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EFFECTS OF NEUTRON IRRADIATION
UPON THE MECHANICAL PROPERTIES OF INCONEL 625

T. T. CLAUDSON

OCTOBER, 1965

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EFFECTS OF NEUTRON IRRADIATION
UPON THE MECHANICAL PROPERTIES OF INCONEL 625

By
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October, 1965

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EFFECTS OF NEUTRON IRRADIATION UPON THE MECHANICAL PROPERTIES OF INCONEL 625

INTRODUCTION

The orderly development of new reactor concepts and technology depends upon the availability of new alloys that are capable of operating in more stringent environments and at higher temperatures than now exist in operating reactors. To provide higher thermal efficiencies, the nuclear superheat reactor concept has been employed, whereby higher operating temperatures are achieved. Operating conditions of the superheat regions of these reactors preclude the use of the common austenitic stainless steels and zirconium alloys. Alloys that have better high temperature strength and oxidation resistance must be used.

As a part of a USAEC-sponsored program to determine the effects of irradiation and environment upon the properties of reactor structural metals, several nickel base precipitation and solution hardenable alloys are being evaluated under various reactor conditions. A survey of the preirradiation mechanical properties and oxidation resistance of these nickel base alloys indicated that Inconel 625 might be suitable for many reactor structural components, including fuel cladding.

Inconel 625 is a relatively new alloy developed by International Nickel Company, Inc. It is a nickel-chromium-iron alloy whose high strength results from adding molybdenum and columbium to the nickel-chromium matrix. Therefore, the material is not a precipitation-hardenable alloy and, in the hot-rolled and annealed condition, does not show the typical ductility decrease in the nickel base alloy range of 760 to 870 °C.

The oxidation resistance of Inconel 625 has been found to be comparable to other nickel base alloys.¹ A tight, adherent, protective, oxide film forms on the metal surface and forms with a minimum amount of intergranular

¹T. T. Claudson and H. J. Pessl. Irradiation Effects on High Temperature Reactor Structural Metals, BNWL-23. February 25, 1965.

attack. Because of its relatively high strength and oxidation resistance, Inconel 625 has been considered as a candidate material for fuel cladding for nuclear superheat reactors. Therefore, it is necessary to determine the effects of irradiation upon its mechanical properties.

SUMMARY AND CONCLUSIONS

The studies within this report have shown the effects of neutron irradiation, test temperature, irradiation temperature, and material condition upon the mechanical properties of the solution hardenable alloy, Inconel 625.

The room temperature tensile properties are characterized by increases in yield strength with some loss in ductility at low (50 °C, 280 °C) irradiation temperatures. Essentially, there was no change within the limits of experimental error in ultimate strength. Material irradiated at these lower temperatures and tested at room temperature maintained good ductility as shown by high uniform and total elongations and large reductions in area. Necking occurred because of plastic instability in a localized region of all specimens irradiated and tested under these conditions.

The major effect found in the room temperature properties of Inconel 625 was caused by irradiation temperature. These data, however, when compared to control specimens, indicate that the effect is primarily because of the thermal environment and not the irradiation since the control specimens also showed a decrease in ductility. This is especially true for the 740 °C irradiation temperature. At the 580 °C irradiation temperature, a further decrease in ductility was observed in the irradiated specimen as compared to the control specimen. This decrease indicated some effect of irradiation. Essentially, no effect was noticed for specimens irradiated at 740 °C. At 740 °C, the only effects were increases in yield and ultimate strength. Although structural components of Inconel 625 will normally operate at elevated temperatures in reactor service, they will also operate satisfactorily under applied stress at lower temperatures during reactor shut down if adequate design considerations are applied.

Unirradiated specimens of Inconel 625 exposed to superheated steam at 1150 °F over long periods of time, increased in yield strength and decreased in room temperature elongations.¹ These effects stabilized after about 4000 hr. The elongation values of over 20%¹ after exposure agree with the values from 580 °C control exposures in this study. Comprelli and Wolff¹ also showed that long term exposure of Inconel 625 to high temperature environments caused precipitation. The precipitates were primarily the AB type, TiN and NbC.

Since the precipitation of inherently brittle second phases occur at elevated temperature (where the alloy is expected to operate) the elevated temperature mechanical properties are of great interest.

The elevated temperature tests on specimens irradiated at 280 °C show a marked decrease in ductility as compared to similar specimens tested at room temperature. It is important to note that this reduction does not seem as dependent upon the irradiation dosage as it is upon the testing temperature. At both test temperatures, only small changes in the percent uniform elongation occurred, compared to a factor of 3 to 4 decrease associated with the difference in test temperature alone.

It appears from the elevated temperature test data that the reduction in strength properties of the material are caused primarily by the effects of the neutron irradiation since significant changes in the yield strengths were found as compared to the control specimens. The reduction in strength may be due to defects in the material relaxing the matrix strain hardening imposed by the solution hardening elements of the alloy, columbium and molybdenum. In comparing the percent uniform elongation data for both the control and irradiated specimens with the as-heat treated value, a marked decrease is observed. This decrease indicates that exposure of the material to its test environment is the primary cause of the reduced ductility because

¹ F. A. Comprelli and U. E. Wolff. Stability of High-Nickel Alloys in Superheated Steam, GEAP-4745. General Electric Company, San Jose, California. November, 1964.

of thermal aging. Thus, it appears that two mechanisms may be operating to cause the observed changes in the mechanical properties of the alloy. One mechanism is associated with the neutron exposure controlling the strength properties, the other is associated with the thermal exposure controlling the ductility of the material.

This apparent small effect of irradiation upon the high temperature ductility of Inconel 625 was not discovered in specimens irradiated at 740 °C. Specimens irradiated to an exposure of 9.2×10^{20} nvt ($E > 1$ MeV) showed a factor of 2 decrease in uniform elongation. This decrease indicates irradiation-induced damage in the material. This observation was also made for the solution hardenable nickel base alloy Hastelloy N at Oak Ridge National Laboratory.¹

Examination of the data for specimens irradiated at 740 °C in the mill-annealed and in the solution-treated and aged conditions and tested both at room temperature and at 650 °C, reveals that the ductility of the material at both test temperatures is lower after 740 °C irradiation than after 280 °C irradiation. For the room temperature tests, the material increased in yield and ultimate strength because of the 740 °C irradiation. The converse was true in material tested at 650 °C where a decrease in the strength properties was observed. In addition, a greater change in strength and ductility due to irradiation was seen at the 650 °C test temperature. It also appears that material in the solution treated condition prior to the irradiation retains more ductility during elevated temperature neutron exposure.

It is concluded from these investigations that thermal exposure plays an important role upon the mechanical properties of Inconel 625 after irradiation. Thermal stability of this alloy at elevated temperatures in absence

¹ W. R. Martin and J. R. Weir. Effect of Elevated Temperature Irradiation on the Strength and Ductility of the Nickel-Base Alloy, Hastelloy N, ORNL-TM-1005, Oak Ridge, Tennessee. February, 1965.

of neutrons is good with most precipitation reactions occurring slowly¹ at temperature of 620 °C. At temperature of 740 °C, these reactions must surely increase in rate; and in the presence of high energy neutrons, may occur even faster. The extent and distribution of the precipitation and their effects upon the mechanical properties of the material should be the subject of further research.

IRRADIATION ENVIRONMENTS AND TEST CONDITIONS

Material used in these studies was obtained from the vendor as 0.250 in. plate stock in annealed and pickled condition. The certified chemical analysis of this material is given in Table I. After hot rolling, the material was annealed at 1150 °C for 1 hr and air cooled. Further plate reduction was necessary to fabricate tensile specimens of the geometry shown in Figure 1. Prior to fabrication of the tensile specimens, the 0.250 in. plate was hot rolled (according to the rolling schedule shown in Table II) to a nominal thickness of 0.110 in. No reheating was required during the rolling. After rolling, the material was sand blasted, washed, roller leveled, and annealed. The material was annealed at 1040 °C for 1 hr and air cooled. Because of slight warpage from the annealing operation, the material was again roller leveled and flattened.

TABLE I
CHEMICAL ANALYSIS OF INCONEL 625

<u>Heat Number NX 8137A</u>									
Chemical, wt%									
<u>C</u>	<u>Ni</u>	<u>Cr</u>	<u>Fe</u>	<u>Cb</u>	<u>Mo</u>	<u>S</u>	<u>P</u>	<u>Si</u>	<u>B</u>
0.033	62.62	21.81	1.46	3.97	8.99	0.006	0.005	0.22	<0.001
<u>Ta</u>	<u>N</u>	<u>H</u>	<u>O</u>	<u>Total rare earths</u>					
<0.01	0.047	0.0006	0.011	20 ppm					

¹F. A. Comprelli and U. E. Wolff. Stability of High-Nickel Alloys in Superheated Steam, GEAP-4745. General Electric Company, San Jose, California, November, 1964.

FIGURE 1

Specimen Geometry Used to Determine the Effects of Irradiation Upon the Mechanical Properties of Inconel 625

TABLE II

ROLLING SCHEDULE FOR INCONEL 625

Heat Number NX 8137A

<u>Pass No.</u>	<u>Gage Width, in.</u>	<u>Temperature</u>
1	0.250	1177 °C*
2	0.210	
3	0.176	
4	0.155	
5	0.135	
6	0.120-0.127	
7	0.113-0.116	

*Starting temperature

The resulting 0.110 in. sheets were given a solution and aging treatment. The material was heated to a temperature of 1040 °C for 1 hr, water quenched, reheated to 705 °C for 24 hr, and water quenched. The properties of the material from this heat treatment were quite similar to the vendor's properties for this specific heat treatment. Additional material was set aside in the as-worked and annealed condition for comparison to the heat treated material.

Flat tensile specimens were then fabricated by surface grinding, to the dimensions shown in Figure 1. To insure the fabrication of reproducible specimens, jigs were used, whereby 30 specimens could be produced at one time. Identical grinding procedures were followed for each set of specimens that were subsequently accurately measured and weighed prior to the irradiation.

Since a reactor material may be used in a variety of temperature ranges, specimens of Inconel 625 were irradiated at 50 and 280 °C in water, and at average temperatures of 580 and 740 °C in a predominately helium environment. Neutron exposure levels in the water environments were between 4×10^{19} and 1.46×10^{21} nvt ($E > 1$ MeV). The exposure levels for the higher temperature irradiations were 9.2×10^{19} nvt and 1.92×10^{20} nvt ($E > 1$ MeV).

Tensile specimens of the identical material were given similar thermal and environmental treatments out-of-reactor and tested as control specimens. However, specimens in the preirradiation condition were tested as control specimens for the 50 °C irradiations and no additional thermal conditioning was given. Control specimens for material that was irradiated in the Engineering Test Reactor (ETR) G-7 hot water loop facility at 280 °C were exposed to an out-of-reactor loop facility which duplicated temperature conditions and water chemistry found in the in-reactor facility. Thermal and environmental conditions for the 580 and 740 °C irradiations were simulated by encapsulating specimens in helium and placing them in a laboratory furnace at the appropriate temperatures for an equivalent number of reactor operating hours. After exposure, the control specimens were tested identically to the irradiated specimens.

After irradiation, specimens were returned to the Radiometallurgy Laboratory for testing. Immediately after discharge from the cask, specimens were weighed, and dimensional and hardness measurements were made. The specimens were then tested under one of two procedures. The procedure depended upon which of three test temperatures was used. The test temperatures used in this investigation were room temperature, 300, and 650 °C.

Specimens tested at room temperature were tested in an Instron tensile machine at a constant strain rate of 0.005 in./in./min. A standard Baldwin PM-3A extensometer was used to measure the specimen extension.

Those specimens tested at 300 °C were placed into a furnace (Figure 2). After the specimen was inserted and the extensometer positioned, 20 min were required to bring the specimen to equilibrium temperature. At equilibrium temperature, the specimen was pulled at a constant strain rate of 0.05 in./in./min. The extension was measured by a modified PM-3A type extensometer calibrated at the test temperature.

FIGURE 2

Experimental Apparatus for 300 °C Tensile Tests

Neg. 0632847-2

The control tests at 650 °C were performed in a 3-stage Marshall furnace (Figure 3) using an extended type extensometer (extensometer not attached). A strain rate of 0.05 in. /in. /min was used. Special grips were

FIGURE 3

Experimental Apparatus for 650 °C Control Tensile Tests

Neg. 0642150-2

developed by using a titanium carbide insert for gripping the specimen. Radioactive specimens, tested at this temperature, were tested at the same strain rate using a similar extensometer in a hard beam Tinius Olson machine. A Marshall tantalum element, resistance furnace was used in these tests, and the tests were performed in vacuum. The test apparatus is shown in Figure 4.

FIGURE 4

High Temperature Vacuum Furnace
Used for Elevated Temperature Tensile Tests

Neg. 0640968-3

TEST RESULTS

A. Effects of Irradiation Upon the Room Temperature Properties of Inconel 625

The results of room temperature (20 °C) tests performed upon irradiated specimens are shown in Figures 5, 6, and 7. Figure 5 shows the effects of neutron exposures of 7.64×10^{20} nvt ($E > 1$ MeV) upon the yield strength, ultimate tensile strength, percent uniform elongation, and percent reduction in area of Inconel 625 after irradiation at 50 °C. As compared to the unirradiated control specimens, the irradiated specimens showed a large increase in strength with a corresponding decrease in uniform elongation. At an exposure of 7.64×10^{20} nvt, the yield strength increased 72% to a value of 160,600 psi. Only a small increase in ultimate strength (7%) was observed. The uniform elongation was only 8.4% at this exposure, but a large reduction in area was evident and indicated local plastic instability at the fracture section of the specimen.

FIGURE 5

Effects of Neutron Irradiation Upon the Room Temperature Properties of Inconel 625 Irradiated at 50 °C in the Solution Treated Condition

Figure 6 summarizes the results of tests performed on specimens irradiated at 280 °C and tested at room temperature. These specimens were irradiated to a maximum exposure of 8.27×10^{20} nvt ($E > 1$ MeV). Although the increases in strength are smaller than for specimens irradiated at 50 °C, a substantially higher percent uniform elongation was found. This elongation exceeded 30% at the maximum exposure tested. There was found little or no effect of the irradiation upon the percent reduction in area or ultimate tensile strength of the material. Also, only small variations in ductility were found throughout the exposure range.

FIGURE 6

Effects of Neutron Irradiation Upon the Room Temperature Properties of Inconel 625 Irradiated at 280 °C in the Solution Treated Condition

Additional specimens were tested at room temperature after irradiation at 580 and 740 °C to an exposure of about 9×10^{19} nvt ($E > 1$ MeV). The results of these tests are shown in Figure 7. For convenience, all the room temperature data for specimens irradiated at about 1×10^{20} nvt have also been plotted for comparison and show the effect of the irradiation temperature upon the room temperature properties of Inconel 625.

FIGURE 7

Effects of Irradiation Temperature Upon the Room Temperature Properties of Inconel 625 Irradiated to an Exposure of About 1×10^{20} nvt ($E > 1$ MeV)

Specimens irradiated at 580 °C and tested at room temperature show a greater percentage increase in yield strength than those irradiated at 740 °C. The lower temperature irradiation increased the yield strength by 27%, compared to a 12% increase for the 740 °C irradiation. However, a marked decrease in uniform elongation (from 27% to about 18%) resulted from an exposure of 9.2×10^{19} nvt ($E > 1$ MeV) at 580 °C. An irradiation temperature of 740 °C caused little if any change to either reduction of area or uniform elongation. It is evident that by comparing the control data to the irradiation data, most of the decrease in uniform elongation is due not to the irradiation but to the thermal exposure of the material in its environment.

As previously mentioned, specimens of Inconel 625 in the mill-annealed condition were irradiated for comparison with those in the solution-treated and aged condition. The results of these tests, performed at room

temperature for specimens irradiated at 740 °C, are shown in Figure 8. For the solution-treated and aged specimen, only small differences in uniform and total elongation, and reduction of area were found between the control and irradiated specimens. A 22% increase in yield strength was found due to the irradiation. Similar statements are true for the annealed specimens.

FIGURE 8

Effects of Irradiation Upon the Room Temperature Properties of Inconel 625 in the Annealed and Solution Treated-Aged Condition Irradiated at 740 °C to an Exposure of 9×10^{19} nvt ($E > 1$ MeV)

Neg. 0643021-6

Comparison of the annealed to the solution-treated and aged specimens shows that solution-treated and aged specimens maintained higher strengths and more ductility before and after irradiation. Specimens in both conditions increased in yield strength about 22% and indicated little relative difference in radiation hardening.

B. Effects of Irradiation Upon the Elevated Temperature Mechanical Properties of Inconel 625

Specimens of Inconel 625 were irradiated at 740 °C to a maximum exposure of 1.9×10^{20} nvt ($E > 1$ MeV) and tested at 300 °C (Figure 9).

FIGURE 9

Effect of Neutron Irradiation Upon the Mechanical Properties of Inconel 625 Irradiated at 740 °C and Tested at 300 °C

The yield strength of the specimens irradiated to 9.2×10^{19} nvt was 100,000 psi as compared to 94,190 psi for the control (an increase of about 7%). Some increase ($\approx 4\%$) was also found in the ultimate strength. A considerable decrease in ductility was found as shown in Figure 9. The uniform elongation decreased from 18.1% for the unirradiated control specimen to 6.19% for the irradiated material. Similar decreases can be seen in the total elongation. The reduction of area of the specimens remained unchanged at approximately 17%.

For the specimens irradiated to 1.9×10^{20} nvt at 740 °C, the 300 °C tensile properties show further reduction in strength and ductility as compared

to specimens irradiated to lower exposure. Most of this decrease is because of the thermal exposure over a longer period of time as shown by the comparison to the control specimen corresponding to the higher exposure. The yield strength of the longer exposure specimens was decreased by the irradiation as compared to the control specimens while the yield strength of the lower exposure specimens was increased slightly (Figure 9).

The effects of irradiation at 280 °C upon the mechanical properties of Inconel 625 at 650 °C have been determined as shown in Figure 10. Results of these tests indicate little change in yield strength of the material at exposures to 1.46×10^{21} nvt ($E > 1$ MeV). The greatest change occurred in the ultimate strength which decreased about 25%. In addition, low ductilities, including the control specimens, were noted throughout the tests. Values of uniform elongation as low as 4.5% at 1.46×10^{21} nvt were found which indicated the material retained about the same ductility as the unirradiated specimen.

FIGURE 10

Effects of Irradiation at 280 °C Upon the Mechanical Properties of Inconel 625 Tested at 650 °C in the Solution Treated Condition

Specimens irradiated at 740 °C to an exposure of 9×10^{19} nvt in the mill annealed and solution treated conditions were also tested at 650 °C. The results of these tests are shown in Figure 11. The effect of the solution treatment in the unirradiated control specimens was to increase somewhat the strength and ductility of the material. After irradiation at 740 °C, the strength and ductility of the material in both the solution heat treated and annealed condition were substantially reduced. Whereas, before irradiation, the solution treated material showed the higher strength, this material exhibited a greater loss in strength after irradiation. However, it is important to note that the solution treated material maintained more ductility after irradiation than did the material in the mill annealed condition even though it had lower strength properties.

FIGURE 11

Effects of Neutron Irradiation Upon the 650 °C Mechanical Properties of Inconel 625 Irradiated at 740 °C in the Mill Annealed and Solution Treated Conditions to an Exposure of 9×10^{19} nvt ($E > 1$ MeV)

Table III lists 650 °C tensile data for irradiated and control specimens of Inconel 625. Comparison of the data for Specimen 59 with Specimen A45X shows that the thermal aging at 740 °C given Specimen A45X does not affect the high temperature strength properties of the material.

As previously explained, the control specimens were given a thermal exposure equivalent to the irradiated specimens. The comparison of data indicates a small (1010 psi) increase in yield strength and a decrease (5000 psi) in ultimate strength. A decrease in ductility was also found as shown by the uniform and total elongation ($\Delta e \approx 9\%$) and reduction of area ($\Delta RA \approx 6\%$) values. Specimen A39X in the mill annealed condition shows a reduction in strength and ductility when compared to Specimen 59. The actual differences in the properties found are then primarily due to both thermal environment and irradiation.

TABLE III
TENSILE DATA FOR IRRADIATED AND CONTROL SPECIMENS OF INCONEL 625

Specimen No.	Temperature, °C		0.2% Yield Strength, psi	Ultimate Strength, psi	% Uniform Elongation	% Total Elongation	% Reduction in Area	Material Condition	Neutron Exposure, nvt
	Irradiation	Test							
59	--	650	83,580	129,600	19.05	19.96	16.92	Solution Treated	0
373-D3	50	650	107,940	125,660	7.16	8.00	17.46	Solution Treated	1.71×10^{21}
A39X	--	650	80,000	112,000	8.17	9.01	11.50	Annealed	0
A40	740	650	72,140	96,770	4.81	5.20	7.96	Annealed	9.2×10^{19}
A45X	--	650	84,570	124,600	10.45	10.79	11.06	Solution Treated	0
A46	740	650	66,150	96,650	6.62	7.00	11.00	Solution Treated	9.2×10^{19}

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