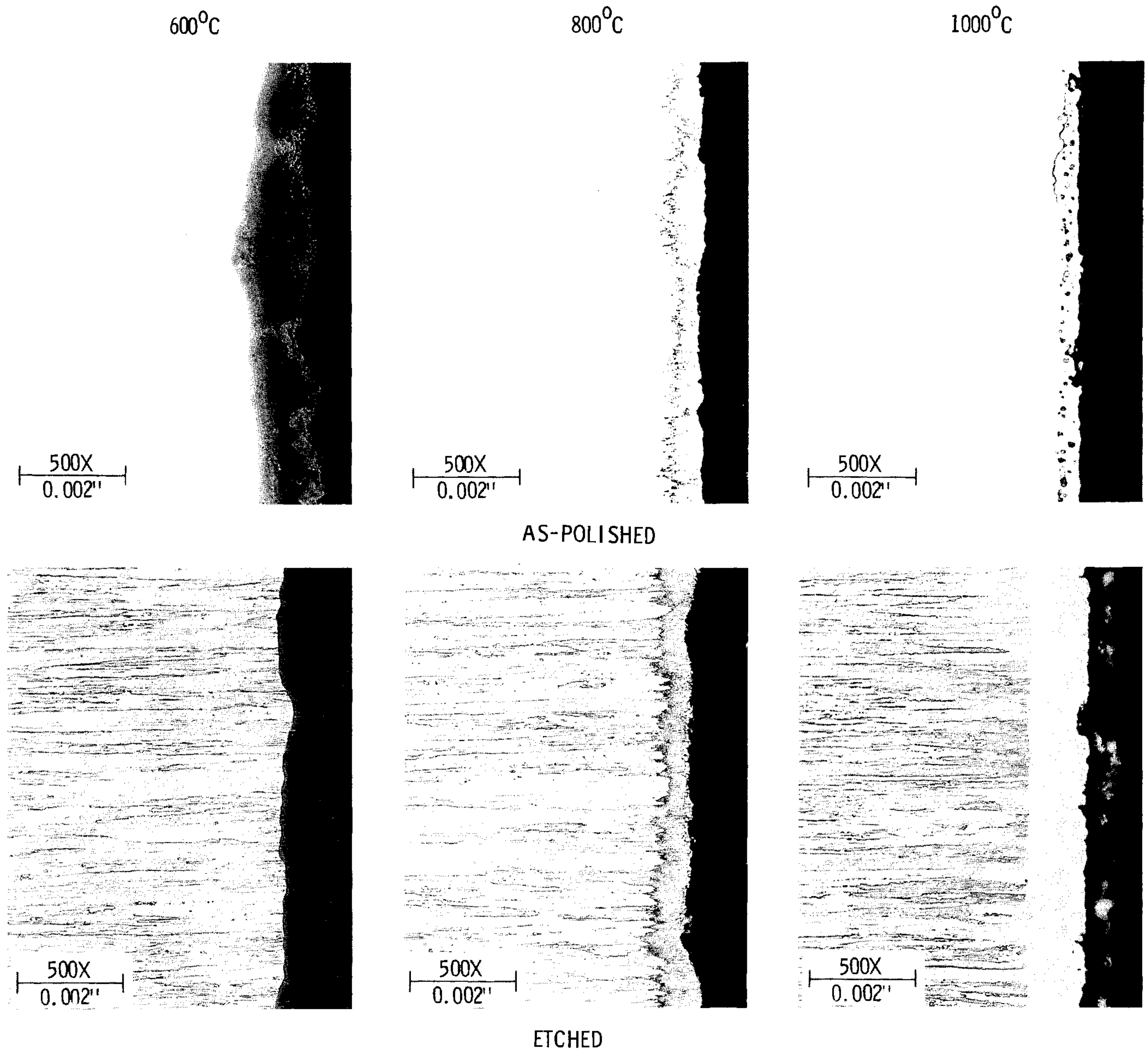

FIGURE 3. TZM Specimens Exposed to Nonradioactive SrF_2 for 12,000 hr - Capsule S/V = 0.9 cm^{-1}

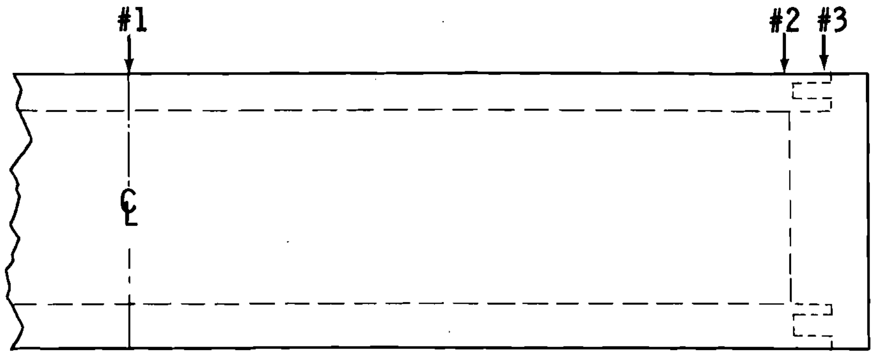

FIGURE 4. Sketch Showing Locations of the Three Puncture Tests on the Steel Stand-In Capsule

sufficient resolution to allow angle-of-strike measurements, and thus will be used for recording subsequent capsule drops. It was also determined that the capsule drop and release mechanism is functioning correctly and will require only slight modification to be ready to use in the upcoming stand-in capsule drops.

Efforts are underway to obtain the Hastelloy S and Hastelloy C-4 to be used in fabricating the prototype outer capsule. It is anticipated that the material will be ordered in January, with a planned delivery time of 3 to 4 months.

Oxidation of Hastelloy S and Hastelloy C-4 (H. T. Fullam)

Tests are continuing to measure the rate of oxidation of Hastelloy S and Hastelloy C-4 in air at 600° to 800°C. The tests will last up to 10,000 hours. The 7500-hr tests have been completed, and examination of the test coupons indicated erratic oxidation behavior of the two alloys. A number of specimens tested for 7500 hr exhibited less weight gain due to oxidation than did corresponding specimens tested for shorter times. None of the coupons showed any evidence of spalling of the oxide layer, and the reason(s) for the erratic behavior has not been determined. Results from the short-term tests (up to 2500 hr) showed that oxidation of the two alloys at 600° to 800°C adhered to parabolic rate relationships; but the longer-term oxidation rates were less than predicted by parabolic rate equations developed from the short-term data. This can be seen by referring to Figures 6 and 7, in which the oxidation data for the two alloys are plotted as Δm^2 (mg^2/cm^4) vs. exposure time in hours. A linear relationship indicates adherence to a parabolic rate relationship. Although the weight gain data indicated erratic oxidation behavior, metallographic examination of the 7500-hr test specimens showed that the oxidation mechanisms were similar to those observed in the short-term tests. Micrographs of specimens from the 7500-hr tests are shown in Figures 8 and 9.

The tensile specimens from the 5000- and 7500-hr oxidation tests were tension tested at room temperature. Results obtained with the Hastelloy S specimens are presented in Table 2 and Figures 10, 11 and 12, while the Hastelloy C-4 results are given in Table 3 and Figures 13, 14 and 15. Evaluation of the test results indicates that oxidation of the two alloys at 600°C to 800°C, for up to 7500 hr, has no significant effect on their room-temperature tensile properties.

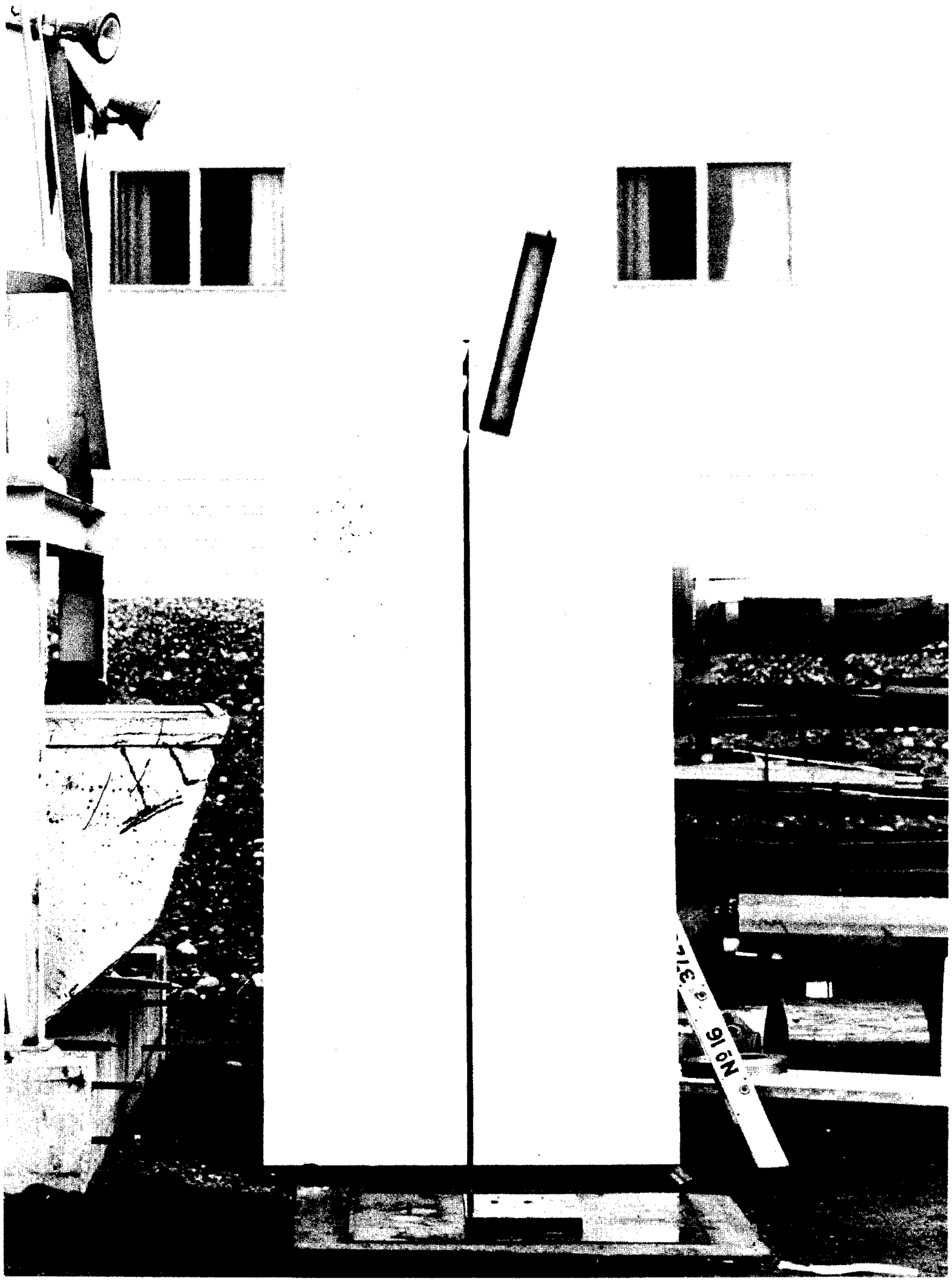

FIGURE 5. Photograph of a 30-Foot Drop Test of an AISI-1018 Steel Capsule

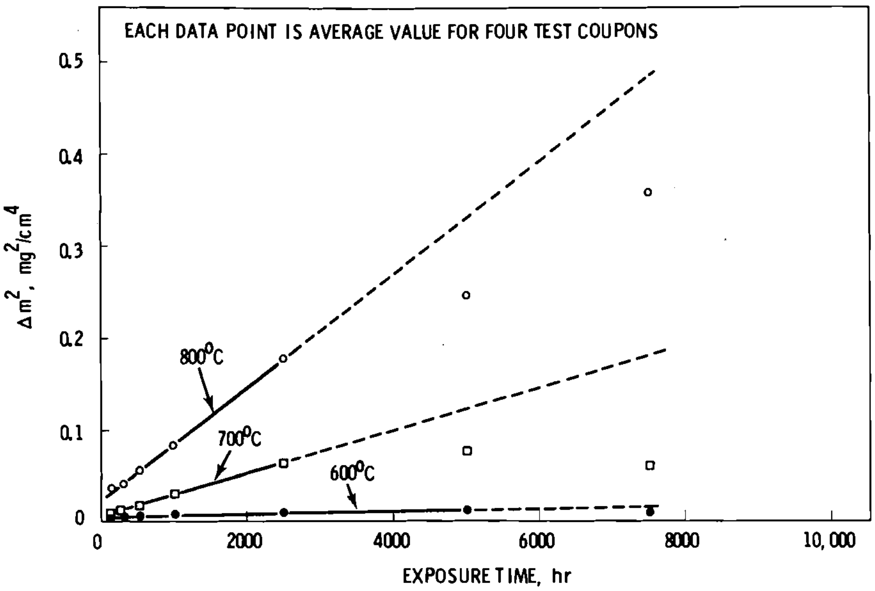

FIGURE 6. Oxidation of Hastelloy S in Air in a Muffle Furnace

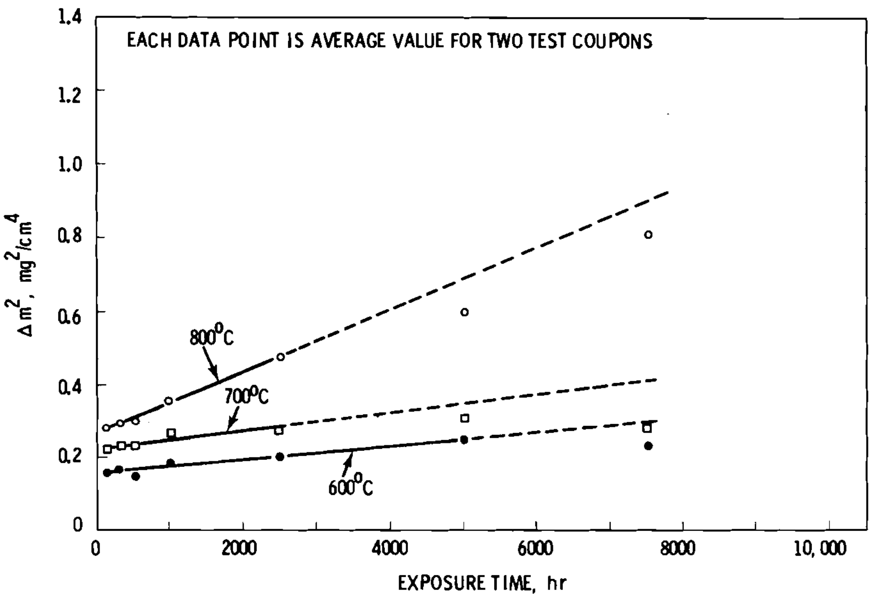

FIGURE 7. Oxidation of Hastelloy C-4 in Air in a Muffle Furnace

Seawater Corrosion of Hastelloy S and Hastelloy C-4 (H. T. Fullam)

The tests to evaluate the resistance of Hastelloy S and Hastelloy C-4 to seawater corrosion are continuing. The 5000-hr tests have been completed, and the test coupons examined. All of the test specimens exhibited some surface discoloration, as shown in Figure 16, but as can be seen in Figures 17 and 18, weight change measurements continue to

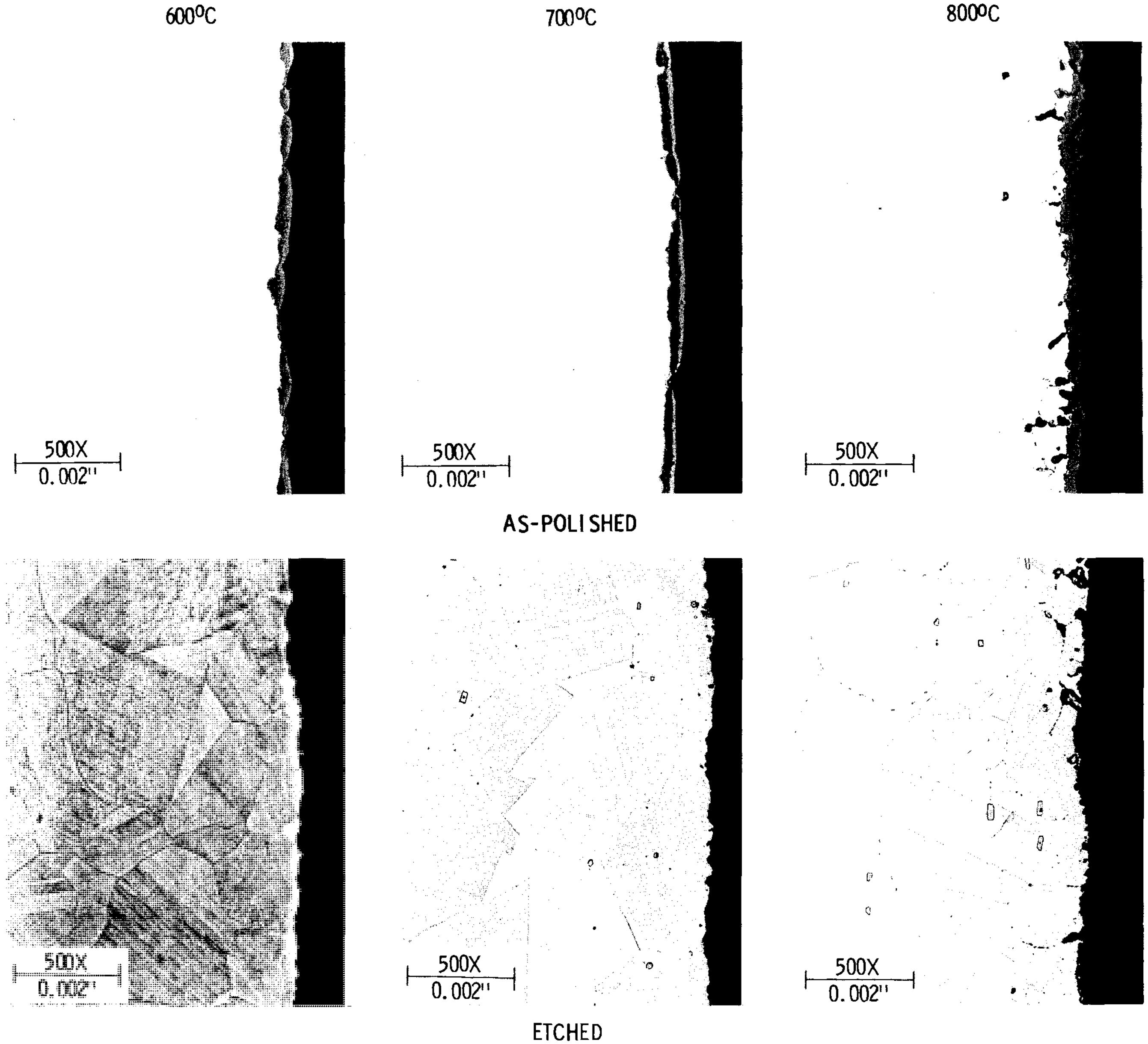

FIGURE 8. Hastelloy S Specimens Oxidized in Air for 7500 hr

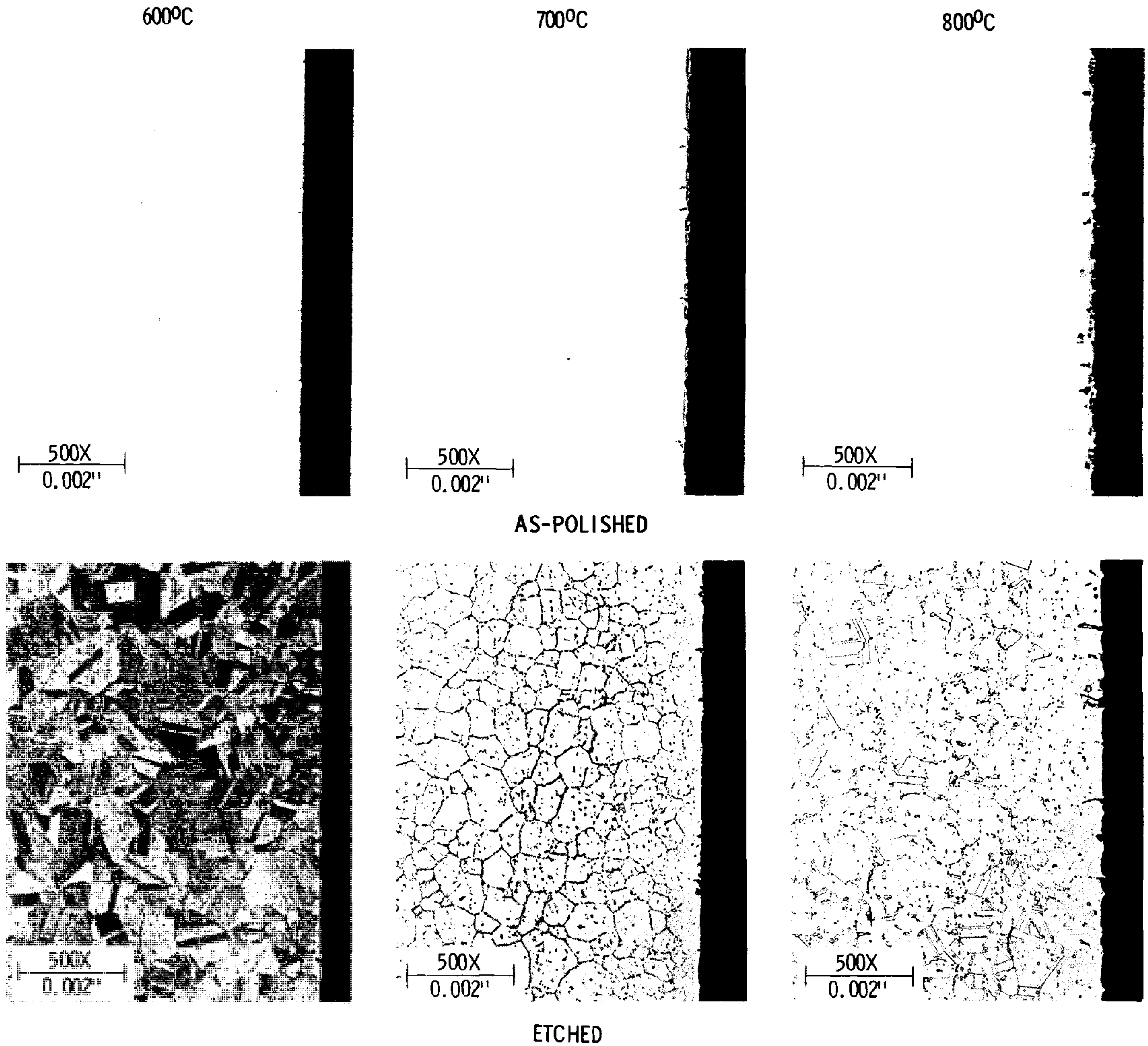

FIGURE 9. Hastelloy C-4 Oxidized in Air in a Muffle Furnace

TABLE 2. Room-Temperature Tensile Properties of Hastelloy S Specimens[(a)] Aged in Air or Vacuum[(b)] at 600°C, 700°C, and 800°C

Aging Temp., °C	Aging Time, hr	Ultimate Tensile Strength, ksi		Yield Strength, ksi		Elongation, %			
						Uniform		Total	
		Vacuum	Air	Vacuum	Air	Vacuum	Air	Vacuum	Air
(Sol'n Annealed Alloy)[(c)]	0	(127.6)		(67.8)		(51.5)		(60.8)	
600	1,000	136.0	137.9	73.1	78.4	50.4	50.8	56.0	56.1
600	2,500	147.8	151.2	89.5	86.7	44.3	44.4	48.8	48.3
600	5,000	181.6	170.5	111.9	108.5	33.6	36.5	37.1	41.4
600	7,500	171.5	169.1	104.8	101.5	35.8	37.8	39.8	41.0
600	10,000								
700	1,000	131.5	127.5	67.2	64.1	53.8	55.4	60.0	61.8
700	2,500	127.8	128.9	61.5	66.3	55.5	47.9	62.6	54.1
700	5,000	134.5	132.1	69.4	68.1	47.6	48.1	53.4	53.7
700	7,500	130.5	129.8	65.7	62.9	50.6	50.4	57.2	57.0
700	10,000								
800	1,000	132.3	127.7	63.3	61.4	53.2	54.9	59.5	63.6
800	2,500	127.2	126.2	59.1	59.1	54.9	54.4	62.7	62.1
800	5,000	130.8	128.4	60.9	61.2	50.9	49.8	59.1	58.2
800	7,500	134.1	125.3	66.3	57.2	47.7	54.0	53.2	62.0
800	10,000								

(a) Four tensile specimens heated in air and 2 tensile specimens heated in vacuum for each set of test conditions.
(b) Tensile specimens heated in evacuated quartz envelopes in the same furnaces as the specimens heated in air.
(c) Solution annealed alloy as received from the vendor.

TABLE 3. Room-Temperture Tensile Properties of Hastelloy C-4 Specimens[(a)] Aged in Air or Vacuum[(b)] at 600°C, 700°C, and 800°C

Aging Temp., °C	Aging Time, hr	Ultimate Tensile Strength, ksi		Yield Strength, ksi		Elongation, %			
						Uniform		Total	
		Vacuum	Air	Vacuum	Air	Vacuum	Air	Vacuum	Air
(Sol'n Annealed Alloy)[(c)]		(121.0)		(55.0)		(60.9)		(71.5)	
600	2,500	182.3	179.3	103.6	99.1	38.7	42.0	43.6	48.6
600	5,000	179.9	180.3	102.1	106.7	39.3	41.2	46.5	46.2
600	7,500	178.3	178.4	104.3	101.6	41.2	39.5	47.4	45.1
600	10,000								
700	2,500	152.9	119.0	60.8	54.0	55.3	63.9	61.6	64.1
700	5,000	123.2	122.2	55.7	55.9	58.7	63.3	65.6	72.7
700	7,500	125.7	126.7	58.0	57.5	58.9	58.3	66.3	66.2
700	10,000								
800	2,500	115.2	116.1	48.5	49.7	63.8	63.2	68.1	72.3
800	5,000	120.2	118.6	51.9	50.4	60.1	59.6	69.5	67.4
800	7,500	120.2	119.3	49.7	50.3	62.9	60.7	71.7	68.7
800	10,000								

(a) Duplicate specimens tested at each set of conditions.
(b) Specimens heated in evacuated quartz envelopes in the same furnaces as the specimens heated in air.
(c) Solution annealed alloy as received from the vendor.

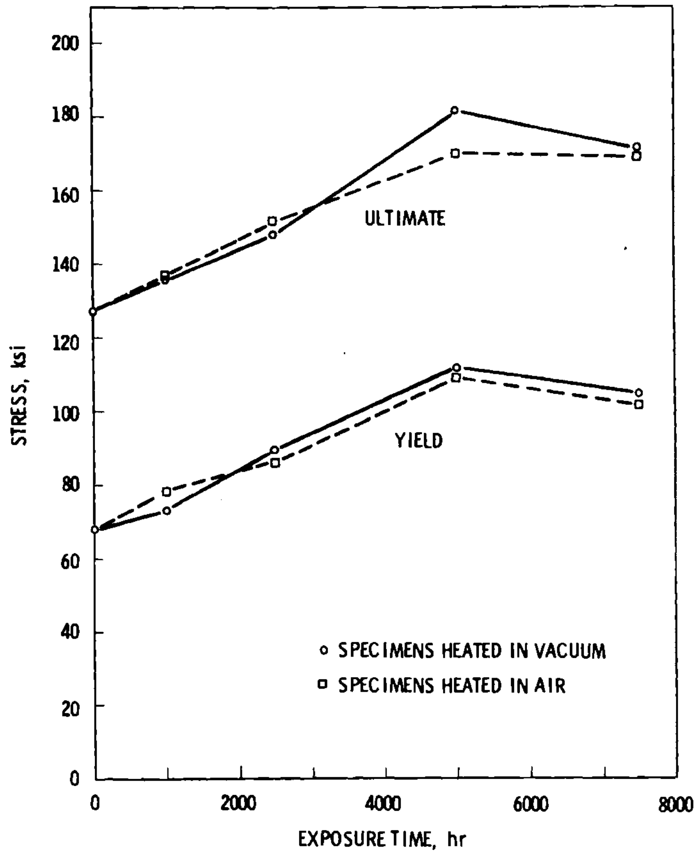

FIGURE 10. Room-Temperature Tensile Properties of Hastelloy S Aged in Air or Vaccum at 600°C

indicate only very slight corrosion. Corrosion of the Hastelloy C-4 coupons was much greater than corrosion of the Hastelloy S coupons (4 to 5X), but it was still very low. If one assumes uniform surface corrosion, the average Hastelloy C-4 specimen weight loss after 5000 hr exposure corresponds to a corrosion rate of about 0.4 μm/yr (1.5 x 10^{-5} in./yr). Since the corrosion is not uniform, the depth of metal affected by seawater attack would be greater than the calculated average corrosion rate, but should still be low. Metallographic examination of the coupons from the 5000-hr tests provided no visual evidence of corrosion of the two alloys by the seawater. Microscopic examination of prestressed specimens from the 5000-hr tests showed no evidence of stress-corrosion cracking of either alloy.

Tensile specimens from the 5000-hr tests were tension tested at room temperature. The results obtained are presented in Table 4. Evaluation of the data shows that the room-temperature tensile properties of the two alloys are unaffected by exposure to flowing seawater for times of up to 5000 hours.

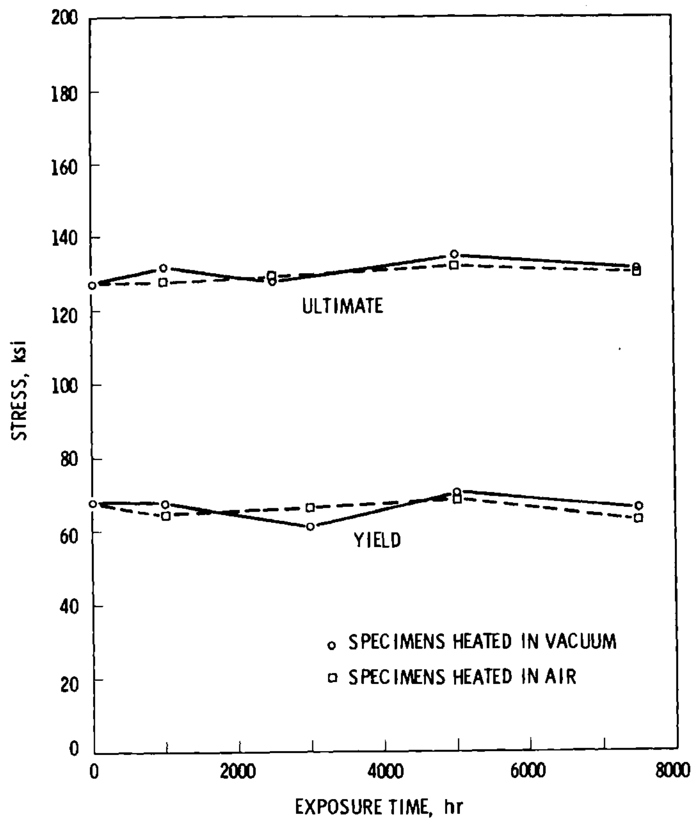

FIGURE 11. Room-Temperature Tensile Properties of Hastelloy S Aged in Air or Vacuum at 700°C

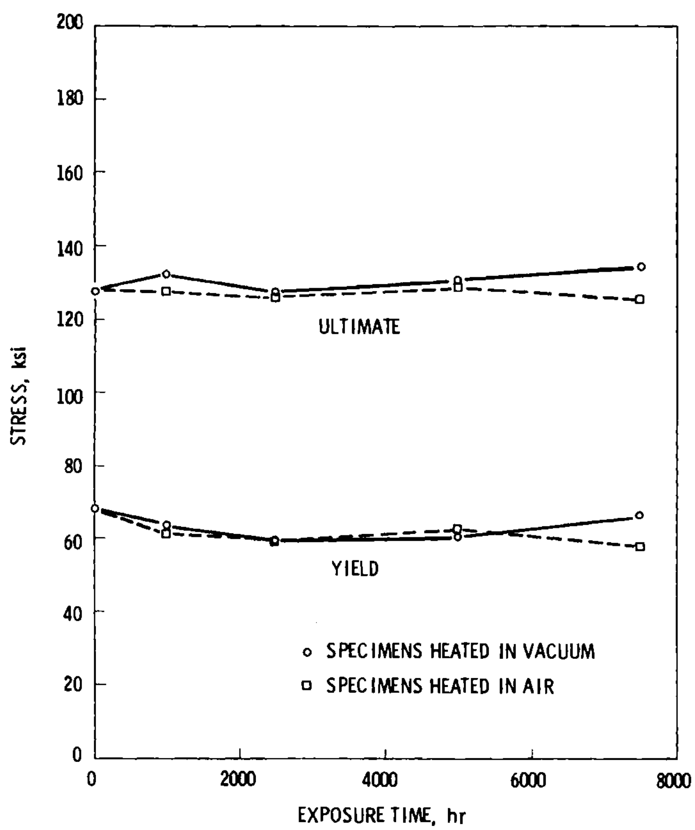

FIGURE 12. Room-Temperature Tensile Properties of Hastelloy S Aged in Air or Vacuum at 800°C

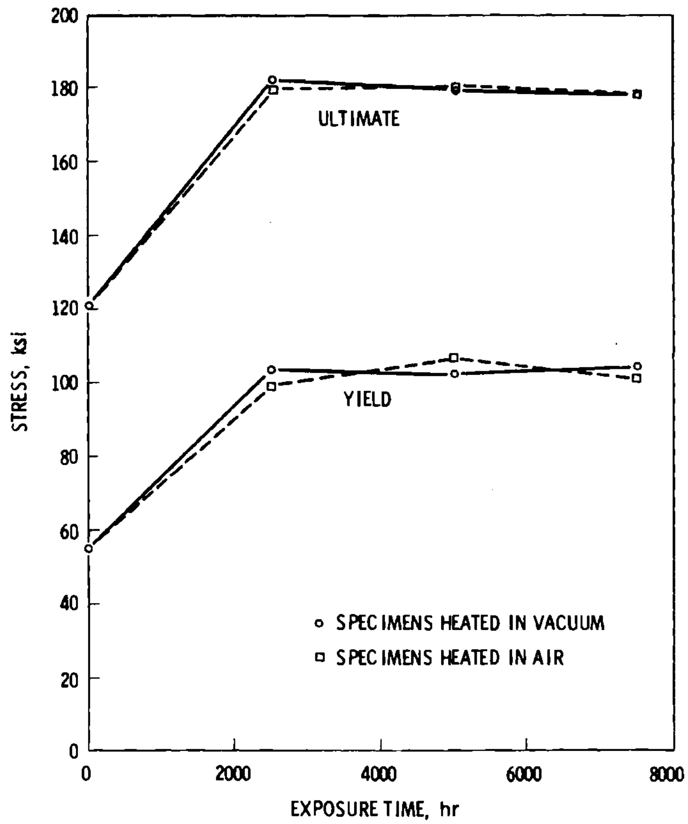

FIGURE 13. Room-Temperature Tensile Properties of Hastelloy C-4 Aged in Air or Vacuum at 600°C

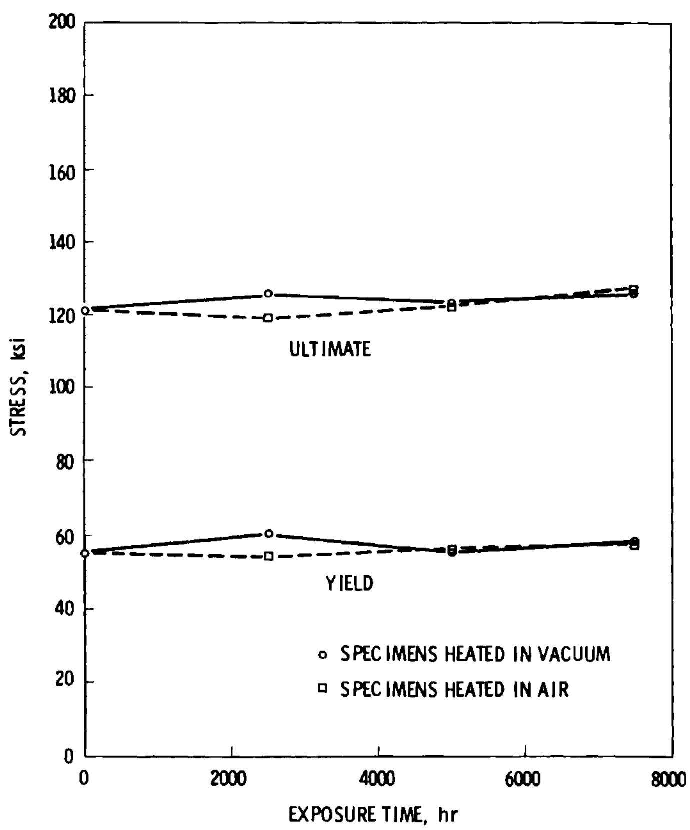

FIGURE 14. Room-Temperature Tensile Properties of Hastelloy C-4 Aged in Air or Vacuum at 700°C

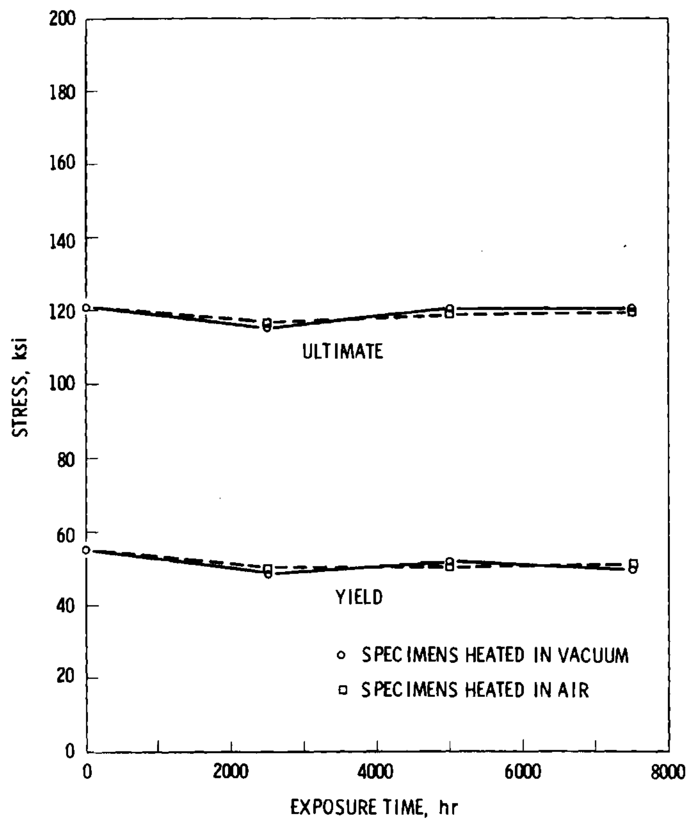

FIGURE 15. Room-Temperature Tensile Properties of Hastelloy C-4 Aged in Air or Vacuum at 800°C

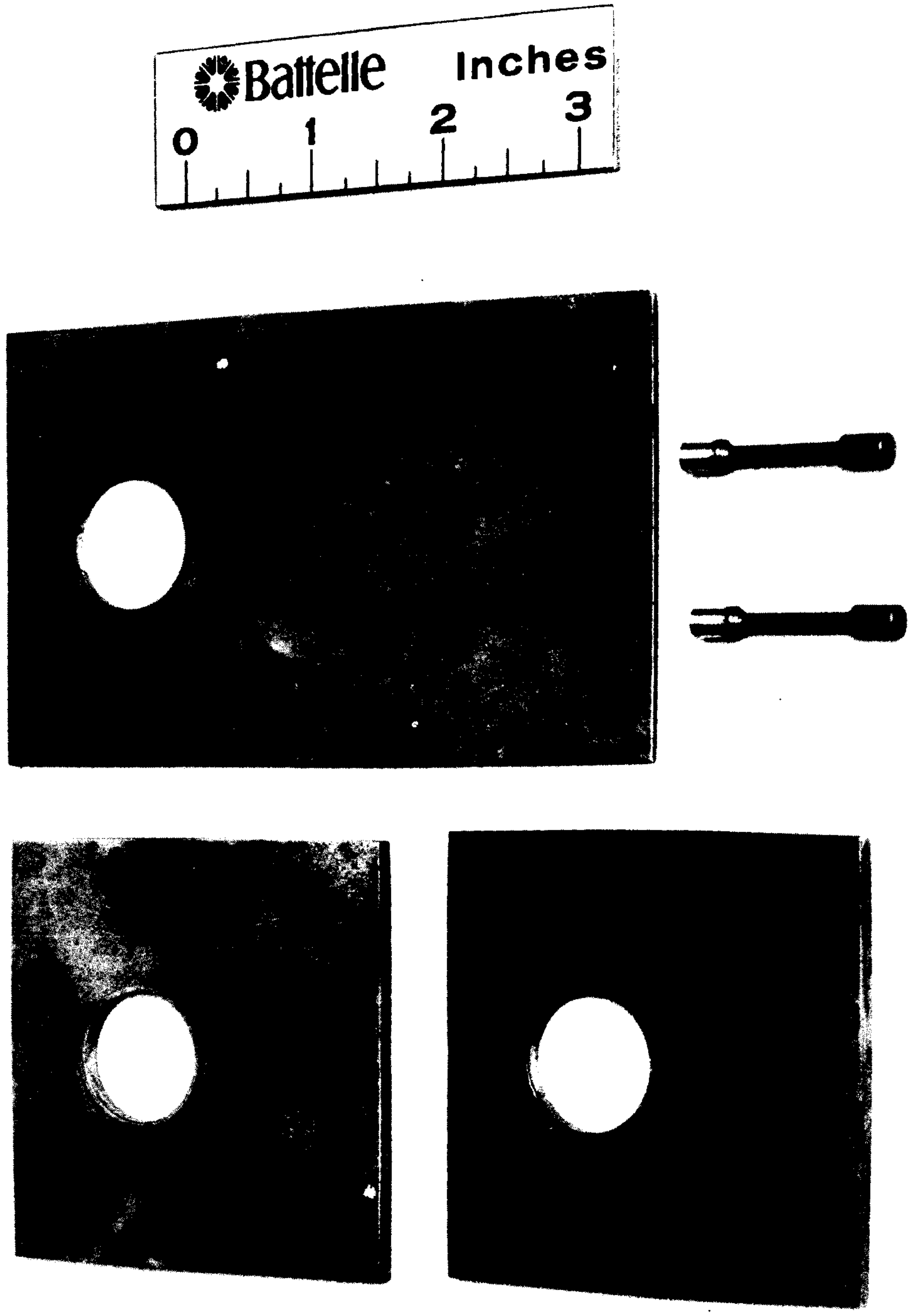

FIGURE 16. Hastelloy S and Hastelloy C-4 Test Coupons, Including Tensile Specimens, Exposed to Flowing Natural Seawater for 5000 hr

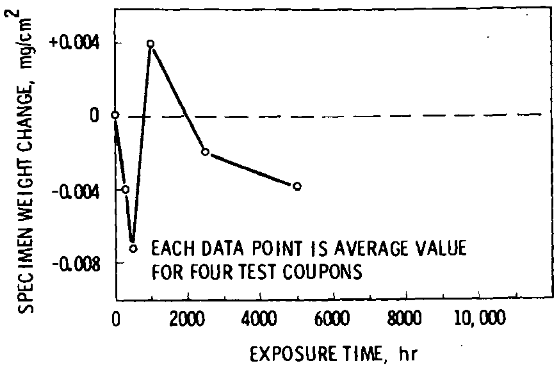

FIGURE 17. Corrosion of Hastelloy S in Flowing Natural Seawater at Ambient Temperature

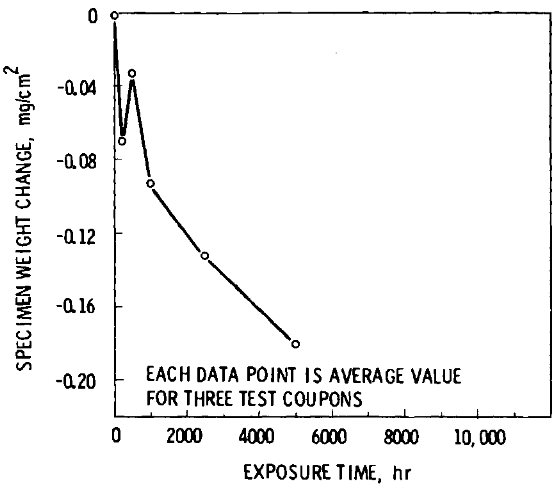

FIGURE 18. Corrosion of Hastelloy C-4 in Flowing Natural Seawater at Ambient Temperature

TABLE 4. Room-Temperature Tensile Properties of Hastelloy S and Hastelloy C-4 Specimens[(a)] Exposed to Flowing Natural Seawater at Ambient Temperature

Material	Exposure Time, hr	Ultimate Tensile Strength, ksi	Yield Strength, ksi	Elongation, %	
				Uniform	Total
Hastelloy S	(Sol'n Annealed Alloy)	(127.6)	(67.8)	(51.5)	(60.8)
	1,000	129.0	70.6	53.3	61.7
	2,500	126.5	69.5	53.2	62.1
	5,000	127.4	68.0	51.3	61.1
	7,500				
	10,000				
Hastelloy C-4	(Sol'n Annealed Alloy)[(b)]	(121.0)	(55.0)	(60.9)	(71.5)
	1,000	123.7	56.5	59.7	69.7
	2,500	120.5	53.1	58.9	69.1
	5,000	120.8	52.9	59.3	69.5
	7,500				
	10,000				

(a) Four tensile specimens of each alloy tested for each time period and average values reported.
(b) Solution annealed alloy as received from the vendor.

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J. R. Keiser
Oak Ridge National Laboratory
Oak Ridge, TN 37830

E. Lamb
Oak Ridge National Laboratory
Oak Ridge, TN 37830

C. L. Ottinger
Oak Ridge National Laboratory
Oak Ridge, TN 37830

J. C. Posey
Oak Ridge National Laboratory
Oak Ridge, TN 37830

A. C. Schaffhauser
Oak Ridge National Laboratory
Oak Ridge, TN 37830

P. Dick
Teledyne Energy Systems
110 W. Timonium Road
Timonium, MD 21093

R. Hannah
Teledyne Energy Systems
110 W. Timonium Road
Timonium, MD 21093

W. A. McDonald
Teledyne Energy Systems
110 W. Timonium Road
Timonium, MD 21093

P. Vogelberger
Teledyne Energy Systems
110 W. Timonium Road
Timonium, MD 21093

C. C. Silverstein
Westinghouse Astronuclear Laboratory
P.O. Box 10864
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