WADC TECHNICAL REPORT 54-40

THE EFFECT OF GRAIN SIZE ON THE FATIGUE AND CREEP PROPERTIES OF STAINLESS STEEL AND INCONEL AT ELEVATED TEMPERATURES

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April 1954

Materials Laboratory
Contract No. AF33-(038)-22808
RDO No. 615-13

Wright Air Development Center
Air Research and Development Command
United States Air Force
Wright-Patterson Air Force Base, Ohio

McGregor & Werner, Inc., Dayton, O.
200 July, 1954

FOREWORD

This report was prepared by the University of California at Berkeley, California, under USAF Contract No. AF 33(038)-22608. The contract was initiated under Research and Development Order No. 615-13, "High Temperature Alloys", and was administered under the direction of the Materials Laboratory, Directorate of Research, Wright Air Development Center, with Lt J.H. DeVan acting as project engineer.

ABSTRACT

This investigation was undertaken to evaluate the effect of grain size, as produced by annealing, on the high temperature creep and fatigue properties of inconel and an 18-8 stainless steel. In order to isolate the effects of grain size and annealing from other metallurgical effects, an attempt was made to select materials which were not particularly prone to extraneous changes such as precipitation or spheroidization of intermediate phases. But, in order to estimate the practical utility of practicing annealing for grain size control of high temperature alloys, the two representative alloys inconel and type 304 extra low carbon 18-8 stainless steel were chosen. Unfortunately both of these materials were found to exhibit structural changes during annealing which might have affected their creep and fatigue properties. The results indicate that to clearly delineate the effect of grain size on the properties of metals it will be necessary to use metals in which all auxiliary microstructural changes are absent.

PUBLICATION REVIEW

This report has been reviewed and is approved.

FOR THE COMMANDER:

[illegible] E. SORTE
Colonel, USAF
Chief, Materials Laboratory
Directorate of Research

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INTRODUCTION

The extensive literature on the effect of grain size on the creep properties of metals was reviewed by Gillett(1) several years ago. In general, the majority of the investigations suggested that the finer-grained metals exhibit superior creep strength at low temperatures and high strain rates in harmony with the effects of grain size on the low temperature tensile properties. But at sufficiently high temperatures and low creep rates, an inversion occurs, and the coarser-grained metals exhibit superior creep resistance. Extensive confirmation of this thesis can be found in the general literature on creep(2-29). And these conclusions have gained support because they are consistent with the general knowledge that grain boundaries behave somewhat like a viscous media at elevated temperatures, thereby permitting grain boundary shearing and intergranular fracturing at elevated temperatures.

Any statement concerning the effect of grain size on the creep resistance, however, requires extensive qualifications. It is well known that single metal crystals have lower creep resistance than polycrystalline aggregates suggesting that if the high temperature creep resistance improves with an increase in grain size, an optimum grain size for highest creep resistance should exist. Weaver(20,23) and Hanson(21) present evidence in support of such optimum grain size. But it is yet uncertain whether their results can be exclusively attributed to grain size, because the auxiliary effects of annealing to achieve the larger grain sizes might well have introduced other structural differences in their test materials. Furthermore much evidence casts doubt on the hypothesis that improved high temperature creep resistance can be achieved by grain coarsening alone:

1. Whereas Burghoff et al(31) found that the creep resistance of a 70-30 brass at 400°F increase with increasing grain size, White and Clark(7) reported that a fine-grained brass was superior in creep resistance to a coarsened specimen at the same temperature.

2. Hanffstengel and Hanemann(15) found that coarse-grained lead does not continue to give improved creep resistance over fine-grained specimens as the creep temperature is raised.

3. Russell, Greenwood, Orr, and Wormser(4,9,12,13,19), observed important variations in the creep resistance of high purity lead even when the grain size was held constant.

4. Jenkins, Tapsell, Mellor, and Johnson(16) report no improvement in the high temperature creep resistance of a killed steel upon grain coarsening.

5. Cross and Lowther(22) observed no improvement in the high temperature creep resistance of a carbon-vanadium steel upon grain coarsening.

6. Parker and Riisness(25) concluded that the creep resistance of OFHC Cu was independent of grain size.

7. Servi and Grant's(32) data suggest that the creep resistance of high purity aluminum is almost independent of grain size.

8. Vicars(27) has shown that the creep resistance of high purity alpha solid solutions of Al and dispersion alloys of $CuAl_2$ in high purity aluminum is insensitive to grain size.

9. Sherby and Dorn(28) have demonstrated that high temperature creep resistance of 2S-O (commercially pure Al) improves substantially upon annealing even when no change in grain size is detectable.

10. McLean(29) has shown that the contribution of grain boundary slip to the total creep strain is rather small.

The persistence of such data as quoted above suggests that the high temperature creep resistance of metals might be only mildly affected by the grain size per se; the commonly observed improvement in high temperature creep resistance with increase in grain size might well be attributable to other microstructural changes that are induced by the annealing treatments commonly used to achieve grain coarsening. Thus the effect of grain size on the creep resistance of metals is yet unknown; and until more fundamental investigations are made in such a way as to isolate a true grain size effect, it will be necessary to refer not only to the grain size but also to the details of the treatments which were used to develop the grain size.

In contrast to the effect of grain size (or annealing treatments) on the creep properties of metals very little work has been done to uncover the possible effect of grain size on the fatigue of metals at elevated temperatures. If the initiation of a fatigue crack is associated with slip damage and the accumulation of dislocations leading to the nucleus of a fatigue crack, the effect of grain size on the endurance limit should parallel somewhat the effect of grain size on the plastic properties. This deduction is in harmony with the known data on the effect of grain size on the plastic properties and endurance strength of metals at low temperatures. Here the endurance limit decreases with increasing grain size(33).

In recent surveys of this field Dolan(34) has shown that the existing data on the effect of grain size on the high temperature fatigue strength of metals are too meager to be conclusive. Cross(8) investigated the fatigue and creep properties of a low carbon and a high carbon 18-8 stainless steel, each as cast and wrought. The mean grain diameters in the two cast metals were about five times greater than those for the wrought metals. Whereas the creep resistances of the cast and wrought specimens of each metal were almost identical, the coarser-grained cast metals gave lower endurance limits at 1200°F. Although these results might imply that the finer-grained materials have higher fatigue strengths, even at elevated temperature, the results are not conclusive since the observed differences might well be attributed to differences arising from the two alternate methods of production that were used. Toolin and Mochel(35) tested the two similar alloys which are described in Table I.

TABLE I

Alloys Tested By Toolin and Mochel

Alloy	Production	C	Si	Cr	Ni	Mo	Co	Fe	Grain Size
1	chill cast	0.37	0.52	25.8	34.8	4.72	33.5	0.65	fine
2	cast in warm mold	0.38	0.52	24.7	33.2	4.62	32.2	4.68	coarse

Endurance Limits (10^7 cycles)

Alloy	1200°F	1500°F
1	63,000 psi.	38,000 psi.
2	51,000 psi.	32,000 psi.

Although the finer-grained specimens again gave superior high temperature endurance limits these differences might be due to differences in composition or methods of casting rather than grain size.

It is the intent of this investigation to evaluate the effect of grain size, as produced by annealing, on the high temperature creep and fatigue properties of inconel and an 18-8 stainless steel in order to extend the existing knowledge in this field.

MATERIALS FOR TEST

In order to isolate the effects of grain size and annealing from other metallurgical effects, an attempt was made to select materials which were not particularly prone to extraneous metallurgical changes such as precipitation or spheroidization of intermediate phases. But, in order to estimate the practical utility of practicing annealing for grain size control of high temperature alloys, the two representative alloys, inconel and Type 304 extra low carbon 18-8 stainless steel were recommended for investigation. Unfortunately both of these materials were found to exhibit structural changes during annealing which might have affected their creep and fatigue properties. Their chemical analyses are given in Table II.

TABLE II

Percent Composition

Metal	C	P	Mn	S	Si	Cr	Ni	Fe	Cu
Inconel	0.05	-----	0.22	0.007	0.22	15.69	76.58	7.03	0.18
Type 304 ELC	0.026	0.023	1.29	0.012	0.58	18.39	8.92	70.76	----

Both alloys were obtained in the form of 3/4 in. diameter bar stock. The grain size produced after annealing the as received stock for one hour in dry hydrogen at various temperatures is shown in Fig. 1. The proposed test temperatures of 1200 and 1500°F, as shown by the broken vertical lines, are well below the grain coarsening temperature for both alloys suggesting that the grain size should be relatively stable at the test temperatures.

The various grain sizes used in this investigation were developed by annealing the specimens machined from the as received stock in dry hydrogen for one hour at appropriate temperatures as shown in Table III.

TABLE III

Grain Size Control

Material	Anneal Temp. $^{\circ}$F	ASTM Grain Size
Type 304 ELC	1652	6
Type 304 ELC	2192	1
Inconel	1292	9
Inconel	1652	9
Inconel	2012	2

The two annealing treatments of 1292 and 1652°F were practiced on inconel in order to ascertain whether annealing alone might affect the fatigue strength even when no change in grain size takes place.

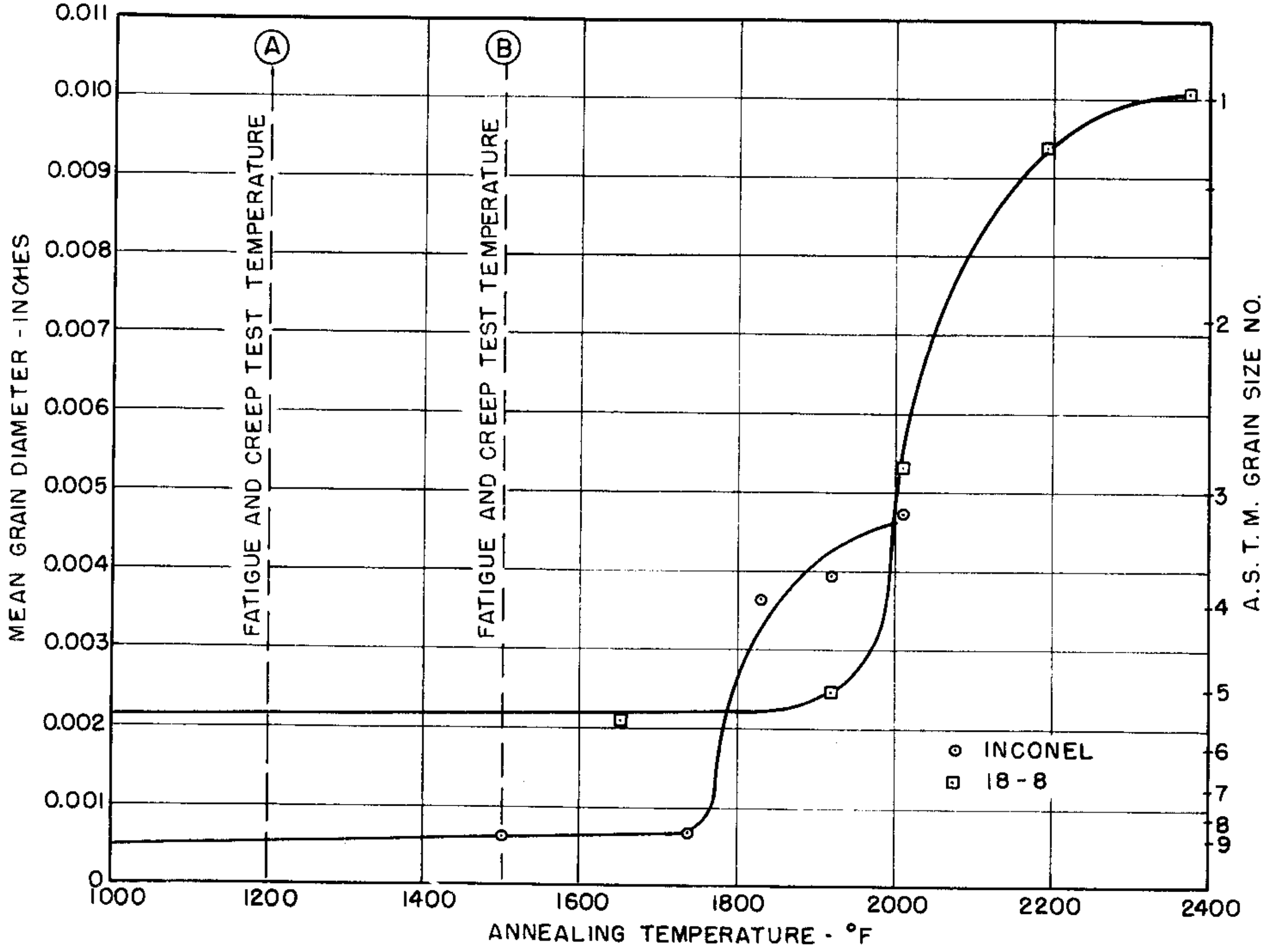


FIG. I EFFECT OF ANNEALING FOR ONE HOUR ON THE GRAIN SIZE OF TYPE 304 18-8 STAINLESS STEEL AND INCONEL.

Typical examples of the microstructures that were developed by annealing are shown in Figs. 2 and 3. Because of furnace limitations it was necessary to anneal the creep and the fatigue specimens separately. Both the creep and fatigue specimens of Type 304 ELC that were annealed at 1652°F gave identical structures, a typical example of which is given in Fig. 2a. But, as revealed by comparison of Figs. 2b and 2c, the creep specimens annealed at 2192°F exhibited slightly greater amounts of carbide precipitation than the fatigue specimens annealed at the same temperature. Furthermore, as documented in the captions below each microstructure, the hardness was higher for the more completely precipitated structure of the creep specimen, although the grain sizes were almost identical. Inasmuch as the creep and the fatigue specimens were selected from different portions of the same bar, it is possible that the observed differences in microstructure might be attributable to variations in chemical composition along the length of the bar. But some slight variations in the details of the heat-treatment program might also have caused the observed differences in structure.

Almost identical structures were obtained by annealing inconel at 1292°F and 1652°F as shown by Figs. 3a and 3b. And the hardness following the 1652°F anneal is only slightly below that obtained after annealing at 1292°F suggesting that the grain size and the states of recovery were almost identical for the two dissimilar annealing conditions that were used. When the microstructures were carefully compared at higher magnifications, however, the specimens annealed at 1652°F appeared to exhibit slightly greater amounts of grain boundary precipitate than those annealed at the lower temperature of 1292°F. Furthermore the coarse-grained specimen produced by annealing at 2012°F exhibited somewhat greater amounts of carbide precipitate than the specimens annealed at the lower temperatures.

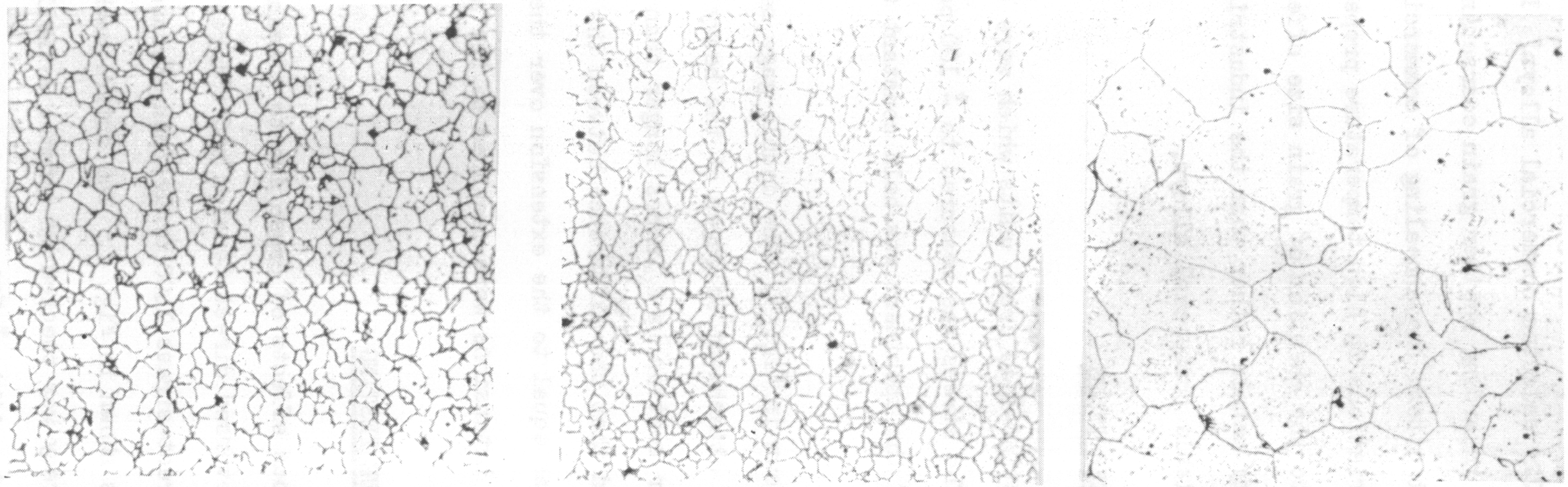
The variations in microstructure which were detected upon annealing inconel and Type 304 ELC 18-8 stainless steel merely emphasize the difficulties that are normally encountered in attempting to control only the grain size of commercial

(a) Anneal Temperature -1652°F
Use: Creep and Fatigue
Rockwell B77 x100

(b) Anneal Temperature -2192°F
Use: Fatigue
Rockwell B66 x100

(c) Anneal Temperature -2192°F
Use: Creep
Rockwell B76 x100

Fig. 2 Microstructures of Annealed Type 304 ELC Stainless Steel



(a) Anneal Temperature - 1292°F
Use: Fatigue at 1200°F
Rockwell B87 x250

(b) Anneal Temperature - 1652°F
Use: Fatigue and Creep
Rockwell B85 x250

(c) Anneal Temperature - 2012°F
Use: Fatigue and Creep
Rockwell B68 x100

FIG. 3 MICROSTRUCTURES OF ANNEALED INCONEL.

alloys by annealing. Undoubtedly more serious problems of structural changes attend the annealing of less carefully selected commercial alloys. It is for this reason that doubt yet persists regarding whether grain coarsening or the auxiliary metallurgical changes that attend the annealing of commercial alloys, are responsible for the observed variations in high temperature properties. Undoubtedly a more definitive answer to the question of grain size effects can be obtained by testing extra high purity metals rather than the industrially significant but complex and poorly controllable commercial alloys.

CREEP TESTS

One of the three identical constant-load creep units which were used in this investigation is shown in Fig. 4. The stress was measured to ± 100 psi and the temperature over the gage section of the specimens was held constant to within ± 2°F throughout each test.

The creep specimen design is shown in Fig. 5. The double gage section type of specimen was used to permit rigid attachment of gages at the heavy shoulder sections of the specimen. The longer section is 2 inches longer than the shorter section. Direct experiments revealed that the difference between the extension of the longer and shorter sections was equal to the extension over the two inch gage section of the longer section. Extensions were measured to the nearest 0.0001 inches.

FATIGUE TESTING

In order to avoid the simultaneous effects of creep due to a mean stress on the fatigue results, completely reversed direct tension and compression stressing was employed. The 5000-pound direct-stress 1400 cycles/min. Krouse testing machine used in this investigation is shown in Fig. 6. The entire machine assembly was carefully realigned and its accuracy was checked by dynamic as well as static tests. The stress was held constant to within ± 500 psi and the temperature of

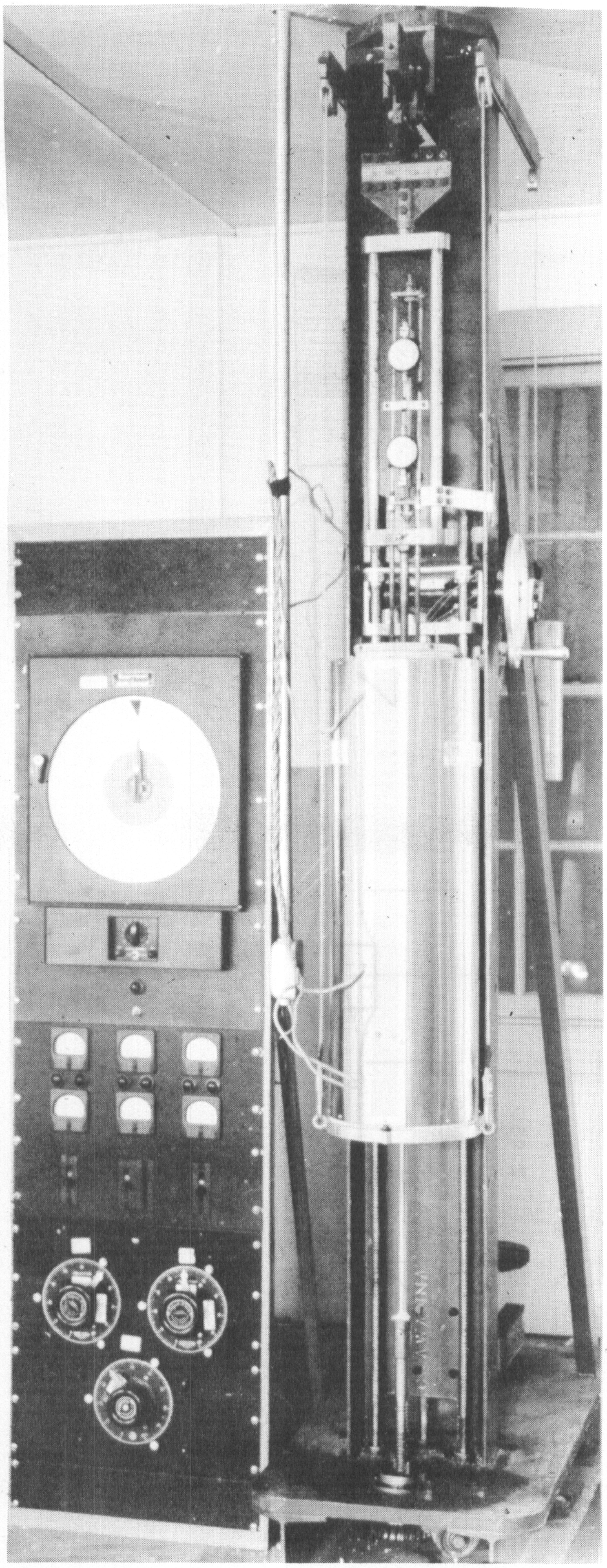


Figure 4 High Temperature Creep Unit

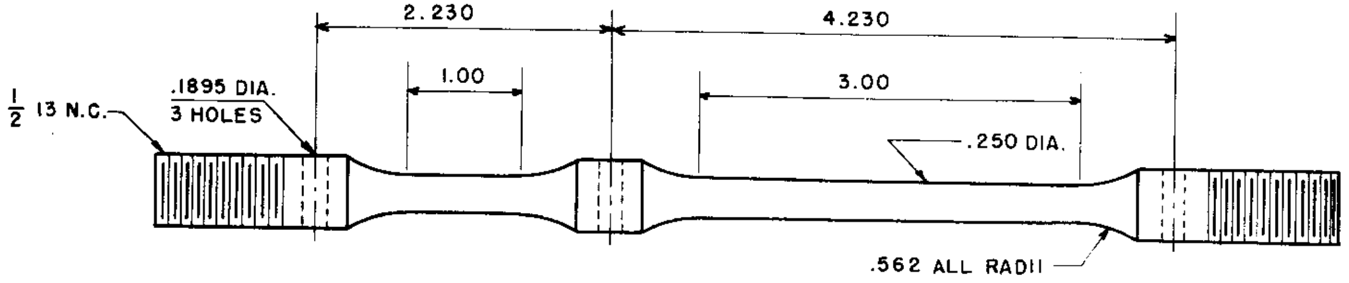


FIG. 5 CREEP TEST SPECIMEN

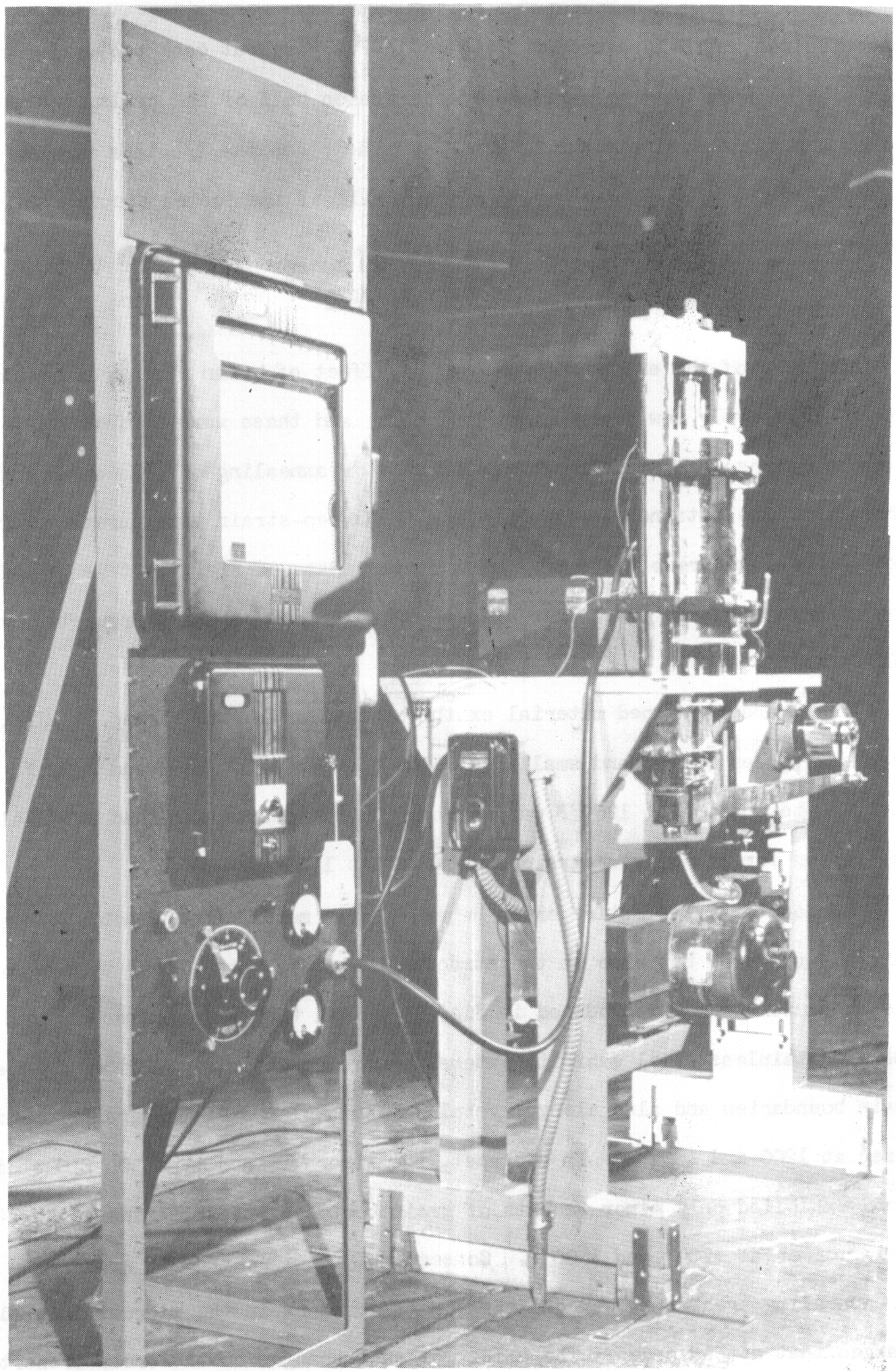


Figure 6 Krouse Direct Stress Fatigue Machine

the specimen was held constant to with ± 2°F throughout each test. In order to minimize plastic bending during the compression half of the cycle, the short column specimen design shown in Fig. 7 was adopted; the 1/4 inch radius induces only a mild stress concentration. All of the tested specimens fractured at the minimum section.

CREEP TEST RESULTS

In view of the extensive data on the effect of grain size or annealing on creep, relatively few creep tests were made; and these were performed for the express purpose of comparing the grain size or annealing effects on fatigue with those that are obtained in creep. From the creep-strain time curves of Fig. 8 and the minimum creep rate and stress rupture data of Fig. 9, it is revealed that there is an apparent grain size effect on the creep resistant properties of 304 ELC stainless steel.

The coarser-grained material exhibited smaller initial creep strains, lower secondary creep rates, and smaller strains to rupture at 1200 and 1500°F. Whereas the time to rupture at 1200°F was shorter for the coarser-grained material, it was longer for the coarser-grained material at 1500°F.

Undoubtedly the entire creep behavior of a metal depends not only on its initial structure but also on the structural changes that attend the creep process. The photomicrographs reproduced in Fig. 10 reveal that the coarser-grained type 304 ELC stainless steel exhibits extensive precipitation of carbides in the grain boundaries and also along crystallographic planes of the grains during creep at 1200 and 1500°F. In contrast, the finer-grained type 304 ELC stainless steel exhibited only minor amounts of grain boundary precipitation of carbides during creep at 1200° and 1500°F. Consequently a difference in grain size (or annealing treatment) can cause major differences in the microstructural changes that attend creep. The effect of such changes on the creep properties, however, cannot justifiably be ascribed to grain size alone. Furthermore the

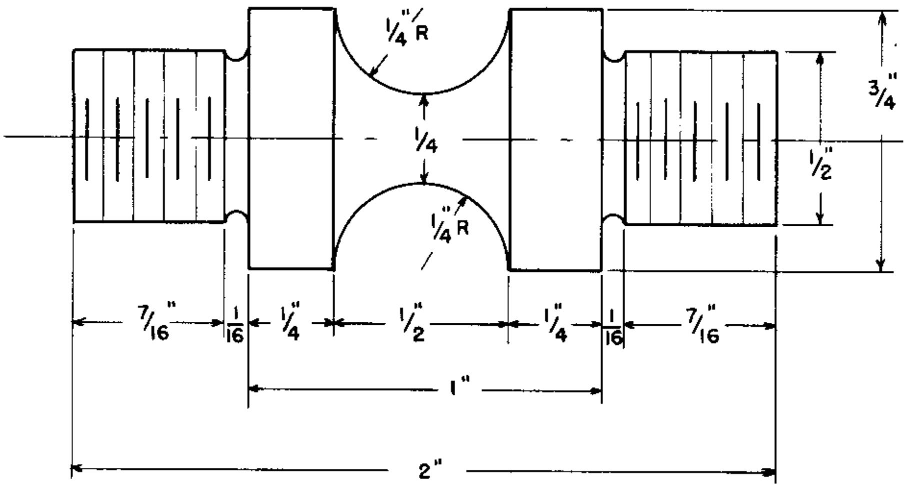
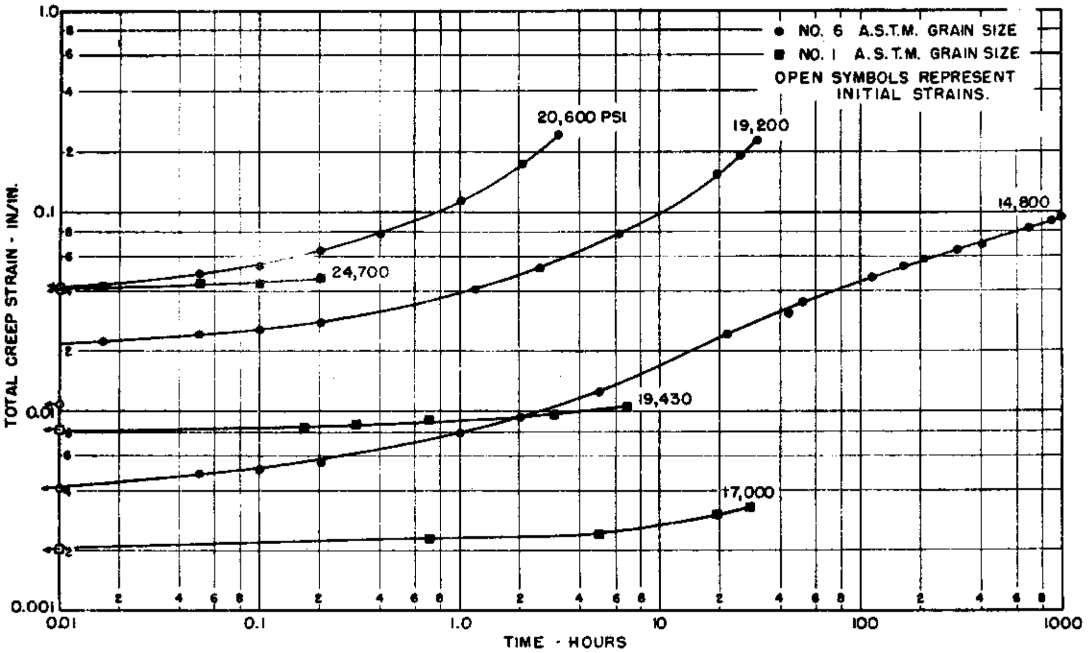


FIG. 7 DIRECT STRESS FATIGUE SPECIMEN.



TOTAL CREEP STRAIN FOR CONSTANT LOADS AS A FUNCTION OF TIME
TYPE 304 ELC STAINLESS STEEL AT 1200°F.

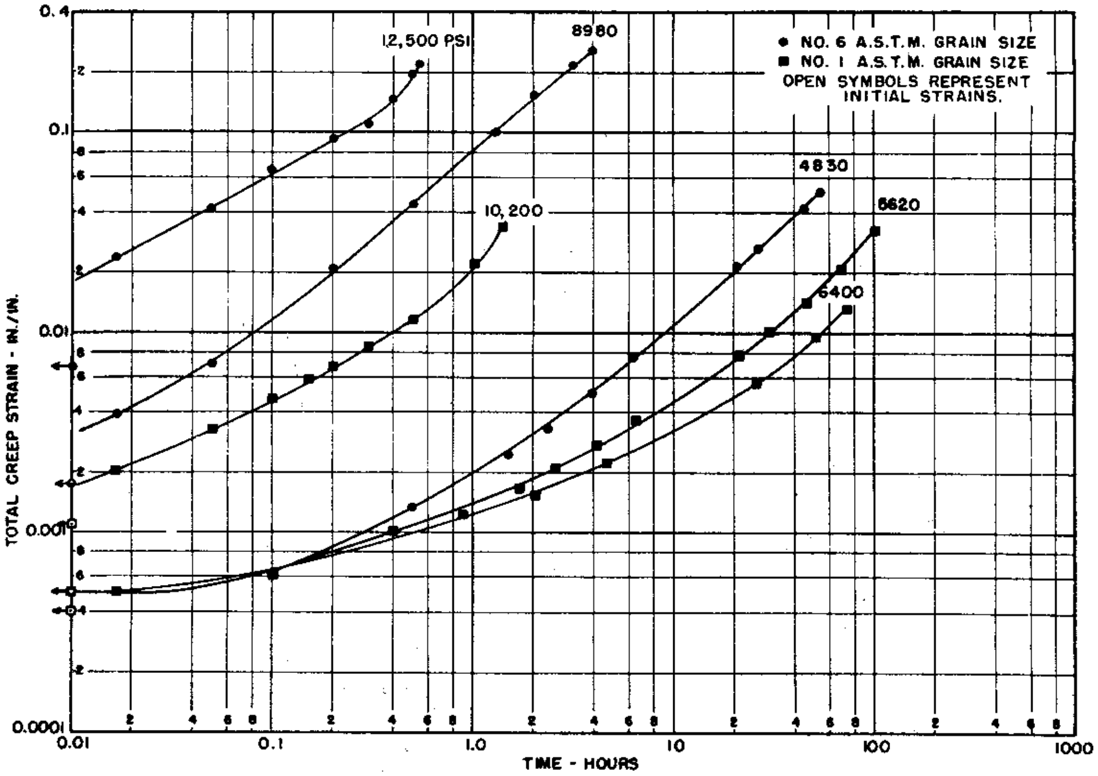


FIG. 8 TOTAL CREEP STRAIN FOR CONSTANT LOADS AS A FUNCTION OF TIME
TYPE 304 ELC STAINLESS STEEL AT 1500°F.

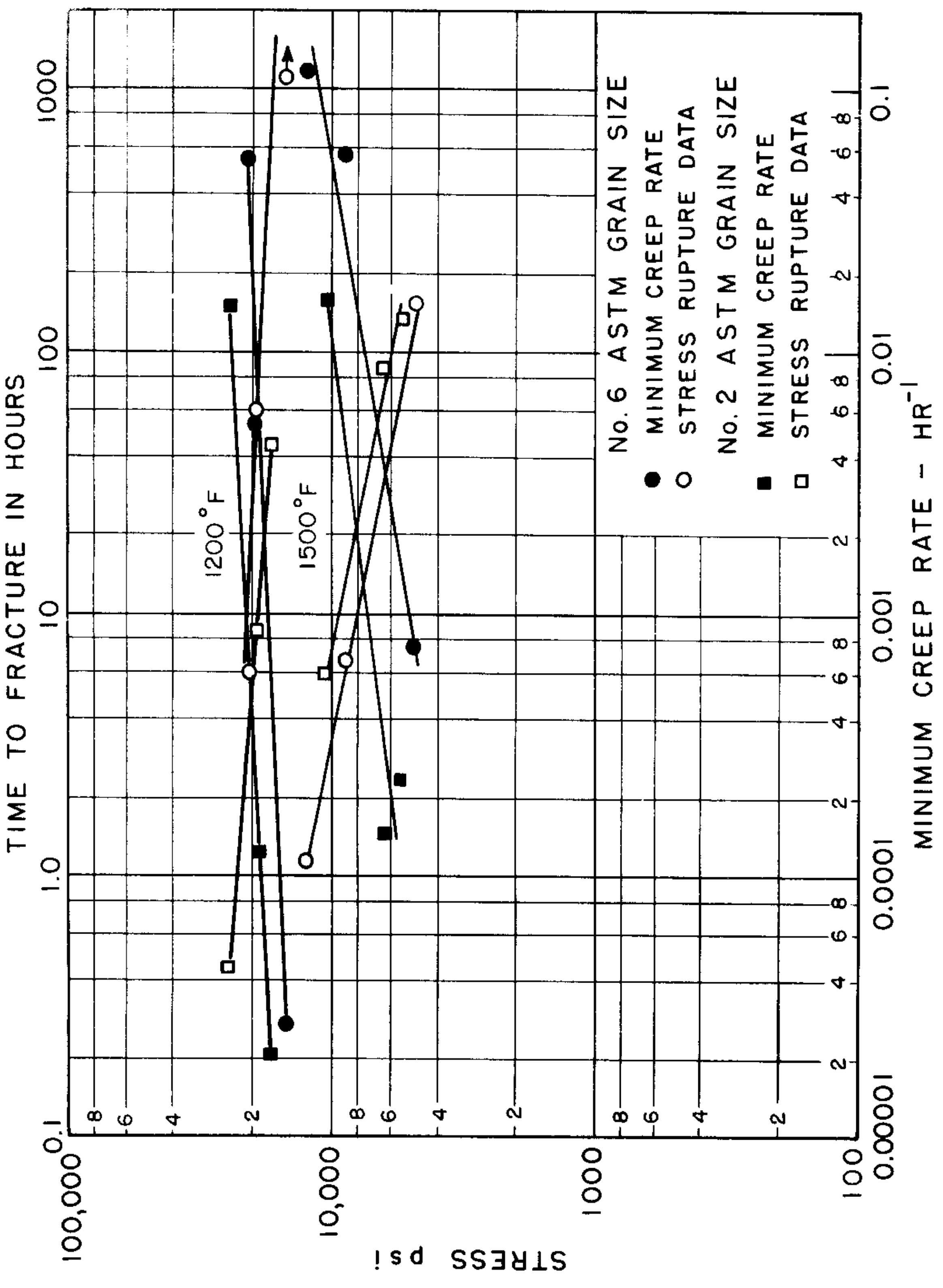
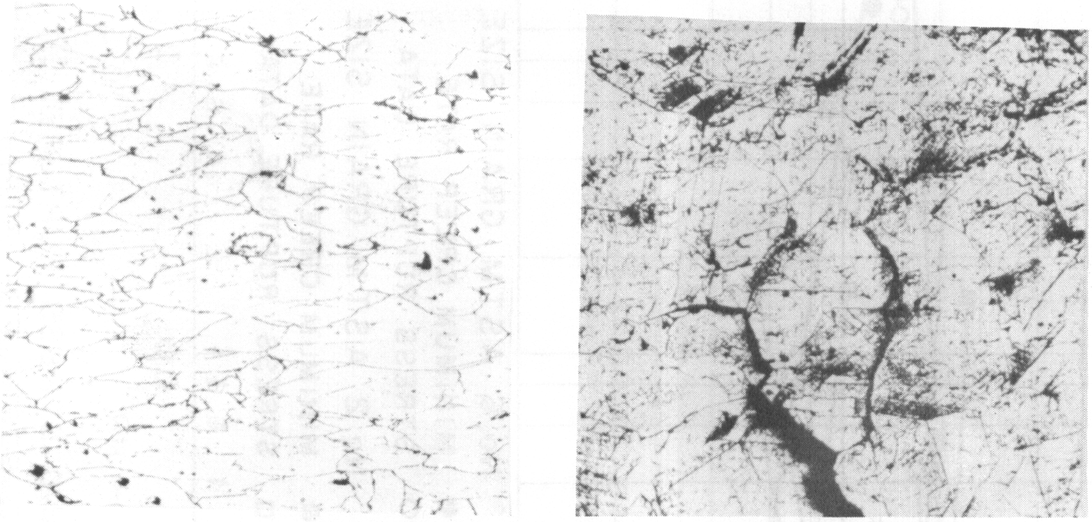
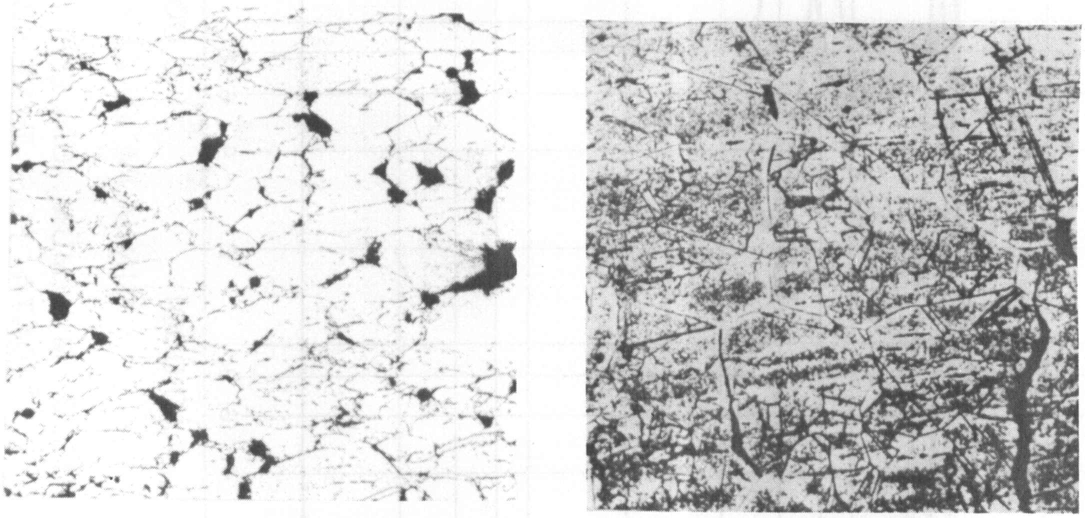


FIG. 9 MINIMUM CREEP RATE AND STRESS RUPTURE DATA FOR TYPE 304 ELC STAINLESS STEEL



No. 6 ASTM Grain Size
Stress 19,200 PSI
Fracture Time 61.2 Hrs.
Test Temperature 1200°F

No. 1 ASTM Grain Size
Stress 17,000 PSI
Fracture Time 44.7 Hrs.
Test Temperature 1200°F.



No. 6 ASTM Grain Size
Stress 8980 PSI
Fracture Time 6.7 Hrs.
Test Temperature 1500°F

No. 1 ASTM Grain Size
Stress 6400 PSI
Fracture Time 88 Hrs.
Test Temperature 1500°F

Figure 10 Photomicrographs of Ruptured Type 304 ELC Stainless Steel Creep Specimens.

microstructural changes that attend creep probably exert a significant effect on the stress-rupture data. This effect is reflected in the fact that the coarse-grained specimens at 1200°F and 1500°F gave intergranular fractures whereas the fine grained specimen fractured in a transcrystalline mode at 1200°F and intergranularly at 1500°F. The two mechanisms of fracturing not only correlated with the amount of grain boundary precipitate after extensive creep but also correlate with the inversion in the grain size effect on the time to rupture at 1200°F as contrasted to 1500°F.

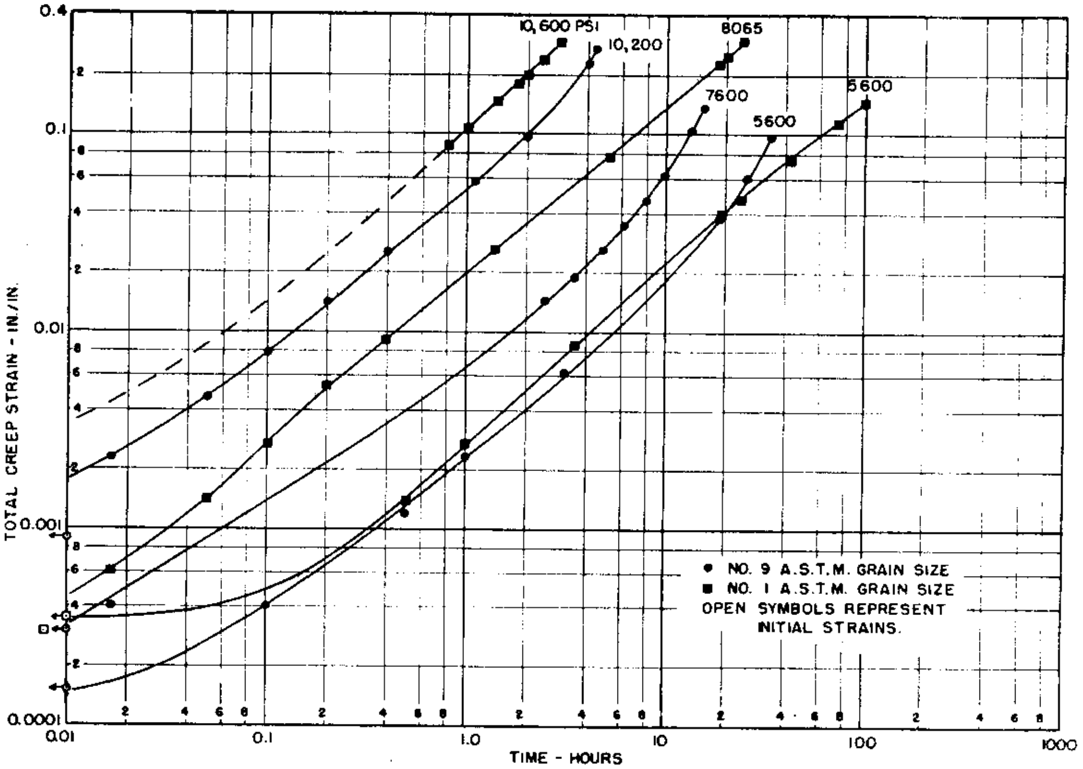
The original creep data for inconel are recorded in Fig. 11. Unlike the case of 18-8 stainless steel, the coarser-grained inconel gives higher initial strains than the finer-grained specimens. As shown in Fig. 12, grain size appears to have little effect on minimum creep rate at either 1200°F or 1500°F though the finer-grained inconel fractures in a shorter period of time at both test temperatures.

Photomicrographs of a series of the ruptured inconel specimens shown in Fig. 13 reveal that almost equivalent amounts of precipitation has taken place in both the fine and coarse grained material. But more extensive precipitation is evident for both grain sizes at the higher testing temperature. It is impossible to conclude, however, that the relatively small and equal amounts of precipitate in the two grain size materials do not obscure the true grain size affect.

The mode of rupturing for the inconel creep specimens appeared to be predominantly transcrystalline, though in some cases the crack may have its origin at an intercrystalline fissure.

FATIGUE TEST RESULTS

Although the coarser-grained Type 304 ELC 18-8 stainless steel exhibited higher creep resistance, its fatigue properties at 1200° and 1500°F were slightly inferior to those of the finer-grained specimen. As shown in Fig. 14, however, the differences in the S-N curves for the fine and coarse grained



TOTAL CREEP STRAIN FOR CONSTANT LOADS AS A FUNCTION OF TIME
INCONEL AT 1200°F.

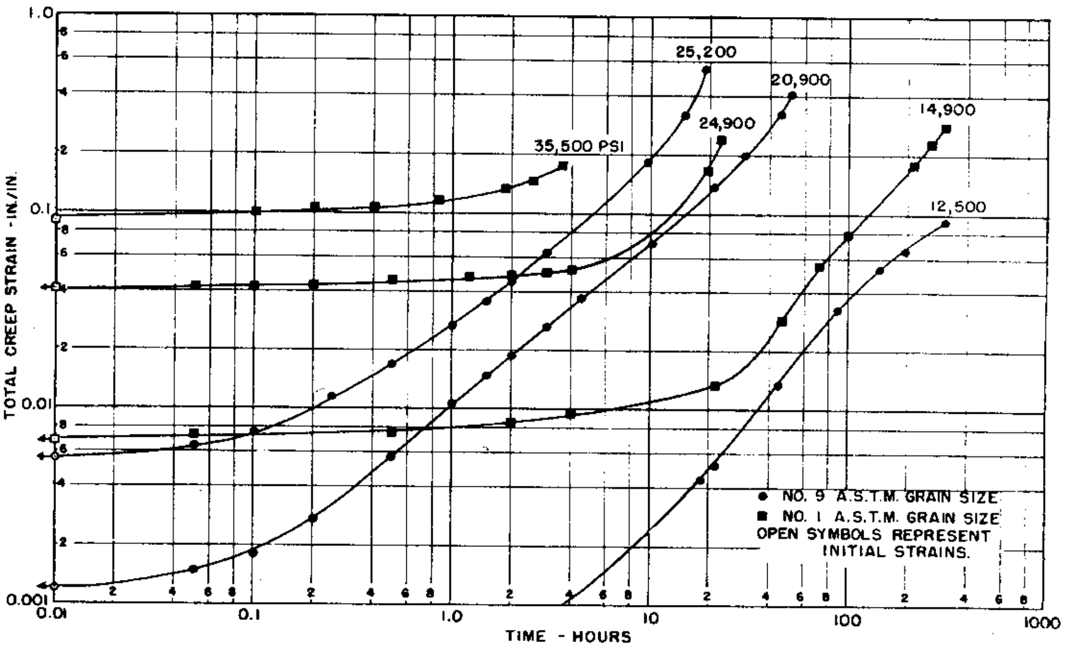


FIG. 11 TOTAL CREEP STRAIN FOR CONSTANT LOADS AS A FUNCTION OF TIME
INCONEL AT 1500°F.

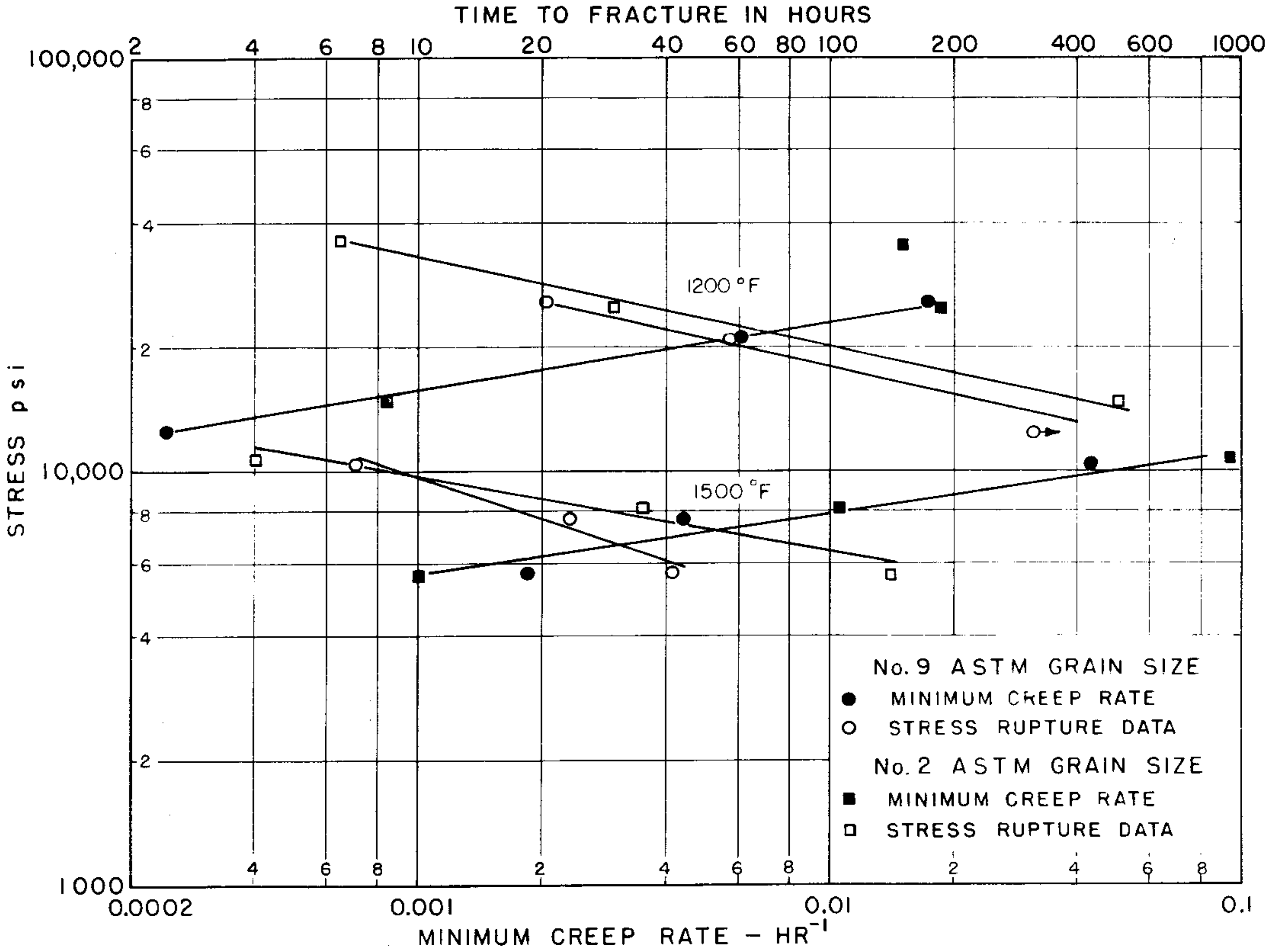
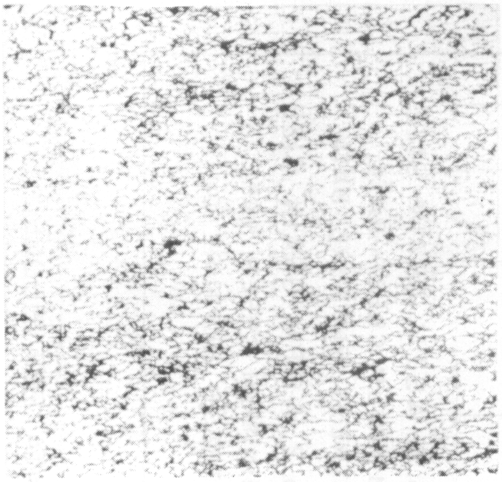
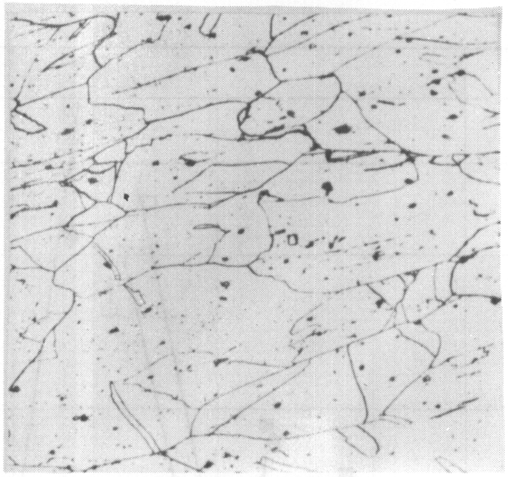


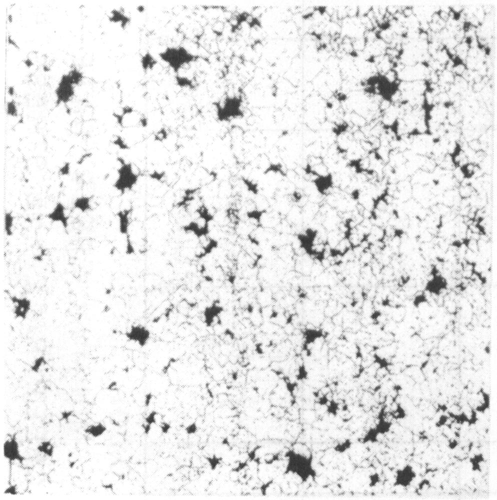
FIG. 12 MINIMUM CREEP RATE AND STRESS RUPTURE DATA FOR INCONEL



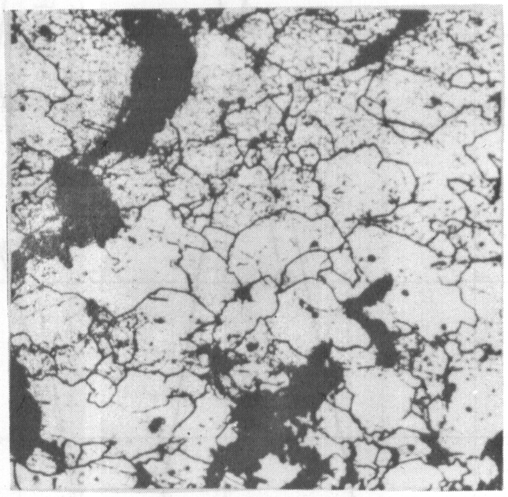
No. 9 ASTM Grain Size
Stress 20,900 PSI
Fracture Time 57 Hrs.
Test Temperature 1200°F.



No. 2 ASTM Grain Size
Stress 24,900 PSI
Fracture Time 30.7 Hrs.
Test Temperature 1200°F.



No. 9 ASTM Grain Size
Stress 7600 PSI
Fracture Time 23.4 Hrs.
Test Temperature 1500°F.



No. 2 ASTM Grain Size
Stress 8065 PSI
Fracture Time 35.00 Hrs.
Test Temperature 1500°F.

Figure 13 Photomicrographs of Ruptured Inconel Creep Specimens.

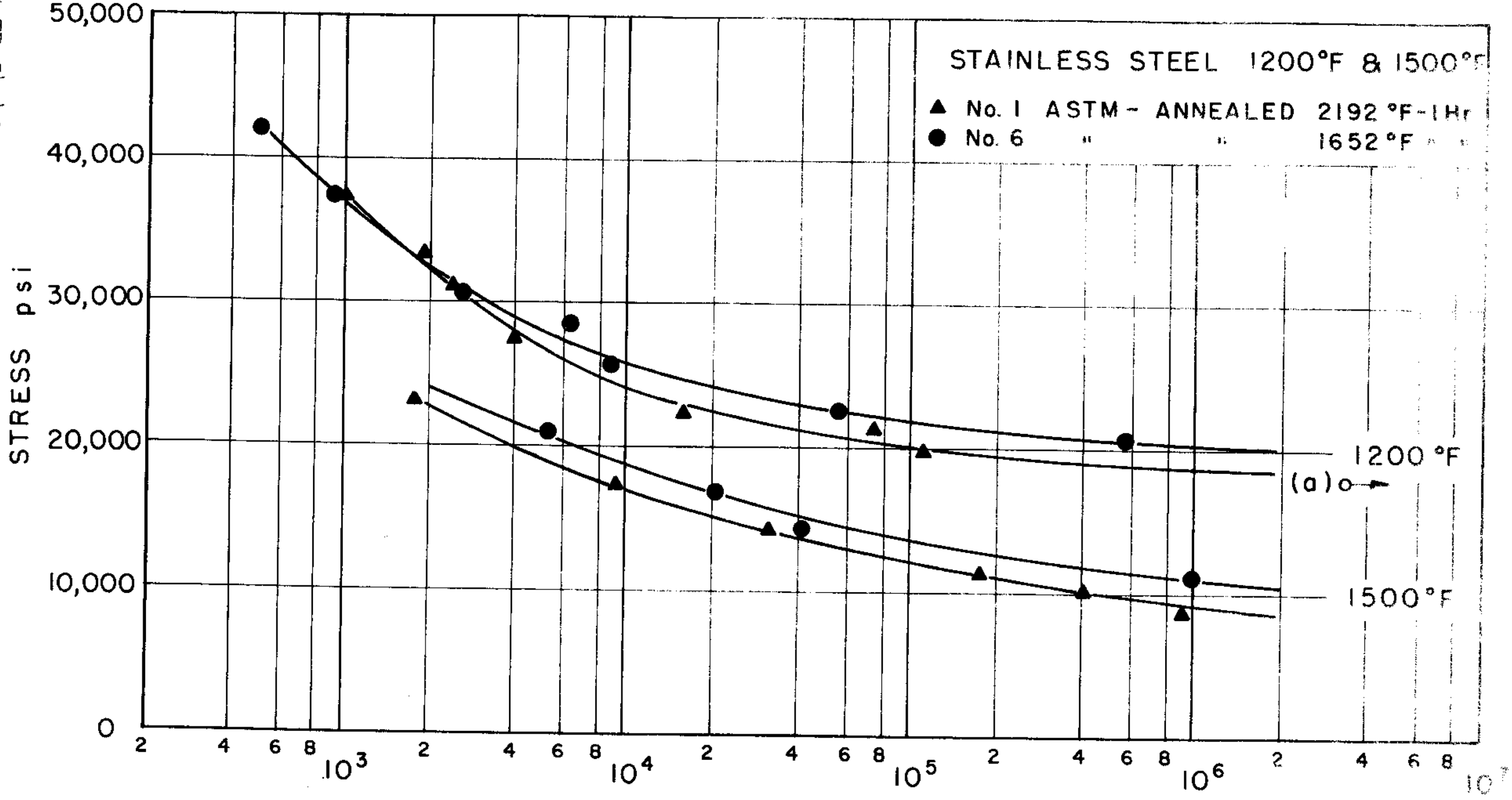


FIG. 14 S-N CURVES OF TWO GRAIN SIZES OF STAINLESS STEEL AT 1200°F & 1500°F ON THE DIRECT STRESS KROUSE FATIGUE MACHINE.

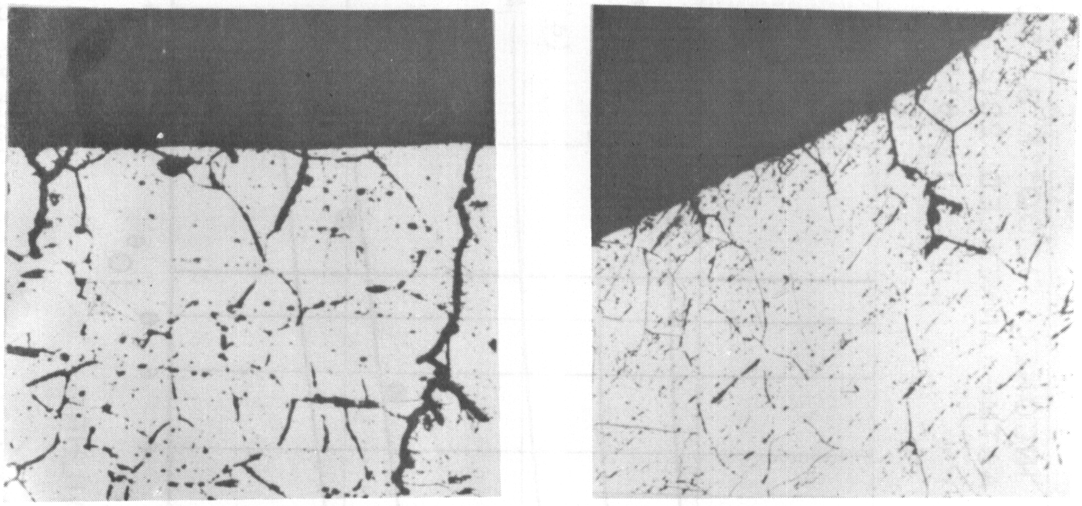
(a) SPECIMEN UNLOADED BEFORE FRACTURE

specimens are negligible.

Since the trends of the S-N curves were almost identical at 1200° and 1500°F, only the microstructures of the failed 1200°F specimens were studied as shown in Fig. 15. No significant differences in the amount of precipitation or the mechanism of crack propagation was detected in the two grain sizes. For both grain sizes, the fracturing was predominantly transcrystalline and noncrystallographic, the cracks being branched and angular. Occasionally the cracks propagated along the grain boundaries. It is significant, however, to note the abnormally high frequency with which a transcrystalline crack crosses the grain boundary at the corners of three adjacent grains. This observation is probably attributable to relaxation along the grain boundaries resulting in a stress concentration at the corners where three grains meet.

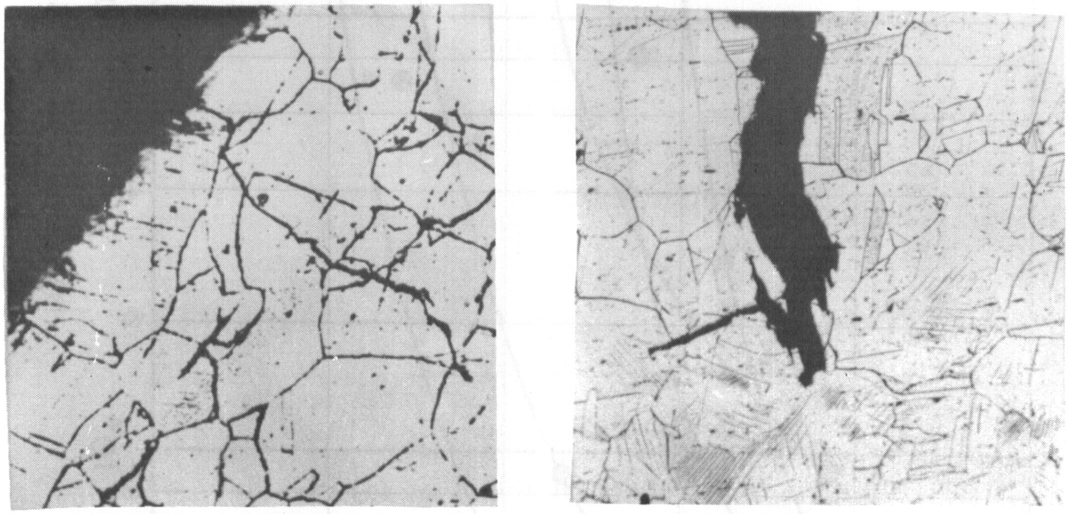
Although the creep resistance of inconel was found to be almost independent of grain size, the fatigue data shown in Fig. 16 reveal that for greater than about $3X10^4$ cycles, the fatigue strength of the finer-grained specimens is superior to that of the coarser-grained material at 1200° and 1500°F. These data also reveal that the fine grained specimen annealed at 1292°F is superior in fatigue strength at 1200°F to the fine grained specimen that was annealed at 1652°F. Thus a difference in annealing treatment induces a difference in the high temperature fatigue properties even when the resulting grain size is the same and the hardness and microstructures following the two anneals are only slightly different.

The photomicrographs of the fractured specimens reveal that the coarser-grain size inconel undergoes extensive crystallographically oriented precipitation during the course of fatiguing whereas the two finer-grained specimens (annealed at 1292° and 1652°F) are almost free from this type of precipitation. It is possible that this difference in precipitation might be associated with the greater plastic strains which were noted in the first few cycles of stressing the coarser-grained material. No major change in microstructure during fatigue of the two



No. 6 ASTM Grain Size
Stress 30,743 PSI
Cycles to Failure 2600
x250

No. 1 ASTM Grain Size
Stress 37,584 PSI
Cycles to Failure 1000
x100



No. 6 ASTM Grain Size
Stress 20,833 PSI
Cycles to Failure 569,000
x250

No. 1 ASTM Grain Size
Stress 21,400 PSI
Cycles to Failure 72,900
x100

Figure 15 Typical Fractures in Fatigue of 304 ELC Stainless Steel at 1200°F.

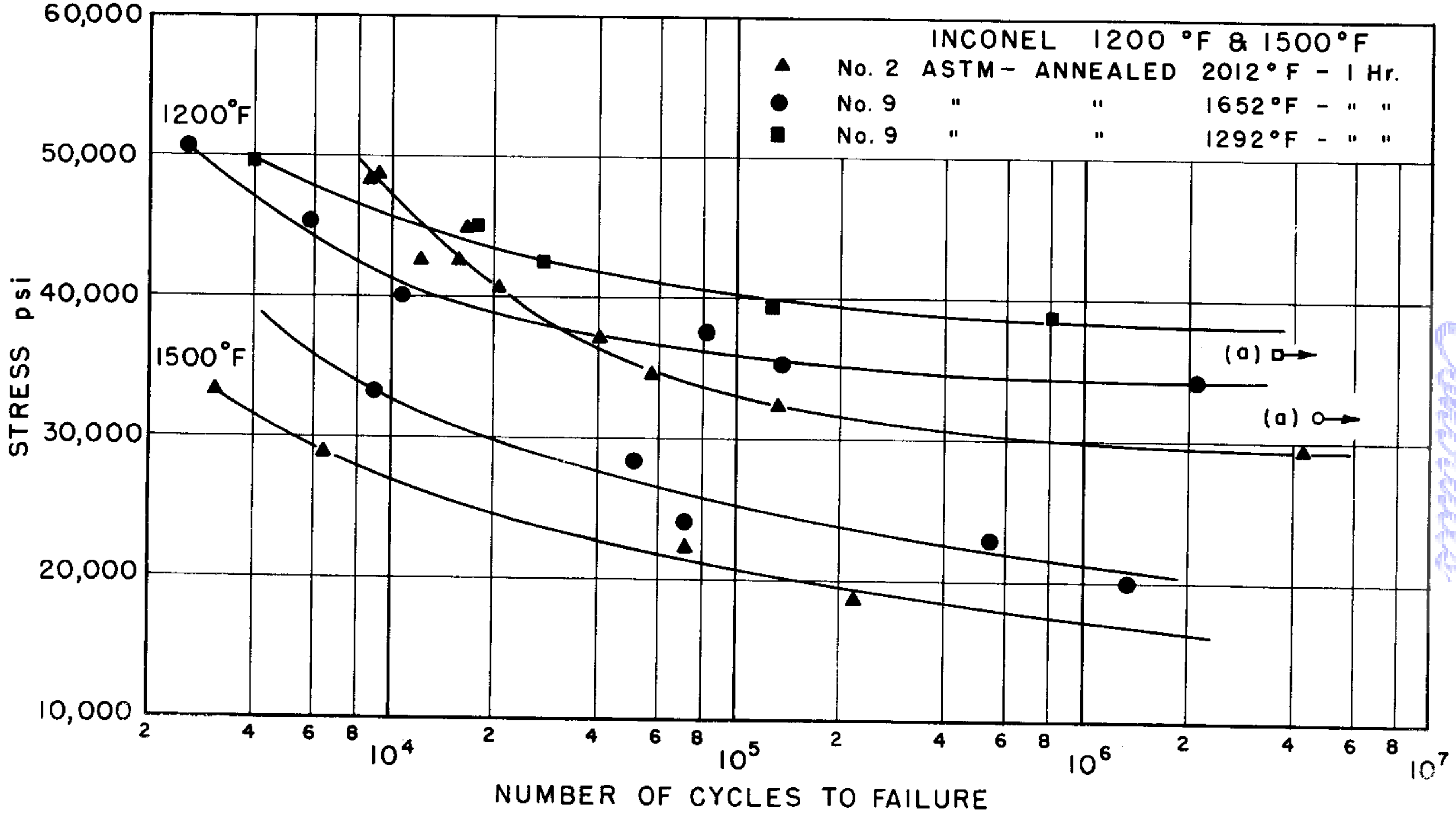
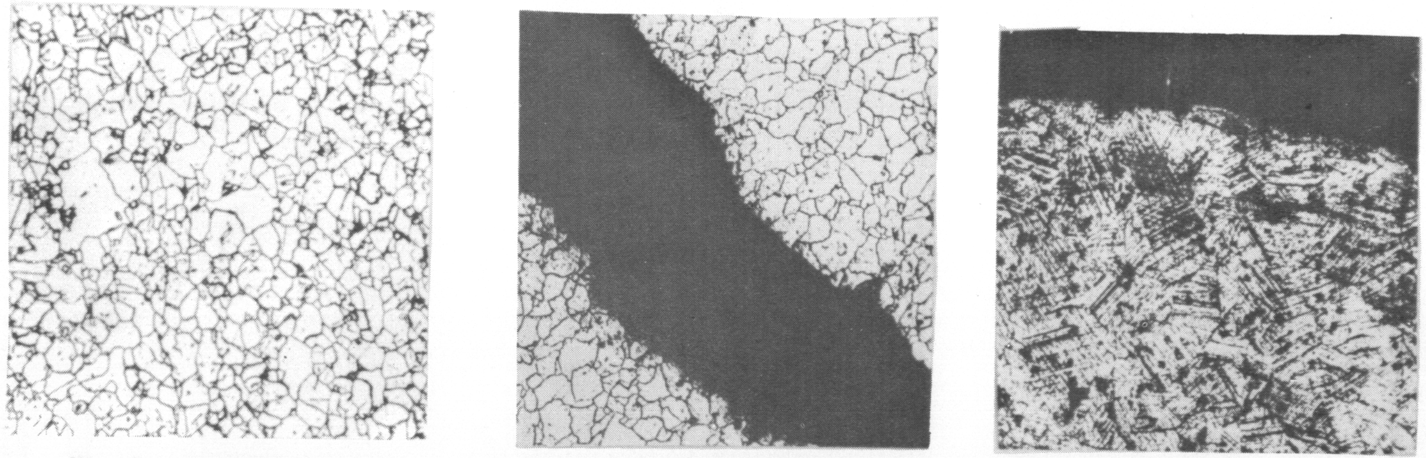


FIG. 16 S-N CURVES OF INCONEL SPECIMENS PERFORMED AT 1200 °F & 1500 °F ON THE DIRECT STRESS KROUSE FATIGUE MACHINE.

(a) SPECIMEN UNLOADED BEFORE FRACTURE.

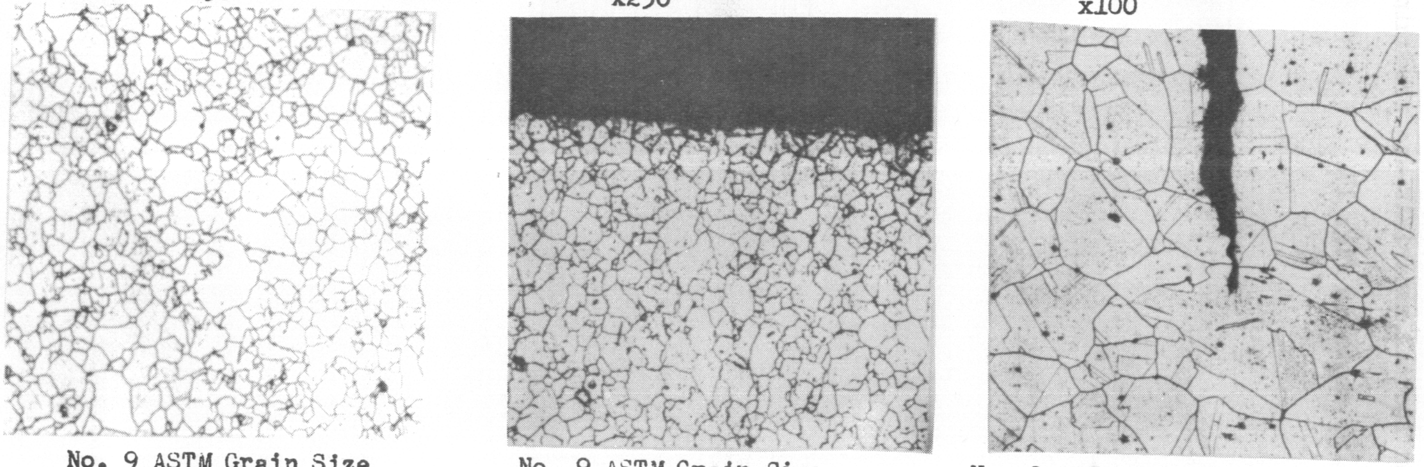
fine grained materials could be detected. Fracturing of the coarse and the two fine grained inconel materials was predominantly transcrystalline at both 1200° and 1500°F, as shown in Fig. 17.



No. 9 ASTM Grain Size
Stress 49,600 PSI
Cycles tc Failure 4000
x250

No. 9 ASTM Grain Size
Stress 50,600 PSI
Cycles to Failure 2600
x250

No. 2 ASTM Grain Size
Stress 48,600 PSI
Cycles to Failure 8700
x100



No. 9 ASTM Grain Size
Stress 38,800 PSI
Cycles to Failure 810,100
x250

No. 9 ASTM Grain Size
Stress 34,050 PSI
Cycles to Failure 2,111,000
x250

No. 2 ASTM Grain Size
Stress 29,800 PSI
Cycles to Failure 4,281,100
x100

Figure 17 Typical Photomicrographs of Fractured Inconel Fatigue Specimens at 1200°F.

CONCLUSIONS

1. The effect of grain size alone on the high temperature creep properties of metals has not yet been clearly delineated:

    a). Many tests, especially those on relatively pure metals, indicate that the creep properties might be relatively insensitive to grain size per se.

    b). In some cases, annealing changes the high temperature creep resistance of metals even in the absence of grain coarsening.

2. The effect of grain size alone on the high temperature fatigue properties of metals has not yet been clearly delineated:

    a). Tests on 18-8 stainless steel reported here suggest that fine grained metals have slightly superior endurance limits to those of coarser-grained metals.

    b). But the data on inconel reported here suggest that annealing alone causes reductions in the high temperature large cycle fatigue strength even when no grain coarsening results.

3. A clear delineation of the effect of grain size on the properties of metals can only be achieved by using metals in which all auxiliary microstructural changes are absent.

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