THE EFFECT OF PHASE INSTABILITY ON THE HIGH TEMPERATURE STRESS-RUPTURE PROPERTIES OF REPRESENTATIVE NICKEL-BASE SUPERALLOYS

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Abstract

Long time testing (up to 25,000 hours) of four representative nickel-base alloys, Udimet 700, 520, 500 and Inco 713C has shown that sigma phase precipitates in each alloy in spite of the relatively safe N_V number of two of these alloys, Udimet 520 and Inco 713C. Moreover, stress-rupture tests have shown that the formation of sigma phase does not necessarily adversely effect the properties. Only in Udimet 700 did sigma phase reduce the rupture strength and result in transgranular, intersigmatic failure.

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Introduction

The development in recent years of more efficient and longer life industrial gas turbines has been dependent on the parallel development of higher temperature and more structurally stable nickel-base superalloys. Although superalloys at normal operating temperatures are in a constant state of metallurgical change, e.g., growth of γ' and reversion of MC to $M_{23}C_6$(1), these changes generally produce predictable variations in mechanical properties so that successful design to 100,000 hours of life is possible. Other structural modifications of the alloy, however, are not as well behaved. The formation of intermetallic phases, such as sigma, has been shown to negate otherwise valid extrapolation of mechanical properties(2). To help control such deleterious phase changes, a method of predicting the equilibrium phases in a quasi-ternary phase system was introduced based on the well known correlation of electronic configuration and structure(3). An extensive test of the various models that have been proposed based on this concept(4,5,6) has shown the method to be generally valid; however, some exceptions have been found(7,8). In addition, recent results have been reported which indicate that the formation of sigma phase may be necessarily be detrimental to an alloy(8,9).

About five years ago, a long-time (up to 25,000 hours) testing program was instituted to determine the effect of phase instability on the creep-rupture behavior of some representative nickel-base superalloys. This report describes the results of this experiment.

Experimental Procedure

From the alloys available at the start of this experiment, four alloys were selected: three forged alloys, Udimet 500, Udimet 520, and Udimet 700; and one cast alloy, Inco 713C. Stress-rupture tests on the Udimet 500 and Udimet 520 alloys were carried out on specimens machined from commercially forged blades while Inco 713C test specimens were machined from cast bars and test specimens of Udimet 700 were machined from bar stack. All the tests were performed following standard ASTM procedures. The wrought blades were given heat treatments which were recommended at the time of the initiation of this experiment. The heat treatments and chemistries of the alloys are presented in Table I. Also included in Table I is the N_v number for each alloy(4). To obtain additional information on long time stability of Udimet 500 and Udimet 520, specimens of these alloys were aged for 9700 and 15,000 hours at 1450°F prior to stress-rupture testing.

The broken stress-rupture specimens were examined by light and electron microscopy. Samples were etched using either Fry's reagent or a solution consisting of six parts HCl and one part HNO_3. To determine the type and amount of the carbide and sigma type phases, x-ray diffraction analysis was carried out on residues obtained by digesting the samples in an electrolyte of 10% HCl in methanol. Transmission electron microscope samples were prepared by electropolishing using an electrolyte consisting of a 20% solution of $HClO_4$ in methanol.

Results

Udimet 700

The results of stress-rupture testing Udimet 700 at 1500°F are summarized in Figure 1. A significant change in the slope of the stress-rupture curve in Figure 1 is noted starting at about 700 hours. This results in a difference between the extrapolated life and the actual life of about 5500 hours at a stress of 30,000 psi. Light micrographs of stress-rupture samples revealed that at 774 hours a phase started to precipitate with a plate-like morphology and dispersed throughout the grains parallel to discrete crystallographic planes, Figure 2. With increasing time of testing progressively more of this phase formed. X-ray diffraction analysis of digested residues identified this precipitate as a sigma-type phase. Examination of the specimens near the fracture surface indicated that voids developed within the grains and at the grain boundaries, Figure 3. Close examination of these voids revealed that they have crystallographic facets parallel to the sigma phase plates, indicating that they are formed predominately at the sigma phase. These voids resulted in a trans-granular type failure.

Inco 713C

The results of stress-rupture testing of Inco 713C at 1500°F are summarized in Figure 4. Up to 10,000 hours of testing the stress-rupture curve closely approximates a straight line. Light micrographs of samples taken at various times of testing are shown in Figure 5. After 319 hours at 50,000 psi, another phase forms predominately in the interdendritic spaces. The amount of this phase increased with increased test time. The phase has a plate morphology and lies parallel to discrete crystallographic planes of the grains in which it forms. X-ray diffraction analysis identified this phase as sigma.

Udimet 520

The results of stress-rupture testing of Udimet 520 at 1450°F both before and after aging at 1450°F are summarized in Figure 6. The stress-rupture curves approximate a straight line for times up to 16,000 hours. It is interesting to note that prior aging for up to 15,000 hours has had no significant effect on the stress-rupture behavior of the alloy. Light micrographs of broken stress-rupture specimens are given in Figure 7. The plate-like precipitate seen in the specimen aged for 15,000 hours prior to testing, Figure 7a, has been identified by x-ray diffraction analysis as sigma phase, with a = 8.96A, c = 4.63A. This phase was also present after 9700 hours of aging at 1450°F and occurred in the specimen that had not been aged after 6476 hours of testing. The relative amount of sigma phase in the specimen tested for 6476 hours was approximately equal to that formed after 9700 hours of straight aging, which was considerably less than that formed after 15,000 hours of aging. Testing the sample aged for 15,000 hours for an additional 7361 hours at 1450°F did not significantly increase the amount of sigma plase.

In addition to the formation of sigma phase, two other phase instabilities were noted after aging and stress-rupture testing. The intermetallic phase Ni_3 (Al,Ti) - γ' coarsened, and the $M_{23}C_6$ carbide phase increased in amount at the expense of the MC carbide phase. The γ' coarsening kinetics followed an approximate expression of the type

$$d = kt^{1/3}$$

where d is the particle diameter, t is time, and k is a constant for a given aging temperature. Particles of γ' grow from 0.09 μm to 0.38 μm after aging for 9700 hours at 1450°F and to 0.40 μm after 15,000 hours, Figure 8. No significant effect of stress on the coarsening kinetics of γ' was detected. The kinetics of $M_{23}C_6$ formation at 1450°F were such that while the predominate phase after the initial heat treatment was MC, after 15,000 hours of aging the MC was almost completely replaced by $M_{23}C_6$. The grain boundaries initially were found to contain a nearly continuous layer of carbides which became slightly thicker with aging, Figure 8. Adjacent to this grain boundary carbide, a layer of γ' formed during aging.

Optical micrographs adjacent to the fracture surface of all specimens of Udimet 520, independent of stress level or aging history, revealed wedge-shaped grain boundary cracks at triple point junctions, Figure 9.

Udimet 500

The results of the stress-rupture testing of Udimet 500 at 1450°F both before and after aging at 1450°F are summarized in Figure 10. Aging of the specimens prior to testing for times of both 9700 hours and 15,000 hours drastically reduced the rupture strength. At 40,000 psi this resulted in a decrease in the rupture life from 2000 hours to 600 hours. Light micrographs of typical rupture specimens are included in Figure 11. The second phase seen in Figure 11b with a plate morphology has been identified by x-ray diffraction as sigma phase, a = 8.88A, c = 4.59A. This phase was also found in this alloy after aging for 9700 hours and 15,000 hours at 1450°F. The amount of sigma phase, however, was considerably less than that found after identical treatments in Udimet 520. In the heat treated condition, the γ' phase was found to be present in two different size spheres, 0.40 μm and 0.08 μm, Figure 12a. With increasing time at 1450°F, the smaller particles grow faster than the larger ones resulting in a normal distribution of γ' with a mean size of 0.6 μm after 15,000 hours, Figure 12b. There again did not appear to be any stress dependence to the growth of γ'.

As in Udimet 520, with increasing time at 1450°F, the MC phase was replaced by the $M_{23}C_6$ carbide phase. In Udimet 500, however, the carbide phase was not present at the grain boundary as a continuous film but rather as discrete particles, and during aging the carbide particles remained discrete, Figure 12b. A γ' film can also be seen to form around these particles as in Udimet 520. Light micrographs taken near the fracture surface reveal the presence of rows of voids along the grain boundaries oriented perpendicular to the stress axis, Figure 13. All specimens of Udimet 500 contained such rows of voids independent of stress level or aging treatment. These voids are generally associated with the carbide particles present on the grain boundaries.

Discussion of Results

In all of the alloys tested, three phase instabilities developed during long-time testing. The γ' phase grew, the $M_{23}C_6$ phase gradually replaced the MC phase, and sigma phase was found to form. While sigma phase has been previously detected in Udimet 700, Udimet 520 and Inco 713C(7,8), this is the first reported incident of this phase in Udimet 500. The method of predicting the formation of sigma phase based on electronic configuration predicts that some sigma may form in Udimet 500 and Udimet 700; however, Udimet 520 and Inco 713C should be well within the γ phase field. In fact, it was found that although Udimet 520 should be a "safer" alloy than Udimet 500 more sigma phase forms in Udimet 520. Thus, this method of predicting the occurrence of sigma phase, while of considerable merit in a great many alloy systems, has serious limitations in other alloys such as Udimet 520 and Inco 713C.

Although sigma phase formed in all four alloys, the presence of this particular phase instability did not have an universal deleterious effect on the stress-rupture behavior. In Udimet 700, the precipitation of this phase correlated directly to a reduction in the stress-rupture strength, as seen in Figures 1 and 2. The presence of sigma phase in this alloy results in the relatively rapid formation of crystallographically shaped voids at the sigma particles. These voids thus formed are an easy avenue for crack propagation resulting in an "intersigmatic" type failure. This behavior closely parallels that reported for the effect of sigma phase in IN 100 by Ross(2).

In the other three alloys tested no such direct effect of sigma precipitation was found. In Inco 713C the formation of this phase during testing had no effect on the rupture strength. Also in Udimet

520, the formation of this phase during testing or aging prior to testing had no effect on the rupture strength. While no break was found in the Udimet 500 stress-rupture curves that could be related to the formation of sigma phase, prior aging at 1450°F did result in a decrease in the rupture strength.

Both Udimet 500 and Udimet 520 form sigma with long-time aging, but only Udimet 500 is deleteriously affected by this treatment. An explanation of this effect appears to lie in the different rupture behavior of these two alloys. As was previously noted, the grain boundary carbide morphology was quite different in the two alloys independent of stress level of testing or aging history. In Udimet 500, the carbide phase existed on the grain boundaries as discrete particles, Figure 12, while in Udimet 520, it formed a much more continuous layer, Figure 8.

Although the replica in Figure 8 would indicate this carbide layer was devoid of structure, transmission electron micrographs indicate the layer is made up of many interconnected carbide particles, Figure 14. This difference in carbide morphology is no doubt the reason for the different mechanisms of crack nucleation and propagation that was found in the broken stress-rupture samples of these two alloys. In Udimet 500, the discrete grain boundary carbide particles result in the nucleation of rows of small voids on grain boundaries oriented perpendicular to the stress axis, whereas in Udimet 520, the continuous carbide film results in wedge-shaped cracks being formed at grain boundary triple point junctions. This difference in the mechanism of crack nucleation and propagation is also reflected in the creep ductilities. Again, independent of testing conditions or aging history, Udimet 500 had a creep elongation of around 5% while Udimet 520 had an elongation of around 11%. This reduced ductility of Udimet 500 compared to Udimet 520 can also be seen by comparing the creep curves of the two materials, Figure 15. It is of particular interest to note that Udimet 500 fails in the steady-state or secondary region of the creep curve before the tertiary stage is reached. This lack of a tertiary stage of creep was a feature which was found in all Udimet 500 tested, independent of testing condition or long time aging history. The fact that failure occurs in the secondary region of creep indicates that once a crack is nucleated by a row of voids it is very unstable, and propagation to failure is quite rapid. Udimet 520, on the other hand, seems able to stabilize the cracks nucleated at wedge type voids such that a tertiary stage of creep is reached and results in considerably more ductility.

From these differences in the creep behavior of Udimet 500 and Udimet 520, an explanation of the deleterious effects of aging on Udimet 500 is suggested. The aging treatment is seen to have no effect on the grain boundary carbide distribution, the creep ductility, the crack nucleation and propagation mechanism, or the type of creep curve that results. It is clear that the formation of sigma with aging does not, in this case, result in intersigmatic failures as in Udimet 700 and IN 100. The primary effect of aging seems to be the coarsening of the γ' phase. As this phase coarsens the steady-state creep rate was found to increase in both alloys. Due to the fact that Udimet 500 has very limited ductility, coupled with the fact that it does not display a tertiary stage of creep, an increase in the steady-state creep rate directly results in a reduction in the observed stress-rupture life. However, due to the greater ductility of Udimet 520 by comparison, an increase in steady-state creep rate by γ' growth has a much smaller effect on the stress-rupture life.

Summary and Conclusions

In spite of the general validity of predicting the occurrence of sigma phase in nickel-base superalloys using concepts based on electronic configurations, some of the predictions of such a model were not found to hold in the present experiment. Two alloys considered safe, Udimet 520 and Inco 713C and one border-line alloy Udimet 500 were found to develop sigma phase with long-time exposure at 1450°F. More importantly, the presence of sigma phase in three of the four alloys tested, Udimet 500, Udimet 520, and Inco 713C was not found to have any deleterious effect on the 1450°F stress-rupture behavior of the alloy. Long-time aging of Udimet 500 did result in a loss of rupture life, but this could not be related to the formation of sigma phase. Rather it is felt that this was a result of the growth of the γ' phase coupled with the limited ductility of this alloy in creep.

References

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2. E. W. Ross, J. Metals, 12, 1967, p 12.

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7. H. E. Collins, Interim Engineering Progress Report # 3, AF33(615)-5126.

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TABLE I

Chemical Compositions

Alloy	Ni	Cr	Fe	Co	Mo	W	Ti	Al	Mn	C	Si	B	Cb	Zr
Inco 713C	Bal	12.43	1.66	0.22	4.48	--	0.70	6.18	0.02	0.16	0.21	0.01	2.13	0.10
Udimet 700	Bal	15.50	0.15	18.80	5.10	--	3.41	4.42	0.10	0.06	0.10	0.03	--	0.05
Udimet 520	Bal	12.1	0.10	12.2	6.20	0.98	3.00	1.98	0.1	0.04	0.1	0.005	--	--
Udimet 500	Bal	19.3	0.24	19.0	4.15	--	2.95	2.99	0.1	0.08	0.13	0.004	--	--

* Calculation based on the method of Boesch and Slaney (4)

Heat Treatments

Inco 713C	As Cast
Udimet 700	2050°F/4 h/A.C. + 1975°F/4 h/A.C. + 1550°F/24 h/A.C. + 1400°F/16 h/A.C.
Udimet 520	2050°F/4 h/A.C. + 1550°F/24 h/A.C. + 1400°F/16 h/A.C.
Udimet 500	1975°F/4 h/A.C. + 1550°F/24 h/A.C. + 1400°F/16 h/A.C.

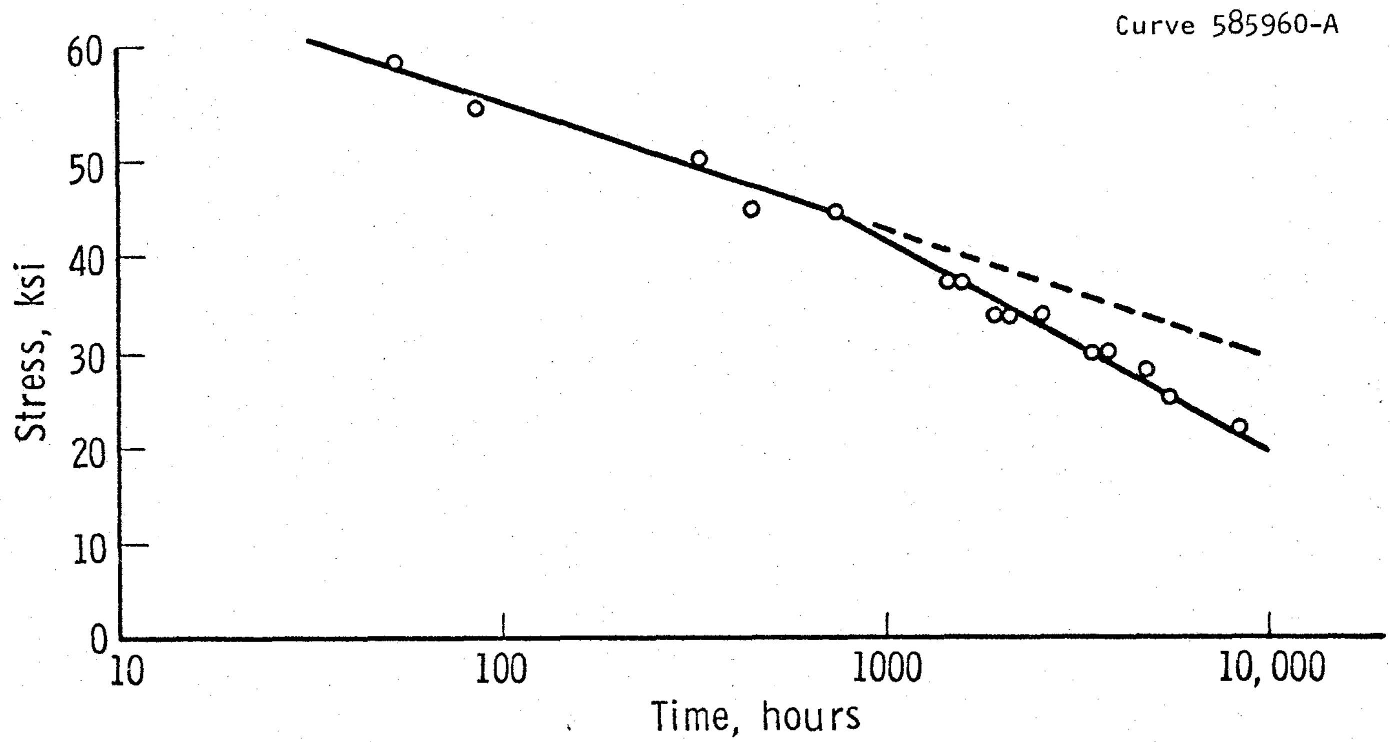


Fig. 1—Stress-rupture curve for Udimet 700 at 1500°F

(a) 51 hours at 60,000 psi

(b) 774 hours at 45,000 psi

(c) 5675 hours at 25,000 psi

Fig. 2. Microstructure of stress-rupture samples of Udimet 700 after various times of testing at 1500°F

-250X

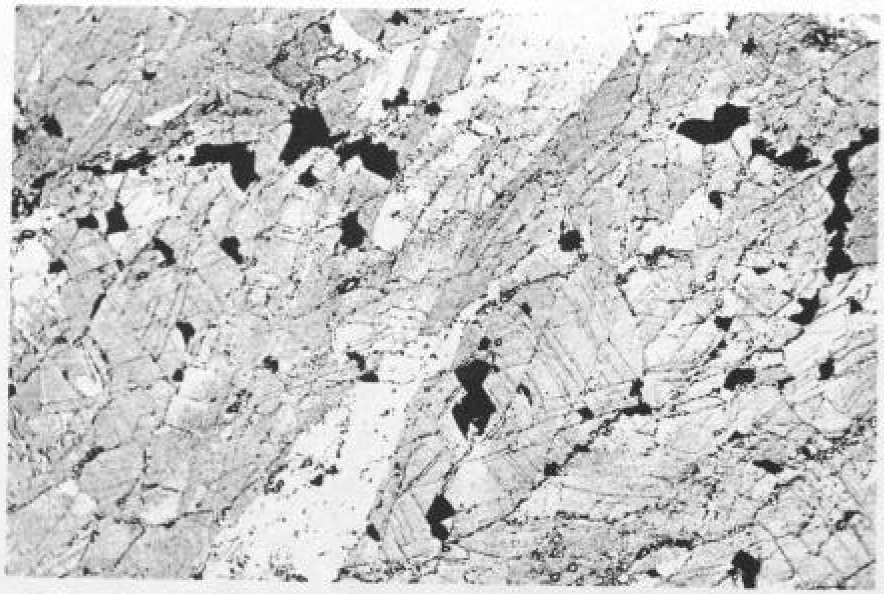


Fig. 3. Microstructure of Udimet 700 near the fracture surface showing void formation on crystallographic planes parallel to the sigma phase -250X

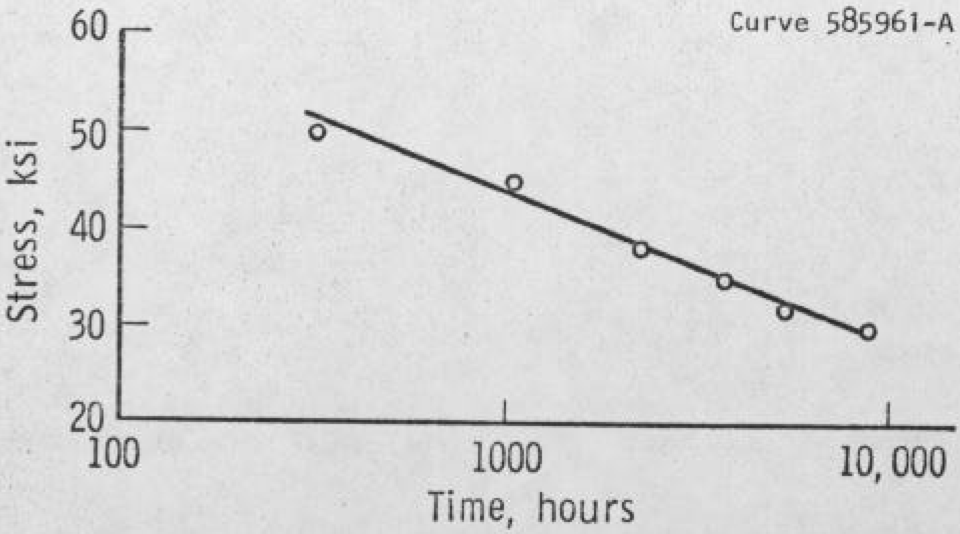
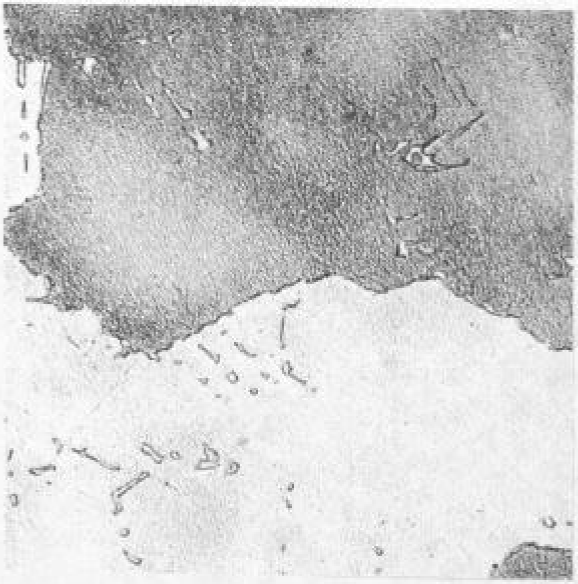
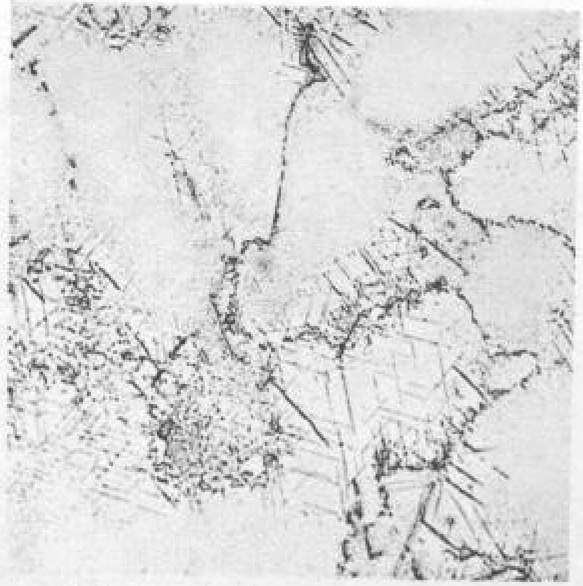


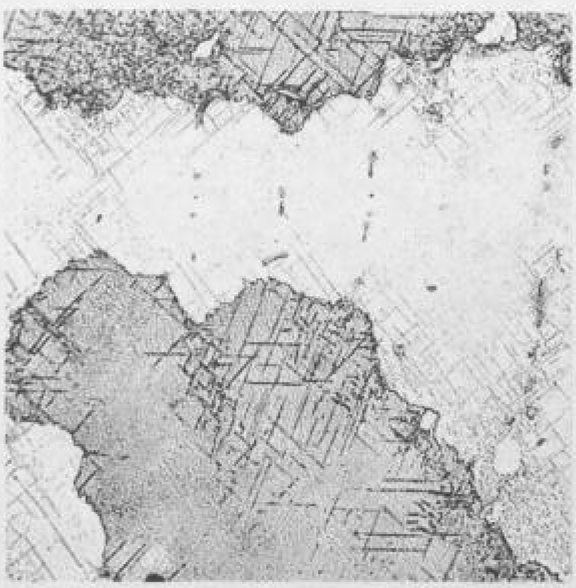
Fig. 4—Stress-rupture curve for Inco 713C at 1500 °F



(a) 91 hours at 60,000 psi



(b) 319 hours at 50,000 psi



(c) 2263 hours at 38,000 psi

(d) 4937 hours at 30,000 psi

Fig. 5. Microstructure of Inco 713C after various times of testing showing the development of sigma phase

250X

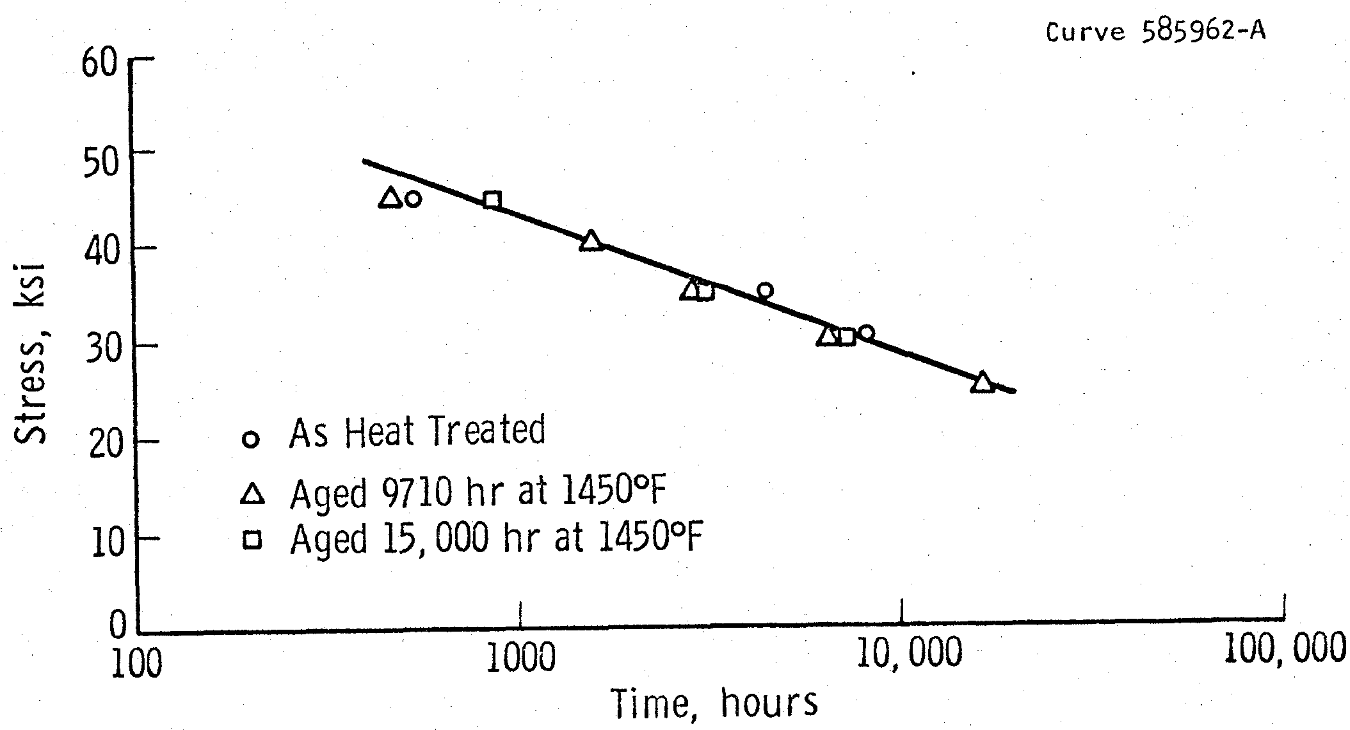
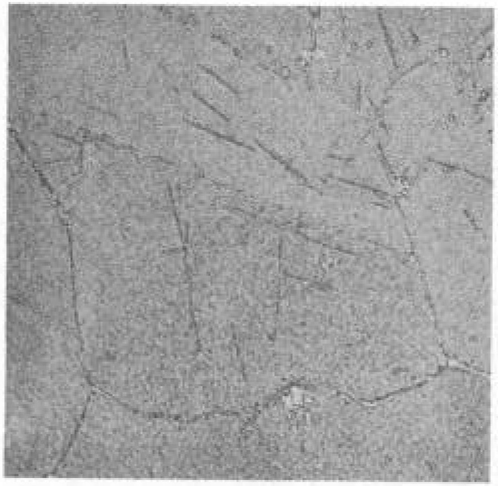
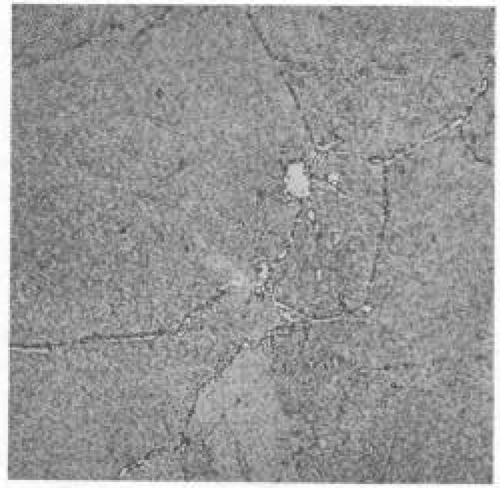


Fig. 6—Stress rupture curves for heat treated and aged Udimet 520 at 1450°F



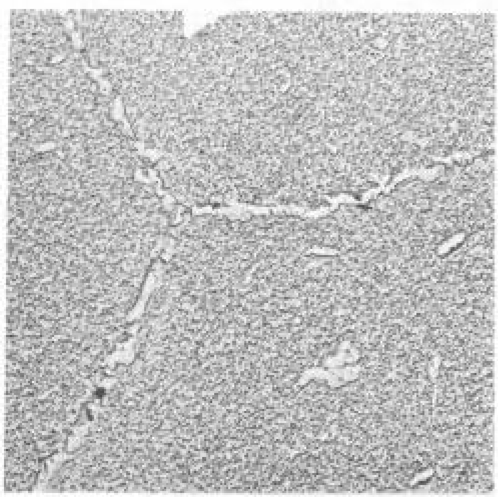


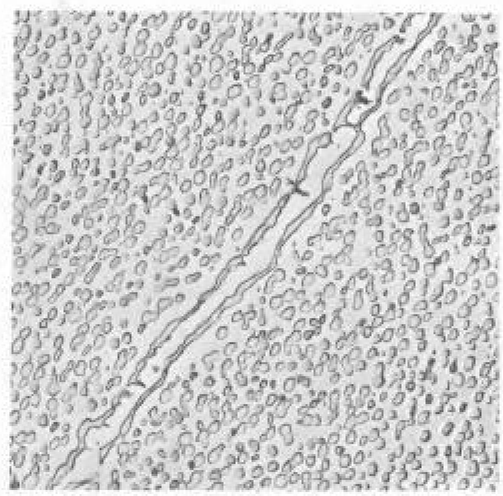
(a) 7361 hours at 30,000 psi aged 15,000 hours at 1450°F prior to test

(b) 6476 hours at 25,000 psi

Fig. 7. Microstructure of Udimet 520 after various aging and testing times at 1450°F

-500X





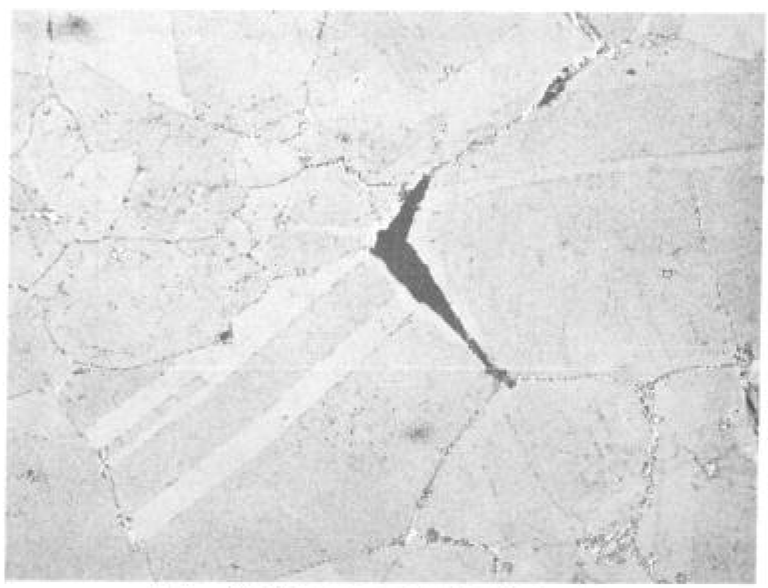
(a) As-heat treated

(b) Heat treated and aged 9700 hours at 1450°F

Fig. 8. Electronmicrographs of Udimet 520 showing the growth of γ' particles and development of a continuous grain boundary carbide film surrounded by a layer of γ'

-3500X

(a) 7361 hours at 30,000 psi aged 15,000 hours at 1450°F prior to test
Unetched 100X



(b) 6476 hours at 25,000 psi
Etched 200X

Fig. 9. Intergranular voids formed near the fracture surface of Udimet 520 rupture specimens in Fig. 7

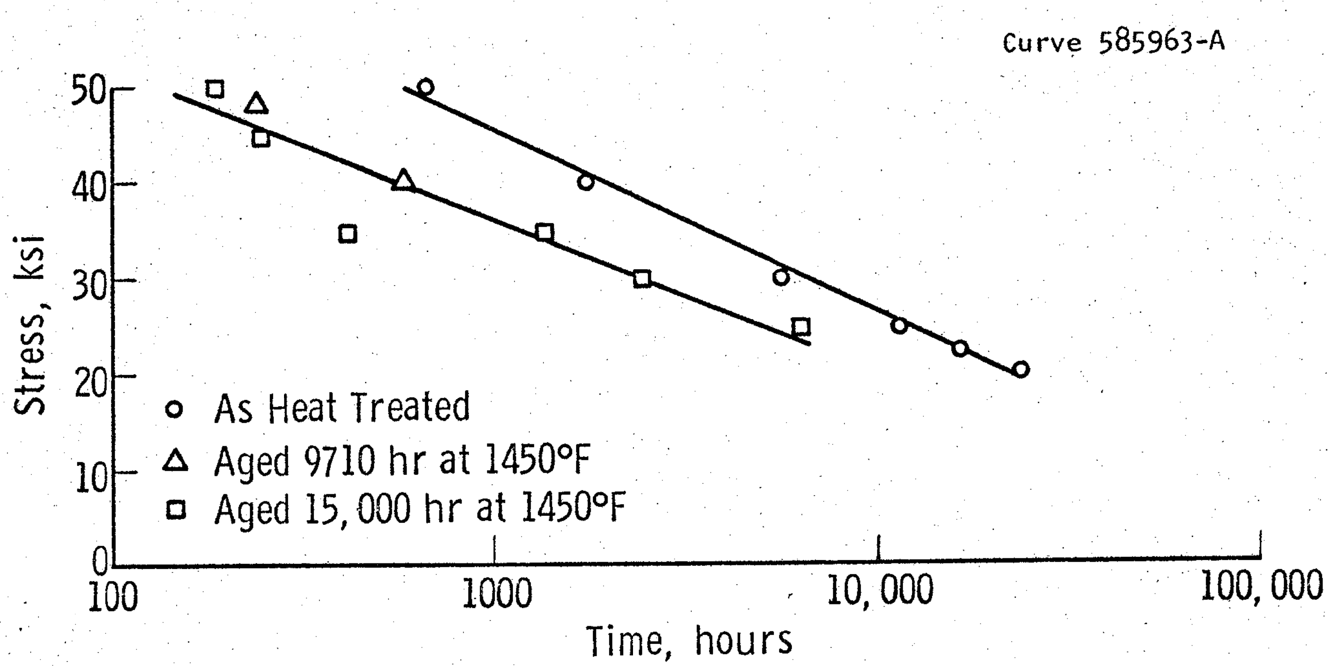
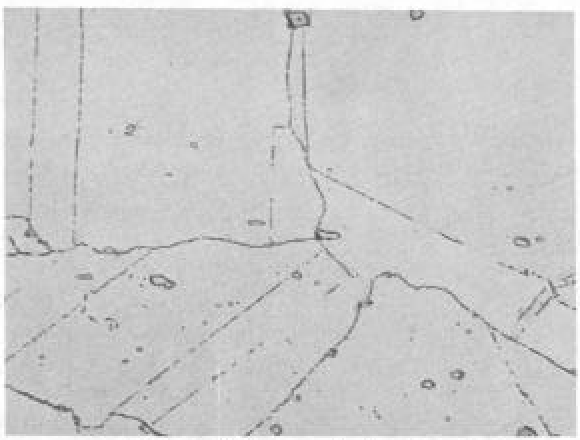
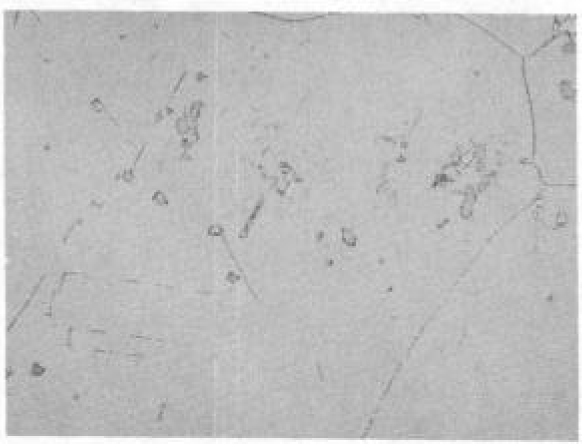


Fig.10—Stress-rupture curves for heat treated and aged Udimet 500 at 1450°F



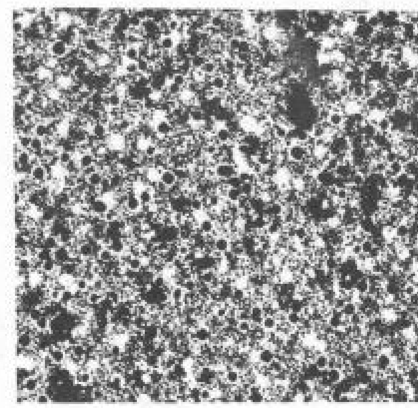
(a) 1729 hours at 40,000 psi



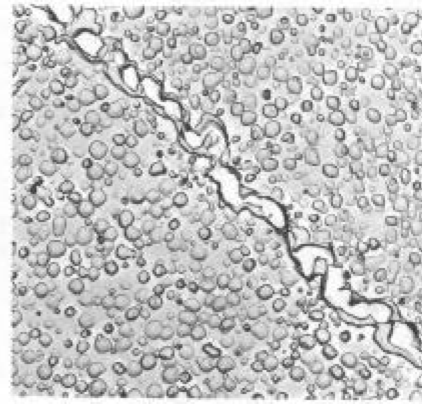
(b) 11,579 hours at 25,000 psi

Fig. 11. Microstructure of Udimet 500 after various testing times at 1450°F

-500X



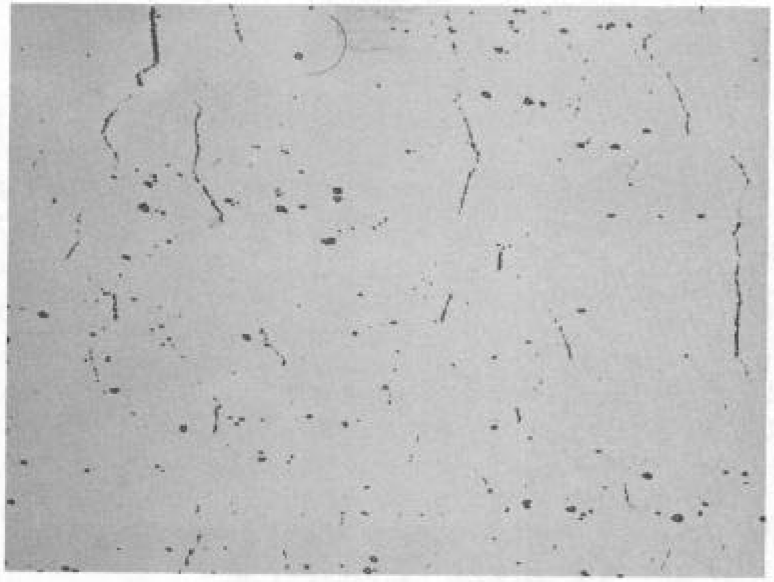
(a) as-heat treated
H_3PO_4 - Extraction Repli
(γ' phase is dark sphere



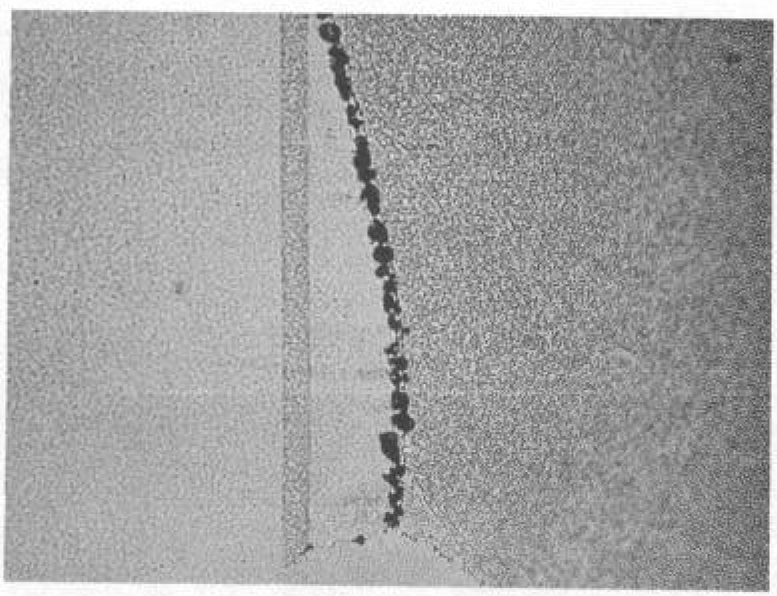
(b) 920 hours at 35,000 psi
aged 15,000 hours at 1450
prior to testing

Fig. 12. Electron micrographs of U
after various times at 1450°F showi
of duplex γ'to normal distribution,

Reduced 25% for Publication



(a) 2468 hours at 30,000 psi
unetched 100X



(b) 2468 hours at 30,000 psi
etched 500X

Fig. 13. Microstructure of Udimet 500 test specimens near the fracture surface showing the development of grain boundary voids.

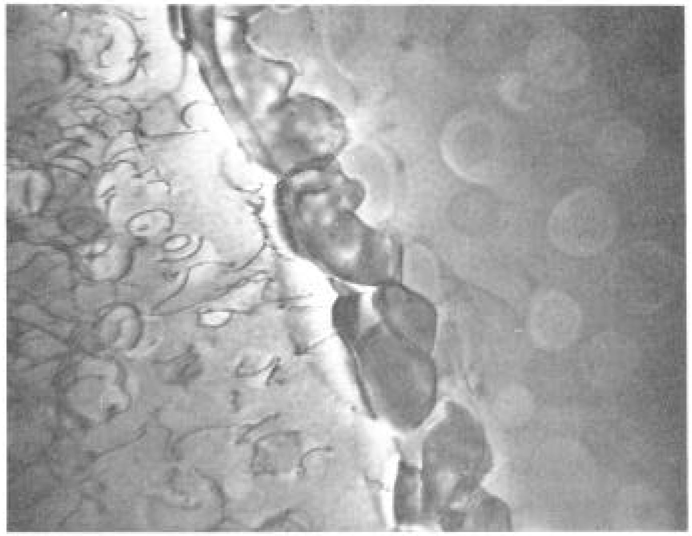


Fig. 14. Transmission electromicrograph of grain boundary in Udimet 520 showing continuous carbide layer made up of small interconnected grains 30,000X

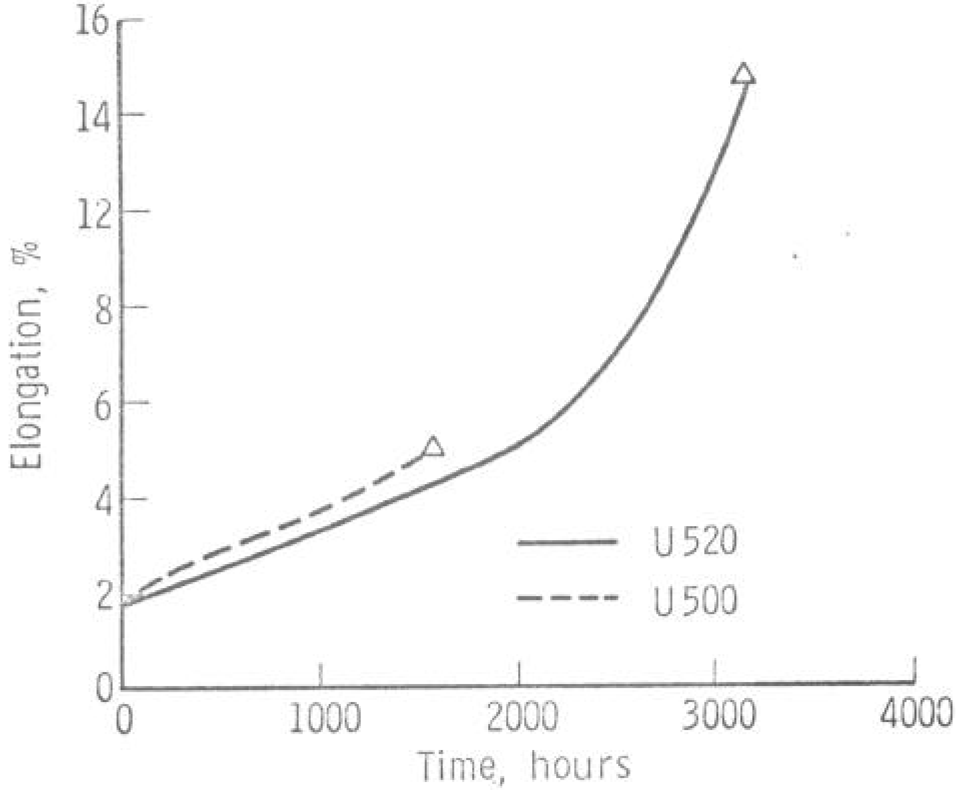


Fig. 15—Creep curves for Udimet 500 and Udimet 520 after aging for 15,000 hours at 1450°F.
Stress - 35,000 psi