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WANL-TME-2799
NOVEMBER 1971

MASTER

**LOW TEMPERATURE TENSILE
PROPERTIES OF IRRADIATED
INCONEL 718**

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**LOW TEMPERATURE TENSILE
PROPERTIES OF IRRADIATED
INCONEL 718**

APPROVED BY:

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Materials

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ABSTRACT

Tensile specimens of Inconel 718 sheet were irradiated at 140°R and tested at temperatures up to 740°R . Maximum fluence obtained was 10^{19} n/cm^2 , $E > 1 \text{ MeV}$. Yield strengths increased below 540°R while ultimate strengths decreased slightly. A significant embrittlement also occurred at all test temperatures. However, the effects noted should not limit the applicability of the alloy over the range of test conditions studied.

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INTRODUCTION

The NERVA reactors present a unique environment for the component materials, including cryogenic to elevated temperatures, high pressure hydrogen and a high neutron flux field. Face-centered cubic alloys are generally selected as the structural materials to preclude cryogenic temperature-induced embrittlement. Previous tests^(1,2) have shown that radiation-induced property changes are intensified at cryogenic temperatures. However, fluences planned for this test have not been previously attained for alloy Inconel 718*.

Inconel 718 is a high strength, tough, nickel-base alloy usable at both high and low temperatures. Strengthening is obtained by precipitation hardening. Previously, this alloy was the prime structural material of the NERVA reactor. However, since the initiation of this test, the alloy has been found to be embrittled by high-pressure hydrogen and, therefore, it is no longer being used. This report is intended to document the work done on this alloy. Because of the other outstanding properties of the alloy, however, it is being considered for application in other nuclear environment programs. In this regard, the data obtained in this report should be a useful addition to the literature.

In a previous test⁽³⁾ similar Inconel 718 specimens were irradiated at room temperature to fluences of 7×10^{18} n/cm² $E > 1$ MeV and 1.5×10^{20} , $E < 0.4$ eV. The present test was intended to extend these data to cryogenic temperatures.

* Trademark of International Nickel Corporation

1.0 TEST PROCEDURE

1.1 MATERIAL

Specimens were taken from 0.022 inch thick Inconel 718 sheet with the axes in the rolling direction. Material was purchased from the Latrobe Steel Company (heat number C50026) to WANL PDS 30030-1 Rev. A. The chemical composition shown in Table 1 meets this specification. Grain size was ASTM No. 8.

The material was received as mill annealed at 1700°F to 1800°F. One half of the finished machined specimens were solution treated for one hour at 1750°F, then aged 8 hours at 1325°F, furnace cooled to 1150°F, and aged an additional 8 hours. The other half were solution treated for one hour at 1950°F, then aged 10 hours at 1400°F, cooled to 1200°F and aged an additional 8 hours.

TABLE 1

CHEMICAL COMPOSITION, w/o

Al - 0.47	Si - 0.09
Cr - 18.61	S - 0.008
C - 0.03	Ti - 0.97
Cu - 0.02	Mo - 2.89
Mn - 0.07	B - 0.004
Ni - 52.28	P - 0.004
Cb - 5.00	Ta - 0.07
Co - 0.07	Fe - bal

1.2 SPECIMENS

Specimen configuration is shown in Figure 1. A pin-loaded design was selected for ease of remote loading and alignment and security of assembly. Doublers were welded to the specimen heads for support. Specimen thickness was that of the as-received sheet.

Since test machine crosshead movement was used to determine specimen deflection, the entire one-inch reduced section was considered as the gage length.

Figure 1. Sheet Tensile Specimen

1.3 TEST PLAN

Because of the wide range of test variables of interest, a factorial experimental test plan was designed which considered all combinations of the variable levels. The variables considered were:

- 1) heat treatment (1750°F or 1950°F solution plus age).
- 2) fast fluence: 0, 5×10^{17} , 2×10^{18} and 1×10^{19} n/cm², $E > 1$ MeV.
- 3) test temperature: 140°R, 340°R, 540°R and 740°R.

One specimen was tested for each combination of the above variables with several spare specimens included as substitutes for damaged specimens. A random test order was used. In addition, specimens were included for irradiation at the highest fluence followed by annealing at 340°R, 540°R and 740°R and testing at 140°R. Additional annealing conditions were used for extra spare specimens. Anneal times ranged from 10 minutes to 1000 minutes.

Properties determined in this test were 0.2 percent offset yield strength, ultimate tensile strength, uniform elongation and total elongation.

1.4 IRRADIATION

Fluence levels were attained in the 10 MW Ground Test Reactor (GTR) at General Dynamics, Fort Worth, Texas in reactor cycle 20C. During irradiation the specimens were immersed in boiling liquid nitrogen (LN₂, 140°R). The reactor ran a total of approximately 6000 MW-hrs.

Fluences were measured using nickel foils and relating the results to preliminary mapping runs. Details have been reported by General Dynamics⁽⁴⁾.

1.5 TESTING

After irradiation, specimens were stored in LN₂ at the Irradiated Materials Laboratory (IML) of the Nuclear Aerospace Research Facility of General Dynamics until tested.

A model TT-D split-console-type Instron machine was used for the tensile testing. To attain the desired test temperature, the specimens were immersed in LN₂ (140°R),

sprayed with a LN_2 - GN_2 mixture (340°R), held at room temperature (540°R) or heated in a Marshall tube furnace (740°R and higher). Times required to heat the specimens from 140°R to the test temperature were 20 minutes to 340°R , 5 minutes to 540°R and 10 minutes to 740°R . The latter two warm-ups were made in a water bath to 540°R .

A temperature calibration between the specimen grip assembly and a thermocoupled specimen was made prior to testing at 740°R . The grip temperature was then used to control the furnace temperature. A similar procedure was used at 340°R . Here the temperature was maintained by controlling the amount of cold nitrogen gas impinging on the specimen from a perforated coil surrounding the specimen and thermocoupled grip assembly⁽⁵⁾.

For the annealing studies, the specimens were heated to the anneal temperature with approximately the same heating ramps as the "at temperature" tests for the desired length of time and returned to LN_2 for testing.

Crosshead speed during testing was 0.02 inch per minute. All data shown in this report were obtained from the Instron load-deflection charts. These are sufficiently accurate for the required data. Uniform elongations obtained are chart plastic deflections to maximum load. Bench measurements of total elongation were made but the results were very similar to the chart values and are not reported here.

Data reduction was by General Dynamics. The raw data and details of the test procedures are given in their report⁽⁴⁾.

2.0 TEST RESULTS AND DISCUSSION

Results were analyzed by the WANL Reliability Department to determine the response of the data to the test variables. Details and results of the analyses can be found elsewhere⁽⁵⁾. Generally, the analysis of variance method was used with response compared at the 0.05 significance level.

Data obtained from each specimen are shown in Table 2. The predicted data values from the statistical analysis are shown for each property in Tables 3 through 6*. These results will be used in the following discussion. Also shown are "pooled" standard deviations, effective sample sizes, degrees of freedom and 95/99 lower limit values. No statistical analysis was made of the annealing specimen data. The test results for these specimens are reported in Table 7.

2.1 YIELD STRENGTH

The yield strength of Inconel 718, as shown in Figure 2, increased rapidly with fluence, particularly at the lowest temperatures. After 10^{19} n/cm², $E > 1$ MeV, the changes ranged from 31% at 140°R to 3.5% at room temperature. In a previous related study on alloy A-286, the yield strength was found to increase as the square root of the fluence⁽⁶⁾. For this material, however, a cube root dependence was found (Figure 3). This dependence was predicted by Blewitt and Coltman⁽⁷⁾ as due to the effect of the mean separation of defects produced by irradiation on yielding.

The interaction of the test temperature and fluence on the yield strength is shown in Figure 4. Only a slight strengthening was found at room temperature and above. In a previous ambient irradiation test, a slight (~5%) reduction in yield strength was found for specimens from the same lot after irradiation in the Plum Brook Reactor⁽³⁾. The slight increase found in this experiment (at 540°R) substantiated earlier cryogenic irradiation test results⁽²⁾. However, the increase was only a few percent and is much lower than that found for other similar materials^(2,6).

* Tables 2 through 6 are taken from DRM 54142.

TABLE 2

TENSILE TEST DATA FOR SPECIMENS OF INCONEL 718 IRRADIATED AT 140°R

Spec. Number	Heat Treat. Temp. (°F)	Neutron* Fluence E > 1 MeV (n/cm ²)	Test Temp. (°R)	0.2% Offset Yield Strength (KSI)	Ultimate Strength (KSI)	Uniform Elong. (%)	Total Elong. (%)
712	1750	0	140	180.6	246.2	23.1	28.1
901	1950	0	140	180.3	238.2	26.2	28.2
716	1750	4.49x10 ¹⁷	140	192.3	240.6	20.5	23.8
905	1950	4.49x10 ¹⁷	140	199.4	234.3	22.2	22.2
720	1750	2.46x10 ¹⁸	140	214.6	250.3	12.6	21.5
909	1950	2.46x10 ¹⁸	140	226.8	251.9	11.5	15.3
724	1750	9.76x10 ¹⁸	140	240.9	268.4	6.38	14.5
913	1950	9.77x10 ¹⁸	140	231.4	251.6	3.63	10.3
713	1750	0	340	181.8	220.5	15.8	22.1
902	1950	0	340	181.3	215.2	15.6	17.5
717	1750	4.49x10 ¹⁷	340	189.8	217.4	15.0	20.8
906	1950	4.49x10 ¹⁷	340	181.6	206.2	18.3	22.2
721	1750	2.46x10 ¹⁸	340	199.1	217.0	9.3	17.0
910	1950	2.46x10 ¹⁸	340	190.5	203.6	13.2	17.7
734	1750	9.84x10 ¹⁸	340	203.9	219.0	3.2	11.0
914	1950	9.80x10 ¹⁸	340	195.9	208.7	2.0	9.5
714	1750	0	540	156.7	204.9	17.9	23.1
918	1950	0	540	175.3	206.2	16.9	18.5
718	1750	4.49x10 ¹⁷	540	166.0	201.5	13.5	13.6
907	1950	4.49x10 ¹⁷	540	164.0	186.1	16.3	18.4
722	1750	2.46x10 ¹⁸	540	167.9	198.1	11.6	19.6
911	1950	2.46x10 ¹⁸	540	172.5	189.0	15.9	19.0
726	1750	9.73x10 ¹⁸	540	173.9	189.4	7.3	14.2
915	1950	9.74x10 ¹⁸	540	175.6	186.7	2.3	11.3
715	1750	0	740	163.6	194.5	14.9	17.6

* Corresponding thermal fluence (E < 0.48 eV) levels were 0, 1.7×10^{16} and 2.1×10^{17} n/cm² for the three fast fluence levels, respectively.

TABLE 2 (CONTINUED)

<u>Spec. Number</u>	<u>Heat Treat. Temp. (°F)</u>	<u>Neutron Fluence E > 1 MeV (n/cm²)</u>	<u>Test Temp. (°R)</u>	<u>0.2% Offset Yield Strength (KSI)</u>	<u>Ultimate Strength (KSI)</u>	<u>Uniform Elong. (%)</u>	<u>Total Elong. (%)</u>
904	1950	0	740	154.1	183.3	17.3	19.3
719	1750	4.49x10 ¹⁷	740	160.5	192.9	16.6	20.1
908	1950	4.49x10 ¹⁷	740	150.6	173.6	15.4	17.7
723	1750	2.46x10 ¹⁸	740	158.8	185.6	13.1	16.3
912	1950	2.46x10 ¹⁸	740	161.1	181.5	14.3	14.6
727	1750	9.83x10 ¹⁸	740	168.1	187.3	9.9	14.6
916	1950	9.81x10 ¹⁸	740	156.4	176.0	6.2	9.7

TABLE 3

0.2% OFFSET YIELD STRENGTH OF INCONEL 718 IRRADIATED AT 140°R

(Average and 95/99 Lower Limit Values in KSI)

Fast Fluence (n/cm²)

Test Temp. °R	0			4.49 x 10 ¹⁷			2.46 x 10 ¹⁸			9.78 x 10 ¹⁸			
	$\bar{X}$	LL	N	$\bar{X}$	LL	N	$\bar{X}$	LL	N	$\bar{X}$	LL	N	
140	180.86	160.67	3.007	199.66	181.05	7.017	218.46	199.85	7.017	237.25	217.25	3.007	N is the effective sample size. s = 5.598 with f = 26 degrees of freedom
340	176.78	157.70	5.195	184.38	166.34	12.121	191.98	173.95	12.121	199.59	180.51	5.195	
540	168.36	149.28	5.195	170.31	152.28	12.121	172.27	154.23	12.121	174.23	155.15	5.195	
740	155.60	135.41	3.007	157.45	138.84	7.017	159.31	140.70	7.017	161.17	140.98	3.007	

TABLE 4

ULTIMATE STRENGTH OF INCONEL 718 IRRADIATED AT 140°R

(Average and 95/99 Lower Limit Values in KSI)

Fast Fluence (n/cm²)

Test Temp. °R	Heat Treat. Temp. °F	0			4.49 x 10 ¹⁷			2.46 x 10 ¹⁸			9.78 x 10 ¹⁸		
		$\bar{X}$	LL	N	$\bar{X}$	LL	N	$\bar{X}$	LL	N	$\bar{X}$	LL	N
140	1750	243.47	227.81	2.532	246.17	231.63	4.878	253.02	238.48	4.878	264.03	248.36	2.532
140	1950	234.63	218.96	2.532	237.33	222.79	4.878	244.18	229.64	4.878	255.18	239.52	2.532
340	1750	223.94	209.09	3.922	218.00	203.87	6.897	216.21	202.08	6.897	218.57	203.73	3.922
340	1950	215.10	200.25	3.922	209.15	195.02	6.897	207.36	193.23	6.897	209.73	194.88	3.922
540	1750	207.68	192.84	3.922	198.69	183.87	6.897	193.86	179.73	6.897	193.18	178.33	3.922
540	1950	198.84	183.99	3.922	189.85	175.72	6.897	185.01	170.88	6.897	184.33	169.48	3.922
740	1750	194.71	179.04	2.532	188.26	173.72	4.878	185.97	171.43	4.878	187.84	172.17	2.532
740	1950	185.87	170.20	2.532	179.42	164.88	4.878	177.13	162.59	4.878	178.99	163.32	2.532

N is the effective sample size. s = 4.205 with f = 24 degrees of freedom.

NOTE: Tables 3 through 6 taken from Reference 5.

TABLE 5

UNIFORM ELONGATION OF INCONEL 718 IRRADIATED AT 140°R
(Average and 95/99 Lower Limit Values in %)

Fast Fluence (n/cm^2)

Test Temp. °R	Heat Treat. Temp. °F	0			4.49×10^{17}			2.46×10^{18}			9.78×10^{18}		
		$\bar{X}$	LL	N	$\bar{X}$	LL	N	$\bar{X}$	LL	N	$\bar{X}$	LL	N
140	1750	23.4	17.2	2.312	19.3	13.5	4.124	13.5	7.7	4.124	6.0	0.0	2.312
140	1950	24.2	18.0	2.312	21.8	16.0	4.124	14.8	8.9	4.124	3.1	0.0	2.312
340	1750	17.2	11.1	2.920	14.5	8.6	4.301	10.0	4.1	4.301	3.7	0.0	2.920
340	1950	18.0	12.0	2.920	16.9	11.1	4.301	11.2	5.4	4.301	0.9	0.0	2.920
540	1750	16.4	10.4	2.920	15.0	9.1	4.301	11.8	5.9	4.301	6.9	0.8	2.920
540	1950	17.2	11.2	2.920	17.4	11.6	4.301	13.0	7.2	4.301	4.0	0.0	2.920
740	1750	15.2	8.9	2.312	15.1	9.2	4.124	13.2	7.3	4.124	9.6	3.3	2.312
740	1950	16.0	9.7	2.312	17.5	11.7	4.124	14.4	8.6	4.124	6.7	0.4	2.312

N is the
effective sample
size.
s = 1.648 with
f = 22 degrees of
freedom

TABLE 6

TOTAL ELONGATION OF INCONEL 718 IRRADIATED AT 140°R
(Average and 95/99 Lower Limit Values in %)

Fast Fluence (n/cm^2)

Test Temp. °R	0			4.49×10^{17}			2.46×10^{18}			9.78×10^{18}		
	$\bar{X}$	LL	N	$\bar{X}$	LL	N	$\bar{X}$	LL	N	$\bar{X}$	LL	N
140	25.8	17.4	6.107	23.0	15.0	13.559	18.3	10.3	13.559	11.7	3.3	6.107
340	23.0	15.0	13.559	21.2	13.3	15.686	17.5	9.6	15.686	11.9	4.0	13.559
540	20.2	12.3	13.559	19.4	11.5	15.686	16.8	8.8	15.686	12.2	4.2	13.559
740	17.5	9.1	6.107	17.7	9.7	13.559	16.0	8.0	13.559	12.4	4.0	6.107

N is the effective
sample size
s = 2.501 with
f = 27 degrees
of freedom

Figure 2. Change in Yield Strength of Inconel 718 vs. Fluence

Figure 3. Yield Strength vs. Cube Root of Fluence at 140°R

Figure 4. Irradiation Effect on the Yield Strength of Inconel 718

The yield strength was not significantly different for the two material heat treatments.

2.2 ULTIMATE TENSILE STRENGTH

The 1750°F solution treatment resulted in statistically significantly higher yield strength values. The difference, however, was only about 5 percent.

Like the yield strength, the ultimate strength increases with fluence when measured at 140°R. However, at higher test temperatures the strength decreases significantly as shown in Figure 5. The data indicate that the turnaround occurs somewhere below 340°R. Again above 340°R there is another turnaround as indicated by the reduced effect at 740°R. The latter effect is credited to removal of defects by annealing while the former is probably due to the effect of defect size and distribution on the ability of the material to yield homogeneously.

There also appears to be a maximum in the fluence effect at 340°R and above at about $5 \times 10^{18} \text{ n/cm}^2$.

The decrease in ultimate strength was found in a previous experiment and has been reported in the literature⁽³⁾. In all cases, this effect is found to be small at the fluences achieved, ~7 percent maximum. The turnaround at 5×10^{18} indicated the effect will not become more serious at higher fluences.

2.3 UNIFORM ELONGATION

The 1950°F heat treatment resulted in slightly higher uniform elongations. However, this condition was also affected to a greater extent by irradiation so that at the highest fluence level the 1750°F solution treated material had a significantly higher uniform elongation. This effect has been credited in a previous report⁽³⁾ as due to an increased grain size in the higher temperature treated material. Grain growth in Inconel 718 occurs above 1850°F and resulted in a grain size of ASTM No. 3 for the 1950°F solution treated material versus ASTM No. 6 1/2 for those solution treated at 1750°F.

The uniform elongation decreases quite drastically with irradiation. The maximum change, 95 percent, occurred for the 1950°F solution treated specimen at 340°R after $9.8 \times 10^{18} \text{ n/cm}^2$, $E > 1 \text{ MeV}$. The absolute value at this condition was only 0.9 percent. Some recovery occurs at higher temperatures.

Figure 5. Percent Change in Ultimate Tensile Strength vs. Fast Fluence at Various Test Temperatures

2.4 TOTAL ELONGATION

The total elongation did not show an effect of heat treatment. The property gradually decreases with increasing test temperature. The effect of fluence was quite pronounced, resulting in a reduction which reached a maximum at the highest fluence and lowest temperature. The maximum change was -55 percent. These effects are shown in Figure 6. While the yield strength values indicate almost complete recovery above 540°R , only about one-half the total elongation was recovered by 740°R .

While the total and uniform elongations decreased with fluence, the difference between the two increased. This increase indicated no loss in the ability of the material to deform inhomogeneously and the total elongation decrease is credited to a loss in homogeneous plastic deformation.

2.5 ANNEALING STUDIES

Several irradiated specimens were annealed at temperatures up to 1140°R for times up to 1000 minutes before being tested at 140°R . Data obtained are shown in Table 7. The yield strength results are plotted in Figure 7. Referring to the 100 minute curve, recovery appears to occur in one large stage between 340 and 1140°R . The maximum slope occurs at about 740°R . These results are significantly different from the "at temperature" results shown in Figure 3 where almost no effect was measured at 540°R . Apparently the defect state in the material interacts with the test temperature.

The ultimate strength increases with anneal temperature to about 540°R before beginning to recover, while the elongation recovers gradually to 940°R where the control and irradiated values are very similar.

Figure 6. Total Elongation of Inconel 718 vs. Test Temperature at Various Fluence Levels

TABLE 7

ANNEALING TEST RESULTS
 Fluence = 10^{19} n/cm² E > 1 MeV
 Test Temperature = 140°R

Specimen No.	Anneal Time (min)	Anneal Temperature °R	Yield Strength (ksi)	U. T. S. (ksi)	Uniform Elongation (%)	Total Elongation (%)
701		No Anneal	232.9	254.3	5.8	14.6
702		No Anneal	234.5	255.7	6.9	14.5
703	10	340	226.3	252.0	10.0	19.4
704	100	340	235.0	261.0	9.7	19.2
705	10	540	226.8	264.7	15.0	23.6
706	100	540	226.8	264.3	13.1	20.5
709	1000	540	214.7	248.0	16.5	21.6
710	100	940	198.1	247.9	21.7	28.6
711	100	1140	191.5	254.3	26.8	29.0
Unirradiated Values			180.9	243.5	23.4	25.8

Figure 7. Recovery of Yield Strength in Irradiated and Annealed Inconel 718

3.0 CONCLUSIONS AND SUMMARY

- 1) The 1750°F solution treatment and associated age is recommended for low temperature applications of Inconel 718. This treatment results in higher strength and improved resistance to radiation embrittlement.
- 2) The yield strength of Inconel 718 increased as the cube root of fluence below 540°R.
- 3) The ultimate tensile strength increases with fluence at 140°R but decreases at higher test temperatures.
- 4) Both the uniform and total elongations are decreased significantly by irradiation over the entire test temperature range. However, the difference between the two did not decrease.
- 5) None of the property changes should seriously degrade the applicability of Inconel 718 in a reactor environment. This conclusion applies to temperatures from 140°R to 740°R and fluences of up to 10^{19} n/cm^2 , $E > 1 \text{ MeV}$.

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ABSTRACT		
<p>Tensile specimens of Inconel 718 sheet were irradiated at 140°R and tested at temperatures up to 740°R. Maximum fluence obtained was 10^{19} n/cm², E > 1 MeV. Yield strengths increased below 540°R while ultimate strengths decreased slightly. A significant embrittlement also occurred at all test temperatures. However, the effects noted should not limit the applicability of the alloy over the range of test conditions studied.</p>		
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