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REPORT TITLE

LOW-CYCLE FATIGUE TESTING OF IRRADIATED HASTELLOY-N

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ATOMICS INTERNATIONAL <small>A Division of North American Aviation, Inc.</small>		NAA-SR- TDR NO-12553	APPROVALS
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STATEMENT OF PROBLEM Perform reverse-bend cycling tests on irradiated Hastelloy-N strips to demonstrate that the Manson method of predicting fatigue properties from tensile data is valid for irradiated Hastelloy-N design criteria.			
ABSTRACT Hastelloy-N strip specimens were irradiated in the Oak Ridge Research Reactor at temperatures of 1330-1390°F, to neutron doses of 1.8 - 2.6 x 10 ²⁰ nvt thermal, 1.4 - 2.0 x 10 ¹⁹ nvt fast. Twelve irradiated specimens were selected for testing from each of two heats, 0143 (0.010 in. thick) and 5911 (0.015 in.). Six specimens in each heat were as-received, the other six were as-processed. Processing steps included chromizing, a simulated coating firing cycle, diffusion treatment, and straining to simulate tube straightening. In-cell fatigue tests were performed by reverse-bend cycling over mandrels of opposing radii at one cycle per minute, in helium, at 1400°F. The twelve specimens tested at strain ranges of 0.61 - 0.75% had an average fatigue life of 97 cycles. Only seven of the specimens tested at strain ranges of 0.31 - 0.37% failed; five remained intact after 25,000 cycles. The average fatigue life for all 0.31 - 0.37% specimens was 15,840 cycles, with no weighting factor used for intact specimens. Experimental results were four times greater than the predicted fatigue life at the higher strain range, fifty times greater at the lower. The Manson method for prediction of Hastelloy-N fatigue properties after irradiation appears valid for use in establishing design criteria.			

I. INTRODUCTION

The design of SNAP-8 fuel elements requires knowledge of the effects of irradiation on the mechanical behavior of the Hastelloy-N cladding. Due to a lack of experimental data, fatigue properties have been predicted from tensile test data, using a correlation developed by Manson¹. The prime objective of this investigation was to demonstrate that the Manson tensile ductility method provides a valid means for prediction of fatigue properties of irradiated Hastelloy-N.

The existing curve of strain range versus cycles to failure was corrected using irradiated Hastelloy-N tensile test data from the ORR S-1 experiment⁷. The strip specimens intended for use in fatigue testing were irradiated as a part of the ORR S-2 irradiation experiment. In-cell tests were performed by reverse-bend cycling over mandrels of opposing radii to provide data for comparison with the predicted curve. This report presents the results of the post-irradiation examination and fatigue tests conducted on the Hastelloy-N strips from ORR S-2.

II. SPECIMEN HISTORY

Strip specimens were 5.0 in. long, 0.5 in. wide, and 0.010 or 0.015 in. thick (nominally). Tubing heats 0143, 0.540 in. ID x 0.010 in. wall, and 5911, 0.540 in. ID x 0.015 in. wall, were used as the raw material. The chemical composition of these heats is shown in Table 1.

The raw stock, received as tubing, was slit axially and rolled flat. Specimens to be tested "as-received" were machined to final dimensions, completing fabrication. Specimens to be tested fully processed were chromized, subjected to a simulated coating firing cycle (no coating was actually applied), machined, diffusion treated and strained to simulate tube straightening. Strips were also available in intermediate processing conditions, as shown in Table 2. A detailed summary of all fabrication steps can be found in another document².

The strip specimens were irradiated with the ORR S-2 Experiment, which measured in-pile stress-rupture properties of Hastelloy-N.

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A Division of North American Aviation, Inc.

NO. NAA-SR-TDR-12553DATE September 28, 1967PAGE 3 OF 25**TABLE 1**Chemical Composition of Hastelloy-N Heats
Used for ORR S-2 Strips

Element	Weight Percent	
	Heat 0143 (0.010 in. thick)	Heat 5911 (0.015 in. thick)
Ni	Balance	Balance
Mo	16.28	16.38
Cr	7.12	6.84
Fe	0.68	0.12
Mn	0.54	0.04
Si	0.23	0.005
Al + Ti	0.19	0.03
Co	0.06	0.005
C	0.04	0.046
Cu	0.03	0.003
W,	0.03	0.12
S	0.011	0.002
P	0.005	0.002
B	0.002	0.001
O ₂	0.009	0.015
N ₂	0.002	0.002
H ₂	0.002	0.050

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TABLE 2

ORR S-2 Strips Shipped to ORNL

Condition	Heat 0143 (0.010 in. thick)	Heat 5911 (0.015 in. thick)
As-received	15	15
Chromized, simulated coating firing cycle (C)	15	15
Chromized, simulated coating firing cycle, strained (CS)	9	9
Chromized, simulated coating firing cycle, diffusion treated, strained (CDS)	9	9

ORR S-2 contained 20 fully processed Hastelloy-N tubes, 10 in capsule ORR-171 and 10 in capsule ORR-172. A total of 72 strip specimens were placed in the ORR-171 capsule by locating bundles of eight strips in the nine gaps between tubes, as shown in Figure 1. The ORR-171 capsule was irradiated in Cycle 70 of the Oak Ridge Research Reactor, from 11-18-66 to 1-8-67. Total irradiation time was 1224 hours, or 4.406×10^6 sec. A summary of neutron doses and irradiation temperatures³ is presented in Table 3.

III. FATIGUE TEST PROCEDURES

The fatigue tests used a completely reversed cycle of strain, with maximum tensile and compressive strains equal in magnitude. The cycle was imposed by bending a strip over mandrels of opposing radii, as shown in Figure 2. Imposition of varying levels of strain was accomplished by changing the radius of the mandrels.

A frame, containing six sets of mandrels mounted in parallel, a single actuator bar which imparted bending motion to six strips simultaneously, and a mesh basket to catch broken strips, was mounted on the inside of the door of a large electric resistance furnace (see Figure 3). The drive mechanism, located on the outside of the furnace door, was connected to the actuator bar by a shaft passing through a graphite seal. The furnace was mounted on rollers so that it could be moved against the door, compressing a gasket to effect a seal. This furnace was a TRENT Model COF VH-18, equipped with a fan to circulate the furnace atmosphere. Two identical units were located in-cell. A gas supply, located outside the cell, fed reactor grade helium to each of the units.

The control console shown in Figure 3 was located outside the cell. This unit contained two furnace controllers, one temperature recorder, on-off switches for furnace power and circulating fans, and limiting switches that prevented operation above or below the allowable temperature range (20°F band). The control console also contained cycle counters, switches that cut the run if the drive motor or fan motor ceased operating or if the shaft seized, and the specimen

Figure 1. Schematic drawing of the ORR-171 capsule of the ORR S-2 experiment.

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TABLE 3

Irradiation Temperatures, Neutron Doses
for ORR S-2 Strip Specimens

Position	Strips	Irradiation Temperature, °F	Thermal Neutron* Dose, nvt x 10 ²⁰	Fast Neutron* Dose, nvt x 10 ¹⁹
1 - 2	54CS, 55CS, 58CS, 59CS, 60CS, 61CS, 63CS, 64CS	1340	1.81	1.41
2 - 3	21, 22, 23, 29, 31, 32, 57, 59	1336	2.06	1.55
3 - 4	52, 60, 61, 62, 14C, 16C, 19C, 22C	1357	2.28	1.72
4 - 5	69C, 75C, 16CDS, 19CDS, 20CDS, 23CDS, 24CDS, 25CDS	1360	2.34	1.85
5 - 6	67C, 70C, 66CDS, 68CDS, 69CDS, 70CDS, 71CDS, 75CDS	1384	2.39	1.93
6 - 7	64C, 65C, 68C, 79C, 80C, 65CDS, 67CDS, 73CDS	1370	2.52	1.96
7 - 8	5C, 8C, 30C, 34C, 36C, 17CDS, 18CDS, 26CDS	1355	2.55	1.98
8 - 9	3, 18, 25, 56, 58, 65, 15CS, 56CS	1356	2.51	1.87
9 - 10	51C, 53C, 54C, 58C, 9CS, 11CS, 12CS, 13CS	1381	2.42	1.73

* Thermal flux (nv) obtained from monitors using the $\text{Co}^{59} (n, \gamma) \text{Co}^{60}$ reaction; fast flux from monitors using the $\text{Fe}^{54} (n, p) \text{Mn}^{54}$ reaction.

Strain Range, %	Mandrel Radius, in.	
	Heat 0143	Heat 5911
2.4	0.416	0.625
1.2	0.833	1.250
0.6	1.666	2.500
0.3	3.333	5.000

Figure 2. Mandrel type used for reverse-bend cycling of ORR S-2 strip specimens.

Figure 3. Fatigue test equipment used for ORR S-2 strip specimens.

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failure detection circuit. This circuit was constructed with the actuator bar being a common leg, with individual legs leading to each of the lower mandrels. Thus, the circuit was complete, and indicating continuity, only if the strip was in place and intact. Lava blocks between the mandrels and the frame insured electrical isolation of each circuit. Each of the twelve circuits (six per furnace) was monitored by a 24-point Minneapolis-Honeywell recorder (two points per circuit). This recorder presented traces of continuity versus time for each strip, so that failures could be detected during unattended periods.

Due to the radioactivity of the specimens, strip handling was performed with master-slave manipulators. Loading was accomplished by placing the hole at the back of each specimen over the pin at the rear of each mandrel, placing the front of the specimen in a slot on the actuator bar, and bolting the upper mandrels to the lower mandrels. The furnace was rolled up to the door assembly, and the gasket compressed by securing several toggle clamps (see Figure 3).

Both furnaces were purged with a $23.8 \text{ ft}^3/\text{hr}$ helium flow while the circulating fan was in operation. The helium introduced during the purge was equivalent to approximately seven furnace volumes. After one hour, the helium flow was reduced to $6.8 \text{ ft}^3/\text{hr}$, and furnace power was applied. The lower flow rate, equivalent to approximately two oven volumes each hour, was maintained throughout the test. After five hours, the temperature stabilized at 1400°F , and cycling was initiated at the rate of one per minute. Each cycle involved movement of a strip from the horizontal position to its maximum upward displacement, movement to its maximum downward displacement, and return to the original horizontal position. Cycling was continued until all strips were broken in each furnace. Specimen failure was indicated by upscale readings on the continuity recorder, and by large resistance readings ($> 1000 \Omega$) obtained with a Simpson meter. After all specimens in a furnace had failed, the cycling was stopped and the furnace power cut. The purge was maintained until the furnace cooled to 200°F . Unloading operations were also performed with manipulators.

The test matrix, shown in Table 4, was satisfied by conducting two runs. The first run contained six strips of each heat and used a nominal 0.6% strain range, while the second run, also containing six strips of each heat, used a nominal 0.3% strain range.

IV. RESULTS

A. Post-Irradiation Examination

The shipping cask was received from ORNL and unloaded on 2-1-67. The 72 strips were in 18 bundles of 4 strips each, secured by 4-5 wraps of Nichrome wire. The bundles were wet, and about one inch of what appeared to be water was found in the bottom of the cask. The bundles were disassembled and individual specimens identified. Figure 4 illustrates the typical post-irradiation condition of the strips.

Flatness measurements were performed to provide a basis for selection of the 24 specimens that were tested. A series of five measurements were made at 3/4-in. intervals along the length of each strip, using a dial indicator device accurate to ± 0.0005 in. The maximum deviation from flatness for each strip is presented in Table 5. Specimens selected for testing are marked with an asterisk.

B. Fatigue Tests

Testing was performed at 1400°F, in helium, at one cycle per minute. Two separate runs were made, the first testing six strips of each heat at a nominal 0.6% strain range, the second testing six strips of each heat at a nominal 0.3% strain range. The results of these tests are presented in Table 6 and Figure 5.

All twelve irradiated specimens tested at the 0.61 - 0.75% strain range failed. The average value for these tests was 97 cycles, about four times the calculated value. Only seven of the twelve irradiated specimens tested at the 0.31 - 0.37% strain range failed; five were intact at 25,000 cycles. These tests were terminated since they had attained a cycle level one decade above the number they were expected to endure. The average value for these tests was 15,840 cycles, about 50 times the calculated value, even using the 25,000 cycle values with

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TABLE 4

Test Matrix for Post-Irradiation Fatigue
Tests with ORR S-2 Strips

Nominal Strain Range, %	Strip Condition	Number of Samples	
		Heat 0143	Heat 5911
0.6	As-received	3	3
	Chromized, diffusion treated and straightened	3	3
0.3	As-received	3	3
	Chromized, diffusion treated and straightened	3	3

a. Photo ORR-5, four unchromized strips.

b. Photo ORR-12, one chromized (69C), and three fully processed strips.

Figure 4. Hastelloy-N strips after irradiation in the Oak Ridge Research Reactor.

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TABLE 5

Flatness Measurements for Irradiated Strip Specimens

Type of Strip	Nominal Thickness, mils	Strip No.	Max. Deviation from Flatness, mils
Unchromized	10	18	2.4
		21	1.8*
		22	1.9*
		23	1.0*
		25	2.8
		29	2.0*
		30	1.3*
		31	1.4*
		32	2.2
	15	52	0.8*
		56	0.9*
		57	0.0*
		58	1.5
		59	0.2*
		60	0.2*
		61	1.5
		62	1.2
		65	0.7*
Chromized, Simulated Fired	10	5C	4.5
		8C	3.3
		14C	5.3
		16C	3.8
		19C	5.7
		22C	4.5
		30C	4.5
		34C	3.3
		36C	2.5
Chromized, Simulated Fired	15	64C	1.5
		66C	4.5
		67C	3.5
		68C	1.0
		69C	3.5
		70C	2.7
		75C	2.3
		79C	2.0
		80C	2.0

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TABLE 5 (Continued)

Type of Strip	Nominal Thickness, mils	Strip No.	Max. Deviation from Flatness, mils
Chromized, Strained, Simulated Fired	10	1CS	3.4
		3CS	1.7
		4CS	6.5
		8CS	2.4
		9CS	3.5
		11CS	3.5
		12CS	4.6
		13CS	2.0
		15CS	2.5
	15	54CS	0.1
		55CS	1.5
		56CS	1.5
		58CS	1.3
		59CS	2.4
		60CS	1.7
Chromized, Diffusion Treated, Strained, Simulated Fired	10	61CS	4.5
		63CS	1.7
		64CS	1.2
		16CDS	5.3*
		17CDS	6.6
		18CDS	4.1*
		19CDS	5.3*
		20CDS	4.0*
		23CDS	3.5*
		24CDS	2.6*
	15	25CDS	5.8
		26CDS	10.0
		65CDS	3.4*
		66CDS	3.8*
		67CDS	4.1
		68CDS	5.8
		69CDS	1.7*
	15	70CDS	2.6*
		71CDS	2.3*
		72CDS	5.3
		73CDS	4.0

* Selected for fatigue testing

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TABLE 6

Results of 1400°F, One Cycle/Minute Bend
Fatigue Tests of Irradiated Hastelloy-N Strips

Heat	Condition	Strip	Thickness, in. ^a	Strain Range, % ^b	Cycles to Failure
0143	As-Received	23	0.0112	0.67	30
		31	0.0113	0.68	138
		22	0.0116	0.69	274
		29	0.0114	0.33	4800
		30	0.0113	0.33	10,124
		21	0.0115	0.33	11,760
	Chromized, Diffusion Treated, Strained, Simulated Fired	16	0.0122	0.73	127
		20	0.0125	0.75	128
		24	0.0118	0.71	130
		18	0.0125	0.36	11,140
		19	0.0126	0.37	25,000 ^c
		23	0.0114	0.33	25,000 ^c
	As-Received	52	0.0154	0.61	7
		60	0.0155	0.62	19
		57	0.0156	0.62	23
		56	0.0169	0.33	25,000 ^c
		59	0.0164	0.32	25,000 ^c
		65	0.0158	0.31	25,000 ^c
		70	0.0156	0.62	68
		69	0.0158	0.63	109
	Chromized, Diffusion Treated, Strained, Simulated Fired	66	0.0158	0.63	113
		73	0.0160	0.31	3410
		65	0.0161	0.31	5440
		71	0.0163	0.32	18,450

^a Measured in-cell with micrometers, verified metallographically.

^b Calculated from the formula: Strain Range = $t \div (r_m + \frac{1}{2}t)$,
where t = measured thickness of the strip, r_m = mandrel radius.

^c Specimen unfailed

Figure 5. Reverse bend fatigue performance of irradiated Hastelloy-N strips tested at 1400°F and one cpm in helium.

no weight factor. These results show the Manson prediction for irradiated Hastelloy-N is valid for use in establishing design criteria, but may be conservative at lower strain ranges.

Figure 6 presents the results of reverse-bend fatigue testing of unirradiated, control specimens. Strips used for these tests were fabricated from heats 0143 and 5911 by methods identical to those employed for the specimens destined for irradiation². Fatigue tests were performed on identical equipment, at 1400°F, in helium, at one cycle per minute, using nominal strain ranges of 2.4%, 1.2%, 0.6%. The six tests at the 0.6% strain ranges yielded an average failure life of about 900, close to a decade above the average failure value for irradiated strips. The three tests at the 0.3% strain range were terminated due to the long lives of similar irradiated specimens, indicating impractically long test duration.

Comparison of fatigue lives for the two heats, 0143 (0.010 in. thick) and 5911 (0.015 in. thick), indicates similar values. However, heat 0143 appears to be clearly superior in the fully-processed condition.

C. Post-Test Examination

A visual examination following testing showed 18 single fractures, one double fracture, and five intact specimens. Photographs of typical specimens following testing are presented in Figure 7.

A total of ten specimens were selected for metallography; one from each of eight matrix conditions (Table 4), and two from the intact specimens. An abrasive cutoff wheel was used to remove a 1/2-inch section surrounding the fracture for failed specimens, and the central section for intact specimens. The sections were mounted in epoxy resin and ground flat with silicon carbide abrasive papers. Polishing steps included use of a water slurry of six micron diamond paste and attack-polishing with alternate applications of three solutions: a 0.3 micron Linde B/H₂O slurry, a HNO₃-HF-H₂O mixture, and a 10% oxalic acid solution. Samples were electrolytically etched in a 10% oxalic acid solution. Photographs were taken as-polished and as-etched.

Typical post-test microstructures of failed Heat 0143 specimens are shown in Figure 8. The large difference in grain size is due to the simulated cladding processing. All of the fractures are inter-

Figure 6. Reverse bend fatigue performance of unirradiated Hastelloy-N strips tested at 1400°F and one cpm in helium.

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Figure 7. Irradiated Hastelloy-N strips after fatigue testing at 1400° F in helium.

a. Failed specimens from heat 0143

ORR-24, as-received specimen 31, failed after 138 cycles at a 0.68% strain range.

ORR-20, as-processed specimen 16, failed after 127 cycles at 0.73%.

ORR-34, as-received specimen 29, failed after 4800 cycles at 0.33%.

ORR-37, as-processed specimen 18, failed after 11,140 cycles at 0.36%.

b. Failed specimens from heat 5911

ORR-27, as-received specimen 60, failed after 19 cycles at 0.62%.

ORR-30, as-processed specimen 66, failed after 113 cycles at 0.63%.

ORR-31, as-processed specimen 71, failed after 18,450 cycles at 0.32%.

c. ORR-38, specimens unfailed after 25,000 cycles at 0.31-0.37%. Strips 56, 59, 65, are as-received heat 5911; strips 19, 23, are as-processed heat 0143.

65 59 23 56 19

Figure 8. Post-test microstructures of
Hastelloy-N Heat 0143.

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114-1-1, attack-polished section
at fracture.

114-1-4, etched section at fracture.

114-2-1, attack-polished section
at fracture.

114-2-4, etched section at fracture.

114-6-3, attack-polished section
at fracture.

114-6-1, etched section at fracture.

114-1-5, etched section away from fracture.

difference in grain size due to processing

preferential reaction of etchant with
the chromized layer

114-2-6, etched section
away from fracture.

114-6-2, etched section
away from fracture.

a. As-received specimen 31, failed after 138
cycles at a 0.68% strain range. Original
photos all at 250X.

b. As-processed specimen 16, failed after 127
cycles at a 0.73% strain range. Original
photos all at 250X.

c. As-processed specimen 18, failed after
11,140 cycles at a 0.36% strain range.
Original photos all at 200X.

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granular, with few secondary grain boundary cracks and little or no grain elongation. Typical post-test microstructures of failed Heat 5911 specimens are shown in Figure 9. Again, the effect of processing on grain size is apparent and all fractures are intergranular. The post-test microstructures of two unfailed specimens are presented in Figure 10. There are no apparent structural differences between these samples and failed samples away from the fracture area.

V. CONCLUSIONS

1. The Manson method prediction of fatigue properties of irradiated Hastelloy-N is valid for establishing design criteria. Observed fatigue life was four times greater than predicted at strain ranges of 0.61 - 0.75%, and fifty times greater than predicted at strain ranges of 0.31 - 0.37%.
2. Irradiation of Hastelloy-N produced a one decade decrease in the fatigue life at strain ranges of 0.6 - 0.7%.
3. Fully processed Hastelloy-N from heat 0143, nominally 0.010 in. thick, had a longer fatigue life after irradiation than heat 5911, nominally 0.015 in. thick.

Figure 9. Post-test microstructures of Hastelloy-N Heat 5911. All original photos at 200X.

114-3-6, attack-polished section at fracture.

114-3-7, etched section at fracture.

secondary crack

114-9-1, attack-polished section at fracture.

114-9-3, etched section at fracture.

artifact

114-10-1, attack-polished section at fracture.

114-10-3, etched section at fracture.

precipitates

114-3-4, etched section away from fracture.

114-9-4, etched section away from fracture.

twinning

114-10-4, etched section away from fracture.

a. As-received specimen 60, failed after 19 cycles at a 0.62% strain range.

b. As-processed specimen 65, failed after 5,540 cycles at a 0.31% strain range.

c. As-processed specimen 71, failed after 18,450 cycles at a 0.32% strain range.

Figure 10. Post-test microstructures of
Hastelloy-N strip specimens
unfailed after 25,000 cycles
at a strain range of 0.31 -
0.37%. All original photos
at 200X.

A. As-processed specimen 19, Heat 0143

114-7-2, typical of attack-polished sample.

114-7-1, typical of etched sample.

B. As-received specimen 56, Heat 5911

114-8-2, typical of attack-polished sample.

114-8-1, typical of etched sample.

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