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**IMPROVED POSTIRRADIATION TENSILE PROPERTIES
OF HASTELLOY X-280**

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OF HASTELLOY X-280

By

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Metallurgy Department

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INTRODUCTION

The combination of oxidation resistance and high temperature strength places the nickel-base alloys among the more promising materials for nuclear reactor application. These alloys, when irradiated at high temperature, however, generally suffer damage which cannot be recovered by postirradiation treatments.⁽¹⁾

Under the sponsorship of the Division of Reactor Development, USAEC, a program has been undertaken to determine the effects of modified microstructures upon the irradiation stability of nickel-base alloys. These structural modifications are made by preirradiation thermal and/or mechanical treatments and are evaluated by tensile and stress-rupture tests and by metallographic examination.

This report describes the improvements found in the postirradiation tensile properties of preconditioned Hastelloy X-280, a low-cobalt, solution-hardenable nickel-base alloy.

SUMMARY

Tensile specimens of Hastelloy X-280, a solution-hardenable nickel-base alloy, were given various experimental preirradiation thermal and/or mechanical treatments. They were subsequently irradiated at 540 °F to exposures of approximately 1×10^{20} nvt ($E \geq 1$ MeV) and then tensile tested at room temperature and 1350 °F in air. When compared with a standard commercial treatment, the experimental treatment generally showed reduced radiation damage. Some treatments increased postirradiation high temperature ductility by 100% while maintaining

strength. One treatment resulted in a 100% increase in postirradiation high temperature yield strength with only 2 to 4% ductility loss. Also, a maximum increase of 57% in postirradiation room temperature yield strength was possible while maintaining 18% uniform elongation.

Possible mechanisms for the property improvement are set forth; these are based on the character of the interaction of the microstructures, formed by the preirradiation treatments, with neutron irradiation.

EXPERIMENTAL PROCEDURE

Material

The Hastelloy X-280 was obtained from 1/2 in. and 1 in. thick plates stocked at Pacific Northwest Laboratory for the AEC-sponsored Irradiation Effects to Reactor Structural Materials Program. Table I gives the chemical composition as obtained by the National Spectrographic Laboratories. Hastelloy X-280, a low-cobalt version of Hastelloy-X, is a representative of the solution-hardenable class of nickel-base alloys. The presence of tungsten, molybdenum, and chromium may be the cause of the thermally unstable microstructure found in other studies^(2,3).

Preirradiation Treatments

The treatments were designed to precipitate and to stabilize generally the carbide phases within the matrix and to reduce the tendency for embrittlement due to precipitate formation at grain boundaries during service.

Plates of suitable size, 12 in. x 3 in. x 1 in. or 1/2 in., were cut with a water-cooled saw from the stock material and examined ultrasonically for

TABLE I. Chemical Composition of Hastelloy X-280, Heat E9518

Wt%							
<u>Ni</u>	<u>Cr</u>	<u>Fe</u>	<u>Mo</u>	<u>C</u>	<u>Mn</u>	<u>Co</u>	<u>Si</u>
Bal (47.3)	22.78	17.51	9.17	0.10	0.71	0.61	0.59
<u>W</u>	<u>Ta</u>	<u>P</u>	<u>S</u>	<u>B</u>	<u>N</u>	<u>H</u>	<u>O</u>
0.56	<0.01	0.007	0.005	<0.001	0.064	0.004	0.016

Total Rare Earths: <20 ppm

Trace Elements, wt%					
<u>Al</u>	<u>Cu</u>	<u>V</u>	<u>Zr</u>	<u>Sn</u>	<u>Ti, Mg, Bi, Pb, Ag, Cb</u>
0.26	0.030	0.12	0.07	0.010	Each <0.01

homogeneity of grain size and absence of voids. They were then solution-treated at 2150 °F for 1 hr and air cooled. These plates were cold rolled 0, 10, 15, or 20% along the original longitudinal fabrication direction. Tensile specimens (Figure 1) were machined from the plates, with their tensile axis parallel to the rolling direction. The machined specimens were examined for quality of finish with zyglo penetrant dye, ultrasonically checked for voids which may have escaped detection in the earlier examination of the plate stock, and finally given a dimensional tolerance examination with an optical comparator. The acceptable specimens were then annealed at either 1000 °F or 1500 °F and air cooled. The standard treated specimens received only the solution treatment at 2150 °F for 1 hr followed by air cooling. (Table II summarizes these treatments.)

Metallographic examination was made of as-heat treated wafers taken transverse to the rolling direction of the plates. The etchant was a solution

of 92% HCl, 3% HNO₃, 5% H₂SO₄ (by volume) where the samples were immersed for 5 to 15 sec (required etching time varied with experimental treatment). Electron micrographs were made by using negative faxfilm and carbon replicas shadowed with UO₂. Figure 2 shows two examples of the experimental treatments. The cold worked specimen shows a less particulate grain boundary precipitate and a dispersion of precipitates within the matrix along the slip lines in contrast to the continuous grain boundary deposition and cleaner matrix of the specimen heat treated without prior cold working.

Irradiation Conditions

Specimens were irradiated in the G-7 hot water loop of the Experimental Test Reactor (ETR) at Arco, Idaho. The irradiation temperature was 540 °F. The average neutron exposure was 1.05×10^{20} nvt ($E > 1$ MeV) and ranged from 0.65 to 1.22×10^{20} nvt for the specimens given the experimental treatments to 1.41×10^{20} nvt for those given the standard treatment. The specimens receiving the experimental treatments had

TABLE 2. *Preirradiation Treatments for Tensile Specimens of Hastelloy X-280*

Standard Treatment:	2150 F/1 hr, A.C.
Experimental Treatment No. 1:	2150 F/1 hr, A.C.; 0% C.W.; 1000 F/24 hr, A.C.
Experimental Treatment No. 2:	2150 F/1 hr, A.C.; 0% C.W.; 1500 F/24 hr, A.C.
Experimental Treatment No. 3:	2150 F/1 hr, A.C.; 10% C.W.; 1000 F/24 hr, A.C.
Experimental Treatment No. 4:	2150 F/1 hr, A.C.; 10% C.W.; 1500 F/24 hr, A.C.
Experimental Treatment No. 5:	2150 F/1 hr, A.C.; 15% C.W.; 1000 F/24 hr, A.C.
Experimental Treatment No. 6:	2150 F/1 hr, A.C.; 15% C.W.; 1500 F/24 hr, A.C.
Experimental Treatment No. 7:	2150 F/1 hr, A.C.; 20% C.W.; 1000 F/24 hr, A.C.
Experimental Treatment No. 8:	2150 F/1 hr, A.C.; 20% C.W.; 1500 F/24 hr, A.C.

FIGURE 1. *Tensile Specimen*

been arranged such that those cold rolled the least also received the least exposure, and vice versa. Room temperature tensile data (Figure 3) showed no effect of this varying exposure, for irradiation damage (ie, property change relative to controls) was found to decrease with increasing exposure rather than to increase with exposure.

Duplicate specimens for use as controls were held in an out-of-reactor

loop under the same conditions of time, temperature, and water chemistry as those irradiated. Figure 4 shows a typical specimen configuration used for in-reactor or out-of-reactor exposure.

Test Conditions

All specimens were tested in air on an Instron tensile testing machine at a crosshead speed of 0.05 in./min. Half of the specimens were tested at room temperature and the other half at 1350 °F. For the room temperature

(Originally 3000X)

2150 F/1 hr, A.C.;
No Cold Work;
1500°F/24 hrs, A.C.

(Originally 3000X)

2150 F/1 hr, A.C.;
20% Cold Work;
1500°F/24 hrs, A.C.

*FIGURE 2. Examples of Experimental
Treatments for Hastelloy X-280*

tests, a standard Baldwin PM-3A extensometer measured specimen extension through the yield point (0.2% offset); for the 1350 °F tests, a "push-rod" extensometer developed at BNW (see Appendix) was used. Extension beyond the yield point was measured by cross-head motion. The furnace setup used for the irradiated specimens is shown in Figure 5. A three-zone Heavi-Duty furnace with a duplicate extensometer was used for testing the control specimens. Set point temperature was maintained within ± 5 °F; temperature variation along the 1.20 in. gage section of the specimen was maintained within 10 °F of set point. Time for furnace temperature to reach equilibrium was approximately 30 min and soak time prior to testing was 15 min.

After testing, the specimens were examined to determine where fracture occurred. If fracture occurred at an extensometer point indentation, the data were rejected. The data from two of the specimens were so rejected.

Except for specimens given the standard treatment and for those given the 15% cold roll and 1000 °F anneal treatment, for which each data point represents two specimens, data points were based on one tensile specimen each. Comparison of the duplicate irradiated specimens show very good agreement. At room temperature, yield strengths agreed within 1.3%, and uniform elongation within 10.5%. At 1350 °F, yield strengths agreed within 3.3%, and uniform elongation within 30% (differences between small values of elongations).

FIGURE 3. Relationship of Neutron Exposure to Radiation Damage for the Room Temperature Tensile Properties of Pre-conditioned Hastelloy X-280

FIGURE 4. Typical Specimen Holder for In-Reactor and Ex-Reactor Exposure

FIGURE 5. In-Cell Tensile Test Apparatus

RESULTS

Test Data

Postirradiation Properties

The results of the postirradiation 1350 °F tensile properties are presented in Figure 6. This shows that all experimental preirradiation treatments resulted in either equal or better high temperature strengths relative to the standard heat treatment, and that strengths increased with the amount of cold work in the treatment. Postirradiation ductility of the "zero cold-worked" material, either 1000 °F anneal or 1500 °F anneal, exceeded that of the standard treated material; ductility decreased with cold work. Comparison of the affects of the two annealing temperatures for the experimental treatments shows that strengths of the 1000 °F annealed material are greater than those of the 1500 °F annealed material. Ductility behaves in a reverse manner. Substantial necking strain occurs for all cold work levels in the 1500 °F annealed material, but only after appreciable cold work in the 1000 °F annealed material.

The results of the postirradiation room temperature properties are presented in Figure 7. Although the experimentally treated material initially starts with yield strengths lower than, and with ultimate strengths equal to, those of the standard treated material, the strengths of the experimentally treated material rise markedly with cold work to far exceed those of the standard treated material. The ductility of the experimentally treated material is initially lower than for that given the standard treatment, and though ductility continues to lower with cold work, it is still appreciable

(18% U.E.) after 20% cold work. Examination of the data for the experimentally treated material shows the specimens annealed at 1500 °F to be weaker and less ductile than those annealed at 1000 °F. Also, the rate of decrease of ductility for the 1500 °F material levels off so that at 20% cold work, it equals that of the 1000 °F material.

Comparison of Control and Irradiated Properties

A comparison of the control and postirradiated 1350 °F tensile properties of specimens given the standard treatment and of those given the experimental treatments is presented in Figure 8. Only the data for 1000 °F anneal specimens is presented since the overall picture for the 1500 °F annealed specimens was found to be similar. There is less difference in UTS between controls and irradiated for the zero cold work treatment than for the standard treatment, though for both, as well as for the cold worked material up to 10% C.W., the effect of irradiation is to decrease strength. After the strength maximum at 10% C.W., the irradiated material becomes stronger than the controls because of a sharp weakening of the controls. The reduction in uniform elongation caused by irradiation is much less severe for the zero cold worked material than for the standard treated material. Also, the reduction in total elongation is a little less severe for the zero cold work material, though there is very little necking strain for either. The large necking strain found for all controls is reduced markedly by irradiation until a minimum occurs at 10% cold work, at which point the drop in total elongation stops.

Standard Treatment: 2150°F/1 hr., A.C.
 Experimental Treatment: 2150°F/1 Hr., A.C.;
 Cold Roll; Anneal/24
 hrs., A.C.

FIGURE 6. Postirradiation 1350 °F Tensile Properties of Hastelloy X-280

FIGURE 7. Room Temperature Postirradiation Tensile Properties of Hastelloy X-280

FIGURE 8. Effect of Irradiation in the
 1350 °F Tensile Properties of Hastelloy
 X-280

The comparison of the control and postirradiation room temperature tensile properties of the standard and experimental material is presented in Figure 9. There is a reduced effect of

irradiation upon the strengths of the zero cold worked material relative to those of the standard treatment and a decreasing effect (both in magnitude and in percentage) with increasing cold work. This decrease is most pronounced for yield strengths. The ductility of the zero cold worked material shows greater radiation damage than does that of the standard treatment, but this effect reduces with cold work. Necking strain is essentially the same for control and irradiated material.

Improvement in Properties Afforded by Some Treatments

The important result of this investigation is that improved postirradiation tensile properties can be obtained through preirradiation treatments. When compared with standard treated irradiated material, some treatments increased high temperature ductility by 100% while maintaining strength levels (Figure 10). One treatment resulted in a 100% increase in postirradiation high temperature yield strength with only a 3% reduction in uniform elongation (Figure 11). Generally, susceptibility to radiation damage was lessened. This is illustrated for the high temperature properties by the comparison in Figure 12 of control and irradiated data for the standard treatment and two experimental treatments. The effect is most dramatic for uniform elongation. Room temperature properties (Figure 7) showed a maximum increase of 57% in yield strength over the standard treated material while maintaining useful ductility of 18%. The reduced effect of irradiation with cold work was noted earlier (Figure 9).

FIGURE 9. Effect of Irradiation on the Room Temperature Tensile Properties of Hastelloy X-280

FIGURE 10. Improved High Temperature Postirradiation Tensile Ductility of Hastelloy X-280

FIGURE 11. Improved High Temperature Postirradiation Strength of Hastelloy X-280

FIGURE 12. Improvement in Radiation Damage Resistance of Hastelloy X-280 High Temperature Properties

DISCUSSION

Improved Properties

It has been shown that the postirradiation ductility, strength, and radiation damage resistance of material irradiated at 540 °F to an exposure of approximately 1×10^{20} nvt can be improved by suitable preirradiation treatments. A detailed analysis of mechanisms must await metallographic examination of the buttonhead and stressed sections of the tensile specimens. This examination is in progress and will be presented in a future topical report. However, comments can still be made on some possible mechanisms.

The improved ductility found for some treatments is quite encouraging,

even though some evidence^(4,5) indicates that the transmutation reaction of boron to form lithium and helium can have embrittling effects upon grain boundaries at elevated temperatures. The material contained 1 ppm boron, a concentration that can cause embrittlement upon transmutation⁽⁵⁾. The ductility observed for an experimentally treated material improved substantially over that for the standard treated material. Since boron has been observed to increase ductility in unirradiated material by controlling precipitation reactions⁽⁶⁾, it is not surprising that these preirradiation treatments which are, in effect, controlling grain boundary precipitates, should also improve ductility. The actual mechanism

whereby the embrittling effect of the lithium and helium produced is offset is not clear. If nucleation of voids by helium agglomeration is the cause of embrittlement, the effect of the treatments might be the reduction of crack propagation paths associated with continuous grain boundary deposits, as a similar effect of certain heat treatments has been found in a precipitation-hardenable alloy⁽⁷⁾. If, instead, the result of the transmutation reaction is primarily that of atomic displacement due to lithium recoil⁽⁴⁾, (which could aid in formation of additional precipitates acting as pins to reduce grain boundary sliding), then the effect of the treatments may be the reduction of the amount of precipitation that could occur at the grain boundary by having already developed pre-precipitation nuclei or precipitates elsewhere in the grain.

The strength increase shown in Figure 11 results (at least in part) from the nucleation and pre-precipitation clustering (1000 °F anneal) and/or precipitation (1500 °F anneal) produced by these treatments. Both Wagoner⁽³⁾ and Kaufman⁽⁸⁾ have suggested that the increase in hardness found after 100 hr exposures of Hastelloy-X at 1300 or 1400 °F is caused by the fine matrix precipitate they observed.

The mechanism for the reduced radiation damage shown in the room temperature properties (Figure 9) may be explained this way. Radiation induces various defects which normally harden and embrittle the material. However, cold work and annealing produce large defect structures which may predominate over the effect of irradiation. In

fact, the figure shows that the yield strength increase caused by irradiation drops from 50% for the standard treated material to only 5% for the 20% cold worked, 1000 °F annealed material. Radiation-induced embrittlement also decreases with cold work.

At the higher test temperature (1350 °F), radiation-induced defects, per se, are probably annealed out during the pretest soak⁽⁵⁾ and their only effect on strength would be the enhancement of normal diffusion controlled reactions such as precipitation. This could result in either lattice strengthening or weakening, depending upon which mechanism, loss of solid solution strengthening or precipitation hardening, became important. If nucleation sites or clusters were already formed by preirradiation treatments, the effect of irradiation would probably increase their size rather than induce further nucleation; Figure 8 does show less reduction in UTS for the zero cold worked material than for the standard material. Also, the reduction in uniform elongation due to irradiation was probably less because of the same mechanism. Actually, the ductility, including the appreciable necking strain (8%) for the zero cold worked material annealed at 1500 °F, is even better. Presumably, this improved ductility results from growth of the even larger particles induced by the higher annealing temperature of this treatment. This amplifies what was discussed earlier regarding the boron effect.

Further Study

A metallographic examination of the tensile specimens must be made to determine the validity of mechanisms

suggested above and other areas of question not considered here. Among these is the drop in the 1350 °F strength of the control specimens cold worked more than 10% and annealed at 1000 °F (Figure 8). (Similar, though less striking behavior was also found for material annealed at 1500 °F.)

Another important area of study is the creep-rupture behavior of these materials. If the effect of these treatments is to cause cluster formation or precipitates to occur after only a 24 hr anneal, what will the subsequent effect be of hundreds or thousands of hours of service at 1350 °F? To answer this question, a test program has been initiated upon irradiated specimens that have received some of the same treatments described here. (The results will be presented in a topical report.)

CONCLUSION

It has been demonstrated that for Hastelloy X-280, irradiated at 540 °F to approximately 1×10^{20} nvt, it is possible to improve certain postirradiation properties. Radiation damage to high temperature properties was generally reduced. One treatment increased uniform elongation at 1350 °F by 100% while maintaining strength, and another improved the 1350 °F yield strength 100% with only 2 to 4% reduction in elongation. Increases in room temperature yield strength up to 57% also occurred while maintaining at least 18% uniform elongation.

It has been demonstrated that the deleterious effect of the transmutation reaction of boron to lithium and helium upon high temperature tensile ductility can apparently be overcome by an appropriate heat treatment.

Some interim suggestions have been proposed for the mechanisms responsible for the improved properties. It is suggested that ductility improvements result from the control, through the preirradiation treatments, of irradiation-induced grain boundary embrittlement reactions. Strength increases are probably due, in part, to the pre-precipitate clustering or precipitation produced by the treatments. The mechanisms for the reduced radiation damage depends on the temperature. At room temperature, the defect structure, resulting from the cold work and annealing treatment, overshadows the effect of the irradiation-induced defects, and as cold work increases, the effect of radiation decreases. At 1350 °F, the effect of irradiation may only increase the size of the clusters of precipitates already formed by the preirradiation treatment rather than nucleate others; the change in strength would then be less.

It is concluded, however, that a more detailed analysis of the mechanisms must await the results of metallographic examination of the tensile specimens.

The effect of long-term service upon the stability of these treatments must be investigated; this investigation is in progress.

APPENDIX

HIGH TEMPERATURE PUSH-ROD
EXTENSOMETER FOR TENSILE
TESTING IRRADIATED SPECIMENS

Those in industry and research involved with measuring the tensile properties of materials have long been concerned with improving the technique of measuring tensile strain during high temperature testing. Ideally, strain should be monitored directly at the gage section of the specimen by a device attached thereto and contained wholly within the furnace heat zone. Such a device for test temperatures to 900 °F has recently been developed here⁽⁴⁾, and techniques for meeting the same requirements at higher temperatures are being extensively investigated.

An interim solution to the problem of measuring tensile elongation remotely at high temperature is the push-rod extensometer shown in the next several figures. This device has undergone simultaneous development here with the "clip-on" devices of lower temperature capability.

This extensometer was developed to meet our particular requirements of light weight (our irradiated specimens are subsized with a 0.125 in. dia gage section), ease of remote manipulation, and high temperature strength, stability, and corrosion resistance. Figure A-1 shows the yokes, which attach directly to the specimen gage section, and the mounting block, which exactly spaces the yoke points 1.00 in. apart. The specimen and yoke points in this figure are not in normal operating positions, and thus show detail more clearly. The points are made of titanium carbide and the yokes are Hastel-

loy X-280. Titanium carbide allows repeated usage at a temperature which results in reduced down-time, and Hastelloy X-280 has excellent corrosion resistance. The points are adjustable to allow correct penetration into the specimen. It has been found that total penetration from both halves of a yoke can be as little as 0.003 in. and still hold the yoke on the specimen. This is extremely important since irradiation drastically embrittles these materials so that any deep notch can result in premature failure. Penetration of the pairs of points is normally preset to the nearest 0.001 in., for the differing diameters allowable within the specimen diameter specification (0.125 ± 0.001 in.), by the use of rods machined to these allowable diameters minus 0.003 in. The desired setting occurs when the points just touch the rod. Data rejection, because of premature failure at the extensometer points, has been drastically reduced by this technique.

Figure A-2 shows the yokes assembled on the specimen, while the extensometer is calibrated with a Boeckeler* precision micrometer as shown in Figure A-3. There are two sets of push rods, each attached to a Baldwin PS-3M microformer. Not only is there an amplified output, but the extensometer becomes an averaging type, and the algebraic sum of the two signals is recorded. This tends to compensate for any extension due to inadvertent bending. The push rod assembly is made of a stainless steel rod within a stainless steel tube. The springs provide a little

*Manufactured by Arizona Tool and Die Company.

FIGURE A-1. Extensometer Yokes and Mounting Block

FIGURE A-2. Assembled Extensometer Yokes

FIGURE A-3. Extensometer Calibration Jig

FIGURE A-4. Assembled Extensometer in Remote Tensile Test Furnace

spring-loaded tension in final assembly at the furnace to reduce unwanted tube motion from air currents. Figure A-4 shows the final assembly within the remote tensile test furnace. The plate seen below the furnace is attached to the bottom Instron pullrod, and it is against this plate that the spring tension is applied. The figure also shows four horizontal rods which have been welded to each of the four tabs connecting the push rods to the yokes. These four rods facilitate remote manipulation of the tabs.

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