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WANL-TME-705

IRRADIATION EFFECTS ON TIE

ROD MATERIALS

(Title Unclassified)

J. S. Thornton

February 28, 1964

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SUMMARY

The tensile and notched tensile properties of four materials were studied for changes as a result of cryogenic irradiation and post-irradiation annealing effects. The program consisted of a total of 280 forged and machined specimens of Inconel X-750, Inconel 718, Hastelloy R-235, and A-286. The Inconel X-750 is the NRX-A tie rod material and is a face-centered cubic, nickel-base, precipitation hardened alloy. The Inconel 718 and Hastelloy R-235 are in the same alloy class as Inconel X-750, while A-286 is an iron base alloy. All four materials are considered suitable for tie rod and other structural applications in NERVA, at least from the standpoint of their properties in a non-nuclear environment.

The irradiation was carried out in liquid nitrogen (-320°F) at the Ground Test Reactor (GTR) of the Nuclear Aerospace Research Facility (NARF) at General Dynamics in Fort Worth, Texas (GD/FW). The irradiation program was designed to emphasize the environment at the thread end of an NRX-A centerline tie rod. Room temperature and 1000°F annealing after irradiation were included from a spectrum of decay heating temperatures typical of flight condition shutdown of NERVA reactors. The dose received by the samples was between 2.4 and $7.1 \times 10^{16} \text{ n/cm}^2$ ($E > 1 \text{ Mev}$), which is low compared to $3 \times 10^{17} \text{ n/cm}^2$ ($E > 1 \text{ Mev}$) for a five-minute full power operation at the thread end of a centerline tie rod. The Inconel X-750 specimens actually received dosage equivalent to that expected on the peripheral tie rods after three to four minutes of operation at 75% of full power.

Slight but significant changes in properties were noted for all materials. Generally, strength increased without loss in ductility for samples tested in situ immediately after irradiation. After storage at room temperature and testing at -320°F , decreases in ductility for Inconel X-750 and Inconel 718 were observed. These decreases in ductility were accompanied by decreases in ultimate strength and increases in yield strength. The original properties of Inconel 718 and Inconel X-750 were recovered after the 1000°F annealing treatment. The reliability of tie rods would not be reduced by the observed changes caused by equivalent dosage, even though the damage mechanism is important and probably applies to all alloys in the class. It is suggested that immediately after irradiation the damage in the material

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consists of a large number of widely scattered point defects consisting of vacancy-interstitial pairs, which inhibit motion of dislocations but do not contribute to nucleation of cracks. This would explain increases in strength without great losses in ductility. After room temperature annealing, the defects have partially clustered and become large enough to pin dislocations causing them to pile up and act as crack nuclei. The observed increases in yield strength and decreases in ultimate strength and ductility can thus be explained. The recovery of original properties after 1000 °F annealing is suggested as being due to the dispersal of the clusters by the uniting of vacancies and interstitials and/or by the diffusion of the two to grain boundaries, sub grain boundaries or other sinks.

Slight property changes in the Hastelloy R-235 were also noted, even though these samples received less than half of the dose of the Inconel X-750 and Inconel 718. Changes in Hastelloy R-235 may have been masked by large variability in starting material. Increases in strength for the in situ tests were especially notable for the Hastelloy R-235 and to some extent for A-286, but properties recovered to original levels after room temperature and 1000 °F annealing. No other significant changes were noted.

It is concluded that all materials had adequate properties after irradiation, and after irradiation plus annealing. However, trends were indicated which could change this conclusion for higher dose levels and/or annealing at a wider variety of temperatures and times. It is necessary that additional work be done in this area, since the dosage achieved was less than that for the test objectives listed in the Appendix for five-minute operation.

INTRODUCTION

Application of Materials Tested

The materials studied for suitability in NERVA structural applications (tie rods in particular) were Inconel X-750, Inconel 718, Hastelloy R-235, and A-286. Inconel X-750 is the NRX-A tie rod material and the others are alternates for this application. The NRX-A tie rod is a most critical application by merit of both its support function in the core and the

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environmental conditions of its exposure. Other potential NERVA applications for these materials include fastening devices, springs, and seals. The reasons for selecting Inconel X-750 as the NRX-A tie rod material with Inconel 718, Hastelloy R-235 and A-286 as alternates are covered in detail in WANL-TME-319. Briefly, the application requires a material which has good ductility and is strong at cryogenic temperatures, and is relatively strong and resistant to corrosive attack by oxygen or hydrogen at elevated temperatures. The only available materials which fit the requirements are precipitation-hardening alloys of the face-centered-cubic class. Among the strongest alloys in this category are Inconel 718 and Hastelloy R-235. Among the alloys about which the most is known are A-286 and Inconel X-750.

The tie rod material receives the highest fast neutron dose of any structural material in the NERVA system, which is $5.2 \times 10^{18} \text{ n/cm}^2$ for a 20-minute full power operation. At the end of the centerline tie rod the dose would be $1.2 \times 10^{18} \text{ n/cm}^2$ ($E > 1 \text{ Mev}$) in 20 minutes. During normal steady state operation, the thread end of the tie rod is exposed to cryogenic temperatures ($\sim -200^\circ \text{F}$), the head end is exposed to moderately elevated temperatures ($\sim 500^\circ \text{F}$), and the tie rod is stressed to levels well below the yield strength. Under maximum conditions of core lateral motion, the tensile and bending stresses can exceed the yield strength causing 1.67% local strain. Evidence is gathering that the combinations of either high or low temperature and/or stress with neutron radiation has an even greater effect on mechanical properties than irradiation of unstressed samples at normal temperatures.^(1, 2) For a flight version of the NRX-A, the tie rod application is complicated by the post operative decay heating cycle during which the maximum tie rod temperature will increase to various levels and have a maximum in excess of 1000°F .

There is legitimate reason for concern that an anneal in this temperature range following irradiation can create a favorable situation for the occurrence of metallurgical changes.

Test Design

The test was designed to provide information on the effect of cryogenic irradiation on the plain bar and notched tensile properties of Inconel X-750, Inconel 718, Hastelloy R-235, and A-286. It was intended that test exposure conditions simulate those of the thread ends of

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the NRX-A tie rods since this is considered the most critical region of the tie rod. Annealing treatments at room temperature and 1000 °F were scheduled to provide information as to the suitability of the material after the decay heating cycle. Two methods of specimen fabrication were employed: 1) hot upsetting and roll threading both ends of a short length of wire and 2) machining to final configuration from bar stock.

Figure 1 is a graphical illustration of the irradiation and testing history of samples. The cryogenic irradiation of 40 samples (two each plain and notched machined samples and three each plain and notched forged samples for each material) to be tested immediately after irradiation (in situ) was preceded by pre-irradiation and post-irradiation control tests on the same number of samples. An additional 96 samples were irradiated in liquid nitrogen and tested at liquid nitrogen temperatures after storage at room temperature, with and without a 1000 °F anneal for one hour, and at room temperature without elevated temperature annealing. Thirty-two specimens were tested for each of the above three conditions (two each plain and notched, forged or machined) and for each of the two control tests for the two temperatures.

PREPARATION FOR TEST

Selection of Materials

Test material was selected from available heats of specification-grade bar and wire. The Inconel X-750 was from the same heat used for tie rods in ND 10202. The chemical specifications together with the mill and check analyses are presented in Table I. It was decided to fabricate specimens from both bar and wire to check the postulate that the fabrication and composition of the material are significant in the final data.

Specimen Design and Fabrication

The configurations of the three basic types of specimens employed in the program are shown in Figure 2. The plain samples were made by both hot upset forging of wire and machining of bar. The small diameter notched sample is the forged sample and the larger diameter

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notched sample is the machined sample. The gage length of the machined samples was 1.9" to differentiate them from the forged specimens which had 2" gage length. The gage length of the forged samples was required to be 2" because of the fabrication procedure. The ratio of minor to major diameter of all the notched specimens was approximately 0.7" to optimize the stress concentration effect.⁽³⁾ The root radii of the notches were between 0.001 and 0.0015" so that the resultant combined stress concentration factor was between 4 and 5. All samples used in this program passed stringent dye penetrant examination.

Heat Treatment

All specimens were heat treated in vacuum using argon gas for backfill to speed cooling rates. The following furnace cycles were employed:

	<u>Solution Treatment</u>	<u>Aging Treatment</u>
Inconel X-750	1900 °F for 30 minutes	1550 °F for 24 hrs. + 1300 °F for 20 hrs.
Inconel 718	1750 °F for 1 hour	1325 °F for 8 hours Cooled to 1150 °F at 100 °F per hour 1150 °F for 8 hours
Hastelloy R-235	1975 °F for 30 minutes	1400 °F for 16 hrs.
A-286	1800 °F for 1 hour	1325 °F for 16 hrs.

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TEST PROCEDURE

NASA Remote Testing Equipment

The forty in situ tensile tests on irradiated samples as well as the forty pre-irradiation control and the forty post-irradiation control tensile tests were performed in liquid nitrogen using the NASA remote tensile testing equipment and Dewar. A schematic diagram of this equipment is shown in Figure 3, and a photograph of the equipment is shown in Figure 4. The force exerted by the Instron crosshead is transmitted to the test specimen through a hydraulic system consisting of a master cylinder, a header, several valves, approximately 100 ft. of copper tubing, and a slave cylinder connected to a pull rod. The remote tensile assembly has ten slave cylinders and pull rods each of which was set up to test four specimens in sequence. The ten pulling units are positioned in two rows of five each. The only load measuring device used was located on the Instron machine, so that the recorded loads included all tare loads in the system. The tare loads were due to the friction between piston rings and seals and the master and slave cylinders, the friction of the hydraulic fluid passing through the valves, header and lines, and the friction between the pull rod and the Dewar seal. Tare loads were recorded at the beginning and end of each test in many cases, but where this was not possible due to insufficient space between specimens, tare was recorded at the beginning and end of each group of four tests. Tare loads will be discussed in more detail in the next section of this report.

Linearly variable differential transformers (LVDT's) were used to measure motion of the pull rods which was assumed to be extension of the test specimens. The LVDT's are capable of measuring motion to an accuracy of $\pm .001$ " over their linear range of 0.600". The LVDT's could be remotely repositioned to achieve measurement of specimen extension over a total rod travel range of 4". The LVDT output was recorded as a function of time through Model 150-100 Carrier preamps and a Sanborn Model 150 recording system. Calibration curves were obtained for each pull rod, so that the recorded LVDT output could be related to rod motion.

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Load-elongation curves of sufficient accuracy to determine the 0.2% offset yield strength for each specimen were plotted from the load vs. time (Instron recorder) and motion vs. time (Sanborn recorder) plots. Ultimate strength was determined directly from the Instron chart. Specimen elongation and reduction in area measurements were made on the broken samples at room temperature. Specimen total elastic and plastic elongation at temperature were noted from the Sanborn charts.

During the tensile tests, the Instron crosshead motion was maintained at a rate of 0.2"/min, but because of the inherent losses of the hydraulic system the motion of the pull rod fluctuated between approximately .01" and .1 in/min.

Standard Testing Equipment

The thirty-two room temperature and thirty-two liquid nitrogen control tests were performed at WANL in a conventional screw-driven 60,000 lb. Wiediman-Baldwin Universal Testing machine. The crosshead speed was maintained at .024"/min for all tests. 0.2% offset yield strength and ultimate strength were obtained from recorded load vs. time curves. Elongation and reduction of area measurements were made on broken samples at room temperature.

The 96 tests on samples which had been irradiated in liquid nitrogen and stored at room temperature for three months were performed at the Westinghouse Waltz Mills site as follows:

- a. 32 tests at room temperature
- b. 32 tests in liquid nitrogen
- c. 32 tests in liquid nitrogen after 1000 °F anneal for 1 hour

The equipment used for these tests was a conventional screwdriven 10,000 lb. Instron machine. The crosshead motion was maintained at .02 in/min for all tests. Ultimate and .2% offset yield strengths were obtained from recorded load vs. time data. Elongation and reduction of area measurements were made on broken samples at room temperature. Total elastic and plastic deformation at temperature was noted from recorded data.

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Description of Irradiation and Tests

In accordance with the test plan presented in the Appendix, 40 specimens were loaded into the NASA equipment and Dewar, submerged in liquid nitrogen and tested in tension to fracture. Figure 5 shows the typical position of samples in the NASA equipment. These tests were performed prior to the irradiation and are designated the pre-irradiation control tests.

After these tests were completed, an additional 40 specimens were positioned in the NASA equipment as shown in the test plan in the Appendix along with 96 samples in four racks of 24 specimens each, which were suspended from the dosimeter racks as shown in Figures 6 and 7. The test plan in the Appendix identifies the specimens in the four racks. Figures 5 and 6 also show the dosimeter locations. The NASA equipment and Dewar with nitrogen added was then placed in the irradiation position and the GTR was brought to power. The specimens were continuously submerged in liquid nitrogen throughout the irradiation, even during the periodic flushing of the Dewar (required to remove any accumulated ozone crystals). After an accumulation of 450 MV hrs. of irradiation time the reactor was shutdown, and the tensile tests were performed. All tests in situ were completed within 12 hours of shutdown.

A second set of 40 control samples (Table IIIc) was tested at the GTR site in the irradiated equipment approximately six weeks after the irradiation when the equipment was radioactively "cool" enough to permit unloading and loading of samples. The purpose of the post-irradiation control tests was to determine if radiation effects on the hydraulic fluid (Oronite 8515) and piston rings and seals (rubber and Teflon) would affect the tensile data.

The 96 irradiated samples from the four racks were removed and shipped to the Westinghouse Waltz Mills site for testing. As stated earlier these samples were at room temperature for approximately three months prior to environmental testing. The test plan in the Appendix identifies the control and irradiated samples tested by Westinghouse at the Large and Waltz Mills sites.

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RESULTS AND DISCUSSION

Test Results

The samples received a total fast neutron dose of between 2.4×10^{16} and 7.1×10^{16} n/cm² ($E > 1$ Mev), which is low in comparison to a five-minute full power NERVA dose of 3×10^{17} n/cm² ($E > 1$ Mev) at the thread end of the centerline tie rod. Table II relates dose to location of specimens. The dose received by the Inconel X-750 specimens is equivalent to that expected during a reactor test run at seventy-five percent of full power lasting one to one and one-half minutes for the thread end of a centerline tie rod or three to four minutes for the thread end of a peripheral tie rod.

Figures 8 through 16 show the test results for notched tensile strength, ultimate tensile strength, notched to unnotched ultimate tensile strength ratio, tensile yield strength (0.2% offset), elongation and reduction in area for all samples tested. The figures are in bar graph form, the height of each bar being the range of data. Data for machined and forged specimens of the same material are presented separately. The replication was two samples for all test points except for the forged samples in the NASA equipment, where the replication was three samples.

The effect of radiation on the NASA equipment seems to be significant in most cases, but is of a variable nature; that is, in some cases the post-irradiation strength data seems higher than the pre-irradiation data, and in other cases lower. For this reason, it is felt that the post-irradiation control data should be considered the baseline data for the NASA equipment test results, even where it is different from the standard control data generated in a conventional hard machine.

The test results for the front row specimens, Figures 8 through 11, (this includes all Inconel X-750 and Inconel 718 specimens) show the following tendencies:

1. No significant change in any property at room temperature (compare columns 7 and 8).
2. No consistent changes in notched tensile strength at -320°F (columns 1 through 6).
3. Consistent increase in ultimate tensile strength for samples tested in situ immediately after irradiation at -320°F (compare columns 2 and 3).

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4. Consistent decrease in ultimate tensile strength for irradiated samples tested at -320°F after storage at room temperature which is effectively an annealing treatment (compare columns 4 and 5).
5. General recovery of -320°F ultimate strength for irradiated samples which were annealed at 1000°F for one hour before testing (compare columns 4, 5, and 6).
6. No consistent changes in notched to unnotched tensile strength ratio (all columns).
7. Consistent increase in -320°F yield strength for irradiated samples tested in situ (compare columns 2 and 3).
8. General increase in -320°F yield strength for irradiated samples after storage at room temperature (compare columns 4 and 5), but a slight decrease from the in situ tests (compare columns 2 and 5).
9. General recovery of original -320°F yield strength for irradiated samples annealed at 1000°F (compare columns 4, 5, and 6).
10. No significant decrease in -320°F elongation for samples tested in situ (compare columns 2 and 3).
11. Consistent decrease in -320°F elongation for irradiated samples stored at room temperature (compare columns 4 and 5 and also 2 and 5).
12. Recovery of original -320°F elongation for samples annealed at 1000°F (compare columns 4, 5, and 6).
13. No consistent changes in reduction of area (all columns).

The test results for the back row specimens (all specimens of Hastelloy R-235 and A-286) are shown in Figures 12 through 15. These specimens received less than half the dose of the front row specimens due to the geometry of the test rig, and, therefore, the results are not as well defined as those for the front row material. The following results were indicated:

1. Large variability in the Hastelloy R-235 forged material as evidenced by comparison of the results on the three control tests (columns 1, 3, and 4).
2. Increases in notched, ultimate and yield strengths for the Hastelloy R-235 machined and forged specimens tested in situ (compare columns 2 and 3).

3. Properties of Hastelloy R-235 recovered after room temperature or 1000 °F anneal (compare columns 4, 5, 6, 7, and 8).
4. No consistent changes in the properties of the A-286 machined samples (all columns).
5. The A-286 forged material showed some increase in notched tensile, ultimate and yield strength at -320 °F after irradiation when tested in situ (compare columns 2 and 3) but recovered after room temperature or 1000 °F anneal (compare columns 4, 5, 6, 7, and 8).

Discussion of Results

The property changes noted in each group of the Inconel X-750 and Inconel 718 specimens were generally too small to be considered very significant if each comparative change is considered alone. However, the fact that the changes noted are so consistent from material to material lends much weight to their significance. One possible mechanism to explain the property changes noted can be suggested. Immediately after the -320 °F irradiation the physical changes in the state materials consist of the addition of a large number of widely scattered point defects. These defects are vacancies and interstitial atoms caused by various neutron collision reactions. The strain energy fields caused by the presence of these defects inhibit but do not stop the motion of dislocations through the lattice when the stress is applied thus increasing the yield strength and ultimate tensile strength. Since the strain energy fields are small because of small defects, dislocations do not pile up sufficiently to provide potential crack nuclei. This process would explain observed increases in strength without loss in ductility. After samples have been annealed at room temperature and then cooled to -320 °F, the state of the material has changed. The point defects have had a chance to cluster slightly by diffusion at room temperature and have become large enough to effectively pin dislocations. The stress field in back of a pinned dislocation will cause other moving dislocations in the same path to pile up and thus create potential crack nuclei.⁽⁴⁾ It is believed that the observed increase in yield strength and decreases in ultimate strength and elongation are explainable by this mechanism. The recovery of original properties after 1000 °F annealing is suggested as being the result of the annihilation of the vacancy-interstitial pairs by recombination and/or diffusion to grain boundaries and other sinks.

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To a lesser extent the same discussion might apply to the A-286 and Hastelloy R-235 materials; however, in some cases, any effects were masked by variability of starting material. The observed increases in strength for in situ tests after -320°F irradiation were comparable to Inconel 718 and Inconel X-750. Annealing appeared to eliminate any other damage effects after this low dosage.

In general, the notched tensile strength data generated was not consistent enough to use as a basis of discussion. It is noteworthy, however, that in no case did the notched to unnotched ultimate tensile strength ratio fall below unity. There were no significant differences in tensile results between machined and forged specimens. Machined specimens were fabricated from 0.375" diameter bar taken from the same heat as the 0.110" diameter wire used for forged specimens. The additional working received by the 0.110" diameter wire had no apparent effect on the properties of the material. Also forged and machined specimens exhibited identical modes of failure and similar fracture locations. It is, therefore, thought that, while forging of tie rod components is desirable due to favorable flow patterns, machined specimens may be used for the further evaluation of irradiation effects on tie rod materials for reasons of economy.

Evaluation of Data Accuracy

A good measure of the accuracy of the data generated in this program is a comparison of the control data generated with the NASA equipment with that with the standard equipment. In most cases, these data fall within 5% of each other, but in several cases the reproducibility is off by as much as 15%. Several possible sources of error should be noted:

1. The diameters of the specimens were small, so that any error in measurement of cross sectional area is magnified over similar errors for larger specimens in the stress calculations. Measurement of the diameter at the root of notched specimens is especially susceptible to this type of error.
2. The clevis arrangement in the NASA equipment is such that some bending can be induced into the sample during testing.

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3. Since the load cell for the in situ tests was located such that it picked up all the tare loads in the hydraulic system, the tare loads had to be subtracted from measured load. The tare load was found to vary as a function of strain rate and may also have varied as a function of load. Because of the fact that strain rate as well as load varied throughout the test, the tare loads recorded at the beginning and end of the test or test series must have been different than the tare during the test. In most cases the tare loads were less than 20% of the net loads at the notched, yield, or ultimate points. In one case, however, the tare was as high as 35% and in another as high as 50%. The two cases were Rod No. 1 (Inconel X-750 machined, irradiated), and Rod No. 9 (Hastelloy R-235 machined, precontrol) respectively. The results in Figure 10, column 2 and Figure 14, column 1 for the two high tare cases do not seem any worse than the other data presented. If tare loads were known to $\pm 25\%$, most data on the NASA equipment should be good to $\pm 5\%$. The tare for the two high tare cases could have been off by no more than 20% as determined by the change in tare required to adjust the data to the control values generated on standard equipment.
4. Because of gas build-up in the hydraulic system due to irradiation effects, some samples were pre-loaded during testing. The effect of preloading a sample below the elastic limit is believed to have negligible effect on tensile properties.
5. Measurements of reduction in area on small samples are susceptible to the magnification of error noted above and especially so because most samples become out of round.

A good measure of the variability of material is the comparison of elongation measurements for the three control tests (columns 1, 3, and 4 in Figures 9 through 16). The elongation measurements were made on the broken specimens by the same person using the same holding and measuring jig and are accurate to within $\pm 1\%$. Because of the greater accuracy of the elongation measurements greater weight can be placed on them. Similar high accuracy is expected for the conventional "hard" screwdriven testing machines used in the program.

Precision (range of data for each test condition) was generally good.

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CONCLUSIONS

1. Tie rod materials irradiated at -320°F to a total integrated dose of 2.4 to $7.1 \times 10^{16} \text{ n/cm}^2$ ($E > 1 \text{ Mev}$) showed changes of mechanical properties both as a function of cryogenic irradiation and the post-operative thermal cycles.
2. The dose received by Inconel X-750 was between 5.4 and $6.8 \times 10^{16} \text{ n/cm}^2$ ($E > 1 \text{ Mev}$) which is low compared to a dose of $3 \times 10^{17} \text{ n/cm}^2$ ($E > 1 \text{ Mev}$) expected at the thread end of a centerline tie rod in a five-minute, full power NRX-A test run. However, for a test run at 75% of full power, the dose received would be equivalent to that expected after one to one and one-half minutes of operation at the thread end of a centerline tie rod or to three to four minutes of operation at the thread end of a peripheral tie rod.
3. The reliability or integrity of Inconel X-750 tie rods would not be reduced by a limited power test run where the materials received comparable dosage even though some changes would be expected in mechanical properties.
4. For the alternate materials, Inconel 718 received 5.1 to $7.1 \times 10^{16} \text{ n/cm}^2$ ($E > 1 \text{ Mev}$); A-286 and Hastelloy R-235 both received 2.4 to $3.1 \times 10^{16} \text{ n/cm}^2$ ($E > 1 \text{ Mev}$).
5. Inconel X-750 and Inconel 718 showed an increase in the -320°F ultimate tensile strength and yield strength but no change in ductility when tested in place immediately after irradiation at -320°F .
6. After annealing at room temperature and retesting at -320°F , the irradiated Inconel X-750 and Inconel 718 showed increased yield strength, decreased ultimate strength and decreased percent elongation as compared to unirradiated samples. The yield strength, even though higher than on control samples, showed slight decrease as compared to samples tested in situ after irradiation.
7. After annealing at 1000°F and retesting at -320°F , the properties of irradiated Inconel X-750 and Inconel 718 recovered and were comparable to the unirradiated material.
8. The -320°F notched tensile strength ultimate tensile strength and yield strength of Hastelloy R-235 and for some A-286 samples increased without loss in ductility after irradiation when tested in situ.

9. Annealing at room temperature and 1000 °F caused recovery of properties in Hastelloy R-235 and A-286.
10. No changes were observed in notched toughness of any materials tested as a result of irradiation or thermal cycling.
11. The observed changes in properties indicate changes in the metallurgical structure or crystal lattice but will not permit prediction of the severity of damage for higher irradiation doses which must be evaluated by further testing.
12. There were no significant differences in results between forged and machined specimens. Machined specimens may be used in lieu of forged specimens for economy.

The changes noted in the properties of the materials tested for the modest fast neutron dose received were not generally large enough to cause concern. The property changes indicated by the results of this testing program would be expected to be larger for larger doses, and perhaps disproportionately larger for much larger doses. The samples received a dose equivalent to approximately one minute or less of NERVA operation at the thread end of the centerline tie rod. This is too small a dose to be considered of value to the NERVA structural materials programs. A detailed examination of microstructure was not made because of high cost of such hot cell work and the lack of any significant dosage with respect to NERVA operating conditions.

RECOMMENDATIONS

1. The importance of annealing and testing at a variety of temperatures has been demonstrated. It is strongly recommended that this practice be continued and expanded to include more temperatures and a variety of times (especially short times) at temperature.

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2. It is imperative that further work be done on these materials and other materials at higher dose levels to insure the reliability of the NERVA system.
3. It is felt that further experiments using the NASA remote hydraulic testing equipment would not be as valuable as those using a method for testing in a hard machine. Any method devised should still employ the feature of holding the specimens at cryogenic temperatures between irradiation and testing.

ACKNOWLEDGEMENT

The efforts of J. A. Begley in the final compilation and editorializing of the report are appreciated.

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TABLE I CHEMICAL COMPOSITION OF TEST MATERIALS

Material	X-750								718							
Form	5/16 $\varnothing$ Rod				0.110 $\varnothing$ Wire				5/16 $\varnothing$ Rod				0.105 $\varnothing$ Wire			
Supplier	Latrobe Steel Company				Latrobe Steel Company				Latrobe Steel Company				Latrobe Steel Company			
Heat No.	H-40387				*H-40387				C-56828				HC-56633			
Spec. and Comp.	PDS 30005-B		Composition		PDS 30005-B		Composition		PDS 30038-B		Composition		PDS 30038-B		Composition	
	Max.	Min.	Mill	Check	Max.	Min.	Mill	Check	Max.	Min.	Mill	Check	Max.	Min.	Mill	Check
Element																
C	.08	--	0.07	0.05	0.08	--	.07		0.10	0.03	0.03	--	0.10	0.03	0.05	0.05
Mn	1.20	--	0.98	0.91	1.20	--	.96		0.04	--	0.25	0.19	0.04	--	0.024	0.05
Si	0.50	--	0.34	0.41	0.50	--	.35		0.45	--	0.24	0.06	0.45	--	0.27	0.43
P	--	--	--	--	--	--	--		--	--	0.003	--	--	--	--	--
S	0.015	--	0.008	0.004	0.015	--	0.008		0.015	--	0.006	--	0.015	--	0.005	0.006
Cr	17.0	14.0	14.96	14.80	17.0	14.0	15.04	Not Determined	21.0	17.0	18.78	18.75	21.0	17.0	18.8	17.5
Ni	--	70.0	71.80	73.00	--	70.0	72.24		55.0	50.0	53.52	53.00	55.0	50.0	53.68	51.0
Mo	--	--	--	--	--	--	--		3.30	2.80	3.05	3.10	3.30	2.80	3.17	2.83
Ti	2.75	2.25	2.67	2.50	2.75	2.25	2.61		1.15	0.65	1.04	1.01	1.15	0.65	0.90	0.71
B	--	--	--	--	--	--	--		0.006	0.001	0.006	.0008	0.006	0.001	0.005	0.002
V	--	--	--	--	--	--	--		--	--	--	--	--	--	--	--
Cu	0.50	--	0.03	0.015	0.50	--	0.030		0.15	--	0.08	0.002	0.15	--	0.09	0.23
Co	0.20	--	0.20	0.050	0.20	--	0.020		0.20	--	0.10	0.09	0.20	--	0.07	0.10
Fe	9.00	5.00	6.53	6.60	9.00	5.00	6.50		Bal	--	Bal	Bal	Bal	--	Bal	--
Nb + Ta	1.20	0.70	0.84	0.97	1.20	0.70	0.79		5.50	4.75	5.36	5.19	5.50	4.75	5.21	5.28
Al	1.00	0.40	0.72	0.68	1.00	0.40	0.70		0.85	0.35	0.67	0.67	0.85	0.35	0.59	0.59

*0.110 $\varnothing$ Wire processed from same heat as 5/16 $\varnothing$ Rod - Mill Analysis Performed on Each Finished Product, check analysis performed only on 5/16 $\varnothing$ rod.

TABLE I CHEMICAL COMPOSITION OF TEST MATERIALS
(Continued)

Material	R-235								A-286							
Form	3/8 Ø Rod				1/8 Ø Wire				3/8 Ø Rod				1/8 Ø Wire			
Supplier	Union Carbide				Union Carbide				Latrobe Steel Company				Latrobe Steel Company			
Heat No.	RV 7931				RV 7247				A-769				C56048			
Spec. and Comp.	Commercial		Composition		Commercial		Composition		AMS 5735E		Composition		AMS 5735E		Composition	
	Max.	Min.	Mill	Check	Max.	Min.	Mill	Check	Max.	Min.	Mill	Check	Max.	Min.	Mill	Check
Element																
C	0.16	--	0.14	0.13	0.16	--	0.15	0.12	0.08	--	0.05	0.03	0.08	--	0.05	
Mn	0.25	--	0.01	--	0.25	--	0.02	--	2.00	1.00	1.61	1.59	2.00	1.00	1.61	
Si	0.60	--	0.16	--	0.60	--	0.08	--	1.00	0.40	0.82	0.93	1.00	0.40	0.82	
P	0.010	--	0.001	--	0.010	--	0.001	--	0.04	--	0.025	--	0.04	--	0.025	
S	0.030	--	0.008	--	0.030	--	0.006	--	0.03	--	0.006	0.006	0.03	--	0.006	
Cr	17.00	14.00	15.09	15.1	17.00	14.00	15.08	14.5	16.0	13.5	15.20	14.8	16.0	13.5	15.2	
Ni	Bal	Bal	Bal	Bal	Bal	Bal	Bal	Bal	27.0	24.0	25.40	24.35	27.0	24.0	25.4	
Mo	6.50	4.50	5.51	5.40	6.50	4.50	5.73	5.29	1.50	1.00	1.19	1.13	1.50	1.00	1.19	
Ti	2.75	2.25	2.61	2.50	2.75	2.25	2.40	2.35	2.30	1.90	1.95	1.96	2.30	1.90	1.75	
B	--	--	--	--	--	--	--	--	0.10	0.001	0.007	0.028	0.10	0.001	0.007	
V	--	--	--	--	--	--	--	--	0.50	0.10	0.33	0.22	0.50	0.10	0.33	
Cu	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	
Co	2.50	--	0.45	0.33	2.50	--	0.20	0.13	--	--	--	--	--	--	--	
Fe	11.00	9.00	9.75	9.70	11.00	9.00	9.73	9.60	Bal	--	Bal	Bal	Bal	--	Bal	Bal
Nb + Ta	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	
Al	2.25	1.75	1.96	1.95	2.25	1.75	2.13	1.90	--	--	0.13	0.17	--	--	0.13	

TABLE II - DOSIMETRY*

<u>Location</u>	<u>Specimen Numbers</u>	<u>Fast Neutron Dose, n/cm²</u>	
		<u>E > 2.9 Mev</u>	<u>E > 1.0 Mev**</u>
Rod No. 1	X-750: 141, 142, 121, 122	1.9×10^{16}	5.4×10^{16}
Rod No. 2	X-750: 101, 102, 81, 82	2.4×10^{16}	6.8×10^{16}
Rod No. 3	718: 21, 22, 1, 2	2.5×10^{16}	7.1×10^{16}
Rod No. 4	718: 61, 62, 41, 42	2.4×10^{16}	6.8×10^{16}
Rod No. 5	718: 37, 17; X-750: 117, 97	1.8×10^{16}	5.1×10^{16}
Front Sample Holders	See Below	2.4×10^{16}	6.8×10^{16}
Rod No. 6	A-286: 301, 302, 281, 282	0.9×10^{16}	2.7×10^{16}
Rod No. 7	A-286: 261, 262, 241, 242	1.0×10^{16}	2.8×10^{16}
Rod No. 8	R-235: 181, 182, 161, 162	1.1×10^{16}	3.1×10^{16}
Rod No. 9	R-235: 221, 222, 201, 202	1.0×10^{16}	2.8×10^{16}
Rod No. 10	R-235: 197, 177; A-286: 277, 257	0.8×10^{16}	2.4×10^{16}
Back Sample Holders	See Below	0.8×10^{16}	2.4×10^{16}

Contents of Sample Holders

<u>Front</u>		<u>Back</u>	
<u>718</u>	<u>X-750</u>	<u>R-235</u>	<u>A-286</u>
3- 8	83- 88	163-168	243-248
23-28	103-108	183-188	263-268
43-48	123-128	203-208	283-288
63-68	143-148	223-228	303-308

*Dosimetry supplied by General Dynamics/Fort Worth has been adjusted as follows:

1. Dose adjusted to account for dosimetry packets being located 1.2" in front of front row specimens and 0.8" behind back row specimens.
2. Dosimetry for rods 2, 7, 4, and 9 has been estimated.

**Dose data multiplied by 2.84 to include neutrons with energies between 2.9 and 1.0 Mev.

FIGURE 1 - HISTOGRAM OF THE IRRADIATION AND TESTING

PLAIN ROUND SPECIMEN (570A686)

NOTCHED ROUND SPECIMEN (570A687)

RADIUS MUST BE TANGENT
WITH REDUCED SECTION
NO UNDERCUT PERMITTED

TOLERANCES UNLESS SPECIFIED

.X $\begin{smallmatrix} + \\ -. \end{smallmatrix}$.1
.XX $\begin{smallmatrix} + \\ -. \end{smallmatrix}$.01
.XXX $\begin{smallmatrix} + \\ -. \end{smallmatrix}$.001

FIGURE 2 - PLAIN AND NOTCHED TENSILE SPECIMENS

FIGURE 3 - SCHEMATIC REMOTE TESTING EQUIPMENT

FIGURE 4 - NASA REMOTE TESTING EQUIPMENT

FIGURE 5 - SPECIMENS AND GRIPS POSITIONED IN NASA REMOTE
TESTING EQUIPMENT

FIGURE 6 - FRONT VIEW OF NASA EQUIPMENT SHOWING SAMPLE HOLDERS

FIGURE 7 - REAR VIEW OF NASA EQUIPMENT SHOWING SAMPLE HOLDERS

Dose: $5.1 - 6.8 \times 10^{16} \text{ n/cm}^2 \text{ (E > 1 Mev)}$

596198C

FIGURE 8 - TEST RESULTS: INCONEL X-750 FORGED SPECIMENS

Dose: $5.4 - 6.8 \times 10^{16} \text{ n/cm}^2 \text{ (E > 1 Mev)}$

FIGURE 9 - TEST RESULTS: INCONEL X-750 MACHINED SPECIMENS

Dose: $5.1 - 7.1 \times 10^{16} \text{ n/cm}^2$ ($E > 1 \text{ Mev}$)

597120C

FIGURE 10 - TEST RESULTS: INCONEL 718 FORGED SPECIMENS

Dose: $6.8 \times 10^{16} \text{ n/cm}^2$ ($E > 1 \text{ Mev}$)

597123C

FIGURE 11 - TEST RESULTS: INCONEL 718 MACHINED SPECIMENS

Dose: $2.4 - 3.1 \times 10^{16} \text{ n/cm}^2$ ($E > 1 \text{ Mev}$)

597122C

FIGURE 12 - TEST RESULTS: HASTELLOY R-235 FORGED SPECIMENS

Dose: $2.4 - 2.8 \times 10^{16} \text{ n/cm}^2 \text{ (E > 1 Mev)}$

FIGURE 13 - TEST RESULTS: HASTELLOY R-235 MACHINED SPECIMENS

Dose: $2.4 - 2.7 \times 10^{16} \text{ n/cm}^2 \text{ (E > 1 Mev)}$

FIGURE 14 - TEST RESULTS: A-286 FORGED SPECIMENS

Dose: $2.4 - 2.7 \times 10^{16} \text{ n/cm}^2 \text{ (E > 1 Mev)}$

597132C

FIGURE 15 - TEST RESULTS: A-286 MACHINED SPECIMENS

APPENDIX

Test Specification - Prepared Prior to Irradiation

I. Experiment Identifications

- A. Test Plan Reference No. - 37/W001 Tie Rod Materials X-750, 718, A-286, R-235
- B. Sponsor - WANL Materials Department
- C. Testing Agency - GTR
- D. Test Date - September 15, 1963

II. Purpose of the Experiment

To evaluate irradiation effects on materials for NERVA applications including tie rods.

III. Drawings

Test Specimens: 570A686, 570A687, 570A688, 570A690

Grips: 799C761, 570A689

Sample Holders: Sketch No. 1, Sketch No. 2, and Sketch No. 3

IV. Equipment List

- A. To be provided by NERVA:
Grips, sample holders, shipping cask
- B. To be provided by Tester:
Remote tensile apparatus and cryogenic chamber, complete

V. Test Environment

- A. Temperature - -320°F
- B. Pressure - 1 ATM
- C. Fast Neutron Flux - $3.4 - 4.8 \times 10^{11} \text{ n/cm}^2\text{-sec.}$ $E > 1 \text{ Mev}$
- D. Thermal Neutron Flux - $6.0 \times 10^9 \text{ n/cm}^2\text{-sec.}$
- E. Gamma Flux - $2.2 \times 10^7 \text{ R/hr.}$
- F. Integrated Fluxes
 - Neutron (Fast) - $1.8 - 2.5 \times 10^{17} \text{ n/cm}^2$ $E > 1 \text{ Mev}$
 - Neutron (Thermal) - $4.3 \times 10^{15} \text{ n/cm}^2$
 - Gamma - $4.4 \times 10^9 \text{ R}$

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G. Fluid Environment - Liquid Nitrogen

H. Duration - 150 hours

VI. Analytical - Not available

VII. Facility Requirements

Those requirements associated with the NASA remote tensile apparatus and cryogenic chamber.

VIII. Dosimetry

Relate dose to each group of 4 tensile specimens in each loading unit and to each group of 24 specimens in sample holders.

IX. Data Handling Requirements - None

X. Test Procedure

The tests referred to below are tensile tests to be performed either in conventional Instron equipment in a controlled area or in the remote tensile equipment (Instron actuated) of the NASA design, as noted. For all irradiated specimens, the time of testing is to be noted. The strain rate shall be 0.05 in/min, and the chart speed shall be 1.0 in/min. The irradiation facility (GTR) will do those tests which are to be performed in the remote equipment and supply WANL the raw data (stress-strain curves and broken tensile specimens) for reduction. WANL will perform the testing on the specimens irradiated in the holding racks and the balance of the control testing. The following procedure includes both GTR and WANL tasks:

A. Condition 1, control: Prior to the irradiation, 40 control specimens shall be mounted in the remote equipment as shown in the following schematic. These control specimens shall be tested in boiling liquid nitrogen (-320°F). No special testing sequence is necessary.

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FRONT
(Facing GTR)

X-750	149 (N,M)	A-286	309 (N,M)
	150 (N,M)		310 (N,M)
	129 (P,M)		289 (P,M)
	130 (P,M)		290 (P,M)
718	109 (N,F)	R-235	269 (N,F)
	110 (N,F)		270 (N,F)
	89 (P,F)		249 (P,F)
	90 (P,F)		250 (P,F)
718	29 (N,F)	R-235	189 (N,F)
	30 (N,F)		190 (N,F)
	9 (P,F)		169 (P,F)
	10 (P,F)		170 (P,F)
718	69 (N,M)	R-235	229 (N,M)
	70 (N,M)		230 (N,M)
	49 (P,M)		209 (P,M)
	50 (P,M)		210 (P,M)
(718)	38 (N,F)	(R-235)	198 (N,F)
(750)	118 (N,F)	(A-286)	278 (N,F)
(718)	18 (P,F)	(R-235)	178 (P,F)
(750)	98 (P,F)	(A-286)	258 (P,F)

Specimen numbers, type, and materials are indicated.

N = Notched, P = Plain, M = Machined, F = Forged

- B. In preparation for the irradiation, 136 specimens shall be mounted in the remote loading units and holding racks as shown in the schematic below:

FRONT
(Facing GTR)

X-750	141 (N,M)	A-286	301 (N,M)
	142 (N,M)		302 (N,M)
	121 (P,M)		281 (P,M)
	122 (P,M)		282 (P,M)
718	101 (N,F)	R-235	261 (N,F)
	102 (N,F)		262 (N,F)
	81 (P,F)		241 (P,F)
	82 (P,F)		242 (P,F)
718	21 (N,F)	R-235	181 (N,F)
	22 (N,F)		182 (N,F)
	1 (P,F)		161 (P,F)
	2 (P,F)		162 (P,F)
718	61 (N,M)	R-235	221 (N,M)
	62 (N,M)		222 (N,M)
	41 (P,M)		201 (P,M)
	42 (P,M)		202 (P,M)
(718)	37 (N,F)	(R-235)	197 (N,F)
(750)	117 (N,F)	(A-286)	277 (N,F)
(718)	17 (P,F)	(R-235)	177 (P,F)
(750)	97 (P,F)	(A-286)	257 (P,F)

24 Specimens for each of
the 4 materials (total 96)
arranged in holding racks,
details on next page.

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Front Rack

Back Rack

718

3-8 (P,F)
23-28 (N,F)
43-48 (P,M)
63-68 (N,M)

R-235

163-168 (P,F)
183-188 (N,F)
203-208 (P,M)
223-228 (N,M)

X-750

83-88 (P,F)
103-108 (N,F)
123-128 (P,M)
143-148 (N,M)

A-286

243-248 (P,F)
263-268 (N,F)
283-288 (P,M)
303-308 (N,M)

- C. After the irradiation at -320°F for 200 hrs., the 40 specimens mounted in the remote loading units shall be tested at -320°F . The liquid nitrogen level shall be maintained so that these specimens will not warm up between irradiation and testing or during testing. No special testing sequence is necessary except that specimens 17, 37, 97, 117, 177, 197, 257, and 277 shall be tested last. These enumerated specimens shall not be tested "in situ" unless a disparity in results for the previous tests is noted. The time of each test shall be noted. (The schematic layout of the specimens irradiated is shown above).
- D. When the condition 1 specimens have been tested, all specimens (tested and untested) shall be permitted to warm up to room temperature (the time at which the specimens reach room temperature should be noted). At this time, the specimens mounted in the holding racks should be returned to WANL as soon as possible. The broken specimens should be retained at GTR to cool down (radioactivity) for two to three weeks.
- E. When the remote equipment has cooled down sufficiently, the post irradiation control testing should be performed also at -320°F . The broken specimens from the irradiation test should be removed, placed in identified sample envelopes and returned to WANL. Additional grips will be supplied for the post-irradiation control test. The loading of samples should conform to the schematic below. No special testing sequence is necessary. Specimens 19, 39, 99, 119, 179, 199, 259, and 279 should be tested only if 17, 37, 97, 117, 177, 197, 257, and 277 were tested previously.

X-750	151 (N,M)	A-286	311 (N,M)
	152 (N,M)		312 (N,M)
	131 (P,M)		291 (P,M)
	132 (P,M)		292 (P,M)
718	111 (N,F)	R-235	271 (N,F)
	112 (N,F)		272 (N,F)
	91 (P,F)		251 (P,F)
	92 (P,F)		252 (P,F)
718	31 (N,F)	R-235	191 (N,F)
	32 (N,F)		192 (N,F)
	11 (P,F)		171 (P,F)
	12 (P,F)		172 (P,F)
718	71 (N,M)	R-235	231 (N,M)
	72 (N,M)		232 (N,M)
	51 (P,M)		211 (P,M)
	52 (P,M)		212 (P,M)
(718)	39 (N,F)	(R-235)	199 (N,F)
(750)	119 (N,F)	(A-286)	279 (N,F)
(718)	19 (P,F)	(R-235)	179 (P,F)
(750)	99 (P,F)	(A-286)	259 (P,F)

F. Condition 2: The 32 irradiated specimens listed below shall be tested at room temperature in conventional equipment. The testing sequence shall be as given below.

Dimensional checks shall be performed on the specimens marked*.

3, 23, 43, 63, 83, 103, 123, 143, 163, 183, 203, 223, 243, 263, 283, 303

4, 24, 44, 64*, 84, 104, 124, 144*, 164, 184, 204, 224*, 244, 264, 284, 304*

G. Condition 3: The 32 irradiated specimens listed below shall be tested at -320 °F in conventional equipment. These specimens shall have been permitted to stand at room temperature for a maximum of 600 hours. The specimens shall be tested in the sequence given. Dimensional checks shall be made on the specimens marked *.

5, 25, 45, 65, 85, 105, 125, 145, 165, 185, 205, 225, 245, 265, 285, 305

6, 26, 46, 66*, 86, 106, 126, 146*, 166, 186, 206, 226*, 246, 266, 286, 306*

H. Condition 4: The 32 irradiated specimens below shall be tested at -320 °F in conventional equipment. These specimens shall have been annealed at 1000 °F $\pm$ 25 °F for one hour and air cooled prior to testing. The specimens shall be tested in the sequence given. Dimensional checks shall be made on the specimens marked *.

7, 27, 47, 67, 87, 107, 127, 147, 167, 187, 207, 227, 247, 267, 287, 307

8, 28, 48, 68*, 88, 108, 128, 148*, 168, 188, 208, 228*, 248, 268, 288, 308*

- I. Condition 2, control: The specimens listed below shall be tested at room temperature.
No special testing sequence is required.

13, 14, 33, 34, 53, 54, 73, 74, 93, 94, 113, 114, 133, 134, 153, 154, 173, 174,
193, 194, 213, 214, 233, 234, 253, 254, 273, 274, 293, 294, 313, 314

- J. Condition 3 and 4, control: The specimens listed below shall be tested at -320°F .
No special testing sequence is required.

15, 16, 35, 36, 55, 56, 75, 76, 95, 96, 115, 116, 135, 136, 155, 156, 175, 176,
195, 196, 215, 216, 235, 236, 255, 256, 275, 276, 295, 296, 315, 316

Summary

Irradiated Specimens:

Load Units -320°F

718 1, 2, 21, 22, 41, 42, 61, 62, (17, 37 - spare)
X-750 81, 82, 101, 102, 121, 122, 141, 142, (97, 117 - spare)
R-235 161, 162, 181, 182, 201, 202, 221, 222, (177, 197 - spare)
A-286 241, 242, 261, 262, 281, 282, 301, 302, (257, 277 - spare)

Racks Rt, -320°F (RT anneal), -320°F (1000 $^{\circ}\text{F}$ anneal)

718 3-8, 23-28, 43-48, 63-68
X-750 83-88, 103-108, 123-128, 143-148
R-235 163-168, 183-188, 203-208, 223-228
A-286 243-248, 263-268, 283-288, 303-308

Control Specimens:

Pre-Irrad Control (Load Units) -320°F

718 9, 10, 29, 30, 49, 50, 69, 70, 18, 38
X-750 89, 90, 109, 110, 129, 130, 149, 150, 98, 118
R-235 169, 170, 189, 190, 209, 210, 229, 230, 178, 198
A-286 249, 250, 269, 270, 289, 290, 309, 310, 258, 278

Post-Irrad Control (Load Units) -320 °F

718 11, 12, 31, 32, 51, 52, 71, 72, (19, 39 - spare)
X-750 91, 92, 111, 112, 131, 132, 151, 152, (99, 119 - spare)
R-235 171, 172, 191, 192, 211, 212, 231, 232, (179, 199 - spare)
A-286 251, 252, 271, 272, 291, 292, 311, 312, (259, 279 - spare)

Standard Control RT and -320 °F

718 13-16, 33-36, 53-56, 73-76
X-750 93-96, 113-116, 133-136, 153-156
R-235 173-176, 193-196, 213-216, 233-236
A-286 253-256, 273-276, 293-296, 313-316

All other specimens are spares

XI. Hazards

- A. Personnel - Established procedures for handling radioactive materials
- B. Facility - None

XII. Data Reduction Required Including Any Special Techniques - None

XIII. Disposition of Hardware

Return to WANL for storage and subsequent disposal through appropriate channels.

XIV. Shipping and Receiving Instructions

Samples must not be bent or damaged in any way.

XV. Appendix

Drawings