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EFFECT OF OVERHEATING ON CREEP-RUPTURE PROPERTIES
OF M252 AND INCONEL 700 ALLOYS AT 1500° AND 1600°F

by

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SUMMARY

The prolongation of rupture life of M252 at 1500°F as a result of repeated brief overheats with stress removed to temperatures of 1900°F to 2100°F previously reported was verified for a second heat and extended to rupture tests at 1600°F. The damage from overheats to 1800°F previously observed did not occur in the second heat. The rupture life of Inconel 700 alloy at 1500° or 1600°F was not changed by overheating to 1800°F and was reduced by overheating to 2000° or 2100°F.

The increased rupture life of M252 alloy was due to a reduction in the rate of increase in creep rate with testing time as a result of overheating above 1900°F. For one heat, creep resistance was not changed by overheating to 1800°F and was reduced for the other. The reduction in rupture life of Inconel 700 was due to reduced ductility from overheats to 2000°F or higher. Creep resistance was not changed nor was the ductility changed by overheats to 1800°F. A reduction in ductility for M252 alloy had little effect on rupture time.

The data obtained to date indicate that creep resistance of γ' alloys is either unaffected by overheats or is increased. Increased creep resistance appears to be associated with increasing overheat temperatures. Increased Al+Ti (increased γ') tends to reduce any changes in creep resistance. In general, therefore, these types of alloys are resistant to temperature induced structural changes reducing creep resistance. Actual rupture times for any one material will be dependent on the combined effect of changes in creep resistance and ductility from overheats. The resistance to creep damage is probably due to the rapid reaction rates for γ' solution and precipitation characteristics of γ' alloys.

The γ' alloys are significantly more resistant to creep damage than alloys of the type of S816 and HS-31 which suffer significant reduction in creep resistance from 1 or 2 overheats of 2 minutes duration as previously reported. M252 alloy required 5 to 10 overheats for a significant increase in life. The loss in ductility of Inconel 700 alloy required 3 to 5 overheats.

INTRODUCTION

During service at elevated temperatures, alloys are often exposed to temperatures higher than the design temperatures. An experimental investigation was carried out to provide information on the response of two "nickel base" Al+Ti hardened alloys (M252 and Inconel 700) to overheating as measured by the influence on their creep-rupture properties at 1500° and 1600°F. The overheats studied were 2 minutes in duration and were applied at 5 hour intervals during creep-rupture tests under stresses normally causing rupture in approximately 100 hours. The load on the specimens was removed during the overheats. Temperatures of overheating were in the range of 1800° to 2100°F.

This report is the fourth in a series (refs. 1, 2 and 3) on the influence of overheating on creep-rupture properties of alloys of the type used for blades in aircraft-gas turbines. In reference 3, it was shown that the creep-rupture strength of M252 alloy at 1500°F could be enhanced by overheats of the type studied when the temperatures were 1900°F or higher. The degree of improvement increased with the number of overheats periodically applied. Overheating to 1650° or 1800°F reduced strength. The enhancement of strength by the higher temperatures of overheating was in contrast to other alloys studied, S816 (ref. 1) and HS-31 (ref. 2), which suffered increasing amounts of damage as the temperature and number of overheats were increased.

The research presented in this report was undertaken to verify the enhancement of creep-rupture properties by the higher temperatures of overheating for M252 alloy by investigating another heat. Secondly, another alloy of the same metallurgical type, Inconel 700, was included to determine if the improvement was characteristic of alloys of this type. In addition, the normal base temperature for the creep-rupture tests was extended from 1500°F to 1600°F to include the effect of increasing service temperatures for turbine blades in turbo-jet engines.

Because the present investigation was in the nature of a verification of previously developed concepts, the study was limited to creep-rupture tests which would normally fracture in about 100 hours. This was also partially dictated by the fact that overheating prolonged rupture life and the relatively short normal rupture time was used in order to keep the testing times within reasonable limits for the overheated tests.

Previous investigations had demonstrated fairly well that overheats can have two major effects. The exposure to higher temperatures can induce microstructural changes which affect subsequent creep when the temperature returns to the normal conditions. If stress is present, the accelerated creep will use up creep-rupture life. In most cases, the two effects had been found to be additive. Accelerated-creep damage can be approximated by determining the percentage of the constant temperature rupture life at the overheat temperature and stress represented by the time of overheating. In most cases, the rupture life at the normal test conditions will be reduced by the same percentage. Because this factor appeared to be fairly straight forward, the investigation for this report was limited to tests with the stress removed during the overheats. The results thus provide information on the influence of temperature induced structural changes.

M252 and Inconel 700 alloys depend primarily on the precipitation of a compound based on $\text{Ni}_3(\text{Al}, \text{Ti})$ (hereafter referred to as γ') for strength at elevated temperatures. This strengthening mechanism is different from that for S816 and HS-31 alloys. These latter alloys depend primarily on solid solution of elements such as Mo, W, Cb and C or the precipitation of fine carbides for strength at elevated temperatures. The γ' reaction results in strong precipitation hardening. S816 and HS-31 alloys, however, exhibit only minor hardening as the result of extensive precipitation of carbides. Presumably then, the difference in response of the two types of alloys to temperature induced structural changes depends on a different effect of overheating on the two strengthening mechanisms. The results of the investigation have the major objective of determining if γ' strengthened alloys generally exhibit improved creep-rupture properties from the higher temperatures of overheating.

The conditions of overheating used in this investigation were established by discussion of the problem with the NACA Subcommittee on Heat-Resistant Materials. The research program was conducted by The University of Michigan Research Institute under the sponsorship and with the financial assistance of the NACA.

MATERIAL

Bar stock of M252 and Inconel 700 alloys from production heats was used. The chemical analyses of all materials are given in table I.

Two heats of M252 alloy from previous work (ref. 3) were used in the study. Both were vacuum melted and supplied gratis for the investigation by their manufacturers in the form of centerless ground 7/8-inch diameter bar stock. To improve the uniformity of the material for testing, these bars were rolled to 1/2 inch round-corner square bars using the University of Michigan rolling mill. Rolling temperatures of both 1950° and 2150°F were used. The rolling was done in nine passes with one reheat to the rolling temperature.

The use of two rolling temperatures resulted from the fact that a considerable effect on properties was possible through varied rolling and heat treating conditions. To evaluate this effect, the materials after rolling were given two heat treatments:

- 1) Solution treat 1 hour 2150°F, air cool,
Solution treat 4 hours 1950°F, air cool,
Age 15 hours 1400°F, air cool;
- 2) The same treatment as (1), omitting the 2150°F solution treatment.

The first treatment was established for production use of M252 at the time the investigation for this report was started. The second treatment was current practice at the time the work for reference 3 was done.

The Inconel 700 alloy used was air-melted by the International Nickel Company and provided through the Curtis Wright Corporation, Wright Aeronautical Division, in the form of 11/16 inch round bar stock. At their request, this alloy was not re-rolled prior to testing. It received the standard heat treatment as follows:

- 1) Solution treat 2 hours 2160°F, air cool;
- 2) Age 4 hours 1600°F, air cool.

EXPERIMENTAL TECHNIQUE

Creep Rupture Testing

The creep rupture testing was carried out in conventional beam loaded creep-rupture units using specimens with a 0.250 inch diameter and 1 inch gage length. Time-elongation data were taken during the tests with a modified Martens-type optical extensometer with a sensitivity of 0.00001 inch. For all tests, the samples were placed in furnaces which were at the test temperature and held for four hours before application of the stress. Temperature variation along the gage length was limited to $\pm 3^{\circ}\text{F}$.

Overheat Testing

All overheats were applied using a two-minute exposure to the desired overheat temperature with the load removed from the sample. These cycles were begun five hours following the application of the load and were repeated until the desired number of cycles was accumulated.

Heating Method

For overheat tests, the conventional units were modified to permit resistance heating of the specimens by passing heavy direct current through the sample. A 400-ampere, direct-current generator was used as a power supply. In order to avoid disturbing the specimen during the test, insulated terminal blocks were fastened to the frame of the unit level with the top and bottom of the furnace. From these terminals, short leads were fastened to the top and bottom specimen holders before the test was started. Then, for overheating, it was merely necessary to attach the power supply leads to the terminal blocks, completing the circuit to the generator field switch. The top specimen holder was insulated from the frame by means of a transite insert. The whole circuit was grounded either through the beam or through an attached ground wire. A photograph of a unit is shown as figure 1.

Temperature Measurement

In order to follow the temperature accurately during an overheat, a welding technique (ref. 4) was employed to attach Chromel-Alumel thermocouples to the specimens. An electronic recorder provided a continuous record of the temperature. A schematic sketch of this arrangement is shown as figure 2. Temperature measurement was complicated by two factors. In order to follow the rapidly changing temperatures during an overheat cycle and effect accurate control, the thermocouple wires had to be welded to the sample. This was done with a percussion-type welder. The welded attachment maintained contact between the thermocouple wires and the specimen as reduction in cross section occurred by creep during the tests. In welding the thermocouple wires on the specimen, however, any minute error in positioning either wire caused the direct current from the generator to impress an electromotive force on the thermocouple circuit. This electromotive force varied with the magnitude of the placement error and appeared on the temperature recorder as a temperature effect. To avoid this, two Alumel wires were employed, one deliberately placed on either side of the single Chromel wire. By connecting these two Alumel wires to the extremes of a variable resistance, the variable tap could be adjusted so that the two electromotive forces obtained cancelled each other, leaving only the thermal electromotive force impressed on the recorder.

Checks were made of the original calibration and the maintenance of calibration of the thermocouples. The system used gave accurate temperature measurements as installed. The overheats did not change the calibration of the thermocouples by more than 1°F for any of the temperatures used.

Procedure for Overheat Cycles

After completion of the five-hour period under stress following load application, the following procedure was followed in performing an overheat:

1. The temperature was checked and an elongation reading made. At this time the generator and recorder were attached to the specimen.

2. The load was removed.

3. After a 60 second time lapse during which the furnace input was cut back and the thermocouple circuit checked, the heating cycle was initiated by applying the maximum generator output of 400 amperes to the specimen. When the desired overheat temperature was attained, the generator output was reduced to a value just sufficient to maintain temperature.

4. At the end of the two minutes at temperature, the power supply was shut off and the specimen allowed to cool. No forced cooling was employed other than that supplied by having allowed the furnace temperature to fall below the base temperature when the input was reduced in step (3).

5. The load was reapplied when the test temperature fell to within 10°F of the base temperature. Although the asymptotic approach of the test temperature to the level of the base temperature introduced some variation in the length of time required to re-establish this level, the samples cooled to 10°F above this level in a reproducible time period. After the load was re-applied, the furnace control was manipulated to bring the test on temperature as soon as possible.

6. When temperature equilibrium was re-established at the base temperature, elongation measurements were taken again and the test continued for five hours until the next cycle. In plotting the time-elongation data, this reading after reapplication of the load was assumed to be at the same total deformation as the reading taken just prior to removal of the load at the beginning of the cycle.

This procedure resulted in reproducible temperature patterns from cycle to cycle and between samples or creep-rupture units. Typical time-temperature patterns for overheats to several of the temperatures used are shown in figure 3.

Metallography

Metallographic samples were mechanically polished through wet papers to 600 grit and then on wet cloths with Linde A and B powders. Etching followed the procedure developed by Bigelow, Amy and Brockway

(ref. 5) using an etchant composed of 12 parts phosphoric acid (85 percent), 47 parts sulfuric acid (96 percent) and 41 parts nitric acid (70 percent). Etching was electrolytically at 6 volts and a current density of about 0.8 amperes per square inch for 5 to 7 seconds.

RESULTS

Normal creep-rupture properties were established out to 100 hours for both the M252 and Inconel 700 experimental materials at 1500° and 1600°F. Some data for M252 were also included from reference 3. In determining the properties of the M252 stock, additional data supplementing that in reference 3 on the influence of hot-working and heat treatment conditions were obtained.

The influence of periodic overheating from 1500° and 1600°F to 1800°, 2000° and 2100°F was established for heat 837 of M252 alloy under the stress causing rupture in 100 hours. Limited tests were conducted on heat HT-28 for overheats from 1600° to 1800° and 2000°F. Data for this same heat for overheats from 1500° to 1650°, 1800°, 1900° and 2000°F are included from reference 3. Overheats on the Inconel 700 stock covered the range of 1800° to 2200°F from base temperatures of 1500° and 1600°F under the stress for rupture in 100 hours.

The analysis of the data is limited to the interrelation of creep resistance and ductility to the rupture time as influenced by overheating. An extensive metallurgical investigation to determine the basic physical metallurgy factors will be the subject of a later report.

M252 Alloy

The base-line normal creep-rupture properties at 1500° and 1600°F will be presented first. The data from the overheat tests will then be presented in terms of changes induced.

Normal Creep-Rupture Properties

The rupture-test data are given in table II and are shown graphically in figure 4. The base conditions selected for overheat tests were as follows:

| <u>Heat</u> | <u>Temperature (°F)</u> | <u>Stress (psi)</u> | <u>Rupture Time (hours)</u> |
|-------------|-----------------------------|-------------------------|---------------------------------|
| 837 | 1600 | 18,000 | 72-79 |
| HT-28 | 1600 | 20,000 | (109-112)* |
| 837 | 1500 | 33,000 | 68-86 |
| HT-28 | 1500 | 34,000 | 65-105 |

* Based on two tests

In each case, the stress was originally selected on the basis of limited testing to cause rupture in 100 hours. After the stress was selected, more extensive testing established the tabulated range in rupture times. The range for heat 837 at 1500°F was based on the scatter indicated by five tests at four different stresses (fig. 4). For 1600°F, six tests at the base stress of 18,000 psi gave the range indicated. The range for heat HT-28 at 1500°F was based on 10 tests (fig. 4) ranging in rupture time from 71 to 736 hours. Only two tests were conducted at 1600°F because the stock remaining from the work for reference 3 was only sufficient to provide four specimens.

The stock used from heat 837 was rolled from 2150°F and heat treated as follows:

- (a) Heated 1 hour at 2150°F and air cooled;
- (b) Heated 4 hours at 1950°F and air cooled; and
- (c) Heated 15 hours at 1400°F and air cooled.

Comparative data from reference 3 are included in table II and figure 4 for the same heat rolled from 1950°F and solution treated only at 1950°F prior to the age at 1400°F. The change in procedure raised the stress for rupture in 100 hours at 1500°F from 27,000 to 31,000 psi. Apparently the major factor causing the difference was the 2150°F solution treatment as is indicated by the following tabulation of rupture-test results for tests from this heat at 1500°F and 34,000 psi:

| <u>Rolling Temperature (°F)</u> | <u>Solution Treatment</u> | <u>Rupture Time (hours)</u> |
|---|------------------------------------|-------------------------------------|
| 1950 | 4 hr. at 1950°F | 38.2, 34.2 |
| 2150 | 4 hr. at 1950°F | 30.0 |
| 1950 | 1 hr. at 2150°F + 4 hrs. at 1950°F | 66.6 |
| 2150 | 1 hr. at 2150°F + 4 hrs. at 1950°F | 60.1 |

Note: All aged 15 hours at 1400°F after solution treatment.

When the research for reference 3 was conducted, the established heat treatment for M252 alloy was 4 hours at 1950°F followed by the 15 hour age at 1400°F. Under these conditions of heat treatment, it was shown in reference 3 that the rolling temperature did have an appreciable effect on rupture strength for heat HT-28 as is indicated by reproducing the data in figure 4b. Possibly the limited testing on heat 837 failed to properly define the effect of rolling temperature with the single treatment.

At the time the research covered by the present report was undertaken, the established heat treatment had been changed to 1 hour at 2150°F, air cooled plus 4 hours at 1950°F, air cooled prior to the age at 1400°F. Consequently, all material studied was given this heat treatment. This includes the limited testing conducted on heat HT-28.

The elongation and reduction of area values for all tests (table II) were high and within the expected degree of reproducibility.

Creep curves for those normal tests conducted at the stresses used for the overheat tests are shown in figures 5a, 6, 7 and 8. In all cases, the creep rate increased from the start of the tests. The following additional comments apply to the creep data:

1. Curves were available for heat 837 at 1600°F for seven tests. These curves indicate a high degree of reproducibility.

2. Only two creep curves at the base condition are available for heat 837 at 1500°F.

3. Only one curve was available (ref. 3) for heat HT-28 at 1500°F. Two tests were conducted at the base condition for 1600°F.

4. The curves show that the large fraction (1/3 to 1/2) of the total elongation accumulated during the last few hours of the tests was not plotted. There are a few tests, usually tests interrupted for structural study, in which the creep curves are additionally abbreviated.

A number of bar stock samples and ruptured specimens were subjected to metallographic examination and found to have a uniform grain size of ASTM No. 6-8. No significant variation in carbides was observed. Typical microstructures before and after testing at 1600°F are shown by figure 9.

Influence of Overheating on Rupture-Test Properties

The experimental results indicated that the rupture life of M252 alloy was prolonged by periodic overheats to temperatures of 2000° or 2100°F from a base temperature of 1600°F. Previously, it had been shown in reference 3 that the same effect resulted for overheats to 1900°F and higher temperatures from a base temperature of 1500°F. Overheats to 1800°F apparently did not alter the strength of heat 837 while strength was somewhat reduced for heat HT-28. In all cases, it was necessary to apply from 5 to 10 overheats before the rupture time was significantly changed. The creep data indicated that the periodic application of the 2 minute overheats reduced the rate of increase of creep rate with time at the base temperatures when overheats were 1900°F or higher. When the overheat temperature was 1800°F, they either did not change creep rate or resulted in a somewhat faster rate of increase in creep rate.

The detailed data on which this general pattern of behavior is based are presented in the following sections.

Rupture life. - The results of the overheat tests (table II and fig. 10) showed the following:

1. Overheating heat 837 to 1800°F from either 1500° or 1600°F did not significantly change rupture time. It caused the rupture times for heat HT-28 to be on the low side or slightly below the range for normal tests. Overheating heat HT-28 from 1500°F to 1650°F resulted in the same effect as the overheats to 1800°F.

2. Overheating to 2000°F increased the rupture time for both heats for normal test temperatures of either 1500° or 1600°F. Overheating to 2100°F for heat 837 resulted in a slightly greater improvement in life than 2000°F overheats. The data from reference 3 for heat HT-28 showed increased life from overheats to 1900° or 2000°F. The extent of this increase was slightly less for 1900° than for 2000°F.

3. Another way of appraising the results is to recognize that from 5 to 10 overheats of 2 minutes duration had no significant effect on rupture time regardless of the overheat conditions. From 5 to 10 overheats to temperatures above 1800°F started to increase strength and if the overheats were continued to rupture, substantial increases in rupture time resulted. When the overheat temperature was 1800°F, a heat-to-heat difference was observed which showed some damage from repeated overheating to rupture in the case of heat HT-28 and no significant change for heat 837.

In conducting the research, data from reference 3 was mainly used to show the effects of overheating from 1500°F. In the present investigation, the major emphasis was placed on overheating from 1600°F. Only survey tests were made to obtain an indication if the same behavior was characteristic of the two heats involved and whether the behavior of the material was similar for both heat treatments. The two tests on heat HT-28 overheated from 1600°F and the 5 tests on heat 837 overheated from 1500°F gave the same general type of results.

Ductility. - The elongation and reduction of area were generally reduced by the overheats applied to the specimens (table II and figure 11). Overheating to 1650° or 1800°F was generally more damaging to ductility than the higher temperatures. As was the case for rupture time, several overheats apparently were necessary to produce a significant effect. The ductility of heat 837 overheated from 1600°F appeared to be less affected by more than five to ten overheats than when this heat was overheated from 1500°F or when heat HT-28 was overheated from either temperature. Overheating to 1800°F about 10 times reduced elongation to 20 or 30 percent. Overheats to higher temperatures applied repeatedly until rupture occurred, reduced ductility below 20 percent, except for heat 837 overheated from 1600°F.

Creep resistance. - Normal creep curves for the tests at either 1500° or 1600°F (figs. 5 to 8) showed that creep rate increased continuously from the start of the tests. Overheating to 1900° to 2100°F reduced the rate of increase in creep rate, whereas overheating to 1650° or 1800°F accelerated creep for heat HT-28 and overheats to 1800°F had little effect on heat 837. These summarized effects are shown in detail by the data, as follows:

1. The curves for tests for heat 837 overheated until rupture occurred for overheats from 1600°F to 1800°, 2000° and 2100°F (fig. 5b) show the overall influence on rate of creep. Overheating to 1800°F had little effect, whereas 2000° and 2100°F had increasing ability to reduce the rate at which creep increased. This pattern of effect is shown for all the tests at each overheat temperature by figures 5c, 5d and 5e.

2. The same general pattern resulted for the creep behavior of heat 837 overheated from 1500°F (fig. 6).

3. The curve for heat HT-28 overheated from 1600°F to 1800°F (fig. 7) showed a possible slight acceleration of creep, whereas overheats to 2000°F definitely retarded the creep. Overheating from 1500°F (fig. 8) to 1650° or 1800°F caused definite acceleration of creep while overheats to 1900° or 2000°F with this base temperature reduced the rate of creep.

4. In those cases where overheating was stopped before rupture occurred, creep continued at the base temperature following the last overheat with no sudden change in rate. Creep gradually accelerated until it achieved a rate approximating that of a normal test at the same amount of creep deformation. This indicates that the overheats had a repeated beneficial effect when the overheat temperatures were above 1900°F or repeated damaging effect when 1800°F reduced rupture life.

Discussion

The rupture life can be considered to be the combined effects of creep resistance and ductility. A number of ways to develop a correlation based on the influence of these two factors to show the effects of overheating were attempted without success.

Inspection of the data shows that the change in creep resistance induced by overheating was in the direction of the change in rupture time. Because creep rates continuously increased with testing time and because the creep, after overheating was stopped, approached the rates shown by normal tests at the same total creep, no way could be found to express the creep resistance changes from overheating. Accordingly, the presentation of this factor has been left qualitative by showing the comparative creep curves of figures 5 to 8.

No correlation with change in ductility was found. The main reason for this involves the relatively small proportion of the total rupture time represented by the time for creep to increase the elongation from 20 or 30 percent to the normal test values of approximately 45 percent. This was so short that the reduction in ductility had very little effect on the overall rupture time. This condition would be characteristic of creep curves with rapidly increasing creep rates towards the end of tests, as was the case for M252 alloy in this investigation. The reduced elongation did slightly reduce potential rupture time in comparison to normal tests. This, however, was insignificant in comparison to the effect on creep resistance.

Two heats involving two conditions of heat treatment were investigated. The general pattern of behavior was the same for both. A heat-to-heat difference was exhibited in that overheating to 1800°F was damaging for one heat and had no effect for the other. It appears then that M252 alloy can be expected to withstand overheats of the type investigated to temperatures of 1900°F or higher without damage and probably improvement. At lower temperatures, the effect appears to vary between heats. In either case, however, repeated overheating is necessary to significantly change properties.

The basic cause for the effects of overheating will require a detailed study of the microstructure with emphasis on the state of the γ' precipitate.

Inconel 700 Alloy

The results for this material were considerably more complex than for the M252 alloy. The normal properties will be presented first, followed by the effects of overheating.

Normal Creep-Rupture Properties

Several tests at varying stresses were originally conducted to establish stress-rupture time curves at 1500° and 1600°F. The results (table III) and figure 12 indicated that the tests to be overheated should be conducted at the following stresses in order to approximate a 100-hour rupture time:

| | |
|--------|------------|
| 1500°F | 43,000 psi |
| 1600°F | 29,000 psi |

As the tests on Inconel 700 progressed, it became evident that a considerable variability in basic creep-rupture properties not disclosed by the original tests was involved. Further investigation and analysis of the data indicated that differences between the individual original bars was the major source of variation. It became evident that the only significant way to express the base rupture time was in terms of the results of actual tests on each bar. This was not recognized until the tests at 1500°F had been completed. However, it was recognized in time so that in most cases normal rupture tests could be conducted on the same individual bar used as a source of specimens for the tests overheated from 1600°F.

Accordingly, the following procedure has been used to define the basic rupture properties:

1. For tests at 1600°F, the results of overheated tests are compared directly with the results of normal tests on specimens from the same bar. All data are included in table III on the basis of the bar number from which the specimens were taken.

2. At 1500°F, no normal test data were available on specimens from the same bar used as a source of specimens for overheat tests. Data were available for three bars at both 1500° and 1600°F. Using these data, an estimated value was established (table III and fig. 12) for the bars used as a source of specimens for the tests which were overheated from 1500°F. The data for tests at both 1500° and 1600°F were plotted as a function of temperature. It was then assumed that the material with the shortest and longest rupture times at 1600°F could be extrapolated to 1500°F by parallel curves to establish the

range at 1500°F. The actual range in rupture time observed for tests at 1600°F and 29,000 psi is included in figure 12.

It should be noted that the data in table III show that four replicate tests on specimens from bar 50 and from bar 51 show a high degree of reproducibility within each bar. In addition, two specimens from bar 19 showed good agreement. This is direct evidence that the specimens from each bar had a high degree of reproducibility. As will be shown later, correlation of the data from tests which had been overheated support the validity of the contention that in most cases all specimens from an individual bar had similar properties.

There was considerable variation in ductility (table III) of the specimens rupture tested at 1600°F. In general, those specimens exhibiting the longest rupture times had the highest ductilities. There was a fair correlation (figure 13) with a few exceptions between rupture time and elongation. Those specimens with short rupture times for their elongation exhibited third stage creep earlier in the tests than the majority of the specimens. Replicate specimens from the same bar had similar ductilities, although there were marked differences between bars. Insufficient data were available from replicate tests at 1500°F to provide a check of this type on the behavior at the lower testing temperature.

The creep curves for the rupture tests at 1600°F and 29,000 psi are shown in figure 14a. The material showed a brief period of decreasing creep rate with testing time followed by a period of nearly constant creep rate. The creep rate then increased quite rapidly leading to rupture. It was evident from figure 14a that a major difference between tests was the time period at which the creep rate increased. The particularly early and rapid third stage creep of bar 50 is evident in this figure.

In plotting the creep curves for figure 14a, the complete curves to rupture are shown in so far as possible. This shows that the rupture time and ductility both increased with the time for the creep rate to increase. The terminal points of the creep curves are the same as those on figure 13.

The minimum creep rates for most of the tests were in reasonably close agreement. It, therefore, seems evident from the data that

the material investigated had quite uniform creep resistance during the early portions of the tests. The major cause of variability in rupture time was the time required for the onset of rapid creep. The variability in rupture time was compounded by the elongation to fracture being lower when the time for the onset of rapid creep was less.

The limited testing done at 1500°F and 43,000 psi prevents a detailed comparison of the creep data for this base temperature. The available data (fig. 15) indicate that the scatter in properties between bars was equally as severe at this temperature as it was at 1600°F. The trends in these tests are the same as those observed for the more detailed results at 1600°F.

The cause of the variable creep-rupture properties was not completely determined. Variability in history prior to final heat treatment was almost certainly the primary cause. It has been recognized in this laboratory for some time that variations in temperature and degree of reduction prior to final heat treatment can have a pronounced effect on the properties of alloys of the type being investigated. It is also known that the influence of prior history is to some degree a function of the conditions of final heat treatment, as previously discussed for M252 alloy.

Evidence that prior working and/or heat treatment conditions caused variability in properties was found in the microstructure. There were differences in grain size within individual specimens (figure 16), the ASTM grain size ranging from 1-2 to as small as 6-7 or finer. Those specimens exhibiting the longer rupture times had more coarse grains than those with the shorter rupture times (figure 16). Such structural variations are characteristic of the prior history variations discussed. No other variations in structure were detected.

Since the variability in the Inconel 700 stock was recognized, highly qualified individuals have confirmed that it could be expected in production material with the heat treatment used. When the investigation of Inconel 700 alloy was originally undertaken, it was requested that production-produced and heat treated material be used. The stock was donated to the program and heat treated in accordance with generally accepted conditions. As the data indicate, variability from this source would be expected to be less within the individual two-foot long bars than between bars.

Influence of Overheating on Rupture-Test Properties

The study of the effect of overheating Inconel 700 alloy was undertaken primarily to determine if lack of damage or improved rupture life found for M252 alloy was characteristic of Ti+Al bearing alloys dependent on γ' precipitation. As previously discussed, the Inconel 700 stock used varied considerably in its creep rupture properties. Accordingly, the results of the overheat tests are evaluated in terms of the normal rupture properties of the individual bars from which the specimens were made.

Rupture life. - The results of tests overheated to 1800°F (table III and fig. 17) from either 1500°F or 1600°F under the conditions studied did not show a significant effect on rupture life. The only exception to this was the specimen overheated 14 times from 1600°F, suggesting the possibility that a large number of overheats would reduce the rupture life.

When the overheat temperature was increased to 2000°F (table III and fig. 17) all tests indicated reduced rupture life. The test on the specimen from bar 13, for which there were no normal tests at 1600°F, gave an intermediate rupture time which did not conflict with the general trend of the other data. The limited data suggested more reduction in rupture life at a base temperature of 1500°F for a given number of overheats than for similar overheating from the 1600°F base temperature.

When the temperature of overheating was raised to 2100°F, the results were somewhat conflicting (table III and fig. 17). Rupture life was reduced in all cases except for the two tests on specimens from bar 23. In general, those specimens which exhibited a loss in rupture life indicated about the same degree of loss as did the tests for overheating to 2000°F.

One specimen (table III and fig. 17a) overheated to 2200°F from 1600°F also exhibited about the same degree of loss in rupture life as those overheated to 2000°F or 2100°F.

Two features of the results require further discussion. Even though the stock exhibited very erratic basic rupture properties under the test conditions, it seems evident that the influence of overheating on the rupture life of Inconel 700 alloy was established. Overheating to 1800°F did not significantly alter rupture life. Higher temperatures of overheating would reduce rupture life. The number of over-

heats to cause a significant effect under the conditions studied is not clearly established. Most of the data indicate that 3 or 5 overheats would significantly reduce the life, with the reduction in life increasing with the number of overheats. The significance of the one exception, bar 23 overheated to 2100°F from 1600°F, has not been determined. No explanation was found in metallurgical studies. The safest assumption is that there was variation of rupture properties within bar 23 so that the normal test gave an abnormally low rupture time. In view of the lack of an adequate explanation, it must be considered, however, that there is the possibility that certain prior treatments of Inconel 700 alloy could develop structures which are not damaged by the overheat conditions studied.

Ductility. - The elongation values for the overheated specimens (table III and figure 18) showed that overheats to 2000°F or higher reduced ductility in nearly every case. Overheats to 1800°F had no significant effect. The tests for bar 23 did not show the decrease in ductility suggested by other tests for these conditions. As discussed previously, this is probably the result of variation between samples from this bar. The limited data for a base temperature of 1500°F did not show a significant difference from that for 1600°F, although the elongation for the tests at 1500°F was lower.

The elongations for the specimens overheated from 1600°F are plotted in figure 13 against the rupture time together with the basic rupture data. These data extend the curve to lower elongations although there is some overlap at intermediate ductilities. This correlation suggests that the ductility is a major factor in controlling rupture life, regardless of the causes for the ductility changes.

The influence of overheating from 1500°F on ductility was not very well established due to the limited data for normal tests at 1500°F. In spite of this, the general pattern was similar to that at 1600°F. There is, therefore, little doubt that the trends established are real. The number of overheats required to significantly reduce ductility for the higher overheat temperatures is the same as was previously discussed for changes in rupture time.

Creep resistance. - Comparison of the creep curves for the overheat tests with those for the basic rupture tests at 1600°F (figs. 14b to 14j) shows no significant change in creep resistance from overheating up to the point when rupture occurred. Due to the lack of creep curves for specimens from the same bar as those used for overheat tests for a base temperature of 1500°F (fig. 15), the effects are not so clear as for 1600°F. However, the available data do not indicate any large effect on creep resistance.

The only exception noted (fig. 14g) was the creep resistance of samples 23A and 23D tested at 1600°F. This difference in creep resistance between the overheated samples and the normal rupture test from this bar have been previously suggested to stem from between-sample variability in this bar.

Discussion

In the case of Inconel 700 alloy, repeated brief overheats did not change creep resistance. Ductility was either unaffected (overheats to 1800°F) or reduced (higher temperature overheats). The changes in ductility controlled changes in rupture life. The creep rates were relatively low (in comparison to M252 alloy previously discussed) and the change in ductility represented a significant proportion of the rupture life.

The influence on ductility was induced by a relatively small number of overheat cycles. Consequently, a few overheats of 2 minutes duration could significantly reduce the rupture life of Inconel 700 alloy. This is in contrast to M252 alloy for which a considerable number of overheats was necessary to noticeably change rupture life.

It is unfortunate that the variability of the test stock caused difficulty in interpreting the results. The general reduction in rupture times from normal tests within each bar for overheats to the higher temperatures seems adequate to demonstrate a damage effect.

Generality of Results

The two alloys studied in this investigation depend primarily on the precipitation $\text{Ni}_3(\text{Al}, \text{Ti})$ type compounds for strength at elevated temperatures. In so far as creep-rupture properties are concerned, however, they responded differently to overheating from 1500° or 1600°F with stress removed during the periodic overheats. The thermally induced structural changes for the two alloys may be summarized as follows:

M252 Alloy - Overheats to temperatures up to about 1800°F either reduced rupture strength or had little effect, mainly through corresponding effects on the creep resistance. Increasing the temperature of overheating above 1800°F increased rupture life due to increased creep resistance. The reduced ductility accompanying these effects did not influence rupture strength very much because of the inherently high ductility of the alloy. This loss in ductility only involved the last few hours of testing.

Inconel 700 Alloy - In this alloy, none of the conditions of overheating appreciably changed creep resistance. Overheating to temperatures of 2000°F and higher did reduce ductility. Because the shape of the creep curves made this alloy highly dependent on the ductility, these higher temperatures of overheating reduced rupture strength.

Both γ' alloys were notably resistant to damage to creep resistance from overheating. If damage is to be induced in this manner, it would only be for the lower temperatures of overheating. It is true, however, that the creep resistance of one alloy was improved by periodic overheats to the higher temperatures while the other was unchanged. Possibly these trends in creep resistance are related to the Al+Ti contents. Alloys with increasing amounts of γ' resulting from larger amounts of Al+Ti may be increasingly resistant to damage from the lower temperatures of overheating but lose the potential for being improved by the higher temperatures. Results from investigations for other sponsors at the University on similar alloys support this concept.

Within these patterns of influence on creep resistance, the additional influence of overheating on ductility must be considered. As

composition is varied among alloys to produce more γ' through the addition of more Al+Ti, ductility tends to become less. Moreover, the creep curves change to render the rupture strength more dependent on ductility. Thus the tendency for overheats to reduce ductility can reduce the rupture strength even though creep resistance is unaffected.

There is considerable question as to the generality of the ductility effects. M252 alloy by virtue of its high Mo content has high ductility in relation to other alloys. Therefore, ductility effects may be the controlling factor in other alloys even though creep resistance may not be damaged by overheats. At present, it appears that the influence of overheating in γ' alloys other than M252 will probably be mainly dependent on the influence on ductility. This must be restricted to alloys with total Al+Ti contents more than about 3.5 weight percent. It is suspected that the smaller amounts of γ' associated with lower amounts of these elements may result in a consequent susceptibility to reduced creep resistance from overheating.

There were several secondary objectives in the investigation. The increase in rupture life for M252 alloy from overheats to 1900°F and higher reported in reference 3 was substantiated on another heat. A loss in life from overheating to 1800°F was not found in the second heat. Neither changing the base test temperature from 1500° to 1600°F nor substantially altering the prior processing and heat treatment for M252 alloy changed the response to overheating. Whether these effects would apply in general to all γ' alloys is not certain. The results of a subsequent structural study of the effects of overheating suggest that they should be at least qualitatively correct so long as some additional factor did not appear in other alloys.

The resistance of the Al+Ti strengthened alloys to damage to creep resistance is in marked contrast to the solid solution - carbide strengthened alloys (ref. 1 and 2). Only 1 or 2 overheats significantly reduced the rupture time of S816 and HS-31 alloys by reducing creep resistance. The amount of the damage increased with overheat temperature and number of overheats. This contrast in behavior illustrates the dependence of the effects of overheating on the strengthening mechanism involved.

In the series of reports on overheating, the effects of repeated overheating has been treated as if the total time of overheating was

controlling. Preceding discussion has indicated that this cannot be done for the increase in creep resistance of M252 alloy - the overheats must be applied periodically. Since the creep resistance of Inconel 700 alloy was unaffected by overheating in the present study, its dependence on periodic application of the cycles is probably less. The data do not indicate, however, whether a single cycle of longer duration would affect creep resistance. The creep damage effect for overheating under stress does seem to be cumulative so that similar results would probably be obtained for one overheat of equivalent duration. Other research at the University suggests that this is the case. It is not known if the total overheat time or repeated overheating controls the reduction in ductility for Inconel 700 alloy. This is somewhat academic, however, since only a few overheats are so damaging.

Application of the results of these studies to service conditions must be done carefully. Although the data demonstrate that the creep resistance of the materials is not affected by overheating, in many cases, other effects such as thermal shock may be far more important in limiting materials which have suffered overtemperature.

CONCLUSIONS

The improvement in creep-rupture strength of M252 alloy previously reported for another heat was verified for a second heat. The general response to overheating was moreover the same for double solution treated material as well as for two conditions of rolling prior to heat treatment.

Inconel 700 alloy did not exhibit increased rupture strength. Reduction in strength was associated with reduced ductility from overheats to 2000°F and higher. Creep resistance up to the point of fracture was not altered by the overheats. This is in contrast to M252 alloy which exhibited increased creep resistance under these conditions.

The behavior of the two alloys indicates that γ' strengthened alloys are resistant to damage to creep resistance from structural changes induced by overheating. In most cases, the influence on future creep-rupture life then depends on the influence of overheating on ductility. M252 alloy may be an exception in that its rupture time is not appreciably influenced by substantial decreases in ductility due to its high initial ductility. Restrictions on these generalities, particularly the amount of Al+Ti in the alloys, are discussed in the report.

The response to thermally induced structural changes from overheating was the same for base temperatures of 1500° or 1600°F for both alloys.

Significant effects on creep-rupture properties required from 5 to 10 overheats for M252 alloy and from 3 to 5 overheats for Inconel 700 alloy.

University of Michigan,
Ann Arbor, Mich., March 25, 1960.

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2. Rowe, J. P., and Freeman, J. W.: Effect of Overheating on Creep-Rupture Properties of HS-31 Alloy at 1500°F. NACA TN 4083, 1957.
3. Rowe, J. P., and Freeman, J. W.: Effect of Overheating on Creep-Rupture Properties of M252 Alloy. NACA TN 4224, 1958.
4. Miller, B. A., Winward, J. M., and Smith, W. K.: High-Heating-Rate Strength of Three Heat-Resistant Metals. NAVORD Rep. 2017, NOTS 670, Naval Ord. Lab., Mar. 16, 1953.
5. Bigelow, W. C., Amy, J. A., and Brockway, L. O.: Electron Microscopic Identification of the γ' Phase of Nickel-Base Alloys. Proc. ASTM, vol. 56, pp. 945-953.

TABLE I. - CHEMICAL COMPOSITION OF EXPERIMENTAL MATERIALS

| Material | Composition (Weight Percent) | | | | | | | | | | |
|-------------------------------|------------------------------|------|------|-------|-------|-------|-------|------|------|------|--------|
| | C | Mn | Si | Cr | Ni | Co | Mo | Ti | Al | Fe | B |
| M252(heat 837) ¹ | 0.16 | 0.82 | 0.60 | 18.70 | 54.15 | 9.70 | 10.00 | 2.71 | 0.96 | 2.20 | 0.0007 |
| M252(heat HT-28) ² | 0.12 | 0.10 | 0.35 | 19.22 | 54.38 | 9.73 | 10.18 | 2.40 | 1.12 | 2.40 | 0.0014 |
| Inconel 700 ³ | 0.12 | 0.07 | 0.24 | 15.70 | 46.30 | 28.69 | 3.08 | 2.02 | 3.13 | 0.65 | 0.0074 |

1. Vacuum melted and supplied by the General Electric Company as 7/8-inch diameter bar stock.

2. Vacuum melted and supplied by the Haynes Stellite Company as 7/8-inch diameter bar stock.

3. Heat Y7952, melted by the International Nickel Company and supplied through the Curtis Wright Corporation, Wright Aeronautical Division as 1 1/16-inch diameter bar stock.

TABLE II
RESULTS OF TESTS ON M252 ALLOY

| Heat Number | Base Conditions | | Overheat Conditions | | Rupture Data | | | | |
|-------------|---------------------|--------------|---------------------|------------------|-------------------------|--------------------------|-----------------------|----|----|
| | Temperature (°F) | Stress (psi) | Temperature (°F) | Number of Cycles | Time (hr) | Elongation (% in 1 inch) | Reduction of Area (%) | | |
| 837 | 1500 ⁽¹⁾ | 15,000 | - | - | 644.8 | 47 | 58 | | |
| | | 20,000 | - | - | 251.7 | 36 | 54 | | |
| | | 25,000 | - | - | 153.8 | 39 | 47 | | |
| | | 30,000 | - | - | 49.9 | 36 | 49 | | |
| | | 30,000 | - | - | 61.6 | 38 | 48 | | |
| | | 30,000 | - | - | 62.7 | 47 | 58 | | |
| | | 34,000 | - | - | 38.2 | 40 | 50 | | |
| 837 | 1500 ⁽²⁾ | 34,000 | - | - | 60.1 | 28 | 43 | | |
| | | 33,000 | - | - | 75.9 | 38 | 39 | | |
| | | 33,000 | - | - | 81.7 | 38 | 45 | | |
| | | 32,000 | - | - | 94.0 | 37 | 42 | | |
| | | 22,000 | - | - | 427.0 | 34 | 46 | | |
| | | 33,000 | 1800 | 17 | 86.7 | 19 | 45 | | |
| | | 33,000 | 1800 | 10 | 77.0 | 27 | 42 | | |
| | | 33,000 | 2000 | 24 | 114.3 | 12 | 16 | | |
| | | 33,000 | 2000 | 10 | 98.5 | 36 | 42 | | |
| | | 33,000 | 2100 | 20 | 125.9 | 26 | 37 | | |
| | | 837 | 1600 ⁽²⁾ | 27,000 | - | - | 11.0 | 38 | 45 |
| 18,000 | - | | | - | 71.9 | 34 | 50 | | |
| 18,000 | - | | | - | 75.4 | 47 | 51 | | |
| 18,000 | - | | | - | 82.5 | 48 | 53 | | |
| 18,000 | - | | | - | 88.3 | 52 | 54 | | |
| 18,000 | - | | | - | 91.5 | 46 | 57 | | |
| 18,000 | - | | | - | 97.0 | 45 | 54 | | |
| 18,000 | - | | | - | 50.0 (Interrupted test) | | | | |
| 18,000 | 1800 | | | 12 | 73.8 | 25 | 55 | | |
| 18,000 | 1800 | | | 10 | 50.0 (Interrupted test) | | | | |
| 18,000 | 1800 | | | 6 | 73.4 | 38 | 39 | | |
| 18,000 | 1800 | | | 3 | 76.7 | 48 | 53 | | |
| 18,000 | 2000 | | | 27 | 137.7 | 40 | 33 | | |
| 18,000 | 2000 | | | 10 | 98.6 | 43 | 47 | | |
| 18,000 | 2000 | | | 10 | 50.0 (Interrupted test) | | | | |
| 18,000 | 2000 | | | 5 | 88.2 | 46 | 47 | | |
| 18,000 | 2100 | | | 20 | 138.6 | 41 | 39 | | |
| 18,000 | 2100 | | | 10 | 100.4 | 35 | 38 | | |
| 18,000 | 2100 | | | 5 | 92.7 | 49 | 50 | | |
| HT-28 | 1500 ⁽³⁾ | | | 21,000 | - | - | 736.0 | 61 | 62 |
| | | | | 24,000 | - | - | 573.9 | 39 | 61 |
| | | 24,000 | - | - | 450.4 | 46 | 55 | | |
| | | 26,500 | - | - | 356.0 | 36 | 59 | | |
| | | 26,500 | - | - | 317.8 | 40 | 56 | | |
| | | 30,000 | - | - | 207.7 | 43 | 58 | | |
| | | 34,000 | - | - | 105.2 | 45 | 54 | | |
| | | 34,000 | - | - | 81.6 | 49 | 58 | | |
| | | 34,000 | - | - | 79.2 | 41 | 55 | | |
| | | 35,000 | - | - | 70.9 | 26 | 41 | | |
| | | 34,000 | 1650 | 18 | 84.9 | 37 | 49 | | |
| | | 34,000 | 1650 | 11 | 54.8 | 34 | 42 | | |
| | | 34,000 | 1650 | 7 | 50.6 | 28 | 44 | | |
| | | 34,000 | 1800 | 10 | 52.7 | 22 | 27 | | |
| | | 34,000 | 1800 | 9 | 48.1 | 25 | 37 | | |
| | | 34,000 | 1800 | 5 | 68.2 | 38 | 52 | | |
| | | 34,000 | 1900 | 30 | 181.6 | 23 | 45 | | |
| | | 34,000 | 1900 | 15 | 137.3 | 35 | 53 | | |
| | | 34,000 | 2000 | 30 | 211.3 | 16 | 50 | | |
| | | 34,000 | 2000 | 15 | 170.9 | 25 | 46 | | |
| HT-28 | 1600 ⁽²⁾ | 20,000 | - | - | 112.4 | 56 | 64 | | |
| | | 20,000 | - | - | 109.4 | 78 | 64 | | |
| | | 20,000 | 1800 | 10 | 89.4 | 28 | 62 | | |
| | | 20,000 | 2000 | 10 | 134.7 | 56 | 51 | | |

(1) Data from ref. (2). Material rolled from 1950°F, solution treated 4 hours at 1950°F, and aged at 1400°F for 15 hours.

(2) Material rolled from 2150°F, solution treated 1 hour 2150°F plus 4 hrs 1950°F, and aged at 1400°F for 15 hours.

(3) Data from ref. (2). Material was rolled from 2150°F, solution treated 4 hours at 1950°F, and aged at 1400°F for 15 hours.

TABLE III
RESULTS OF TESTS ON INCONEL 700 ALLOY

| Sample Number | Base Conditions | | Overheat Conditions | | Rupture Data | | |
|---------------|------------------|--------------|---------------------|------------------|------------------------------|--------------------------|-----------------------|
| | Temperature (°F) | Stress (psi) | Temperature (°F) | Number of Cycles | Time (hr) | Elongation (% in 1 inch) | Reduction of Area (%) |
| 11D | 1500 | 34,000 | - | - | 803.5 | 11 | 12 |
| 12A | 1500 | 40,000 | - | - | 208 | 7 | 10 |
| 16D | 1500 | 43,000 | - | - | 122.6 | 6 | 9 |
| 17C | 1500 | 45,000 | - | - | 44.5 | 4 | 2 |
| 19- | 1500 | 43,000 | - | - | 92 Estimated ⁽¹⁾ | | |
| 19C | 1500 | 43,000 | 2000 | 4 | 18.6 | 3 | 5 |
| 20- | 1500 | 43,000 | - | - | 80 Estimated ⁽¹⁾ | | |
| 20D | 1500 | 43,000 | 1800 | 13 | 71.8 | 4 | 4 |
| 20C | 1500 | 43,000 | 1800 | 10 | 70.7 | 6 | 5 |
| 20B | 1500 | 43,000 | 2000 | 7 | 34.5 | 1 | 2 |
| 21B | 1500 | 43,000 | 2100 | 4 | 18.5 | 2 | 2 |
| 23B | 1500 | 43,000 | - | - | 129.5 | 7 | 12 |
| 24D | 1500 | 43,000 | - | - | 65.4 | 6 | 7 |
| 25- | 1500 | 43,000 | - | - | 102 Estimated ⁽¹⁾ | | |
| 25B | 1500 | 43,000 | 1800 | 10 | 104.8 | 4 | 7 |
| 11A | 1600 | 20,000 | - | - | 633.0 | 18 | 21 |
| 12B | 1600 | 28,000 | - | - | 103.6 | 16 | 22 |
| 13B | 1600 | 29,000 | 2000 | 6 | 54.7 | 10 | 10 |
| 14A | 1600 | 29,000 | - | - | 89.2 | 17 | 22 |
| 14B | 1600 | 29,000 | 1800 | 14 | 69.5 | 10 | 15 |
| 14D | 1600 | 29,000 | 2000 | 6 | 30.0 (Interrupted test) | | |
| 15C | 1600 | 29,000 | - | - | 90.0 | 18 | 20 |
| 15D | 1600 | 29,000 | - | - | 30.0 (Interrupted test) | | |
| 15B | 1600 | 29,000 | 2000 | 12 | 55.7 | 4 | 8 |
| 16A | 1600 | 29,000 | - | - | 83.8 | 14 | Data Not Available |
| 16B | 1600 | 29,000 | 2100 | 11 | 53.1 | 4 | 5 |
| 18A | 1600 | 25,000 | - | - | 229.1 | 13 | 17 |
| 19A | 1600 | 29,000 | - | - | 76.6 | 13 | 20 |
| 19B | 1600 | 29,000 | - | - | 63.1 | 8 | 13 |
| 20A | 1600 | 29,000 | - | - | 62.7 | 10 | 14 |
| 22C | 1600 | 16,500 | - | - | 977.3 | 8 | 10 |
| 22D | 1600 | 29,000 | 2000 | 3 | 34.5 | 4 | 5 |
| 23C | 1600 | 29,000 | - | - | 65.4 | 13 | 21 |
| 23D | 1600 | 29,000 | 2100 | 6 | 79.4 | 8 | 14 |
| 23A | 1600 | 29,000 | 2100 | 3 | 71.8 | 13 | 16 |
| 24C | 1600 | 29,000 | - | - | 53.8 | 8 | 14 |
| 24B | 1600 | 29,000 | 2000 | 6 | 33.7 | 4 | 4 |
| 24A | 1600 | 29,000 | 2000 | 3 | 35.2 | 3 | 5 |
| 25A | 1600 | 29,000 | - | - | 73.6 | 12 | 20 |
| 25C | 1600 | 29,000 | 1800 | 7 | 78.9 | 14 | 15 |
| 25D | 1600 | 29,000 | 2100 | 3 | 62.5 | 9 | 15 |
| 50A | 1600 | 29,000 | - | - | 50.2 | 12 | 14 |
| 50C | 1600 | 29,000 | - | - | 49.0 | 13 | 13 |
| 50D | 1600 | 29,000 | - | - | 47.9 | 12 | 15 |
| 50B | 1600 | 29,000 | - | - | 45.7 | 12 | 15 |
| 50F | 1600 | 29,000 | 1800 | 8 | 44.9 | 9 | 11 |
| 50G | 1600 | 29,000 | 2100 | 5 | 25.4 | 3 | 6 |
| 50H | 1600 | 29,000 | 2200 | 4 | 32.5 | 4 | 6 |
| 51C | 1600 | 29,000 | - | - | 86.8 | 13 | 15 |
| 51B | 1600 | 29,000 | - | - | 83.1 | 15 | 19 |
| 51A | 1600 | 29,000 | - | - | 72.9 | 19 | 18 |
| 51D | 1600 | 29,000 | - | - | 65.6 | 15 | 18 |

(1) Values estimated from data at 1600°F (see text pg. 15). Range in time at 1500°F and 43,000 psi estimated to be 60-140 hours.

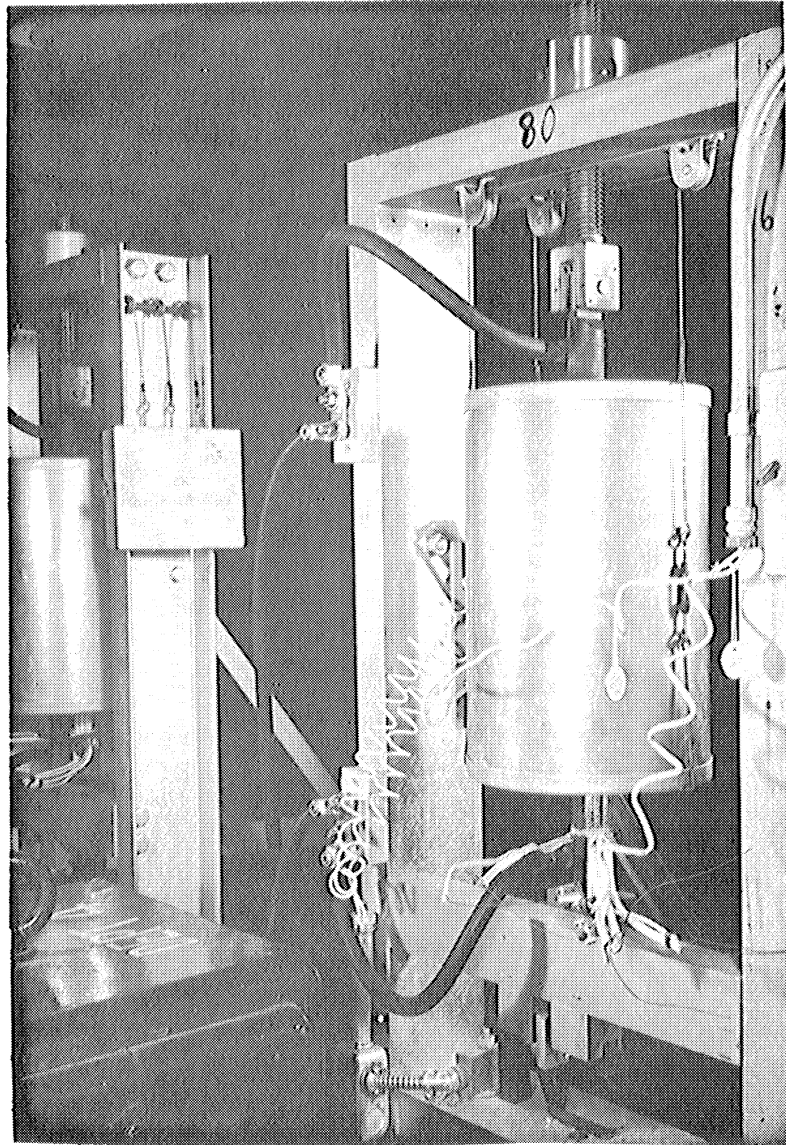


Figure 1. - Photograph showing creep-rupture unit modified for use in overheating by resistance heating.

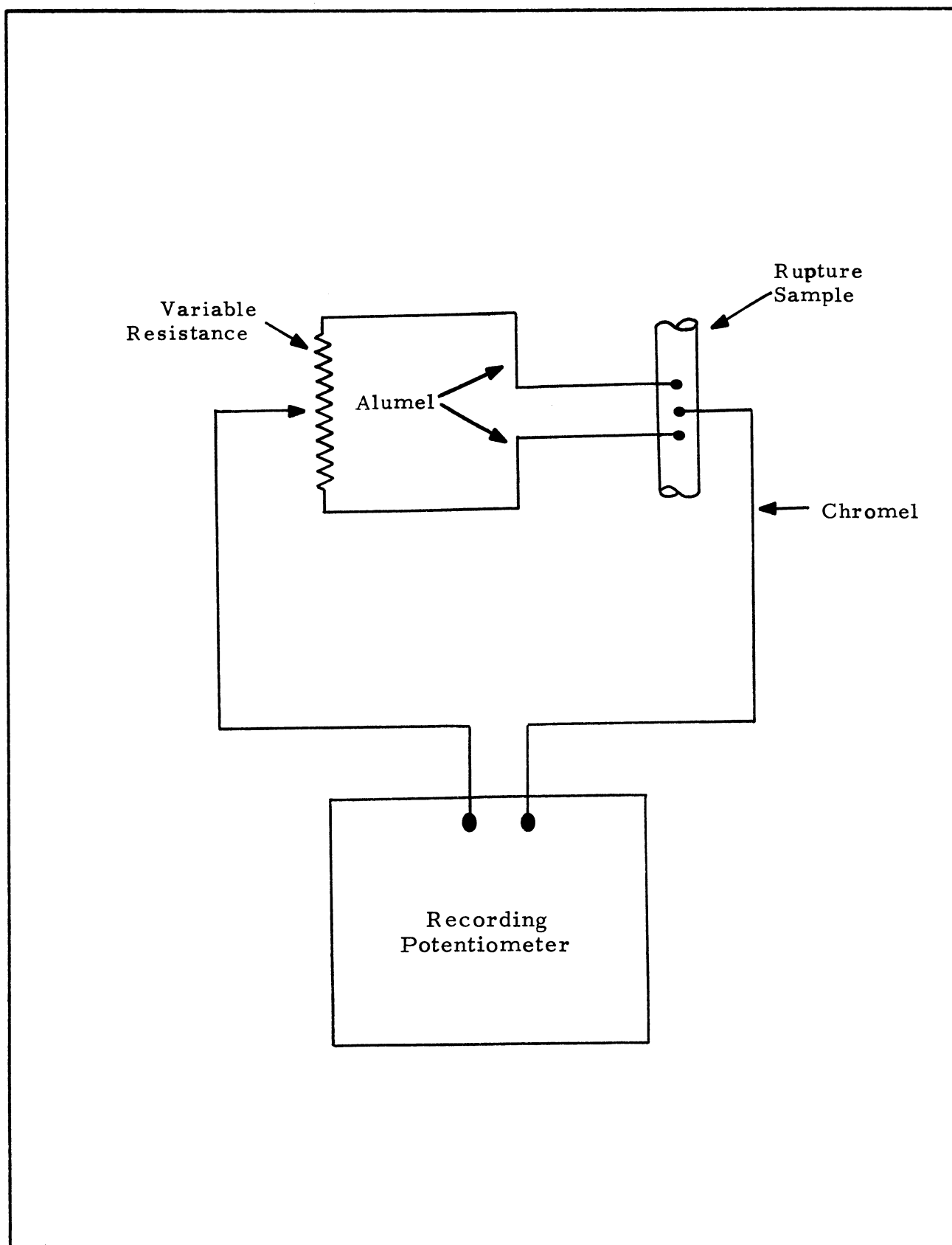


Figure 2. - Schematic wiring diagram of the system used for measurement of temperature during overheats to avoid extraneous emf from heating current.

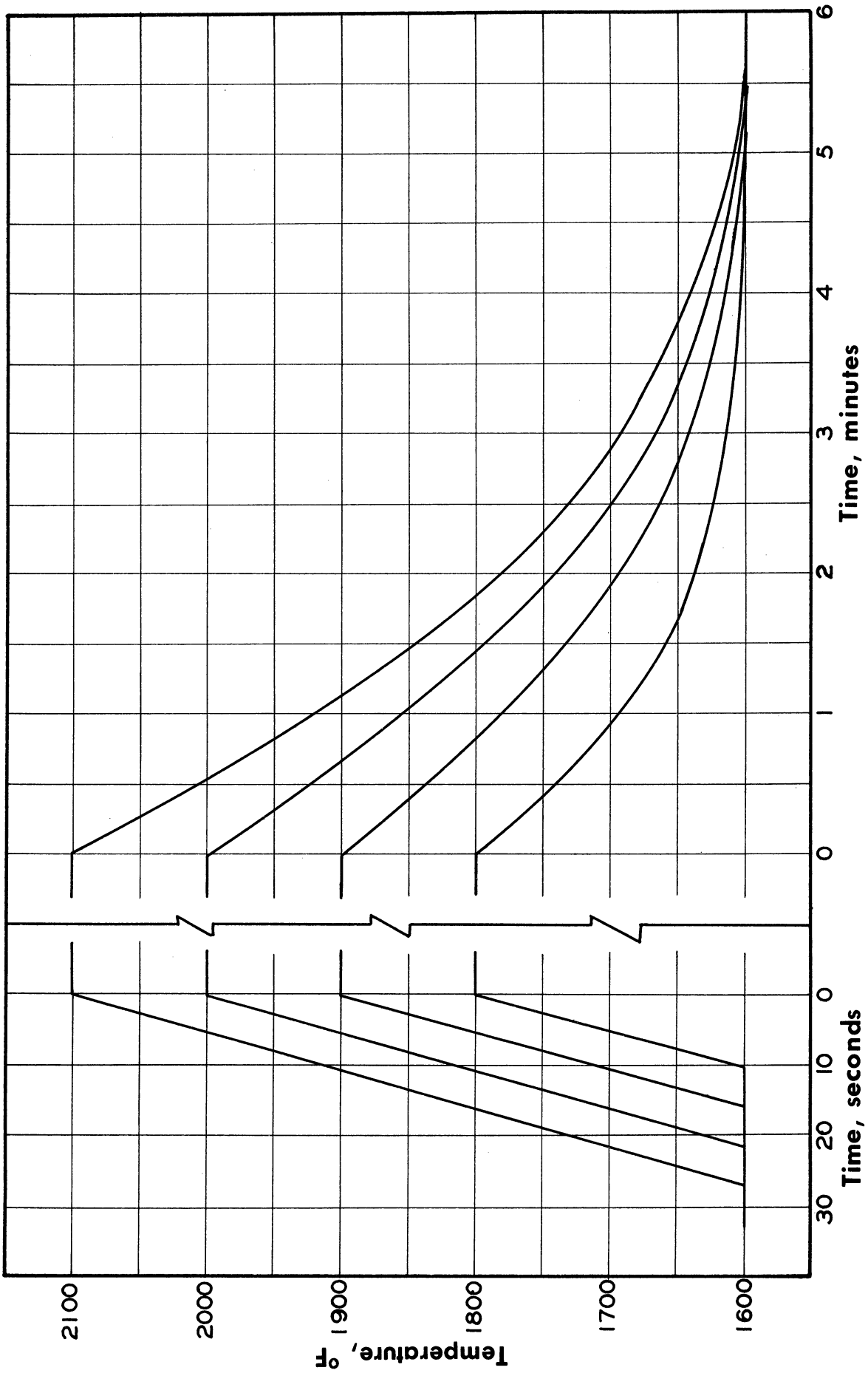
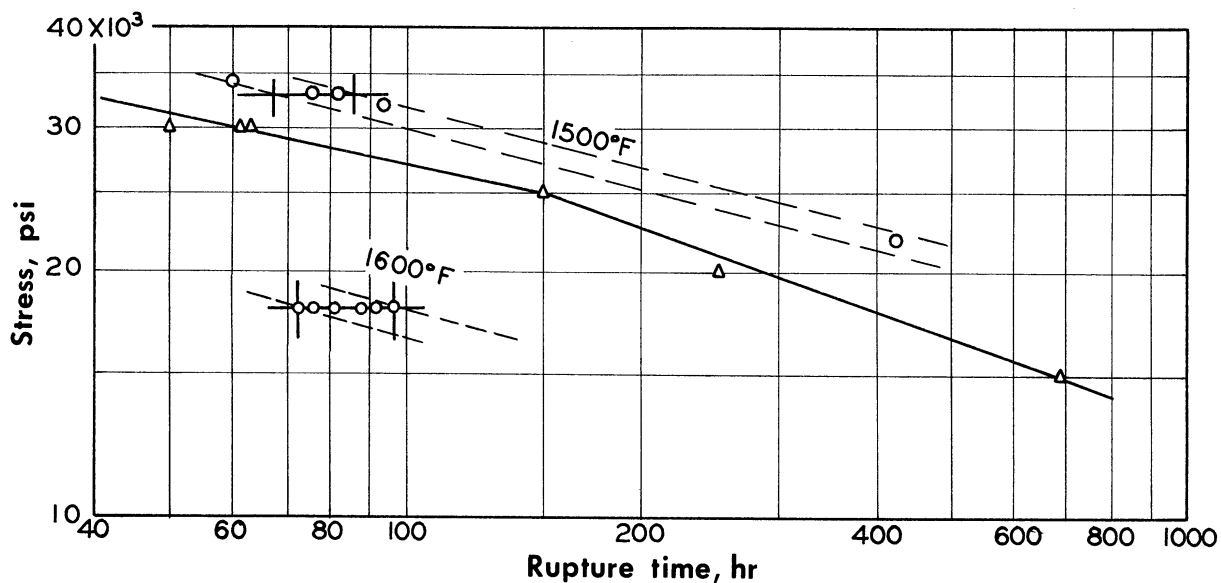
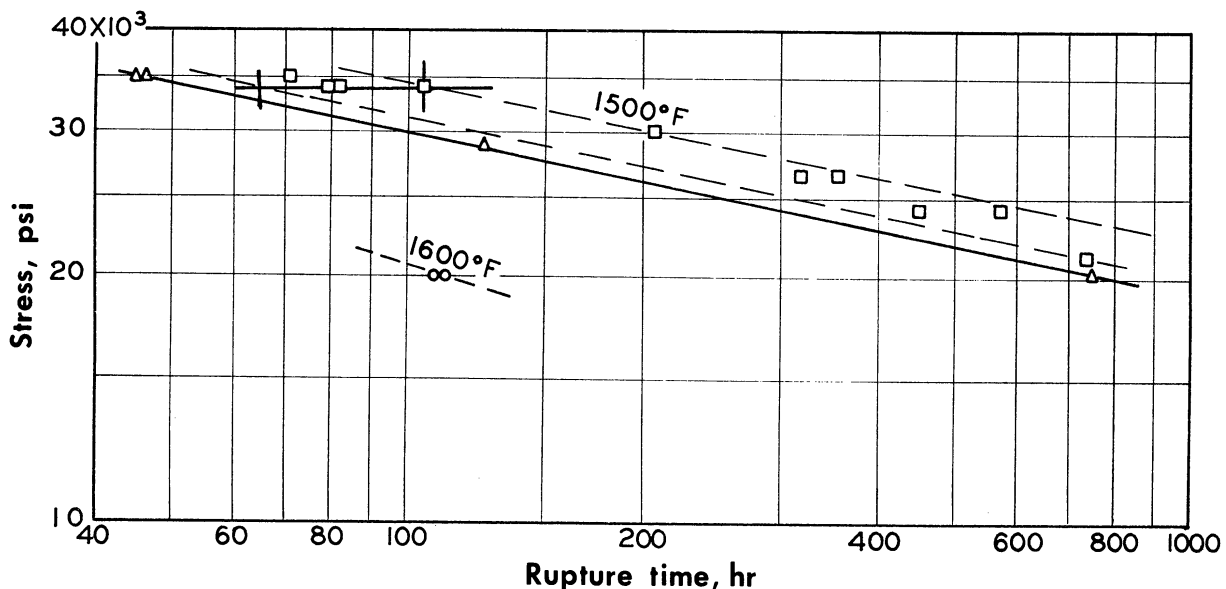


Figure 3. - Typical time-temperature curves for several of the overheat temperatures employed.

- Rolled from 2150°F, solution treated 1 hour at 2150°F plus 4 hours at 1950°F, and aged 15 hours at 1400°F; air cooled from all treatments.
- △ Rolled from 1950°F, solution treated 4 hours at 1950°F, and aged 15 hours at 1400°F; air cooled from all treatments. (Data from ref. 3)
- Rolled from 2150°F, solution treated 4 hours at 1950°F, and aged 15 hours at 1400°F; air cooled from all treatments. (Data from ref. 3)

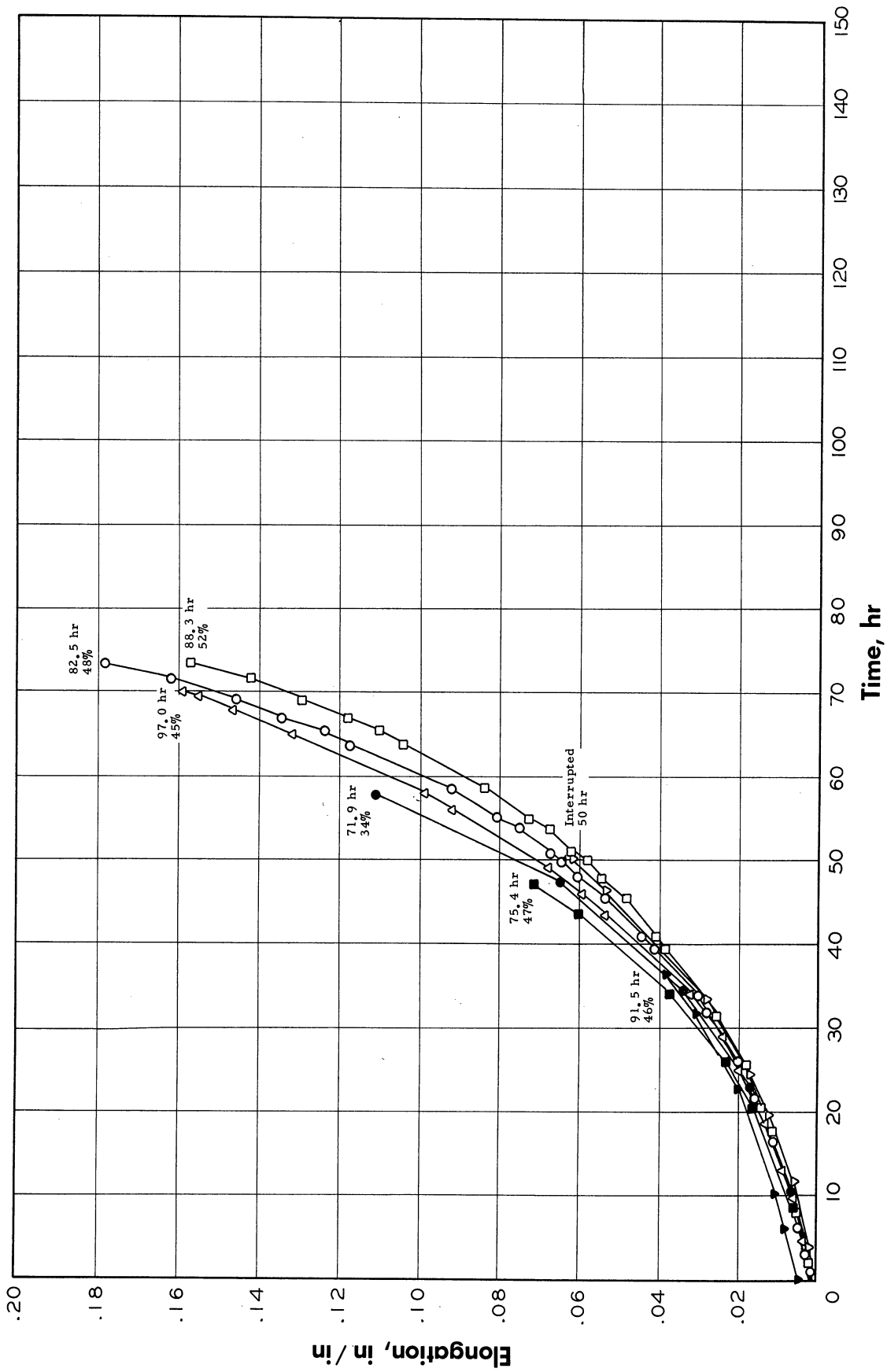


(a) Heat 837.



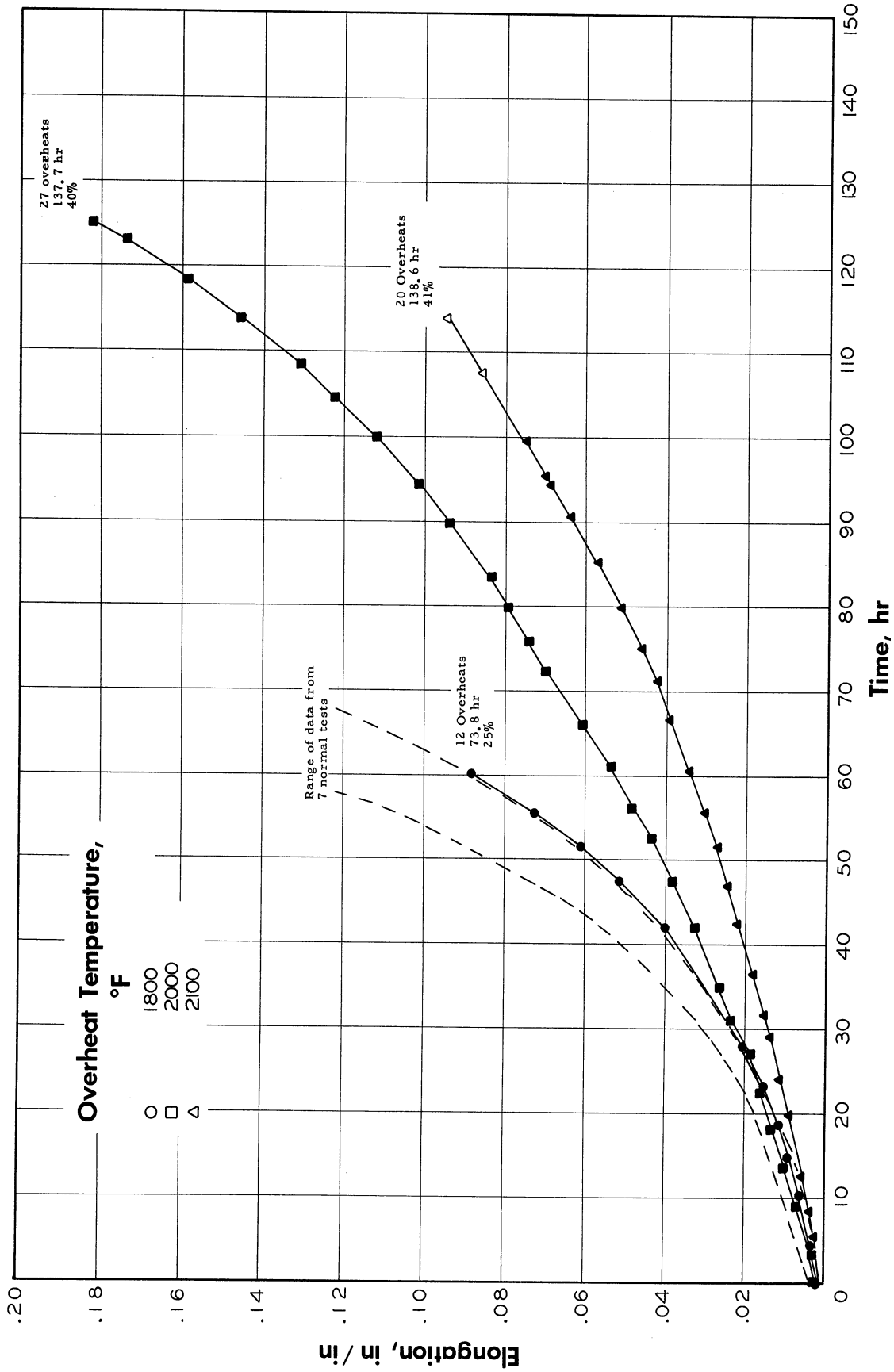
(b) Heat HT-28.

Figure 4. - Curves of stress against rupture time at 1500° and 1600°F from normal rupture tests on the two heats of M252 alloy used in the investigation, showing at the stresses used for overheat tests, the ranges in rupture time predicted by the available data.



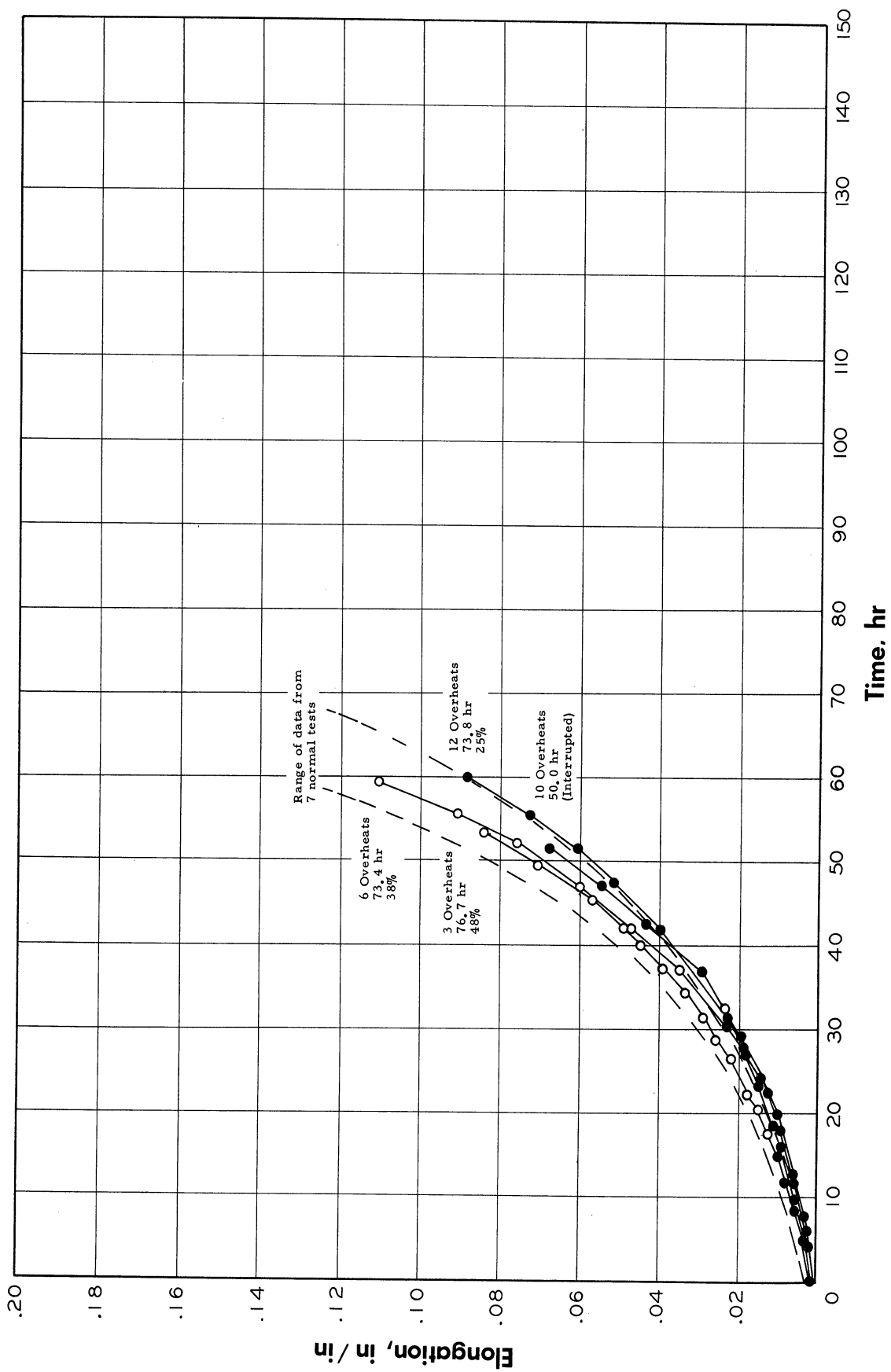
(a) Normal creep rupture tests.

Figure 5. - Comparative creep curves at 1600°F and 18,000 psi for heat 837 of M252 alloy for the indicated test conditions. Solid points indicate period of overheats. Number of overheats, time for rupture and elongation at rupture indicated for each curve.



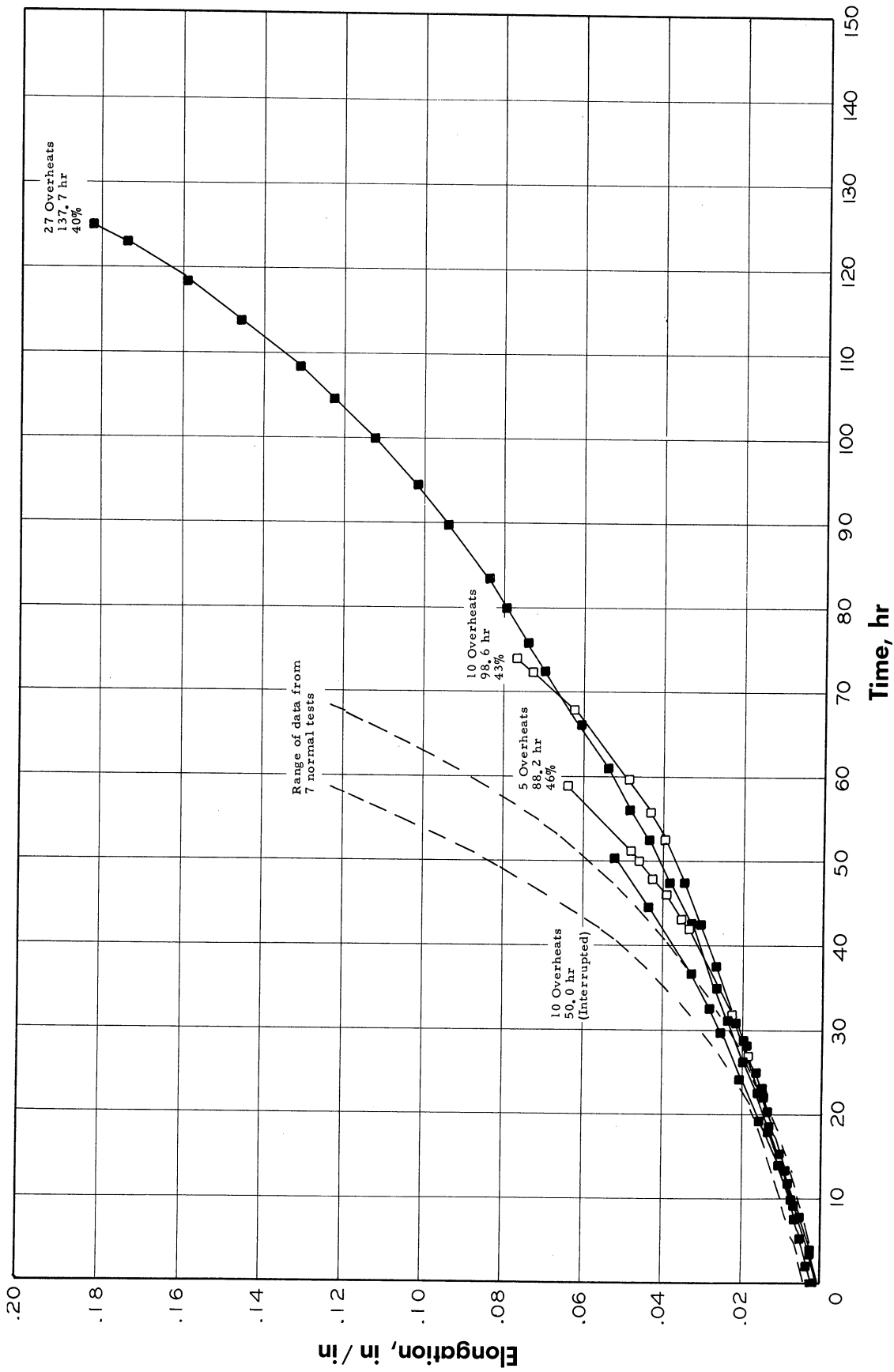
(b) Tests receiving the greatest number of overheats to each temperature.

Figure 5. - Continued. Comparative creep curves at 1600°F and 18,000 psi for heat 837 of M252 alloy for the indicated test conditions. Solid points indicate period of application of overheats. Number of overheats, time for rupture and elongation at rupture indicated for each curve.



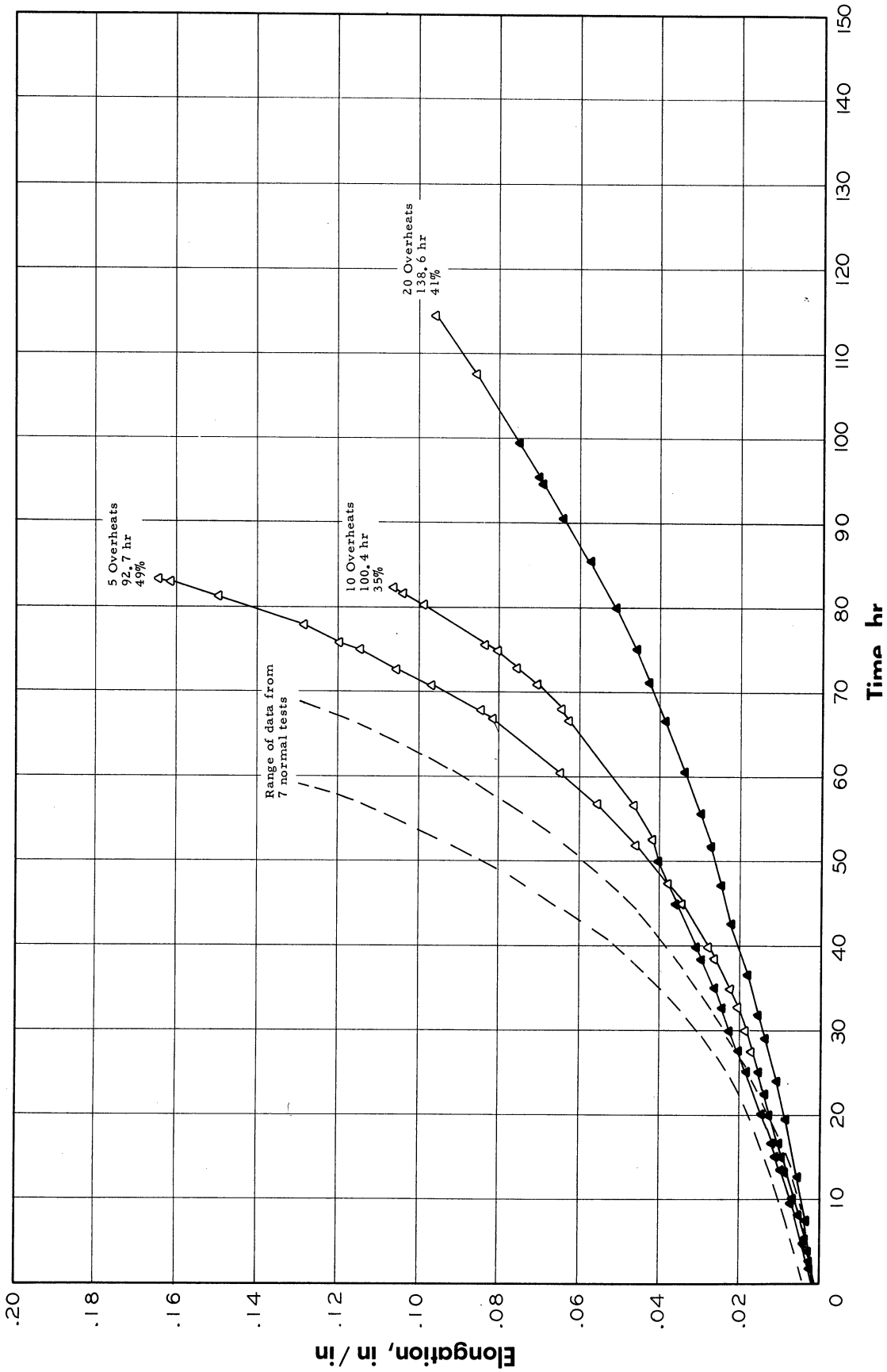
(c) Tests overheated to 1800°F.

Figure 5. - Continued. Comparative creep curves at 1600°F and 18,000 psi for heat 837 of M252 alloy for the indicated test conditions. Solid points indicate period of application of overheats. Number of overheats, time for rupture and elongation at rupture indicated for each curve.



(d) Tests overheated to 2000°F.

Figure 5. - Continued. Comparative creep curves at 1600°F and 18,000 psi for heat 837 of M252 alloy for the indicated test conditions. Solid points indicate period of application of overheats. Number of overheats, time for rupture and elongation at rupture indicated for each curve.



(e) Tests overheated to 2100°F.

Figure 5. - Concluded. Comparative creep curves at 1600°F and 18,000 psi for heat 837 of M252 alloy for the indicated test conditions. Solid points indicate period of application of overheats. Number of overheats, time for rupture and elongation at rupture indicated for each curve.

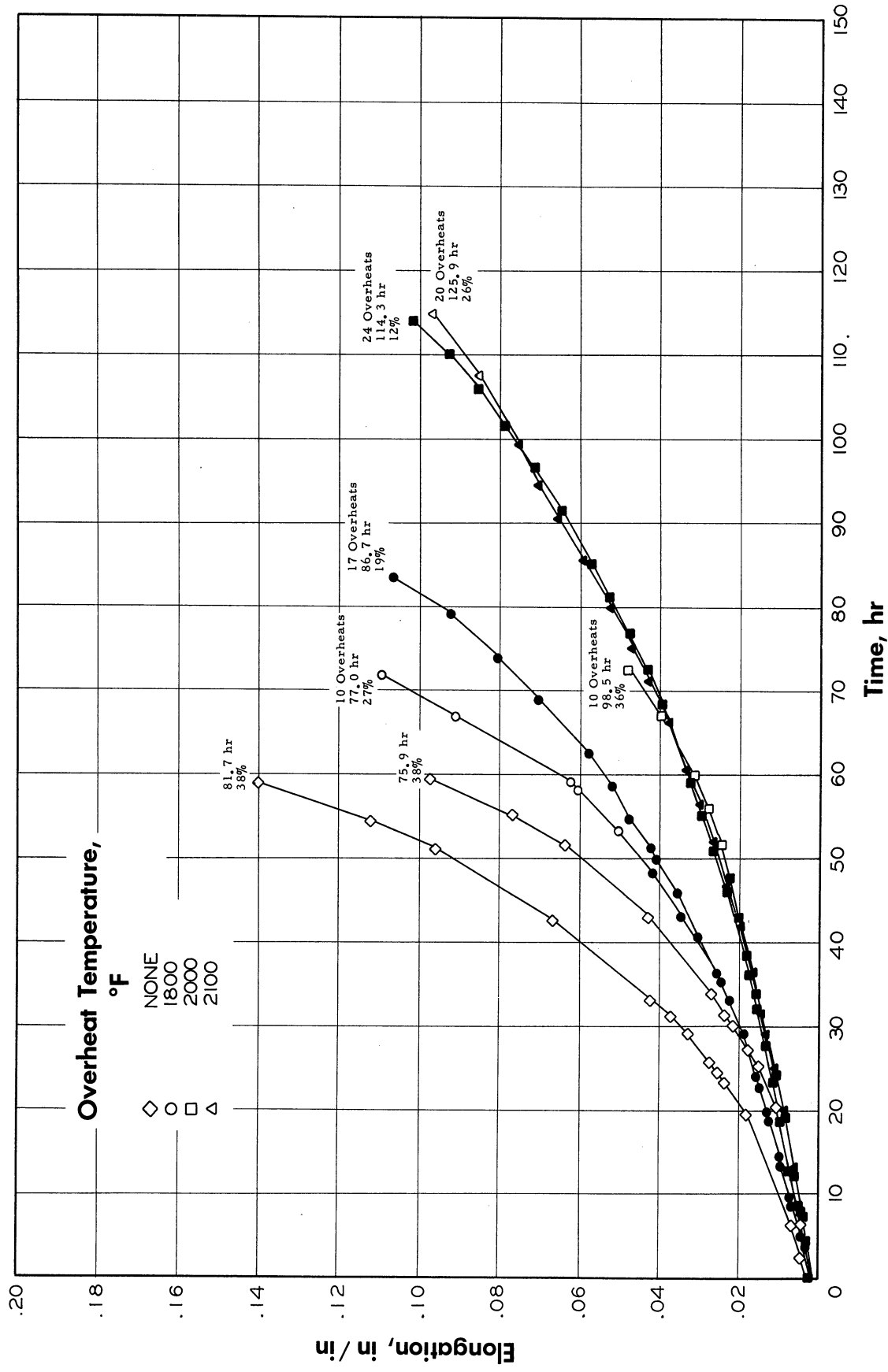


Figure 6. - Comparative creep curves at 1500°F and 33,000 psi for heat 837 of M252 alloy for the indicated test conditions. Solid points indicate period of application of overheats. Number of overheats, time for rupture and elongation at rupture indicated for each curve.

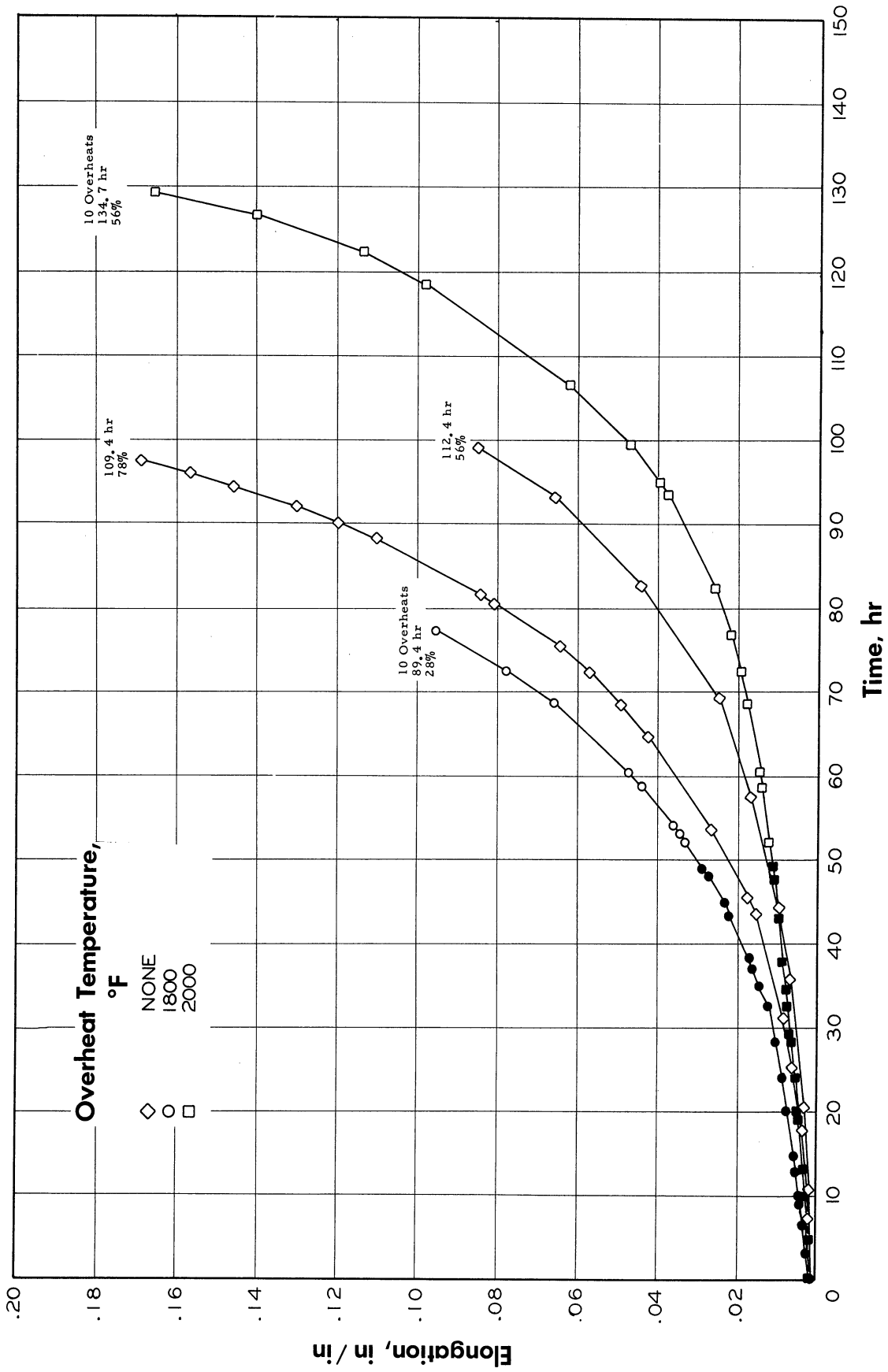


Figure 7. - Comparative creep curves at 1600°F and 20,000 psi for heat HT-28 of M252 alloy for the indicated test conditions. Solid points indicate period of application of overheats. Number of overheats, time for rupture and elongation at rupture indicated for each curve.

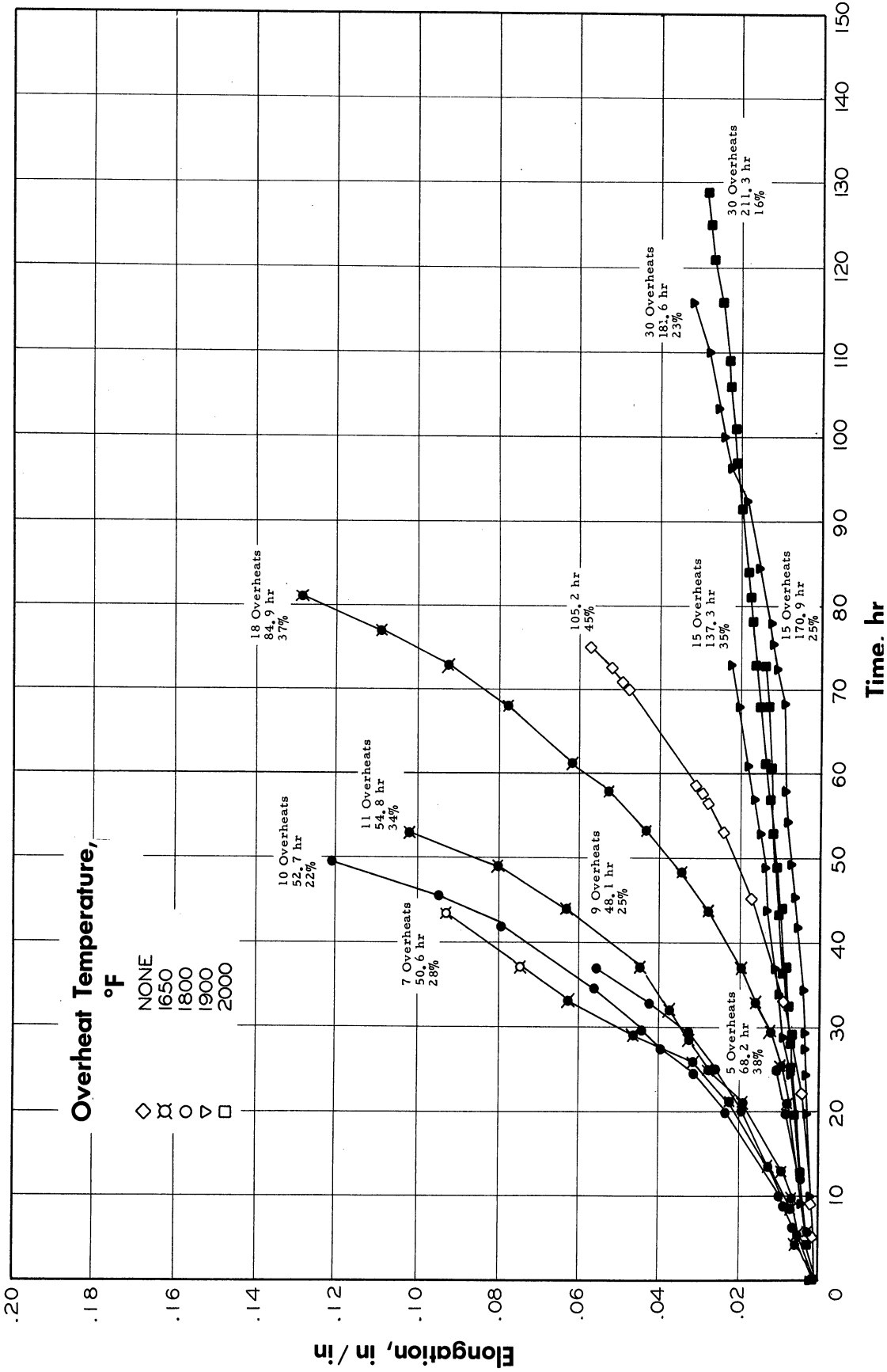
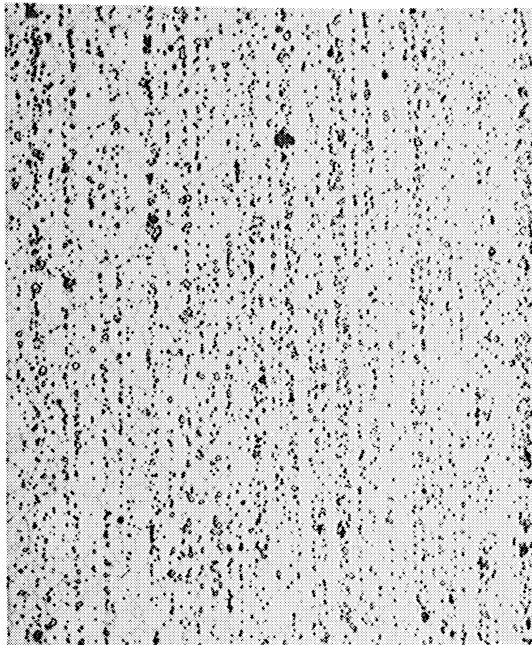
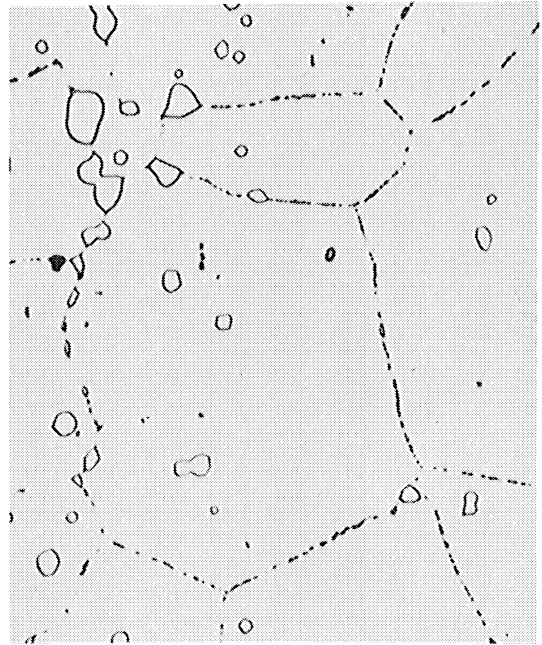


Figure 8. - Comparative creep curves at 1500°F and 34,000 psi for heat HT-28 of M252 alloy for the indicated test conditions. Solid points indicate period of application of overheats. Number of overheats, time for rupture and elongation at rupture indicated for each curve.

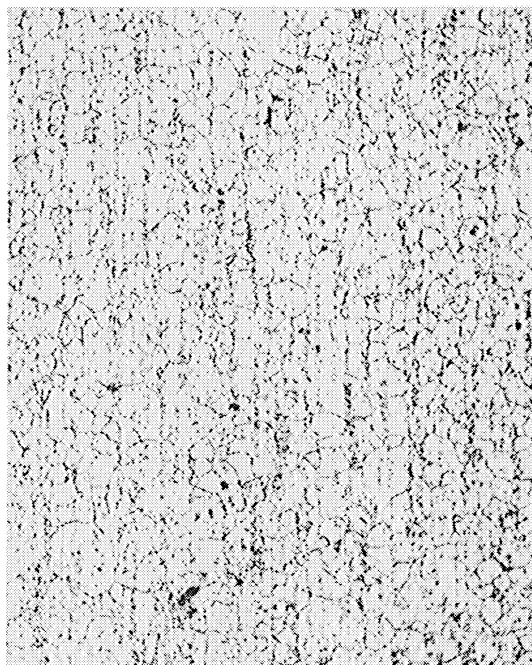


X100

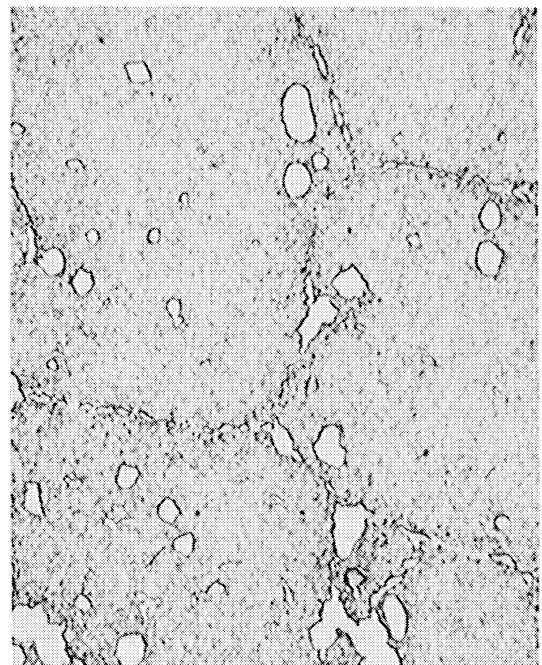


X1000

(a) Heat 837, as heat-treated.



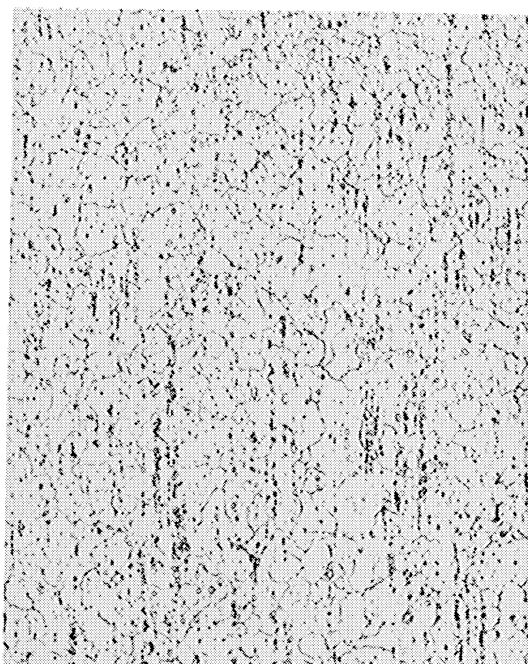
X100



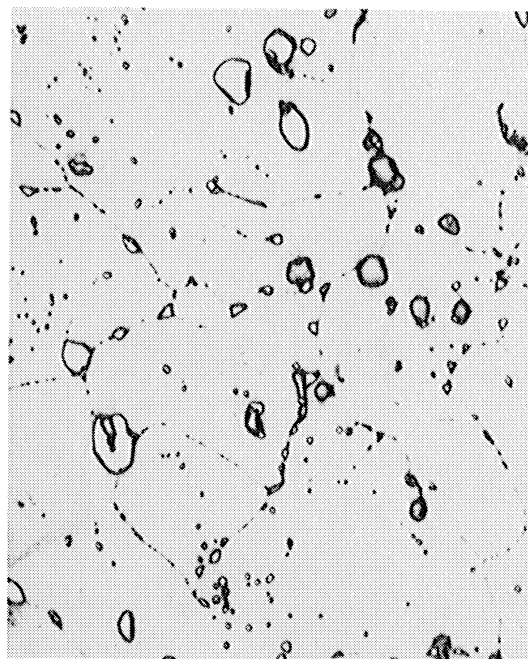
X1000

(b) Heat 837 after rupture testing at 1600°F and 18,000 psi.
Interrupted at 50.0 hours.

Figure 9. - Microstructures of M252 heats after heat treatment and after rupture testing at 1600°F.

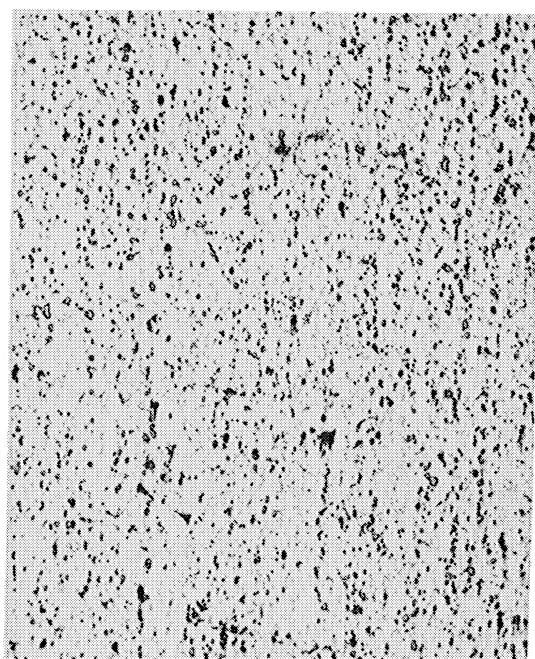


X100

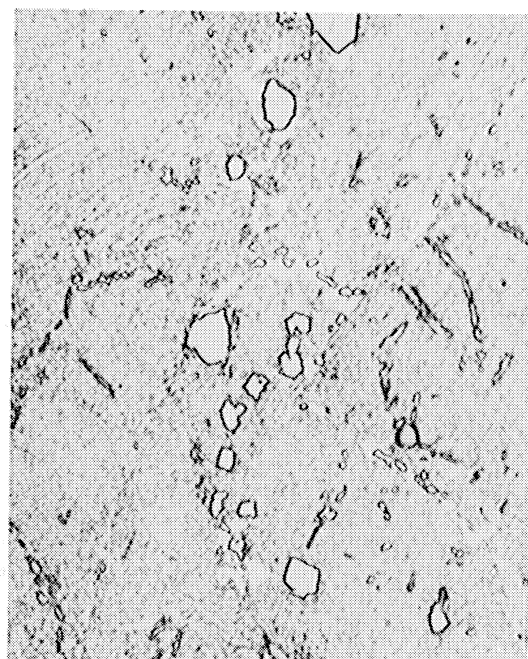


X1000

(c) Heat HT-28, as heat-treated.



X100

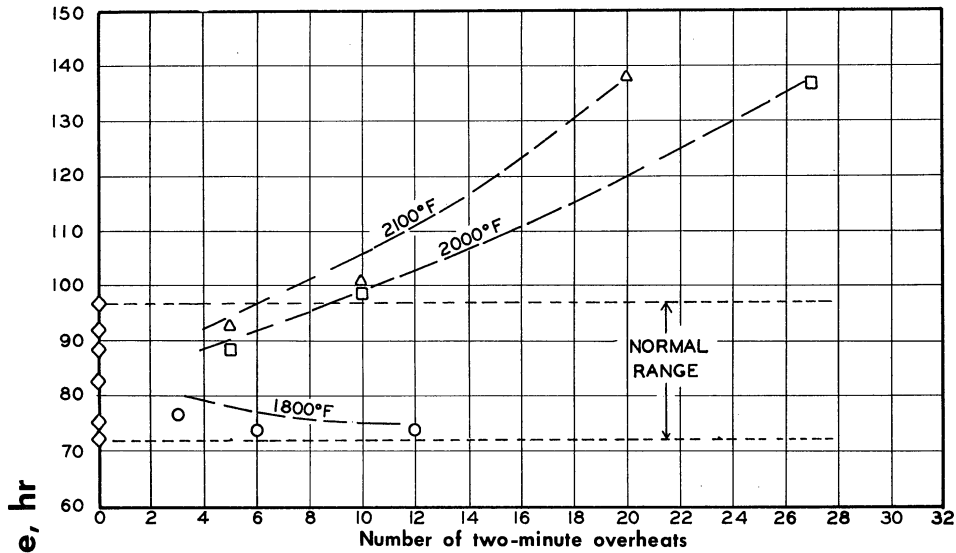


X1000

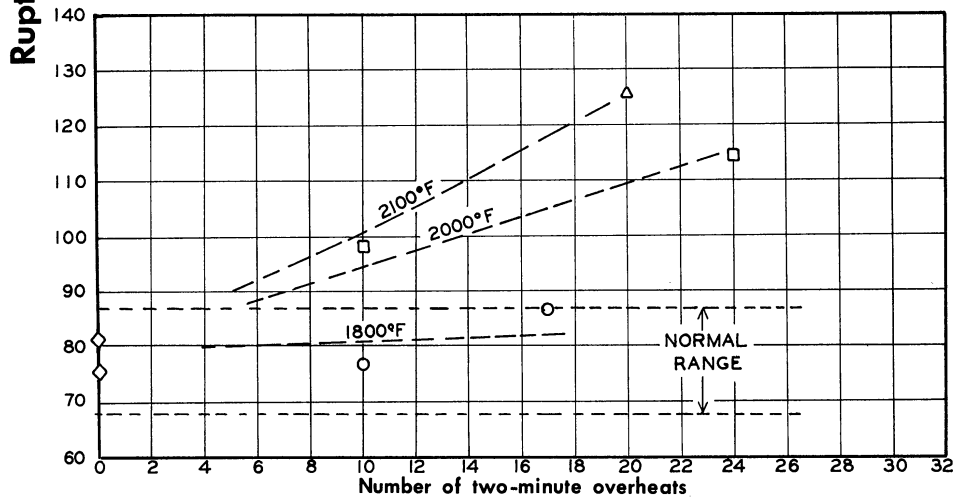
(d) Heat HT-28 after rupture testing at 1600°F and 20,000 psi.
Rupture time, 109.4 hours.

Figure 9. - Concluded. Microstructures of M252 heats after heat treatment and after rupture testing at 1600°F.

| Overheat Temperature, °F | | | |
|--------------------------|------|---|------|
| ◇ | NONE | ▽ | 1900 |
| ⊗ | 1650 | □ | 2000 |
| ○ | 1800 | △ | 2100 |

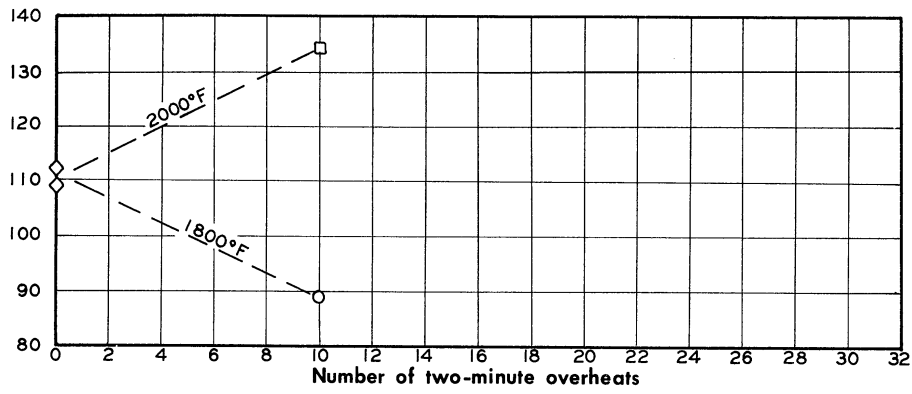


(a) Heat 837 tested at 1600°F and 18,000 psi.

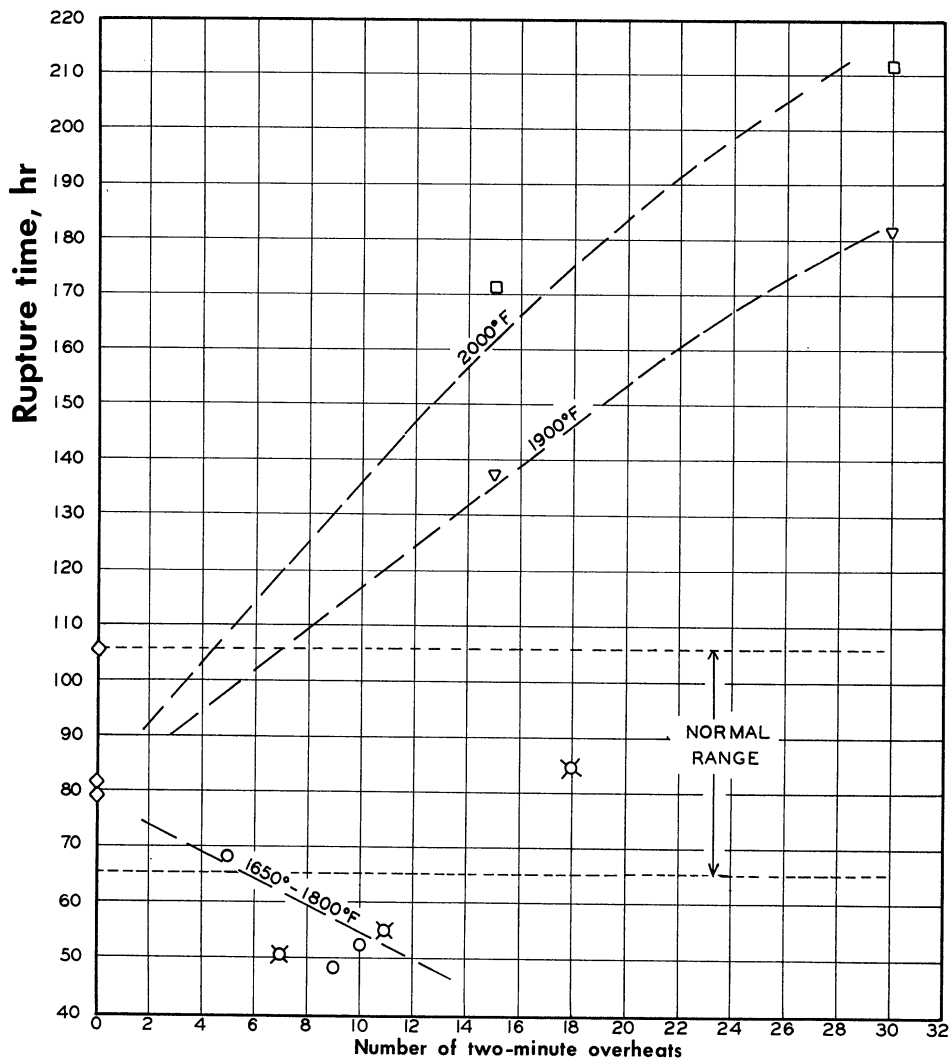


(b) Heat 837 tested at 1500°F and 33,000 psi.

Figure 10. - Effect of number of overheats on the rupture time of M252 alloy for the overheat temperatures and base conditions indicated. Overheats were of two minutes duration applied every five hours from the beginning of the test.

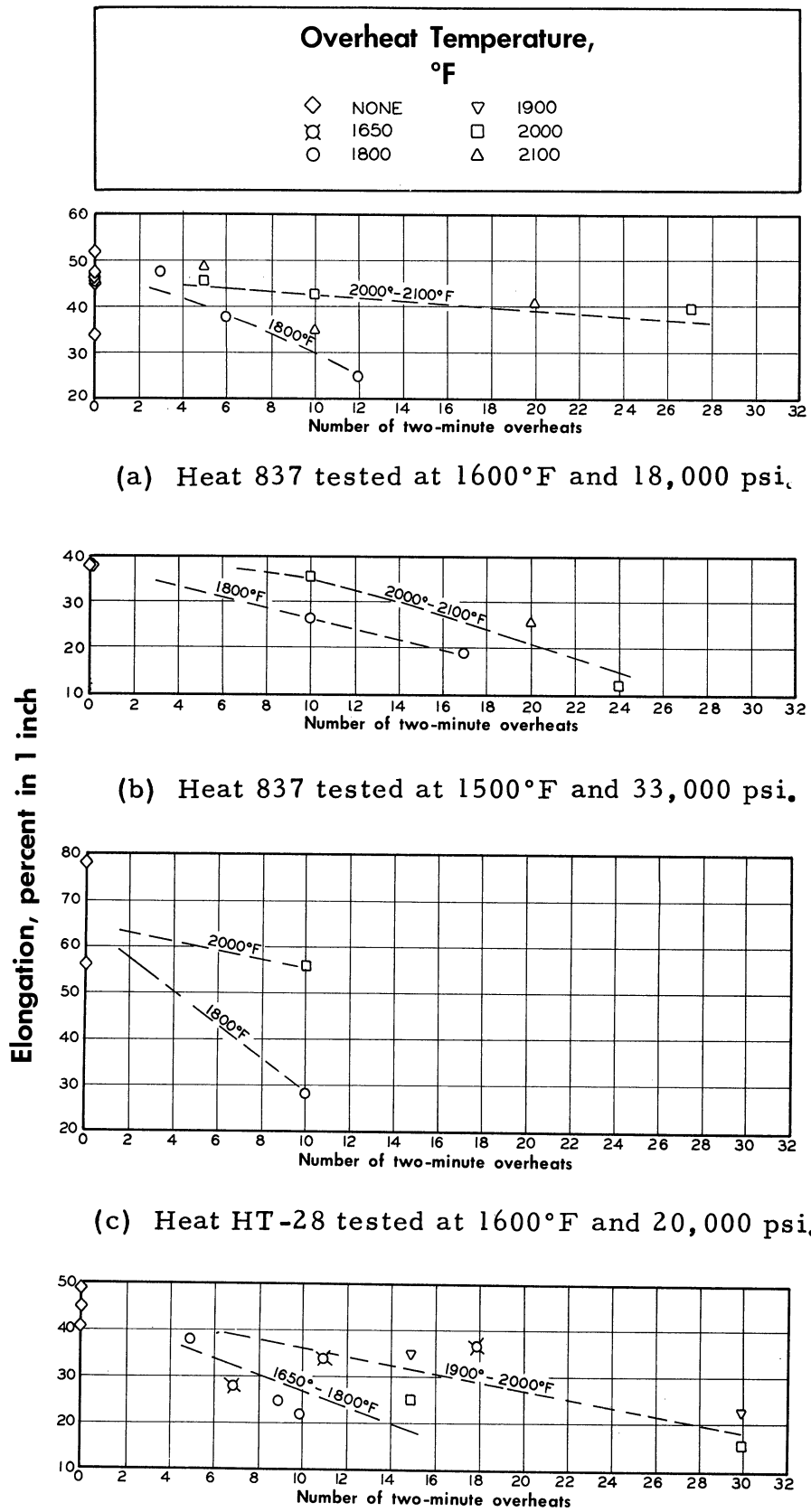


(c) Heat HT-28 tested at 1600°F and 20,000 psi.



(d) Heat HT-28 tested at 1500°F and 34,000 psi. (Data from ref. 3).

Figure 10. - Concluded. Effect of number of overheats on the rupture time of M252 alloy for the overheat temperatures and base conditions indicated. Overheats were of two minutes duration applied every five hours from the beginning of the test.



(d) Heat HT-28 tested at 1500°F and 34,000 psi. (Data from ref. 3).

Figure 11. - Effect of number of overheats on the elongation at fracture of M252 alloy for the overheat temperatures and base conditions indicated. Overheats were of two minutes duration applied every five hours from the beginning of the test.

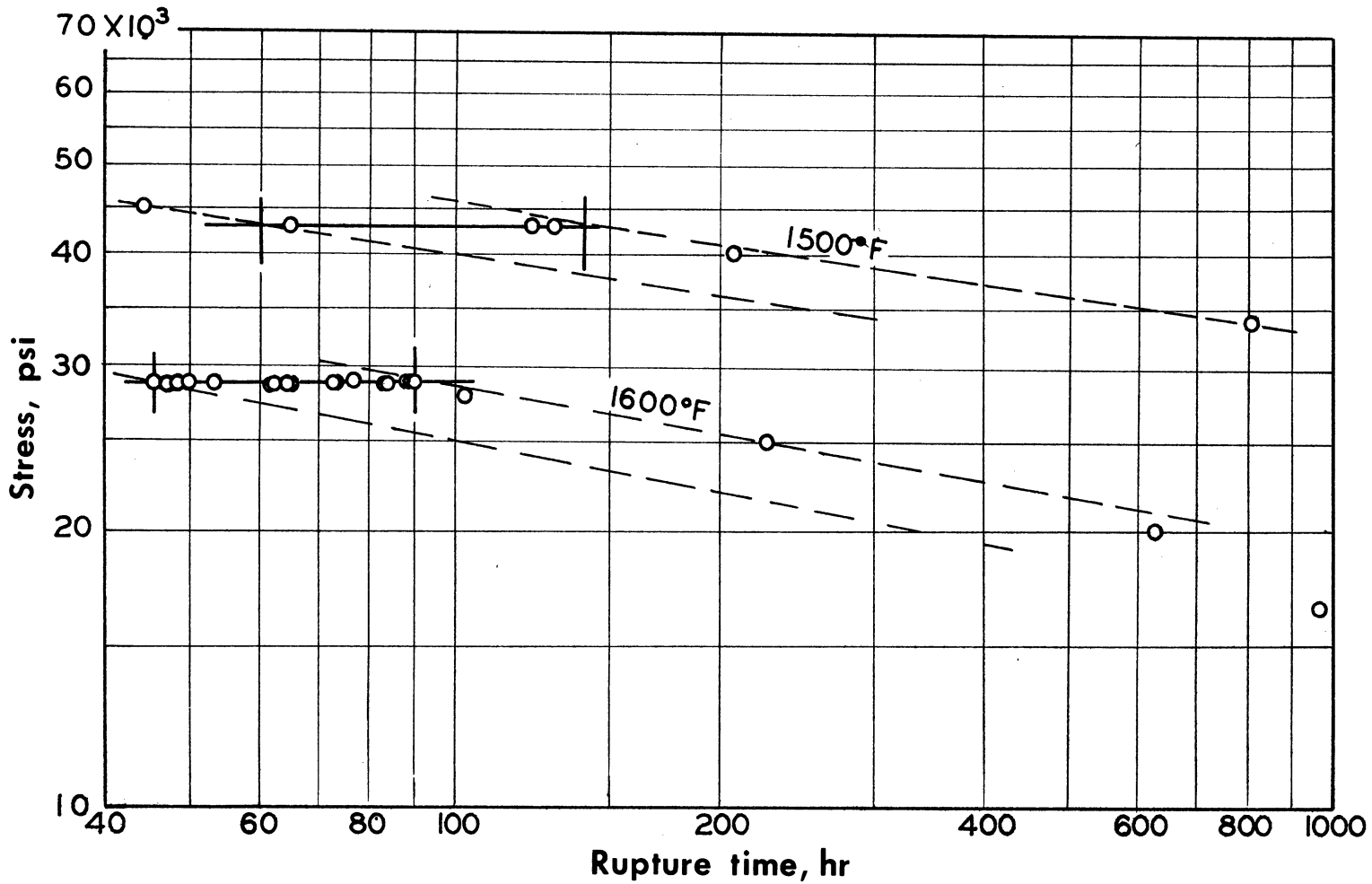


Figure 12. - Curves of stress against rupture time at 1500° and 1600°F from normal rupture tests on the heat of Inconel 700 alloy used in the investigation, showing at the stresses used for the overheat tests, the ranges in rupture times predicted by the available data. (See text for explanation of range at 1500°F.)

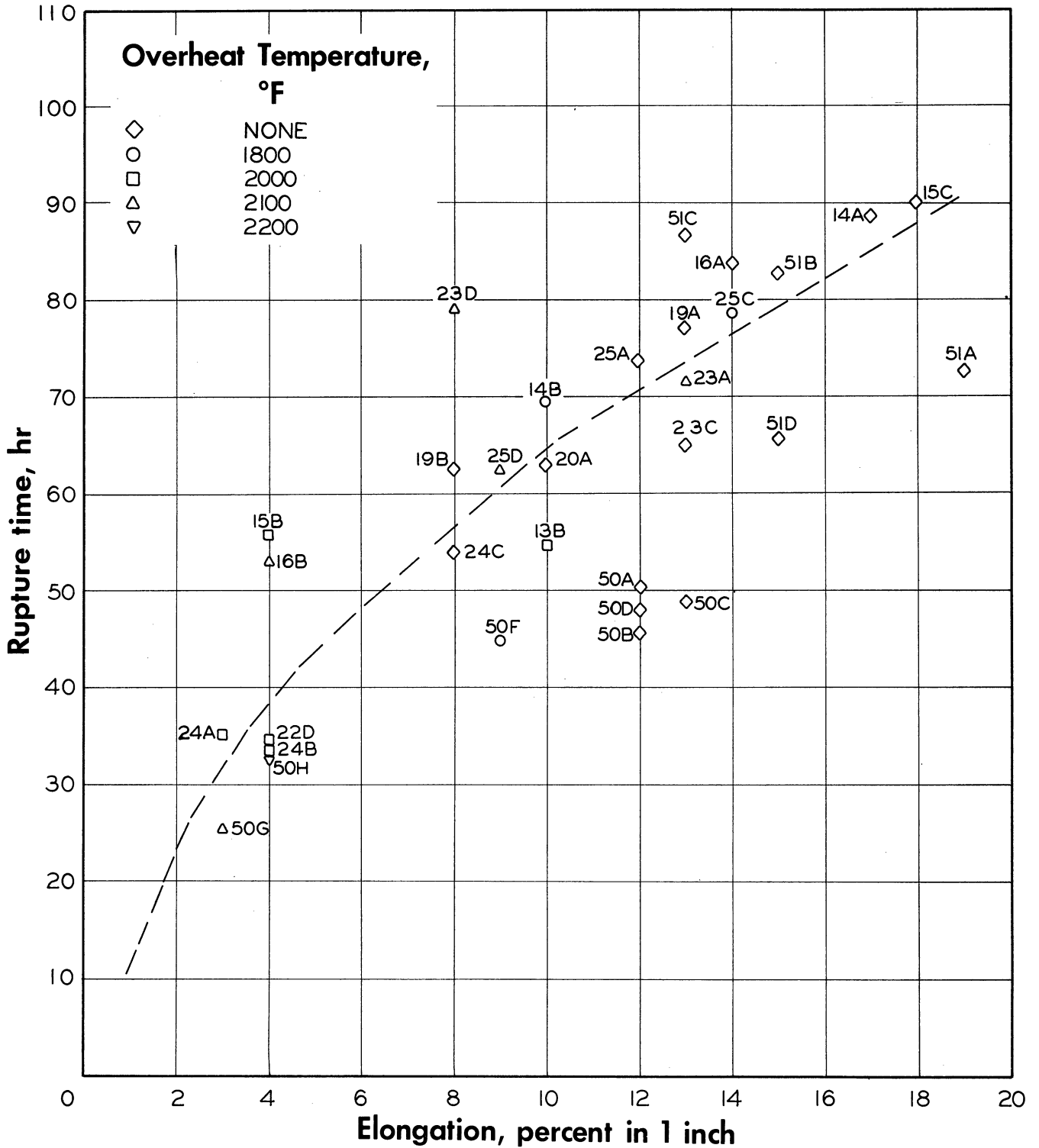
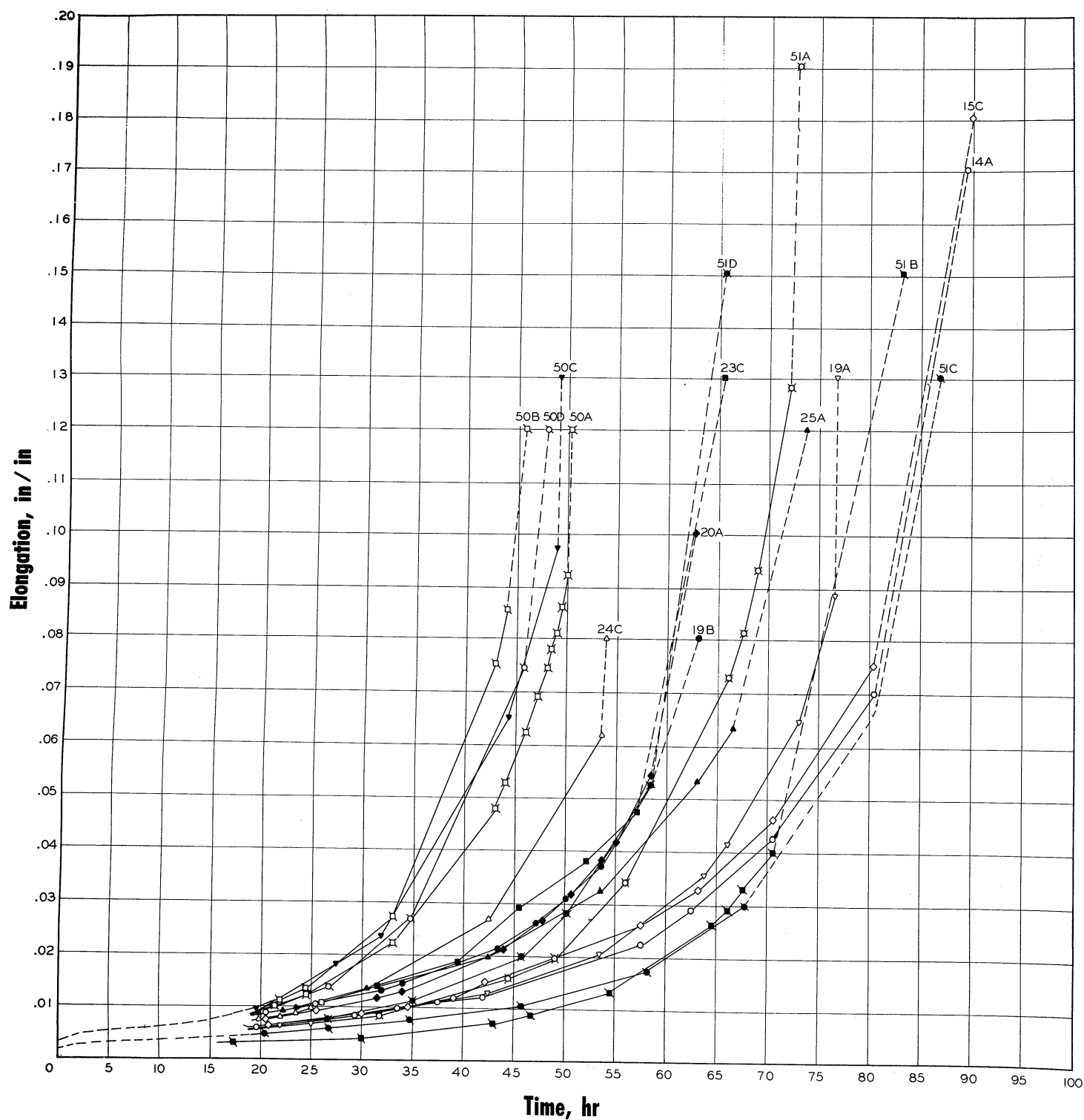
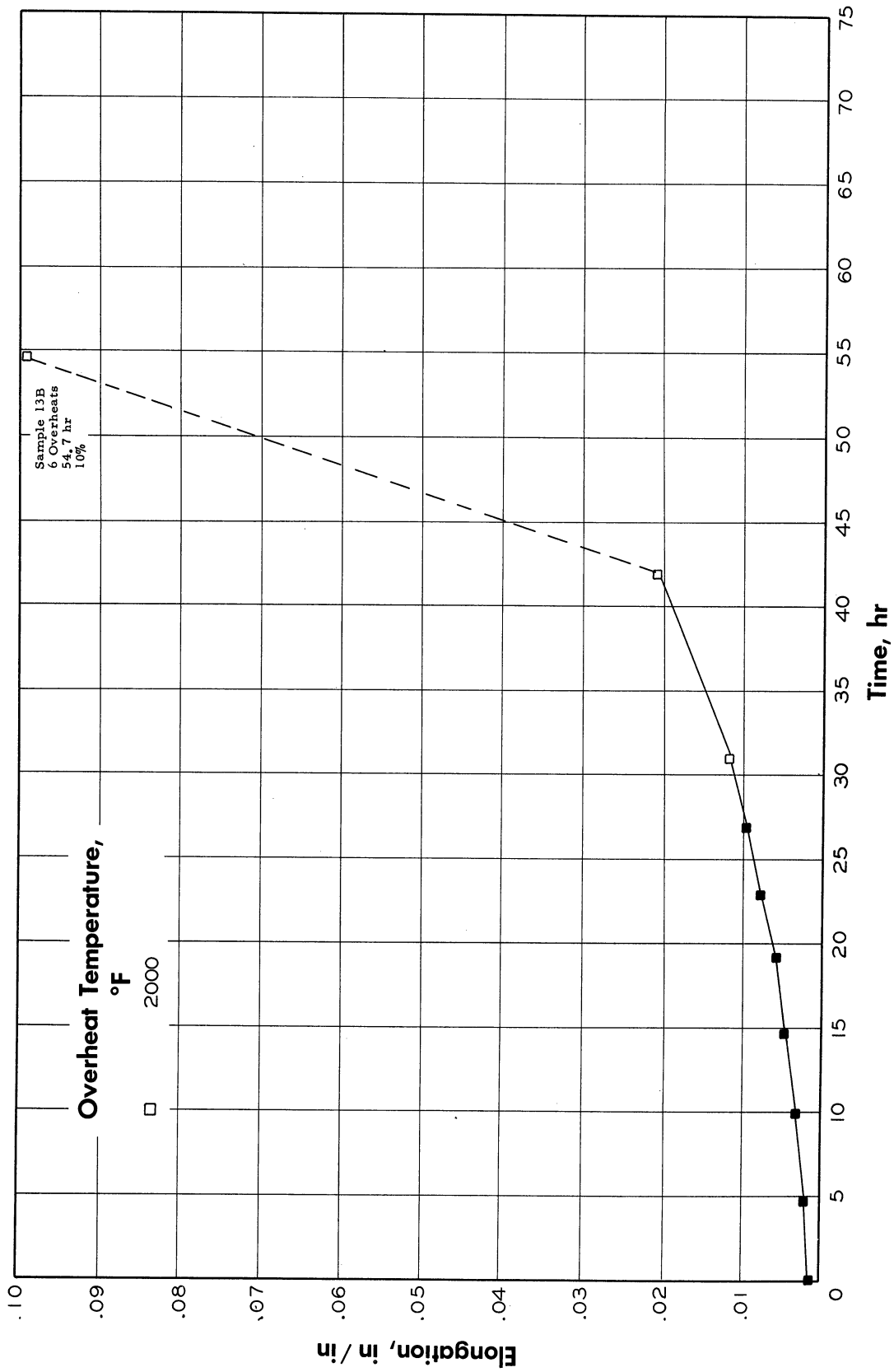


Figure 13. - Rupture time against elongation at fracture for tests on Inconel 700 alloy at 1600°F and 29,000 psi under the indicated test conditions. Specimen numbers indicated for each point.



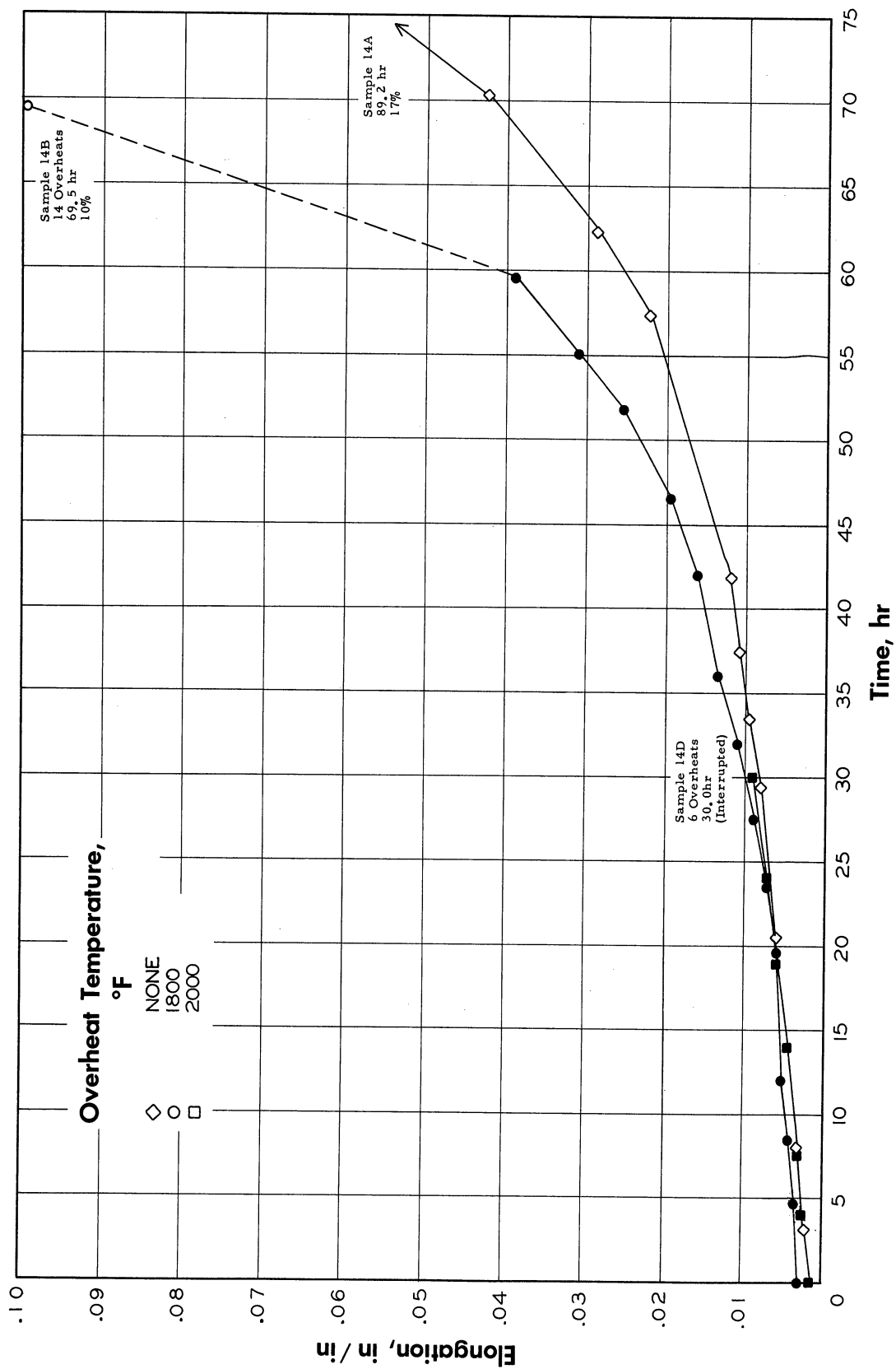
(a) Normal creep rupture tests showing complete curves terminating at final rupture time and elongation. (Data omitted for first 20 hours for clarity.)

Figure 14. - Comparative creep curves at 1600°F and 29,000 psi for Inconel 700 alloy for the indicated test conditions. Solid points indicate period of application of overheats. Sample number, number of overheats, rupture time, and elongation at fracture indicated for each curve.



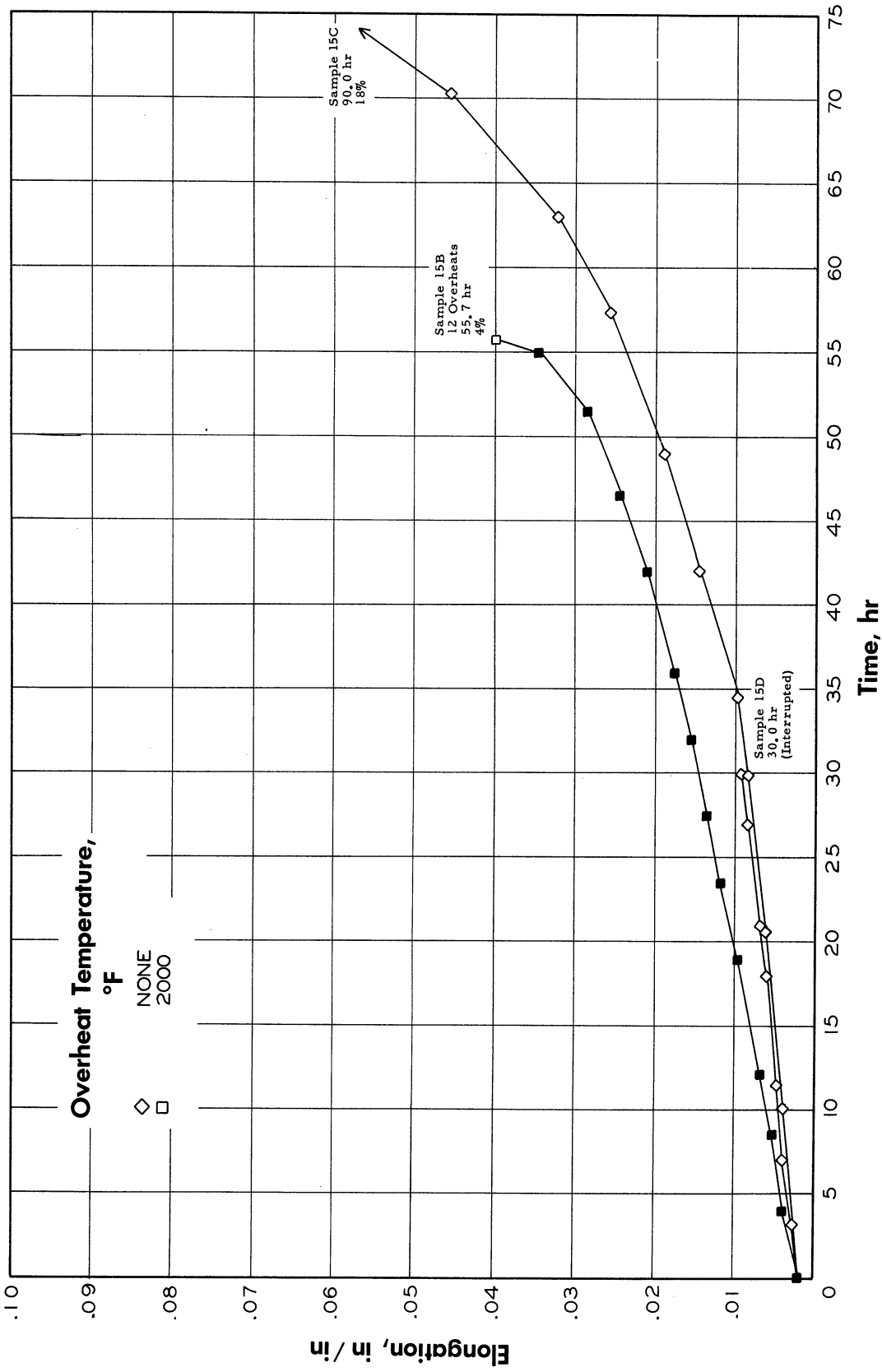
(b) Tests from bar 13.

Figure 14. - Continued. Comparative creep curves at 1600°F and 29,000 psi for Inconel 700 alloy for the indicated test conditions. Solid points indicate period of application of overheats. Sample number, number of overheats, rupture time, and elongation at fracture indicated for each curve.



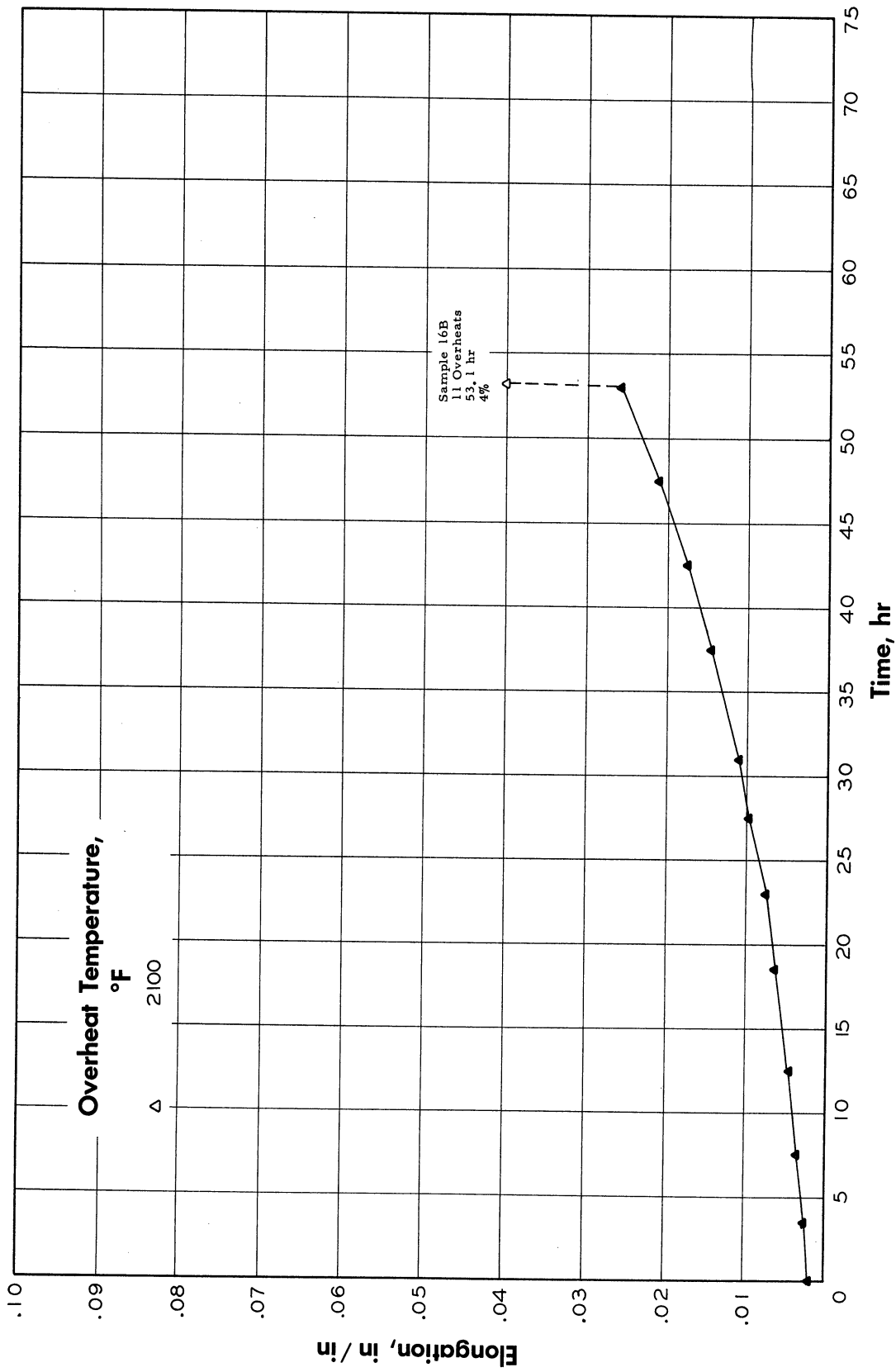
(c) Tests from bar 14.

Figure 14. - Continued. Comparative creep curves at 1600°F and 29,000 psi for Inconel 700 alloy for the indicated test conditions. Solid points indicate period of application of overheats. Sample number, number of overheats, rupture time, and elongation at fracture indicated for each curve.



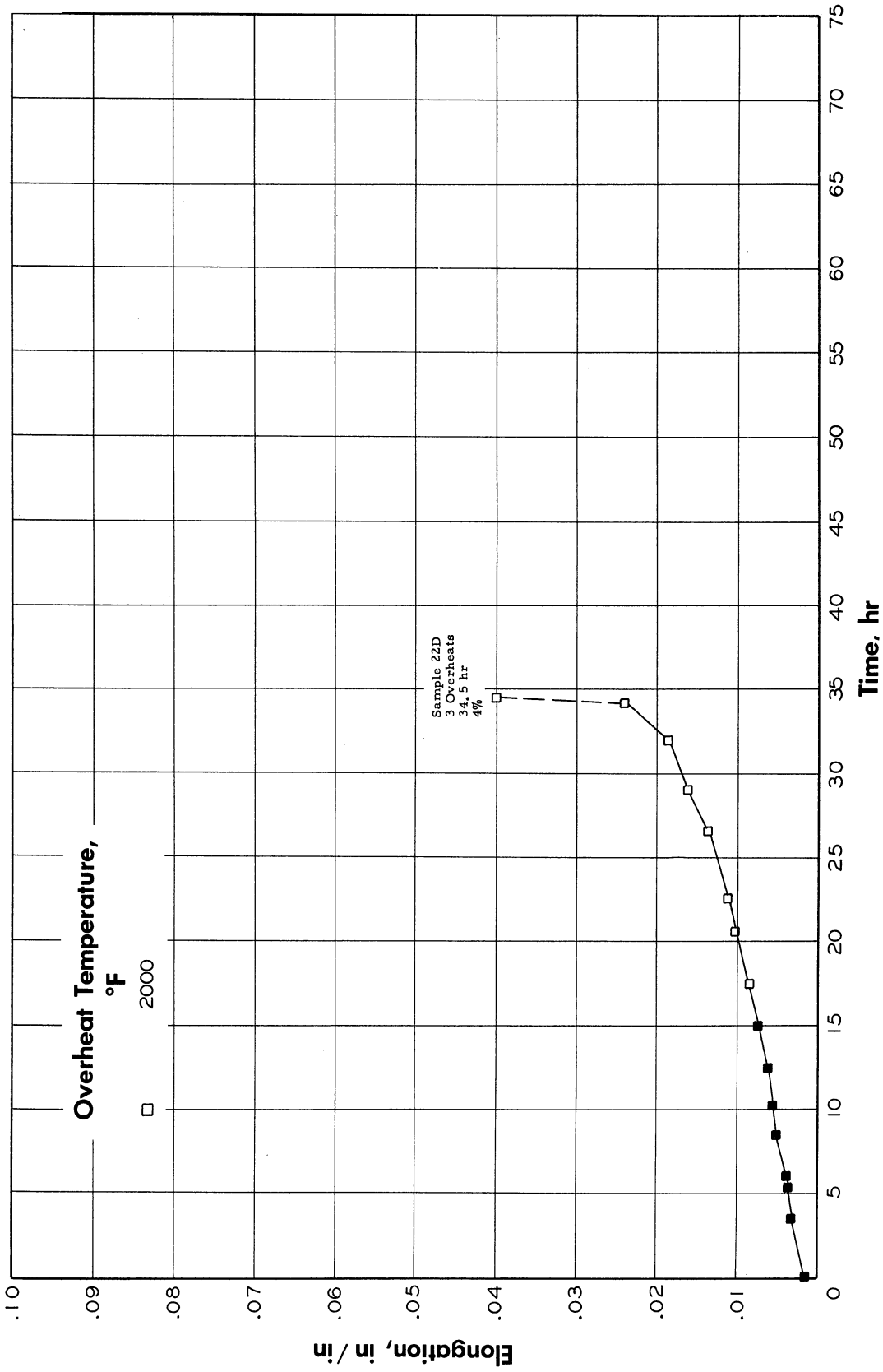
(d) Tests from bar 15.

Figure 14. - Continued. Comparative creep curves at 1600°F and 29,000 psi for Inconel 700 alloy for the indicated test conditions. Solid points indicate period of application of overheats. Sample number, number of overheats, rupture time, and elongation at fracture indicated for each curve.



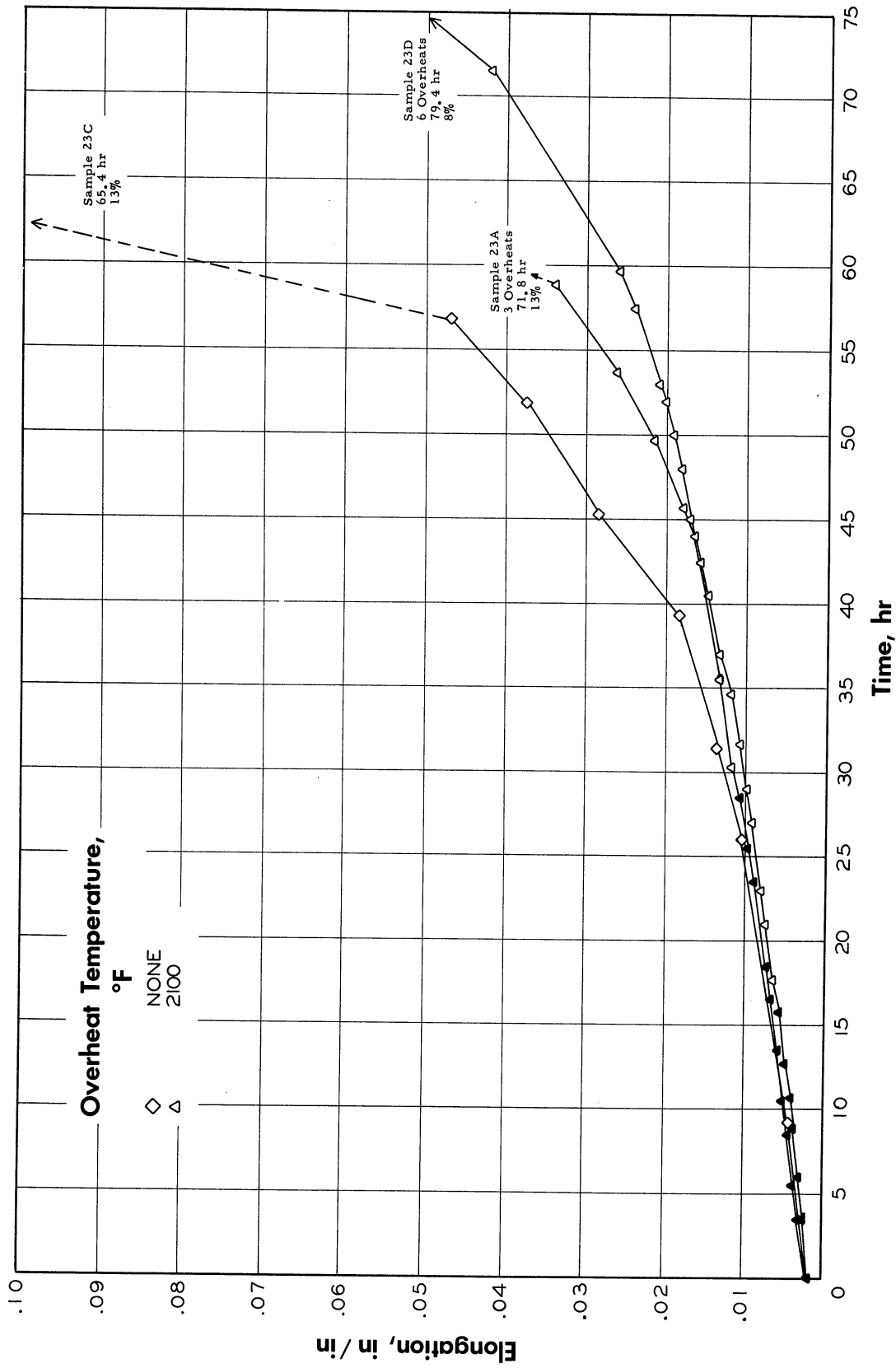
(e) Tests from bar 16.

Figure 14. - Continued. Comparative creep curves at 1600°F and 29,000 psi for Inconel 700 alloy for the indicated test conditions. Solid points indicate period of application of overheats. Sample number, number of overheats, rupture time, and elongation at fracture indicated for each curve.



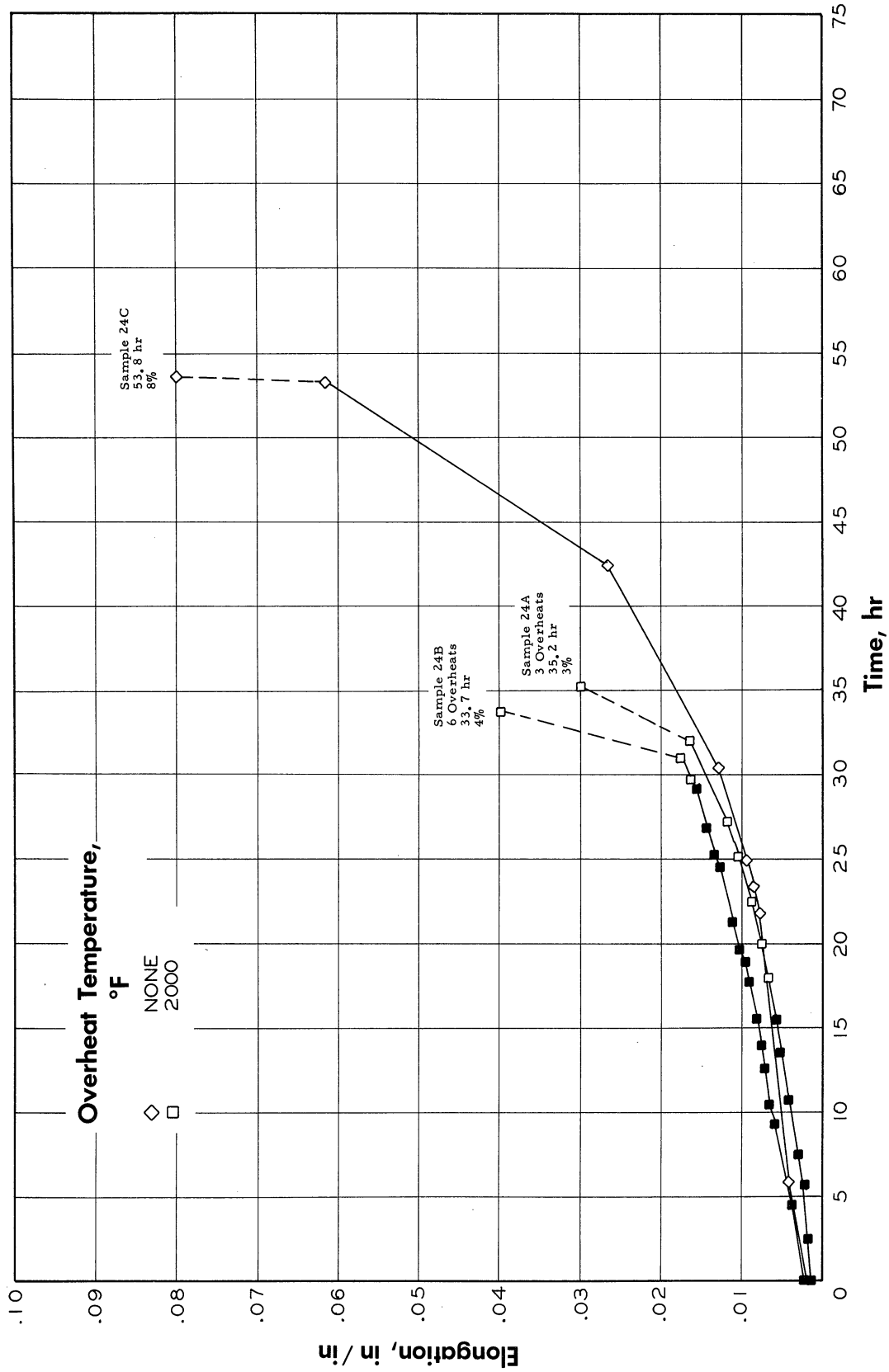
(f) Tests from bar 22.

Figure 14. - Continued. Comparative creep curves at 1600°F and 29,000 psi for Inconel 700 alloy for the indicated test conditions. Solid points indicate period of application of overheats. Sample number, number of overheats, rupture time, and elongation at fracture indicated for each curve.



(g) Tests from bar 23.

Figure 14. - Continued. Comparative creep curves at 1600°F and 29,000 psi for Inconel 700 alloy for the indicated test conditions. Solid points indicate period of application of overheats. Sample number, number of overheats, rupture time, and elongation at fracture indicated for each curve.



(h) Tests from bar 24.

Figure 14. - Continued. Comparative creep curves at 1600°F and 29,000 psi for Inconel 700 alloy for the indicated test conditions. Solid points indicate period of application of overheats. Sample number, number of overheats, rupture time, and elongation at fracture indicated for each curve.

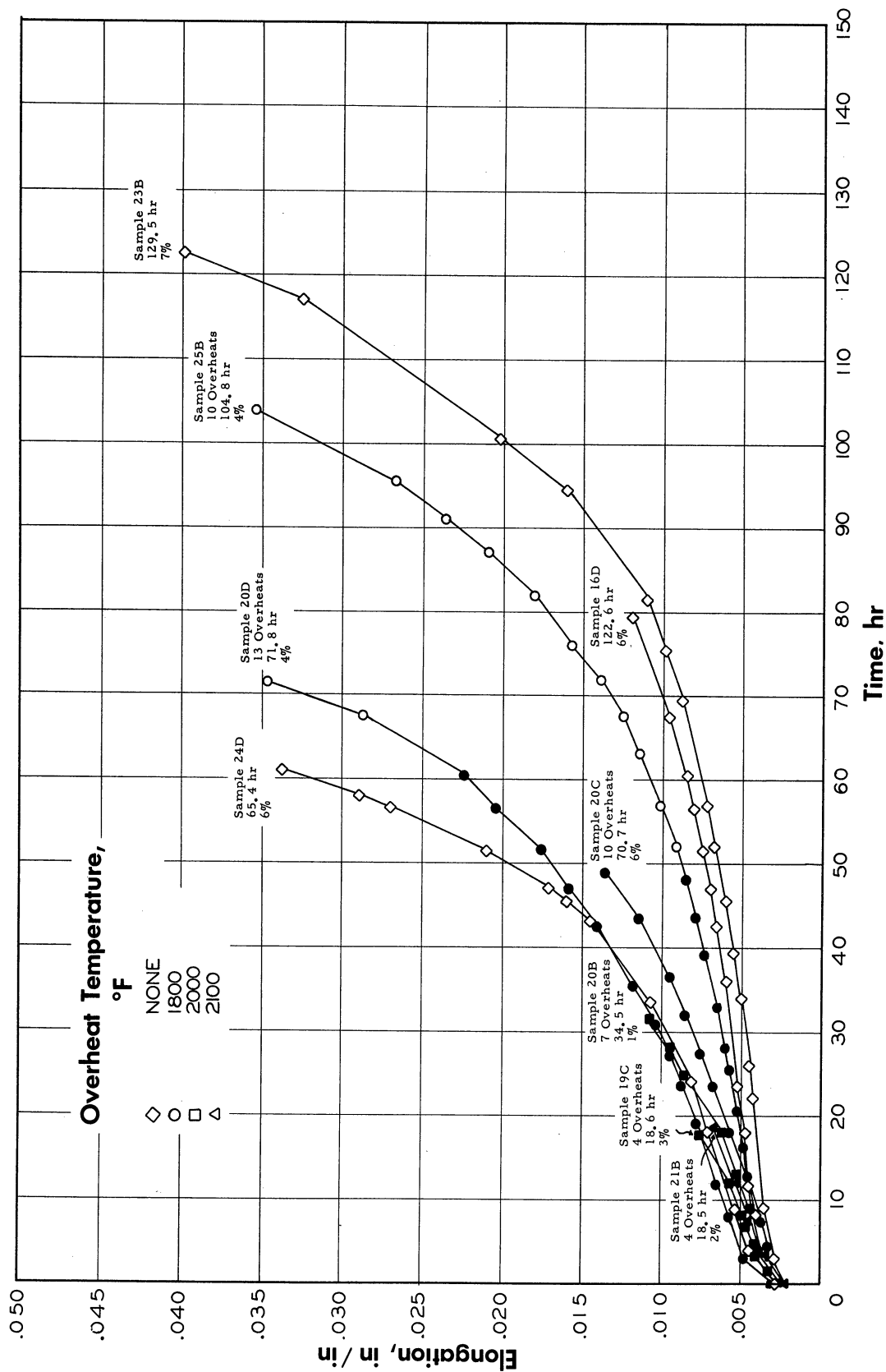
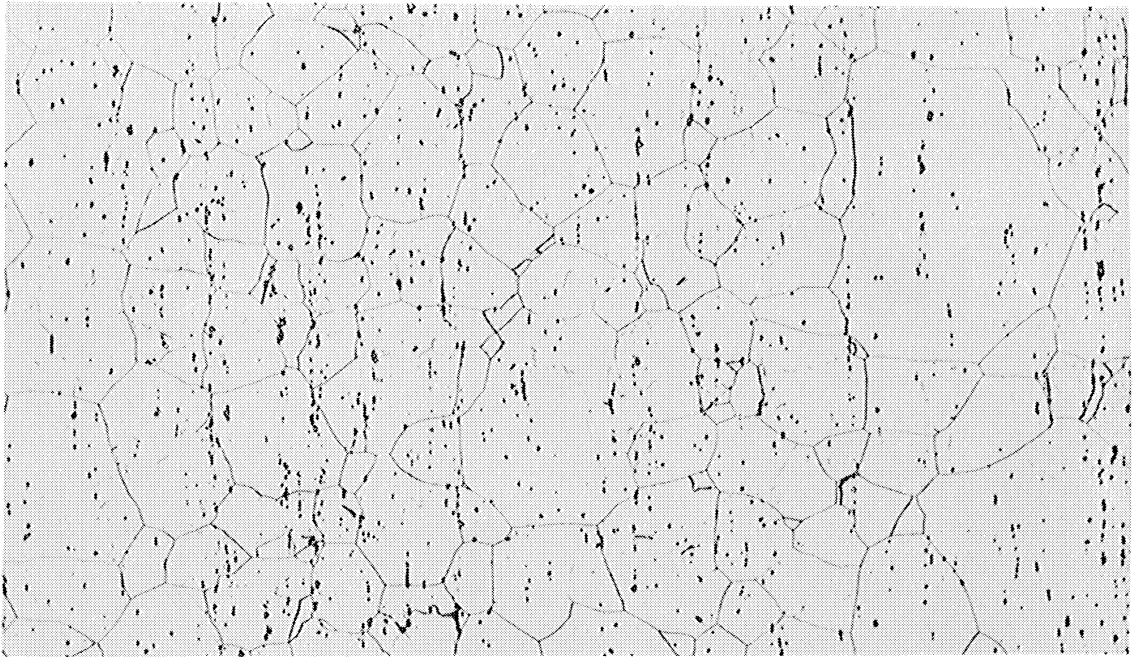
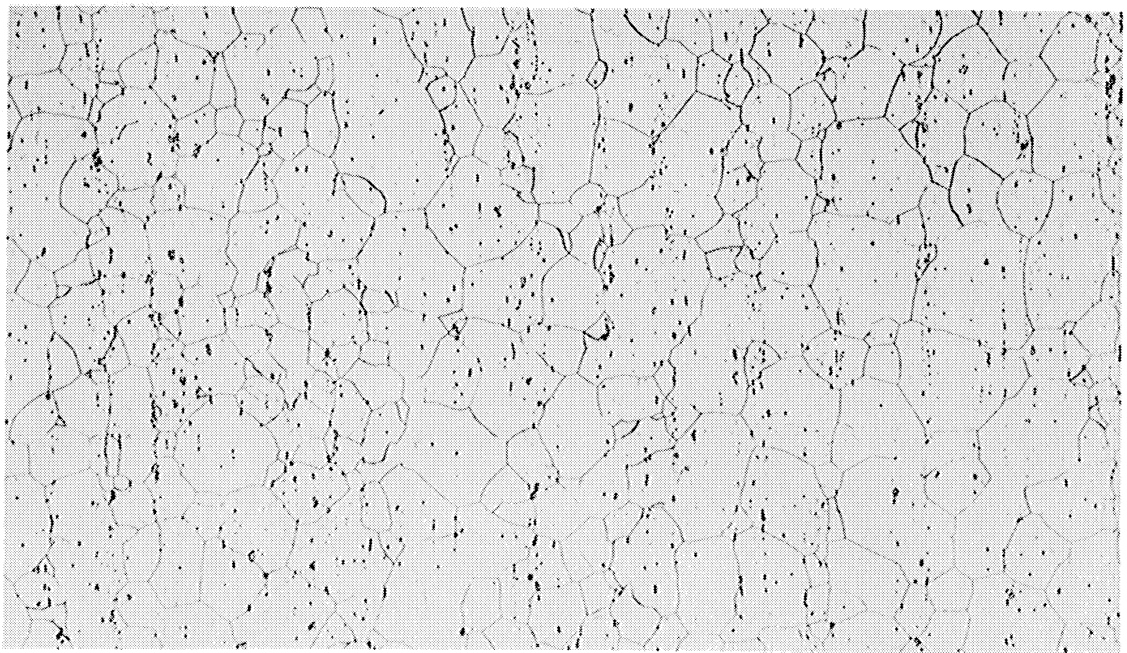


Figure 15. - Comparative creep curves at 1500 °F and 43,000 psi for Inconel 700 alloy for the indicated test conditions. Solid points indicate period of over- heats. Sample number, number of overheats, rupture time, and elongation at frac- ture indicated for each curve.



X100

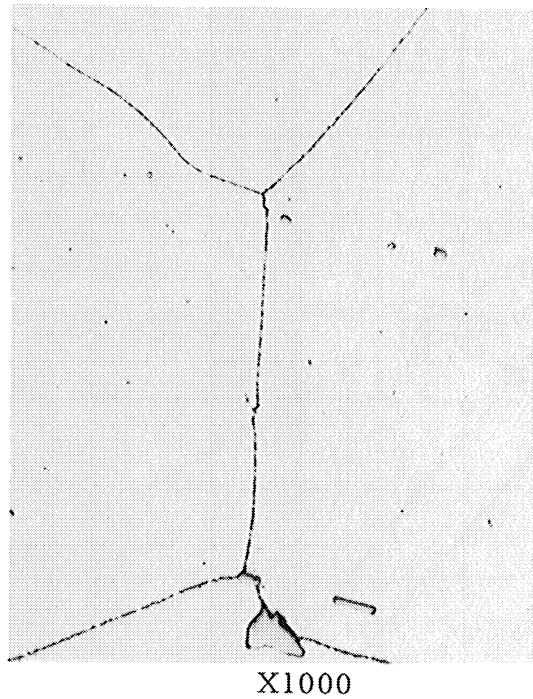
(a) Bar 14, as heat-treated. Rupture time 88-90 hours at 1600°F, 29,000 psi.



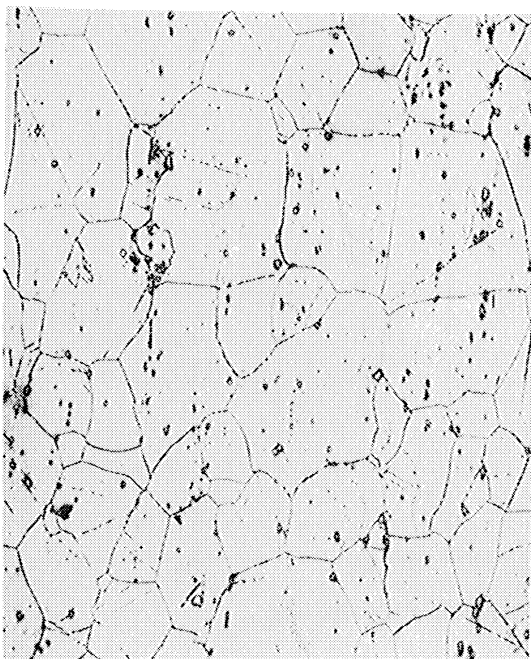
X100

(b) Bar 50, as heat-treated. Rupture time 45-50 hours at 1600°F, 29,000 psi.

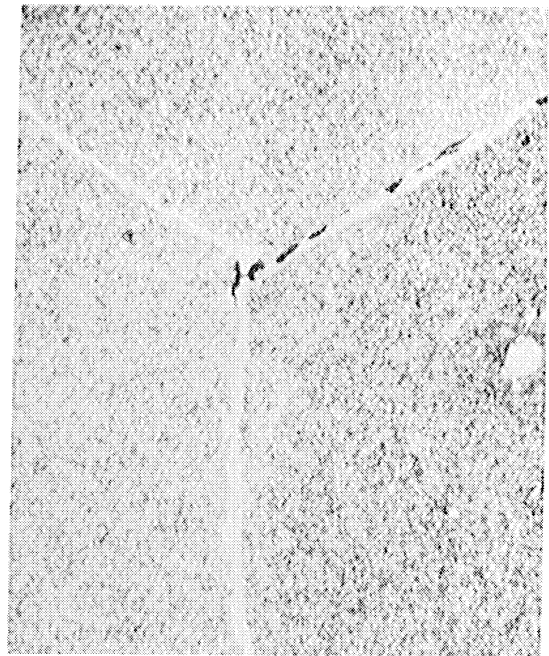
Figure 16. - Microstructures of Inconel 700 alloy after heat treatment and after rupture testing at 1600°F.



(c) Typical grain boundary appearance, as heat-treated.



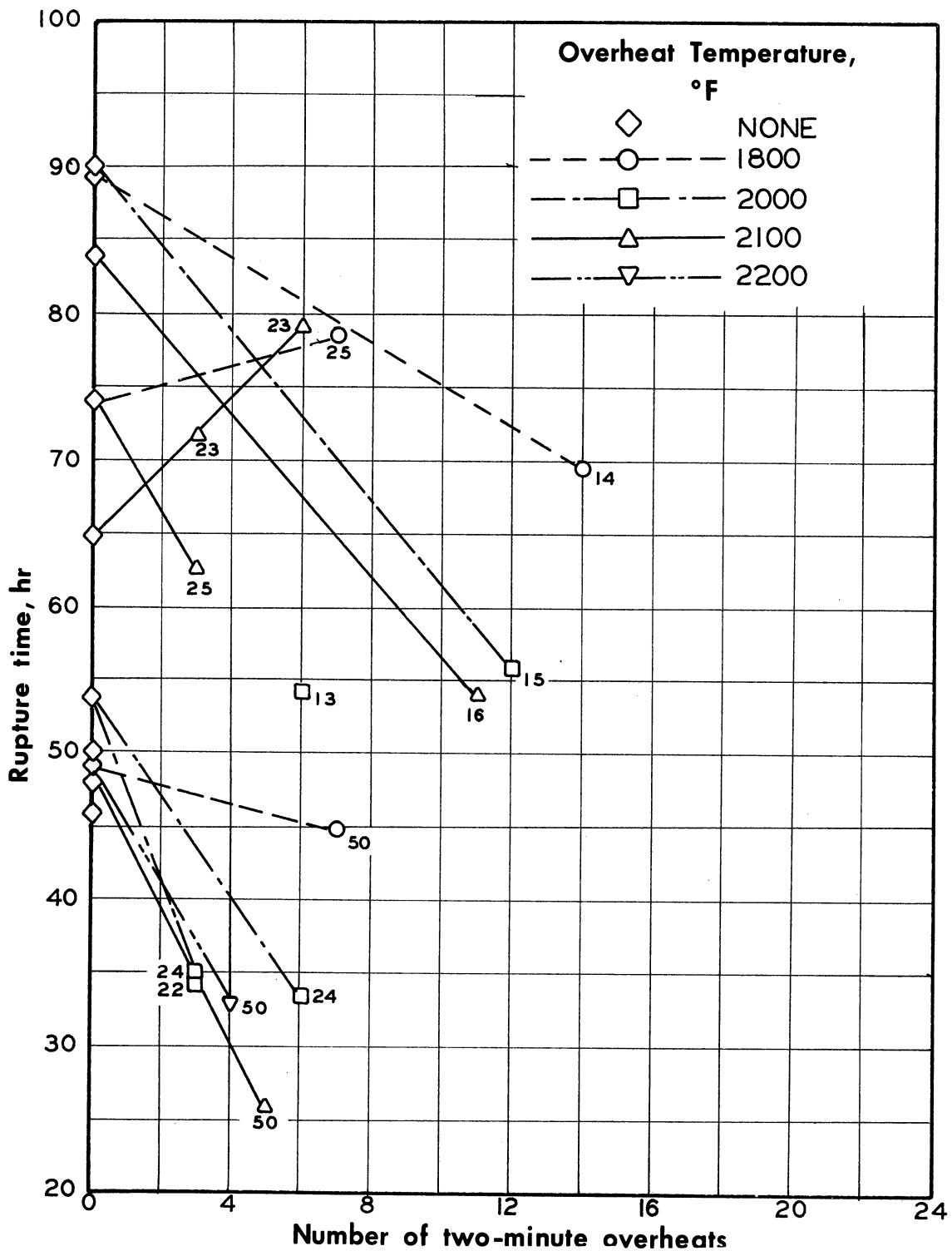
X100



X1000

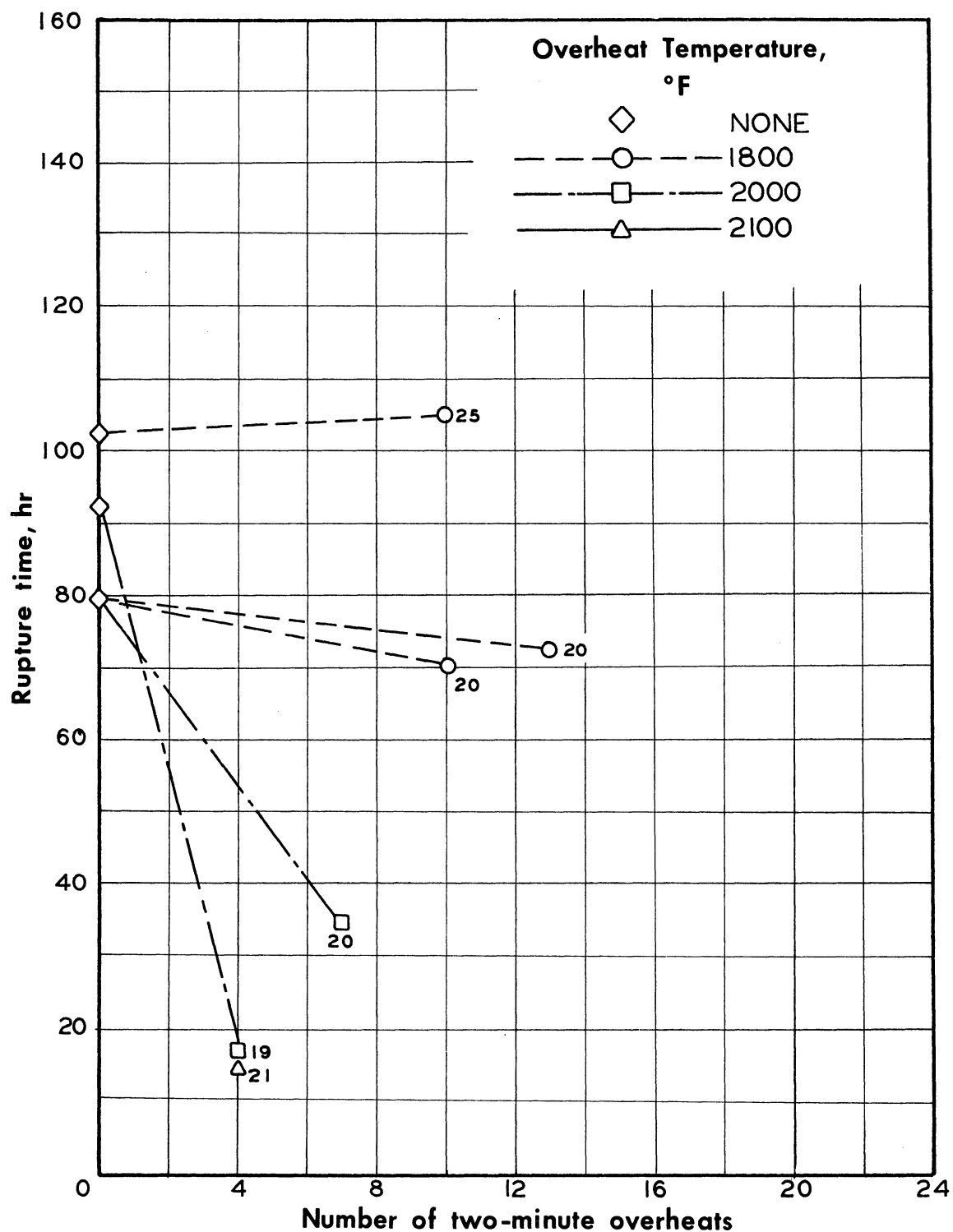
(d) Bar 15 after rupture testing at 1600°F and 29,000 psi.
Rupture time 90.0 hours.

Figure 16. - Concluded. Microstructures of Inconel 700 alloy after heat treatment and after rupture testing at 1600°F.



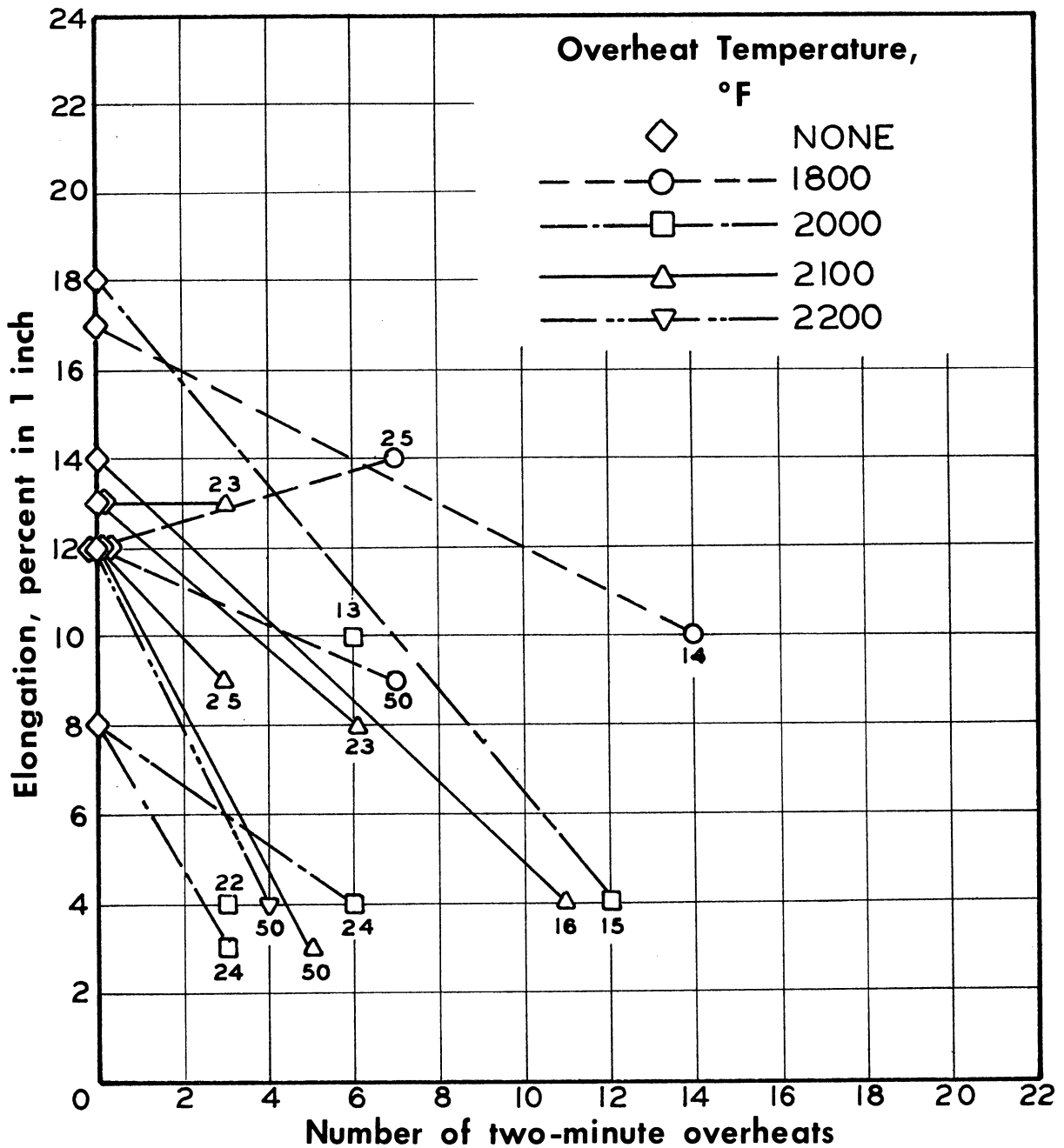
(a) Tests at 1600°F and 29,000 psi.

Figure 17. - Effect of number of overheats on the rupture time of Inconel 700 alloy for the overheat temperatures and base conditions indicated. Overheats were of two minutes duration applied every five hours from the beginning of the test. Bar number indicated for each overheat test. Overheat results compared to normal test from same bar wherever possible.



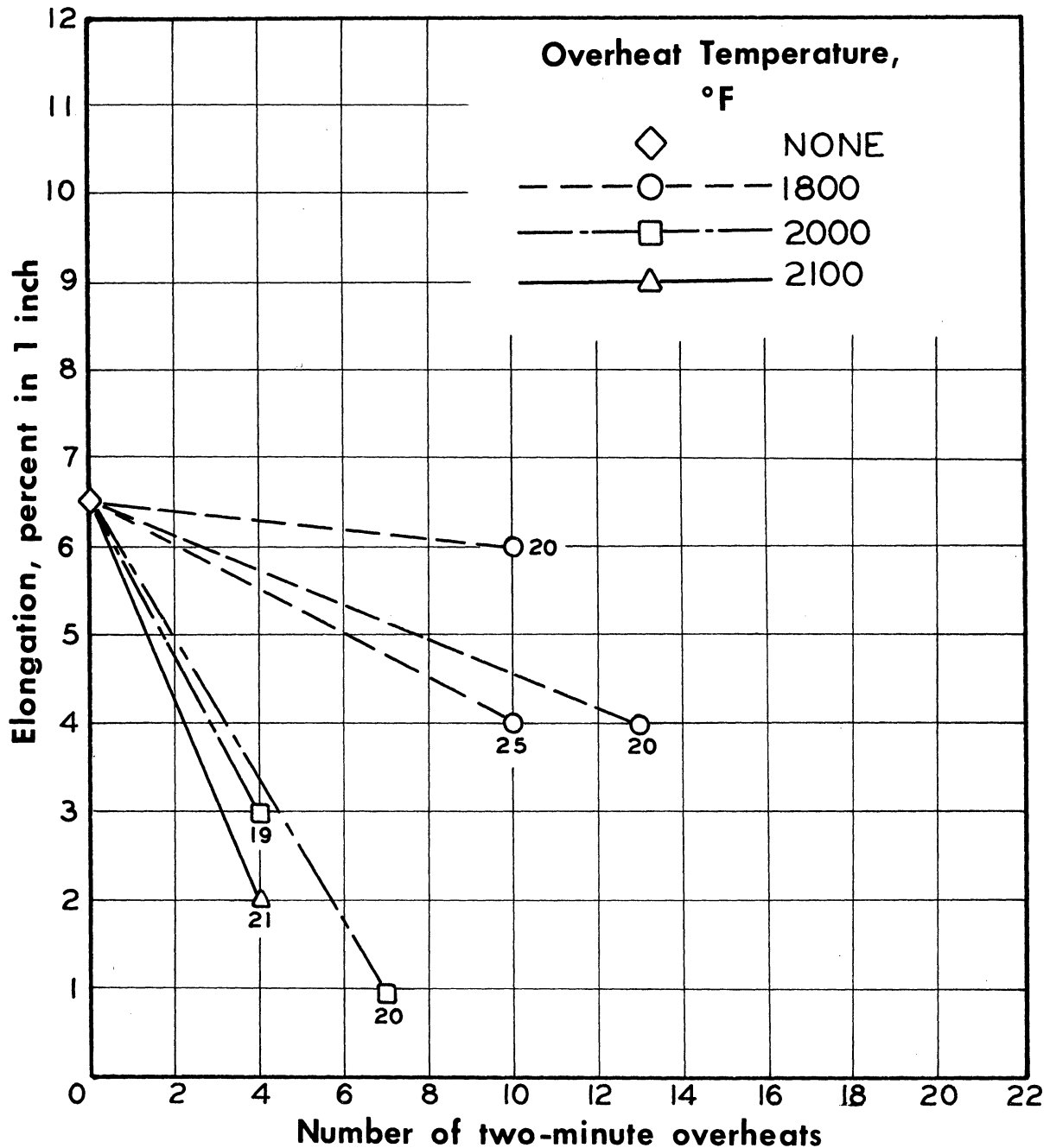
(b) Tests at 1500°F and 43,000 psi. (Values for normal tests estimated.)

Figure 17. - Concluded. Effect of number of overheats on the rupture time of Inconel 700 alloy for the overheat temperatures and base conditions indicated. Overheats were of two minutes duration applied every five hours from the beginning of the test. Bar number indicated for each overheat test. Overheat results compared to normal test from same bar wherever possible.



(a) Tests at 1600°F and 29,000 psi.

Figure 18. - Effect of number of overheats on the elongation at fracture of Inconel 700 alloy for the overheat temperatures and base conditions indicated. Overheats were of two minutes duration applied every five hours from the beginning of the test. Bar number indicated for each overheat test. Overheat results compared to normal test from same bar wherever possible.



(b) Tests at 1500°F and 43,000 psi. (Values for normal tests estimated.)

Figure 18. - Concluded. Effect of number of overheats on the elongation at fracture of Inconel 700 alloy for the overheat temperatures and base conditions indicated. Overheats were of two minutes duration applied every five hours from the beginning of the test. Bar number indicated for each overheat test. Overheat results compared to normal test from same bar wherever possible.

