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REPORT

**METALLOGRAPHIC EVIDENCE OF STRAIN - AND
IRRADIATION - INDUCED PRECIPITATION IN
PRECONDITIONED HASTELLOY X-280**

I. S. LEVY and K. R. WHEELER

APRIL, 1966



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3 3679 00060 2203

BNWL-211

UC-25, Metals, Ceramics,
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METALLOGRAPHIC EVIDENCE OF STRAIN- AND IRRADIATION-INDUCED
PRECIPITATION IN PRECONDITIONED HASTELLOY X-280

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I. S. Levy and K. R. Wheeler

Metallurgy Research Section

Metallurgy Department

April, 1966

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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, however, when irradiated at high temperature, generally suffer damage in the form of reduced ductility and strength unrecoverable by postirradiation treatments.

A program, under the sponsorship of the Division of Reactor Development, USAEC, 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.

Among the alloys being investigated is Hastelloy X-280, a low-cobalt version of the solution-hardenable nickel-base alloy Hastelloy X. Tensile specimens of this alloy, representing various experimental preirradiation treatments, were irradiated at 540 °F to approximately 1×10^{20} nvt, and then tensile tested at room temperature and 1350 °F. Several treatments have been shown to improve postirradiation properties when compared to a standard treatment.⁽¹⁾ The investigations upon this alloy continued with metallographic examination of the broken tensile specimens. This report describes some

significant results of these metallographic examinations.

SUMMARY

Tensile specimens of Hastelloy X-280, a solution-hardenable nickel-base alloy, were given various experimental preirradiation thermomechanical treatments. They were subsequently irradiated at 540 °F to approximately 1×10^{20} nvt, tensile tested at 1350 °F, and subjected to metallographic examination. Comparisons were made with metallographic examinations performed on control specimens given similar treatments.

Evidence is presented of the occurrence of precipitation induced by strain, by irradiation, and by the combination of both strain and irradiation. Tentative identification of the phases produced is made.

EXPERIMENTAL PROCEDURE

Material

The Hastelloy X-280 was obtained from 1/2- 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 of the material tested 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

TABLE 1. Chemical Composition of Hastelloy X-280, Heat E-9518

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>A</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
<u>Total Rare Earths</u>							
< 20 ppm							
Trace Elements: <u>Al</u> <u>Cu</u> <u>V</u> <u>Zr</u> <u>Sn</u> ; <u>Tr</u> , <u>Mg</u> , <u>Bi</u> , <u>Pb</u> , <u>Ag</u> , <u>Cb</u>							
0.26 0.030 0.13 0.07 0.010 Each < 0.01%							

may be the cause of the thermally unstable microstructure shown by this alloy in several studies. (2,3,4,5,6)

Preirradiation Treatments

The treatments were designed to precipitate, and stabilize generally within the matrix, the carbide phases which would normally tend to form deposits at, and embrittle, 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 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 1500 °F and air-cooled (Table 2 summarizes these treatments).

Irradiation Conditions

Specimens were irradiated for approximately 18 days in the G-7 hot water loop facility of the Experimental Test Reactor (ETR) at National Reactor Testing Station (NRTS), Arco, Idaho. The irradiation temperature was 540 °F. The average neutron exposure was 0.89×10^{20} nvt ($E > 1$ MeV), ranging from .646 to 1.22×10^{20} nvt. As previously reported, the tensile properties showed no effect of this exposure variation.¹ To determine the effects of the irradiation upon the properties and microstructure of the material, duplicate specimens were held in an ex-reactor loop under the same conditions of time, temperature, and water chemistry, and used as controls.

Tensile Test Conditions

Specimens were tested at 1350 °F in air on an Instron tensile testing

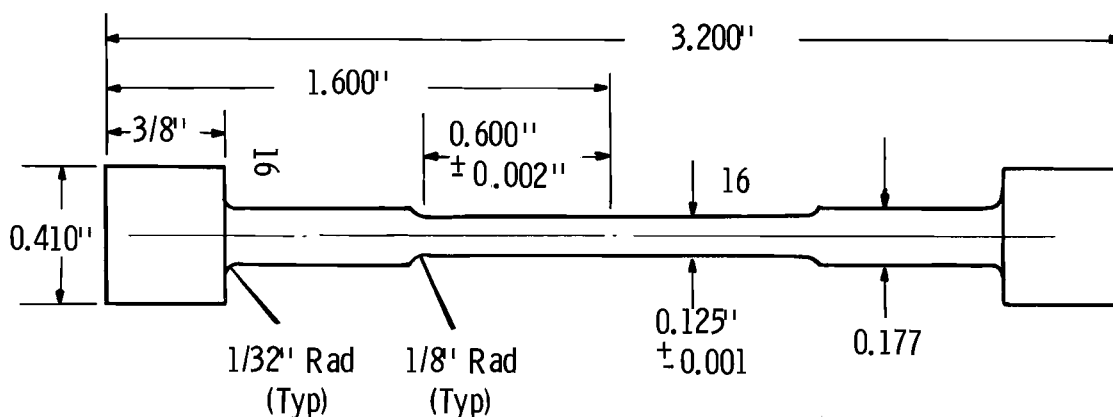


FIGURE 1. Tensile Specimen

TABLE II. Preirradiation Treatments for Tensile 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., 1500 F/24 hr, A. C.
Experimental Treatment #2	2150 F/1 hr, A. C.	10% C. W., 1500 F/24 hr, A. C.
Experimental Treatment #3	2150 F/1 hr, A. C.	15% C. W., 1500 F/24 hr, A. C.
Experimental Treatment #4	2150 F/1 hr, A. C.	20% C. W., 1500 F/24 hr, A. C.

machine at a crosshead speed of 0.05 in./min. Time for the furnace temperatures to reach equilibrium was approximately 30 min., soak time prior to testing was 15 min., and test duration approximately 5 min.

Metallographic Procedures

Subsequent to tensile testing at 1350 °F, metallographic examination was made of both control and irradiated specimens. Sections, transverse to the tensile axis, were taken from the buttonhead and from the gage section adjacent to the fracture area. The metallographic procedures used were as follows:

- The specimens were placed in metallographic mounts--bakelite for the unirradiated specimens and stainless steel for the irradiated ones. The specimens

were ground flat using a 120 grit wet belt sander.

- Rough polishing was initiated by using emery polishing paper wetted with kerosend, first with the 2/0 grade paper, then with the 3/0 grade.
- The specimens were then polished on a Syntron vibratory polisher for approximately 20 hr. Metcloth and Linde A abrasive with distilled water were used in the polishing.
- Final polishing was accomplished on the Syntron in three stages: 30 min. on Microcloth with Linde A abrasive and distilled water; 30 to 45 min. on Gamal cloth with Metpolish No. 3 and distilled water; and 30 min., or until no scratches appeared upon examination under 500 X magnification,

of electronic grade MgO on Gamal cloth with distilled water. After the final polish, precipitate particles appeared in relief at 100 X.

- To prevent staining, it was determined that etching had to proceed immediately after the MgO polish. If a delay was unavoidable, etching was preceded by a short polish with the MgO.
- The etchant used was a solution of 92% HCl, 3% HNO₃, and 5% H₂SO₄ by volume. The samples were immersed from 5 to 15 sec., with the time variation due to the influence of heat treatment and irradiation upon reaction rates.
- Electron micrographs were made from UO₂ shadowed, negative, fax-film and carbon replicas by using a Philips EM-100B microscope.

RESULTS

Comparisons were made of the micrographs from transverse sections of the buttonhead and gage sections of control and irradiated specimens. These comparisons produced evidence that precipitation could be induced by strain, by irradiation, and by a combination of strain and irradiation.

Strain-Induced Precipitation

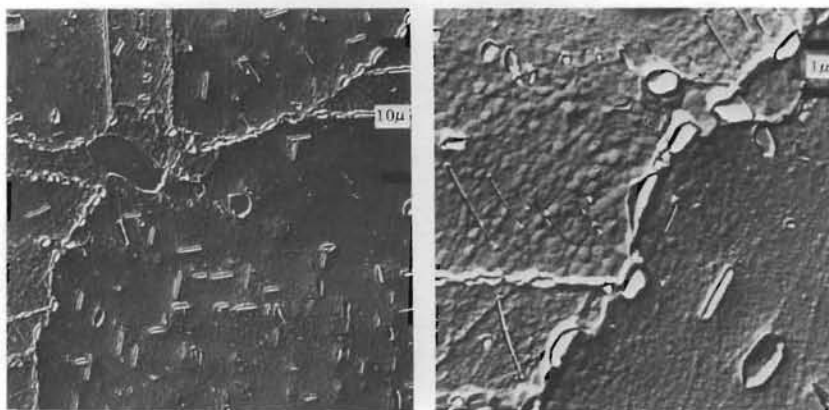
Figure 2 shows the evidence of strain induced precipitation. The figure compares the microstructure of unirradiated material from the unstrained buttonhead portion of the tensile specimen with that from the strained gage section. The specimens had been given an experimental treatment that included 20% cold-rolling prior to the 1500 °F anneal. An increase in precipitate volume in the stressed area over that which is in

the buttonhead is immediately evident. This increased precipitation appears to take the form of small triangular- or tetrahedral-shaped particles, and occurs in both the matrix and the grain boundaries. Though these particles are etched out in this figure, transmission electrons microscopy in progress on this sample shows them to be blocky and triangular.⁽⁷⁾ The amounts of the larger globular and rod-shaped matrix structures and the amounts of the globular deposits in the grain boundaries appear not to have undergone much change due to strain.

Irradiation-Induced Precipitation

Figure 3 shows evidence of irradiation-induced precipitation. The figure compares an unirradiated buttonhead with an irradiated buttonhead from specimens each of which had received an experimental treatment that included 15% cold-rolling prior to the 1500 °F anneal. Since these are sections from the unstrained portions of the specimens, no precipitation due to strain is involved. An increase in precipitate volume in the irradiated buttonhead is evident on close examination of the 3000 X micrograph. At the 10,000 X magnification, this increase is shown to be due to the formation of an extremely small, mainly triangular shaped matrix precipitate. It appears here also that there has been no increase in the volume of globular and rod-shaped matrix structures, nor of the grain boundary deposits. The apparent denuding of the area adjacent to one of the grain boundaries in the irradiated buttonhead is not wholly representative of the sample,

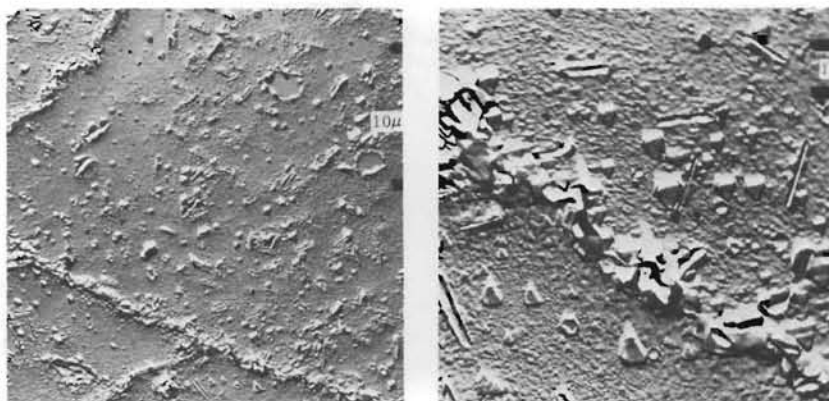
UNIRRADIATED BUTTONHEAD



(a)
Neg. 3366A (Originally 3000X)

(b)
Neg. 3366B (Originally 10,000X)

UNIRRADIATED STRESSED SECTION

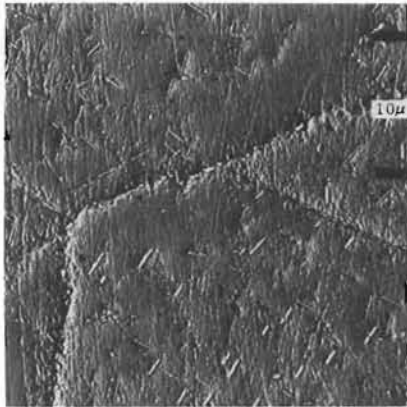


(c)
Neg. 3506A (Originally 3000X)

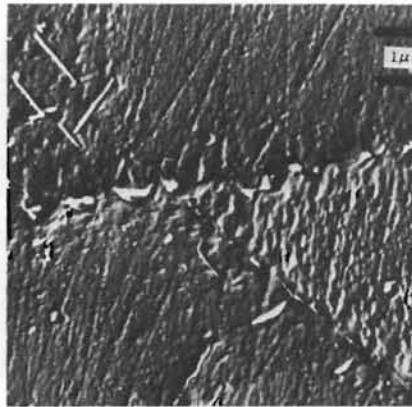
(d)
Neg. 3506B (Originally 10,000X)

FIGURE 2. Strain-Induced Precipitation
(Heat Treatment: 2150 °F/1 hr, A.C.; 20%
C.W.; 1500 °F/24 hr, A.C.)

UNIRRADIATED BUTTONHEAD

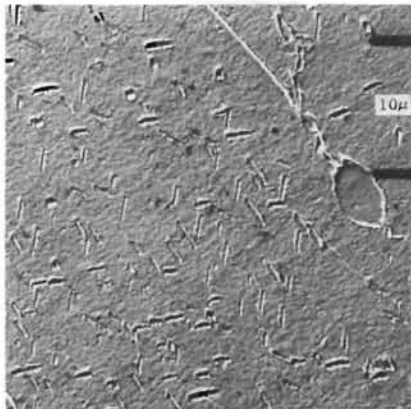


(a)
Neg. 3360A (Originally 3000X)



(b)
Neg. 3360B (Originally 10,000X)

IRRADIATED BUTTONHEAD



(c)
Neg. 3383A (Originally 3000X)



(d)
Neg. 3383B (Originally 10,000X)

FIGURE 3. Irradiation-Induced Precipitation
(Heat Treatment: 2150 °F/1 hr, A.C.; 15% C.W.;
1500 °F/24 hr, A.C.)

though the narrow, continuous grain boundary deposit that is seen is as common as the globular type.

Combined Strain- and Irradiation-Induced Precipitation

Figure 4 shows evidence of precipitation induced by the combined effect of strain and irradiation.

The figure compares the buttonheads and gage sections of an unirradiated tensile specimen with those of an irradiated specimen, with each specimen having received the same treatment. In this instance, the treatment did not involve cold-rolling prior to the 1500 °F anneal. The

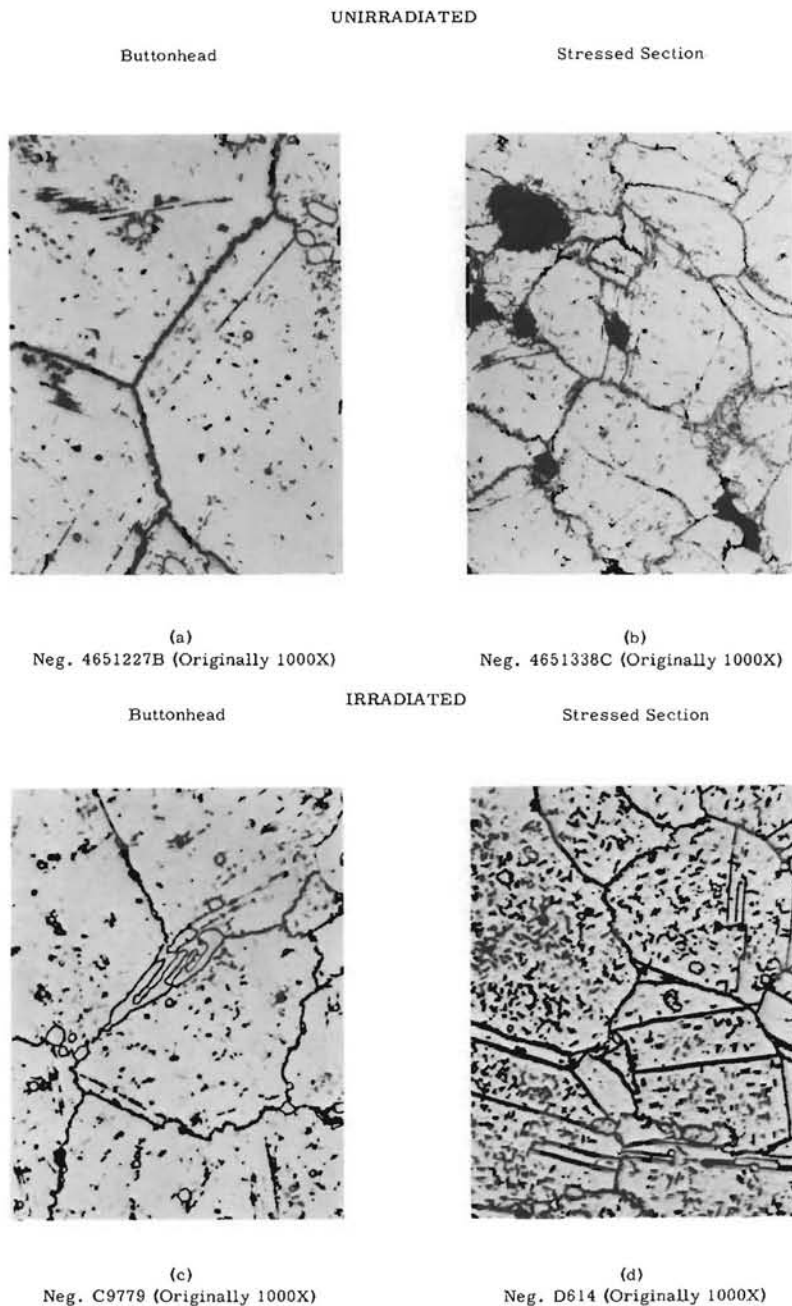


FIGURE 4. Combined Irradiation-Induced and Strain-Induced Precipitation (Heat Treatment: 2150 °F/1 hr, A. C.; 0% C.W.; 1500 °f/24 hr, A.C.)

irradiated gage section shows a large increase in precipitate over that seen in any of the other three sections. While there appears to be a finer precipitate in the background of the irradiated gage section micrograph, the gross effect of irradiation followed by tensile strain at 1350 °F was a massive precipitation of rod-shaped particles.

DISCUSSION

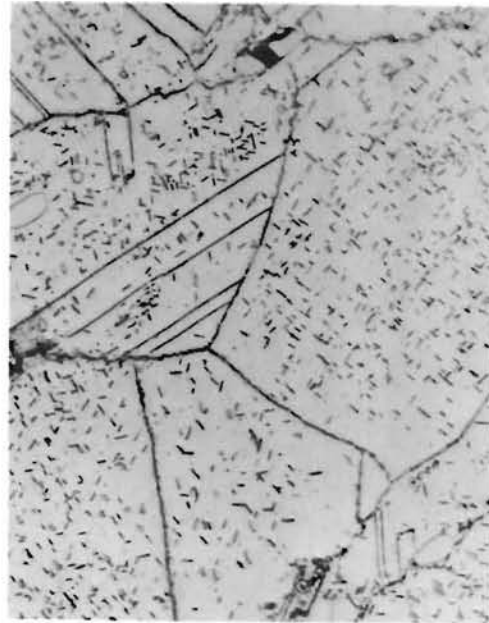
The Combined Effect of Strain and Irradiation Upon Precipitation Reactions

The material in which evidence was found of combined strain- and irradiation-induced precipitation had not received cold-rolling during the experimental preirradiation treatment. For those where evidence was found of either strain- or irradiation-induced precipitation, the experimental treatment included either 15 or 20% cold work.

In the "zero cold-worked material", the gross effect of irradiation and strain was a precipitation of the rod-like structures. This precipitate was not in evidence prior to either irradiation or strain in this material (Figure 4: a,b,c), but was already evident in that material receiving the prior cold work (Figure 2: a,b; Figure 3: a,b).

It would appear, therefore, that the combined effect of strain and irradiation on material given only the 1500 °F anneal and then strained at 1350 °F is to reproduce the effect of cold work followed by a 1500 °F anneal and a soak at 1350 °F. As evidence for this reasoning, Figure 5 shows a unstrained buttonhead section from a specimen that had received a prior

treatment that included 20% cold work, and then tensile tested at 1350 °F. The similarity of microstructure with that of the irradiated and strained section shown in Figure 4d is quite apparent. Therefore, in the temperature range of concern here, namely 1350 °F to 1500 °F, precipitation reactions result from the combination of strain and irradiation similar to those caused by extensive cold work.



4651228B

(1000X)

FIGURE 5. Buttonhead Section of Hastelloy X-280 Tensile Specimen

(Heat Treatment; 2150 °F/1 hr, A.C.; 20% C.W.; 1500 °F/24 hr. A.C.)

The precipitates shown in the Figure 4 micrographs are identified as follows. The massive, globular precipitate found in the matrix and grain boundaries is M_6C which has been shown to develop during the 2150 °F phase of the treatment.^(3,4,8) The major grain boundary phase is additional M_6C brought out by the annealing

treatment.^(4,8) The same precipitates are seen in Figure 2: a,b, and in Figure 3: a,b. Smaller amounts of M_6C also precipitate along dislocations,⁽⁸⁾ examples of which can be seen in Figure 4: a,c decorating dislocations formed around the larger M_6C globules. The identification of the rod-shaped structures resulting from the combination of strain and irradiation (or from prior cold work in the other figures) is less certain. It could be sigma phase ($FeCr$). This phase had been shown earlier to form in this alloy as long Widmanstätten platelets after aging for 100 hr at 1700 °F,⁽⁴⁾ but after only 10 hr at that temperature, this phase more often appears in a shorter form similar to that seen here.⁽⁹⁾ An investigation by Comprelli and Lewis⁽⁶⁾ of the aging behavior of Hastelloy X at 1400 °F indicated that after 1025 hr a major matrix phase of M_6C , a minor amount of AB_2 Laves types phase, and a trace of $M_{23}C_6$ had formed; positive identification of the constituents or of the morphology of these phases could not be made with the selective etching technique used. Beattie and Hagel⁽¹⁰⁾ studied the effect of a 1000 hr age at 1500 °F upon complex austenitic alloys, several of which had compositions similar to that of Hastelloy X-280. These alloys were found to precipitate both (Fe_7MO_6) and M_6C phases abundantly, with a small amount of $M_{23}C_6$. Further, their data indicated that, of these alloys compositionally similar to Hastelloy X, sigma phase appeared only in those alloys having both high titanium and high aluminum. Also, the μ phase is usually found in platelet form.⁽¹¹⁾

Therefore, it would appear that the rod-shaped structures are either μ or an M_6C -type phase.

Strain-Induced and Irradiation-Induced Precipitation

Figures 2 and 3 showed, respectively, strain-induced and irradiation-induced precipitation for specimens having received 15 and 20% cold work prior to the 1500 °F anneal.

The induced precipitate has a morphology completely distinct from the rod-shaped precipitate discussed above. Also, from the higher magnification necessary to see this phase it would appear that the amount of it is smaller. Kaufman⁽³⁾ found evidence of trace amounts of a Laves phase in Hastelloy X after aging at 1400 °F for 100 hr or at 1550 °F for 48 hr. Comprelli and Lewis⁽⁶⁾ found a minor amount of an AB_2 type Laves phase and a trace of $M_{23}C_6$ also after aging at 1400 °F. Beattie and Hagel⁽¹⁰⁾ found a small amount of $M_{23}C_6$ after 1000 hr at 1500 °F. It is conceivable that after the initial precipitation of the rod-shaped phase (M_6C or μ) had resulted from the experimental treatment, the individual effect of strain or irradiation was the precipitation of a Laves and/or a $M_{23}C_6$ phase.

Since neither of the buttonhead specimens showed this phase at 10,000 X (Figure 2, b; Figure 3, b), the individual effect of strain or irradiation was apparently an increase of the rate for the precipitation reaction.

The smaller size of the irradiation-induced precipitation, relative to that induced by strain, might be related to the smaller amount of prior cold work (15% versus 20%), to the smaller combined amount of cold-work and tensile

strain (15% plus 0% for the irradiated buttonhead versus 20% plus 15% for the strained section), or to differences in the effects of irradiation and strain upon the mechanisms of precipitate growth.

Further Study

A correlation is being made between the post-irradiation tensile behavior of preconditioned Hastelloy X-280⁽¹⁾ and the microstructures of the specimens in order to rationalize the mechanisms involved. Such an understanding could lead to improved preirradiation treatments. This study will be the subject of future reports.

An area of investigation that must be given serious consideration is that of the creep-rupture behavior of these materials. If the effect of irradiation and strain is to induce precipitation after less than 1 hr at 1350°F during a tensile test, what will be the effect of hundreds or thousands of hours of service at 1350° F? To answer the question, a creep-rupture 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

- Evidence has been presented for the occurrence in Hastelloy X-280 of precipitation resulting from the combined effect of strain and irradiation.

The precipitate induced due to the combined effect of strain and irradiation on non-cold worked specimens appears to be similar to that found in specimens that had been cold-worked but were neither strained nor irradiated. The induced precipitate

is considered to be either an M_6C or a μ phase.

- Evidence has been presented for the occurrence in Hastelloy X-280 of strain-induced and irradiation-induced precipitation. The precipitate has a very fine, triangular or tetrahedral form and may be a Laves phase, similar to that found subsequent to long thermal exposure. Strain or irradiation appears to increase the rate of this precipitation. The smaller size of the irradiation-induced precipitate relative to that induced by strain may be due to the smaller amount of prior cold work, the smaller combined amount of cold-work and tensile strain, or to differences in the effects of irradiation and strain upon precipitation growth.
- A correlation of the tensile behavior of preconditioned Hastelloy X-280 with specimen microstructure will be the subject of future reports.
- The effect of long-term service upon microstructural stability of irradiated specimens that have received the experimental treatments must be investigated; this investigation is in progress.

ACKNOWLEDGMENTS

The authors wish to acknowledge the invaluable assistance of those who performed the metallography. The preparation and optical metallography of the unirradiated specimens was performed by I. R. Robertson; M. H. Brady prepared the replicas. The preparation, optical metallography, and replication of the irradiated specimens was performed by D. H. Parks. H. C. Bowen performed all the electron microscopy.

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