

WANL-TME-1817 July 10, 1968

# MASTER

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EXPERIMENT DESIGN FOR GTR-20 RADIATION EFFECTS TEST OF INCONEL-718, 37/W410, 15/W101, & 15/W202



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# MASTER



#### EXPERIMENT DESIGN FOR GTR-20

#### RADIATION EFFECTS TEST OF

INCONEL-718, 37/W410, 15/W101, & 15/W202







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#### I. Experimental Purpose

The purpose of this irradiation is to define the limitations of Inconel-718 as it is used in the NERVA program. The principal applications of this material in the R-1 design are as counter-flow tie tubes (support stems) and lateral support springs (Figure 1). The range of temperatures that the support stems will experience during steady-state operation are from 200°R to 1000°R (Reference 1). The range of fast (E > 1 MeV) neutron fluences that the tie stems will experience are from 1 x  $10^{18}$  to 2.9 x  $10^{19}$  nvt (Reference 2). This is based on a ground test having 100 minutes of equivalent time at full power. The uncertainty in the fast fluence as determined by WANL Thermal and Nuclear Design is +50%. The thermal neutron fluence experienced by tie stems ranges from 2.1 x  $10^{15}$  to 5.3 x  $10^{18}$  nvt (Reference 2). These fluences are also subject to a +50% uncertainty as defined by WANL Thermal and Nuclear Design. Previous test data have indicated a loss in ductility of 718 at cryogenic temperature irradiation. This is due primarily to the fast neutron displacement generation and its stability in the material at the cryogenic temperature. There is also a loss in ductility of this material at temperatures above 1500°R (Reference 6). This is a thermal neutron fluence effect resulting from helium production originating in boron impurities as  $B^{10}(n, \alpha) Li^7$ . At temperatures near 1500°R this helium migrates toward the Inconel 718 grain boundaries. This irradiation will define the extent of



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Strategic Contraction

### CONFIDENTIAL RESTRICTED DATA



#### I. (Continued)

these losses of ductility as well as other material properties useful for NERVA design conditions. The tie stems will utilize a silicone cement (RTV-891) as a sealant in the end caps. The sealing effectiveness of this sealant must be known for a pressure of 210 psid after a gamma dose of  $10^{12}$  ergs/gram carbon.

The lateral support springs are limited to a temperature of  $1300^{\circ}$ R (Reference 1). The fast neutron (E > 1 MeV) fluence experienced by these lateral support springs ranges from 1.3 x  $10^{18}$  to 7.2 x  $10^{18}$  nvt (Reference 2). Here again, there is a  $\pm 50\%$  uncertainty on these fluences. There is evidence that some materials undergo stress relaxation during irradiation to neutron fluences of  $10^{17}$  nvt and higher (Reference 3). It is necessary to determine the extent of stress relaxation in the Inconel-718 lateral support springs and therefore to determine any changes in bundling forces on the NERVA core during operation.



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II. Experimental Objectives

Following are objectives for 37/W410.

To determine for Group C-I:

- 1. 0.2% tensile yield strength
- 2. ultimate tensile strength
- 3. elongation and reduction of area
- As a function of:
- 1. test temperature between 1250 and 2000°R
- 2. thermal neutron fluence between 0 and 5 x  $10^{19}$  nvt
- 3. grain size from 5 to 7 ASTM units
- 4. heat treatment at 1750 versus heat treatment at 1950°F

To determine for Group C-II, C-III, and C-IV:

- 1. 0.2% tensile yield strength
- 2. ultimate tensile strength
- 3. elongation and reduction of area

As a function of:

- 1. test temperature between 140 and 740°R
- 2. fast neutron fluence between 0 and 1 x  $10^{19}$  nvt
- 3. grain size from 5 to 7 ASTM units
- 4. heat treatment at 1750 versus that at 1950°F
- 5. annealing temperatures between 340 and 740°R (at a fast neutron fluence of  $1 \times 10^{19}$  nvt)



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II. (Continued)

- 6. annealing times between 10 and 1000 minutes (at a fast neutron fluence of  $1 \ge 10^{19}$  nvt)
- 7. notched versus unnotched specimens

To determine for Group E-I, 15/W101

- 1. stress relaxation in lateral support springs
- As a function of:
- 1. irradiation temperature between 140 and 1200°R
- 2. fast neutron fluence between 0 and 5 x  $10^{18}$  nvt
- 3. spring load between 0 and 130 lbs.
- To determine for Tubular I and II, 15/W202
- 1. 0.2% tensile yield strength
- 2. ultimate tensile strength
- 3. elongation and reduction of area

As a function of:

1. test temperature from 140 to 2000°R at a thermal neutron fluence of  $1 \times 10^{19}$  nvt and a fast neutron fluence of  $1 \times 10^{19}$  nvt

To determine for Tubular III, 15/W202

 the sealing effectiveness of RTV-891 silicone cement used in the cemented end caps of these specimens at a gamma dose of 10<sup>12</sup> ergs/gm-carbon for hydrogen pressures of 210 and 315 psig.







II. (Continued)

There will be at least seven specimens of Inconel 718 bar material, coarse grain size, 1750°F solution anneal, 10<sup>19</sup> nvt, tested at 140°R with no intervening warm-up.

The distribution of these specimens identified as specimen numbers 291, 489, 490, 494, 497, 498, and 502 will also be treated to a separate statistical analysis.







#### III. Analysis Procedures

Standard techniques to perform an analysis of variance will be used to determine the material properties as a function of the independent variables discussed in Section II and the random variability of the specimens. These analyses will be performed by the WANL Reliability Department, and reference as to their use was found in many statistics text books, such as References 4 and 5. The matrices for each group of specimens used in the analysis of variance are given in Appendix A. A fractional factorial design is used. Results will be given in the form of functional equations expressing the property of interest as a function of the variables considered in the experiment plans. The standard deviation associated with the random variability of each property will also be given.







#### IV. Requirements for Experimental Data

#### A. Accuracy Requirements

The neutron fluence for specimens with the same fluence requirement in any given group shall be within  $\pm 5\%$ . Whenever a group has two required fluences (for example, group A-I has 24 specimens that require  $5 \times 10^{18}$  nvt and 12 specimens that require 1 x  $10^{19}$  nvt), the ratio of high to low fluence shall be 2.00  $\pm 10\%$ . Variation in fluence between groups shall be within  $\pm 30\%$ . The gamma dose for sealant specimens shall be within  $\pm 10\%$ .

The gamma dose of all specimens shall be reported to within  $\pm 50\%$  to comply with the NERVA Trend Data Retrieval Program.

The Instron machine load shall be recorded within +0.5%.

All elongation measurements shall be made to +0.5%.

Reduction of area measurements shall be to +1%.

Room temperature shall be recorded to the nearest °R.

The Instron machine grip temperatures shall be monitored with thermocouples, and they should be recorded to within  $\pm 1^{\circ}R$ . The specimen temperature shall be accurate to  $\pm 5^{\circ}R$ .

Annealing temperatures shall be accurate to  $\pm 2^{\circ}$ R in the cryogenic range and to  $\pm 5^{\circ}$ R at temperatures equal to or greater than room temperature.







The annealing times shall be accurate to +1%.

The temperature of the three Inconel 718 springs to be irradiated at 1200°R (See Appendix A, Group E-1) shall be accurate to within  $\pm 10^{\circ}$ R.

#### B. Prediction of Results

The ultimate and yield strengths will increase at cryogenic temperatures and also slightly at elevated temperatures after irradiation (Reference 6). Elongation at cryogenic temperatures is expected to decrease as previously determined at lower fluences. Elongation will also decrease above 1500°R as indicated previously and elongations <5% are expected at the high fluences.







#### V. Experimental Procedures

#### A. Specimen Irradiation Fluence and Temperature

All specimens shall be irradiated at 140°R in  $IN_2$  except 5 Inconel 718 springs, 3 of which shall be irradiated at 1200°R and 2 of which shall be irradiated at 540°R. The following table shows the required neutron fluence for each specimen to be irradiated and the group it is in. These group numbers correspond to those in Appendix A, the tabulation of specimen groups.

Specimen Group	Number of Specimens To Be Irradiated	Required Fast Neutron Fluence (nvt)
C-I	12 (Including 4 spares)	$5 \times 10^{18}$ *
C-I	24 (Including 8 spares)	5 x 10 <sup>19</sup> *
C-II	12 (Including 4 spares)	$5 \times 10^{18}$
C-II	24 (Including 8 spares)	$1 \times 10^{19}$
C-III	36	1 x 10 <sup>19</sup>
C-IV	12 (Including 4 spares)	1 x 10 <sup>19</sup>
Tubular I	9	5 x 10 <sup>19</sup> *
Tubular II	9	1 x 10 <sup>19</sup>
Tubular III	6	$1 \times 10^{12} \text{ ergs/gm-carbon}$
E-I (Springs)	7	$5 \times 10^{18}$

\* Thermal Neutron Fluence



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The specimens scheduled to receive the high fluence of  $1 \ge 10^{19}$  nvt shall be placed on the inner surface of the dewar nearest the North face of the core within a 5 inch radius circle, as shown in Figure 6.\* The center of the circle represents the projection of the core center. The dashed lines indicate the regions containing Inconel-718 tubular specimens Group II, which are directly underneath (or slightly farther away than) the Group II and III bar specimens. The specimens scheduled to receive the low fluence of  $5 \ge 10^{18}$  nvt shall be placed on a spherical surface (as shown in Figure 7) that is 5 inches North of the inner surface of the dewar on a horizontal line through the core center perpendicular to the core North face and that intersects a 10 inch radius circle on the inner surface of the dewar. This surface may be modified slightly and its deviation from spherical defined as a result of further dosimetry analysis and calculation of the attenuation from the high fluence specimens.

The neutron fluence for specimens with the same fluence requirement in any given group shall be within  $\pm 5\%$ . The ratio of high to low fluence shall be 10  $\pm 10\%$  and 2  $\pm 10\%$  for groups C-I and C-II, respectively. Variation in fluence between groups shall be  $\pm 30\%$ .





<sup>\*</sup> Based on estimated flux distribution provided by General Dynamics, Fort Worth





Specimen dimensions are approximated in the following table to demonstrate the space each specimen will occupy. The table also gives the drawing number(s) associated with each specimen for the exact dimensions. These drawings are Figures 8 through 12.

	Type of Inconel 718 Specimen			
Approximate	Bar	Springs	Tul	bular Look Test
Dimensions (inches)	Specimens	Springs	Teustre	Leak lest
Length	3.60	5.0	7.15	5.2
Width	1.0		1.0	1.0
Thickness (or Diameter)	0.26	1.13	0.5	0.5
Drawing Number(s)	8010377	979D482	981D477	<b>981</b> D477
		Fig. 10	388D407	388D407
Number at Indicated Neutron Fluence (nvt)				
Fast				
5 x 10 <sup>18</sup>	12	7		
1 x 10 <sup>19</sup>	72		9	
Thermal				
5 x 10 <sup>18</sup>	12			6
5 x 10 <sup>19</sup>	24		9	
Number of Unirradiated	36	7	8	4









#### B. Tensile Testing (Instron Machine)

Each specimen shall be held at its planned test temperature for 15 minutes before starting the crosshead. This time is approximate and may be changed by the WANL monitor at the time of testing. The Instron machine crosshead speed shall be 0.05 inches/min. for specimens tested at temperatures between 140 and 740°R and 0.002 inches/minute for specimens tested at temperatures between 1250 and 2000°R. Alignment checks shall be made intermittently during tensile testing. This shall be accomplished with three strain gages mounted 120° apart around the circumference of the gage section of a dummy specimen. The WANL monitor will determine whether the alignment of the system is adequate.

#### C. Metallography

Macrographs with a magnification of 2 to 4 shall be taken of the fractured surface of specimens numbered 295, 313, 316, 318, 363, 364, 371, 386, 387, and 393. (See Appendix A for specimen numbering.)

Electron fractographs (having suggested magnification 6000) of the fractured surface and microphotographs (having magnification 100 and 500) of the polished longitudinal area including the fracture shall be taken of specimens numbered 291, 300, 387, and 393. WANL shall receive replicas of the electron fractographs.



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#### D. Data Reduction

The data to be taken shall be that outlined in Sections A and C plus the following: The ultimate tensile and 0.2% offset tensile yield strengths, the Instron chart elongation, the plastic elongation at maximum stress, the total plastic elongation obtained with bench measurement, and reduction of area using photography methods and/or micrometer measurements. Data shall be based on actual WANL reported gage length. Original or legible reproduced copies of all loaddeflection curves, temperature recording curves, and raw data tabulations are to be made available to WANL as soon after testing as possible.

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#### E. Pedigree Specimens

Several unirradiated Inconel-718 specimens will be tested at WANL at various temperatures after fabrication. These specimens will be manufactured to the same specifications as those shipped to GD/FW.

The following characterization of the Inconel-718 material scheduled for inclusion into the GTR-20 irradiation program has been obtained to date. Two heats of material will be used. One is Allvac Metals Co. Heat No. 5734 and the other is Latrobe Steel Company Heat No. C-57275. The chemistry of these materials is as follows:





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	WANL PDS	HT	. No. 5734	HT.	No. C-57275
Element	<b>30038-</b> D	Vendor	Check Analysis	Vendor	Check Analysis
В	0.006 max.	0.004	0.0022	0.004	0.0025
Р		0.002	0.001	0.003	0.001
S	0.015 max.	0.005		0.004	
C	0.10 max.	0.052		0.03	
Cu	0.15 max.	0.03	0.008	0.01	0.023
Co	0.20 max.	0.17	0.19	0.14	0.14
Mn	0.50 max.	0.07	0.011	Trace	0.033
Si	0.75 max.	0.12	0.04	0.07	0.09
Al	0.40-1.00	0.50	0.74	0.46	0.72
Ti	0.65-1.15	0.97	1.17	1.00	1.12
Мо	2.80-3.30	2.98	3.06	3.01	3.04
Nb+Ta	4.50-5.75	5.10	5.05	5.48	5.31
Cr	17.00-21.00	18.7	18.7	18.77	18.7
Ni	50.00-55.00	52.0	52.2	53.1	53.6
Fe	Remainder				

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Two heat treatments will be used. Heat Treatment No. 1 consists of a one (1) hour solution treatment at 1750°F, an air cool to room temperature then eight (8) hours at 1325°F followed by a furnace cool to 1150°F and a hold for a total age time of 18 hours before air cooling to room temperature. Heat Treatment No. 2 consists of

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a solution treatment for one (1) hour at 1950°F followed by an air cool to room temperature. The material is then aged for 10 hours at 1400°F, furnace cooled to 1200°F and held for a total age time of 20 hours before air cooling to room temperature. These heat treatments produce the following grain sizes in the two heats of material:

Heat No.	Heat Treatment	<u>Grain Size</u>
Allvac 5734	No. 1	51
Allvac 5734	No. 2	4
Latrobe C-57275	No. l	8
Latrobe C-57275	No. 2	5

#### F. Disposition of Spare Specimens

Spare specimens will be used to replace damaged specimens if there are any. If spares are not used to replace damaged specimens they will be used for additional replication at the most stringent test temperature (140°R, or as determined by the WANL monitor).

#### G. Inconel 718 Leak Test Specimens

The 10 Inconel 718 leak test specimens shall be pressurized with hydrogen or helium to 210 and 315 psig with room temperature hydrogen. The leakage rates shall be determined before and after irradiation.



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#### H. Inconel 718 Springs (Stress Relaxation)

The free length and compressed length shall be measured for specimens irradiated at  $IN_2$  before and after irradiation at  $IN_2$  temperature. The spring constants shall be measured at WANL before irradiation and shall be measured at GD/FW after irradiation. These specimens and all others will be measured for the same properties at room temperature.

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#### APPENDIX A

The following pages give tabulation of all Inconel-718 specimens, the group number of each group, and the independent variables.





#### C-I Inconel-718 - Plain Tensile

#### Test Conditions

Specimen No. & Test Order	Grain Size	Heat <u>Treatment</u>	Fluence (Thermal) (nvt)	Test Temperature (°R)
304	<5	1750°F	0	1250
<b>39</b> 3				1750
292			5 x 10 <sup>18</sup>	1500
377				2000
386			5 x 1019	1250
374				1500
387				1750
363				2000
379		1950°F	•	1500
384				2000
389			5 x 10 <sup>18</sup>	1250
383				1750
365			¥ 5 x 10 <sup>19</sup>	1250
394				1500
366				1750
372		V		2000
370	>7	1750°F	¥ o	1500
371			$\checkmark$	2000
390	¥	V	5 x 10 <sup>18</sup>	1250



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#### C-I Inconel-718 - Plain Tensile (continued)

#### Test Conditions

Specimen No. & <u>Test Order</u>	Grain Size	Heat Treatment	Fluence (Thermal) (nvt)	Test Temperature (°R)
388	>7	1750°F	5 x 10 <sup>18</sup>	1750
381			5 x 10 <sup>19</sup>	1250
385				1500
376				1750
378			V	2000
373		1950°F	0	1250
380			$\checkmark$	1750
367			5 x 10 <sup>18</sup>	1500
369			$\checkmark$	2000
368			5 x 10 <sup>19</sup>	1250
382				1500
391				1750
375	V	¥	$\checkmark$	2000
			-	

#### Spare Specimens

#### C-I Inconel-718 - Plain Tensile

Specimen No.	Grain Size	Heat Treatment	Fluence (Thermal) (nvt)
471	<5	1750°R	0
472			5 x 10 <sup>18</sup>
473	V	$\checkmark$	5 x 10 <sup>19</sup>
NEO III		A-3	
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#### Spare Specimens

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No.

#### C-I Inconel-718 - Plain Tensile (continued)

Specimen No.	Grain Size	Heat Treatment	Fluence (Thermal) (nvt)
474	<5	1750°F	5 x 10 <sup>19</sup>
475		1950°F	0
476			5 x 10 <sup>18</sup>
477			5 x 10 <sup>19</sup>
478			$5 \times 10^{19}$
479		♥ 1750°F	0
480			5 x 10 <sup>18</sup>
481			5 x 10 <sup>19</sup>
482			5 x 10 <sup>19</sup>
483		<b>V</b> 1950°F	0
484			5 x 10 <sup>18</sup>
485			5 x 10 <sup>19</sup>
<b>48</b> 6	V		5 x 10 <sup>19</sup>
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#### C-II Inconel-718 - Plain Tensile

#### Specimen No. Fluence Test Grain Fast Temperature Heat & Test Order Size Treatment (nvt) (•R) 1750°F <5 n 5 x 10<sup>18</sup> 1950°F 5 x 10<sup>18</sup> 1750°F >7 5 × 10<sup>18</sup>

#### Test Conditions

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#### C-II Inconel-718 - Plain Tensile (Continued)

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Test	Condi	Lti	ons
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#### C-II Inconel-718 - Plain Tensile



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#### Spare Specimens

#### C-II Inconel-718 - Plain Tensile (Continued)

Specimen No.	Grain Size_	Heat Treatment	Fluence Fast _(nvt)
493	<5	1950°F	1019
494		1750°F	1019
<b>49</b> 5		9	0
496			5 x 10 <sup>18</sup>
497			1019
498		$\checkmark$	1019
499		1950°F	0
500			5 x 10 <sup>18</sup>
501			10 <sup>19</sup>
502		₹ 1750°F	10 <sup>19</sup>
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#### C-III Inconel-718 - Plain Tensile - Annealing Study (Fast Fluence = 1 x 10<sup>19</sup> nvt)

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#### Test Conditions

Specimen No. & Test Order	Grain Size	Heat Treatment	Annealing Temperature (°R)	Annealing Time (min.)
333	<5	1750°F	340	10
342				100
328			↓	1000
347			540	10
351				100
355			$\checkmark$	1000
335			740	10
344				100
332		Į.	V	1000
358		1950°F	340	10
362				100
361			J.	1000
334			540	10
340				100
329			$\checkmark$	1000
359			740	10
360				100
348	V	Į.	$\checkmark$	1000
338	>7	1750°F	340	10
343				100
327	V	V	V	1000
			<u>s</u>	
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#### C-III Inconel-718 - Plain Tensile - Annealing Study (Fast Fluence = $1 \times 10^{19}$ nvt) (Continued)

#### Test Conditions

Specimen No. & Test Order	Grain Size	Heat Treatment	Annealing Temperature (°R)	Annealing Time (min.)
346	7	1750°F	540	10
352				100
349			$\checkmark$	1000
337			740	10
341				100
330		V	$\checkmark$	1000
350		1950°F	340	10
357				100
353			$\checkmark$	1000
336			540	10
339				100
331			$\checkmark$	1000
354			740	10
356				100
345	V	$\checkmark$	$\checkmark$	1000
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#### C-IV Inconel-718 - Notched Tensile

Specimen No.	Grain	Heat	Fluence Fast	Test Temperature
Test Order	Size	Treatment	(nvt)	(°R)
304	5	1750°F	0	340
322			V	740
290			1019	140
285		V		540
301		1950°F	0	140
287			V	540
311			1019	340
325			$\checkmark$	740
299		1750°F	0	140
290			V	540
306			1019	340
317		V	$\checkmark$	740
314		1950°F	0	340
319			V	740
294			10 <sup>19</sup>	140
282	V	$\checkmark$	$\checkmark$	540

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#### Test Conditions



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#### Spare Specimens

C-IV Inconel-718 - Notched Tensile -  $IN_2$  Irradiated

#### Test Conditions

Specimen No.	Grain Size	Heat Treatment	Dose (Fast) (nvt)
511	5	1750°F	0
512		$\checkmark$	1019
513		1950°F	0
514		V	1019
515		1750°F	0
516		$\checkmark$	1019
517		1950°F	0
518	$\checkmark$	$\checkmark$	1019



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E-I Inconel-718 - Spring Test

Spring No.	Load (1b)	Temp. During Irradiation (°R)	Fluence (Fast) (n/cm <sup>2</sup> )
l	0	1200	0
2	$\checkmark$		5 x 10 <sup>18</sup>
3	65		0
4		V	5 x 10 <sup>18</sup>
5		540	0
6		$\checkmark$	5 x 10 <sup>18</sup>
7		140	0
8			$5 \times 10^{18}$
9	130	1200	0
10			5 x 10 <sup>18</sup>
11		540	0
12		$\checkmark$	5 x 10 <sup>18</sup>
13		140	0
14	V	$\checkmark$	$5 \times 10^{18}$

Inconel-718 Tubular Specimens

Irradiation in  $LN_2$  (140°R)

Group	Neutron Fluence (nvt)	Irradiated	Number of Unirradiated Control Specimens		
Tubular I	$1 \times 10^{19}$ fast	9	2		
Tubular II	$1 \times 10^{19}$ thermal	9	2		
Tubular III (Sealant)	l x 10 <sup>12</sup> ergs/gm	6	4		









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FIGURE 1 R-1 Reactor Assembly









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FIGURE 4 Inconel-718 Bar Specimens

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DESIGN DATA						WORKING CONDITIONS										
ITE_IA	NIRE SI	ZE 5>	00		1.D.	FREE	NO. OF	COILS	IN DIA	ON DIA	SOLID	DIRECTION	RATE (REF)	WORKING	LOAD	2 CONDITIONS
NO.	A	В	AS WOUND	GROUND	REF	LENGTH	ACTIVE	TOTAL	HOLE	SHAFT	HEIGHT	OF COILS	LBS/IN.	LOAD LBS	± LBS	AT LENGTH
	.112	.179	6871005	£ 73+.005	400	3.802	15.4	17.4	-	_	3.120	LEFT	265.0	81.6	4.0	3.494 BASIC
Ľ	±.001	±001	00/2005	012-000	.405	REF	REF	REF			REF	HAND	283.0	133.0	6.0	3.300 BASIC

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FIGURE 5 Inconel-718 Springs



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