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REPORT TITLE POST-IRRADIATION EXAMINATION OF IN-PILE STRESS RUPTURE
SPECIMENS (ORR S-2 AND ORR S-3)

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NAA-SR-MEMO 12577

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I. INTRODUCTION

1. Thirty Hastelloy-N test specimens were biaxially stress-rupture-tested in-pile at the Oak Ridge National Laboratory (ORNL), as part of the SNAP cladding evaluation program. Selection of Hastelloy-N reference heats was based on the results of elevated temperature uniaxial creep-ductility testing (ORR S-1). The purpose of testing was to compare the performance of Hastelloy-N in a coated tube configuration to existing data on Hastelloy-N bars.

ORR S-2

Cladding tube specimens were fabricated of Hastelloy-N of two reference heats: 281-4-0143 and 5911. After appropriate pre-irradiation measurements were made, each tube was placed in a controlled temperature-flux radiation environment at the ORNL and stressed by internal pressure. Specimen stress levels were established, using the hoop stress relationship:

$$\sigma_H = \frac{PD}{2t}, \text{ where } \begin{array}{ll} \sigma_H & \text{is hoop stress (psi),} \\ P & \text{is hydrostatic pressure} \\ & \text{above atmosphere (psi),} \\ D & \text{is mean specimen} \\ & \text{diameter (in.),} \\ \text{and } t & \text{is average specimen} \\ & \text{wall thickness} \\ & \text{(in.).} \end{array}$$

Specimen internal pressures, operating temperatures, thermal and fast flux, and rupture times were monitored during testing. After the specimens had failed, they were shipped to the Atomics International Hot Laboratory (AIHL), for visual examination, dimensional measurement, and metallographic analysis. The dimensional measurements included specimen outer diameter profiles and wall thickness determinations.

ORR S-3

Preliminary ORR S-2 in-pile test data indicated that specimens with the higher internal pressures were failing before they had acquired the desired neutron dosage. Additional specimens (ORR S-3)

were similarly tested in-pile, at lower internal pressures. Four of the specimens were stressed to failure, four remained unstressed, and two were stressed and unfailed. The specimens were shipped to the AIHL for visual examination and non-destructive dimensional measurements. The unfailed specimens were returned to the ORNL for further stress-rupture testing; the failed specimens were examined similarly to the ORR S-2 specimens.

The scope of this report includes presentation and evaluation of the results of the in-pile testing and the post-irradiation examination.

II. SPECIMEN HISTORY

Figure 1 shows the in-pile stress-rupture specimen design used. The stainless steel tube provided for specimen internal pressurization producing the desired hoop stresses.

Tubing of heat no. 281-4-0143 had a nominal wall thickness of 10 mils, while heat no. 5911 was used in both 10 and 15 mil wall thicknesses. Tubing from heat no. 5911, originally of 15 mil wall thickness was redrawn to obtain 10 mil material. The chemical compositions of the heats before processing are shown in Table 1. The fabrication and quality assurance procedures and the inspection data for the specimens are included in References 1 and 2.

In-pile tests were assembled and tested under the guidance of Dr. H.E. McCoy of the ORNL staff. The twenty ORR S-2 specimens were tested in two irradiation capsules (ten specimens each) at the ORNL; the ten ORR S-3 specimens were similarly tested in a third capsule. Figure 2 shows a typical irradiation capsule configuration. The fatigue strip packages shown between adjacent specimens were irradiated within one capsule only. Three thermocouples and a three-zone resistance furnace surrounded each specimen. The test temperature over the specimen center three inches was controlled to $1400^{\circ}\text{F} \pm 5.4^{\circ}\text{F}$. Helium was supplied through each specimen pressurization tube, producing the desired hoop stresses. Specimen external surfaces were exposed to a helium 1% oxygen environment.

FIGURE 1 ORRS-2 and ORRS-3 in-pile stress-rupture specimen design.

TABLE I
CHEMICAL COMPOSITIONS (WEIGHT PERCENT)

Element	HT # 5911	HT # 281-4-0143
Cr	6.84	7.12
Mo	16.38	16.28
Fe	.12	.68
Ni	Bal.	Bal.
W	.12	.03
C	.046	.04
Si	.005	.23
Co	.005	.06
Mn	.04	.54
P	---	.005
S	---	.011
Al + Ti	.03	.19
B	.001	.002
Cu	.003	.03
O ₂	.015	.0088
H ₂	.002	.002
N ₂	.005	.0021

FIGURE 2 ORRS-2 and ORRS-3 irradiation capsule configuration.

The ORR S-2 stress-rupture specimens were irradiated at the ORNL from 11/18/66 to 1/1/67; the ORR S-3 specimens were irradiated in the next reactor cycle. Average thermal and fast flux for each specimen are included in Table 2.

III. POST-IRRADIATION EXAMINATION

ORR S-2 Specimens

1. Visual Examination

On February 24, the cask containing the ORR S-2 stress-rupture specimens arrived at the AIHL. All twenty specimens appeared in satisfactory condition.

Kollmorgen periscope photographs were taken of each specimen. Figures 3 through 6 show typical specimens in the as-received condition. Longitudinal failures (approximately one-half in. long) could be seen on 4550 and 5040R. Failures on the remaining specimens were not visible, and ORNL used small circles of red paint to pinpoint the failure areas (Figures 5 and 6).

2. Dimensional Measurements

Diameter measurements of each specimen were taken, using a helical scan diameter profilometer. Three modes of output were produced: (1) a continuous X-Y plot (of diameter as a function of curvilinear distance along the specimen length), (2) a printed digital form of the X-Y plot, on paper tape, and (3) a punched binary form of the X-Y plot, on Friden tape which is compatible with the data processing system at Atomics International. Table 4 includes the heat numbers and diameter data for each of the identified specimens; locations under consideration were those of maximum diametral strain. Tube diameter measurement precision was ± 0.0003 in.

3. Metallography

After completing the profilometer measurements, the specimens were sectioned for wall thickness measurements and metallography. A transverse met sample, one-half in. to 5/8-in. long was obtained through the failure zone of each specimen. Where the failure could not be directly located, the failure zone was determined by evaluating the

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TABLE 2

EXPERIMENT AVERAGE THERMAL AND FAST NEUTRON FLUX

<u>Specimen Number</u>	<u>Experiment Average Thermal Neutron Flux (n/cm²/sec)</u>	<u>Experiment Average Fast Neutron Flux (n/cm²/sec)</u>
4504	4.82 x 10 ¹³	4.06 x 10 ¹²
4550	↓	↓
4588		
4596		
5034R		
5036R		
5037R		
5039R		
5040R		
5046R		
4602	5.30 x 10 ¹³	3.40 x 10 ¹²
4616	↓	↓
4621		
6002		
6003		
6004		
6005		
6006		
6007		
6008		
4639	5.06 x 10 ¹³	3.73 x 10 ¹²
5053	↓	↓
5065		
5066		
5067		
5071		
5085		
5090		
5094		
5096		

171-4550

FIGURE 3 Kollmorgen photograph of Specimen No. 4550, showing longitudinal failure.

171-5040

FIGURE 4 Kollmorgen photograph of Specimen No. 5040R, showing raised longitudinal failure.

171-4504

FIGURE 5 Kollmorgen photograph of Specimen No. 4504, showing pinhole failure marked with a raised film spot.

171-5039-R

FIGURE 6 Kollmorgen photograph of Specimen No. 5039R, showing pinhole failure marked with a raised film spot.

diameter profile of the particular specimen with failure assumed to be in the localized area of maximum diameter change.

After cold mounting, the met samples were ground on silicon carbide papers and polished with 0.3 micron alumina. Photomicrographs, taken of the sections, appear in Figures 7, 8, 9, 10, 13 and 15. Figure 8 shows a typical met sample area in the as-polished condition. By using a calibration scale (Figure 11), wall thickness measurements were obtained for each specimen. Measurement precision was at least ± 0.001 in. The samples were then etched in an HCl-HNO_3 solution, Figure 12 shows a typically etched surface. The specimen chromized layers were quite discernable, and appeared to vary in thickness between specimens; additional specimen fracture areas, in the etched condition, are shown in Figures 14 and 16.

ORR S-3

1. Visual Examination

On April 28, the cask containing the ORR S-3 stress-rupture specimens arrived at the AIHL. The specimens were identified and visually examined. The ten specimens appeared in satisfactory condition.

Kollmorgen photographs were taken of each specimen. Figures 17 through 19 show typical specimens in the as-received condition. A large longitudinal failure can be seen on 4639; failures on the remaining specimens were not visible. No attempt at marking such failures was evident. It should be noted that, of the six unfailed specimens, the pressure tubes for 5053 and 5094 (unfailed) had been removed before the cask arrived at the AIHL, although these specimens were to be returned to the ORNL for further stress-rupture testing.

2. Dimensional Measurements

Diameter measurements of each specimen were taken as before, using the profilometer. Table 4 includes the heat numbers and diameter data for each specimen; locations considered were those of maximum diametral strain. Detailed specimen diameter-length relationships appear in Appendix A.

3. Metallography

After completing the profilometer measurements, the specimens

FIGURE 7 Photomicrograph of Specimen No. 4550 in the as-polished condition. Heat 281-4-0143, 250X.

FIGURE 9 Photomicrograph of Specimen No. 6002 in the as-polished condition. Heat 5911, 200X.

100-8-4

FIGURE 11 Photomicrograph of microscope stage micrometer at 200X, providing calibration for determining specimen wall thicknesses.

100-5-6

FIGURE 8 Photomicrograph of Specimen No. 4602 in the as-polished condition. Heat 281-4-0143, 250X.

FIGURE 10 Photomicrograph of Specimen No. 6007 in the as-polished condition. Heat 5911, 200X.

101-3-7

100-1-9

FIGURE 12 Photomicrograph of Specimen No. 4504 in the etched condition, showing secondary cracking in the specimen wall. Heat 281-4-0143, 250X.

100-1-7

FIGURE 14 Photomicrograph of Specimen No. 4504 in the etched condition, showing primary failure apparently enlarged as a result of etching. 250X.

101-6-7

FIGURE 16 Photomicrograph of Specimen No. 5036R in the etched condition, showing primary failure apparently enlarged as a result of etching. 250X.

100-1-1

FIGURE 13 Photomicrograph of Specimen No. 4504 in the as-polished condition, showing primary failure. 250X.

101-6-4

FIGURE 15 Photomicrograph of Specimen No. 5036R in the as-polished condition, showing primary failure. Heat 5911 Redrawn, 250X.

FIGURE 17 Kollmorgen photograph of Specimen No. 4639, showing large raised longitudinal failure.

FIGURE 18 Kollmorgen photograph of Specimen No. 5071. No failure location is visible.

FIGURE 19 Kollmorgen photograph of Specimen No. 5085, typical of the unfailed specimens.

were sectioned, as before, for wall thickness measurements and metallography. After sample preparation, wall thickness measurements were obtained. Photomicrographs were taken of samples in the etched condition. Fracture areas and chromized layer conditions were similar to those of the ORR S-2 specimens. Failure areas, in the etched condition appear in Figures 20, 21 and 22.

IV. RESULTS AND DISCUSSION

Test results for the ORR S-2 stress-rupture control specimens^{3,6} are presented in Table 3; test results for the ORR S-2 stress-rupture in-pile specimens are presented in Table 4. Considering two sets of profilometer measurements (unirradiated and irradiated), each accurate to ± 0.0003 in., the combined error would be ± 0.0006 in. or $\pm 0.1\%$ ductility. Inspection of the data in Tables 3 and 4 indicates that (1) irradiation reduced the creep ductility of Hastelloy-N from a range of 3.9% to 17.2% to a range of 0.3% to 2.6%, and (2) average creep rates of processed Hastelloy-N are relatively unaffected by irradiation, for the rates encountered in the ORR S-2 and ORR S-3 specimens. Data from Tables 3 and 4 were used in preparing a series of plots showing the relationships between creep ductility, rupture life, creep rate, and hoop stress.

The plot of hoop stress versus rupture life (Figure 23) includes values for unirradiated and irradiated material. Inspection of this plot indicates that (1) no differences exist between the hoop stress versus rupture life relationships for the two thicknesses of heat 5911 (0.010 in. and 0.015 in.) or between the two heats (0143 and 5911) for unirradiated and irradiated material, and (2) irradiation reduced the rupture life of processed Hastelloy-N by about a factor of ten.

The plot of hoop stress versus creep rate (Figure 24) for unirradiated and irradiated material indicates that no differences exist between the hoop stress versus creep rate relationships before and after irradiation, for heats 0143 or 5911, except that significant differences exist between the two thicknesses of heat 5911 after irradiation.

107-1-19

FIGURE 20 Photomicrograph of Specimen No. 4639 in the etched condition, showing severe cracking typical of failure section. Heat 281-4-0143, 100X.

107-1-22

FIGURE 22 Photomicrograph of Specimen No. 4639 in the etched condition, showing opposite edge of primary failure. Specimen fracture occurred during profilometer measurement. 100X.

107-1-21

FIGURE 21 Photomicrograph of Specimen No. 4639 in the etched condition, showing edge of large primary failure. 100X.

TABLE 3

ORR S-2 CONTROL TEST RESULTS

Heat No.	Specimen No.	Hoop Stress (psi)	Failure Temperature (°F)	Rupture Life (Hours)	Ductility (%)	Average Creep Rate (Hours ⁻¹)
281-4-0143	5069	50,400	1400	~0.1	17.2	1.7
281-4-0143	5057	35,900		2.2	6.7	3.0×10^{-2}
281-4-0143	5097	29,800		4.2	7.7	1.8×10^{-2}
281-4-0143	5068	24,100		12.7	4.6	3.6×10^{-3}
281-4-0143	5098	21,100		15.5	9.6	6.2×10^{-3}
281-4-0143	5048	18,600		63.7	8.9	1.4×10^{-3}
281-4-0143	5072	14,900		153.2	8.2	5.3×10^{-4}
281-4-0143	5062	12,410		788.5	11.1	1.4×10^{-4}
281-4-0143	5078	9,930		2160.0	10.6	4.9×10^{-5}
5911	6025	38,200		0.9	8.4	9.3×10^{-2}
5911	6016*	27,200		30.1	6.7	2.3×10^{-3}
5911	6029	27,100		5.0	3.9	7.8×10^{-3}
5911	6019	19,400		30.3	9.4	3.1×10^{-3}
5911	6020*	14,500		61.2	0.9	Did not rupture- leak in system
5911	6018	15,520		100.2	6.4	6.4×10^{-4}
5911	6026	14,500		343.5	9.0	2.6×10^{-4}
5911	6017*	11,610		160.9	7.6	Temperature ran high
5911	6012*	11,940		523.3	5.3	
5911	6014	11,630		334.7	7.63	2.3×10^{-4}
5911	6031	9,690		2974.5	4.6	1.5×10^{-5}
5911R	5127R	36,600		0.4	9.1	2.3×10^{-1}
5911R	5126R	31,400		5.85	4.4	7.5×10^{-3}
5911R	5057R	26,300		19.8	8.2	4.1×10^{-3}
5911R	5050R	19,700		~86.9	8.2	9.5×10^{-4}
5911R	5049R	19,700		87.5	8.9	1.0×10^{-3}

TABLE 3 (Continued)
ORR S-2 CONTROL TEST RESULTS

Heat No.	Specimen No.	Hoop Stress (psi)	Failure Temperature (°F)	Rupture Life (Hours)	Ductility (%)	Average Creep Rate (Hours ⁻¹)
5911R	5038R	17,730	1400	20.9	7.8	3.7×10^{-3}
5911R	5130R	15,920	↓	142.4	6.2	4.4×10^{-4}
5911R	5128R	13,180	↓	372.5	7.8	2.1×10^{-5}
5911R	5123R	10,470	↓	1731.7	6.8	3.9×10^{-5}
5911R	5054R	10,490	↓	866.7	7.7	8.9×10^{-5}

* Test Results Deemed not Valid by ORNL

TABLE 4

ORR S-2 and S-3 Irradiated Test Results

Heat No.	Specimen No.	Post-Irradiation Diameter (in.)	Wall Thickness (in.)	Post-Irradiation Mean Diameter (in.)	Internal Pressure (psi)	Hoop Stress (psi)	Failure Temperature (°F)	Thermal Neutron Dose (n/cm ²)	Fast Neutron Dose (n/cm ²)	Rupture Life (hours)	Ductility (%)	Creep Rate (Hours ⁻¹)
281-4-0143	4504	.5664	.0114	.5550	1000	24,342	1400	2.04 x 10 ¹⁹	1.72 x 10 ¹⁹	1.25	0.69	5.54 x 10 ⁻³
	4550	.5774	.0110	.5664	800	20,596				2.25	2.65	1.18 x 10 ⁻²
	4588	.5658	.0114	.5544	700	17,021				6.7	0.52	7.69 x 10 ⁻⁴
	4596	.5654	.0112	.5542	600	14,845				5.7	0.41	7.16 x 10 ⁻⁴
	4602	.5654	.0111	.5543	500	12,484		2.24 x 10 ¹⁹	1.44 x 10 ¹⁹	56.3	0.61	1.07 x 10 ⁻⁴
	4616	.5663	.0121	.5542	450	10,305				105.9	0.68	6.38 x 10 ⁻⁵
	4621	.5672	.0121	.5551	400	9,175				88.3	0.76	8.65 x 10 ⁻⁵
5911	6002	.5715	.0150	.5565	1000	18,550				8.17	0.35	4.30 x 10 ⁻⁴
	6003	.5729	.0149	.5580	800	14,980				22.75	0.63	2.78 x 10 ⁻⁴
	6004	.5736	.0150	.5586	700	13,034				46.0	0.72	1.57 x 10 ⁻⁴
	6005	.5755	.0151	.5604	600	11,134				133.1	1.02	7.65 x 10 ⁻⁵
	6006	.5787	.0151	.5636	500	9,331				244.3	1.47	6.03 x 10 ⁻⁵
	6007	.5794	.0148	.5646	450	8,583				572.0	1.70	2.98 x 10 ⁻⁵
	6008	.5784	.0149	.5636	400	7,565				632.0	1.55	2.44 x 10 ⁻⁵
5911 redrawn	5034R	.5638	.0108	.5530	1000	25,603		2.04 x 10 ¹⁹	1.72 x 10 ¹⁹	0.7	0.68	9.70 x 10 ⁻¹
	5036R	.5675	.0105	.5570	800	21,219				4.7	1.32	2.81 x 10 ⁻¹
	5037R	.5694	.0105	.5589	600	15,969				25.5	1.59	6.23 x 10 ⁻²
	5039R	.5645	.0113	.5532	450	11,015				109.5	0.30	2.76 x 10 ⁻³
	5040R	.5718	.0105	.5613	400	10,691				145.8	2.03	1.40 x 10 ⁻²
	5046R	.5716	.0106	.5610	500	13,231				83.2	2.00	2.40 x 10 ⁻²
281-4-0143	4639	.5740	.0113	.5627	362	9,000	1400	2.23 x 10 ¹⁹	1.64 x 10 ¹⁹	505.1	2.04	4.05 x 10 ⁻⁵
	5053	.5725	*	--	---	--						
	5065	.5636	**	--	---	--						
	5066	.5670	.0113	.5557	488	12,000***				40.6	0.72	1.78 x 10 ⁻⁴
	5067	.5623	**	--	---	--				--	--	---
	5071	.5750	.0114	.5636	405	10,000				409.6	2.16	5.27 x 10 ⁻⁵
	5085	.5624	**	--	---	--				--	--	---
	5090	.5738	.0111	.5627	335	8,500				930.0	2.09	2.25 x 10 ⁻⁵
	5094	.5625	**	--	---	--				--	--	---
	5096	.5625	**	--	---	--				--	--	---

* Unfailed
** Unstressed, Unfailed
*** Held at Temperature for 950 hours before applying stress

FIGURE 23 Hoop stress vs. rupture life for un-irradiated and irradiated cladding tube specimens.

FIGURE 24 Hoop stress vs. creep rate for un-irradiated and irradiated cladding tube specimens.

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Those irradiated specimens stressed at lower levels had lower creep rates (see Table 4), more creep ductility, (Figure 25) and longer rupture lives (Figure 26). It should be noted that significant differences exist in the ductility and rupture time versus creep rate relationships between the two thicknesses of heat 5911 and that specimens of redrawn heat 5911 show longer rupture lives and greater ductility values at similar creep rates. Thus, the redrawn heat 5911 appears superior to the other materials tested. However, only a few specimens of each type of material were tested at comparable stress levels and creep rates. Further testing, with more statistically significant sampling should be performed to verify this observation.

Specimen fractures were completely intergranular, which should be expected at 1400°F. Specimens of high ductility and early rupture times tended to have secondary grain boundary cracking; specimens of longer rupture lives tended to have fewer, but larger cracks in their grain boundaries.

V. CONCLUSIONS

1. Irradiation reduces creep ductility in Hastelloy-N. Average creep ductility for all unirradiated cladding specimens was about 4%; ductility for all irradiated specimens was about 1%. Greater pre-post irradiation ductility changes were noted, when specimens of similar hoop stress levels were considered.
2. Irradiation reduces rupture life in Hastelloy-N. Average unirradiated and irradiated rupture times, for all comparable hoop stress levels, were 480 hours and 175 hours, respectively.
3. The creep rate in Hastelloy-N is not affected by irradiation. Creep rates, for unirradiated vs irradiated specimens, fall within the same range, for similar hoop stress levels.

FIGURE 25 Ductility vs. creep rate for irradiated cladding tube specimens.

FIGURE 26

Rupture time vs. creep rate for un-irradiated and irradiated cladding tube specimens.

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3. G.G. Allaria, "ORR S-2 Irradiated Hastelloy-N Biaxial Stress-Rupture Data", NAA-SR-TDR-12457, May 1967.
4. Atomics International Laboratory Notebook B-345001.
5. Atomics International Laboratory Notebook B-320451.
6. J.D. Stearns, "Determination of Uniaxial Mechanical Properties of Unirradiated and Irradiated Hastelloy-N Bar and Biaxial Stress-Rupture Properties of Chromized and Coated Unirradiated Hastelloy-N", NAA-SR-TDR-12556, September 1967.

APPENDIX A**Computer Summary of Diameter
Data for Failed ORR S-3 Tubes***

* Computer data for ORR S-2 tubes may be found in Reference 3,
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COMPARISON DATA FOR ORNL BURST SPECIMEN □ 4639 ⁷⁷⁰⁰⁻²⁴ _{0007 0000}

ORNL STRESS RUPTURE TUBE COMPARISON DATA NO 5086

7766-15
00000000

PRE-TEST AVERAGE DIAMETERS

POST-TEST AVERAGE DIAMETERS

RESULTANT DIAMETER CHANGES

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ORNL STRESS RUPTURE TUBE COMPARISON DATA NO 5071 7766-15
9999

PRE-TEST AVERAGE DIAMETERS

POST-TEST AVERAGE DIAMETERS

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ORNL STRESS RUPTURE TUBE COMPARISON DATA NO 5090

7766-13
0000

PRE-TEST AVERAGE DIAMETERS

POST-TEST AVERAGE DIAMETERS

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