FOR FIGURES I thru I8 see file copy.

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Report

to

The International Nickel Company, Inc.
Research Laboratory

on

INFLUENCE OF OVERHEATING ON THE CREEP-RUPTURE PROPERTIES OF AS-CAST INCONEL 713-C ALLOY

by

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SUMMARY

An experimental investigation was carried out to determine the influence of overheating on the creep-rupture properties of as-cast Inconel 713-C alloy at 1500°, 1700°, and 1800°F. Overheats of 0.5-hour duration to temperatures 100° and 200°F above the normal test temperature were applied after 48 hours during the course of nominal 200 hour tests.

The following results were obtained:

1) Thermal effects from overheating prolonged rupture life, mainly by delaying third-stage creep.

2) The rupture times for 100°F overheats with the stresses present were no different than when the specimens were overheated with the stress removed. This was expected because calculations from normal-constant temperature data indicated that because the time of overheating was less than 3 percent of the times for rupture at the overheat conditions, the reduction in rupture life at the base temperatures from accelerated creep during the overheats would be negligible.

3) The rupture times for 200°F overheats under stress from 1500° or 1700°F were similar to those for normal-constant temperature tests. Calculations based on normal test data indicated that about 30 percent of the rupture life would be "used-up" during the overheats. The strengthening from the thermal effect was sufficient to offset the accelerated creep and prevent a reduction in rupture time compared to normal-constant temperature tests.

4) Calculations based on normal test data indicated that rupture should occur during overheats from 1800° to 2000°F with stress present. Two tests agreed with the prediction. Two other tests were not reduced in rupture life, apparently because the individual specimens had sufficient strength at 2000°F to greatly reduce the creep damage during overheating.

5) Some 20 overheated tests indicated actual rupture times equal to or above the range of rupture times for normal tests. This validates the improvement from thermal effects being real and not due to scatter of properties of the specimens.

6) Any reduction in rupture life of Inconel 713-C from overheating under conditions simulated by the investigation would require that stress and time of overheating be sufficient to use-up more than 30 percent of the rupture life. Calculations of creep damage from stress during overheating based on "addibility-of-life-fractions" yield conservative values for this alloy.

7) The improved creep resistance of Inconel 713-C alloy from overheating agrees with the general response of nickel-base γ' strengthened alloys. Either no change or improved creep resistance is imparted by overheating. For rupture strength to be reduced, there must be a significant reduction in ductility. Ductility of Inconel 713-C was not reduced and, therefore, rupture strength was increased.

8) The report discusses the mechanisms of the observed effects and points out the limitations of the generalities due to the varied mechanisms involved in overheat effects.

INFLUENCE OF OVERHEATING ON THE CREEP-RUPTURE PROPERTIES OF AS-CAST INCONEL 713-C ALLOY

An experimental investigation was carried out to determine the response of as-cast Inconel 713-C alloy to overheats to temperatures 100° and 200°F above the normal temperature of creep-rupture tests at 1500°, 1700°, and 1800°F. The overheats were one-half hour in duration and were applied when the tests had been in progress for the first 48 hours of nominal 200 hour tests. In part of the tests, the stress was present during the overheats. In the other tests, the stress was removed during overheating and reapplied when the temperature returned to normal. The two types of data were used to determine the relative effects of thermally-induced structural changes during overheats and the damage from accelerated creep at the overheat conditions when stress was present.

The conditions of the experiments had been selected to approximate overheating which might be experienced by the alloy during service as rotating blades of aircraft gas turbines. By consideration of the relative effects of thermally-induced structural changes and accelerated creep from stress during overheating, the generality of the results was extended beyond the specific test conditions. The reliability of the information was extended further by comparison to the results of investigations of overheating for other alloys. Particular attention was given to the metallurgical principles governing the effects of overheating.

EXPERIMENTAL MATERIAL

Standard investment cast specimens having a diameter of 0.250 inch and a 1-inch gage length were used. The melting and casting were carried out in vacuum using vacuum-melted master heat stock by an investment casting foundry. Ninety-five specimens were supplied on an order from The International Nickel Company, Inc.

The master heat, No. 147, was certified to have the following chemical composition (weight percent):

C	Mn	Si	Cr	Al	Ti	Mo	Cb+Ta	Co	Ni	Fe
0.13	0.02	0.13	12.26	6.23	0.69	4.49	1.98	0.13	bal.	1.58

B	Zr	S
0.015	0.129	0.007

The tests were conducted on specimens in the as-cast condition.

GENERAL PROCEDURE

Normal-constant temperature stress-rupture time curves at 1500°, 1700°, and 1800°F were established. These curves served to define stresses for rupture in approximately 200 hours. Some replicate testing was carried out to indicate the scatter in rupture times to be expected.

Tests were then started in exactly the same way as in the normal tests under the stresses for rupture in approximately 200 hours. At 48 hours, however, the temperature was increased 100° or 200°F for one-half hour, returned to the normal test temperature, and the test then continued to rupture. The influence of the overheat was then established by comparing the rupture time with that of the normal tests.

In part of the overheat tests, the stress was not changed. In these tests, therefore, there was accelerated creep during the overheats as well as thermally-induced structural changes. In other tests, the stress was removed during the overheats to restrict the influence of overheating to thermally-induced structural changes. By thus defining the cause of observed changes in creep-rupture properties, a basis for extending the results to specific cases having other conditions of overheating than those studied was developed.

Normal-constant temperature rupture data are usually available. Data for overheats at a wide range of specific overheat conditions are generally not available. In conducting the research program, experiments were also included to obtain an indication of the usefulness of normal test data to compute the effects of specific overheats. To do this, normal rupture tests were conducted at the overheat temperatures to establish the normal rupture times under the stresses present during the overheats. These tests were conducted in the usual way using virgin specimens. Using these data, the percentage of the normal rupture time at the overheat conditions, represented by the half-hour overheats, was calculated.

If no other factor was involved, it could be expected that the rupture time at the base temperature would be reduced by the percentage of rupture life "used-up" during the overheats. The ability to calculate the effect of a stressed overheat was, therefore, checked by comparing this computed reduction in rupture life with the experimental value. This uses a so-called "addibility-of-life-fractions" concept, which assumes that the actual rupture time under varying stresses and/or temperatures will be in accordance with the sum of the life fractions represented by the individual conditions of exposure.

Experience has demonstrated that there can be, in addition to the life fractions, an additional factor of thermally-induced structural changes. Some success has been obtained in this laboratory in computing actual rupture times in the presence of thermally-induced structural changes by determining the influence of overheating with the stress removed during the overheats. The rupture time computed from the addibility-of-life-fractions is corrected for any changes due to the thermally-induced structural changes.

In conducting the tests, creep data were measured. This enabled the determination of whether thermally-induced structural changes were influencing creep-rupture properties by altering creep resistance. When combined with ductility measurements, it is possible to determine the mechanical mechanism for observed effects. Such information is useful in extending the generality of application of the data.

In an investigation of this type, the reliability of the results is considerably improved if the mechanism by which observed effects occur is understood. This generally requires a detailed extensive study of the microstructural features controlling creep resistance and fracture. Very little work of this type was carried out for this investigation. A considerable analysis of these features, however, was made on the basis of extensive experience with other alloys.

EXPERIMENTAL TECHNIQUES

The techniques used to conduct the tests are described in this section.

Creep-Rupture Testing

Creep-rupture testing was carried out in conventional beam-loaded creep-rupture units. Each sample was accurately measured before testing. Time-elongation data were obtained during the tests with a modified Martens-type optical extensometer system having a sensitivity of $\pm 1 \times 10^{-5}$ inch. The units were equipped with automatically controlled furnaces for heating the specimens. Temperature variations along the gage length were held to ±3°F. For all tests, the furnaces were turned on and allowed to come up to temperature overnight. The specimens were then placed in the hot furnace, brought on temperature, and loaded within a maximum of four hours.

Stress-strain data were obtained from measurements during the application of the load. The extensometers were read at sufficiently close intervals during the tests to provide data adequate to plot creep curves. The time for rupture was obtained from an electric timer, automatically actuated to record total elapsed time. Elongation and reduction of area were measured from the ruptured specimens after removal from the test units.

Considerable difficulty in attaching the extensometers was experienced. The final solution was to drill holes through the shoulders and insert pins to which the extensometer bars could be attached. Rather small diameter pins had to be used to avoid fracture at the point where the holes were drilled. This in turn led to problems of avoiding bending of the pins. It would have been easier to have threaded collars on the specimens ahead of the adapters. To do this, however, it would have been necessary to have a special mold made up to increase the length of the threaded portion of the specimen.

Overheat Tests

Conventional units were modified for resistance heating of the specimens for overheating. A suitable direct current was passed through the specimens using a 400 ampere D-C generator as a power supply. To prevent any disturbance of the specimen during the test, leads were fastened to the specimen holders and to insulated terminal blocks attached to the frame of the test unit before the load was applied. When the time came for application of the overheat, the generator leads were connected directly to the terminal blocks completing the circuit.

To follow and control the rapidly changing temperature during overheating, a special thermocouple arrangement was necessary to:

a) maintain intimate contact with the specimen while expansion, reduction in cross-section, and contraction occurred, respectively.

b) to eliminate the measurement of electromotive force impressed on the thermocouple circuit due to the passage of direct current.

This thermocouple arrangement consisted of spot welding two Alumel wires on either side of a single spot-welded Chromel wire. Intimate contact was maintained by the welds and the two impressed electromotive forces obtained cancelled each other out when a variable resistance, to the ends of which the two Alumel wires were connected, was properly adjusted. The calibration of the circuit was checked constantly during each overheat.

This investigation included basically two types of overheats: Overheats in the absence of stress and overheats in the presence of stress. For both types of tests, the specimens were prepared with a thermocouple welded at the center as described previously, and an additional thermocouple mechanically attached to each end of the reduced section for checks on the temperature distribution along the gage length. The tests were started exactly as in a normal creep-rupture test except that the power leads were attached to the specimen holders before application of the load. The time period at the base temperature and stress before overheating was 48 hours for both the stressed and unstressed overheat tests. The time at the overheat temperature was also kept constant in all tests at one-half hour.

In performing an overheat in the absence of stress, the following procedure was followed:

1) The furnace temperature was checked; an extensometer reading taken; the power leads from the generator were attached to the unit; and the welded thermocouple was attached to the variable resistance-indicating potentiometer set-up.

2) The load was removed.

3) After a one-minute time interval in which the furnace output was cut back and the thermocouple circuit checked, overheating was begun by applying a direct current of approximately 200 amperes from the generator to the specimen. Upon attainment of the

desired overheat temperature, the generator output was reduced to a value just sufficient to maintain this temperature.

4) The thermocouple circuit was checked constantly during the overheat period to assure elimination of measuring impressed electromotive force.

5) At the end of the one-half hour of overheating, the generator was shut off and the specimen was allowed to cool. The only forced cooling was that obtained due to the fact that cutting back the furnace output in step 3 allowed the furnace temperature to fall below the base temperature.

6) When the specimen temperature was within about 10°F of the desired base temperature, the load was reapplied. This was done because of the asymptotic nature of the approach of the specimen temperature to the base temperature. The time for the specimen to reach a temperature 10°F above the base was relatively constant and short for a given set of base conditions; whereas the time required to reach the actual base temperature was somewhat long and varied. The furnace input was then manipulated to obtain the base temperature as soon as possible.

7) Upon reaching the base temperature, extensometer readings were taken again and the test continued at base conditions to rupture.

For overheats in the presence of stress, the procedure was exactly the same as that for overheating in the absence of stress with the exception of the steps involving removal and reapplication of the load, which were omitted. Creep measurements were made during the overheats.

Metallographic Procedures

One specimen was split lengthwise and macroetched. Several were macroetched without splitting to show surface grain size.

The specimen used for macroetching was also polished and examined microscopically. In addition, typical normal rupture test and overheat test specimens were polished and examined. The specimens were cut so that a longitudinal cross-section near the fracture was polished. The specimens were mounted and mechanically polished. An electrolytic etch using 5 percent H_2SO_4 was used. The examination was confined to magnifications of 100 and 1000 diameters. The only exception was a small amount of work attempting to examine the structures under the electron microscope using collodion replicas shadowed with palladium.

RESULTS

Data are presented for normal creep-rupture tests and for specimens which had been overheated.

Normal Creep-Rupture Properties

A range of stresses was used for normal rupture tests at 1500°, 1700°, and 1800°F to provide data for stress-rupture time curves indicating the stresses for rupture in 200 hours to be used for the overheat tests. Replicate tests were then conducted at or near this stress to obtain an indication of the scatter in test data. The results (Table 1 and Fig. 1) led to the following stresses being selected for the overheat tests with the indicated range in rupture times:

Normal Test Temperature (°F)	Stress (psi)	Range in Rupture Times (hours)
1500	56,000	150 to 270
1700	26,000	165 to 265
1800	17,000	125 to 210

These ranges in rupture times are based on the curves defining the upper and lower limits of the test data in Figure 1. The number of tests was too few for a true statistical determination of the range at the stresses to be used in the overheat tests. However, the use of all the data by means of the upper and lower limiting curves improved the reliability of the ranges. The mean values for the ranges deviated somewhat from 200 hours because the stresses were necessarily selected for the overheat tests before the replicate testing indicated the ranges. However, they are sufficiently close for the objectives of the investigation.

The ranges in rupture time were no greater than had been expected for investment castings. The strength and ductility levels are understood to be normal for specimens melted and cast in a vacuum.

Rupture Properties at the Overheat Temperatures

Before any overheat testing was begun, rupture tests were conducted at the overheat temperatures under the stresses to be used. The objectives were first to determine if the pre-specified overheat conditions could be expected to yield suitable test data; and, secondly, to provide data which could be used to determine to what degree normal test data could be used to estimate the reduction in life to be expected from an overheat under stress. Only two tests were conducted at each condition to obtain an indication of the rupture times rather than a well-established range.

The test data (Table 2) are treated in the following tabulation to show the results:

Temperature (°F)	Stress (psi)	Average Rupture Time (hours)	Ratio Overheat Time (0.5 hour) to Average Rupture Time
1600	56,000	20.0	0.025
1700	56,000	1.57	0.32
1800	26,000	17.8	0.028
1900	26,000	1.85	0.27
1900	17,000	17.2	0.029
2000	17,000	0.63	0.80

When the overheat temperatures were to be 100°F above the normal temperature, this tabulation indicates that the half-hour overheat represents only 2.5 to 2.9 percent of the time normally expected for rupture at the overheat temperatures under the stresses to be used. It would, therefore, be expected that the rupture time at the normal test conditions would be reduced by no more than this amount as a result of accelerated creep at the overheat conditions. This is less than the scatter in normal test results. It was, therefore, concluded that the presence of the stresses during overheats to 100°F above the normal test temperatures should not noticeably influence rupture times.

When the overheat temperatures were 200°F above the normal test temperature, however, the half-hour would be for a larger proportion of the total rupture time at the overheat conditions. The tabulation indicated that overheating from 1500° to 1700°F would "use-up" about 32 percent of the rupture life, from 1700° to 1900°F about 27 percent, and from 1800° to 2000°F about 80 percent.

The first two are large enough so that the rupture life at the normal test conditions could be expected to be reduced sufficiently to be noticeable, in spite of the scatter in rupture times. When overheated from 1800° to 2000°F, the specimens should rupture during the overheats. The 48 hours before overheating "used-up" a nominal 25 percent (actually between 23 and 38 percent based on range of rupture times) of the rupture life. Because the overheat time represents about 80 percent of the rupture life at the overheat temperature, the nominal 75 percent of the rupture life remaining at the time of overheating should not be enough to last through the half-hour overheat.

Influence of Overheating

The results of the overheat tests (Tables 3 and 4) are evaluated in terms of the influence on rupture time by Figures 2 and 3. The rupture times are superimposed on the bands of the stress-rupture data from Figure 1. Only a limited number of tests were overheated 100°F because initial testing indicated that the more severe 200°F overheats were not reducing rupture strength. Only sufficient tests were conducted to verify that the 100°F overheats would not unexpectly have a larger effect. The data show that:

1) When specimens were overheated for one-half hour to temperatures 100°F above the normal base temperature with or without stress present, the rupture life was not reduced (Fig. 2). In all but one test, the rupture times were at or above the high side of the range for the normal tests. The one test on the low side of the range (overheated to 1600°F from 1500°F without stress) almost certainly represents a case where the specimen had low strength for other reasons than the effect of the overheat. The relatively prolonged rupture time for the specimen overheated from 1800° to 1900°F with stress present probably represents a case where the combined effect of overheating and stress actually prolonged rupture life.

2) Elongation and reduction of area values (Table 3) were not significantly changed by overheating to 100°F above the normal test temperatures.

3) When specimens were overheated to 200°F above the normal test temperature, rupture times were not reduced (Fig. 3), except for two of four tests overheated under stress from 1800° to 2000°F. The specimens overheated without stress were all at or above the upper side of the range for normal tests. Overheating from 1800° to 2000°F, in particular, tended to increase rupture time. The specimens overheated with stress present all had shorter rupture times than those for which the stress had been removed during the overheats. Of the two tests which ruptured in short time periods when overheated to 2000°F under stress, one ruptured during the overheat, and the other 7.5 hours after overheating. However, two other tests survived the overheats and had rupture times in or above the range for normal tests.

4) Elongation and reduction of area values were not significantly changed (Table 4) by overheating to 200°F above the normal temperature with exception of the specimen which ruptured during the overheat from 1800° to 2000°F.

Creep data from the overheated tests are interpreted as follows:

(a) When overheated from 1500° to 1600°F (Fig. 4), all three of the samples appeared to have slightly lower creep rates than before overheating. The main effect, however, was an indication that third-stage creep was delayed by the overheats.

(b) When samples were overheated from 1500° to 1700°F (Fig. 5) with the stress removed, there appeared to be some increase in creep resistance, particularly as a delay of increase in creep rate in the latter part of the tests. When stress was present, about 1-percent creep occurred during the overheats. Although this raised the level of the subsequent creep curves, they were similar to those for normal tests. It is evident, however, that the accelerated creep during overheating reduced the amount the specimens could creep after overheating, in comparison to those overheated with the stress removed, and thereby limited their rupture time.

(c) When samples were overheated from 1700° to 1800°F (Fig. 6), the creep rates were definitely reduced after overheating, in comparison to the rates before overheating. The data also suggest that third-stage creep was delayed, particularly when stress was not present.

(d) When samples were overheated from 1700° to 1900°F (Fig. 7), there may have been a slight reduction in creep rate, in comparison to that before the overheat. The creep resistance of the specimen overheated with stress which ruptured in 197 hours may have been decreased. The specimen apparently underwent considerable creep during the overheat and, therefore, entered third-stage creep sooner than the others. There was no indication that it had less creep resistance than the other tests before overheating. The onset of rapid creep appeared to be delayed by overheating in the other tests. The creep that occurred during the overheats with stress present apparently reduced rupture life, in comparison to those overheated without stress.

(e) When samples were overheated from 1800° to 1900°F (Fig. 8), the limited data suggest that the overheating with stress increased creep resistance and delayed third-stage creep. The one test overheated without stress showed little change.

(f) When samples were overheated from 1800° to 2000°F (Fig. 9), it seems evident that the onset of third-stage creep was greatly delayed with some reduction in creep rate before third-stage creep. This was true for the specimens which underwent limited creep during the overheats under stress as well as those overheated without stress. The rapid and extensive creep during the overheats under stress for the two tests which had short rupture times is shown by the creep data.

The influence of overheating on creep resistance is summarized as follows:

1) There was a thermally-induced delay in third-stage creep. This increased with temperature and was somewhat more extensive for 200° than for 100°F overheats. At the same time, there was a slight but considerably less significant indication of reduced creep rate.

2) When the stresses were present during the overheats to 100°F above the normal test temperatures, the amount of creep during the overheat was quite small. Any influence of this accelerated creep was small in comparison to the thermally-induced delay of third-stage creep. Its effect was much less than the scatter in creep resistance between specimens.

3) When the stresses were present during the overheats to 200°F above the normal test temperatures, the amount of creep during the overheats was sufficient to limit rupture life, in comparison to specimens overheated with the stress removed. The thermally-induced delay of third-stage creep, however, was sufficient to offset the influence of accelerated creep so that rupture life was not reduced except for part of the tests overheated from 1800° to 2000°F. Apparently, there was a wide variation in creep resistance at 2000°F between specimens. In two cases, the creep was sufficient to cause immediate rupture. Two other specimens were sufficiently resistant to creep at 2000°F so that there was no reduction in life in comparison to normal tests.

Microstructures

The attempts to relate observed responses to overheats to microstructural characteristics were not very successful. The major microstructural features varied a great deal within any one individual specimen so that it was nearly impossible to draw conclusions. The photomicrographs shown in Figures 10 through 18, therefore, are not strictly comparable. What appear to be differences often merely reflect the difficulty of choosing the same type of structure between specimens. With this reservation, the following observations were made:

1) The macrostructure of the specimens (Fig. 10) showed the expected grain size. There were, however, definitely coarser grains on one end than on the other. This was particularly true near the the surface of the gage section.

2) The as-cast microstructure (Fig. 11) consisted of the following:

   (a) A dendritic pattern outlined by precipitated carbides.

   (b) The matrix exhibited all the characteristics which Decker (Ref. 1) described as about 80-percent γ' in the form of cubes arranged to give a "flagstone" structure in a γ matrix.

   (c) The precipitates in the boundaries exhibited the same characteristics as those identified by Decker as (Cb, Ti)C.

3) When subjected to normal rupture testing, the most striking effect was the change of the matrix to an aligned pattern (Figs. 12, 13, and 14). The extent and coarseness of this pattern increased with testing temperature and, to a lesser extent, with time. A white phase, which Decker assumed to be γ', appeared in the boundaries and was more extensive the higher the testing temperature. The carbides may have increased somewhat in size and in number with increasing temperature. The effects of testing conditions on structure were not evident at 100X and, therefore, only a limited number of pictures were included.

4) No change in microstructure from overheating was observed (Figs. 15, 16, 17, and 18). The structures under the optical microscope were similar to those which were found in the normal rupture test.

5) Fracture apparently passed through the white phase or at the boundary of the white phase with the matrix. It was not possible to be sure whether the boundaries were those of the macrograins or the interdendritic boundaries. The probability is that it followed the macrograin boundaries.

DISCUSSION

The outstanding feature of the test results was the absence of any thermally-induced reduction in creep-rupture properties and the indication that rupture life was actually being prolonged by thermally-induced delay of third-stage creep. Accelerated creep during overheating caused no more reduction in rupture life than was anticipated from normal test data. These findings were based on 20 tests, a sufficient number to preclude chance variation in specimen strength indicating an erroneous result. While the number of tests at any one specific test condition was insufficient for statistically sound proof of lack of damage, the general overall pattern validates the individual test results, in spite of the scatter in properties between specimens.

The data, therefore, indicate that overheats within the range of conditions studied would not adversely influence creep-rupture properties from thermally-induced structural changes. If stress was present during the overheats, the reduction in life would be less than that computed from normal-constant temperature rupture data by addibility-of-life-fractions. In fact, the thermally-induced delay in third-stage creep appeared to be sufficient to offset the creep damage from exposure under stress during overheats for approximately 30 percent of the rupture life at the overheat conditions. Apparently, the rupture life of Inconel 713-C can be expected to be no shorter than in normal tests unless the time of exposure under stress during overheating exceeds 30 percent of the normal rupture time at the overheat conditions. The data, in fact, suggest that the presence of stress during an overheat actually enhanced the thermally-induced strengthening effect.

The observations concerning the influence of stress during overheats were based on the estimates from normal test data given on Page 8 for the amounts of creep life expected to be used up during the overheats with stress. As predicted, the accelerated creep had no significant effect for 100°F overheats because the less than 3 percent of the life involved was too small to be significant. When the overheats were 200°F above the normal test temperatures of 1500° and 1700°F, the influence of accelerated creep during the overheats was noticeable, as predicted by the calculated approximately 30 percent of rupture life expended during the overheats. When the overheats were from 1800° to 2000°F, the calculations predicted that the specimens ought to rupture during the overheats because the 0.5 hour overheat represented about 80 percent of the rupture life and was being applied to specimens which had already used up about 25 percent of their available rupture life. In two tests, this was borne out. In two other tests, due to scatter in properties, the specimens apparently had sufficient strength at 2000°F to reduce the creep damage to a sufficiently small amount that it was offset by the thermally-induced strengthening.

The limited structural investigation carried out did not disclose a reason for the lack of damage to creep-rupture properties from overheating. Enough work was done, however, to indicate that the answer would require a more extensive study than was originally contemplated.

COMPARATIVE RESPONSE OF SUPERALLOYS TO OVERHEATING

Several alloys have been investigated at the University of Michigan for the effects of overheating on creep-rupture properties. The results for Inconel 713-C are evaluated in terms of the prior studies.

General Responses to Overheating

Most nickel-base alloys strengthened by the precipitation of γ' have exhibited either an increase in creep-rupture strength from thermally-induced structural changes during overheating, or at least no reduction in strength (Refs. 2, 3 and 4). The one exception was wrought Inconel 700 alloy, which was reduced in rupture strength as a result of the adverse effects on ductility by overheating to temperatures of 2000°F or higher. The creep resistance has been increased or at least not reduced.

When stress was present during overheats, the accelerated creep reduced rupture life. This reduction, however, was no more than would be expected from addibility-of-life-fractions, and even less if the thermally-induced structural changes increased creep strength.

The data from the present investigation show that as-cast Inconel 713-C responded to the conditions of overheating studied generally as would be expected for nickel-base γ' strengthened alloys. As will be discussed later, there can be exceptions to the general response of these type alloys. Therefore, some care should be exercised in applying the results to specific cases other than those studied in this investigation.

The general response of nickel-base γ' strengthened alloys to overheating does not necessarily extend to other types of alloys. A wrought and a cast cobalt-base alloy both underwent extensive reduction in creep resistance (Refs. 5 and 6) and, therefore, rupture life from repeated overheats of two minutes duration.

Limitations to General Response

Exceptions to the general pattern of response and uncertainties due to incomplete data exist for the nickel-base γ' strengthened alloys.

In the results for Inconel 713-C, it was noted that there were increased rupture times from overheats under stress which suggested that the presence of stress in itself during some of the overheats increased creep resistance. A similar effect was observed for M252 alloy (Ref. 2) for which the rupture times were considerably longer than computed from addibility-of-life-fractions when overheated from 1500° to 1650° or 1800°F. In both cases, the overheat temperatures were below the temperature for complete solution of γ'. For M252 alloy, this deviation did not continue when the overheat temperature was increased. Inconel 713-C was not investigated for overheats near to or above the solution temperature for γ'. From the viewpoint of practical applications, the deviation for Inconel 713-C was fortunately on the side of longer-than-expected life.

In the course of a limited experimental investigation of composition effects (Ref. 4), it was observed that creep resistance could be reduced to a marked extent in alloys somewhat low in γ' content. As will be discussed later, this appeared to be due to a slow rate of reprecipitation of the γ' dissolved during overheating. This left the alloy weak after overheating until there was time for the γ' to reprecipitate. Thus, the general rule of no damage to creep resistance from overheating for γ' strengthened alloys probably does not hold for low Al+Ti alloys. In fact, it would not hold for any alloy which, for any reason, did not have time to reprecipitate dissolved γ' after overheating before stress was reapplied.

There is one other general case where it is expected that overheating could reduce creep resistance. The creep resistance of alloys with relatively low amounts of γ' is quite sensitive to the state of the γ'. Thus, exposure to overheating at temperatures below the solution temperature of γ' under conditions which accelerated γ' agglomeration would be expected to reduce creep resistance.

There is an uncertainty arising from differences in conditions of overheating. Inconel 713-C was only investigated for overheats of one-half hour duration. The degree to which the delay of rapid third-stage creep was dependent on the time of exposure is not known. Only one case of prolonged exposure of other alloys is available for direct comparison. Overheating Inconel 700 alloy for time periods of 15 to 60 minutes from 1600° to 1700° and 1750°F (Ref. 7) resulted in no indication of thermally-induced structural changes influencing creep resistance or ductility. When stress was present, the rupture times were in accordance with the addibility-of-life-fractions. This agreed with the results of repeated brief overheats which indicated no change in creep resistance or ductility for these temperatures of overheating. It appears, therefore, that Inconel 713-C differed from Inconel 700 in that there was some increase in creep resistance from the half-hour overheats in the same temperature range. There is no way, however, to determine from the available data to what degree this was dependent on the time of overheating and whether or not it would have been intensified from repeated overheats.

In the case of M252 alloy, there were no comparative data for prolonged exposures. There is, therefore, no certainty that the absence of a significant effect from repeated brief overheats to temperatures below the γ' solution temperature would have held for prolonged overheats in the same temperature range.

As will be discussed in the next section, the influence of overheats can be closely related to solution effects. Therefore, this must be considered in general applications of the results. Temperature, time of overheating, and the number of overheats could all influence the results.

Mechanism of Overheat Effects

All of the temperatures of overheating studied for Inconel 713-C were well below the temperature for complete solution of γ'. Decker (Ref. 1) estimated this temperature to be 2100° to 2150°F. This was due to the high Al content of Inconel 713-C with its correspondingly large volume percent of γ' (estimated to be 80 percent by Decker).

In M252 and Inconel 700 alloys, overheats to 2000°F or higher for two minutes resulted in complete solution of γ' (Ref. 4). Because the rate of γ' reprecipitation was rapid enough to occur during cooling from overheating, this resulted in repeated refinement of the γ' particles. In the case of M252 alloy, this resulted in a substantial increase in creep resistance in comparison to normal tests. The repeated refinement of the γ' prevented the decrease in creep resistance associated with the agglomeration of γ' in normal tests. Several overheats, however, were necessary to introduce a significant effect. For Inconel 700, the overheats had little or no effect on creep resistance, even though the γ' was refined the same as it was for M252 alloy. This was shown to be due to the influence of volume percent of γ'. Fundamental considerations of the creep process indicated that creep resistance was insensitive to the size of the γ' particles at the level of volume percent of γ' in Inconel 700 alloy, whereas it would be expected to be sensitive for the smaller volume percent in M252 alloy.

Due to the large volume of γ' in Inconel 713-C, it seemed probable that its creep resistance would also be insensitive to the state of the γ'. It is not certain from this investigation whether or not the thermally-induced increase in creep resistance from overheating was due to an influence on γ'. Unlike the other alloys studied, the γ' was initially in oriented cubes and changed to alternating layers of γ' and γ during testing. The theoretical considerations are not developed to the extent to allow a prediction as to what the effect of changes in γ', if any occurred, would have on creep resistance.

There must have been increasing solution of γ' in Inconel 713-C with increasing temperature of the half-hour overheats. This could have been responsible for the increased creep resistance. It should be recognized that in the case of Inconel 713-C, the major influence of overheating was the delay of rapid third-stage creep. This could be due to an influence of overheating on the mechanism of fracture rather than creep resistance. More detailed studies would have to be carried out to determine the actual mechanism. Such studies would have to take into account that creep in the γ' itself could be important at the high volume level of γ' in Inconel 713-C alloy.

When Inconel 700 was overheated from 1600° to 2000°F or higher, there was a loss in ductility which limited rupture life (Ref. 3). This was shown (Ref. 4) to be related to the solution temperature being below 2000°F for the predominant carbide, $M_{23}C_6$, forming in this alloy. The repeated overheats not only kept carbides from forming in the grain boundaries, but dissolved those which had formed prior to testing. In normal tests at 1600°F, the mechanism of fracture involved the nucleation of microcracks between carbide particles in the grain boundaries and the matrix. The microcracks grew in number and size, eventually linking to cause fracture. The prevention of carbide precipitation removed this source of fracture. However, the resulting clean grain boundaries had little resistance to crack growth, once a crack started. Fracture then occurred rapidly with little or no microcracking at less ductility than in normal tests. When the overheat temperatures were below the solution temperature for $M_{23}C_6$, there was little or no change in the carbides and, therefore, little or no change in ductility.

In M252 alloy, the predominant carbide was M_6C as a result of a 10-percent Mo content. The solution temperature for this carbide is above 2100°F. Overheats to 2000° or 2100°F had little effect on carbides. The mechanism of fracture was not altered. There was some reduction in ductility due to the increased creep resistance. This did not, however, significantly reduce the rupture life of this high-ductility alloy.

In Inconel 713-C, Decker (Ref. 1) reported the predominant carbide to be (Cb, Ti)C. It would be expected that this carbide would not readily dissolve under the overheat conditions. It seems doubtful, therefore, that carbide solution occurred. This could, perhaps, be responsible for the absence of a change in ductility. The limited study carried out did not indicate that microcracking was prevalent in the alloy. The mechanism of fracture may, therefore, be different than in M252 and Inconel 700 alloys. Possibly, it occurs by nucleation and growth of surface cracks, and the overheats somehow altered the process. Again, more study would be necessary to determine the mechanism.

This brief review of the mechanisms involved has been presented to show that the nickel-base γ' strengthened alloys do not all respond to overheating in the same way. Even though they seem to be highly resistant to loss in creep resistance from overheating, care should be exercised in too widespread application of the generality without regard to the details of the overheat conditions and the mechanisms involved. Secondly, the influence of overheating on ductility depends on the structural features controlling fracture. This again requires attention to detail in specific applications. As-cast Inconel 713-C differs from other nickel-base γ' strengthened alloys in its structural features. Care must, therefore, be exercised in applying the principles developed from other alloys to other conditions of overheating than those specifically studied in this investigation.

CONCLUSIONS

The creep resistance of as-cast Inconel 713-C was not reduced by thermal effects for the overheats studied. Rupture time was increased principally as a result of delayed third-stage creep while ductility was not changed. The improvement in creep resistance was in accordance with the response of other nickel-base γ' strengthened alloys which have either not undergone loss or have increased in creep resistance as a result of thermal effects from overheating.

Accelerated creep due to the presence of stress during the overheats can be expected to reduce rupture life. The time of overheating divided by the rupture time under the overheat conditions, as calculated from normal test data, is a practical measure of the reduction in rupture life to be expected. For as-cast Inconel 713-C alloy, the prolongation of rupture time from thermally-induced effects was sufficient to offset overheating under stress for 30 percent of the rupture life at the overheat conditions. Exposure at the overheat conditions of greater than 30 percent of the available rupture life would be required to reduce rupture life in comparison to normal tests.

These conclusions are based on tests overheated to temperatures 100° and 200°F above normal test temperatures of 1500°, 1700°, and 1800°F. The overheats were 0.5 hour in duration and were applied 48 hours after the start of nominal 200 hour tests. The conclusions are discussed in the report from the viewpoint of comparison with the response of other alloys, the metallurgical mechanisms involved, and the limitations of the test conditions.

REFERENCES

1. Bieber, C. G. and Decker, R. F.: "Effect of Heat Treatment on Inconel "713C" Age-Hardenable Nickel-Chromium Cast Alloy." Tech. Report, Development and Research Division, The International Nickel Company, Inc., July 21, 1959.

2. Rowe, John P., Freeman, J. W.: "Effect of Overheating on Creep-Rupture Properties of M252 Alloy." NACA TN 4224, March 1958.

3. Rowe, John P., Freeman, J. W.: "Effect of Overheating on Creep-Rupture Properties of M252 and Inconel 700 Alloys at 1500° and 1600°F." Final Report to National Aeronautics and Space Administration, Washington, D. C., from University of Michigan Research Institute, March 25, 1960.

4. Rowe, J. P. and Freeman, J. W.: "Relations Between Microstructure and Creep-Rupture Properties of Nickel-Base Alloys As Revealed by Overtemperature Exposures." Report to be submitted to NASA by The University of Michigan Research Institute.

5. Rowe, John P., Freeman, J. W.: "Effect of Overheating on Creep-Rupture Properties of S-816 Alloy at 1500°F." NACA TN 4081, December 1957.

6. Rowe, John P., Freeman, J. W.: "Effect of Overheating on Creep-Rupture Properties of HS-31 Alloy at 1500°F." NACA TN 4083, December 1957.

7. Rowe, John P. and Freeman, J. W.: "Effect of Overheating to 1700°F and 1750°F on the Creep-Rupture Properties of Inconel 700 Alloy at 1600°F and 22,000 psi." Eng. Res. Inst., University of Michigan Report to The International Nickel Company, Inc., December 15, 1956.

Table 1

NORMAL CONSTANT TEMPERATURE RUPTURE TEST DATA FOR AS-CAST INCONEL 713-C

Temperature (°F)	Stress (psi)	Rupture Time (hours)	Elongation (% in 1 in.)	Reduction of Area (%)
1500	60,000	123.1	4.0	4.4
	60,000	80.2	6.0	8.8
	56,000 (B)	256.7	4.2	6.0
	56,000 (B)	256.0	4.5	4.2
	56,000 (B)	156.1	6.0	8.3
	55,000	304.5	4.0	7.1
	55,000	207.6	3.0	7.5
	50,000	472.3	3.7	7.1
1700	28,000	116.3	6.5	8.7
	28,000	143.4	6.0	10.3
	26,000 (B)	224.4	10.0	16.7
	26,000 (B)	197.3	7.5	21.9
	25,500	190.7	8.0	21.9
	25,500	214.2	11.0	18.1
	23,500	469.6	8.0	16.0
1800	18,000	107.6	15.8	30.7
	17,000 (B)	229.1	10.2	27.5
	17,000 (B)	199.5	7.0	20.7
	17,000 (B)	163.3	7.1	18.4
	16,500	265.6	9.8	17.9
	16,500	226.9	11.0	27.3
	15,500	299.3	9.0	17.9
	15,500	195.2	13.0	22.6

(B) - Stress selected for use in overheat tests.

Table 2

CONSTANT TEMPERATURE RUPTURE TEST DATA AT OVERHEAT CONDITIONS FOR AS-CAST INCONEL 713-C

Temperature (°F)	Stress (psi)	Rupture Time (hours)	Elongation (% in 1 in.)	Reduction of Area (%)
1600	56,000	25.3	6.0	5.5
		19.1	5.5	8.7
		15.5	6.8	7.9
1700	56,000	1.5	9.0	12.0
		1.63	8.0	8.3
1800	26,000	18.4	12.5	19.9
		17.2	15.1	40.5
1900	26,000	2.0	14.9	27.3
		1.7	12.7	31.5
1900	17,000	13.4	13.0	36.5
		21.0	21.2	18.9
2000	17,000	0.75	21.0	44.3
		0.50	19.5	65.5

Table 3

RUPTURE TEST DATA FOR SPECIMENS OVERHEATED 100°F FOR 0.5 HOUR

Base Test Conditions		Overheat Conditions		Rupture		
Temperature (°F)	Stress (psi)	Temperature (°F)	Stress (psi)	Time (hours)	Elongation (% in 1 in.)	Reduction of Area (%)
1500	56,000	1600	Zero	273.3 164 ± 3	3.3 6.0	7.1 9.4
1500	56,000	1600	56,000	249.7	5.9	6.2
1700	26,000	1800	Zero	371.3	9.2	10.6
1700	26,000	1800	26,000	264.6	12.7	20.8
1800	17,000	1900	Zero	208.3	10.0	31.9
1800	17,000	1900	17,000	378.4	8.0	26.4

Table 4

RUPTURE TEST DATA FOR SPECIMENS OVERHEATED 200°F FOR 0.5 HOUR

Base Test Conditions		Overheat Conditions		Rupture		
Temperature (°F)	Stress (psi)	Temperature (°F)	Stress (psi)	Time (hours)	Elongation (% in 1 in.)	Reduction of Area (%)
1500	56,000	1700	Zero	166.9*	2.0	4.4
				325.1	4.5	5.5
				258.5	5.0	6.7
1500	56,000	1700	56,000	210.7	7.0	6.0
				233.3	7.0	10.0
				212.0	6.5	8.7
1700	26,000	1900	Zero	265.2	11.0	19.6
				274.2	10.2	20.5
1700	26,000	1900	26,000	197.0	13.0	15.8
				248.8	9.2	12.7
1800	17,000	2000	Zero	447 ± 5	13.0	27.5
				391.7	10.1	23.4
1800	17,000	2000	17,000	348.8	11.0	20.6
				218.8	18.0	19.7
				56.3	11.5	28.5
				48.8**	22.0	59.0

* Flaw in specimen

** Fractured during overheat