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# FOURTH PROGRESS REPORT

TO

MATERIALS CENTRAL

WRIGHT AIR DEVELOPMENT DIVISION

ON

# EFFECT OF PRIOR CREEP ON MECHANICAL PROPERTIES OF AIRCRAFT STRUCTURAL MATERIALS

by

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Project 02902

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SUMMARY

This report covers progress under Contract AF33(616)-6462 for the period from April 1, 1960 to June 30, 1960 on a study of creep damage to short-time mechanical properties of aircraft structural materials. The materials under study included Rene' 41 (a complex nickel-base alloy) and a binary alloy of 80 Ni-20Cr. A molybdenum-base alloy is to be added to the program. The aim of these studies is to define the creep-exposure conditions leading to significant alterations in short-time mechanical properties and then determine the mechanism responsible for the damage.

During this reporting period, equipment modifications were started for vacuum and atmosphere creep-exposure testing. Experimental work consisted of a study of the effects of surface attack on tensile properties of Rene' 41 and the continuation of metallographic studies of this alloy. The inclusion in tensile tests of the attacked surface layer on samples exposed without stress was found to significantly reduce the ductility. Previous studies were conducted on remachined samples to define the primary effects of time and temperature. Surface condition was found to have almost as great an effect as did thermally-induced changes. The study of the effect of surface was extended to specimens exposed to creep at 1300°F.

Metallurgical studies of Rene' 41 included the attempts to obtain dilatometric aging curves, observations of the nature of the surface attack, and establishment of procedures for obtaining extraction replicas. It is hoped to use the latter procedure in conjunction with electron diffraction analysis in order to establish the location of the various carbide phases in ductile and brittle samples.

# INTRODUCTION

This report, covering the period from April 1, 1960 to June 30, 1960, is the fourth progress report to be issued under Contract AF33(616)-6462. The research is a study of the effect of creep damage to mechanical properties of aircraft structural materials.

The present investigation is an extension of work previously conducted at the University of Michigan under Contract AF33(616)-3368. In the earlier work, studies were made of the effects of relatively limited amounts of prior creep on the mechanical properties of a number of established sheet materials. This research was limited since the creep-exposures were confined to the nominally useful temperature ranges for materials acceptable for service, in part, due to their immunity to creep damage. The short-time properties determined after creep-exposure were the tension, compression, and tension-impact properties either at room temperature or the exposure temperature.

The present investigation differs from the previous one in several respects. First, the new work is carried out on materials presently available for use at the highest of operating temperatures, and secondly, the research is oriented so that the basic mechanism of creep damage can be studied rather than the evaluation of specific exposure conditions. Finally, the tests are conducted primarily on wrought bar stock. This permitted the introduction of conventional notch bar impact tests in addition to the short-time tension and compression tests previously used to evaluate the effect of creep. An additional advantage of cylindrical specimens is that greater reliability can be placed on reduction of area measurements in evaluating ductility. In the first year of the present contract, the emphasis of the research was directed towards the interaction of creep and thermally-induced structural changes to the exclusion, as far as possible, of the effects of surface damage and environment.

Two materials were studied. The first was Rene' 41, a complex nickel-base, heat-resistant alloy. The second was a binary alloy of 80 percent nickel and 20 percent chromium, as an example of a material relatively free from thermally-induced structural changes. A third material scheduled for study was a binary alloy of molybdenum - 0.5 percent titanium, as an example of the newer class of high melting point or refractory alloys. Procurement of this alloy was delayed pending availability of equipment for vacuum creep testing.

The results of the first year's studies of Rene' 41 are being summarized. Among conclusions reached were that the alloy was subject to damage to room temperature tensile and compression properties through the combined effects of creep-strain in inducing both the Bauschinger effect and strain hardening; through microstructural changes primarily thermally-induced but also probably stress-activated; and through surface attack propagated deep into the structure by exposure to stress at relatively low temperatures. The major thermally-induced structural change identified was the agglomeration and volume decrease of dispersed γ' particles in the matrix. $M_{23}C_6$ carbides appearing during creep may be related to reduced ductility. The relative distribution of the carbides was postulated as an important factor in controlling ductility.

The present phase of the contract was authorized to extend these studies along the lines revealed by the first year's investigation and, in addition, conduct new research in the following general areas:

1. Extend the investigation to include the complex stresses and variable stresses and temperatures of actual service.

2. Study more carefully the events taking place during creep and leading up to fracture with a view towards investigating the feasibility of "healing" creep damage.

3. Consider, in greater detail, the role of environmental effects during creep in causing damage to mechanical properties.

## TEST MATERIALS

Stock of both the 80 Ni-20 Cr alloy and Rene' 41 alloy was obtained in the form of 1/2-inch nominal diameter wrought bar stock. Detailed descriptions of the form and chemical analysis of the test materials was given previously and will not be repeated here.

The Rene' 41 was tested primarily in a heat treatment condition specified for parts limited in service by tensile strength. This consisted of the following steps:

1. Solution treatment: 1950°F - 1/2 hour, plus air cool.

2. Aging treatment: 1400°F - 16 hours, plus air cool.

For the purpose of this investigation, this treatment was designated as the "R" condition.

Since this progress report contains no data for the 80 Ni-20 Cr alloy, discussion of its heat treatment will not be repeated.

As discussed in the next section, the design of equipment for vacuum creep-testing has been finalized and construction begun. Procurement of a molybdenum alloy was held in abeyance pending the availability of test equipment and a knowledge of the type of specimens required. It should now be possible to initiate procurement of the molybdenum alloy of most current interest. Procurement of the molybdenum alloy was delayed to eliminate the danger of obtaining an expensive material that might become obsolete before the test equipment became available.

## TEST SPECIMENS, EQUIPMENT, AND PROCEDURES

The creep exposures in air are conducted on specimens having a 0.350-inch gage section diameter. These specimens are machined from the 1/2-inch diameter bar stock and have a reduced gage section of approximately 2 inches. The specimen is gripped by threaded holders fitting 1/2-13 threads machined on the ends of the specimen. A drawing of the test specimen was presented in Reference 1.

For tests of mechanical properties following creep-exposure, the specimen can be machined either to a tensile specimen having a gage diameter of approximately 0.3-inches, to a compressive specimen approximately 0.3-inches in diameter by 1-1/2-inches long, or to a Type-W subsized notched impact specimen.

A description of the test equipment and procedures was presented previously (Reference 1) and will not be repeated in detail. Wherever applicable, ASTM Recommended Practices are adhered to. The creep-exposures are conducted in individual creep testing units. The load is applied by a third-class lever system and extension is measured by a modified Martens optical extensometer system.

During the period covered by this report, work was begun to modify several creep units for vacuum or atmosphere testing. The major modification is the substitution of a vacuum retort for the existing creep-furnace. The existing frame, control system, load application system, outside holders, etc., are to be retained. Heating is by means of a tantalum-wire resistance element inside the vacuum retort. The retort is approximately 2 feet high by 8 inches outside diameter and is made up of standard steel pipe fittings welded together. Molybdenum radiation shields are employed to reduce heat loss and eliminate the need for outgassing a bulky insulating material. All working parts in the hot zone are made of molybdenum. Rene' 41 is used for the other working parts. The load will be applied to the specimen holders by pull rods operating through a specially designed O-ring seal. This eliminates the need for using space-consuming bellows and will also eliminate the necessity of accounting for the bellows force in low load tests. Both direct loading and beam loading will be possible. The provision of 1/2-inch threads in the specimen holders will permit the use of specimen gage section diameters of 0.350-inches on down. A separate mechanical pump and diffusion pump will be used for evacuating each retort. Thermocouple and ionization gages are provided for measurement of the vacuum. Deformation will be measured with the conventional Martens optical extensometer system, observed through a window in a forward facing port. A compound mirror system, i.e., several offset mirrors on one stem, obviates the necessity for re-setting the extensometer system.

All parts of the first furnace have been fabricated. When assembly is complete, the design of the radiation shields will be confirmed before the remaining sets are made.

## RESULTS AND DISCUSSION

The major portion of this reporting period was occupied with analyzing and correlating the data obtained during the first year's study of Rene' 41 and the 80 Ni-20 Cr alloy. As discussed above, equipment modifications were also undertaken to prepare for vacuum and atmosphere tests. Experimental work on Rene' 41 was confined to the initiation of a study of surface damage and a continuation of the metallographic investigations outlined in Reference 1. No new experimental work was started on the 80 Ni-20 Cr alloy.

### Effect of Surface Damage on Tensile Properties of Rene' 41

The first year's surveys study of the effect of creep-exposure on Rene' 41 was designed to investigate the effects of creep and thermally-induced structural changes to the exclusion, as far as possible, of surface effects. This was accomplished by machining approximately 0.025-inch from the specimen gage section diameter prior to tensile testing. Deep surface cracking, extending well below the remachined layer, was believed to be the factor causing low ductility in two specimens crept to fairly large amounts in 10 hours at 1200° and 1300°F. It was theorized that small surface discontinuities were propagated well into the material because the low plasticity of the alloy at these temperatures prevented redistribution by creep of the stress concentrations around the discontinuities.

Check tests were also run to confirm the need for remachining creep-exposure specimens prior to tensile tests. These were confined to 10 hour exposures without stress at 1400°, 1600°, and 1800°F and to 10 hour creep-exposures at 1400°, 1500°, and 1600°F. In subsequent room temperature tensile tests, the "as-is" (unmachined) samples all had lower ultimate tensile strengths and lower elongations and reductions of area than did coresponding remachined samples. The losses of strength and ductility due to lack of remachining were as large, in some cases, as those due to creep and/or thermally-induced structural changes.

These observations pointed out the importance of extending the previous studies to include surface damage effects. During the present reporting period, additional unstressed exposures of Rene' 41 in the condition "R" treatment were conducted for 10 hours at 1200°F and for 100 hours at temperatures between 1000° and 1800°F. Creep-exposure tests were started at 1200° and 1300°F. The exposed specimens were then tensile tested at room temperature in the "as-is" condition. The data for remachined specimens were supplemented by a test on a specimen exposed 100 hours at 1000°F. The test data are summarized in Table 1 and plotted in Figures 1 and 2. Time-elongation curves for the 1300°F creep-exposures are plotted in Figure 3.

Figure 1 presents plots of room temperature tensile properties versus exposure temperature for both 10 hour and 100 hour unstressed exposures. The curves for remachined specimens were taken from Reference 1. Remachining had no significant effect on the tensile yield strengths, however, appreciable effects are evident on both the ultimate tensile strength and ductility values for the 10 hour exposures at 1400° to 1800°F and the 100 hour exposure at 1400° and 1600°F. To extend the comparison, a remachined specimen will be tested after exposure for 10 hours at 1200°F. The detrimental effects of the "as-is" surface appear to be somewhat less for the 100 hour exposures than for the 10 hour exposures. This indicates that the effects of overaging in 100 hours were of relatively greater importance in reducing the strength and ductility. This is particularly evident from a comparison of the 1800°F data. In other words, surface attack had a smaller additional effect on a sample embrittled by a metallurgical reaction. Where the inherent ductility was unaffected by exposure (as in the 10 hour exposure), surface attack caused the ductility to be cut almost in half.

The effect of creep-exposure in accentuating the effects of surface attack is indicated in Figure 2. As mentioned previously, a specimen exposed to 4.60 percent creep in 10 hours at 1300°F and then remachined, exhibited very low tensile ductility. Oxidized areas were observed on the fracture surface indicating that deep cracks had extended well below the normally remachined zone. A similar effect was noted in a specimen exposed at 1200°F. A study was undertaken of this effect by exposing, for subsequent "as-is" tensile testing, additional specimens at 1200° and 1300°F. The results of two tests conducted at 1300°F are plotted in Figure 2 together with the data previously obtained on remachined specimens. These data indicate that surface condition had no effect on the increase in tensile yield strength caused by increased creep (the Bauschinger effect) but that the ultimate tensile strength and ductility may have been affected by creep-accentuated surface attack. The difference in both properties observed between "as-is" and remachined specimens with increased creep strain appears to be greater than that indicated from Figure 1 as solely due to surface condition in the unstressed exposures. The curve for "as-is" specimens appears to extend, as a limiting case, to the point for the remachined specimen with the oxidized fracture surface. Additional tests on both "as-is" and remachined samples will be required to confirm these indications. Similar tests are also under way at 1200°F.

Creep curves for the two "as-is" samples and the "oxidized" sample are presented in Figure 3. These curves show that greatly increased creep accompanied only a small increase in stress. The acceleration in creep rate at a point some time after the mid-point of the test suggests that propagation of surface discontinuities well into the specimen may have taken place. This would reduce the cross-sectional area, raise the effective creep stress, and thus increase the creep rate. Such an explanation would be consistent with the presence of deep cracks in specimen R-63. A few deep cracks were also observed in specimen R-95, however, oxidation was not observed on the fracture surface. Metallurgical instability, as a source of accelerating creep, is also possible, however, evidence from the unstressed exposures, stressed exposures, and metallographic investigations summarized in Reference 1 suggest that overaging is not a serious factor in this short a period at 1300°F.

The preceding observations suggest that the role of creep in the propagation of surface discontinuities at relatively low temperatures may be significant. Additional tests of "as-is" and remachined specimens will be required after creep-exposure at 1200° and 1300°F in order to investigate this premise.

## Metallurgical Studies of Rene' 41

Metallurgical studies of Rene' 41 conducted during this reporting period included attempts to obtain dilatometric aging curves, observations of surface attack, and attempts to use electron diffraction in conjunction with extraction replicas for carbide identification.

The unstressed exposure tests reported in Reference 1 and the present progress report were conducted in individual creep furnaces using normal threaded-end creep specimens. As an expedient, a standard Martens optical extensometer system was employed to measure dimensional changes with exposure time. Shrinkage of a few hundredths of a percent was observed in almost all specimens over the entire exposure period, with the higher the temperature the greater the shrinkage.

This procedure differed from a regular dilatometer test in that a uniformly cylindrical specimen was not used and also, the extensometer assembly subjected the specimen to a light load. In addition, extension measurements were not started until the end of the 4-hour pre-heat period. Although the results obtained showed fairly good internal consistency, it was deemed desirable to check them by normal dilatometric procedures. Standard cylindrical specimens, 4.0-inches long by 0.4-inches in diameter were prepared. A quartz-tube dilatometer using an Ames dial was available for extension measurement. Due to equipment difficulties, satisfactory data has not yet been obtained, although qualitative confirmation of shrinkage has been obtained.

The importance of the effect of surface removal on mechanical properties was discussed previously (page 5). Metallographic examination was subsequently made of these samples in order to determine the nature of the attack. Optical micrographs of the "as-is" samples exposed 100 hours at 1400°, 1600°, and 1800°F are presented in Figures 4, 5 and 6. The edge attack on specimen D-2, exposed 474 hours at 1700°F, is shown in Figure 7. These pictures show that both surface cracking and alloy depletion occurred as the exposure temperature was increased. Of particular interest were the clear, apparently completely depleted, layers adjacent to the overaged gamma prime phases in Figures 6 and 7. Specimen D-2 appears to have four distinct layers before the gamma prime is reached. Layer one is a light grey material, probably oxide, immediately at the surface. Layer two contains surface cracks and indeterminant particles. Layer three contains a needle-like phase, but no cracks, while layer four is a clear depleted zone. Attempts have been made to obtain electron micrographs of the surface layers in these samples, however, it has been difficult to obtain satisfactory replicas. Either the replicas have torn in being stripped off the cracked areas, or if a replica could be stripped, the electron beam in the microscope caused the edge to curl up. Modifications to the normal stripping procedures are being investigated and it is hoped that these difficulties can be overcome. In addition, the surface layers will be machined off one-by-one and subjected to X-ray diffraction analysis.

Metallographic examination has been made of the "as-is" specimen R-95, creep-exposed 10 hours at 1300°F (see Table 1 and Figure 3). Figure 8 shows an isolated deep crack observed in this sample, approximately 0.3-inches from the fracture. It appears to extend from the surface as a narrow intergranular crack and then widens considerably about 0.002-inches below the surface. As discussed on page 7, the ductility of this sample appears to be lower than normally expected for this amount of creep on a remachined specimen.

A third area of metallographic investigation conducted during this reporting period was concerned with electron diffraction analysis on extraction replicas. This again was a problem of developing techniques and tangible results were not obtained. The reason for undertaking these studies was the discovery (reported in Reference 1) that the mere presence of $M_{23}C_6$ carbides did not account for embrittlement of Rene' 41. $M_{23}C_6$ was identified in ductile tensile-tested samples by X-ray diffraction analysis of the extracted minor phases. This led to questions of the location and forms of the different types of carbide as being probable factors affecting ductility. Electron diffraction of extraction replicas has the advantage of being able to study the various phases in situ. It has the disadvantages of being a time-consuming, rather delicate operation, particularly sensitive to preparation techniques, whose results once obtained, must be analyzed carefully. After considerable experimentation, it appears that the extraction by a bromine-methyl alcohol reagent of a carbon replica, offers best hopes for success. Towards the end of the reporting period, a few promising replicas were obtained. An example of one is shown in Figure 9 from a sample exposed without stress for 100 hours at 1800°F. The tensile elongation of this sample was 4.2 percent and the reduction of area was 5.9 percent. The dark particles are extracted carbides, while the grey area is the carbon replica showing the matrix and the agglomerated gamma prime phase. Gamma prime envelopes an isolated carbide particle at the top of the picture. The white areas are places where the replica is torn. Diffraction spots have been obtained from the carbides in this replica, however, analysis of the pattern is not complete. It is hoped that these studies will yield information on the relative locations of $M_6C$ and $M_{23}C_6$ carbides in both ductile and brittle samples.

## FUTURE WORK

Work planned for the next three-month period includes the following:

1. Completion of equipment modifications for vacuum and atmosphere testing.

2. Final selection and ordering of a molybdenum alloy.

3. Continuation of metallographic studies of Rene' 41 -- including dilatometry, surface phases, and extraction replication.

4. Continuation of studies to establish the role of creep in crack propagation in Rene' 41 at low temperatures.

5. Completion of analysis of the results obtained from previous tests on Rene' 41 and 80 Ni-20 Cr alloy and initiation of new studies as warranted.

## REFERENCES


1. Gluck, J. V. and Freeman, J. W., "Effect of Creep-Exposure on Mechanical Properties of Rene' 41" (dated April 1960) - to be issued as WADD Technical Report on Contract AF33(616)-6462.

TABLE I

CREEP-EXPOSURE TEST DATA FOR RENE' 41 ALLOY

| | Exposure Conditions | | | | | | | | | | Room Temperature Tensile Properties After Exposure | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Spec. No. | Temp (°F) | Time* (hrs) | Stress (psi) | % of Rupture Life (est) | Total Load Def. (%) | Plastic Load Def. (%) | Creep Def. (%) | Total Plastic Def. (%) | Total Def. (%) | Amount Remachined from Diam. (in.) | Ult. Tensile Strength(psi) | 0.2% Offset Yield Strength (psi) | Elongation (%) | Reduction of Area(%) | Modulus, E $x10^5$ psi |
| As treated | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | 189,800 | 129,733 | 20.5 | 27.9 | 30.5 |
| R-89 | 1000 | 100.0 | None | Nil | -- | -- | na | na | -- | 0.025 | 187,000 | 130,000 | 24.4 | 32.4 | 31.6 |
| R-90 | 1000 | 100.0 | None | Nil | -- | -- | na | na | -- | Nil | 187,500 | 129,500 | 22.9 | 23.2 | 31.2 |
| R-96 | 1200 | 10.0 | None | Nil | -- | -- | na | na | -- | Nil | 190,500 | 133,200 | 21.9 | 27.4 | 32.4 |
| R-97 | 1200 | 100.0 | None | Nil | -- | -- | na | na | -- | Nil | 194,500 | 133,000 | 21.4 | 25.5 | 31.6 |
| R-95 | 1300 | 10.0 | 108,000 | 83 | 0.52 | 0.07 | 2.64 | 2.27 | 3.16 | Nil | 199,800 | 173,000 | 10.2 | 12.9 | 31.2 |
| R-99 | 1300 | 10.0 | 106,000 | 67 | 0.49 | 0.04 | 0.59 | 0.63 | 1.08 | Nil | 196,200 | 160,000 | 17.6 | 17.2 | 30.4 |
| R-93 | 1400 | 100.0 | None | Nil | -- | -- | na | na | -- | Nil | 181,000 | 128,200 | 14.9 | 14.9 | 31.6 |
| R-92 | 1600 | 100.0 | None | Nil | -- | -- | na | na | -- | Nil | 149,000 | 107,500 | 6.8 | 8.3 | 31.1 |
| R-91 | 1800 | 100.0 | None | Nil | -- | -- | na | na | -- | Nil | 131,000 | 102,500 | 2.8 | 5.0 | 30.4 |

* Plus 4 hour pre-heat

na - Not available

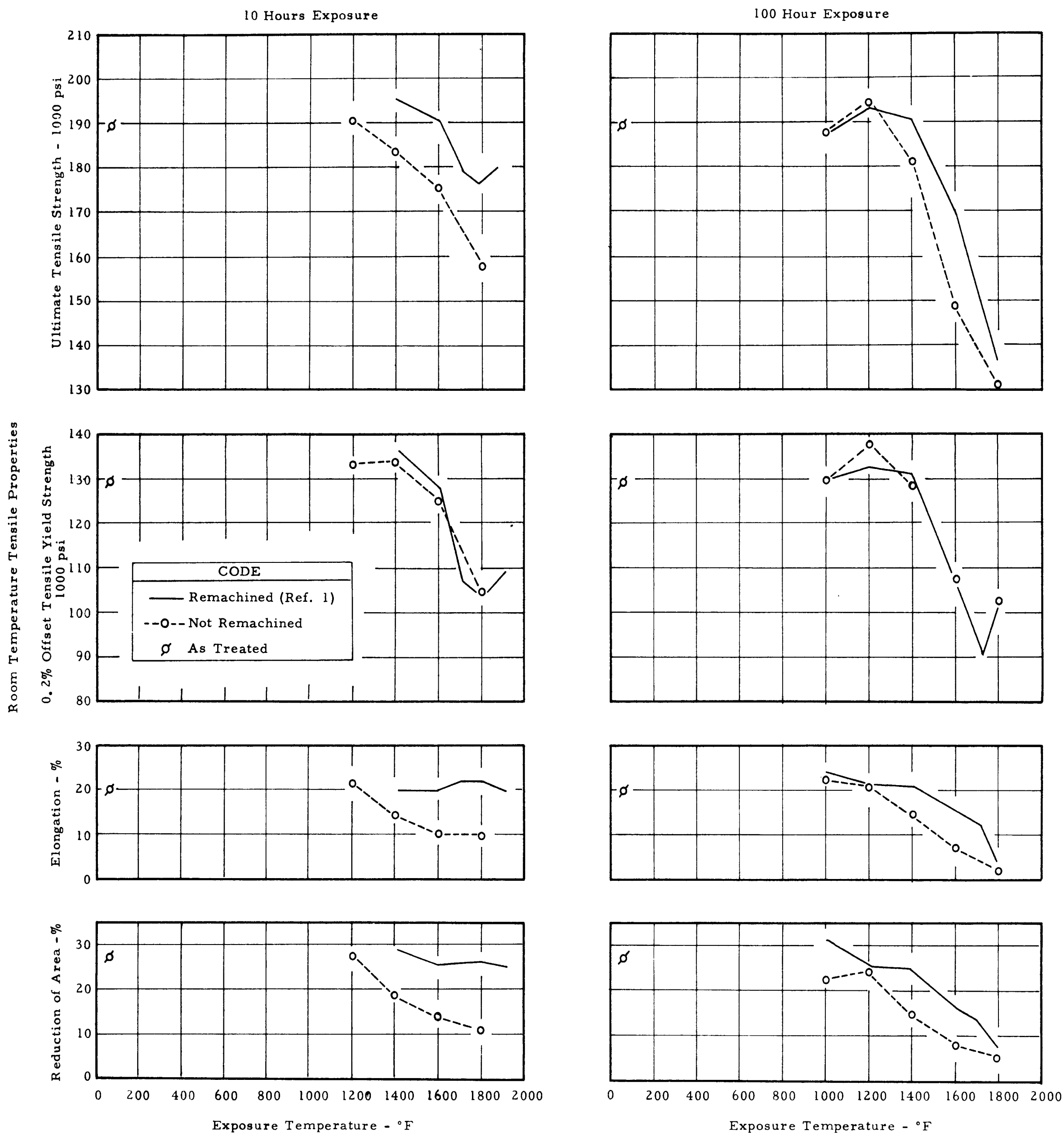


Fig. 1 Effect of Remachining on Curves of Room Temperature Tensile Properties Versus Exposure Temperature of Rene' 41 Exposed Without Stress for 10 or 1000Hours.

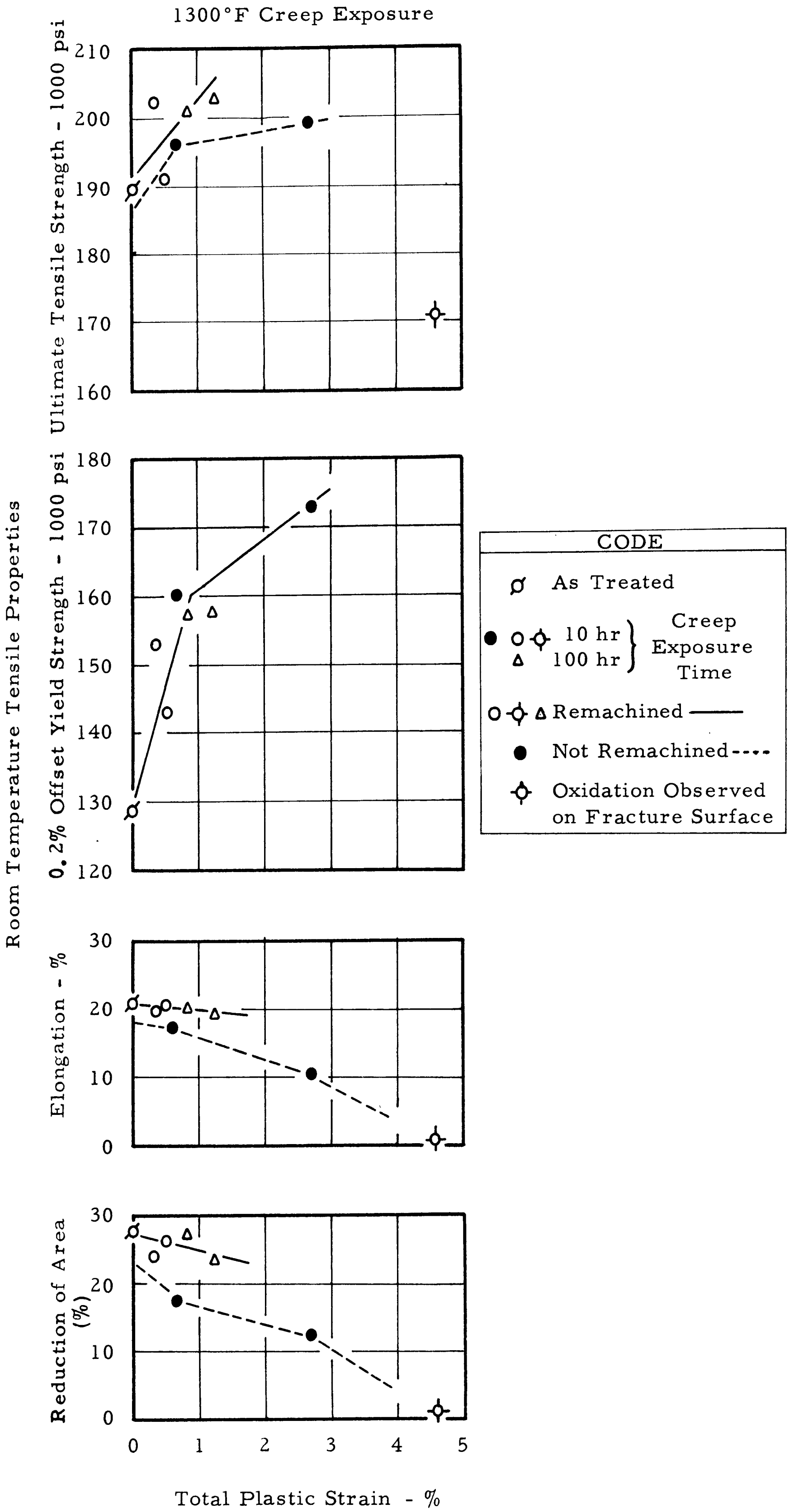


Fig. 2 Effect of Remachining on Curves of Room Temperature Tensile Properties Versus Total Plastic Strain in 10-Hour Creep-Exposures of Rene' 41 at 1300°F.

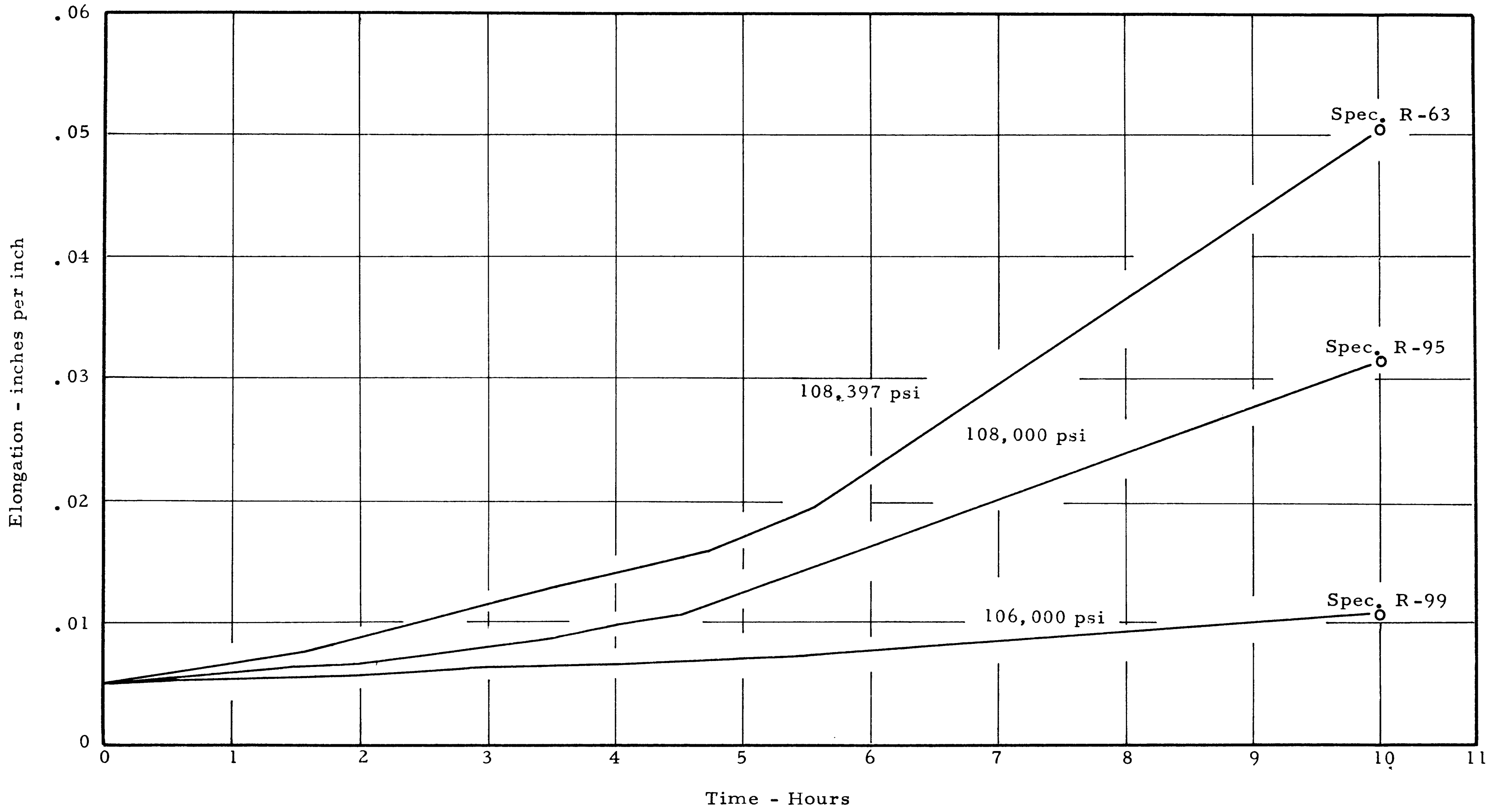


Fig. 3 Time-Elongation Curves for Rene' 41 Subjected to Creep-Exposure at 1300°F.

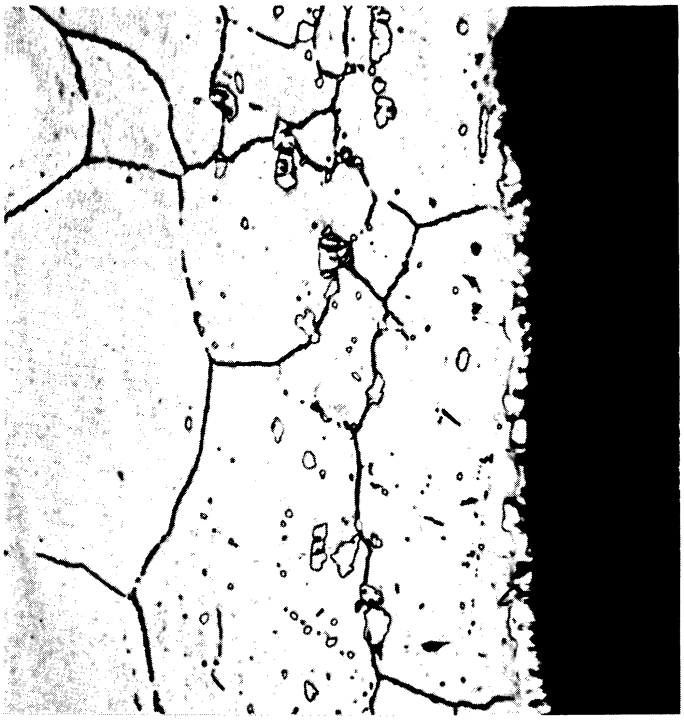

Fig. 4 Spec. No. R-93 (Section Showing Surface Attack)
Exposed Without Stress 100 Hours at 1400°F
Subsequent Tensile Properties
Elongation: 14.9%
Reduction of Area: 14.9%

Corresponding Remachined Spec.
Elongation: 22.0%
Reduction of Area: 24.9%

500X

Fig. 5 Spec. No. R-92 (Section Showing Surface Attack)
Exposed Without Stress 100 Hours at 1600°F
Subsequent Tensile Properties
Elongation: 6.8%
Reduction of Area: 8.3%

Corresponding Remachined Spec.
Elongation: 15.1%
Reduction of Area: 16.6%

500X

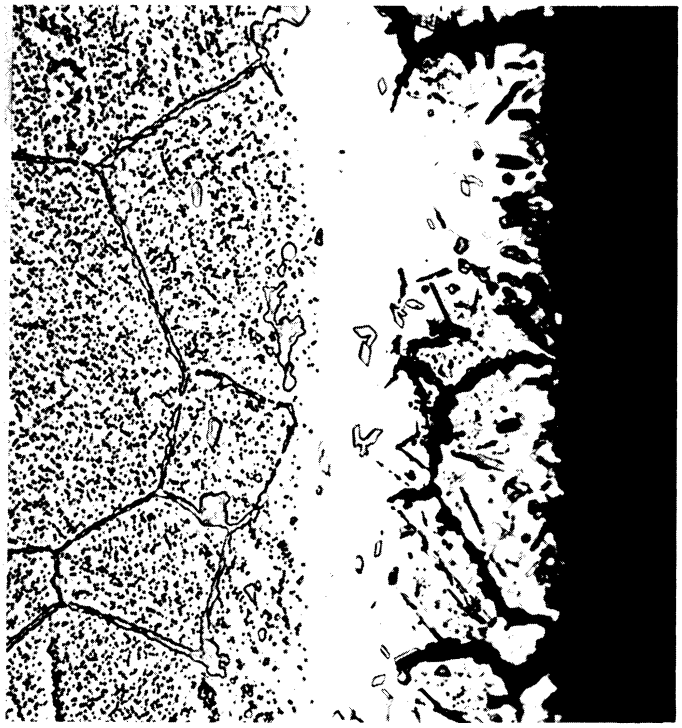

Fig. 6 Spec. No. R-91 (Section Showing Surface Attack)
Exposed Without Stress 100 Hours at 1800°F
Subsequent Tensile Properties
Elongation: 2.8%
Reduction of Area: 5.0%

Corresponding Remachined Spec.
Elongation: 4.2%
Reduction of Area: 5.9%

500X

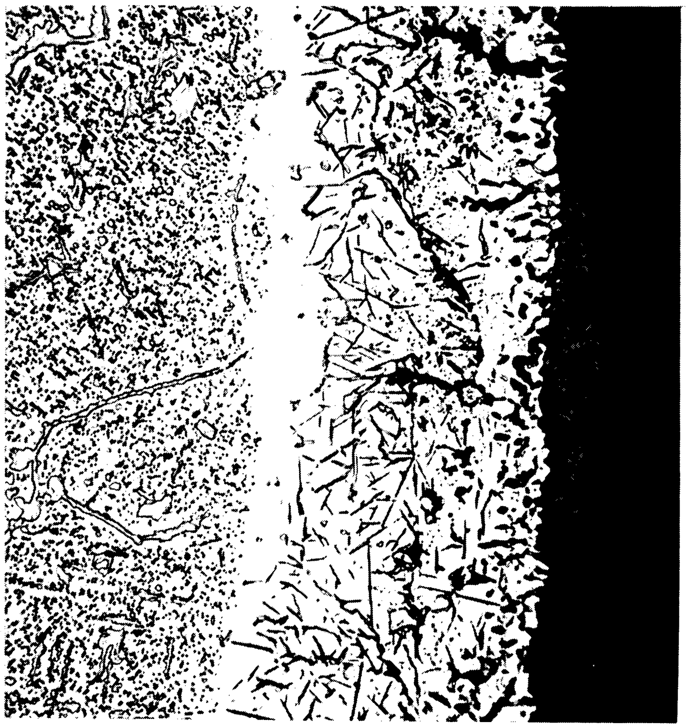

Fig. 7 Spec. No. D-2 (Section Showing Surface Attack)
Exposed in Dilatometer Furnace 474 Hours at 1700°F

(No mechanical properties available)

500X

Fig. 8 Spec. No. R-95 (Section Showing Surface Attack and Isolated Crack) Creep-Exposure at 1300°F for 10 Hours to 2.71 Percent Deformation
Subsequent Tensile Properties
Elongation: 10.2%
Reduction of Area: 12.9%

100X

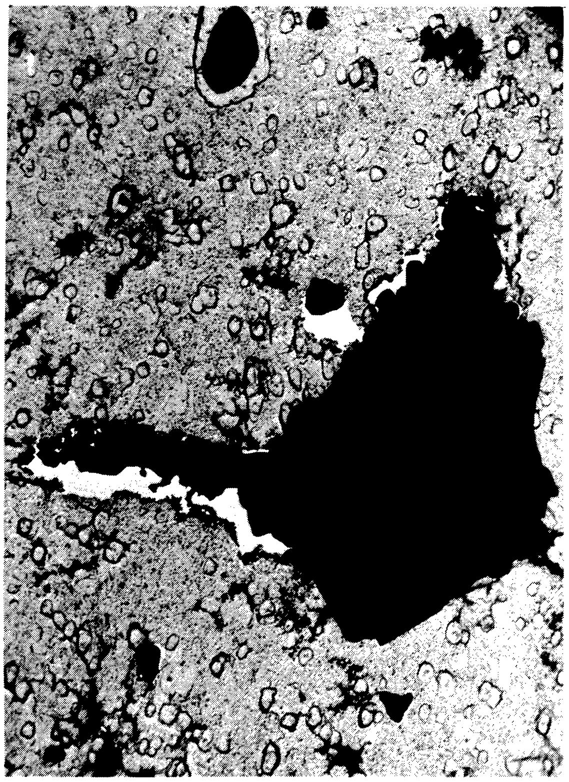

Fig. 9 Spec. No. R-32 (Electron Micrograph of Extraction Replica from Longitudinal Section)
Exposed Without Stress 100 Hours at 1800°F
Subsequent Tensile Properties
Elongation: 4.2%
Reduction of Area: 5.9%

4800X