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CREEP-RUPTURE PROPERTIES OF Cb-752 ALLOYS AND THEIR RESPONSE TO HEAT TREATMENT

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ABSTRACT

Creep-rupture properties for Cb-752 (Nb–10% W–2.5% Zr) to 1000 hr are presented for test temperatures of 982°C (1800°F), 1093°C (2000°F), and 1204°C (2200°F). Substantial improvements in strength at 982°C (1800°F) and 1204°C (2200°F) can be achieved by pretest annealing 1 hr at 1593°C (2900°F). These improvements are shown to be stable for at least 1000 hr. Pretest annealing at higher temperatures yields inferior properties. A tentative explanation for these effects is offered in terms of precipitate distribution.

INTRODUCTION

A large number of refractory-metal alloys are currently under consideration for high-temperature structural applications. In most cases, the only mechanical property data available on these materials are tensile-test data and very short-time creep data. Since long-time ($\geq$ 1000 hr) creep properties must be considered for many high-temperature applications, an evaluation of several promising refractory-metal alloys was undertaken. This evaluation included the determination of creep properties to 1000 hr, their stability, and their response to heat treatment, so that a valid comparison of their suitability for high-temperature structural applications could be made. This report describes such an evaluation of Cb-752 (Nb–10% W–2.5% Zr).

MATERIAL

The heat of material tested was produced by Stellite Division of Union Carbide Corporation (heat No. 52227) from powder which was consolidated by electron-beam melting. The resulting material was consumable arc-melted to an 11-in.-diam ingot.

The ingot was conditioned and extruded to a rectangular billet, then "hot-cold" rolled to a 1/4-in. sheet bar. The sheet bar was cold rolled to 1/8-in. sheet. This sheet was spot conditioned, pickled, and vacuum annealed 1 hr at 1371°C (2500°F). The material was then cold rolled to penultimate gage and solution annealed for 1 hr at 1538°C (2800°F), after which it was cold reduced to final gage (0.030 in.), pickled, and annealed 1 hr at 1316°C (2400°F).

The vendors analysis of this material is listed below:

Element	Wt %
W	9.9
Zr	2.6
C	0.0034[a]
O	0.0072[a]
N	0.0099[a]

[a]Determined on finished sheet.

The material was given a fluorescent-penetrant inspection which showed it to be free of surface flaws greater than 0.0005 in. deep, a transmission-ultrasonic inspection which showed it to be free of laminations greater than 0.125 in. in diameter, and a shear-wave ultrasonic inspection which showed it to be free of transverse discontinuities in excess of 3% of the material thickness.

EXPERIMENTAL DETAILS

The apparatus used in this work is described in a previous report.[1] The tests were performed at pressures lower than 2×10^{-7} torr. Every test specimen was analyzed for interstitials. The after-test oxygen content of most specimens was between 100 and 400 ppm. In this region the creep-rupture properties could not be correlated with the oxygen contents.

The metallographic specimens were prepared by vibratory polishing in the manner described by Long and Gray.[2]

RESULTS AND DISCUSSION

The creep-rupture properties of as-received Cb-752 alloy at 982°C (1800°F) are given in Fig. 1. Times to 1, 2, 5, and 10% elongation are plotted as a function of stress along with the time to rupture. Similarly the creep-rupture properties at 1093°C (2000°F) and 1204°C (2200°F) are given in Figs. 2 and 3 respectively. Figure 4 shows the secondary creep rate as a function of stress for each of these temperatures. Isochronous stress-strain curves for the alloy at all three temperatures are shown in Figs. 5, 6, and 7 respectively. It can be seen from Figs. 2 and 3 that at long times and high temperatures the curves exhibit a pronounced curvature. The ductilities seem to be adequate at all of the temperatures investigated, the lowest being an average of approximately 37% for the 982°C (1800°F) tests.

In order to determine the effect of annealing temperature on the creep-rupture properties, duplicate specimens were annealed at various temperatures. After annealing, one specimen from each pair was loaded to 35,000 psi at 982°C (1800°F) while the other was loaded to 17,500 psi at 1204°C (2200°F). The times to selected percent elongations and to rupture are plotted as a function of pretest annealing temperature for the

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[1] R. L. Stephenson, Comparative Creep-Rupture Properties of D-43 and B-66 Alloys, ORNL-TM-944 (November 1964).

[2] E. L. Long and R. J. Gray, Metals Progr. 74(4), 145–48 (October 1958).

982°C (1800°F) tests in Fig. 8. Figure 9 gives the influence of pretest annealing temperature on the properties at 1204°C (2200°F). It can be seen that the creep-rupture properties can be improved substantially by annealing at moderately high temperatures while annealing at still higher temperatures yields inferior properties. In order to determine the long-time stability of these improvements, a number of specimens were annealed at 1593°C (2900°F), the apparent optimum temperature, and tested at 982°C (1800°F) and 1204°C (2200°F). The results of these experiments are shown in Figs. 10 and 11. (Curves showing the time to 1% creep and to rupture for the as-received material are included for comparison.) Isochronous stress-strain curves for the pretest annealed material at 982°C (1800°F) and 1204°C (2200°F) are shown in Figs. 12 and 13 respectively. Annealed specimens tested at 982°C (1800°F) average approximately 28% ductility.

A possible explanation for the effect of pretest annealing temperature on the mechanical properties and for the severe curvature of the creep-rupture curves (Figs. 2 and 3) at high temperatures is suggested by the microstructures of the creep specimens. Figure 14 shows the microstructures of specimens tested at 982°C (1800°F). Figure 14a shows a representative view of the microstructure at high magnification and a view of the fracture at low magnification of a specimen tested a very short time. Figure 14b shows similar views of a specimen tested for a long time at the same temperature. Analogous views of short- and long-time specimens tested at 1093°C (2000°F) and 1204°C (2200°F) are shown in Figs. 15 and 16 respectively. All specimens give some indication, however inconclusive, of a precipitate. At longer times and higher temperatures a precipitate is distinctly visible in the grain boundaries. The appearance of this precipitate in the grain boundaries is roughly concurrent with the onset of the accelerated creep observed at longer times and higher temperatures. Figure 17 shows photomicrographs of specimens tested at 982°C (1800°F) and 1204°C (2200°F) after a pretest anneal of 1 hr at approximately 1760°C (3200°F). In contrast to the specimens shown in the preceding figures, which showed substantial fracture ductility, these specimens are seen to fail with very little deformation of the matrix material. In the case of the 1204°C (2200°F) test the fracture is clearly intergranular. It is possible that increasing pretest annealing temperatures places progressively more of this precipitate in solution, allowing it to reprecipitate in a finely dispersed state at the test temperature and hence produce the higher strength properties. At still higher temperatures it is possible that increased atom mobilities allow more rapid coalescence of the remaining precipitate in the grain boundaries while grain growth reduces the grain-boundary area and thus decreases the amount of precipitate needed to cause significantly reduced fracture ductility.

Precipitates which are identical in appearance have been observed in D-43 (Nb–10% W–1% Zr–0.1% C) (Ref. 3) and FS-85 (Nb–27% Ta–10% W–1% Zr) also.[4] Attempts have been made to identify this precipitate by electron diffraction.[5] The results are inconclusive but preliminary data indicate that it is ZrO_2.

SUMMARY

The creep-rupture properties have been determined to 1000 hr for a heat of Cb-752 alloy at 982°C (1800°F), 1093°C (2000°F), and 1204°C (2200°F). It has been shown that the pretest annealing temperature can have a pronounced effect on the creep-rupture properties at the test temperatures investigated. Strengths were progressively increased by pretest anneals at increasing temperatures up to approximately 1593°C (2900°F). With higher pretest annealing temperatures, inferior strength and ductility are observed. A tentative explanation of this behavior in terms of the distribution of precipitates in the alloy is offered. The improved properties resulting from a 1-hr pretest anneal at 1593°C (2900°F) are shown to be stable for at least 1000 hr at 982°C (1800°F) and 1204°C (2200°F). These improvements are achieved at the expense of

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[3]R. L. Stephenson, to be published.

[4]R. L. Stephenson, <u>Creep-Rupture Properties of FS-85 Alloy and Their Response to Heat Treatment</u>, ORNL-TM-1456 (July 1966).

[5]T. E. Wilmarth, private communication.

a slight reduction in ductility. In view of the pronounced curvature of some of the creep-rupture curves at long times it is concluded that very short-time data frequently do not provide an adequate evaluation of an alloy.

ACKNOWLEDGMENTS

The author wishes to acknowledge the contribution to this work by others. Those who merit specific mention are J. R. Weir for invaluable suggestions and guidance during the course of the work, W. O. Harms and H. Inouye for reviewing the manuscript, C. W. Houck for the preparation of metallographic specimens, and the Metals and Ceramics Division Reports Office for the preparation of this report.

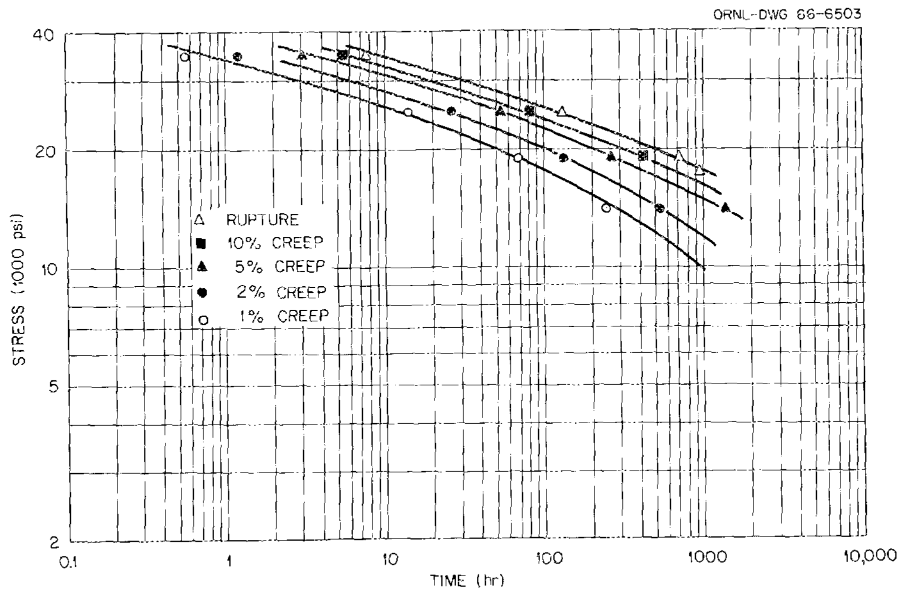


Fig. 1. Creep-Rupture Properties of Cb-752 Alloy at 982°C (1800°F).

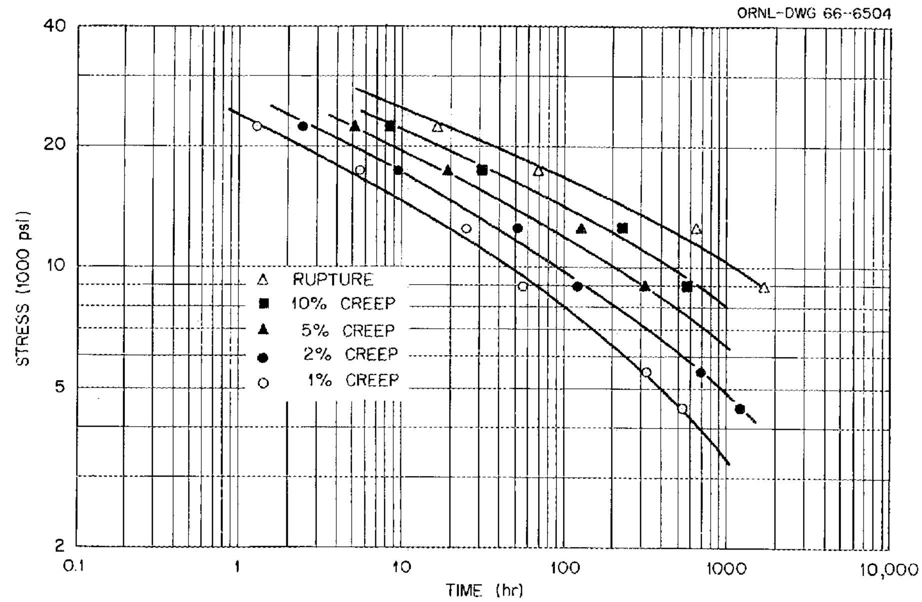


Fig. 2. Creep-Rupture Properties of Cb-752 Alloy at 1093°C (2000°F).

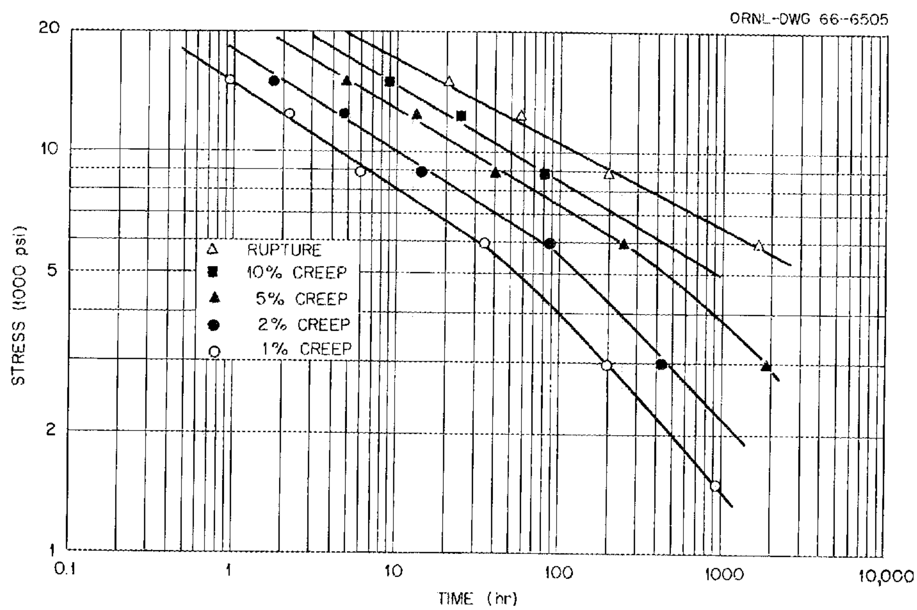


Fig. 3. Creep-Rupture Properties of Cb-752 Alloy at 1204°C (2200°F).

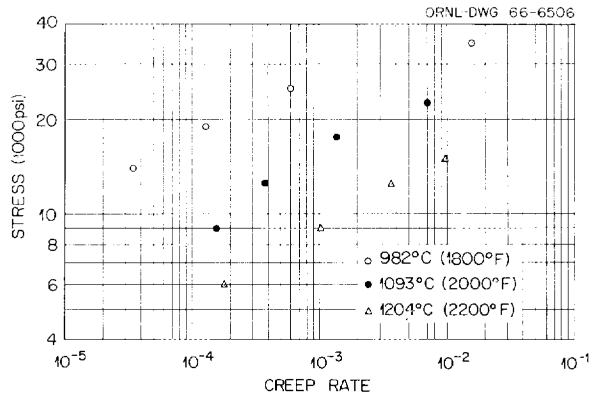


Fig. 4. Secondary Creep Rate vs Stress for Cb-752 Alloy at 982°C (1800°F), 1093°C (2000°F), and 1204°C (2200°F).

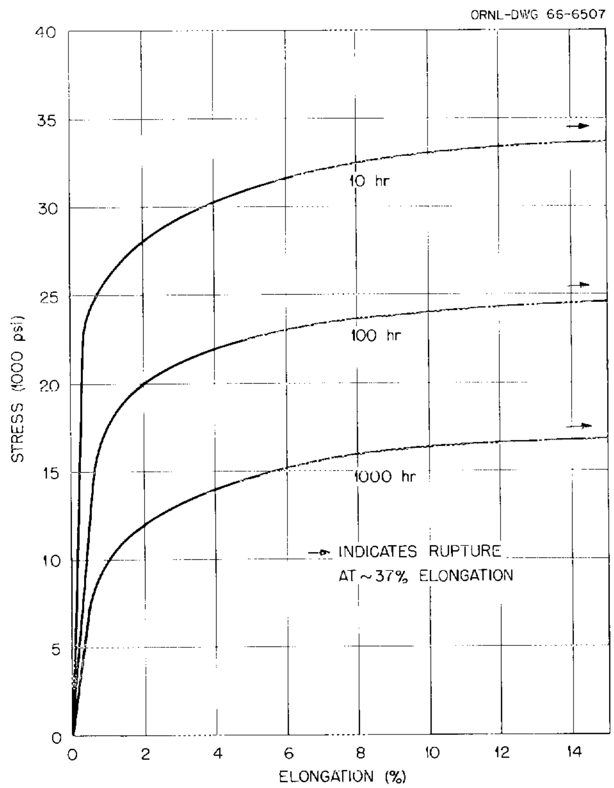


Fig. 5. Isochronous Stress-Strain Curves for Cb-752 Alloy at 982°C (1800°F).

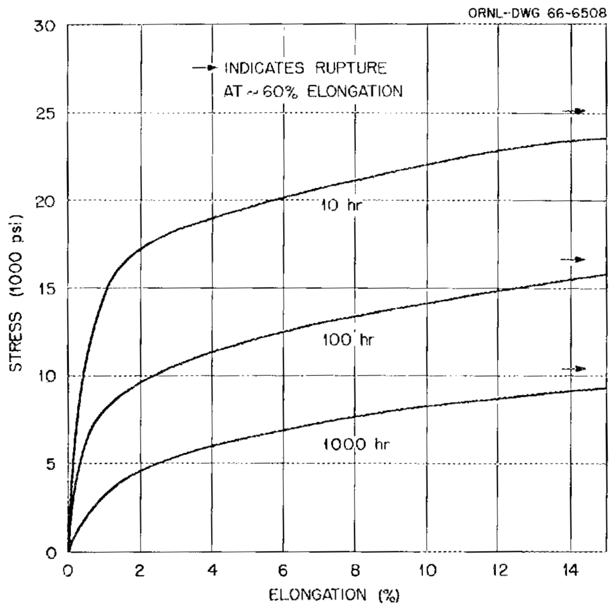


Fig. 6. Isochronous Stress-Strain Curves for Cb-752 Alloy at 1093°C (2000°F).

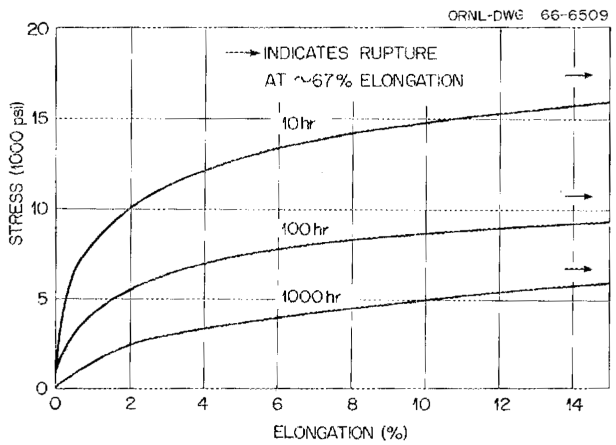


Fig. 7. Isochronous Stress-Strain Curves for Cb-752 Alloy at 1204°C (2200°F).

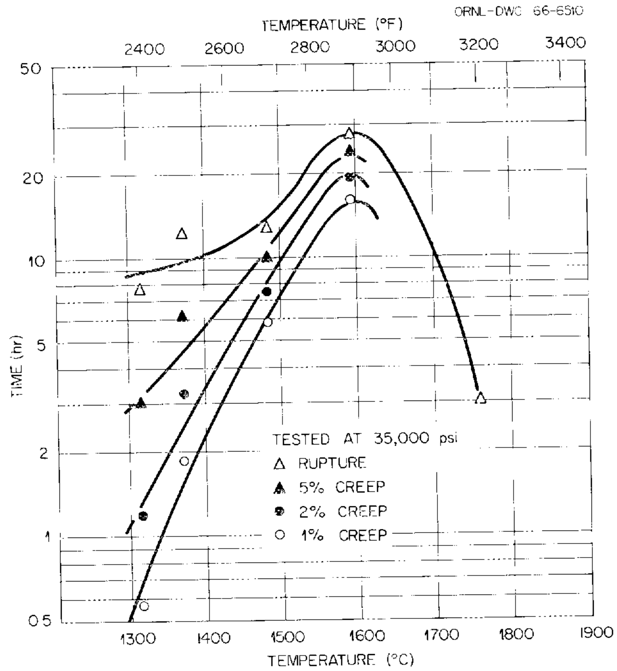


Fig. 8. Effect of Annealing Temperature on Creep-Rupture Properties of Cb-752 Alloy at 982°C (1800°F).

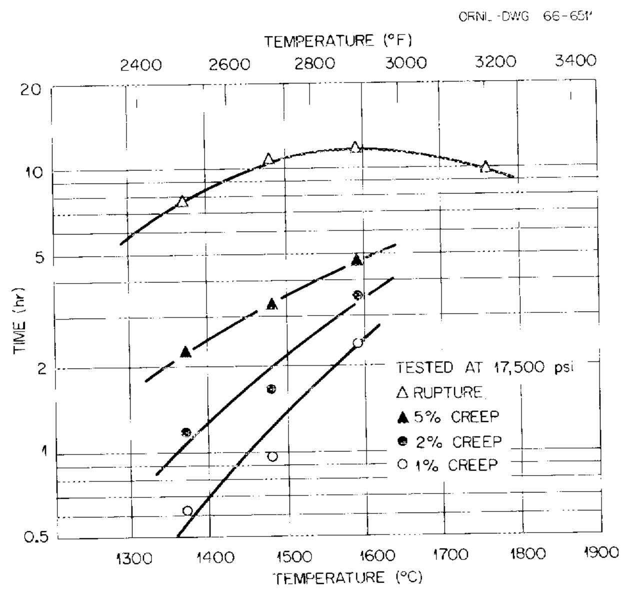


Fig. 9. Effect of Annealing Temperature on Creep-Rupture Properties of Cb-752 Alloy at 1204°C (2200°F).

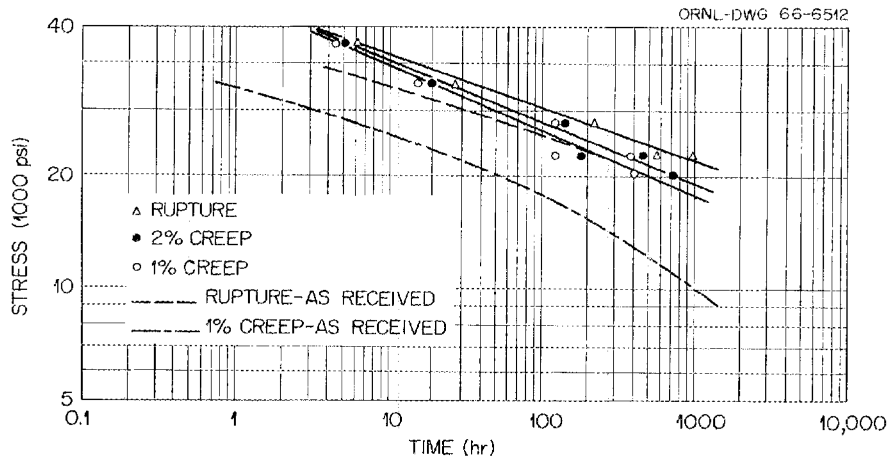


Fig. 10. Comparative Creep-Rupture Properties of Cb-752 Alloy Tested at 982°C (1800°F) After 1-hr Pretest Anneal at 1593°C (2900°F).

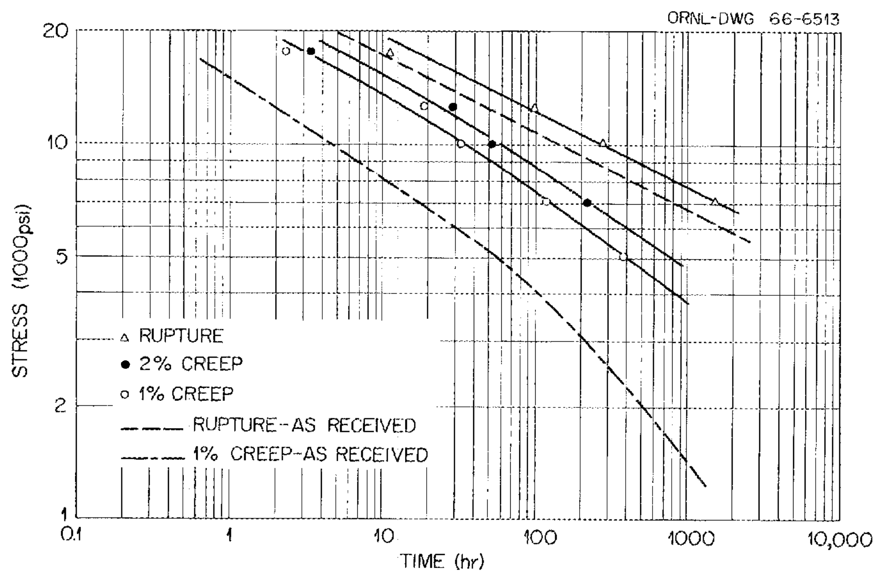


Fig. 11. Comparative Creep-Rupture Properties of Cb-752 Alloy Tested at 1204°C (2200°F) After 1-hr Pretest Anneal at 1593°C (2900°F).

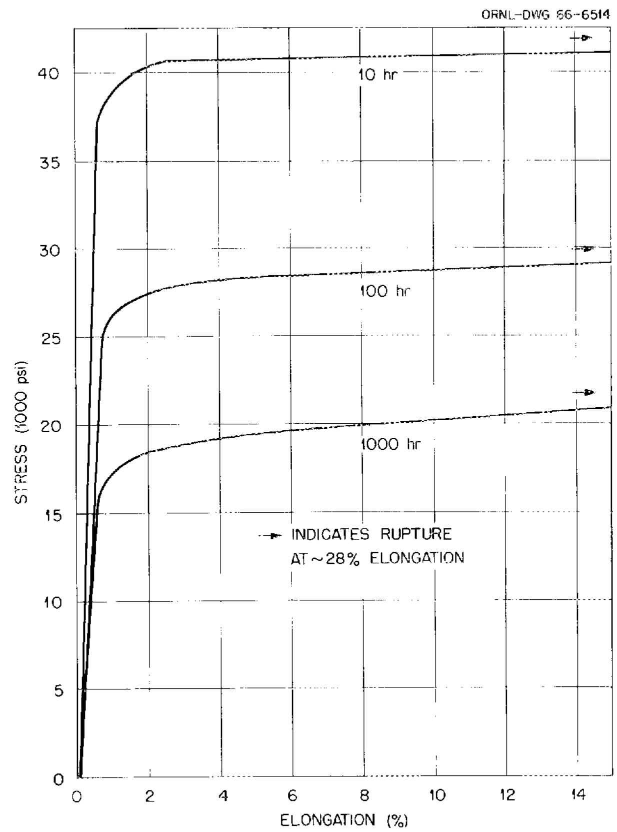


Fig. 12. Isochronous Stress-Strain Curves for Cb-752 Alloy Tested at 982°C (1800°F) After 1-hr Pretest Anneal at 1593°C (2900°F).

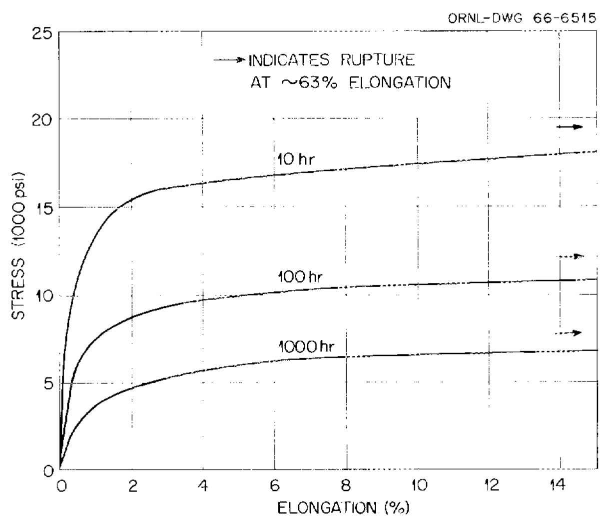
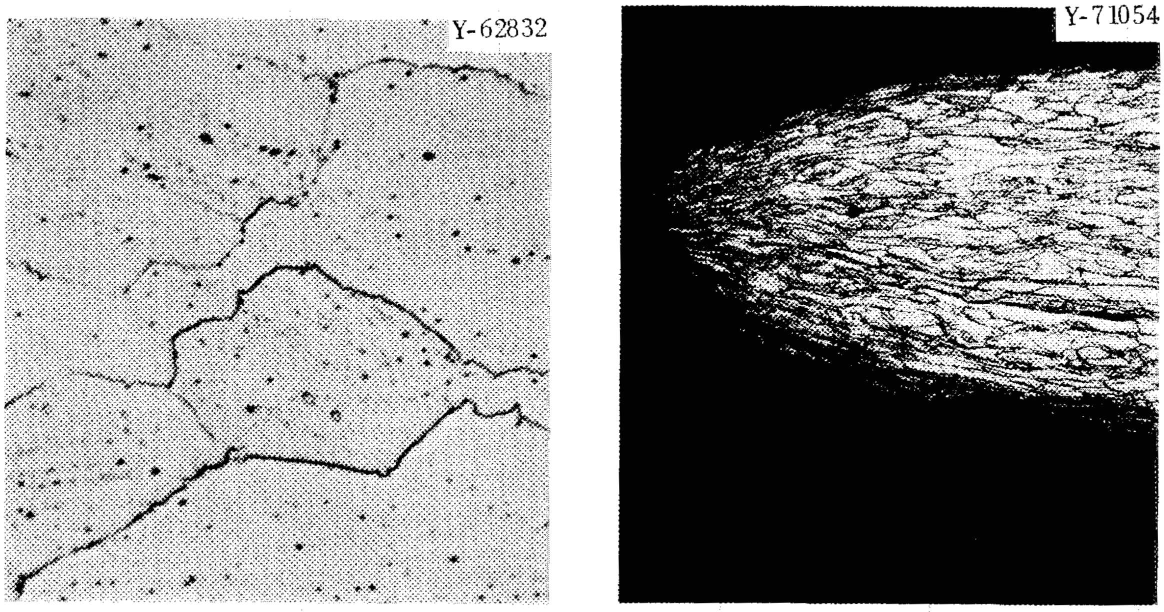
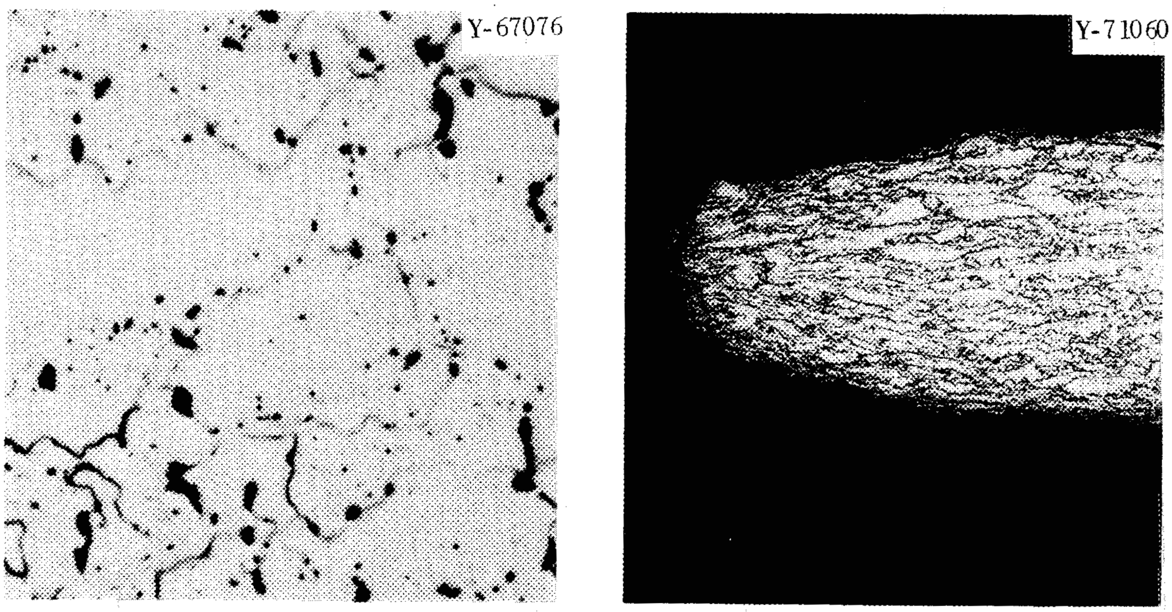


Fig. 13. Isochronous Stress-Strain Curves for Cb-752 Alloy Tested at 1204°C (2200°F) After 1-hr Pretest Anneal at 1593°C (2900°F).

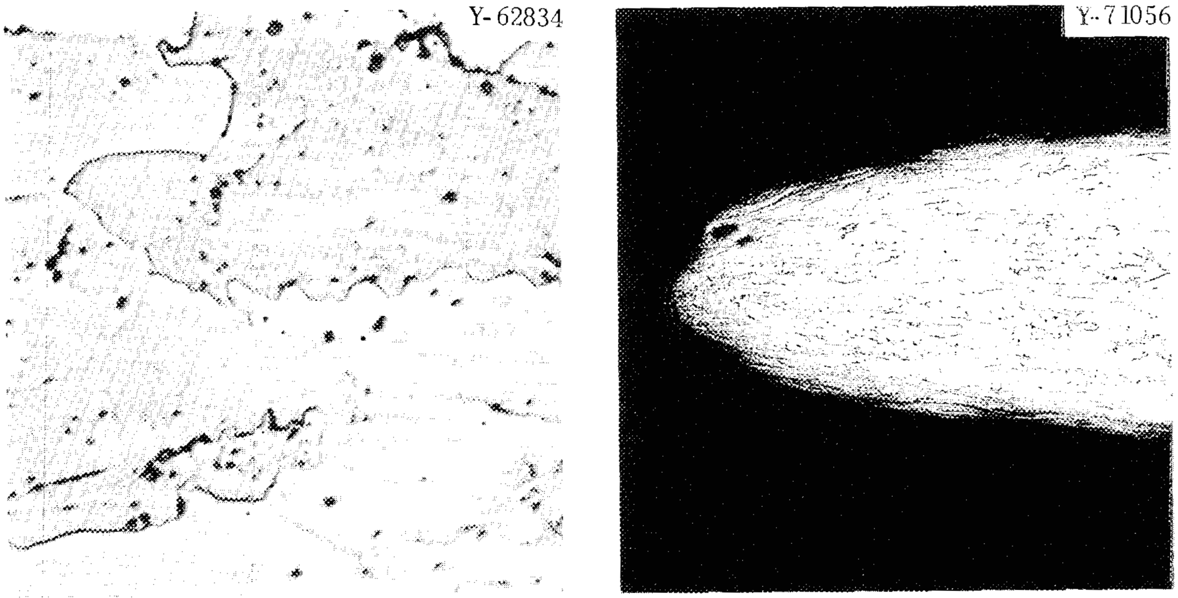


(a) 7.7 hr

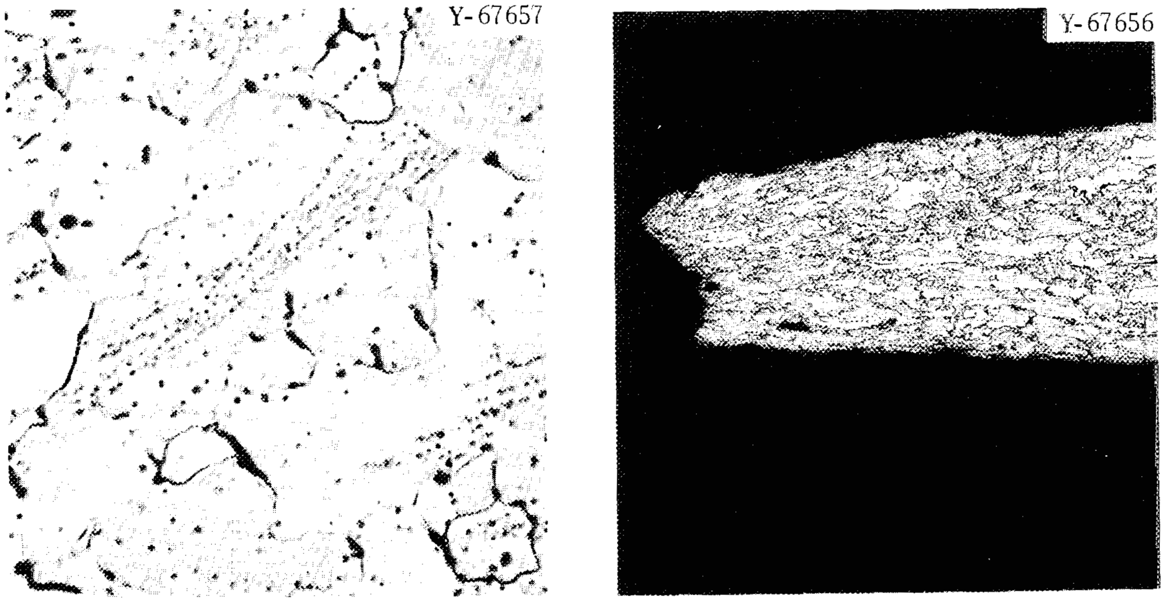


(b) 976.6 hr

Fig. 14. Microstructures of Specimens Tested at 982°C (1800°F).

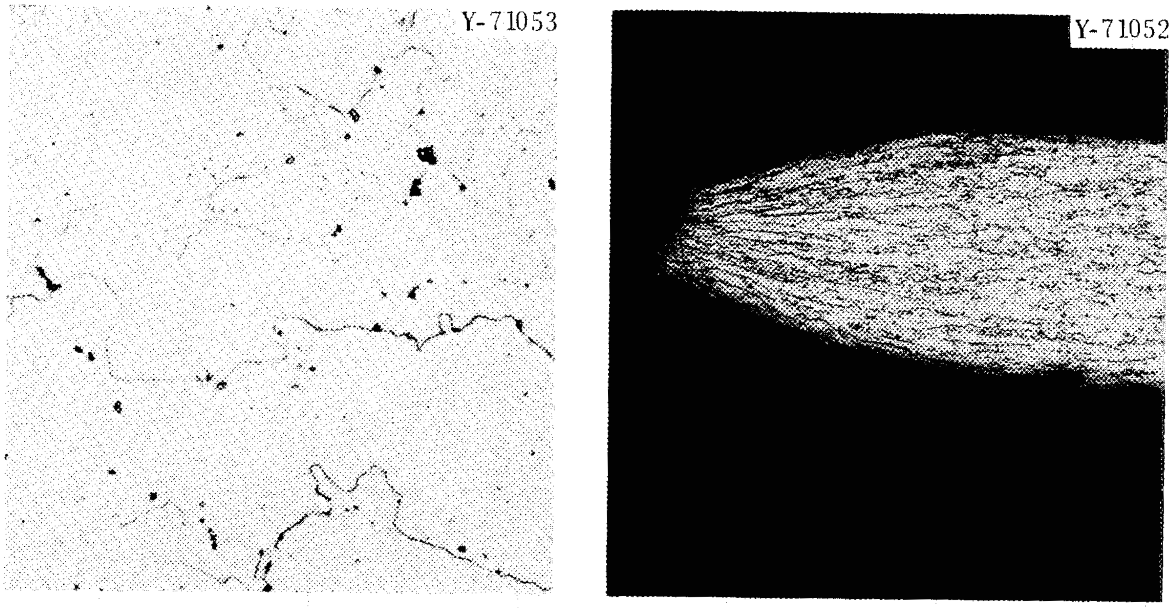


(a) 16.1 hr

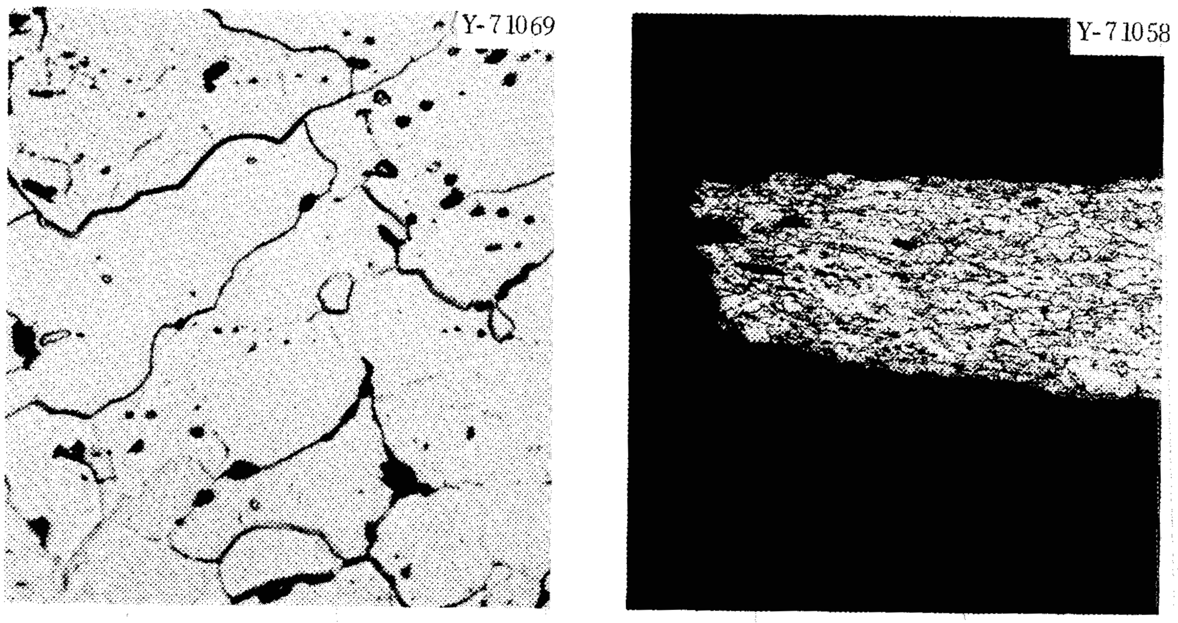


(b) 1750.7 hr

Fig. 15. Microstructure of Specimens Tested at 1093°C (2000°F).

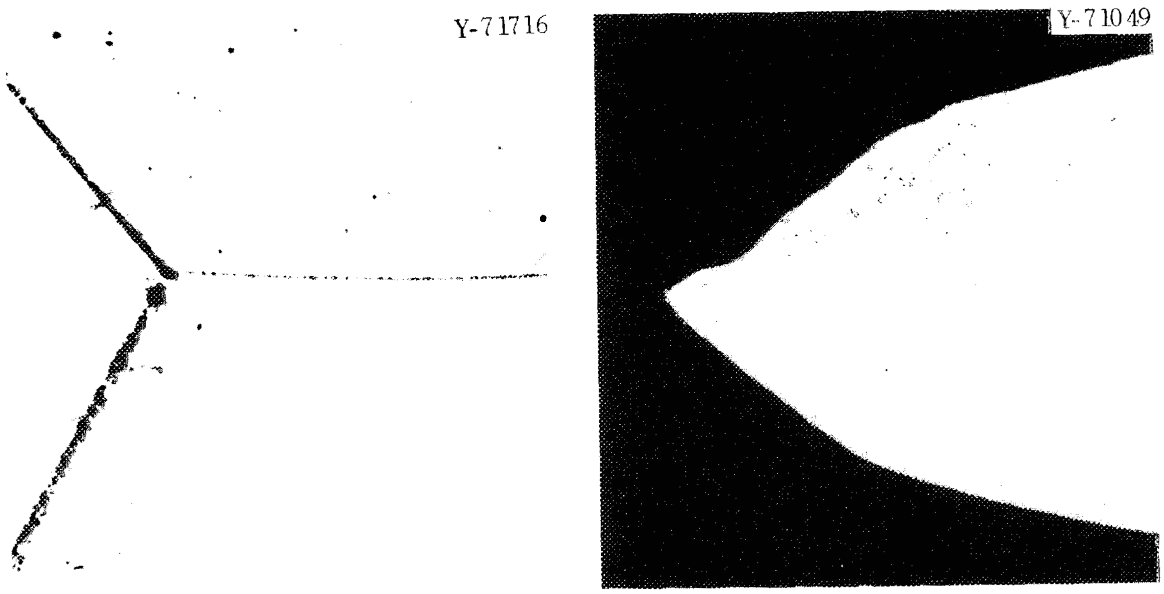


(a) 20.4 hr

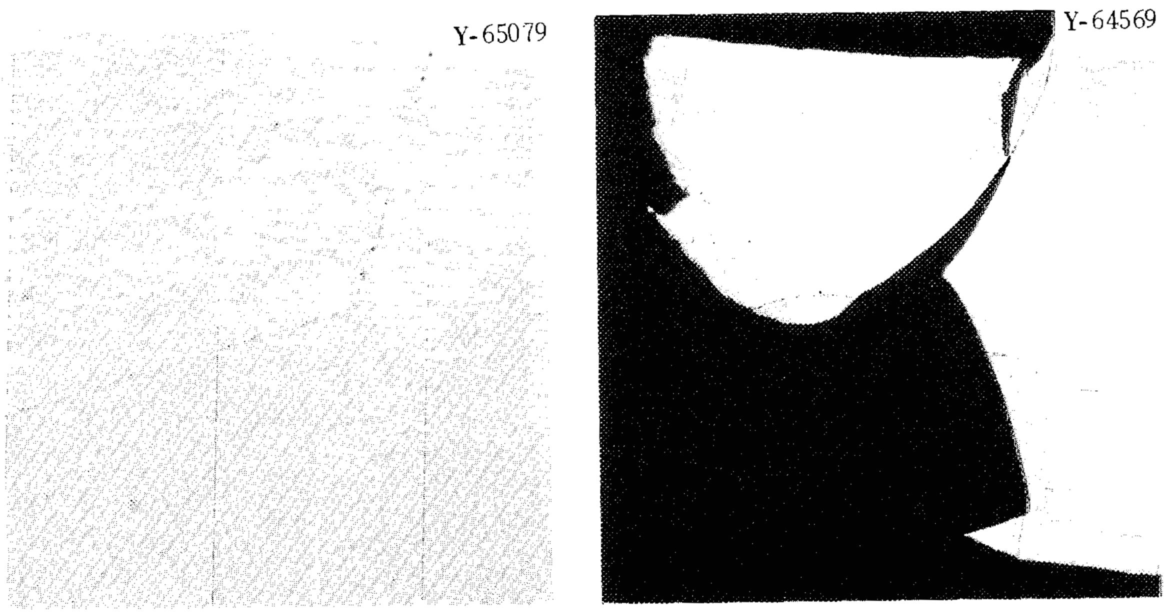


(b) 1656.0 hr

Fig. 16. Microstructures of Specimens Tested at 1204°C (2200°F).



(a) 982°C (1800°F)



(b) 1204°C (2200°F)

Fig. 17. Microstructures of Specimens Tested at Indicated Temperature After 1 hr Pretest Anneal at 1760°C (3200°F).

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