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**IMPROVED POSTIRRADIATION STRESS —
RUPTURE PROPERTIES OF HASTELLOY X-280**

I. S. Levy

July, 1966

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RUPTURE PROPERTIES OF HASTELLOY X-280

By

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Metallurgy Research Section
Metallurgy Department

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INTRODUCTION

The requirements of oxidation resistance and high temperature strength place the nickel-base alloys among the more promising materials for nuclear reactor applications. When these alloys are irradiated at high temperature, however, they generally suffer damage which cannot be remedied by postirradiation treatment.⁽¹⁾

As part of a program sponsored by Division of Reactor Development and Technology, USAEC, to determine the effects of irradiation on the mechanical properties of materials, efforts have been undertaken to determine the extent to which modifications to microstructures can improve the irradiation stability of nickel-base alloys. These microstructural modifications are made by preirradiation thermomechanical treatments. Tensile and stress-rupture tests and metallographic examination provide criteria for evaluating the effectiveness of the treatments.

Among the alloys being investigated is Hastelloy X-280, a low-cobalt version of the solution-hardenable nickel-base alloy Hastelloy X. Several experimental thermomechanical treatments have been shown to improve postirradiation tensile properties, at room temperature and 1350 °F, when compared to

a standard treatment.⁽²⁾ Metallographic examination of some of the tensile specimens, subsequent to testing at 1350 °F, showed evidence of precipitation induced by strain and irradiation.⁽³⁾

This report describes the improvements found in the postirradiation stress-rupture properties of preconditioned Hastelloy X-280.

SUMMARY

Stress-rupture specimens of Hastelloy X-280, a solution-hardenable nickel-base alloy, were given various experimental preirradiation thermomechanical treatments. The specimens were subsequently irradiated at 540 °F to exposures of approximately 1×10^{20} nvt ($E > 1$ MeV) and 3×10^{20} nvt thermal. When compared to a standard treatment, some experimental treatments increased postirradiation rupture life up to 106%, with no change in postirradiation minimum creep rate. These treatments increased duration of postirradiation secondary creep up to 115% and increased the duration of postirradiation tertiary creep up to 107%.

Property improvements are interpreted based on the interaction of the microstructures formed by the preirradiation treatments with the neutron

environment and subsequent thermal and stress environments associated with the testing. Relationships of these results to reactor material fabrication are considered.

EXPERIMENTAL PROCEDURES

Material

The Hastelloy X-280 was obtained from 1/2 in. and 1 in. thick plates (in the mill annealed condition) stocked at Battelle-Northwest Laboratory for participants in the AEC-sponsored Irradiation Effects on Reactor Structural Materials Program. Table I presents the chemical composition as reported 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 shown by this alloy in several studies.⁽³⁻⁸⁾

Preirradiation Treatments

The treatments were designed to reduce embrittlement due to carbide deposition at grain boundaries during service by precipitating and stabilizing carbide phases within the matrix.

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>li</u>	<u>O</u>
0.56	<0.01	0.007	0.005	<0.001	0.004	0.004	0.016
Total Rare Earths, 20 ppm							
Trace Elements:							
<u>Al</u>	<u>Cu</u>	<u>V</u>	<u>Zr</u>	<u>Sn</u>			
0.26	0.030	0.12	0.07	0.010	;		
<u>Ti, Mg, Bi, Pb, Ag, Cb</u>							
Each <0.01%							

Plates with dimensions of 12 in. x 3 in. x either 1 in. or 1/2 in. were cut from the stock material with a water-cooled saw. Uniformity of grain size and absence of voids was established by ultrasonic tests.

The plates were then solution-treated at 2150 °F for 1 hr, air cooled, and cold rolled to 0, 10, 15, or 20% reduction in thickness. The rolling direction was parallel to the original longitudinal fabrication direction, and stress-rupture specimens (Figure 1) were machined with their tensile axes parallel to the rolling direction. The machined specimens were examined for quality of finish with a penetrant dye; ultrasonically examined for voids, which may have escaped detection in the earlier examination of the plate stock; and finally checked for dimensional tolerance with an optical comparator. The acceptable specimens were then annealed at either 1000 °F or 1500 °F and air cooled. Reference specimens received only the standard solution treatment at 2150 °F for 1 hr followed by air cooling. Previous metallographic studies⁽²⁾ showed that heavy cold-working provided lattice strain and that subsequent aging resulted in precipitation at the strain sites. Table II summarizes the heat treatments given the test specimens.

FIGURE 1. Stress-Rupture Specimen

TABLE II. Preirradiation Treatments for Stress-Rupture Specimens of Hastelloy X-280

Standard Treatment:		2150 °F/1 hr, A.C.	
Experimental Treatment #1:		2150 °F/1 hr, A.C.;	0% C.W.; 1000 °F/24 hr, A.C.
"	"	#2:	" 1500 °F/24 hr, A.C.
"	"	#3:	" 10% C.W.; 1000 °F/24 hr, A.C.
"	"	#4:	" 1500 °F/24 hr, A.C.
"	"	#5:	" 15% C.W.; 1000 °F/24 hr, A.C.
"	"	#6:	" 20% C.W.; 1500 °F/24 hr, A.C.

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. Fast neutron exposures ($E > 1$ MeV) ranged from 1.05×10^{20} nvt for those specimens that had received the standard treatment to 1.22 to 1.42×10^{20} nvt for those that had been given the experimental treatments. Thermal neutron exposures ranged from 2.79×10^{20} nvt for the standard treated specimens to 3.13 to 3.50×10^{20} nvt for the experimentally treated specimens.

Test Procedures

All stress-rupture specimens were tested in air at 1350 °F and 18,000 psi (the 1000 hr rupture stress for standard treated unirradiated material.) The tests were performed at Battelle-Columbus on four lever-arm units having lever arm ratios of from 9.35:1 to 9.62:1. A dial gage was attached to the load pan to provide creep data for most of the specimens. Though the data so obtained could have only an approximate absolute value, it would have important internal correlative value for determining the effects of the preirradiation treatments. Bending due to nonalignment was determined to be less than 6%. The furnaces were a single

zone, multiple tap type, with an overall length of 9 in. Temperature was maintained along the gage section of the specimen to within ± 5 °F of set point. The specimen was heated under a 1 lb load (50 psi) and allowed to soak at test temperature for 1 hr. The 1 lb load was then removed, and a bucket of lead shot totaling 20% of the desired final weight was placed on the pan. The remainder of the final weight in lead shot was placed in the bucket at the rate of 20 lb/min. Duration of the test was determined by a timer connected through a switch to the load pan.

Duplicate specimens were used for three of the seven preirradiation treatments tested.

RESULTS

Effect of Varying Neutron Exposure

Time-to-rupture data representing all treatments tested showed no correlation between the small variation in neutron exposure, either thermal or fast ($E \geq 1$ MeV), and rupture life at 1350 °F.

Postirradiation Properties

Comparison of the three sets of duplicate specimens is given in Table III. Between duplicate specimens, time-to-rupture was found to vary from 11.2 to 44% and minimum creep rate to vary from 1.5 to 13%.

TABLE III. Comparison of Stress-Rupture Data for Duplicate Specimens

Specimen Number	Heat Treatment	Time-to-Rupture, hr	Minimum Creep Rate	Duration of Creep Stages, hr		
				Primary	Secondary	Tertiary
B001	*	316.2	6.9×10^{-5}	55.0	200	61.2
B002	*	280.2	6.8	57.5	210	12.7
B051	3	216.1	5.5	5.0	195	16.1
B052	3	150.1	6.2	0.	138	12.6
B053	4	599.2	7.4	30.0	440	129.
B054	4	539	4.9	15.0	455	69.3

* Standard Treatment

The effect of the preirradiation treatments on postirradiation time-to-rupture is presented in Figure 2. The specimens given the preirradiation treatments that included the 1500 °F anneal had better rupture lives than did either the standard treated specimens or those given the treatments that included the 1000 °F anneal. Time-to-rupture decreases as the amount of cold work in the preirradiation treatment is increased.

The effect of the preirradiation treatments on various postirradiation creep properties was examined. No significant effect of preirradiation

FIGURE 2. Effect of Preirradiation Treatments on Postirradiation Time-to-Rupture for Hastelloy X-280

treatment upon postirradiation minimum creep rate was found. These rates varied only from 4.9 to $8.7 \times 10^{-5} \%$ /hr and, considering the inadequacies of the measuring technique used, these differences cannot be considered significant.

The effect of the preirradiation treatments upon the postirradiation duration of primary creep is shown in Figure 3. The standard treatment,

FIGURE 3. Effect of Preirradiation Treatments on Duration of Postirradiation Primary Creep for Hastelloy X-280

which included no preirradiation cold work or annealing, resulted in a much longer primary creep stage than did the experimental cold work and annealing treatments.

The effect of the preirradiation treatments upon the postirradiation duration of secondary creep is shown in Figure 4. As with time-to-rupture, the 1500 °F annealed material exhibits longer duration than the standard treated or 1000 °F annealed material, and the duration of secondary creep is decreased as the amount of cold work in the treatment is increased.

The effect of the preirradiation treatments upon the postirradiation

duration of tertiary creep is shown in Figure 5. Again, the 1500 °F annealed material exhibits the longest duration.

Improvement in Stress-Rupture Properties Afforded by Preirradiation Treatments

An important result of this investigation is that improved postirradiation stress-rupture and creep properties can be obtained through preirradiation treatments. When compared with standard treated material, some treatments increased high temperature time-to-rupture up to 106%, secondary creep duration up to 115%, and tertiary creep duration up to 107%. This is summarized in Figure 6. Since creep data were not available for the specimen having the best rupture life (616.8 hr for the 0% cold work, 1500 °F anneal treatment), the average data from the specimens with the next highest rupture life (H.T.#4) were used in the figure. Based on the effect of cold work on secondary creep (Figure 4), a higher value for the duration of secondary creep for the 0% cold worked material would be expected but a slight reduction might be expected in duration of tertiary creep (Figure 5).

DISCUSSION

Improved Properties

The effect of the treatments upon the high temperature postirradiation stress-rupture properties is the increase in time-to-rupture through the increase of the durations of secondary and tertiary creep. The material receiving the experimental treatments that included the 1500 °F anneal showed the best properties. Improvement in grain boundary ductility is probably the prime cause of these property improvements. Improved postirradiation

FIGURE 4. Effect of Preirradiation Treatments on Duration of Postirradiation Secondary Creep for Hastelloy X-280

FIGURE 5. Effect of Preirradiation Treatments on Duration of Postirradiation Tertiary Creep for Hastelloy X-280

FIGURE 6. Improved Postirradiation Stress-Rupture and Creep Properties of Hastelloy X-280

1350 °F tensile necking strain (i.e. total elongation minus uniform elongation) was shown to be a result of these same treatments.⁽²⁾ This improved tensile ductility is associated with heavy irradiation- and strain-induced matrix precipitation.⁽³⁾ Correlation of time-to-rupture and duration of secondary creep with total tensile elongation and of duration of tertiary creep with tensile necking strain can be made (Figures 7 and 8). This indicates similar mechanisms are acting for improvement of both tensile and creep ductility.

The improved ductility that results from some of the treatments is quite encouraging, particularly in view of some evidence^(9,10) that the transmutation reaction of boron, forming

FIGURE 7. Correlation of Time-to-Rupture and Second Stage Creep Duration with Tensile Total Elongation for Hastelloy X-280

FIGURE 8. Correlation of Duration of Tertiary Creep with Tensile Necking Strain for Hastelloy X-280

lithium and helium, can have embrittling effects upon grain boundaries at elevated temperatures. The material contained 10 ppm boron, a concentration that can cause embrittlement upon

transmutation.⁽⁹⁾ The actual mechanism for the reduction in the embrittling effect of the lithium and helium produced is not clear. If nucleation of voids by helium agglomeration at grain boundaries is the cause of embrittlement, the effect of the treatments could be the reduction of crack propagation paths associated with continuous grain boundary deposits, which has been the effect of certain heat treatments in a precipitation-hardenable alloy.⁽¹¹⁾ The massive matrix precipitation found in irradiated and strained experimentally treated specimens⁽³⁾ probably resulted in less grain boundary deposition. If the result of the transmutation reaction is, instead, primarily that of atomic displacement in the grain boundary area due to lithium recoil⁽¹⁰⁾ (which could aid in formation of additional precipitates to act as pins to reduce grain boundary sliding), then the effect of the treatments may be that of reducing 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 effect of the cold work in some of the treatments is the reduction of rupture life (Figure 2) and the duration of secondary creep (Figure 4). The studies of the postirradiation tensile properties showed that cold working generally reduced total elongation.⁽²⁾ This is probably due to dislocation tangling caused by the cold work. Transmission microscopy of an unstressed section of a tensile specimen given a treatment consisting of 20% cold work and 1500 °F anneal shows

that massive dislocation networks are still in evidence after 24 hr at 1500 °F and 20 min at 1350 °F during the tensile test.⁽¹²⁾ From the relationship of the duration of secondary creep to total tensile elongation (Figure 7), it would then appear that the decreases in rupture life and duration of secondary creep with cold work result from the reduction in ductility caused by extensive dislocation tangling.

Since there is no significant variation in minimum creep rates among the several treatments, prior cold working has not increased the postirradiation creep strength of the material, though it did increase postirradiation tensile yield and ultimate strengths.⁽²⁾ However, the large difference in primary creep duration (Figure 3) between the standard treated material (no cold work) and the cold worked material is related to the tensile strength levels induced by the cold work (Figure 9). During primary creep, substructure is formed, and the amount is primarily dependent upon that produced during the preirradiation treatment. The duration of primary creep is dependent upon the amount of substructure formation required after the preirradiation treatment. Once the requisite structure is developed, the secondary stage of creep rate now is essentially that resulting from the highly worked structure developed by the combination of the preirradiation treatment and primary creep.

The stress-rupture tests point up an important fact; that is, for actual reactor applications, the 1500 °F annealing treatment (the treatment consisting of zero cold work and a 1500 °F anneal is chosen here since it gave the best rupture life) must be given to the

FIGURE 9. Correlation of First Stage Creep Duration with Yield Strength and Ultimate Tensile Strength for Hastelloy X-280

material prior to placement in the reactor to achieve improvement in postirradiation properties. It is not sufficient to anneal the material in situ in an irradiation environment at some high service temperature. This follows from a comparison of the behavior of the standard-treated and 1500 °F-annealed material. Both were irradiated at 540 °F after treatment. Each was then annealed at the 1350 °F test temperature for 1 hr and then stressed. The 1500 °F-annealed material had double the rupture life of the standard-treated material. The results of microstructural examinations reported earlier⁽³⁾ and of those currently in progress have indicated possible mechanisms. The unirradiated standard-treated material undergoes matrix precipitation reactions during tensile

testing at 1350 °F. No matrix precipitate is seen in the irradiated specimen, and ductility of the irradiated material is very low. For the 1500 °F annealed, irradiated material, gross matrix precipitation occurred during tensile testing. Ductility is improved over that of the standard treated material. Those observations suggest that for standard-treated material, the combined effects of irradiation at 50 °F and applied strain at 1350 °F allows those alloy constituents which normally precipitate in the matrix to migrate to the grain boundaries. However, when solute-atom clusters or nuclei are induced by aging prior to irradiation, grain boundary precipitation after irradiation is reduced and matrix precipitation is promoted. A mutual interaction, as observed for the 1500 °F annealed material between clustered solute atoms and irradiation-induced defects at 540 °F to form complex aggregates, would be expected. These aggregates would serve as incipient nuclei for precipitation under the influence of temperature and strain during post-irradiation testing. Such an explanation is consistent with available microstructural observations and with correlations between tensile ductility and stress-rupture life.

Further Study

The mechanisms of the ductility improvement need to be more thoroughly investigated not only to gain a better understanding of the nature of radiation damage in this alloy, but also to develop better preirradiation treatments. Transmission microscopy, X-ray diffraction, and microprobe analysis will be used to establish the conditions for solute-atom clustering and precipi-

tation before and after irradiation and for interactions between these metastable structures and radiation-defect states.

CONCLUSIONS

- It has been demonstrated that for Hastelloy X-280 irradiated at 540 °F to approximately 1×10^{20} nvt ($E_{\gamma} > 1$ MeV) it is possible to improve post-irradiation stress-rupture properties. Postirradiation time-to-rupture at 1350 °F was increased by some treatments up to 106% with no apparent change in minimum creep rates relative to standard treated material. The improved time-to-rupture was caused by increases in the duration of postirradiation secondary and tertiary creep of up to 115% and 107%, respectively.
- These improvements in rupture and creep properties are due to improved grain boundary ductility that results from the interaction of the microstructures, developed by the pre-irradiation treatments, and subsequent neutron irradiation and creep strain.
- For reactor structural or cladding applications, the material must be given the treatments prior to its placement in an irradiation environment.
- Further work is required to elucidate the mechanisms of ductility improvement and irradiation damage; transmission microscopy and microprobe analysis will be used.

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