SUMMARY REPORT

TO

MATERIALS LABORATORY

WRIGHT AIR DEVELOPMENT CENTER

ON

STUDIES OF HEAT-RESISTANT ALLOYS

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FOREWORD

This report was prepared by The University of Michigan Research Institute under USAF Contract Number 33(616)-5466. The contract was initiated under Task Number 73512, with Mr. C. B. Hartley acting as project engineer for the Materials Laboratory, Wright Air Development Center. This report covers work done from March 15, 1958 to June 15, 1959.

The A-286, the "A" Nickel, and the "17-22-A"V Steel used in the work reported under Phase A were supplied gratis by The Allegheny-Ludlum Steel Corporation, The International Nickel Company, and The Timken Roller Bearing Company, respectively. The A-286 used in the work reported under Phase B was supplied by the Materials Laboratory, WADC, and the carbon steels used in Phase B were supplied by the Chemical and Petroleum Panel of the ASTM-ASME Joint Committee on the Effect of Temperature on the Properties of Metals.

ABSTRACT

Results are reported for an investigation that was carried out to show the effect f hot working on the structure and creep-rupture properties of heat-resistant lloys. Three types of materials were studied: a commercially-pure metal ("A" ickel), a precipitation-strengthened, austenitic alloy (A-286), and a low-alloy, rritic steel ("17-22-A"V). The influence of hot-working conditions on properties as determined for "A" Nickel and A-286 Alloy under a previous contract. This eport describes extensive work aimed at correlating the structures of these alloys ith the properties.

In the case of "A" Nickel rupture tested at 1100°F, near the static recrystallization mperature, the studies indicated that substructures were the major factor affecting rength and that internal lattice strains as measured by hardness were relatively nimportant. At 800°F the creep resistance of "A" Nickel appeared to depend on nternal strains and substructures more or less equally. Some uncertainty remained s to the exact influence of substructures because they could not be measured in most the specimens, presumably because the sub-boundaries in most cases were not harp and well-defined. An etch-pit technique, electron microscopy, Berg-Barrett -ray reflection microscopy, micro-beam back-reflection X-ray diffraction, and ansmission electron microscopy were used in the attempt to measure substructures.

For A-286 Alloy the rolling temperature influenced rupture properties at 1200°F nd 1350°F more than the amount of reduction. Rough correlations between strength nd grain size were found, but it was not clear whether solution of excess phases grain size *per se* was the strength-determining factor. Small variations in the loy's response to a standard heat treatment as measured by hardness and electron icroscopy did not appear to affect the creep-rupture properties. A small effect G phase in the grain boundaries was observed.

The rolling experiments on "17-22-A"V Steel demonstrated that beneficial fects on both rupture strength and ductility at 1100°F can be produced by working oroughly-solutioned austenite at temperatures too low for simultaneous recrystallization. he results suggested that the more severely strained the austenite the greater the mprovement in properties. Electron microscopic examination indicated that changes the bainite structure and a reduction in the amount of carbides at prior austenite ain boundaries were related to the property changes.

A second phase of the investigation dealt with the effects of strain-aging-type enomena on strength at high temperatures. Constant-strain-rate tension tests dicated that carbon steels showing the familiar strain-aging peak at about 400°F strength versus temperature curves also have higher-than-average strengths under e slow-strain-rate and small-strain conditions of 100- to 1000-hour creep-rupture sts. Evidence of strain aging in A-286 Alloy was found as serrated stress-strain rves obtained in standard tensile tests. After an 1800°F solution treatment serrated rves were observed up to 1200°F, whereas 1000°F was the highest temperature r serrations in material solution treated at 1650°F. This was interpreted to mean at titanium- and nitrogen-bearing compounds experienced greater solution at 1800°F, creasing the alloy's susceptibility to strain aging. It was suggested that forces of emical attraction between nitrogen atoms and silicon, aluminum, or titanium atoms abilize Cottrell-type atmospheres, permitting them to be effective at higher-than-rmal temperatures.

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INTRODUCTION

This summary report covers research conducted at The University f Michigan Research Institute for the Materials Laboratory, Wright ir Development Center. The work was authorized under Air Force ontract No. AF 33(616)-5466, and it covered the period of March 15, 1958 o June 15, 1959.

There is invariably a range in creep-rupture properties of heat-esistant alloys under production conditions. This can lead to several ifficulties. The restriction from the need to design to minimum properties o be expected is obvious. More insidious is the possibility that properties ay be even lower than expected. The advantages of being able to produce aterials consistently and reliably with maximum properties are obvious. he ranges in possible properties are large enough to be highly significant n air or space vehicles.

There is no question but that a major cause of the range in properties is ue to variations in the microstructure arising from variations in the conditions production. Usual practice is to produce alloys in their final form using the ost economical means consistent with freedom from cracks and other flaws. "standard" heat treatment is then applied. It is now well recognized that e response to the heat treatment can vary with the production conditions nd that this is a major source of variability. Usually this source of variability large enough to mask variation arising from normal variation in chemical omposition.

In the work presented in this report, controlled hot working was used as source of initial structural variation. Both the influence of hot working on roperties in the hot-worked condition and its influence on response to heat eatment were studied for appropriate materials. Heat treatments when used ere not varied for a given alloy and were intended to show the influence of orking conditions on response to heat treatment. The experimental materials ncluded (1) a commercially pure metal ("A" Nickel), (2) a precipitation-trengthened, austenitic alloy (A-286), and (3) a low-alloy, ferritic steel 17-22-A" V).

The influence of hot-rolling conditions on the creep-rupture properties of A" Nickel in the as-rolled condition and the influence of hot-rolling conditions n the creep-rupture properties of A-286 alloy with a standard heat treatment d been determined in previous work (Ref. 1). The study of the influence of hot-orking conditions on "17-22-A" V steel was conducted for this report, except for ome previous preliminary work.

In addition to the study of hot-working effects, another phase of research was carried out on a subject that has been receiving more and more attention, namely, the influence of strain-aging-type reactions on high-temperature properties. The strain aging study was done on two heats of AISI 1020 carbon steel with known differences in susceptibility to strain aging and on a heat of A-286 alloy that Captain Domian had used in a hot hardness study at the Materials Laboratory of the Wright Air Development Center. Constant-strain-rate tension tests were run over a wide temperature range so that curves of stress for a given strain versus temperature could be constructed for each alloy under various conditions of heat treatment and strain rate. The occurrence of a peak or an inflection in the curve was assumed to be caused by strain aging. Electron microscopy was used in an effort to detect the structural features controlling the strain-aging-type reactions. During the year an automatic constant-strain-rate unit constructed under an earlier part of the contract was adjusted to operate satisfactorily for strain rates ranging between tensile tests and creep tests, and was used to obtain part of the data.

EXPERIMENTAL MATERIALS

Three types of materials were used to investigate the influence of hot working on high-temperature properties: (1) A commercial grade of pure nickel ("A" Nickel) was used to study the influence of working conditions on properties in the as-worked condition. It was assumed that the behavior of "A" Nickel would be typical of metallurgically simple materials where solid-solution strengthening, precipitation strengthening, and other complex phase relationships are minor. (2) A precipitation-strengthened, austenitic alloy (A-286) was chosen as an example of those alloys that are customarily solution treated and aged after hot working. The study of this material was designed to show the extent to which prior history effects can influence creep-rupture properties by altering the alloy's response to a standard heat treatment. (3) A ferritic, low-alloy, steel ("17-22-A" V Steel) was included in the investigation for the purpose of testing certain hypotheses for using controlled hot working to produce microstructures with a superior combination of high-temperature strength and ductility, unattainable by heat treatment alone.

For the strain aging study, two AISI 1020 carbon steels and A-286 alloy (Heat 82073) were used. One of the carbon steels (Steel C) was deoxidized with silicon and was susceptible to strain aging in all conditions of heat treatment. The other carbon steel (Steel F), deoxidized with both aluminum and silicon, and, in contrast, was not susceptible to strain aging, except after certain heat treatments. The influence of strain aging on the properties of A-286 alloy had not been established previously, except that hot hardness tests by Captain Domain had indicated strain aging.

The materials for the hot-working study were supplied gratis by the following organizations: "17-22-A" V steel from The Timken Roller Bearing Company, A-286 alloy (Heat 21030) from The Allegheny-Ludlum Steel Corporation, and "A" Nickel from The International Nickel Company, Inc.

The plain carbon steels (Steel C and Steel F) used in the strain aging study were obtained from the Chemical and Petroleum Panel of the ASTM-ASME Joint Committee on the Effect of Temperature on the Properties of Metals. The A-286 alloy (Heat 82073) used in the strain-aging experiments was supplied by the Materials Laboratory, Wright Air Development Center from the stock used by Captain Domian to study strain aging by hot-hardness tests.

The chemical compositions were reported by the manufacturers as follows:

Alloy	C	Mn	Si	Cr	Ni	V	Fe	Other
'A" Nickel Ht N9500A)	0.06	0.27	0.06	-----	99.46 (Ni+Co)	----	0.09	0.03Cu;0.004Mg 0.008S;0.024Ti $<$0.005Zr;0.003B
-286 Ht 21030)	0.06	1.35	0.47	14.58	25.3	0.21	Base	2.00Ti;1.38Mo 0.17Al;0.004B
-286 Ht 82073)	0.03	1.27	0.62	14.58	25.44	0.59	Base	0.008N;0.12Al 0.37Co:Nom. Ti
'17-22-A" V Ht 11833)	0.29	0.70	0.71	1.43	0.31	0.81	Base	0.51Mo
.020 Steel C	0.20	0.68	0.27	-----	-----	----	Base	0.015Al;0.0048N 0.028P;0.034S
.020 Steel F	0.19	0.68	0.24	-----	-----	----	Base	0.053Al;0.0046N 0.026P;0.036S

PHASE A - INFLUENCE OF HOT WORKING ON STRUCTURE AND CREEP - RUPTURE PROPERTIES

EXPERIMENTAL PROCEDURES

The general experimental approach used may be summarized as follows: (1) The subject alloys were rolled with wide ranges of temperature and reduction and allowed to air cool. (2) The nickel was left in the as-rolled condition, the A-286 alloy was solution treated and aged, and the "17-22-A" V steel was tempered. (3) The alloys were creep-rupture tested in the temperature range of maximum interest. (4) Structural studies were carried out before-- and sometimes after-- creep-rupture testing to correlate the properties with the structures.

Two of the materials studied, "A" Nickel and A-286 alloy, were carried through the first three steps under a previous WADC contract (Ref. 1). All of the work on the third material, "17-22-A" V steel, except for some preliminary experiments, was done under the present contract.

Study of Structures in "A" Nickel and A-286 Alloy

Light Microscopy

The gross structural features of rolled "A" Nickel and rolled plus heat treated A-286 were examined previously and described in Reference 1. The observations at that time were limited primarily to variations in grain size resulting from heating for rolling and from recrystallization during rolling. In the present work light microscopy was used to determine the extent to which recrystallization occurred during rupture testing "A" Nickel at 1100°F and 20,000 psi. Representative test specimens were sectioned, polished, and etched after rupture, and the approximate volume percent of recrystallization was determined by a lineal analysis.

In the A-286 alloy, quantitative grain size measurements were made to test for the possibility of correlations between grain size and rupture life at 1200° or 1350°F. The grain sizes were determined by counting grains in photomicrographs at convenient magnifications and converting the data into the number of grains per square inch at 100 diameters. These numbers were then converted to ASTM Grain Size Numbers.

Electron Microscopy

The electron microscope was used to check for possible structural details in "A" Nickel not visible under the light microscope. However, the major use for electron microscopy was to study the age-hardening precipitate and excess phases in the A-286 alloy.

Two problems were encountered in the preparation of A-286 specimens for lectron microscopy. The first was to find a new method for preparing a olished surface free of cold worked metal; the usual method of electropolishing esulted in excessive attack on a certain grain boundary phase. The second roblem was to find an etchant that would reveal the age-hardening precipitate hich in A-286 is very small and closely spaced. The following procedure was ound to give satisfactory results:

1. Wet grind on successively finer grades of silicon carbide abrasive paper, finishing with the No. 600 grit.

2. Remove the No. 600-grit scratches with a short-nap cloth wheel charged with a diamond abrasive.

3. Subject the specimen to argon-ion bombardment until the outer layers of disturbed metal are removed.

4. Etch the specimen for 20 seconds by swabbing with glyceregia composed of five parts glycerine, four parts concentrated HCl, and one part concentrated HNO_3.

ollodion replicas were stripped from specimens prepared in the manner ove, and after 0.34 micron diameter polystyrene balls were placed on the eplicas they were shadowed with palladium metal.

echniques Investigated for Studying Substructures in "A" Nickel

A major expenditure of time and effort was spent in an effort to establish chniques for identifying and measuring substructures in "A" Nickel. Data the literature (Ref. 2) and data from previous work in this laboratory (Ref. 1) dicated that the substructure as influenced by rolling conditions was the ntrolling factor influencing creep-rupture properties. The techniques vestigated included etch pits in conjunction with light microscopic examination, o X-ray diffraction techniques, and transmission electron microscopy.

Etch-Pit Technique: Under special conditions which are not fully understood bgrain boundaries sometimes can be seen under the light microscope as a w of etch pits in polished and etched specimens. Factors such as thermal story and soluble impurity content seem to be important (Ref. 3). This chnique was tried on specimens from all of the rolled nickel bars. A 40-rcent aqueous solution of H_3PO_4 was the most successful etch although sub-ain boundaries were revealed in only a very few specimens. For reasons scussed later in the report, completed creep specimens were also examined this method.

Berg-Barrett X-ray Reflection Microscopy: Each grain is composed of a number of small crystalline volumes differing slightly in orientation. The character of these constitutes the substructure. Because they do differ in orientation each diffracts X-rays in slightly different directions and offers the possibility of measuring substructures. In Berg-Barrett X-ray reflection microscopy, the diffraction spots from an individual grain on a photographic film are magnified for studying the diffraction effects of substructures.

The procedure is to record images of diffraction spots on high-resolution photographic plates (see Figure 1a) and then to magnify the images optically up to the limit of resolution of the diffraction conditions or the photographic emulsion, whichever is lower. In this case, the diffraction conditions, i.e., the extremely small angular differences between subgrains, made it impossible to resolve the spots from the finest subgrains that were visible under the light microscope.

Micro-Beam, Back-Reflection X-ray Diffraction: In back-reflection X-ray diffraction, a Debye ring is obtained. The rings are composed of diffraction spots from the individual grains. Orientation differences due to substructures in each grain would cause each spot to be composed of a number of smaller spots from the subgrains. The use of a monochromatic microbeam reduces the number of grains diffracting to a small number so that overlapping of spots from many grains is prevented. The proper arrangement of the recording photographic film takes advantage of the divergence of the diffracted X-ray beam to resolve the smaller spots related to the substructure. Figure 1b shows schematically the specific arrangement used in determining the possible usefulness of this method in studying substructures in the rolled "A" Nickel specimens.

Transmission Electron Microscopy: A research technique developed rather recently for studying the internal structure of metals is transmission electron microscopy. It is identical to ordinary electron microscopy, except that the metal specimen itself is used instead of a replica of the specimen. Since an electron beam cannot penetrate metals very deeply the specimen must be reduced to a very thin foil. In properly thinned foils individual dislocations can be seen (Ref. 4); hence, any type of substructure consisting of an array of dislocations presumably could be studied by this method.

The main task in applying transmission electron microscopy to the present problem was to cut a wafer from a bar of rolled nickel and reduce it to a foil by grinding and electropolishing in such a way that the original structure was not altered by unintentional heating or deformation. The grinding was carried out with special diamond abrasive wheels on a grinder specially designed and built for a University dental laboratory for the study of thin sections of teeth. Electropolishing was done in an electrolyte consisting of 80 percent glacial acetic acid and 20 percent perchloric acid. The foils were examined in a Phillips electron microscope with an accelerating voltage of 75KV.

Hardness Measurements

Hardness measurements were taken on completed creep and rupture specimens of "A" Nickel to determine the relative amounts of softening by recovery during the test. The measurements were Vickers Hardness Numbers taken with a 30 Kg load, and the indentations were made in the threaded portion of the specimen near the fillet where little or no creep deformation occurred. The objective was to obtain a measure of thermally-induced recovery independent of hardness changes induced by creep.

Hot Rolling Experiments on "17-22-A" V Steel

The object of the experiments on "17-22-A" V steel was to produce varying austenite grain sizes after a thorough solution treatment above the coarsening temperature of the austenite. The austenite was then allowed to transform from the varying grain size conditions, tempered and subjected to survey creep-rupture tests. The following experimental steps were used:

1. The 1-inch bar stock was held at 2200°F for 1 hour to obtain thorough solution of excess phases. The grain size was coarsened by this treatment.

2. Preliminary experiments were conducted to determine conditions of rolling to give a fine austenitic grain size, partial recrystallization, and no recrystallization. (Fig. 2)

3. The varying degrees of grain refinement were obtained by allowing the bars to cool in air to temperatures of 2100°, 2000° and 1900°F then rolling to a reduction of 50 percent in two passes, and finally being allowed to air cool o room temperature. A master cooling curve obtained by placing a thermocouple n the center of a bar and measuring temperature versus time automatically from 2200°F was used to establish the appropriate time in air before the reduction by rolling. In one additional experiment a bar was cooled to 1600°F from 2200°F and reduced 60 percent in five passes to produce a severely strained austenite.

4. The bars after air cooling from rolling were tempered six hours at 250°F.

5. Survey creep-rupture tests were conducted at 1100°F under stresses of 19,000, 40,000 and 55,000 psi.

6. The grain sizes of the as-rolled materials were determined by light microscopy. Structures resulting from the various treatments were studied t magnifications varying from 250 to 22,000 diameters using light and electron microscopy. The specimens were polished on a Syntron vibratory polisher and tched with five percent nital. Palladium-shadowed collodion replicas were sed for electron microscopy.

Previous research on "17-22-A" V steel under a WADC contract (Ref. 5) showed that high creep-rupture strength was associated with heating to temperatures above the coarsening temperature of the austenite during heat treating. This however resulted in low ductility under rupture test conditions. Heat treating below the coarsening temperature of the austenite resulted in low creep-rupture strength and high ductility. This suggested that heating above the coarsening temperature followed by working under conditions refining the austenite grain size might result in high creep-rupture strength with the high ductility associated with transformation from a fine-grained austenite.

EXPERIMENTAL RESULTS

Extensive structural studies were carried out on "A" Nickel and A-286 alloy with the objective of explaining the influence of hot-rolling conditions on creep-rupture properties as established in Reference 1. Also, creep-rupture tests and structural studies were used to check the theory that improved creep-rupture properties could be obtained in a hardenable ferritic steel by using working to refine the austenite grain size after a high-temperature treatment so as to induce transformation from fine-grained but thoroughly solution-treated austenite.

Structural Analysis of "A" Nickel

"A" Nickel was selected for the study of the structural changes introduced into a metal matrix by hot working on the basis that it would be free from complicating structural changes, particularly those associated with precipitation reactions. It was recognized that impurities are present in "A" Nickel and would be expected to increase creep resistance over very pure nickel. At worst however they would be expected to intensify the effect of the structural changes induced by working the metal. If the assumption is correct and in the absence of recrystallization during testing, the influence of working conditions should operate only through the alteration of the crystalline perfection of the individual grains. This could be either or both the introduction of internal strain or the development of substructures. The increased creep resistance in the absence of recovery during testing from internal strain developed by "cold work" is well known. Limited studies of specially induced substructures in nickel has shown a marked increase in creep resistance with increase in substructure density. (Ref. 6)

As stated in Reference 1 consideration of the creep-rupture data strongly indicated that substructure was the controlling factor for creep at 1100°F. At 800°F internal strain was recognized as a major factor contributing to strength. Because the data from Reference 1 is so essential to the arguments developed in this report, certain key figures have been reproduced in this report.

Recrystallization During Testing at 1100°F

Rupture life at 1100°F and 20,000 psi increased uniformly with the amount of isothermal reduction (Fig. 3) independent of the temperature of reduction up to limiting amounts dependent on the temperature of reduction. It was recognized n Reference 1 that the limiting reduction at 1800°F was associated with recrystallization during rolling. The larger limiting isothermal reductions at he other temperatures had been found to be associated with recrystallization during testing.

To determine the recrystallization effects in more detail, all of the samples ested at 1100°F and 20,000 psi were examined for this report. Those samples which deviated from a uniform increase in rupture life with the amount of reduction were found to be undergoing considerable recrystallization during testing Fig. 3). Accordingly it seems clear that recrystallization during rolling or during testing was the limiting factor for increasing strength by isothermal reduction. When recrystallization occurred during rolling at 1800°F it prevented a build-up in strength even though the amount of recrystallization varied from a trace for 5.8 percent reduction to 75 percent for a reduction of 7.5 percent. Increasing amounts of recrystallization during testing first reduced the increase in strength from increasing amounts of reduction and when sufficiently extensive caused the strength to fall off from optimum values.

It is also important to note that reductions on a falling temperature could produce longer rupture times than could be obtained by isothermal reduction. Accordingly, some additional factor must be introduced by working on a falling emperature. The data are not sufficiently extensive to be sure of the relative role of recrystallization during testing after rolling on a falling temperature. The available data indicate little difference between isothermal and falling emperature reductions from 1400°F. However, at 1600° and 1800°F the trengths were built up by reduction on a falling temperature. The complications etween recrystallization during rolling and testing made it impossible to stablish recrystallization effects during testing for rolling on a falling temperature rom 1800°F. The limited data for rolling from 1600°F suggest that there was ess weakening from a given amount of recrystallization for rolling with a alling temperature than for isothermal reduction.

The increased amount of recrystallization during testing for a given reduction with decreasing temperature of reduction is quite evident for the non-isothermally olled samples. The rupture life was quite closely related to the amount of ecrystallization.

Recovery During Testing

The influence of recrystallization during testing at 1100°F suggested that recovery during testing was a factor. Recrystallization is the most advanced form of recovery in that it eliminates internal strain and much of the substructures.

However, internal strain can be reduced by recovery without recrystallization. Such recovery leaves a substructure characteristic of the prior working and recovery conditions and having sharp boundaries. Accordingly, limited tests were undertaken to study this factor.

The rupture times at 1100°F and 20,000 psi had been found in Reference 1 to correlate with as-rolled hardness with three separate curves. Figure 4 shows that one curve represented the data for rolling at 1800° or 1600°F, another for 1400° and a third for 80°F. Thus the rupture time varied over a wide range for a given initial hardness, depending on the rolling temperatures. Moreover the samples rolled over a falling-temperature range generally correlated with the isothermal data for the same initial rolling temperature. The correlations held whether or not recrystallization occurred during rolling. The only deviations were those samples which underwent extensive recrystallization during testing.

The correlation of rupture time with initial hardness even though recrystallization occurred during rolling or the samples were rolled on a falling temperature seems to be furthur evidence that the initial internal strain is probably indicative of some structural change which controls rupture strength. Apparently the initial temperature of reduction governs the influence on the structural feature controlling rupture strength. Certainly the degree of internal strain (in the absence of recrystallization during testing) is not the controlling factor.

The correlation (Fig. 4) for a given temperature of reduction between rupture life and initial hardness even though recrystallization occurred during rolling suggested that recovery from internal strain was involved in all the tests. When this possibility was recognized, the easiest way to check it seemed to be the measurement of the hardness of the threaded ends of the specimens. These portions of the specimens had been exposed to temperature with little effects of creep due to the low stress. Thus a measure of the thermally-activated recovery from internal strain could be obtained.

The results (Fig. 5) showed that all of the rupture times plotted as a function of the residual hardness gave one relatively narrow band that showed a very large increase in rupture life for a small increase in residual hardness. This was rather suprising because it included tests which recrystallized during testing as well as those which did not. Detailed study of the data indicates that the hardness values for specimens rolled at 1600° or 1800°F changed very little during the tests. The only appreciable change involving these temperatures of working were in those specimens rolled on a falling temperature and even then the changes were fairly small. The samples rolled at 1400°F and room temperature, however, underwent large changes in hardness when the initial hardness was high. These latter samples constitute those which had rupture lives on the low side of the correlation. The following can be deduced from the correlation with residual hardness:

1. Some other factor than hardness (internal strain) was the controlling factor for rupture life at 1100°F. This other factor had very little influence on hardness.

2. Those specimens undergoing substantial changes in hardness during testing probably had strengths low for their residual hardness because recovery or recrystallization during the tests accelerated creep.

A limited study of recovery by heating unstressed slugs was made with the results shown in Figure 6. These indicate that recovery or recrystallization could have been complete fairly early in the rupture tests, especially if creep accelerated these processes. However, the indications are that it continued during a substantial portion of the tests. Examination of the threaded ends of samples undergoing substantial recrystallization during testing indicated that the threaded ends also recrystallized. The data in the figure indicate that this must also have occurred gradually during the tests unless creep in the gauge section accelerated it markedly.

The significance of the correlation with residual hardness (Fig. 5) is difficult to understand. It suggests strongly that some other factor than initial hardness was the controlling factor for specimens rolled at 1600° and 1800°F. Recovery during testing after rolling at 1400°F and room temperature eliminated most of the hardening from internal strain but left the samples with the hardness characteristic of the controlling structural factor. The strengths were on the low side for their hardness due to the weaking caused by recovery or recrystallization during testing.

The only structural feature of which the authors are aware which could account for the observed affects of rolling conditions and recovery or recrystallization on rupture life at 1100°F is the nature of the substructures. It could be expected that increasing amounts of reduction would reduce the size of the substructural units and increase the number of grains having fine substructures (Ref. 7). It can further be hypothesized that partial recrystallization during rolling or during testing limited the number of grains containing fine substructures. The strengths however were in accordance with the volume of metal containing fine substructures. On this basis the various relationships investigated can all be accounted for. It would have to be assumed that higher strengths with a higher initial hardness for rolling on a falling temperature involves the opportunity for development of a more favorable substructure without recrystallization limiting this feature.

Because rupture time involves both creep resistance and the amount a material can creep, the elongation and reduction of area of the specimens should not be ignored. The ductility values for the rupture tests at 1100°F (Ref. 1) decreased with increasing rupture time. This indicates that creep resistance was the predominate factor controlling rupture time changes as influenced by prior rolling. The creep data in Reference 1 proves this. The lowest elongations of any specimens was in the order of 20 percent. Certainly working did not improve rupture life by increasing ductility. Moreover the ductiltiy was high enough to indicate that creep resistance was the predominate factor in all cases and not fracture mechanisms.

The dependence of creep resistance at 800°F on rolling conditions was very similar to that of rupture time at 1100°F (Fig. 7)even though there were a number of differences. The range of reductions with equal strength independent of temperature was however much lower. Secondly there was a greater increase in strength for a given larger reduction as the temperature of rolling was reduced. Creep strengths could be built up by rolling on a falling temperature but apparently not as high as could be obtained from an equivalent cold reduction. This behavior suggests that internal strain is a major factor at 800°F.

The correlation between as-rolled hardness and creep strength (Fig. 8), however, indicated that internal strain was not the only factor. The creep strength for a given hardness increased with the temperature of reduction up to 1600°F. Secondly, the difference increased with increasing as-rolled hardness.

Recrystallization during rolling at 1800°F limited the increase in strength as it did for rupture life at 1100°F. Recrystallization however did not occur during testing at 800°F. However, since recovery could occur during testing the hardness of the shoulders of the completed creep specimens were measured and plotted against the creep strength (Fig. 9). Recovery narrowed the difference between rolling temperatures but did not eliminate it as it did for rupture testing at 1100°F (Figs. 4 and 5). Accordingly it appears that internal strain from working persisted during the 800°F tests to increase creep resistance. Internal strain, however, was not the only factor because the creep strength for a given hardness was still highly dependent on the temperature of reduction. (Fig. 8)

For the reasons discussed in relation to the rupture test results at 1100°F, substructures appear to be the most likely factor in addition to internal strain influencing the creep resistance at 800°F. Presumably the higher creep resistance for a given hardness with increasing temperature of reduction is related to the degree of reduction involved. The larger reductions at the higher temperatures for a given hardness would presumably develop a finer substructure with more creep resistance.

Substructure Evaluation

The characteristics of "A" Nickel as previously discussed indicated that substructures from working should be the predominate factor controlling creep-rupture properties. Accordingly, extensive research effort was devoted to the development of means of measuring substructures as a function of working conditions. This roved to be very difficult and complete success was not achieved even hough most of the research effort on "A" Nickel was placed on this phase of he investigation.

Etching techniques in conjunction with light and electron microscopic xamination, transmission electron microscopy, Berg-Barrett X-ray eflection microscopy, and a micro-beam, back-reflection X-ray diffraction echnique were used to study substructures.

Light Microscopic Examination: For a reduction of 5.8 percent at 1800°F here was a clearly-defined substructure (Figs. 17 and 18 of Ref. 1). The ubstructures however became indistinct for a 10.8 percent reduction at 1800°F nd were not at all visible at higher reductions. Further, the substructures ere not visible for all reductions at 1600°, 1400° or 80°F. The etching echniques used did not define substructures in the latter samples even though heory (Ref. 8) and the properties of the samples indicated that substructures ere present and a controlling factor.

A review of the small amount of literature on light-microscopic studies f substructures in nickel indicated the main requirement was to satisfy the onditions necessary to cause etch pits to form at dislocation sites because ubgrain boundaries are basically configurations of dislocations. There were wo necessary conditions, namely, (1) a certain minimum degree of segregation f foreign atoms at the dislocation (called decoration of the dislocation) and 2) an etching technique that would produce a preferential attack of the metal t the dislocation site. There were sufficient foreign atoms present in the "A" ickel to satisfy the first requirement. However as discussed later there were number of treatments that left the atoms unavailable for decorating dislocations. everal etchants have been reported successfully used, but the best all-round tchant was found to be a 40-percent aqueous solution of ortho-phosphoric acid H_3PO_4) used in an electrolytic etching cell. The advantages of this etchant ere that (1) its action under 1-1/2 to 3 volts was slow and easily reproduced, 2) its usefulness was not appreciably altered by age, or minor variations in e concentration of H_3PO_4 or metallic ions, and (3) it left the other structural atures clearly visible.

Unfortunately, the ability to develop etch pits at dislocations was not the only equirement for showing substructures by the etch-pit technique. The second ecessary condition was that the sub-boundaries by sharp and well defined; for easons that are not clear a sub-boundary made up of a broad band of dislocations ould not be shown by the several etches used even after extensive efforts to do o. It was concluded that the failure to develop sharply outlined substructures "A" Nickel rolled large amounts and/or at low temperatures was due to these road, ill-defined sub-boundaries which are characteristic of deformed but not lly polygonized structures. This belief was substantiated when sharp sub-oundaries were found in some creep-tested specimens that had not shown substructures in the as-rolled condition. The loss of hardness and the sharpening of the sub-boundaries during creep could be explained as resulting from stress-accelerated recovery and polygonization. Recovery and polygonization are those processes by which (1) dislocations of opposite sign annihilate each other and (2) the remaining net excess of dislocations of like sign align themselves by a process of climb to form low-angle boundaries perpendicular to the slip planes. The former process is probably associated with the rapid loss in hardness observed during the first few hours in slugs heated to 1100°F (Fig. 6). The latter process, polygonization, is probably the controlling factor in reducing broad boundaries to sharp, narrow boundaries. Polygonization is known to be highly temperature dependent and proceeds rapidly only at temperatures well above half the melting point. At 1800°F it is quite rapid in nickel, but at 1100°F it is probably fairly slow, even under stress. In short, recovery and ploygonization reduce hardness and convert broad, ill-defined zones of dislocations into sharp, well-defined configurations of dislocations called subgrain boundaries.

It remains to be explained why, after creep testing, only a few of the specimens showed evidence of a sharpening of sub-boundaries while most of the specimens did not show sharp boundaries after creep. Supposedly all of the specimens experienced comparable amounts of polygonization during the creep test. Etch pits were visible in all samples after creep, except where they were not visible even before creep, so it may be assumed the etching technique had not failed unexpectedly. Apparently, even though the removal of internal strain as measured by hardness was rapid, the process of converting broad boundaries to narrow boundaries by polygonization was quite slow at 1100°F even under a stress of 11,000 psi. Perhaps the slow decrease in hardness that follows the initial rapid decrease (Fig. 6) is associated with the polygonization process. Further, the breadth of the sub-boundaries in the as-rolled condition probably varied, depending on the severity of the working conditions. Under these conditions it is conceivable that only those specimens with the narrowest sub-boundaries in the as-rolled condition had sufficiently narrow sub-boundaries after creep to be visible by the etch-pit technique. If this is true it must be concluded that the rolling temperature is a major factor in determining the breadth of sub-boundaries in the as-rolled state because after creep testing the specimen rolled 27.3 percent at 1800°F showed a clearer substructure than did the specimen rolled 5.3 percent at 1600°F and creep tested under the same conditions.

Failure to develop etch pits was experienced only in instances where precipitation of an excess phase occurred, or where a previously precipitated phase was not put into solution by the rolling process. Specifically, etch pits were not produced in the following instances:

1. In all of the annealed and annealed plus creep-tested specimens.

2. In new grains formed by simultaneous recrystallization during rolling at 1800°F.

3. In narrow zones adjacent to all grain boundaries of all specimens in the as-rolled condition. (After several hundred hours of creep at 1100°F and 11,000 psi this was not the case; etch pits formed right up to the boundaries.)

4. In specimens rolled at room temperature and up to 36 percent reduction at 1400°F.

The assumption is that all of these cases can be explained on the basis that the excess phase which precipitates under certain conditions ties up the foreign atoms which otherwise would decorate the dislocations. In annealed specimens and in the new grains of partially recrystallized specimens the excess phase precipitated at grain boundaries as small, discrete particles and also within the grains as clusters of platelets with definite angular relationships (Figs. 13, 15 and 16 in Ref. 1). In all specimens where the excess phase was dissolved during rolling, the phase re-precipitated only in grain boundaries and occasionally, on slip lines during cooling from rolling. Re-precipitation did not occur in the grain interiors, probably because the many dislocations introduced by rolling provided so many sites for the foreign atoms that distinct precipitates did not form. Supposedly the boundary precipitation that occurred during air cooling left the adjacent areas depleted of the foreign atoms needed to decorate dislocations. This would explain observation No. 3 above, and, also, the fact that no depleted zones were observed after several hundred hours of creep at 1100°F and 11,000 psi can be explained by diffusion of the foreign atoms (probably interstitials) from the grain interiors into the depleted regions where they then decorated the dislocations.

Creep at 1100°F resulted in the substructure in the material rolled 5.8 percent at 1800°F becoming much clearer (Figs. 10a-10d). Substructures were also visible in the samples isothermally reduced up to 27.3 percent at 1800°F (Figs. 10e and 10f). An indistinct substructure was also observed in the material reduced 5.3 percent at 1600°F. Samples which were not rolled or were rolled under other conditions did not show substructures after creep.

It was very unfortunate that those samples showing substructures after creep all had about the same rupture time. Consequently it was not possible to verify whether or not the hypothesis regarding the relation of substructure to properties was correct.

The presentation of four different areas on the same specimen in Figures 10a-10d for the sample reduced 5.8 percent at 1800°F was to show the variation in the appearance of the substructures with orientation of the individual grains. Similar variations were found in the other samples in which substructures could be seen. Accordingly it would be necessary that the differences in substructure be rather arge before a definite difference could be detected by optical microscopic means. Figures 10e and 10f also show the recrystallized grains to be free from fine substructures. So far as could be determined there was no change in the substructure as a result of creep for the sample reduced 5.8 percent at 1800°F where the substructure was visible before creep.

Electron Microscopic Examination: Electron microscopic examinations using replicas were carried out to ascertain if there were significant features of the microstructure too fine to be shown by the light microscope. The only significant result was the determination that the sub-boundaries in the material rolled 5.8 percent at 1800°F appeared first as a row of closely spaced pits (Fig. 11a); as etching was continued the pits grew until they overlapped to form continuous narrow valleys (Fig. 11b). No indications of substructures other than those visible under the light microscope were found.

Transmisson Electron Microscopy: The examination of metals in the electron microscope by direct transmission through the specimen itself has been shown capable of revealing individual dislocation (Ref. 4). The principle on which this works is dependent on the differential effect on transmitted electrons by the atomic arrangement at the dislocation in comparison to the surrounding matric free from dislocations. Because it offered a means of studying dislocations without the need for "decoration" by foreign atoms it seemed worthwhile to investigate. Moreover it offered the opportunity to study the broad, diffuse sub-boundaries hypothesized for the larger reduction and reductions at the lower temperatures.

The experimental difficulties of the technique are, however, very great. The most severe problem is the production of foils from the rolled bars with a thickness appreciably less than one micron. This must be done without plastically deforming the thin sections. Secondly the electron beam must not heat the specimen sufficiently to allow recovery.

The technique was carried far enough in the time available so that a recognizable substructure was developed in one transmission electron micrograph (Fig. 12) from a rolled sample. It was taken from the material rolled 5.8 percent at 1800°F and creep tested at 1100°F, the material with the most clearly evident substructure under light microscopy. The structure observed closely resembled that found in light microscopy, except that the apparent boundaries acted to reduce the number of electrons passing through the foil. In light microscopy the boundaries become visible as a result of being etched out preferentially. The process therefore does work without requiring etching.

Due to difficulties in preparation of samples and the lack of time, successful thin sections of other rolled samples were not obtained. The technique of preparing the thin sections requiresimprovement by a talented technician before the utility of the method can be established. In addition, a higher power electron microscope than the 75 KV unit available is necessary. Perhaps the most important problem even after the technique difficulties are solved will be the interpretation of the transmission micrographs. It is recognized that Figure 12 shows an array of dislocations and not individual dislocations. It can be forseen that interpretation difficulties would be large for samples rolled large amounts with high dislocation densities.

Berg-Barrett X-ray Reflection Microscopy: X-ray diffraction micrographs aken by the Berg-Barrett technique are shown in Figure 13 for material annealed t 1800°F and material reduced 5.8 percent by rolling. The splitting of the iffraction spot for the annealed condition shows a coarse type of substructure in he annealed condition. The reduction of 5.8 percent at 1800°F smeared the iffraction spots indicating that the original, coarse subgrains had been modified n crystal perfection by the rolling.

The most significant result, however, is the lack of adequate resolution of he method. Figure 13c, a light micrograph of the sample reduced 5.8 percent at 800°F, was included to show the large number of fine subgrains present in a eformed grain. For the present study the method would have had to be capable f resolving the number of individual spots within one diffraction spot corresponding o the number of subgrains shown by Figure 13c to be measuring the same substructure shown by the light micrograph. It is clear that this is not being ccomplished.

The method shows a coarse substructure present in the unreduced material. his coarse structure was not observed in the light micrographs, perhaps because he precipitated impurities were not available to decorate dislocations. After a eduction of 5.8 percent at 1800°F, these original coarse subgrains had undergone he development of a finer substructure, apparently the one more closely related o properties.

Micro-Beam, Back-Reflection X-ray Diffraction: In a back-reflection X-ray hotogram taken under the conditions described on page 6 and in Figure 1b, single grain in an annealed, polycrystalline specimen will produce a rather harp spot. If the specimen is cold worked, the diffraction spot will spread both adially and circumferentially with respect to the Debye ring. The radial roadening is due to residual lattice strains which cause the lattice parameter to ary slightly from point to point, and the circumferential spreading is caused by ttice rotations within the grain. If the specimen is then heated to allow recovery nd polygonization to occur, the radial breadth decreases and the arc formed by ircumferential spreading breaks up into a series of small spots corresponding to ubgrains having slightly different orientations. This cycle of events can occur lmost simultaneously when a metal is deformed by rolling at a temperature just elow the simultaneous recrystallization range.

In short, three kinds of data can be obtained from a micro-beam, back-eflection X-ray photogram of a rolled nickel specimen:

1. The degree of radial broadening is a measure of residual lattice strains. he presence of extremely small (less than 10^{-6} cm in diameter) subgrains is nother possible cause of radial broadening, but this is likely only in severely old worked metals.

2. The extent of circumferential spreading is determined by the over-all ange of subgrain orientations within one grain which, in turn, is determined by e severity of the deformation.

3. The degree of separation of any two subgrain spots within an arc is related to the angle of misorientation between the subgrains.

Figures 14 and 15 are typical examples of X-ray photograms obtained from rolled samples. Less initial strain resulted from rolling at 1600°F than at 1400°F or at room temperature causing a sharpening of the Debye rings as the rolling temperature increased. The relatively few photograms taken were not sufficient to definitely establish the relationship between circumferential spreading and rolling conditions. The few results available, for example Figure 15, suggest that circumferential spreading was a function of degree of reduction and not temperature of reduction (in the absence of recrystallization during rolling).

The limited amount of work done by this method was undertaken with the hope that a very small beam would result in better resolution of the small diffraction spots arising from the substructures. This was not achieved. Figure 16 shows the only spots observed. In this case they were apparently due to the same coarse substructure that was shown by the Berg-Barrett method for annealed material (Fig. 13). The diffraction spots from the finer type of substructure introduced by rolling were not resolved. When this was certain no more work was done by this method.

It is interesting to note that the indications were that the circumferential spreading of the arcs was independent of temperature of reduction. This corresponds to the rupture life at 1100°F and 20,000 psi being independent of the temperature of reduction and a function only of degree of reduction for isothermal rolling. The circumferential spreading of the diffraction spots is a function of the over-all variations in orientation of the substructures. It is possible therefore that this is a major factor influencing creep-rupture strength.

Discussion and Summary of "A" Nickel Structure Studies

The microstructural studies demonstrated that recrystallization during working or during subsequent exposure to creep was the limiting factor for increasing strength in "A" Nickel by hot working. Apparently the build-up in strength from working on a falling temperature to higher levels at higher hardness values is due to a lower tendency to recrystallize under these creep-rupture test conditions.

Recrystallization during working at a relatively constant temperature limits the build-up in strength so that the strength remains essentially constant with further reduction. Recrystallization during creep, however, reduces strength in proportion to the degree of recrystallization. Thus a small degree of recrystallization during rolling may reduce the amount of increase in strength from increasing reduction but still leave the strength higher than for a smaller reduction which did not recrystallize. The amount of recrystallization during testing had to be substantial before it began to reduce strength below that of the optimum reduction. Certainly there was less recrystallization and higher strengths the higher the initial temperature of rolling on a falling temperature.

So far as was ascertained from the data, internal strain contributed very little o rupture strength at 1100°F. Apparently recovery from internal strains occurred uite rapidly. The strong dependence of rupture time on amount of reduction ndicates that some other factor than internal strain or recovery from internal train controlled properties. At 800°F, however, internal strain contributed ubstantially to strength because recrystallization did not occur and recovery was low.

The correlations between as-rolled hardness and creep-rupture properties Figs. 4 and 8) demonstrate strikingly how inaccurately hardness measures these roperties for "A" Nickel rolled at widely differing temperatures. However, nitial hardness did correlate with properties for specimens rolled at the same emperature. This resulted in wide variations in properties for a given hardness epending on the rolling temperature. When rupture life at 1100°F and 20,000 psi as plotted versus residual hardness as measured in the threaded portion of ompleted test specimens a single correlation band was obtained independent of olling temperature that was nearly vertical so that a small change in residual hardess was associated with a very large change in rupture life. One possible explantion for this is that residual hardness is a measure of the substructure and that hile the substructure has little effect on the hardness it has a very large effect n the rupture life. The residual hardness measured in specimens creep tested t 800°F showed they apparently still contained appreciable amounts of internal train because three separate correlation lines were obtained that were dependent n the rolling temperature.

At 1100°F where internal strain effects were apparently eliminated by recovery, he major factor governing strength was the degree of reduction. Recrystallization uring working or during testing apparently was the only thing that prevented the trength from increasing continuously with the amount of reduction independent of e working temperature. At 800°F the contribution to strength from internal train and the absence of recrystallization during testing resulted in some dependence n the temperature of reduction.

The dependence of creep-rupture strength at 1100°F on amount of reduction sugests that the structural feature varied by working was influenced much more by eduction than by the temperature of rolling. A small amount of qualitative X-ray ata indicates the range of mis-orientation of the substructures within each grain as also independent of the temperature of reduction. In general it can be expected at the degree of misorientation increases with the degree of reduction. The results uggest that this is independent of the temperature of reduction. It is not, however, lear whether it is the degree of mis-orientation or the probable accompanying efinement of the substructure that is the controlling factor governing strength.

The greatest effort of the investigation was expended in exploration of techniques measure the substructures as a function of working conditions and creep-rupture roperties. The substructures were, however, so fine (1-3 microns in diameter) even after a small reduction at 1800°F, that most of the techniques used previously by others to measure substructures were inadequate. Apparently, most of the previous substructure studies dealt with the larger substructural units such as are visible in the Berg-Barrett X-ray micrographs (Figs. 13a and 13b) and the back-reflection, X-ray photograms (Fig. 16).

It is clear that the Berg-Barrett technique or the micro-beam, back-reflection technique as used in this investigation will not resolve the smallest substructures established by the rolling conditions. Techniques and equipment such as that developed by Weissmann (Ref. 9) has a high resolving power. However it must be carried out on fully recovered samples. It could not therefore be used on as-rolled samples, except perhaps those samples rolled very small amounts at very high temperatures. Its usefulness would depend on the degree to which samples could be recovered after rolling without inducing substantial subgrain growth. Even then the resolving power might be inadequate if extreme refinement of the substructures resulted.

Light microscopic means for studying the substructures also seems limited in scope. The need to decorate the dislocations with impurities seems to be a limiting factor. Only the samples rolled at 1600° and 1800°F responded to this. A more serious limitation seems to be the broad, diffuse sub-boundaries resulting from low rolling temperatures and/or large reductions.

Transmission electron microscopy offers considerable promise. The experimental and interpretation problems are however a major limitation. Any attempts to use this technique should clearly recognize that a major effort involving unpredictable time periods would be required to develop a method of preparing suitable thin slices and proper interpretation of the transmission electron micrographs.

The role of the impurities in the "A" Nickel is not clear. Certainly they did not delineate the substructure as well as might have been expected. It is not clear whether or not this was due to the complexity of the substructures. Because the impurity atoms should theoretically segregate to the substructure boundaries it would be expected that they could have a major effect on properties. There is still therefore an important question as to whether the marked effects of rolling conditions on creep-rupture strength is due to substructures alone or to an interaction of impurity atoms with substructures.

Structural Analysis of A-286 Alloy

In Reference 1 it was shown that the creep-rupture properties of the particular lot of A-286 stock investigated exhibited marked dependence on temperature of rolling even though it was solution treated at 1650°F and aged at 1325°F subsequent to rolling. The effect of degree of reduction was minor in comparison to the temperature effect. Moreover, the initial temperature of heating controlled properties of samples rolled on a falling temperature. These conclusions were tempered, however, by recognition that the properties of the experimental stock were not onsistent with the results of the experimental rolling. The strength of the original ar stock used for the investigation was lower than would have been predicted by ıe experiments. It was hypothesized that this was associated with the larger eduction used in producing the bar stock under conditions which refined the rain size.

The results being reported here are those obtained in an effort to relate tructural features as governed by prior rolling conditions to the creep-rupture roperties. Recrystallization characteristics during rolling and during heat eatment, the age-hardening γ' reaction, and other precipitation characteristics uring heat treating and during testing were considered.

The results of creep-rupture tests from Reference 1 are included as Figures 17 nd 18 as background information.

ecrystallization and Grain Size

Simultaneous recrystallization occurred during rolling only at 2200°F. (Table 1). his was limited to a maximum of about 20 percent recrystallization for the largest -pass reduction of 35.1 percent. The data in Table 1, however, show that there as a marked increase in grain size when the rolling temperature was raised from 700° to 1950°F.

Recrystallization during heat treatment at 1650°F occurred in A-286 after rolling ıder the following conditions:

1. All material rolled at 80°F.
2. All material rolled at 1700°F, except the one reduced 3.9%.
3. The specimens reduced 38.7 percent in 5 passes from 1950°F.

one of the other samples recrystallized during heat treatment. The determination recrystallization during heat treatment was based on grain shape, grain size, rain markings and the basic principles of recrystallization and grain growth. No ıdication of grain growth during testing was observed.

When the log of the rupture time was plotted against the average grain size igs. 19 and 20) a general trend of increasing rupture life with increasing grain size as obtained. The variation in rupture times for a given grain size was substantial, ırticularly for the larger grain sizes (larger rupture times).

It is not possible to determine from the data obtained to what degree grain size as responsible for the observed effects of rolling conditions on properties because e following additional variables are present:

1. Partial recrystallization during rolling in some cases.

2. Recrystallization during heat treatment at 1650°F in part of the samples and not in others.

3. Variations in the degree of solution of excess phases with temperature of heating for rolling.

Under the conditions of rolling studied, none of the samples recrystallized more than about 20 percent during rolling. Figures 19 and 20 show that the rupture strength was in accordance with the predominant, large, unrecrystallized grains. There was little indication that increasing amounts of recrystallization would reduce strength. In fact the rupture time tended to increase as the amount of reduction increased at 2200°F above that required to induce recrystallization. This resulted in a range of average grain sizes with constant rupture life or even higher rupture life at the smaller average grain size.

Recrystallization during heat treatment was confined to materials which were initially fine grained. The only possible exception was the material reduced 38.7 percent from 1950°F in 5 passes. The rupture life was low and possibly in accordance with the grain size although one sample which did not recrystallize had still lower strength at 1200°F. There were also samples which recrystallized to a grain size slightly larger than the majority of specimens rolled at 1700°F which had no higher strength than the finer-grained samples.

In the specimens rolled from 2200°F, there was a fairly wide range in grain size for essentially constant rupture times. Material rolled from 1950°F showed quite wide variations in rupture times for essentially constant grain size.

A review of the grain characteristics of the specimens subjected to creep testing at 1200°F and 35,000 psi, as reported in Reference 1, indicated that the same general relationships to creep strength existed as for rupture strength.

The relationships between strength and recrystallization effects certainly are not clear cut from this data. It appears that the grain size itself, the absence of recrystallization during heat treatment, or the solution effects of the higher temperature treatments could be involved. Samples which underwent recrystallization and grain growth when they were heated but not rolled had as high or higher strength than the samples subsequently rolled. This indicates that structural effects of the type found in rolling "A" Nickel did not persist in the unrecrystallized A-286 materials to cause an increase in strength. The recrystallization and grain growth in the samples with no reduction would have removed any possible substructural effects present in the as-received stock. There the cause of the variation in the strength of A-286 alloy seems to be related either to the grain size or the solution effects accompanying heating for rolling.

Age-Hardening Precipitate

The major strengthening reaction in A-286 alloy is generally considered to be the precipitation of an age-hardening phase identified by several investigators as $Ni_3(Al, Ti)$ and designated as γ'. Because the precipitation reaction is generally considered so predominant in determining the strength of A-286 the influence of the rolling conditions on this reaction was studied.

The hardness of the as-heat treated samples (Table 1) did not indicate any pronounced effect of rolling conditions after aging. The samples heated to 1950° or 2200°F were somewhat softer than the rolled samples. The bars rolled in several passes from 2200°F were the hardest. Yet they were no stronger if as strong as the samples which were not rolled but heated to 2200°F. Thus there was nothing in the hardness data to account for the increased strength from rolling at 1950° and 2200°F.

Representative specimens were examined by electron microscopy (Figs. 21-24) in an attempt to determine if this would show a significant effect on the precipitation reaction. No definite effects attributable to either heating or working alone were found in so far as the general matrix precipitate was concerned. However, the following three effects were noted which appeared to have no relation to these two variables:

1. The precipitate appeared more clearly in some samples than in others. It appeared most clearly in the samples from which Figures 21c, 22a, 22c, 23b, 23e, and 24c were taken.

2. Some samples showed a marked roughening of the surface that varied considerably from grain to grain, e.g., Figures 21e, 23c, and 23d.

3. Some samples, such as in Figures 21a, 22b, and 22d, showed only random etch pits.

When well-defined precipitate particles were found, very little difference in size or spacing could be found. The possible variation in the degree to which the precipitate developed during heat treatment did not appear to be related to properties.

Grain Boundary Phases

Phases developed in the grain boundaries during heat treatment. The predominant one is shown in Figures 21-24. It was very similar in appearance to a phase discovered by Beattie and Ver Snyder (Ref. 10) and analyzed by Beattie and Hagel to be $Ni_{13}Ti_8Si_6$ (Ref. 11). It is referred to as G phase because of its prominent appearance at grain boundaries.

G phase appears in all of the electron micrographs of Figures 21 through 24, excepting Figures 21a, 21b, 21e, 22b, 22d, 23c, and 23d. These exceptions correspond to those specimens that do not show well-defined age-hardening particles (except for Figure 22c), suggesting that the tying up of titanium in G phase may retard or delay the precipitation of gamma prime.

Evidence of a second grain boundary phase was found in some of the specimens that were electropolished. In these specimens deep, narrow crevices were formed along the grain boundaries during the electropolishing operation. The possibility that the crevices were pre-existing microcracks was ruled out when the same specimens contained no crevices after preparing the surface by argon-ion bombardment. Unfortunately, this phase did not respond to the etchant (5 Glycerine-4HCl-1HNO_3) that was used to reveal the G phase and the age-hardening precipitate. However, enough of the electropolished specimens were examined under the electron microscope to establish that the unknown boundary precipitate was (1) virtually absent in samples heated to 2200°F, (2) present in some of the 1950°F specimens, and (3) present in practically all of the 1700° and 80°F specimens.

Random comparisons of structures between strong and weak specimens failed to show whether the most prominent boundary precipitate, G phase, had any effect on the creep-rupture properties. When comparisons were made at constant grain size, however, a slight strengthening effect of G phase was evident in specimens rolled at 80°, 1700°, and 1950°F and rupture tested at 1200°F and 65,000 psi and at 1350°F and 40,000 psi. This judgement was based on the observation that in Figures 19 and 20 the points for specimens containing no G phase or only trace amounts of G phase were consistently on the weak side of the scatter band while points for the specimens containing appreciable amounts of G phase were always on the high side of the band. The only exception noted was the specimen rolled 3.9 percent at 1950°F (rupture life of 143 hours). This specimen showed only trace amounts of G phase precipitated at grain boundaries, but its rupture life was at the top of the scatter band (Fig. 19). The influence of G phase in specimens rolled at 2200°F was not clear. All of these samples contained appreciable amounts of G phase and yet there was a significant variation in strength.

In the slow strain rate tests at 1200°F and 35,000 psi there did not appear to be any influence of G phase. A similar study of the second, unidentified grain boundary phase indicated this phase did not appreciably influence properties. The unknown phase could be detected only in electropolished samples by the short crevices left after the precipitate was dissolved during the electrolytic polishing action. The method of specimen preparation (see page 5) used for the electron micrographs in Figures 21-24 did not reveal the unidentified phase.

Globular Excess Phases

Excess phases in globular form were widely spaced throughout most of the specimens. The particles frequently were found in elongated groups or stringers. They also occurred as single particles randomly scattered throughout the structure.

The identity of these particles is not critical for this investigation, but it may be assumed that they were mainly titanium carbide (and/or carbonitrides) and agglomerated G phase ($Ni_{13}Ti_8Si_6$).

The number of particles was found to decrease somewhat on heating to 1950°F and considerable more solution occurred on heating to 2200°F. These changes coincided with rather large increases in the average grain diameter indicating that the particles were effective grain growth inhibitors up to some temperature between 1700° and 1950°F.

The highest strengths generally occurred in the samples experiencing the greatest solution of excess phases and the largest increase in grain size. Because the two effects occur simultaneously it is not clear which was responsible for the strength increase.

The main effect of solution with the higher temperature treatments appears to be solution of carbides or carbonitrides. In the absence of a direct indication of any disturbance of the general precipitation of γ', it appears that if solution effects were responsible for the increased strength, it must involve these elements. The γ' is essentially dissolved at 1650°F and apparently should not be involved in the effects of higher temperature.

The mechanism by which carbon and nitrogen could improve strength is not known. Possibly it operates through a strain-aging type of mechanism as discussed in another section of this report.

Discussion and Summary of A-286 Structure Studies

The structural studies of A-286 alloy as related to working conditions and properties indicated that the major effect of working conditions was the initial temperature of heating. There was no clear-cut indication that partial recrystallization during working or recrystallization during subsequent heat treatment at 1650°F resulted in marked differences in properties from those of samples rolled at higher temperatures which did not recrystallize.

The marked effect of heating temperature alone indicated that there was no influence of working conditions such as was found for "A" Nickel which persisted in the absence of recrystallization.

The generality of the indication that heating temperature was the controlling variable must still be tempered by the recognition that low strengths are found with high initial heating temperatures. This apparently is related to extreme recrystallization during rolling and possibly to temperature reduction relations during large reductions.

The strength increased with grain size in an approximate correlation. However solution of excess phases, mainly carbides or carbonitrides occurred concurrently. Either or both of these factors could be involved. The solution effect is probably the moreimportant.

No evidence was found to indicate that the strength properties were altered by an influence of heating and rolling on the general precipitation of γ' in the matrix. Likewise no general effect of grain boundry precipitates was found, although samples containing the larger amounts of a phase believed to be G phase tended to have high strength for their grain size.

Influence of Hot Rolling on Creep-Rupture Properties of "17-22-A"V Steel

The original experiments were designed to determine if a thorough solution treatment followed by working under conditions which refined the austenite grain size would give high strength combined with the high ductility considered characteristic of structures transformed from fine-grained austenite. When it was found that refining the austenite grain size did not improve ductility, experiments were undertaken to determine if working without recrystallization before transformation would improve ductility.

It was determined that to obtain a combination of a reasonably coarse grained austenite and a thorough solution treatment it was necessary to heat the steel to 2200°F. The resulting austenite grain size was ASTM 4.5. Preliminary experiments were carried out to establish conditions of rolling which would refine the austenite grain size after treatment at 2200°F. The results (Fig. 2) indicated that within the limits of stock size and the rolling mill a reduction of 50 percent after allowing the bar to cool from 2200°F to 2100°F gave the most suitable refinement of austenitic grain size. This resulted in a grain size of ASTM 7.5. In addition, for comparative purposes, bars were cooled to 2000°F and reduced 50 percent with a resultant partial recrystallization of the austenite. Further, material cooled to 1900°F and reduced 50 percent did not recrystallize.

An additional experiment was undertaken in which the austenite was severely strained by cooling to 1600°F from 2200°F and reducing 60 percent in 5 passes through the rolling mill.

All samples were air cooled after rolling and tempered six hours at 1250°F. The resulting Brinell hardness values (Table 2) were in the range of 326 to 355.

Rupture-Test Characteristics

The influence of the treatments were evaluated by rupture tests at 1100°F with the results in Table 2 and Figure 25.

The usual heat treatment of the "17-22-A" V steel is to air cool from 1850°F and temper. Data for this treatment has been included in Table 2 and Figure 25 for comparative purposes. The special rolling conditions had the following effects in comparison to this standard treatment:

1. Recrystallization of the austenite by the reduction of 50 percent at 2100°F after heating to 2200°F resulted in about the same rupture strength as for the standard 1850°F heat treatment. The ductility was, however, very low while the standard treatment had high ductility.

The 50-percent reduction resulted in an austenitic grain size of 7.5 while the austenitic grain size during the standard treatment at 1850°F was much finer (ASTM 11.5). Neither high strength nor high ductility was obtained by the austenite grain refinement. If refinement of austenite grain size is responsible for combinations of high strength and high ductility in as-worked and tempered steels, a much finer grain size than that obtained in this work would be necessary.

2. The material with partial recrystallization of the austenite as a result of a 50-percent reduction at 2000°F, had appreciably higher strength but the ductility was still low.

3. The material reduced at 1900°F had still higher rupture strength. Although the ductility in the shorter time tests showed improvement it was still low in the longer time tests.

4. The 60-percent reduction from 1600°F was included in the experiments because the results previously discussed suggested that worked but unrecrystallized austenite was the key to combinations of high strength and high ductility. The 60-percent reduction at 1600°F was selected as the most severe working of the austenite which could be introduced within the limits of the stock available for the experiments.

The results showed a further substantial increase in rupture strength and an appreciable increase in ductility. The ductility was still low, however, in comparison to that which it was hoped could be developed by the experiments.

5. One test on material simply heated to 2200°F and air cooled without working had low rupture strength and extremely low ductility.

6. The creep data (Table 2) from the rupture tests indicate that reducing the rolling temperature of the austenite from 2100° to 2000°, 1900°, and 1600°F resulted in some increase in creep resistance. The material simply heated to 2200°F was, however, most creep resistant, but its low ductility limited rupture life. In the materials reduced by rolling, the rupture life was increased by the combined effect of increased creep resistance and increased ductility.

7. The results indicate quite definitely that a relatively fine austenitic grain size in combination with a thorough solution treatment is not the key to combinations of high strength and ductility in rupture tests at 1100°F. They do indicate however that working of the austenite under conditions where recrystallization does not occur does markedly improve strength and can improve ductility. It now appears that combinations of very high strength and ductility would result if working were carried out under the following conditions:

(a) Initial heating for working resulting in thorough solution effects.
(b) Initial working to refine the austenite grain size.
(c) Working continued to temperatures far below those where the austenite recrystallizes so as to strain the fine-grained austenite before transformation to bainite.

Microstructures of Worked and Transformed Samples

Optical micrographs (Figs. 26-29) show the effect of the working conditions on structure. While it is difficult to judge bainitic structures it appears that the more the austenite is worked before transformation the finer the bainite. The influence of working on the austenitic grain structure is indicated in the 250X magnification pictures. The grain refinement from the reduction at 2100°F (Fig. 26) and the grain distortion from reduction at the lower temperatures (Figs. 28 and 29) is evident.

All of the as-rolled and tempered samples and the sample normalized from 2200°F were examined by electron microscopic means. At a magnification of 22,000X no definite effects of working on the structures was evident. As shown by the electron micrographs (Fig. 30) there were alternate areas of rough and fine texture with small carbides. Examination of the replicas at 2,200X, however, indicated two differences. The visible carbides shifted from the old austenite grain boundaries to a random distribution as the rolling temperature was reduced.. Secondly the bainite structure appeared to become finer as the rolling temperature was reduced.

Discussion and Summary of "17-22-A"V Studies

The results indicated that improved creep-rupture properties can be obtained in Cr-Mo-V steels by working thoroughly-solution-treated austenite below its recrystallization temperature and then allowing it to transform to bainite during air cooling from rolling. The working was more effective the lower the temperature of reduction.

Refinement of austenitic grain size by recrystallization during rolling after a thorough solution treatment was not effective in increasing strength and ductility in rupture tests at 1100°F. If refinement of grain size is to be effective the grain size must be reduced to a considerably smaller size than the ASTM 7.5 obtained in this investigation.

The evidence points to the probability that extremely high rupture strength combined with good ductility will develop in as-worked and tempered steel if certain conditions are met. These are (1) thorough solution by a relatively high temperature of heating for working, (2) large reductions refining the austenite grains to a small size, and (3) continuation of working to temperatures far below the simultaneous recrystallization temperature so as to severely strain the small austenite grains.

It should be recognized that the conditions set forth in the previous paragraph may be important in response to subsequent heat treatment as well as for properties in the as-worked and tempered condition. It is known that strength and ductility after a normalize at 1850°F are dependent on the prior processing. It is suggested that the conditions of the previous paragraph are necessary for combinations of high strength and ductility. If the structure after working is coarse the probability is that the alloys will not respond as well as if relatively fine structures of initially well-solutioned material were heat treated.

The structural studies indicated that working the austenite caused carbides to be distributed more randomly and reduced the tendency to segregate to the old austenite grain boundaries. This is probably responsible for the improved ductility. In combination with the refined bainite it is also probably responsible for improved creep resistance.

Straining the austenite prior to transformation could be expected to initiate transformation at many more centers than in strain-free austenite. This should have refined the bainiteas the structures indicated. It should also tend to reduce the tendency for carbides to segregate to the old austenite grain boundaries. Transformation from many centers would be expected to reduce the prominence of the old grain boundaries.

It is important to recognize that secondary hardening is very important to the properties of Cr-Mo-V steels. It could be, therefore, that the observed effects on properties operate through the secondary hardening reaction. This was not studied in this investigation.

GENERAL RELATIONSHIPS SHOWN BY STUDIES OF EFFECTS OF HOT-WORKING CONDITIONS

In a simple, single-phase alloy working conditions can have a pronounced effect on properties. The results for "A" Nickel indicate that two factors can be involved. At relatively low creep temperatures, internal strain induced by working can raise creep strength substantially. As the test temperature increases strain becomes less important. Plastic working however still has a major effect, probably through the substructures established. The data indicate that this is dependent on the amount of reduction and not the temperature of reduction. Recrystallization during working or during subsequent creep is the limiting factor for improving strength by this mechanism. Very sparse data indicate that the improvement in strength occurs as a result of the development of very fine substructures probably as a result of refinement with increasing reduction and/or increasing mis-orientation of the sub-grains.

In an alloy subject to a strong age-hardening reaction, A-286, very little evidence of influence from strain or substructure was found. The predominant factor was the temperature of heating. It is recognized that the data are incomplete since they do not account for low strength resulting from large reductions after a high initial working temperature. The indications are that grain size and solution effects are the controlling factors. Evidence was found of appreciable solution effects from higher temperatures of working than those related to the precipitation reaction. At the same time, however, grain size increased. Because it is known that large reductions from a high temperature can result in a fine grain size and low strength it is not clear which factor predominates. No evidence was found to indicate that varying amounts of rolling significantly influenced the general precipitation reaction in the A-286 alloy insofar as properties are concerned. There was evidence however to indicate that heating or heating and working influenced phases forming in the grain boundaries which had some effect on properties.

In a Cr-Mo-V steel undergoing transformation to bainite after working it was found that better properties resulted from lowering the working temperature so as to induce increasingly severe strains in the austenite prior to transformation. Refining the austenitic grain size by simultaneous recrystallization was not effective under the conditions of these experiments. Presumably best all-around properties would result from transformation of strained, fine-grained austenite. In this case prior working conditions appeared to be important through their effect on the transformation to bainite. The strained austenite appeared to transform from more sites than the unstrained austenite with consequent influences on the bainite structure and carbide distribution. In a sense then the same factors that influenced "A" Nickel were involved except that they operated through their influence on transformation structures rather than through a direct effect on the crystalline perfection of the matrix. There is no reason to expect that either the internal strain or substructures developed by working the austenite survived the transformation.

HASE B-RELATIONSHIP BETWEEN STRAIN-AGING-TYPE PHENOMENA AND STRENGTH AT HIGH TEMPERATURES

Strength at high temperatures on the high side of the range for many alloys s associated with manifestations of strain aging. Usually this is found for high emperatures of heat treatment where it is probable that there is solution of carides, nitrides, etc. These elements could be expected to participate in straingging-type reactions.

Usually the maxima in strength versus temperature curves, yield points, or errated stress-strain curves indicating strain aging are exhibited at lower emperatures than those at which creep is significant. However, the tendency or high creep and rupture strengths to occur in those conditions of alloys which xhibit strong evidence of strain aging suggests that the same structural and ompositional features which are responsible for strain aging can also be esponsible for the high strength at high temperatures.

There appears to be two possible mechanisms by which strain aging could ncrease creep resistance:

1. As the temperature is increased, the strain-aging mechanism remains ffective for decreasing amounts of strain and slower strain rates.

2. The small atoms which participate in strain aging are attracted chemically o large atoms in the alloy. This inhibits diffusion of the small atoms and causes stabilization of the Cottrell-type atmospheres at the dislocations blocking their novement and thereby reducing creep.

It seemed probable that this type of reaction could be responsible for a substanial proportion of the unexplainable heat-to-heat variation in properties of similarly eat treated alloys at high temperatures. Furthurmore, the influence of prior istory on properties of an alloy could be due to the influence of the prior history n the solution of phases containing elements which participate in strain-agingype reactions and to its influence on the substructure which the reaction could tabilize.

Both possible mechanisms for the strain-aging-type reactions involve the hift of the manifestations of the reaction to higher temperatures under creep onditions. It appeared therefore that an investigation of the shift of manifestaions of strain aging with strain rate would demonstrate whether or not the ostulated mechanisms were operating.

BASIS FOR INVESTIGATION

The most common way in which strain aging exhibits itself is through maxima r inflection points in strength versus temperature curves. Experimenters usually onduct tensile tests at constant strain rates and plot the stress for a fixed strain versus temperature. The strain rates for these tests are several orders of magnitude faster than those involved in normal creep and rupture testing. Moreover the strains at which maximum effects are found in tensile tests are usually several percent. In general the strain rates and total strains involved are much higher than those involved in creep and rupture testing in the range of 100 to 1000 hours or longer.

Both theory and experimental evidence demonstrate that maxima in stress for a given strain versus temperature curves in constant-strain-rate tensile tests shift to lower temperatures as the strain rate is reduced. However these maxima are for quite large strains.

There is ample experimental evidence to demonstrate that the strengthening due to strain aging in tensile tests disappears as the temperature is raised above the temperatures at which strength maxima are observed. For instance, there will be no difference in the stress for a fixed strain for carbon steel subject to strain aging and a non-aging carbon steel when the temperature is raised to 800° to 1000°F. The aging steel will however show a marked strengthening at about 400°F. The strain rates involved are however very rapid in comparison to those in creep and rupture tests. Moreover the strains involved are large in comparison to those usually considered in creep tests.

Alloys subject to strain aging exhibit so-called yield point phenomena. These may be the sharp yield points commonly found in some carbon steels or may be serrations in the stress-strain curve for tensile tests. Both effects disappear for tensile tests as the temperature is raised to the point where creep becomes significant.

For the same type of mechanism to be effective in increasing creep and rupture strength as causes the usual manifestations of strain aging, some type of modification must exist. The dragging effect of interstitial atom atmospheres could be effective at higher temperatures under conditions of slow strain rates and small strains. As previously discussed, however, the effect would shift to lower temperatures for large strains. For this mechanism to be effective, it would have to be confined to small strains.

The other possibility is that the interstitial atoms are stabilized in Cottrell-type atmospheres as the result of chemical attraction to larger atoms present in the alloy. There is some precedent for this. In carbon steel it is known that undeoxidized steel has relatively high creep strength to about 800°F. However, the addition of silicon raises the strength markedly above 800°F. Aluminum added under conditions which avoid the formation of aluminum nitride also has the same effect. It has furthermore been demonstrated that there is a direct correlation between nitrogen in solution and creep resistance of carbon steel containing silicon and/or aluminum at 850°F. Both elements tend to react with N to form nitrides of Si or Al. Thus the tendency for chemical reaction between Si or Al and N could stabilize the N atoms in Cottrell-type atmospheres by chemical attraction. To be effective the reaction would have to be limited and not proceed to the point where discrete compound particles formed. It is known that treatments causing massive Si or Al nitrides result in less than half the creep strength observed in the same steel with nitrogen in solution at the start of the test.

In view of the known marked effect of nitrogen on the creep properties of carbon steel, it was selected as one of the materials to check the influence of strain rate and total strain on the temperature of maximum strain aging effect. There were, however, many alloys which exhibited seemingly similar creep behavior for which the relationship to strain aging was not as clearly indicated. One of these was A-286 alloy which exhibits a very wide range in rupture properties between heats for a given standard heat treatment. Accordingly this material was selected for investigation as a more general case. The same stock was found to exhibit strain aging in an investigation using hot-hardness tests at the Materials Laboratory, WADC. It should be noted that a hot-hardness test is essentially a rapid-strain-rate test involving large strains.

EXPERIMENTAL PROCEDURES

Property Measurements

The studies were carried out by subjecting the experimental materials to constant-strain-rate tension tests over a wide range of temperature and measuring the stress at several given strains. The final data were plotted as iso-strain curves of stress versus temperature.

For carbon steels strain rates of 0.25 and 0.004 percent per hour were used. For A-286 standard tensile tests and constant-strain-rate tests at rates of 0.1 and 0.004 percent per hour were used. The specimens were brought to temperature and the load applied rapidly until the strain rate approached the required level. The load was then adjusted to keep the rate constant. The tests were continued until two percent strain was attained. From curves of stress versus time, values of stresses corresponding to strains of 0.25, 0.5, 1.0 and 2.0 percent were obtained. The tests were conducted every 100°F so as to cover the temperature range where the manifestations of strain aging are normally observed in tensile tests, to the upper part of the creep temperature range of the subject alloys.

Part of the tests were conducted using manual stress adjustment in standard creep units. The rest of the tests were conducted in the automatic constant-strain-rate machine. The automatic machine kept the strain rate more constant than in the manual tests. More important, however, was the fact that it did not require constant attention of the operator over long time periods in the faster strain rate tests.

The two carbon steels investigated were heat treated as follows:

Steel C was a silicon-deoxidized steel known to be subject to strong strain aging reactions at 400° to 600°F. Furthermore it was known that its creep resistance was nearly independent of whether it was normalized or annealed, and independent of the temperature of heat treatment. Accordingly, it was first normalized after one hour at 1650°F as the simplest treatment. The aluminum-deoxidized Steel F was annealed from 1650°F to permit nitride precipitation and to keep strain aging at a minimum. In this condition it was referred to as Steel FN. In the tests at a rate of 0.25 percent per hour, the normalized Steel C proved to be considerably stronger than the annealed Steel FN at the lower test temperature due to the difference in carbide structures. Accordingly Steel C was annealed from 1650°F for the tests at 0.004 percent per hour to bring the steels closer together in strength. Also, Steel F was normalized from 2100°F (then referred to as Steel FA) to put nitrogen in solution and make the steel susceptible to strain aging. This resulted in the FA material being stronger at the lower temperature.

A-286 alloy was first oil quenched from 1800°F, the same treatment used by Captain Domian in his hot-hardness study at the Materials Laboratory. Subsequently it was oil quenched from 1650°F in an attempt to lower strength and improve ductility. The data cited in the first section of this report suggested that the 1650°F treatment should result in less solution of the phases which contain elements expected to contribute to strain aging. As discussed in the RESULTS section this was probably not attained due to prior history of the stock.

The serrated nature of the stress-time curves for the carbon steels at the lower temperatures of testing caused a great deal of difficulty in maintaining constant strain rates. The stress required for a given strain rate would build up and then a relatively rapid yielding would occur with the strain rate increase requiring rapid stress reduction. The values at the lower temperatures for carbon steel are therefore only approximate. In the A-286 alloy at the lower temperatures the stress fluctuated up and down but there was no undue amount of the yield point phenomenon found for carbon steel. Consequently the values of stress for the isostrain curves were better established for A-286 at the lower temperatures.

Structural Studies

The isostrain curves of the carbon steels indicated that the strength fell off rapidly between 800° and 1000°F. This suggested that if compounds of nitrogen with silicon and aluminum were forming, they might be "overaged" to the extent that they would be visible by electron microscopic techniques. Accordingly the specimens of Steel C and F in the conditions subject to strain aging were examined after two percent deformation introduced in constant-strain-rate tests at a rate of 0.004 percent per hour.

RESULTS

Constant strain rate tests over wide ranges of temperature were carried out or several strain rates on both the carbon steels and the A-286 alloy.

Carbon Steel

Constant-strain-rate tests were first conducted at a strain rate of 0.25 ercent per hour. The isostrain curves obtained did not show evidence of the train aging peak moving to higher temperature than the usual 400° to 600°F ange for the aging steel C (Fig. 31). The curves for the aging Steel C and he non-aging Steel F remained parallel. There are two features of the results hich require explanation. The "non-aging" Steel F exhibited serrated stress-train curves at the lower test temperatures even though it would not exhibit narked increases in the stress for a given deformation in rapid-strain-rate ests. The second is that the air-cooled Steel C had higher strength than the nnealed Steel F, presumably as a result of the difference in carbide structure.

On the basis that a rate of 0.25 percent per hour might have been too rapid o show the expected effects at small strains, tests were repeated at the slower ate of 0.004 percent per hour. The isostrain curves (Figs 32-34) showed that he strength did not fall off uniformly with temperature and showed distinct nflections at about 800°F for the aging Steel C and the aluminum-deoxidized teel FA treated to dissolve aluminum nitride and exhibit aging characteristics. he latter steel when annealed from 1650°F (Steel FN) to reduce strain aging did ot exhibit the inflection and the strength fell off smoothly with temperature. he differences are shown even more clearly by the comparative curves for he three materials in Figures 35-38.

The solid points in Figure 32 represent tests run at 0.004 percent per hour on ging Steel C which was air cooled after one hour at 1650°F. The previous series f constant-strain-rate tests at 0.25 percent per hour were conducted on Steel C n this condition. The two tests were carried out at 600° and 800°F to illustrate he effect of heat treatment, by altering the carbide structure, on the strength nder conditions of constant strain. The high strength of Steel FA air cooled from 150°F compared to Steels C and FN which were both furnace cooled from 1650°F s attributed mainly to the stronger carbide structure which forms on faster ooling.

Figures 35-38 also illustrate that the strengthening phenomenon prevalent in ging Steels C and FA is strongly dependent on the amount of strain as well as he temperature of testing. Looking at Steels C and FN the maximum difference t 0.25 percent deformation occurs at about 900°F (Fig. 35). As the total strain is ncreased the temperature of maximum difference in stress moved toward lower temperatures reaching 800°F at a deformation of two percent (Fig. 38). The same inflection characteristics are observed for Steel FA only at slightly lower temperatures than for Steel C.

In Figure 33 it is evident that the strengthening phenomenon has become inactive where the strength drops off rapidly for the two-percent total strain at about 800°F. In this temperature range it is observed that the strength for two-percent deformation falls below that for smaller deformations. This condition arises because the material has become weakened to the point where load must be removed to maintain the constant-strain-rate. This is equivalent to third-stage creep for constant-load creep tests.

It is evident from Figures 35-38 that after passing the inflection point the strengths of the aging steels drop off rapidly approaching the lower strength of non-aging Steel FN with increasing deformations and temperatures. The mechanism which is responsible for the strain-activated strengthening seems to become inactive or lose its effectiveness when either the temperature or plastic strain becomes too high.

Structural Studies

Typical electron microstructures of the matrix are shown in Figure 39. The small particles in the matrix of Steel C are assumed to be silicon nitride while those in Steel FA are assumed to be aluminum nitride. They closely resemble the particles identified as nitrides by Leslie et al. (Ref. 12). The polystyrene balls included in the pictures were 3400 A in diameter. They thus indicate that the particles were 200 to 500 A in size.

Steel FA developed recognizable particles in the matrix in less than 500 hours at 1000°F with out stress or plastic strain (Fig. 40). No recognizable particles were present in the as-heat treated material and material heated for 72 hours. They may have been present after 144 hours and definitely were present after 500 hours. Very little change could be seen in Steel C after 500 hours (Fig. 41). This indicates that silicon nitride does not form as readily as aluminum nitride. A more drastic treatment of 240 hours at 1200°F appeared to develop extreme general precipitation in Steel FA and very little in Steel C.

Attempts to obtain electron diffraction patterns of the particles were not successful. The method used was to extract the particles on carbon replicas.

A-286 Alloy

Tensile tests as well as slower constant-strain-rate tests were conducted on A-286 alloy. Material solution treated at both 1650° and 1800°F was investigated to determine if the greater solution effects from an 1800°F treatment would show up in the results.

The way that normal manifestations of strain aging appear in this alloy is rough serrated stress-strain curves over a limited temperature range. This mperature range was from 500° to 1200°F after solution treating at 1800°F, ith maximum serrations in both frequency and intensity occurring at 900°F. ensile testing on this alloy after solution treating at 1650°F showed a marked ecrease in the serrated nature of the stress strain curves. The behavior was uite similar in that sharp yield points were not observed at any temperature nd that the serrations, when they occurred, were similar in nature to those bserved for the 1800°F solution treatment. However, the temperature range here serration was evident was smaller and the severity of the serration bserved was markedly less than that observed for the 1800°F solution treatment. t 500°F only very minor irregularities or serrations were observed; these creased in intensity with increasing temperature at a maximum at about 800°F en decreased rapidly above 900°F to an almost completely smooth curve at 1000°F nd higher.

From the stress-strain curves obtained values of stress for deformations of .5, 1.0 and 2.0 percent were plotted as a function of temperature and appear in igure 42. In comparing these curves it can be seen that strengths are maintained t a high level for both conditions over the range where strain aging is evidenced y the serrated curves. The strengths seem to drop off at a lower temperature r the 1650°F condition which fits in with the fact that serrated stress strain urves cease to be evident at a temperature of 100° to 200°F lower than for the 800°F condition.

Figure 43 shows the comparison of the ultimate and breaking strength for the vo conditions as a function of temperature. It is evident that up to a temperature f 1000°F there is no significant difference between the two conditions. At this oint the strength dropped off markedly for the 1650°F condition, whereas trengths remained high up to almost 1200°F for the 1800°F condition before ropping off sharply.

Figure 44, presenting the reduction of area and elongation from tensile sting, shows the effect of solution temperature on these properties. The eduction of area curve indicates that the 1650°F condition exhibits higher reduction f area at low temperatures but falls below the 1800°F condition at temperatures igher than 900°F. Since the differences are not large and no significant ifference is evident in elongation measurements the differences in ductility from ne condition to the other appear to be negligible over the temperature range vestigated.

Constant-strain-rate tests were run at 0.1 and 0.004 percent per hour up to 400°F. At all temperatures between 1000° and 1400°F a decrease in stress was ecessary to maintain a constant strain rate which is the equivalent of third-stage creep for constant-load creep tests. Figures 45 and 46 showing the comparative results of the constant-strain-rate tests for the two treatments illustrate that very little difference in strength occurs at deformations of 0.5, 1.0 and 2.0 percent up to a temperature of 1200°F.

These tests indicate a strain-aging effect during tensile testing which was somewhat more intense in the material treated at 1800°F than in that treated at 1650°F. However there was no evidence of a contribution from strain aging in either condition for slower tests at rates of 0.1 or 0.004 percent per hour. The isostrain curves started to fall off regularly with temperature. The only unusual features of the curves were related to the onset of third-stage creep causing a reversal of the strength order from 2.0 to 0.5 percent deformation at the lower temperatures to 0.5 to 2.0 percent at the higher temperatures.

There are, however, features of the data which raise questions as to whether or not the strain-aging reaction was contributing to strength in the slower strain rate tests. The early onset of third-stage creep would tend to obscure movement of peaks or inflections to higher temperatures. The evidence of strain aging in the tensile tests was most marked at temperatures below 1200°F, whereas the most interest in creep properties for A-286 alloy is at 1200° to 1350°F. Secondly, there was no difference between the material treated at 1650° or 1800°F. Usually A-286 alloy shows a marked increase in strength from the increase in solution temperature. Some unknown factor in the prior history of this heat resulted in equal strength for the two treatments. The creep resistance was therefore abnormal. It is suspected that the greater solution of carbonitrides had already occurred in the material before treatment at 1650°F. It was shown in the first section of this report that heating or working at high temperatures followed by heat treatment at 1650°F could result in much higher strength than in initially weak material treated at 1650°F. Even though the tensile tests indicated reduced susceptibility to strain aging after treatment at 1650°F the material could have had more than adequate solution effects for the contribution to creep resistance.

Finally, samples treated at both temperatures fractured with extreme brittleness during the constant-strain-rate tests at 0.004 percent per hour at 1000° and 1100°F. The A-286 alloy in both conditions was apparently severely embrittled in this temperature range by the strain-activated reaction, even in the slow-strain-rate tests.

GENERAL DISCUSSION AND SUMMARY

Creep Properties

Fairly good evidence was obtained to indicate that the same type of reaction which causes strain aging in carbon steel operates at higher temperatures under the slow-strain-rate conditions of creep and rupture tests. Although marked evidence of strain aging was found in tensile tests for the A-286 material for temperatures below 1200°F, evidence was not found to show an extension of the reaction to temperatures of 1200°F or higher in constant-strain-rate tests.

The characteristics of the A-286 stock investigated suggested that this may ave been due to the fact that prior history effects made the as-received stock usceptible to strain-aging and the treatment at 1650°F did not sufficiently reduce he strain-aging characteristics so that an effect would be evident by contrast ith the constant-strain-rate characteristics of material treated at 1800°F. The irst section of this report contains evidence that certain prior working conditions ould cause this to happen.

The results for carbon steel supported the original concepts under which he investigation was undertaken. They strongly point to a close connection etween strain-aging and superior creep strength. The data for A-286 alloy ere encouraging only in the sense that the presence of strain-aging was emonstrated. It is expected that additional experiments on A-286 alloy treated o reduce susceptibility to strain aging would also show evidence that there is close connection between strain-aging and high creep strength.

Structural Considerations

It has long been recognized that the creep resistance of carbon steel is ighly dependent on deoxidation practice. More recently it has been recognized hat the effects of certain heat treatments is related more to the deoxidation ractice than to the carbide structure insofar as creep resistance is concerned. n general the relations are as follows:

1. Undeoxidized steel has high creep resistance up to about 800°F independent f heat treatment.

2. Silicon-deoxidized steel has creep resistance on the high side of the range r carbon steel to at least 1000°F. Again it is nearly independent of the prior eat treatment.

3. The use of aluminum for deoxidation results in strength on the low side of e range for carbon steel unless the steel is heat treated by rapidly cooling from bove the grain coarsening temperature of the austenite. Usually more than 1.5 ounds of Al per ton must be added to cause this effect. Air cooling of small ection sizes is sufficient to give high strength.

Dickinson (Ref. 13) showed that these variations in creep resistance were overned by the nitrogen in solution after heat treatment. Removing the nitrogen rom solution in silicon-bearing steels resulted in low strength regardless of heat reatment. Nitrogen was removed from solution by vacuum melting, vacuum xtraction and by prolonged heating. Air cooling aluminum-deoxidized steel om above the coarsening temperature of the austenite kept nitrogen in solution.

One of the most striking features of the creep resistance of carbon steel is the close association between strong strain aging response and high creep strength. Because the theory of strain aging and Cottrell atmospheres requires that slowing down the strain rate should move the strain-aging effects to lower temperatures, some other features must be present to have strain-aging-type reactions improve creep resistance.

The hypotheses under which the experiments were undertaken were substantiated by the results. In materials subject to strain aging, the strain-aging peaks or inflections of isostrain stress versus temperature curves are maintained to higher temperatures as the strain rates are reduced and the total strains are kept low. Moreover, particles were observed in the matrix of carbon steel samples which were probably silicon or aluminum nitrides developing under conditions where the strain-aging-type reaction apparently overaged. This lends support to the theory that nitrogen segregates to the dislocations. When elements reactive with nitrogen are present they are chemically attracted to the nitrogen to make the Cottrell-type atmospheres effective to higher temperatures than would be the case for nitrogen alone. Undeoxidized steel does not retain its creep resistance to high temperatures because reactive elements are not present to stabilize the nitrogen. Slow strain rates and small strains are probably required to avoid rapid overaging of the nitrides. As is well known silicon nitride does not form as readily as aluminum nitride. Consequently silicon-deoxidized steels generally maintain their creep resistance better than aluminum-deoxidized steels because of their greater resistance to overaging.

It should be recognized that the combination of silicon or aluminum with nitrogen giving maximum strengthing would not be visible under the electron microscope under the conditions used for this investigation. When they did become visible they were probably overaged well beyond their most effective state. In fact it is not yet clear if actual compounds are present when the nitrogen is most effective or if there is some sort of pre-compound chemical attraction to stabilize the odd-size atoms around the dislocations to inhibit creep.

REFERENCES

1. A. P. Coldren and J. W. Freeman, "An Investigation of the Relationship Between Microstructure and Creep-Rupture Properties of Heat-Resistant Alloys," Wright Air Development Center Technical Report 58-204, 1958.

2. T. H. Hazlett and R. D. Hansen, "Influence of Substructure on the Shape of the Creep Curve," *Trans. ASM*, *47* (1955) 508.

3. L. C. Lovell, F. L. Vogel, and J. H. Wernick, "Etching Reagents for Dislocations in Metal Crystals," *Metal Progress*, *75* (May, 1959) 96.

4. F. W. C. Boswell and E. Smith, "Examination of Metals by Transmission Electron Microscopy," ASTM Symposium on Advances in Electron Metallography, ASTM-STP No. 245, 1958.

5. A. P. Coldren and J. W. Freeman, "An Investigation of Three Ferritic Steels for High-Temperature Application," Wright Air Development Center Technical Report No. 57-40, ASTIA Document No. 118204 (April, 1957).

6. B. Ancker, T. H. Hazlet, and E. R. Parker, "Relationship Between Small-Angle Boundaries and Creep," *J. Appl. Phys.*, *27* (1956) 333.

7. S. Weissmann, "Quantitative Study of Substructure Characteristics and Correlation to Tensile Properties of Nickel and Nickel Alloys," *J. Appl. Phys.*, *27* (1956) 1335.

8. P. Gay, P. B. Hirsch, and A. Kelly, "X-ray Studies of Polycrystalline Metals Deformed by Rolling. III. The Physical Interpretation of the Experimental Results," *Acta Cryst.*, *7* (1954) 41.

9. S. Weissmann, "Method for the Study of Lattice InhomogeneitiesCombining X-ray Microscopy and Diffraction Analysis," *J. Appl. Phys.*, *27* (1956) 389.

10. H. J. Beattie, Jr, and F. L. Ver Snyder, "A New Complex Phase in a High-Temperature Alloy," *Nature*, *178* (1956) 208.

11. H. J. Beattie, Jr. and W. C. Hagel, "Intermetallic Compounds in Titanium-Hardened Alloys," *Trans. AIME*, *209* (1957) 911.

12. W. C. Leslie, R. L. Rickett, C. L. Dotson, and C. S. Walton, "Solution and Precipitation of AlN in Relation to the Structure of Low Carbon Steels," Trans. ASM, 46 (1954) 1470.

13. C. D. Dickinson, "A Study of the Fundamentals of the Effect of Deoxidation on the Creep Characteristics of Plain Carbon Steel," Doctoral Thesis, University of Michigan, June, 1956.

TABLE 1

Influence of Rolling Conditions on Grain Structure and Hardness of Rolled and Heat Treated A-286 Alloy

Rolling Conditions			Recrystallization		After Heat Treatment	
perature 'F) (a)	Reduction of Area(%)	No. of Passes	During Rolling	During Solution Treatment (b)	Over-all Mean Grain Size (ASTM No.)	Hardness (DPH)
80	0.0	0	--	--	10.2	307
	9.4	3	--	Yes	10.1	304
	19.7	5	--	Yes	10.0	306
	28.8	7	--	Yes	10.2	305
	39.5	9	--	Yes	9.8	306
700	0.0	0	--	--	9.9	309
	3.9	1	No	No	10.0	318
	8.4	2	No	Yes	7.5	319
	21.5	2	No	Yes	9.8	299
	31.7	2	No	Yes	9.9	304
	11.5	3	No	Yes	7.6	299
	17.7	3	No	Yes	8.0	302
	37.6	5	No	Yes	9.5	305
950	0.0	0	--	--	6.2	284
	3.9	1	No	No	5.9	301
	9.7	2	No	No	5.9	302
	18.0	2	No	No	5.6	318
	23.5	2	No	No	5.8	312
	34.2	2	No	No	5.6	316
	12.2	3	No	No	5.8	306
	18.8	3	No	No	5.7	302
	38.7	5	No	Yes	7.5	303
200	0.0	0	--	--	0.9	286
	5.4	1	Partial	No	1.6	312
	10.4	2	Partial	No	2.5	325
	20.2	2	Partial	No	4.3	305
	26.1	2	Partial	No	4.7	306
	35.1	2	Partial	No	5.0	311
	14.5	3	Partial	No	2.9	319
	20.8	3	Partial	No	3.6	331
	39.8	5	Partial	No	3.9	335

a) Initial Temperature; no reheats were used.

b) Solution treatment was 1 hour at 1650°F, O.Q., followed by an aging treatment of 16 hours at 1325°F.

TABLE 2

Influence of Hot Working on the Creep-Rupture Properties of "17-22-A" V Steel at 1100°F

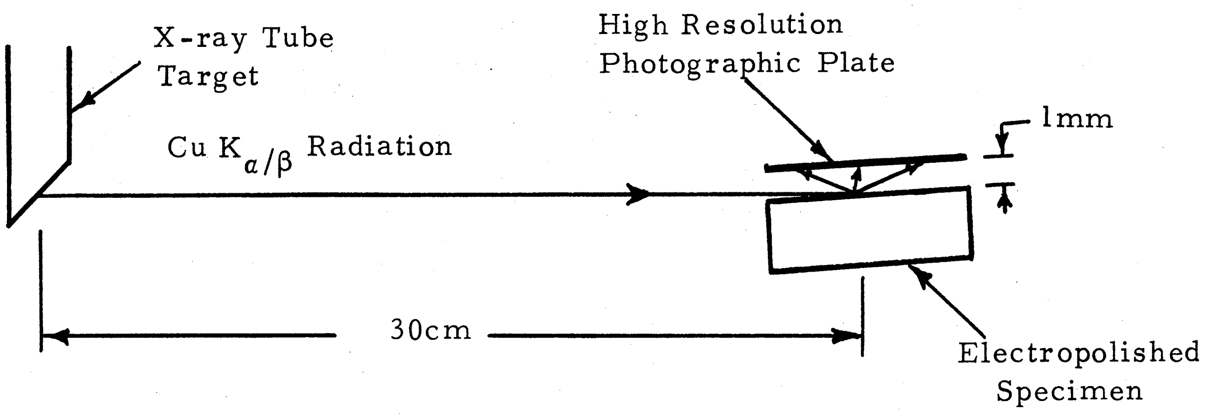
Processing Conditions	Stress (psi)	Rupture Life(hrs)	Elong. (%in 4D)	R.A. (%)	Min. Creep Rate(%/hr)	Def. on Load(%)	Time to Reach Indicated Total Deformation(hrs)			
							0.2%	0.5%	1.0%	2.0%
1 hr. 2200°F, AC* tempered 7 hrs, at 1250°F, AC. G.S. -4.5; BHN-362	40,000	86.0	1.0	1.9	0.0012	0.30	b	80.0	---	---
1 hr. 2200°F, AC to 2100°F and rolled 50% reduction, AC + tempered 6 hrs. 1250°F, AC. (Austenite 100% recrystallized during rolling) G.S. -7.5; BHN-326	55,000	23.8	0.9a	2.0	0.025	0.36	b	<1.0	14.2	---
	40,000	131.0	1.1a	1.6	0.0056	0.30	b	12.0	98.0	---
	29,000	459.8	0.7a	1.2	0.0010	0.16	20.0	280.0	---	---
1 hr. 2200°F, AC to 2000°F and rolled 50% reduction, AC + tempered 6 hrs. 1250°F, AC. (Austenite partially recrystallized during rolling) G.S. -7.5 and 4.5; BHN-349	55,000	38.5	2.7	3.6	0.029	0.37	b	<1.0	11.0	35.0
	40,000	230.7	1.5a	3.2	0.0038	0.27	b	10.0	125.0	---
	29,000	832.6	1.2a	2.4	0.0008	0.22	b	200.0	650.0	---
1 hr. 2200°F, AC to 1900°F and rolled 50% reduction, AC + tempered 6 hrs. 1250°F, AC (No recrystallization during rolling) G.S. -4.5; BHN-355	55,000	32.2	9.1	16.8	<0.63	0.36	b	<1.0	1.5	12.0a
	40,000	324.6	1.5a	4.8	0.0026	0.33	b	3.0	150.0	---
	29,000	1229.1	0.9a	3.6	0.0005	0.22	b	390.0	1135.0	---
1 hr. 2200°F, AC to 1600°F and rolled 60% reduction in 5 passes, AC + tempered 6 hrs. 1250°F,AC. (No recrystallization during rolling) G.S. -4.5; BHN-346	55,000	72.7	6.4	12.7	0.0229	0.37	b	2.0	14.5	49.0
	40,000	493.8	2.7	5.6	0.0020	0.25	b	22.0	258.0	475.0

*AC - air cooled.

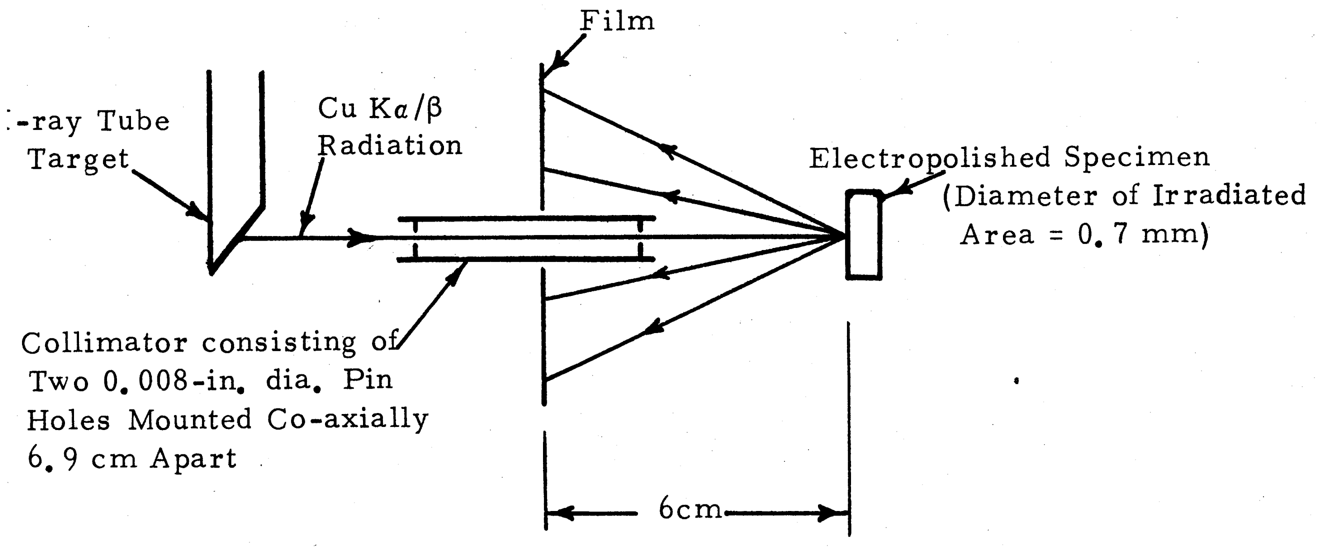
a - Obtained through small extrapolation of elongation versus time curve.

b - Deformation exceeded on loading.

< - "Less than".



(a) Arrangement Used for Berg-Barrett X-ray Reflection Microscopy



(b) Arrangement for Taking Micro-Beam, Back-Reflection X-ray Diffraction Photograms.

Figure 1. Schematic Diagrams of X-ray Diffraction Experiments.

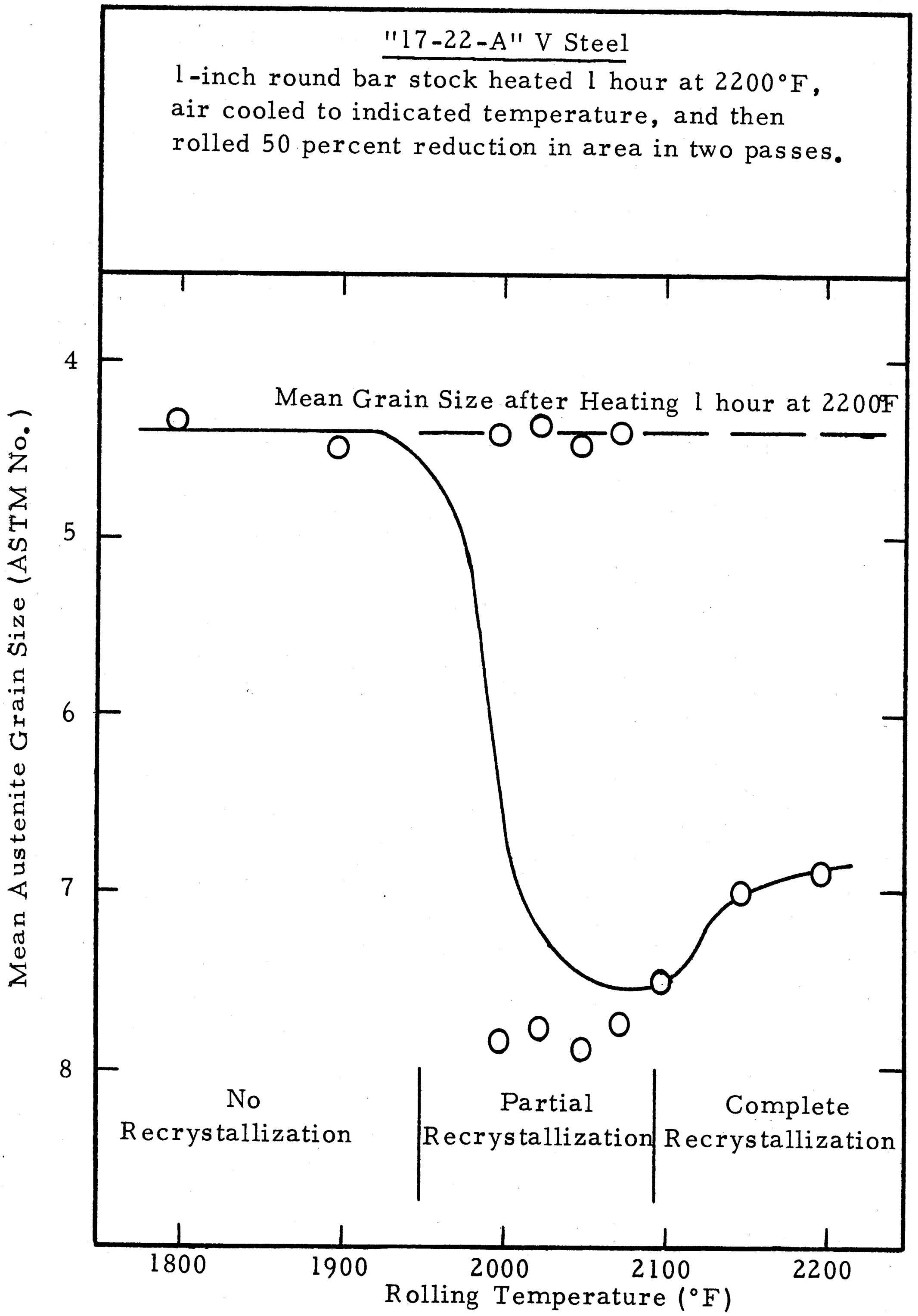


Figure 2. Austenite Grain Size of "17-22-A" V Steel after Heating 1 Hour at 2200°F, Cooling to Indicated Rolling Temperature, and Reducing Area 50 Percent by Rolling.

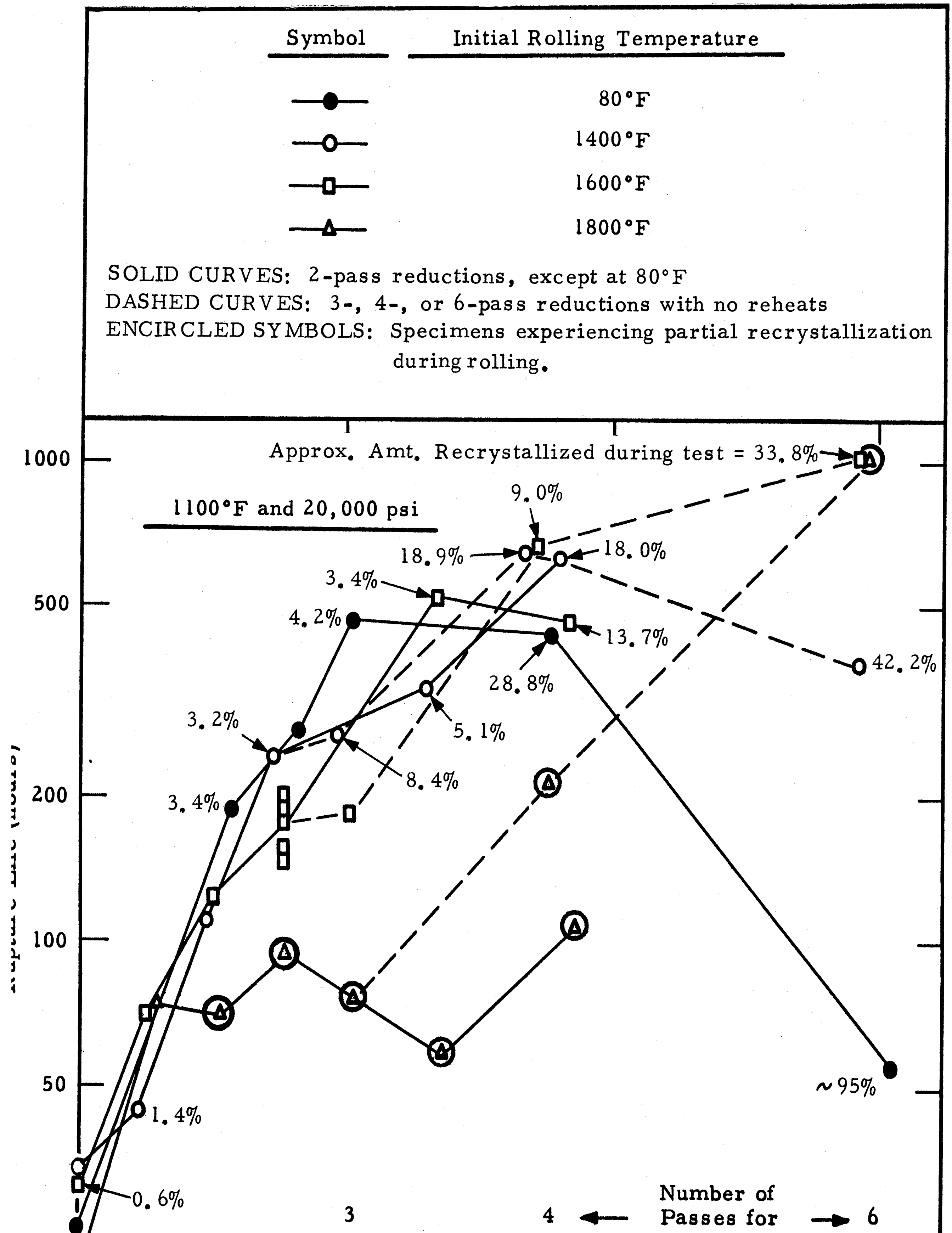


Figure 3. Variation of Log Rupture Life with Rolling Conditions for "A" Nickel Tested at 1100°F and 20,000 psi, Showing Approximate Extent of Recrystallization in Representative Specimens During Testing

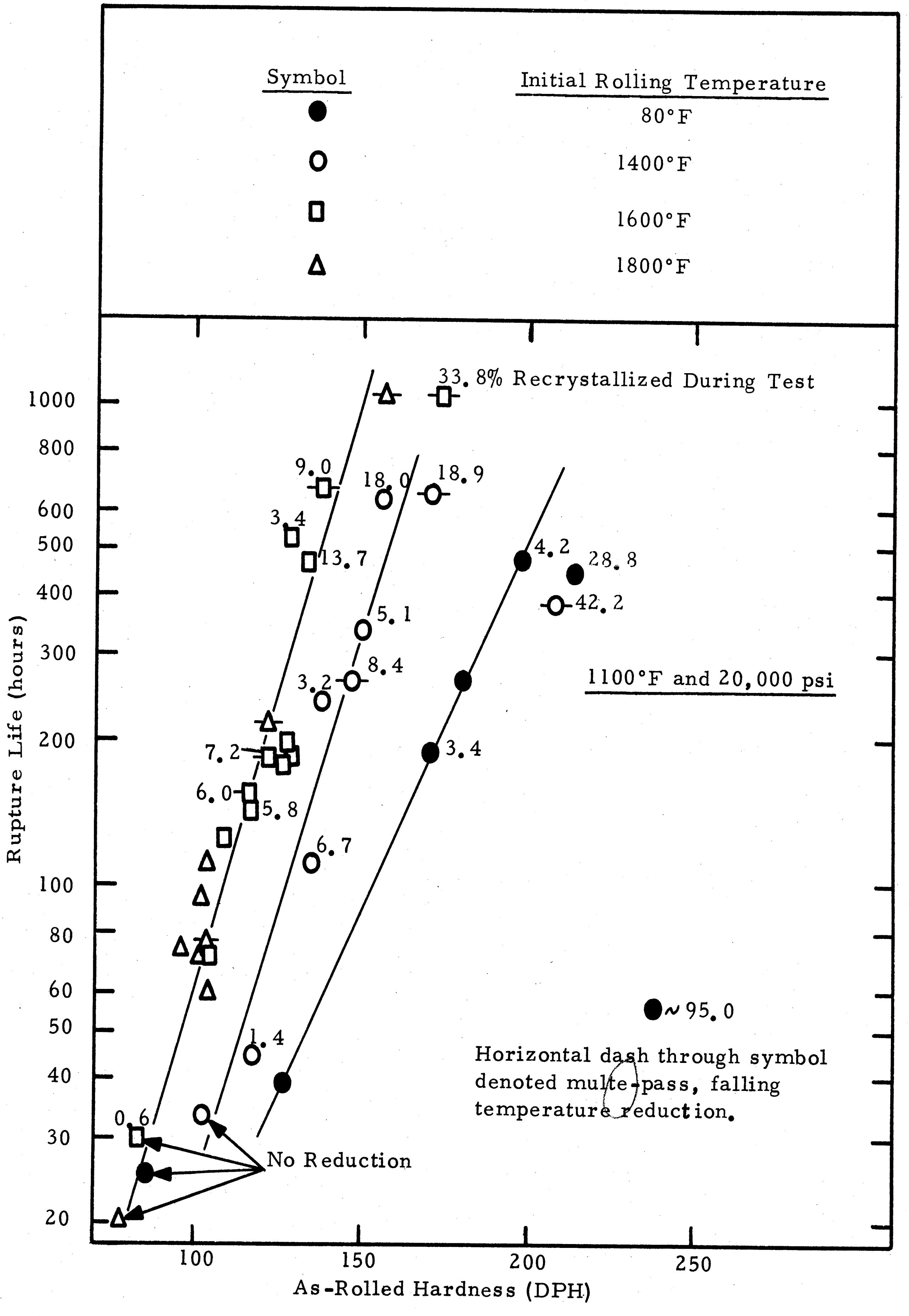


Figure 4. Correlations of Log Rupture Life with As-Rolled Hardness for "A" Nickel Rolled at 80°, 1400°, 1600° and 1800°F and Rupture Tested at 1100°F and 20,000 psi.

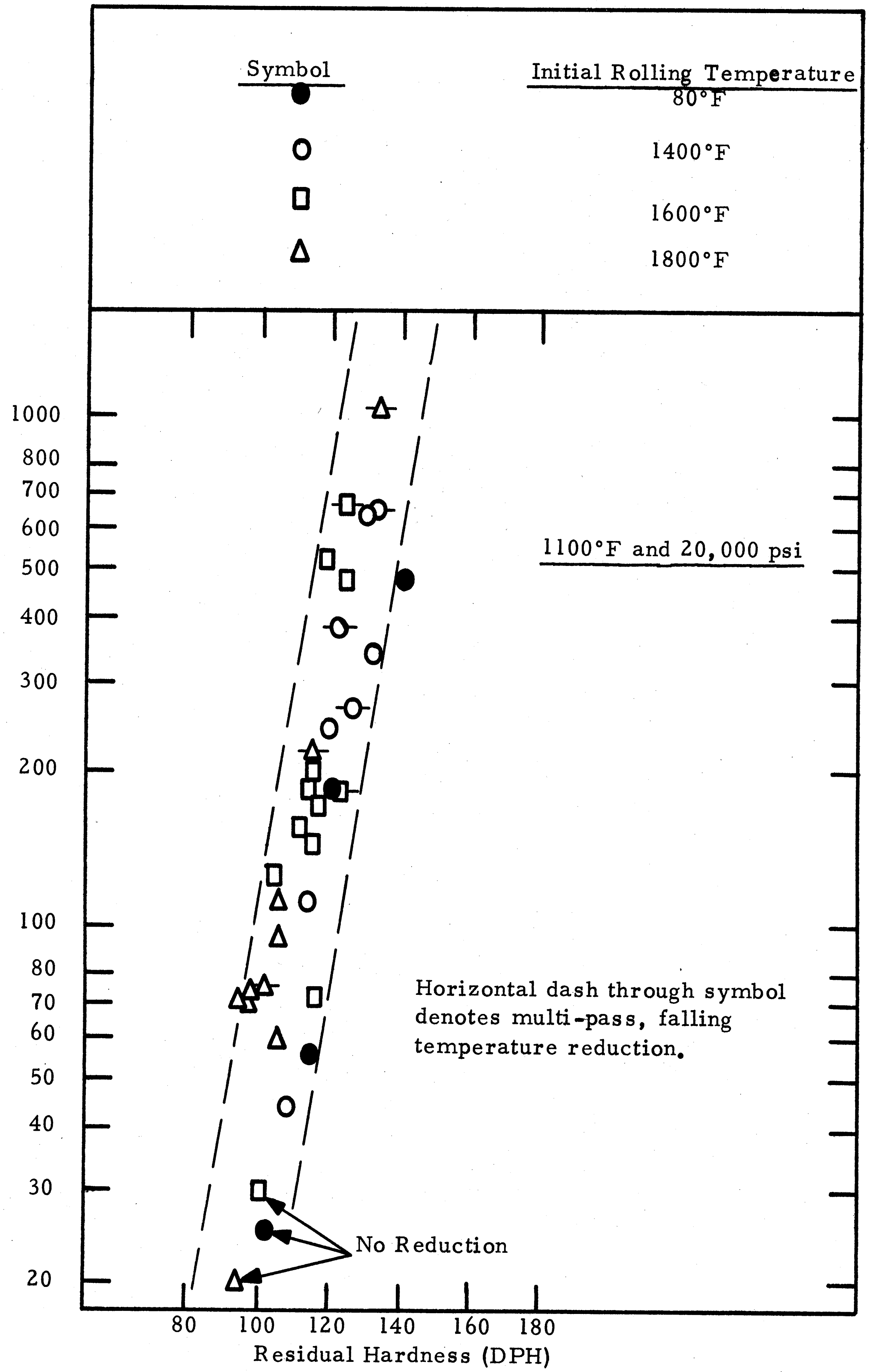
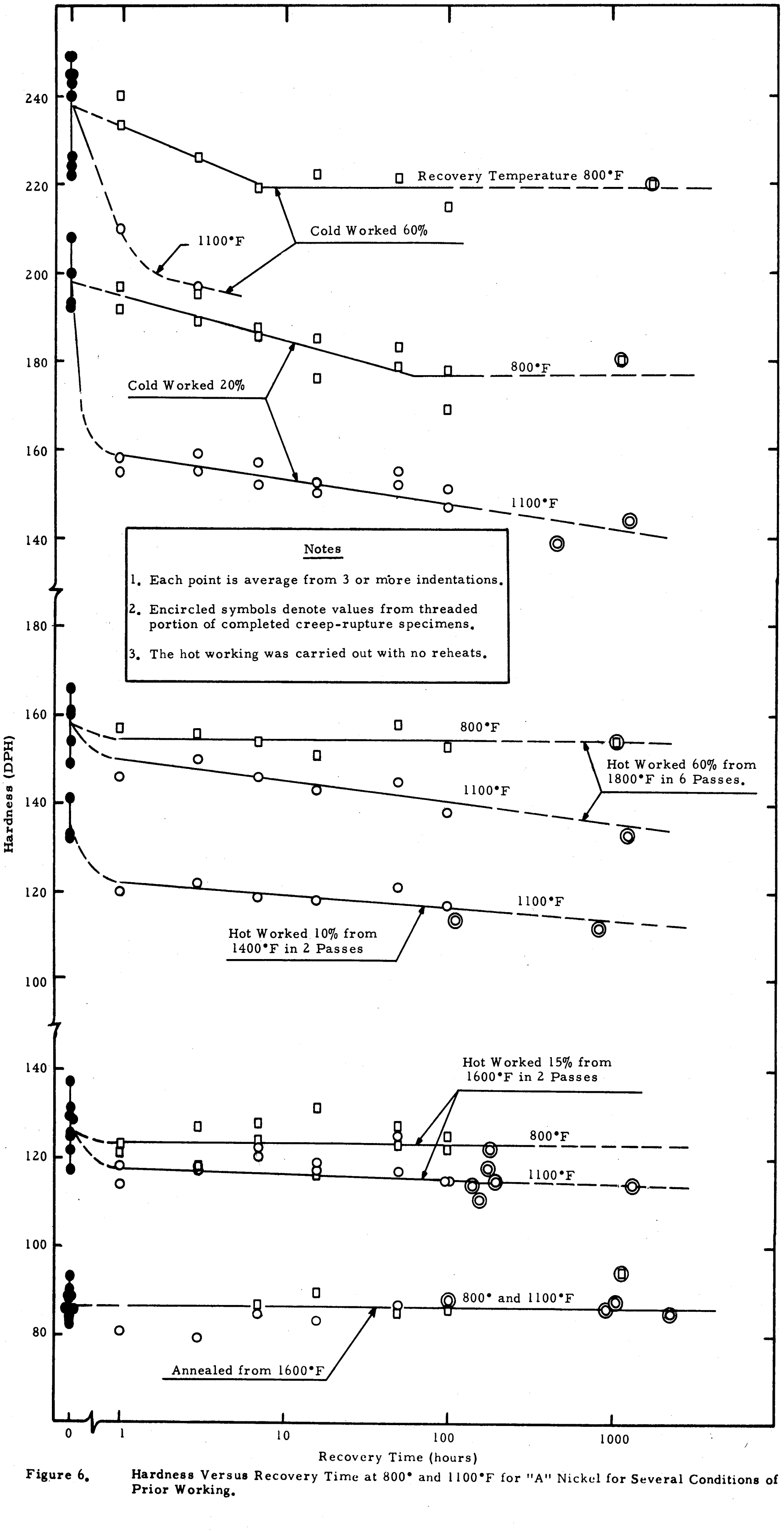


Figure 5. Correlation Between Log Rupture Life and Residual Hardness for "A" Nickel Rolled at 80°, 1400°, 1600°, and 1800°F and Rupture Tested at 1100°F and 20,000 psi.



Notes

1. Each point is average from 3 or more indentations.
2. Encircled symbols denote values from threaded portion of completed creep-rupture specimens.
3. The hot working was carried out with no reheats.

Figure 6. **Hardness Versus Recovery Time at 800° and 1100°F for "A" Nickel for Several Conditions of Prior Working.**

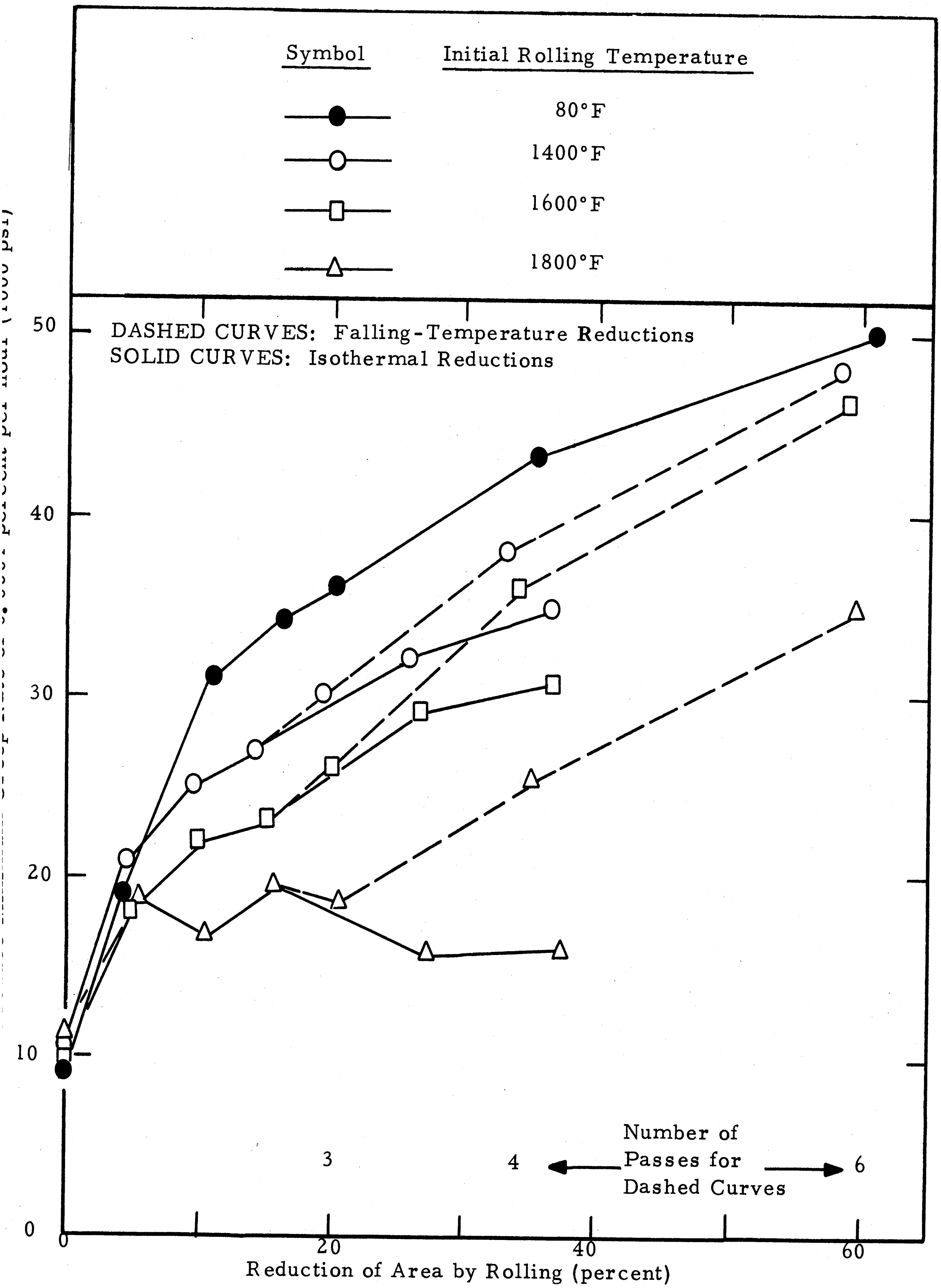


Figure 7. Estimated Stress to Produce Minimum Creep Rate of 0.0001 Percent per Hour at 800°F for "A" Nickel.

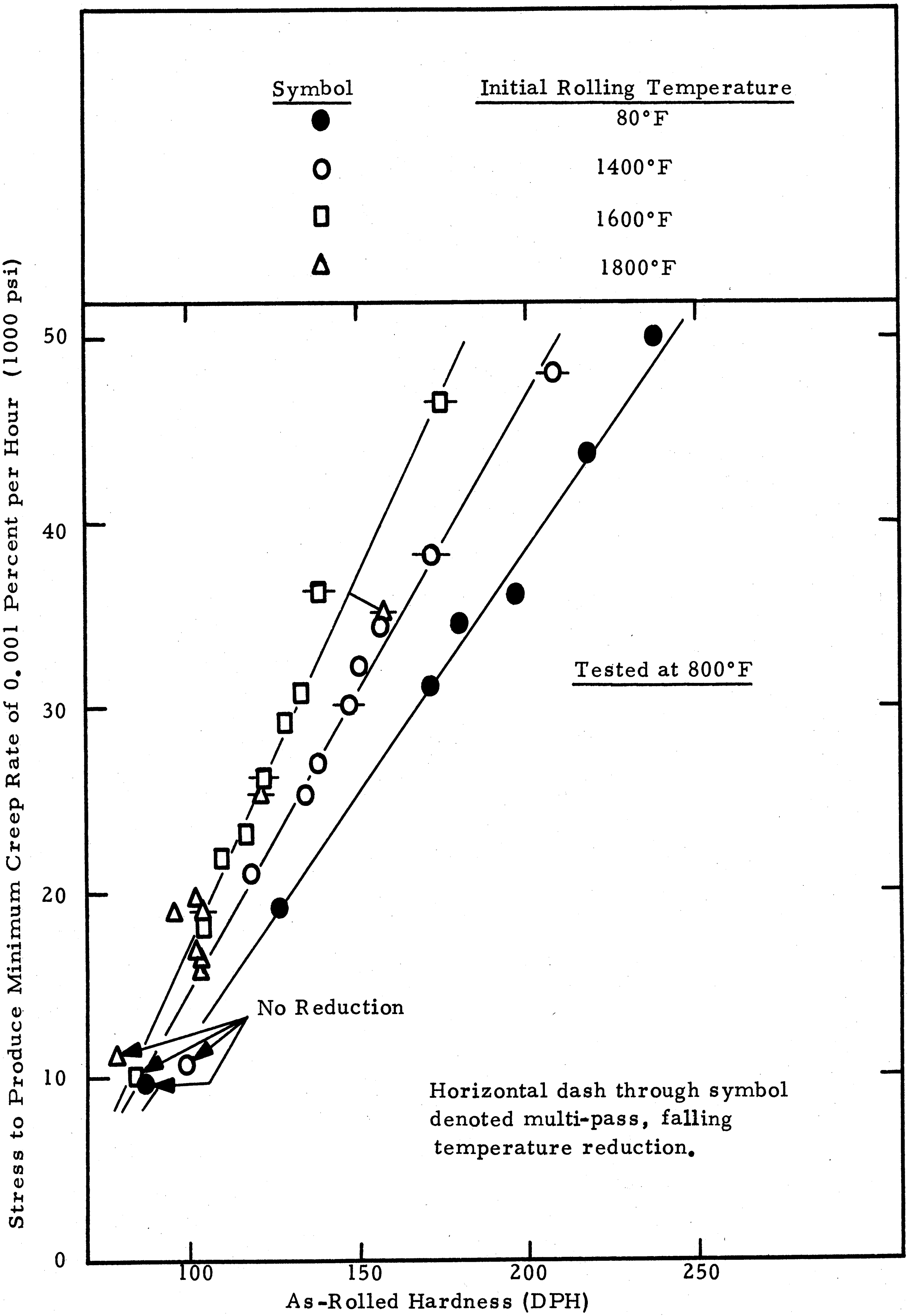


Figure 8. Correlations Between As-Rolled Hardness and the Stress to Produce a Minimum Creep Rate of 0.0001 Percent per Hour for "A" Nickel Rolled at 80°, 1400°, 1600°, and 1800°F and Creep Tested at 800°F.

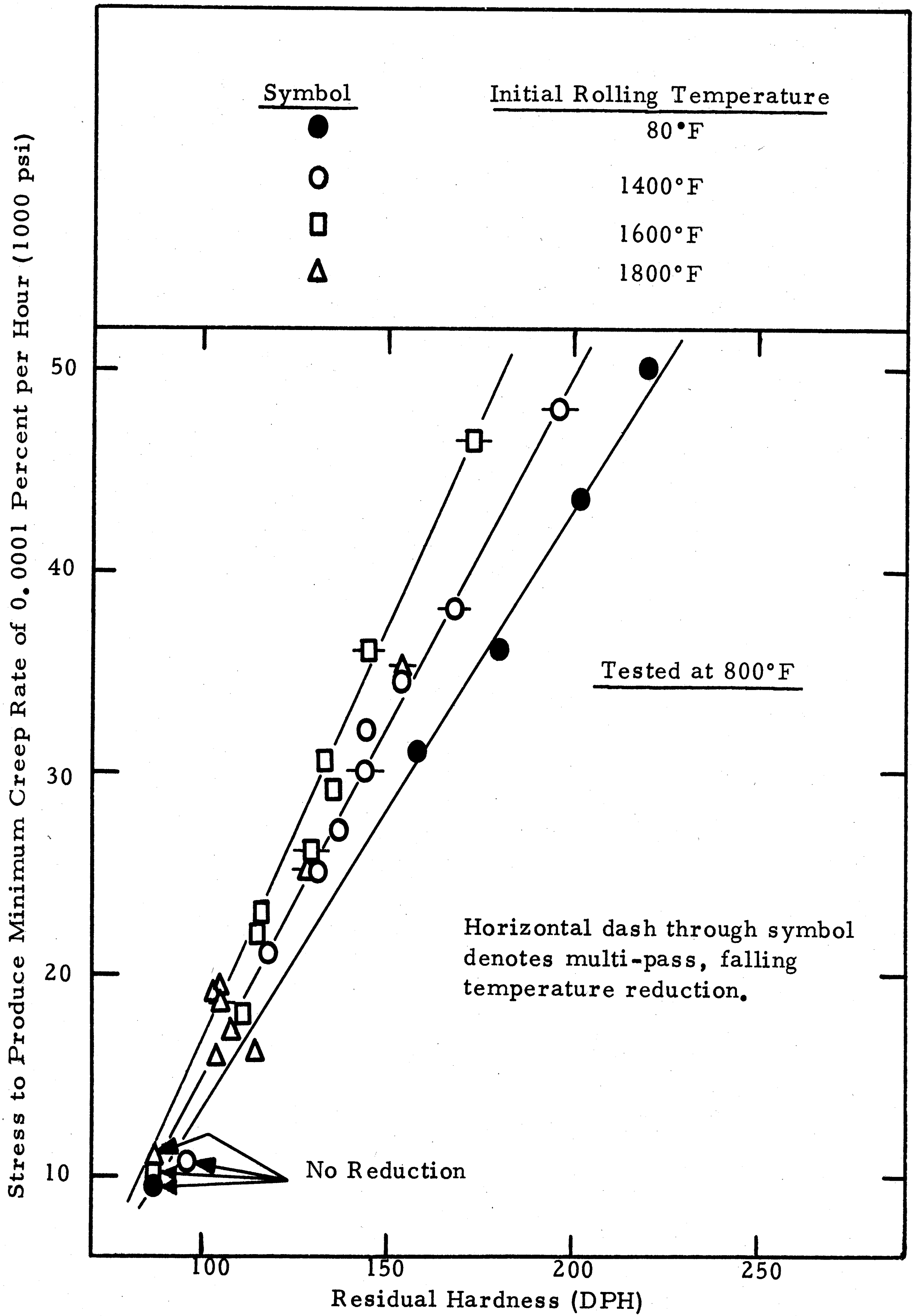
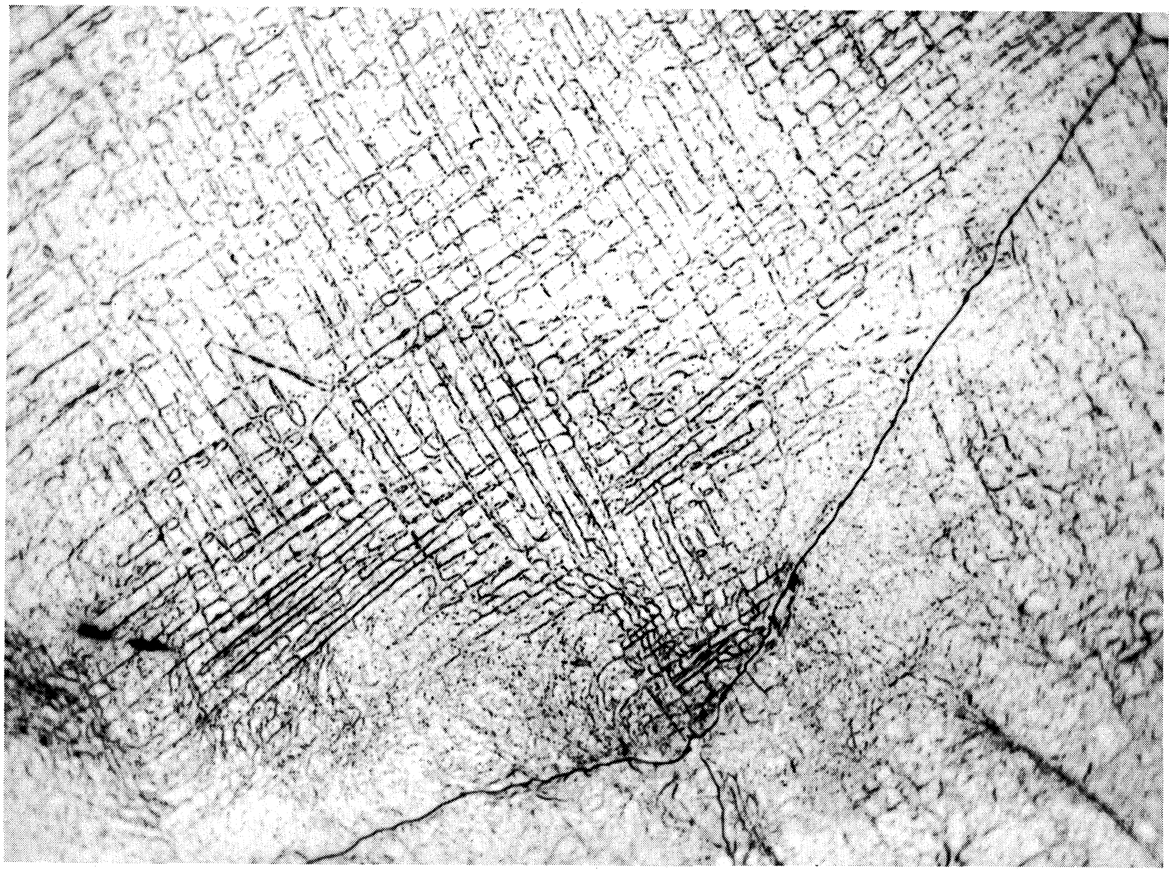
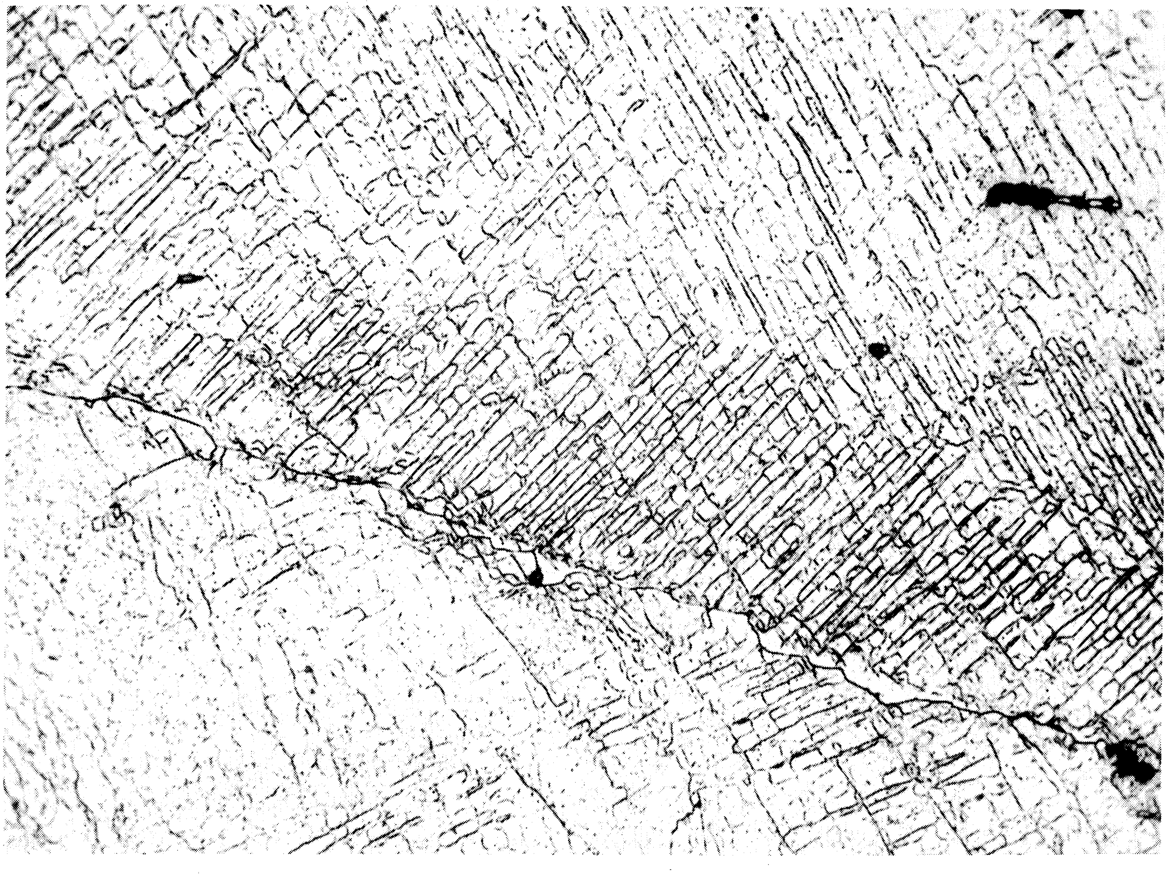


Figure 9. Correlations Between Residual Hardness and the Stress to Produce a Minimum Creep Rate of 0.0001 Percent per Hour for "A" Nickel Rolled at 80°, 1400°, 1600° and 1800°F and Creep Tested at 800°F.



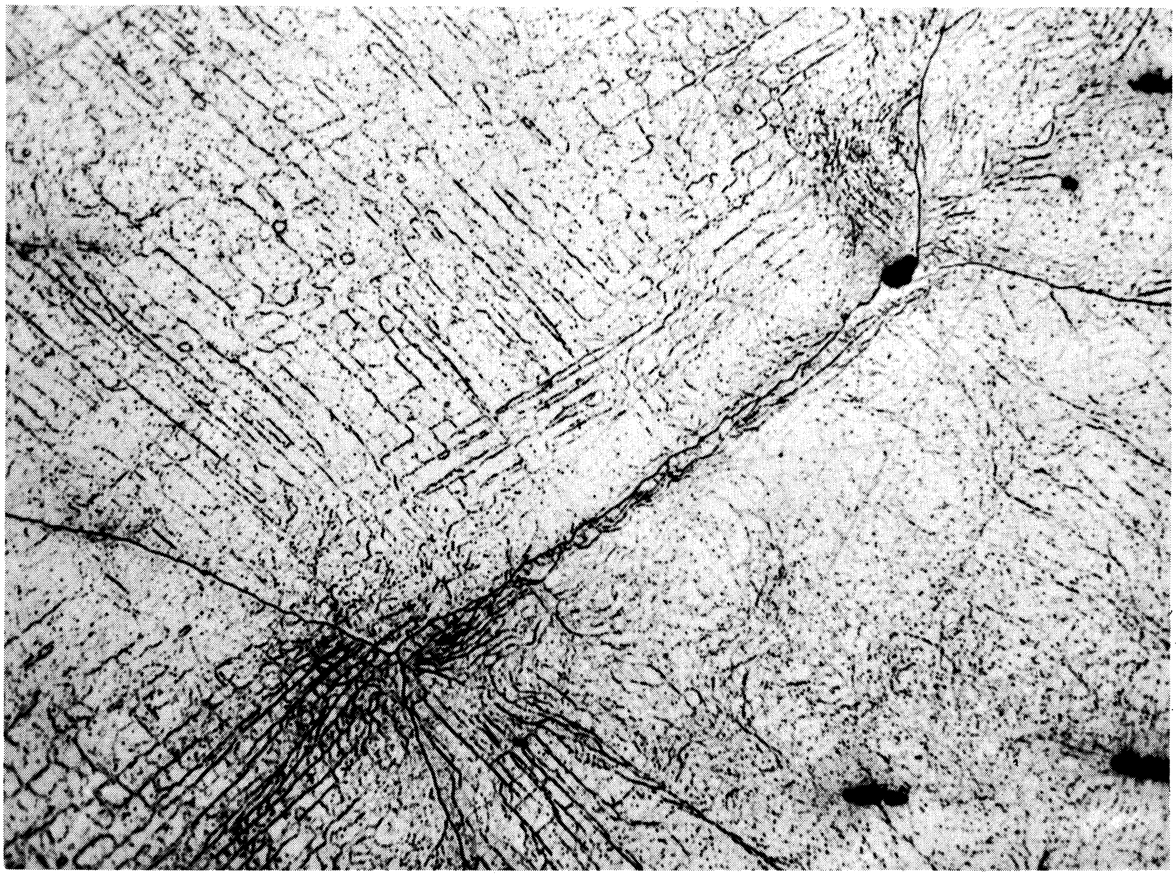
(a) Rolled 5.8 Percent at 1800°F + Tested 702 Hours at 1100°F and 11,000 psi.



(b) Same as in (a) except different area.

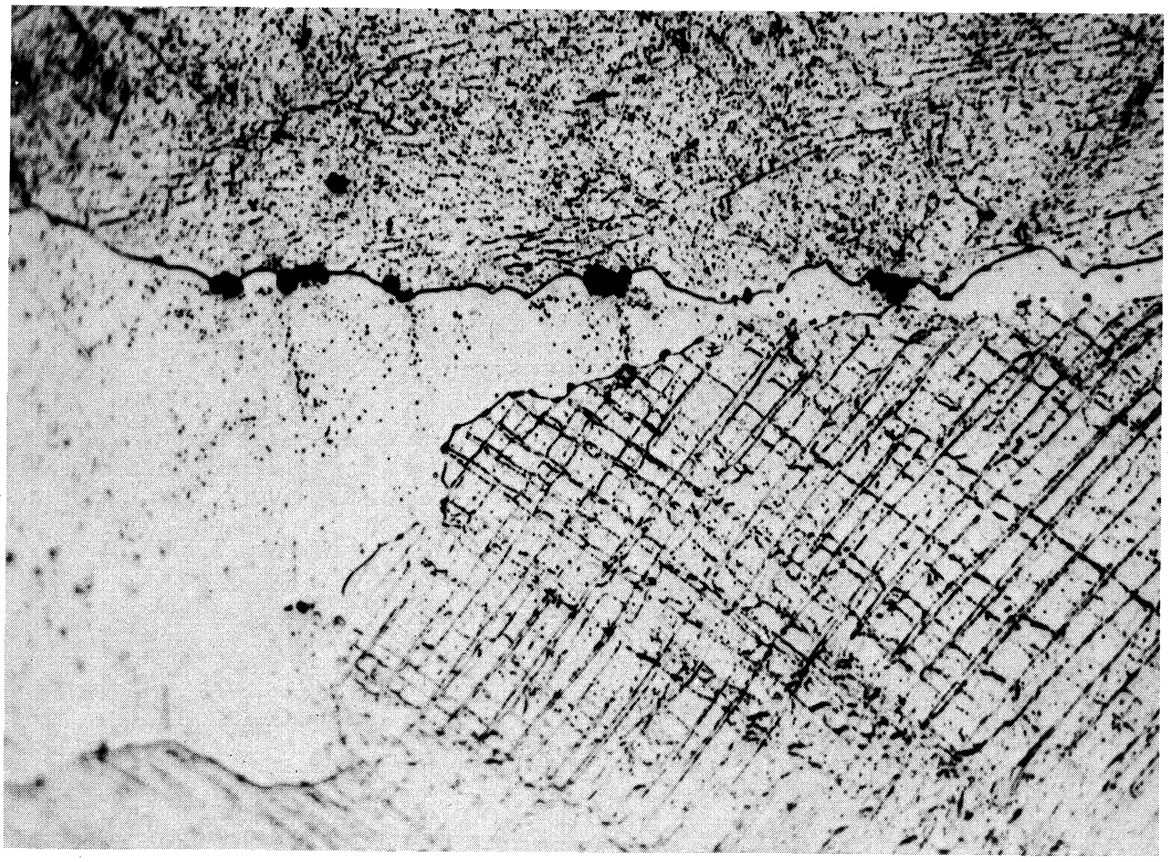
Figure 10. Substructures in Rolled "A" Nickel after Creep Testing. All Micrographs are at X1000 Magnification.

(c) Same as in (a) except different area.

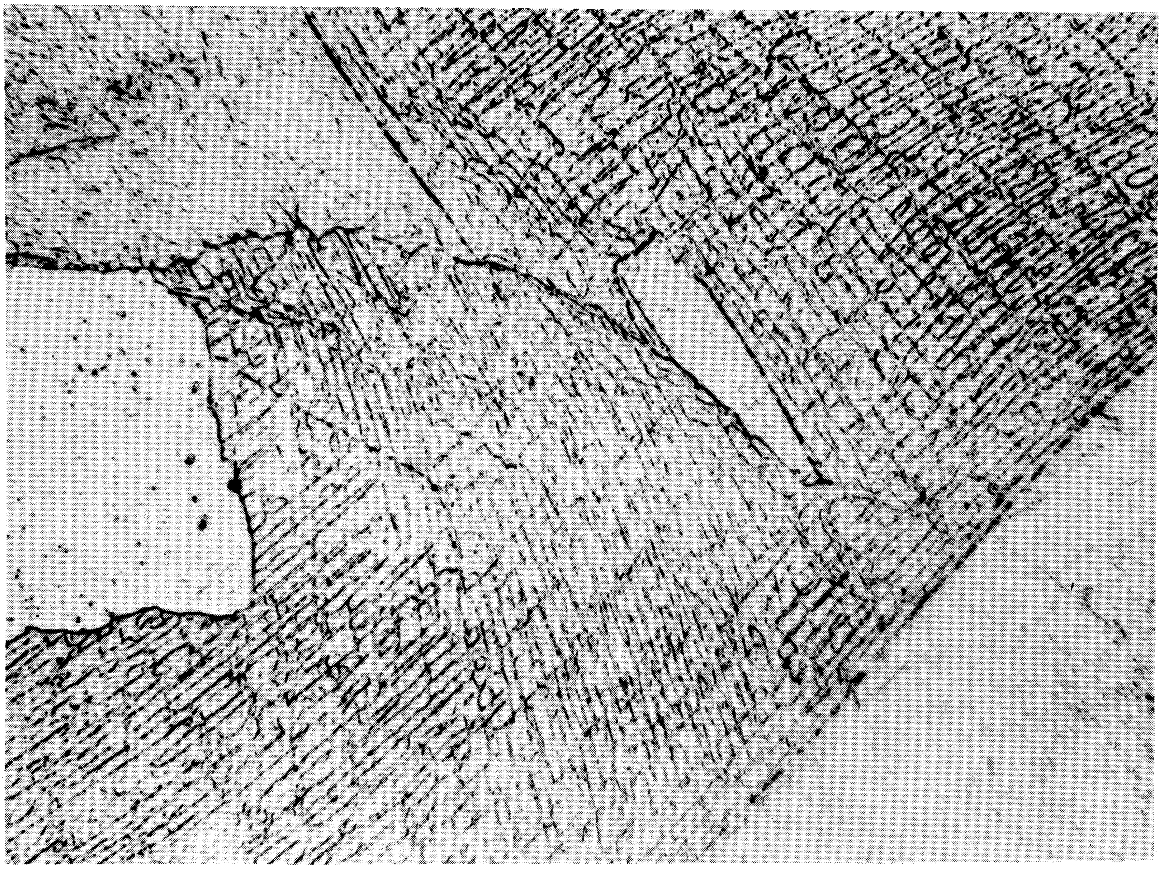


(d) Same as in (a) except different area.

Figure 10. (continued)

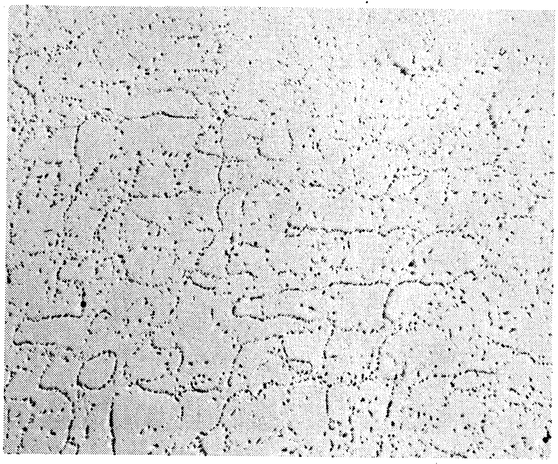


(e) Rolled 15.7 Percent at 1800°F + Tested 843 Hours at 1100°F and 11,000 psi.

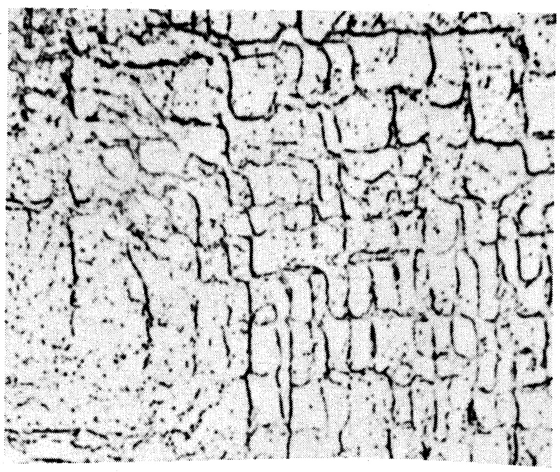


(f) Rolled 27.3 Percent at 1800°F + Tested 988 Hours at 1100°F and 11,000 psi.

Figure 10. (concluded)

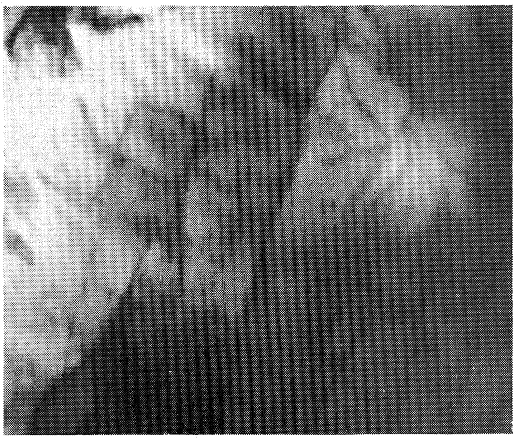


(a) Electron Micrograph from Specimen with Light Electrolytic Etch in Aqueous Solution of 40 Percent H_3PO_4. Mag. 2,200X



(b) Light Micrograph from Same Specimen as in (a), Except More Heavily Etched. Mag. 2,000X

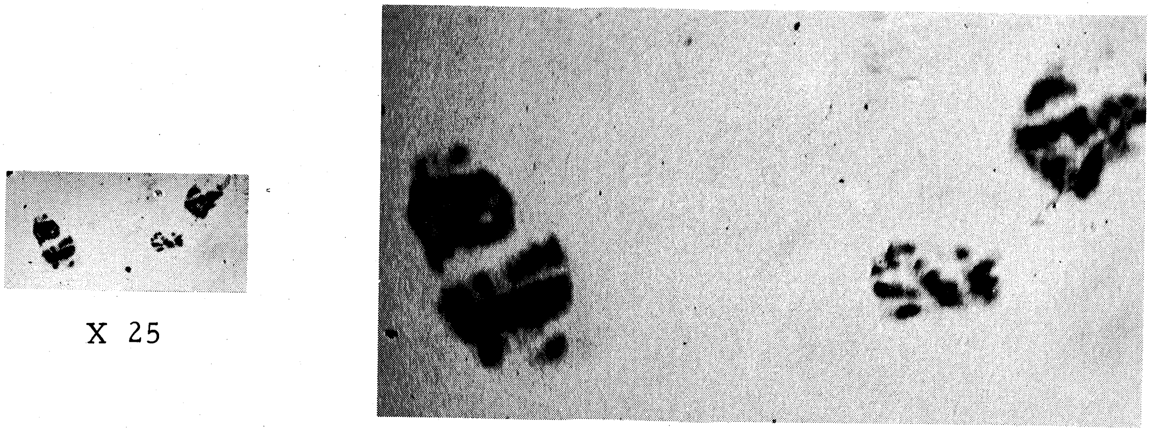
Figure 11. Comparison of Electron Micrograph and Light Micrograph Taken from "A" Nickel Specimen Rolled 5.8 Percent at 1800°F Showing that Solid Black Lines in Light Micrograph are Formed by Rows of Overlapping Etch Pits.



Transmission Electron Micrograph
X 12,000

Ordinary Light Micrograph
X 1,000

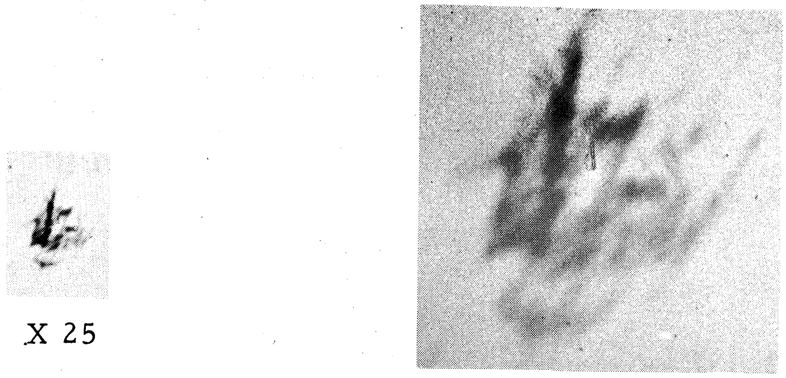
Figure 12. Transmission Electron Micrograph and Ordinary Light Micrograph From An "A" Nickel Specimen Rolled 5.8 Percent Reduction at 1800°F + Creep Tested 702 Hours at 1100°F and 11,000 psi.



X 25

X 100

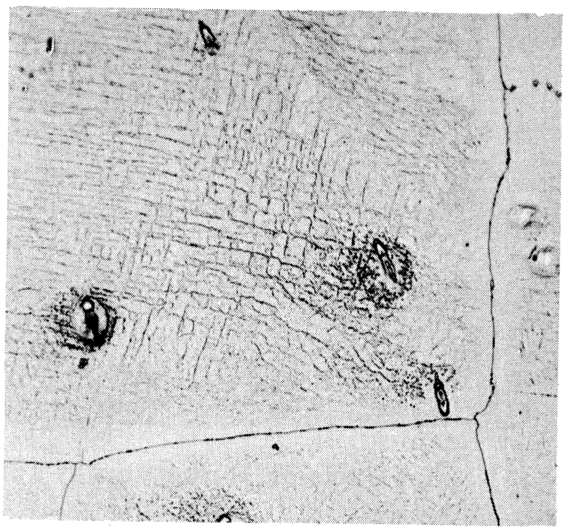
(a) X-ray micrographs of specimen annealed at 1800°F.



X 25

X 100

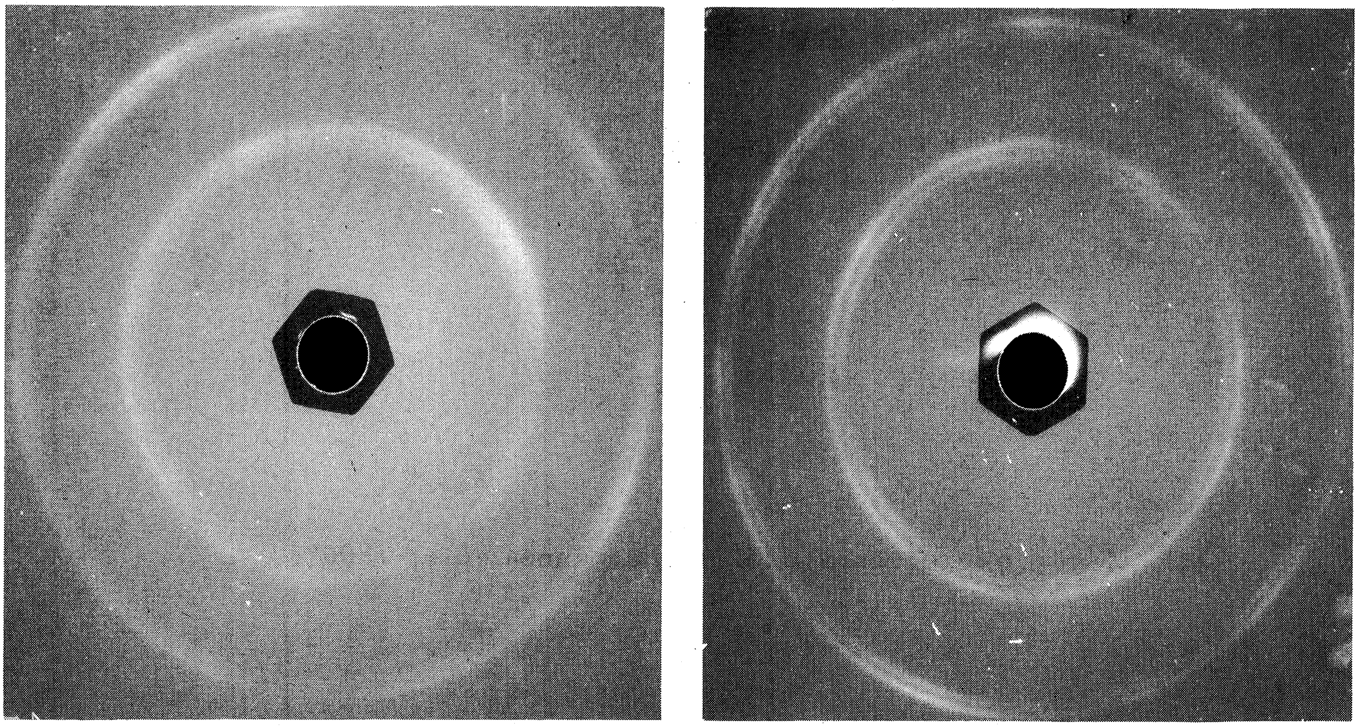
(b) X-ray micrographs of specimen rolled 5.8 percent at 1800°F.



X 500

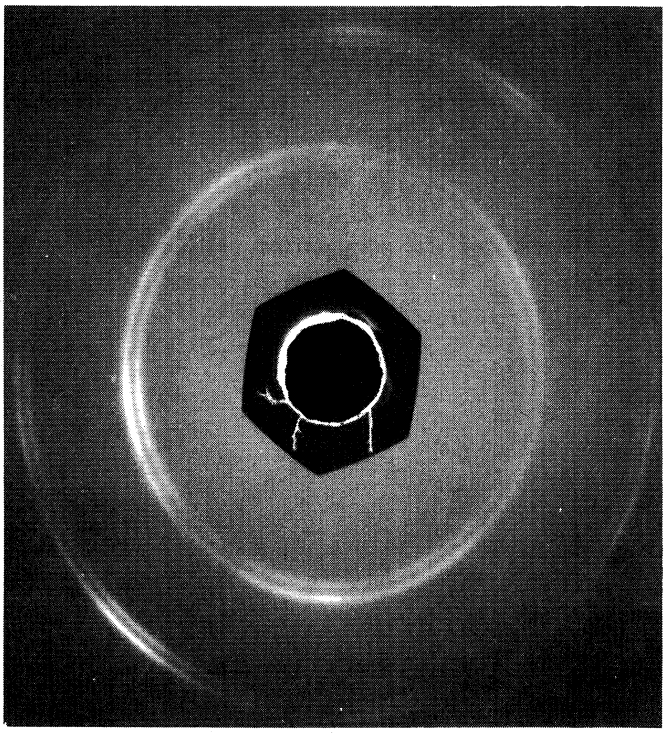
(c) Light micrograph of specimen rolled 5.8 percent at 1800°F.

Figure 13. Comparison of Berg-Barrett X-Ray Micrographs With a Light Micrograph Taken from "A" Nickel Specimens.



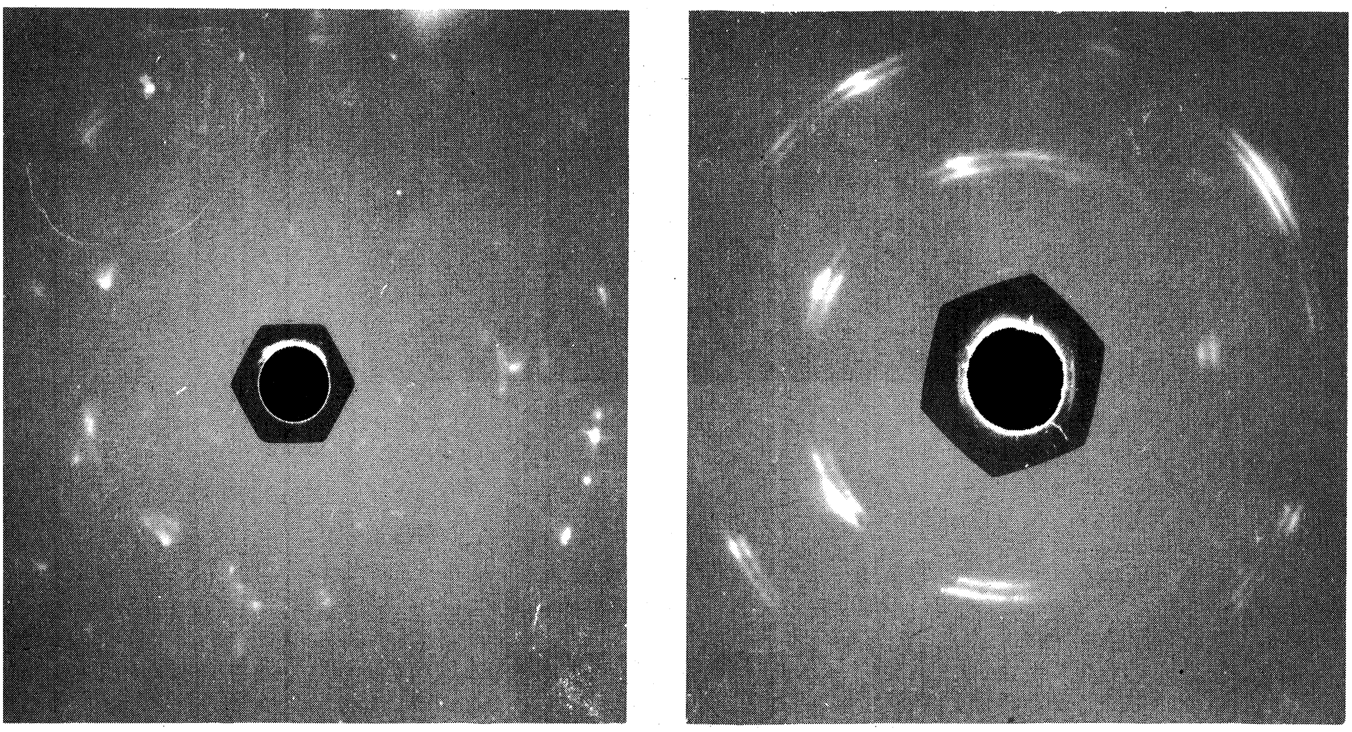
(a) 35.5 Percent Reduction at Room Temperature

(b) 36.1 Percent Reduction at 1400°F



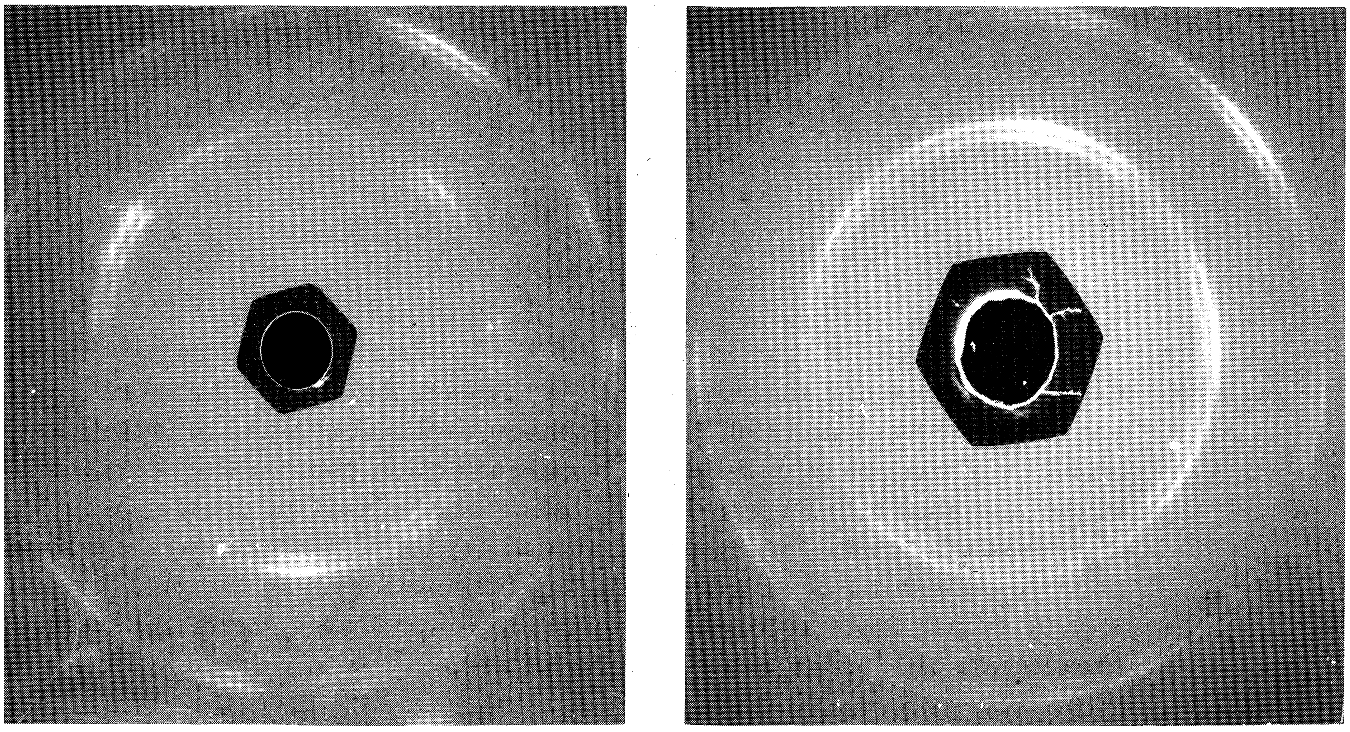
(c) 34.4 Percent Reduction at 1600°F

Figure 14. Micro-Beam, Back-Reflection X-ray Photograms Showing the Effect of Rolling Temperature on the Diffraction Rings from the (331) Planes (Outer Pair of Circles) and the (420) Planes (Inner Pair of Circles) of "A" Nickel Rolled 35 Percent Reduction of Area. Radiation: CuK$_{\alpha/\beta}$. Specimen-to-Film Distance: 6 cm. Diameter of Irradiated Area on Specimen: 0.7mm. Mean Grain Diameter: 0.13 mm.



(a) Annealed at 1600°F

(b) 5.3 Percent Reduction at 1600°F



(c) 9.9 Percent Reduction at 1600°F

(d) 34.4 Percent Reduction at 1600°F

Figure 15. Micro-Beam, Back-Reflection X-ray Photograms Showing the Effect of Percent Reduction of Area by Rolling on the Diffraction Spots from the (331) Planes (Outer Doublets) and the (420) Planes (Inner Doublets) of "A" Nickel Rolled at 1600°F. Radiation: CuKα/β Specimen-to-Film Distance: 6cm. Diameter of Irradiated Area on Specimen: 0.7 mm. Mean Grain Diameter: 0.13 mm.

Figure 16. X-ray Evidence of Substructure in "A" Nickel Rolled 5.8 Percent Reduction of Area at 1800°F. The photographs above are enlarged (2.6X) portions of micro-beam, back-reflection photograms similar to the one shown in Figure 15 (b). Each arc or pair of doublet arcs corresponds to one grain, and the small spots within each arc correspond to subgrains of slightly differing orientations. In the photographs above, small spots 1mm apart would correspond to subgrains having an orientation difference of 20 minutes of arc.

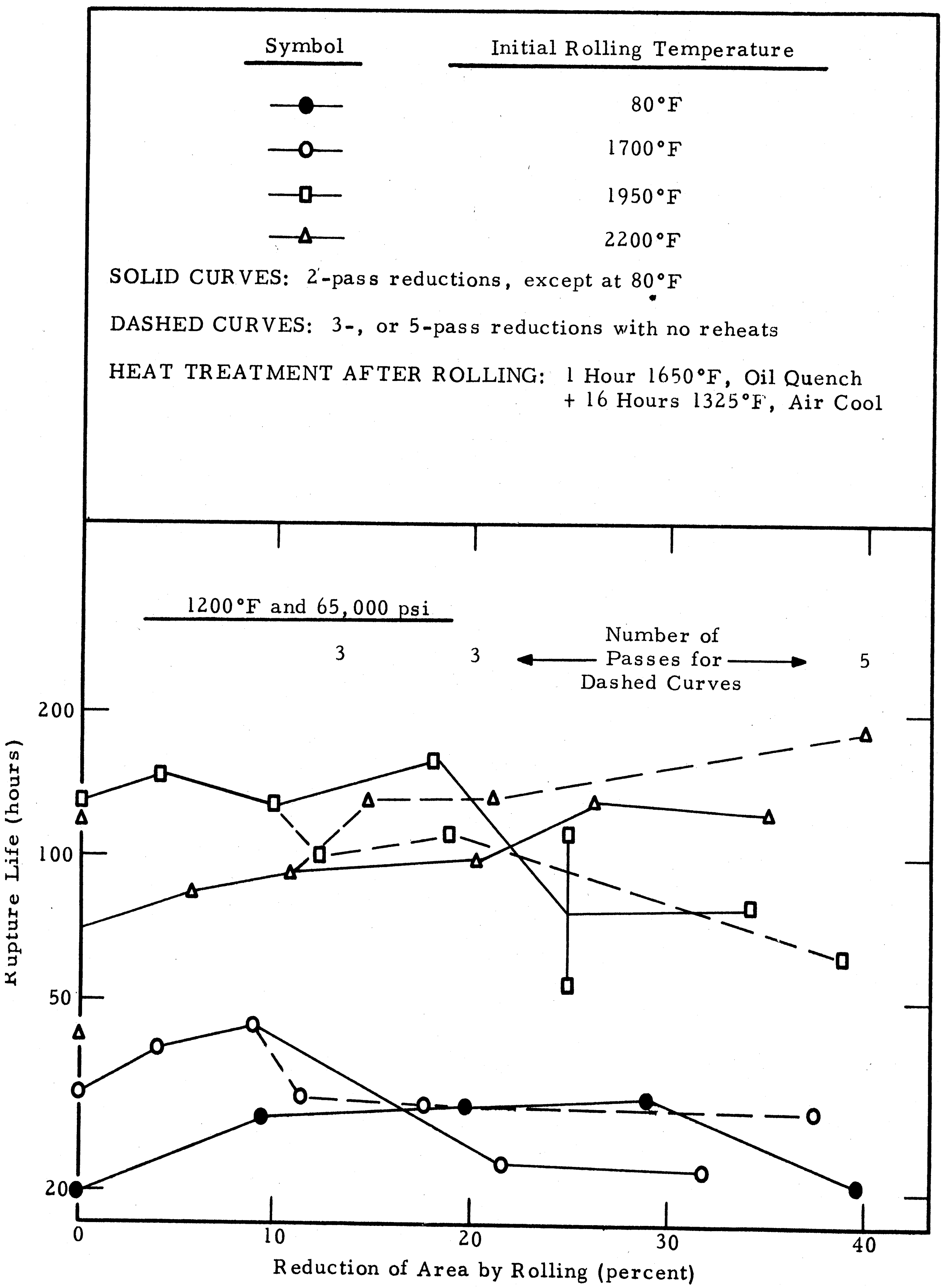


Figure 17. Variation of Log Rupture Life with Prior Rolling Conditions for Heat Treated A-286 Alloy Tested at 1200°F and 65,000 psi.

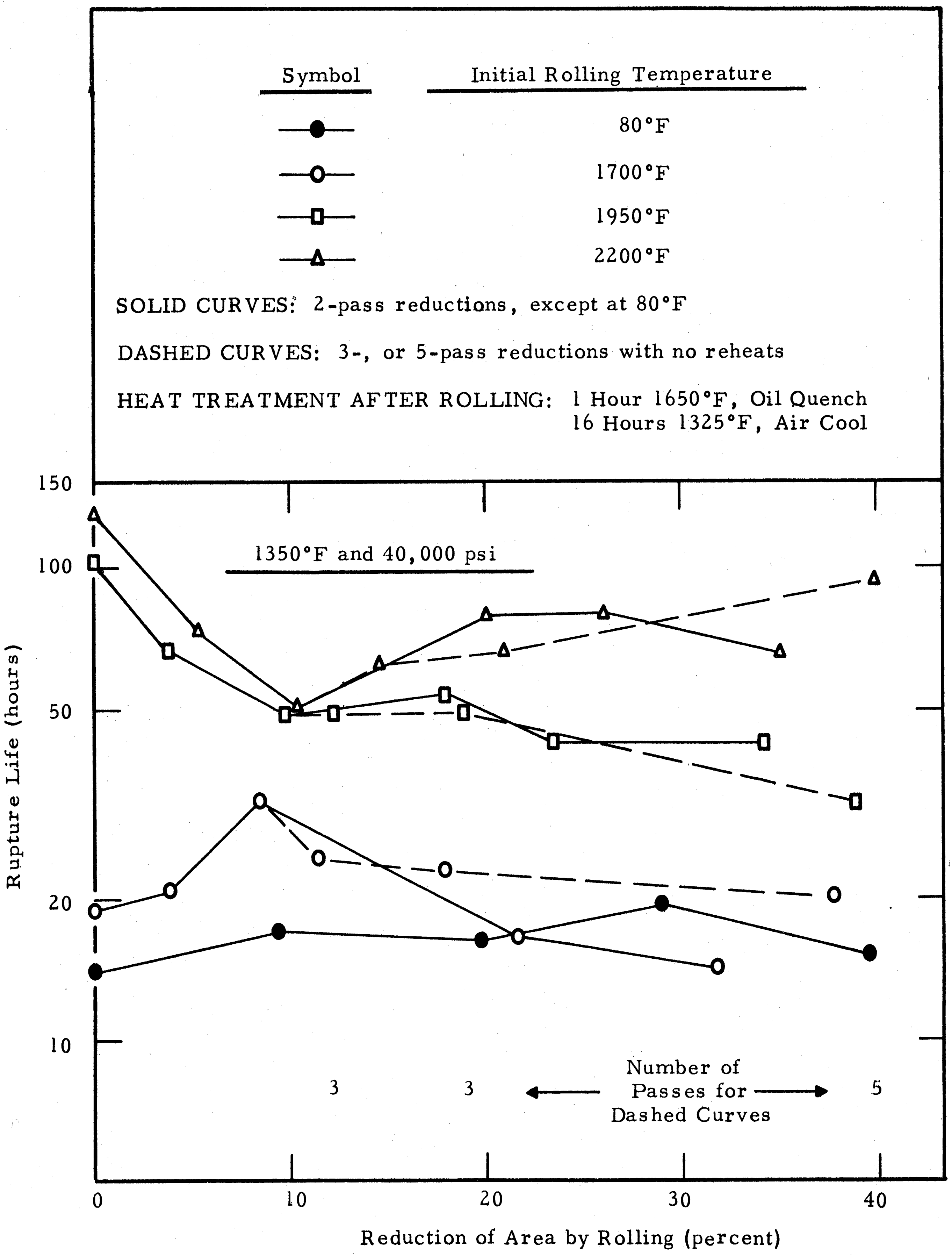
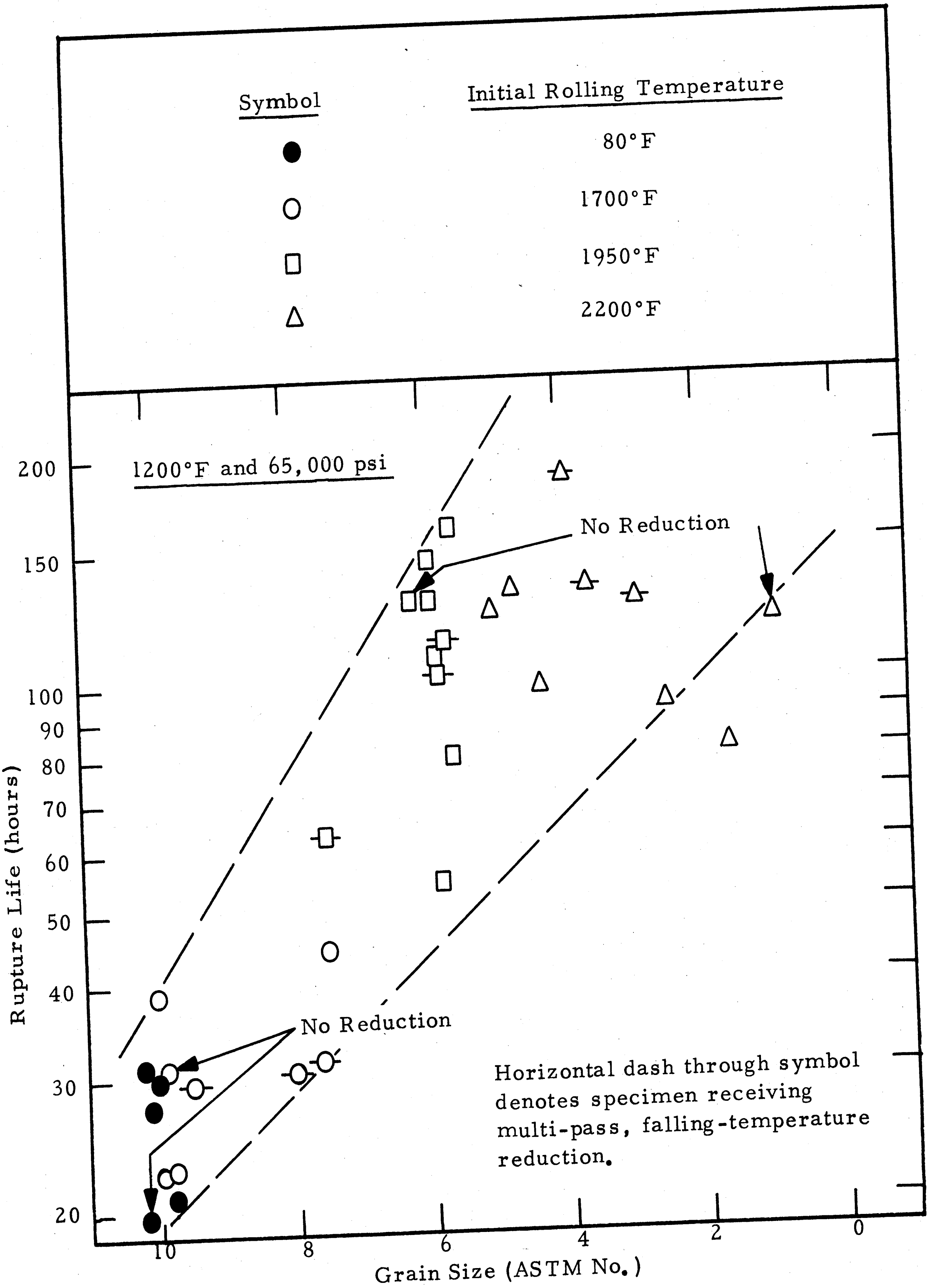


Figure 18. Variation of Log Rupture Life with Prior Rolling Conditions for Heat Treated A-286 Alloy at 1350°F and 40,000 psi.



Horizontal dash through symbol denotes specimen receiving multi-pass, falling-temperature reduction.

Figure 19. Log Rupture Life Versus Grain Size for A-286 Rolled at 80°, 1700°, 1950°, and 2200°F, Heat Treated, and Tested at 1200°F and 65,000 psi. Heat Treatment after rolling was 1 hour at 1650°F, oil quench, plus 16 hours at 1325°F, air cool.

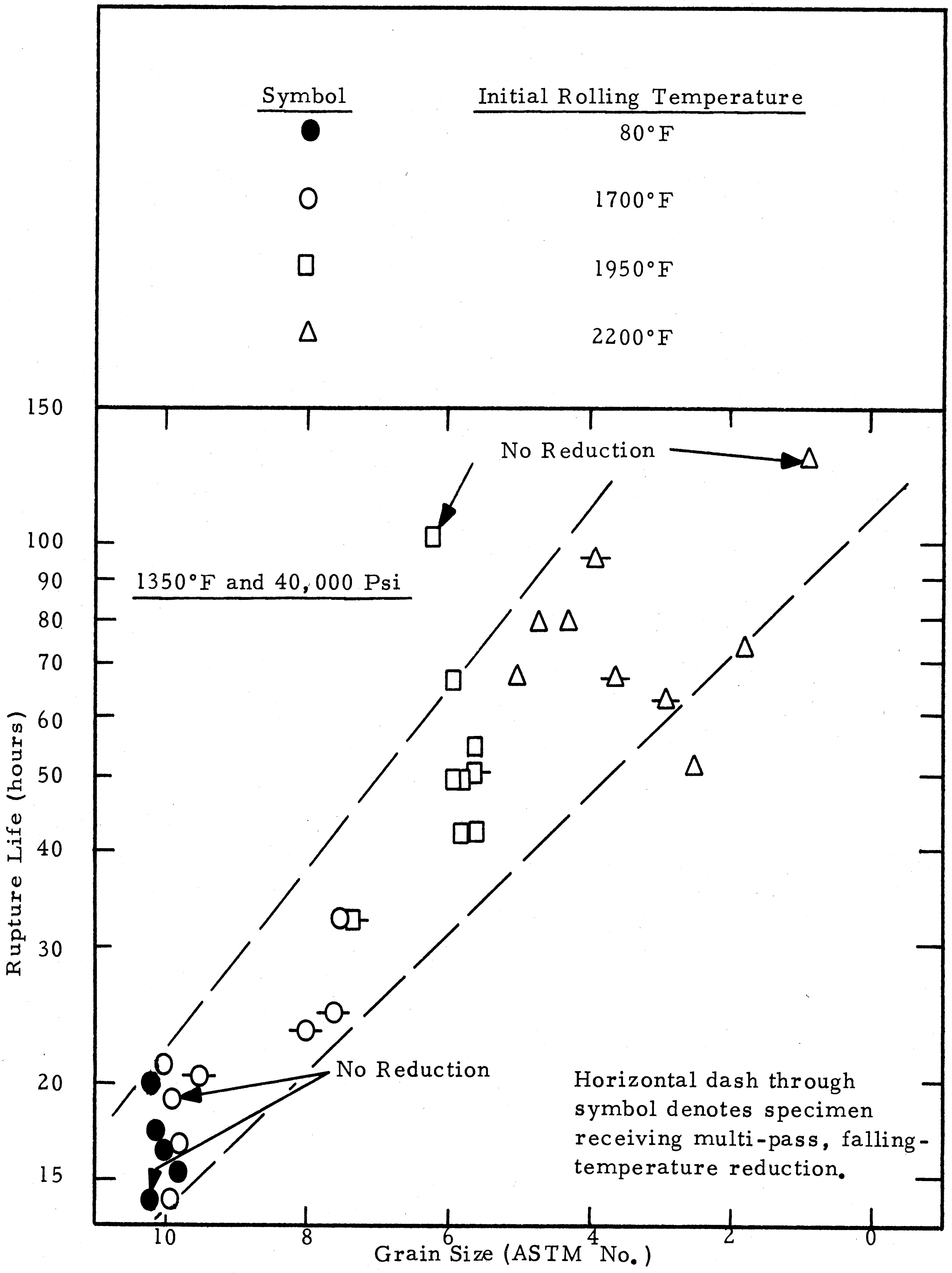
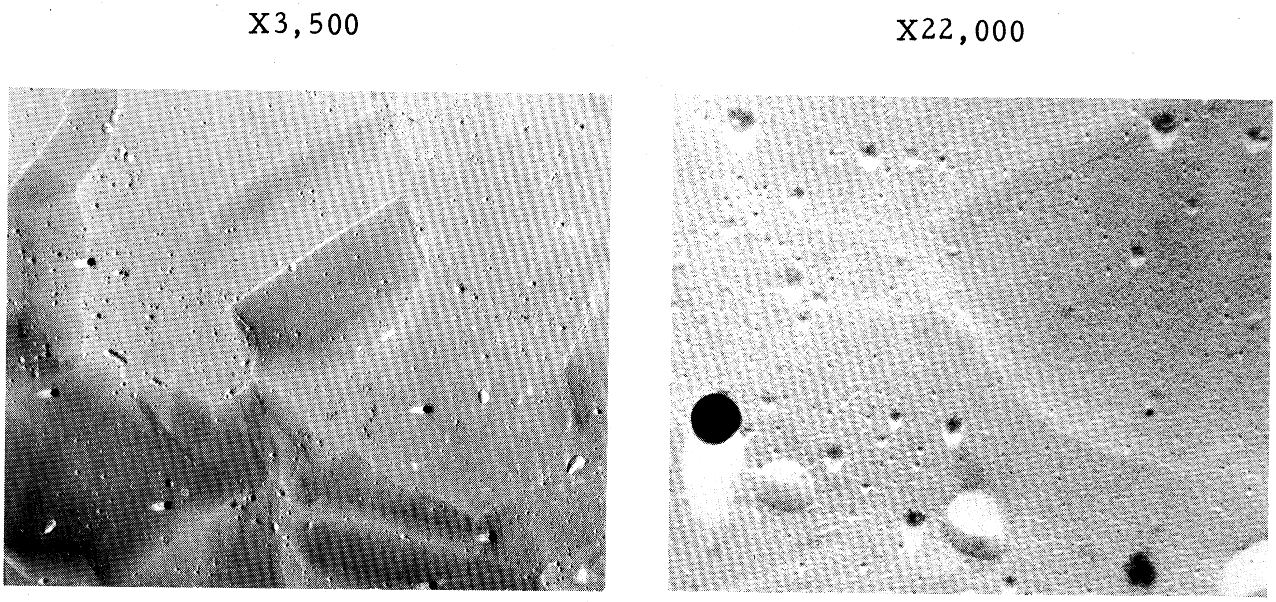
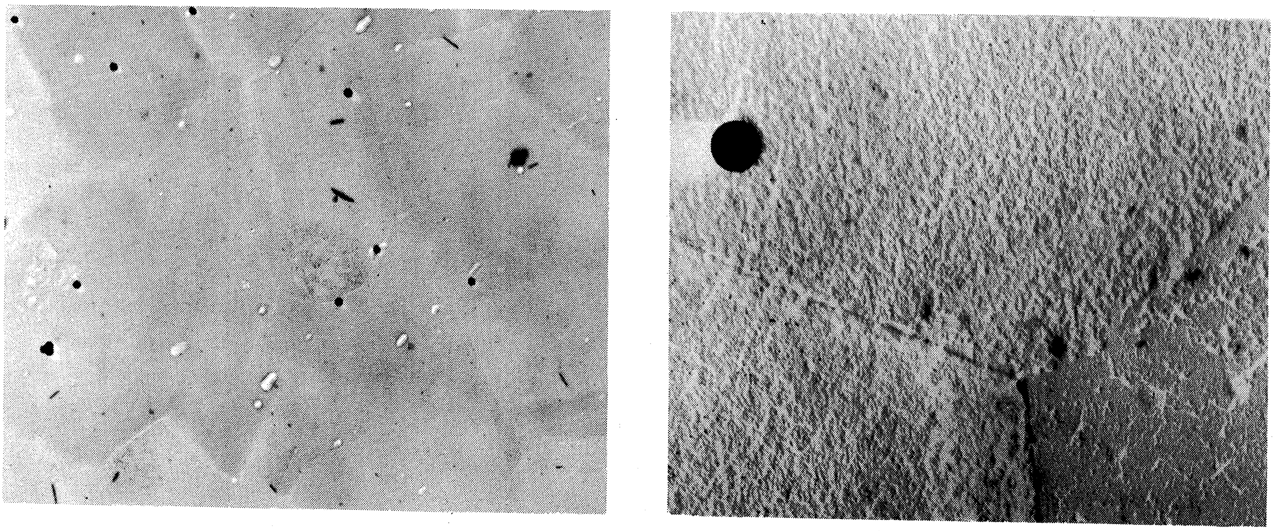


Figure 20. Log Rupture Life Versus Grain Size for A-286 Alloy Rolled at 80°, 1700°, 1950° and 2200°F, Heat Treated, and Tested at 1350°F and 40,000 psi. Heat treatment after rolling was 1 hour at 1650°F, oil quench, plus 16 hours at 1325°F, air cool.



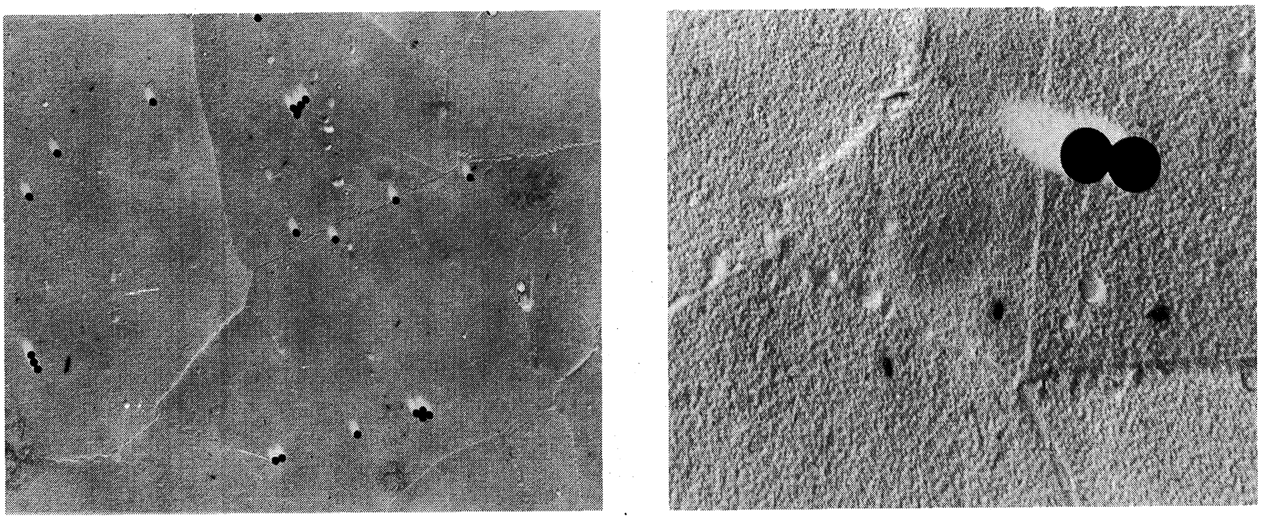
(a) No reduction (base condition).



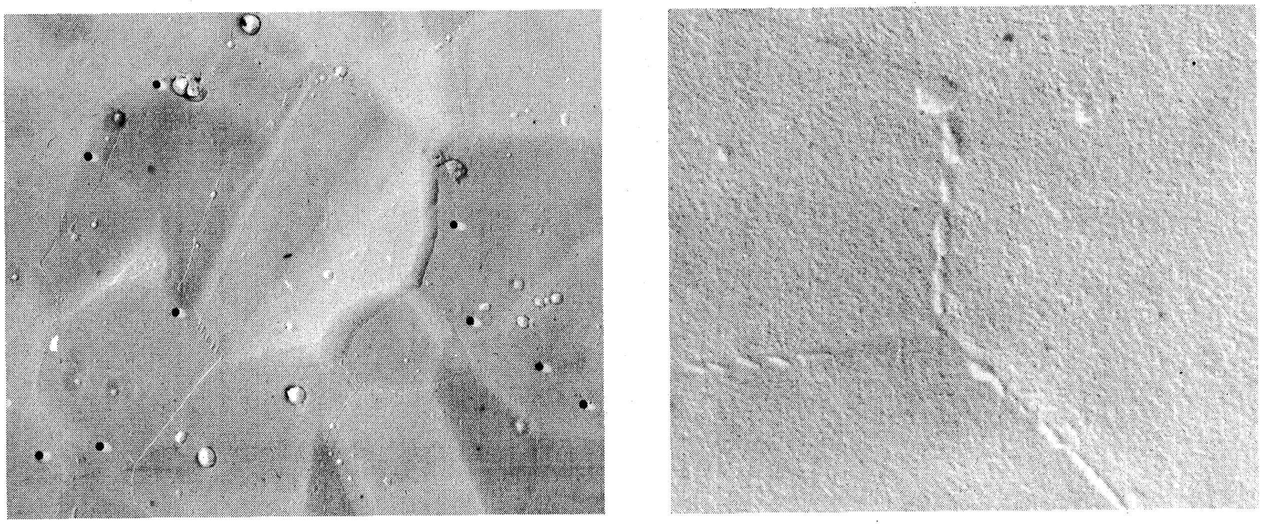
(b) 9.4-percent reduction of area.

Figure 21. Electron Micrographs of A-286 Alloy Rolled at Room Temperature + Solution Treated 1 Hour at 1650°F, Oil Quenched, and Aged 16 Hours at 1325°F.

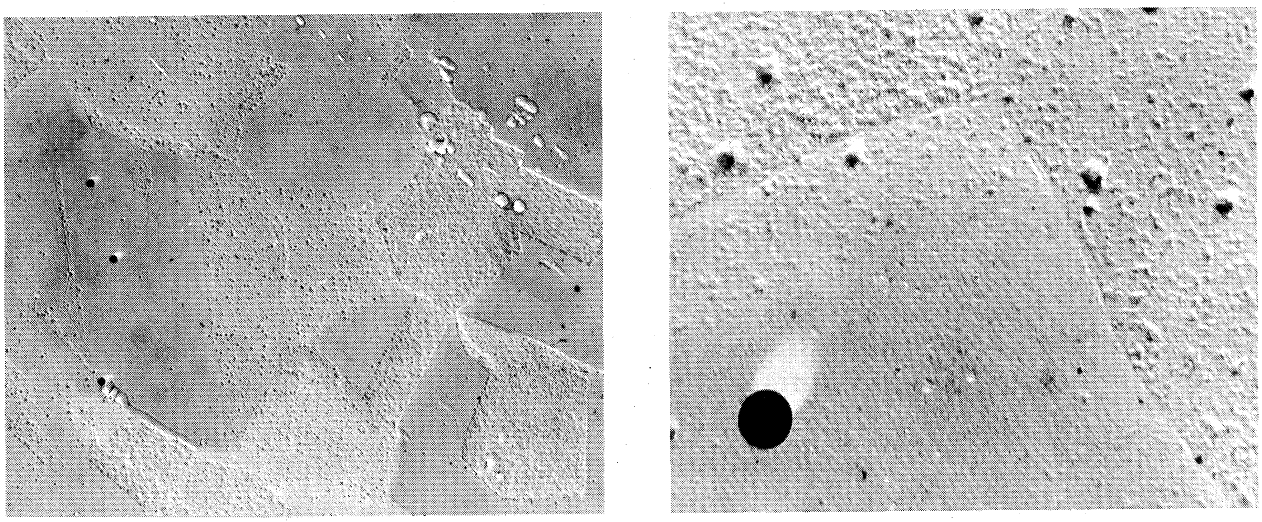
X3,500 X22,000



(c) 19.7-percent reduction of area.

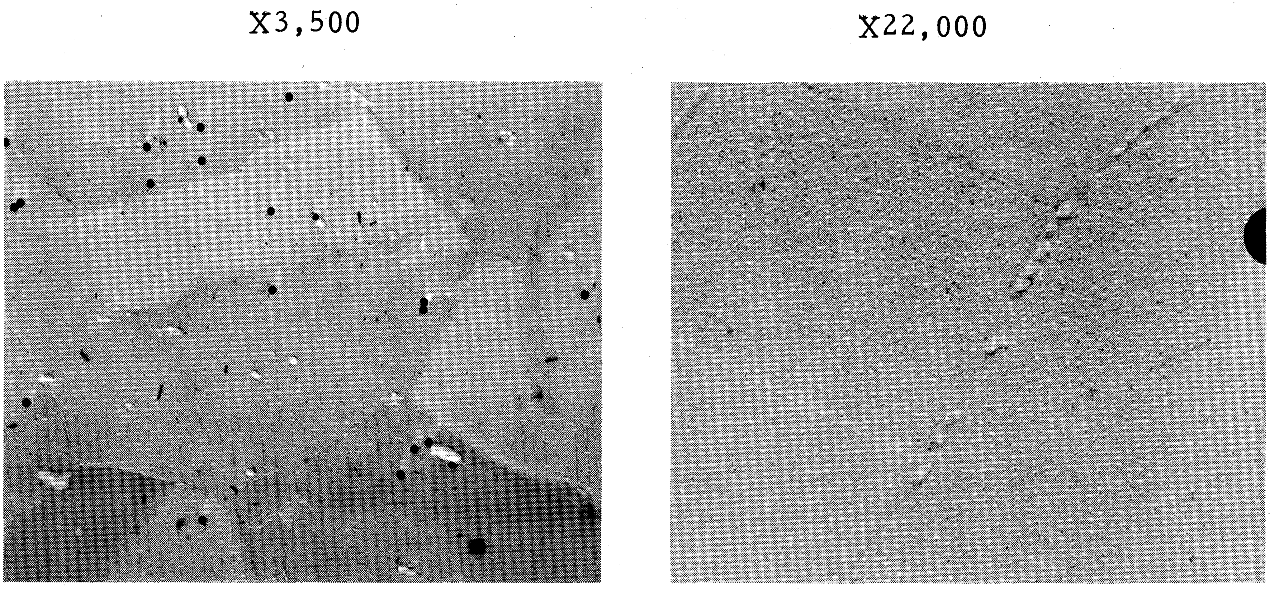


(d) 28.8-percent reduction of area.

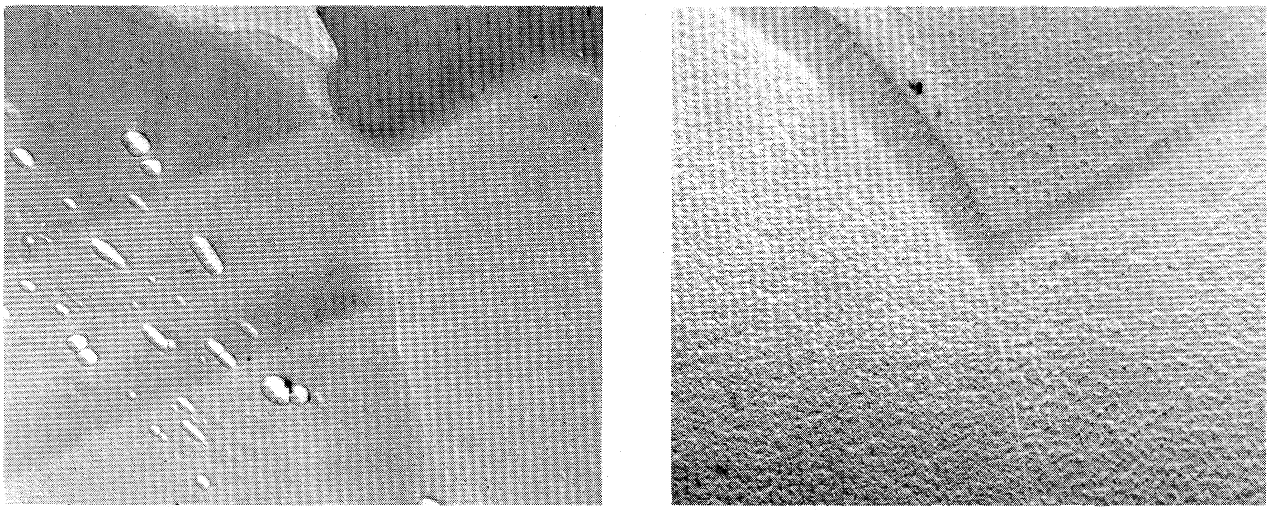


(e) 39.5-percent reduction of area.

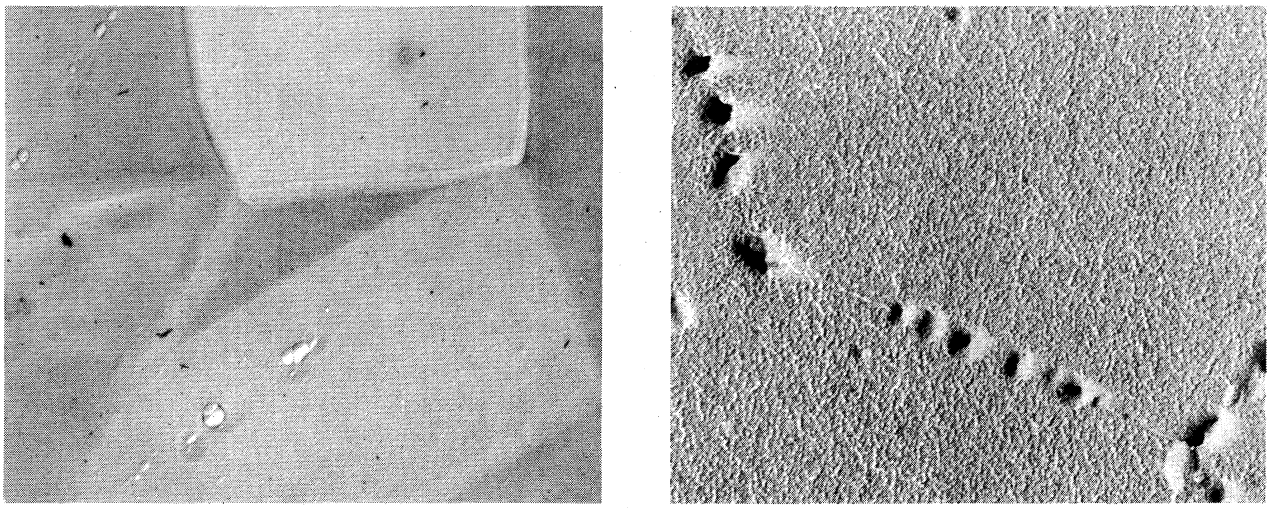
Figure 21. Concluded.



(a) No reduction.

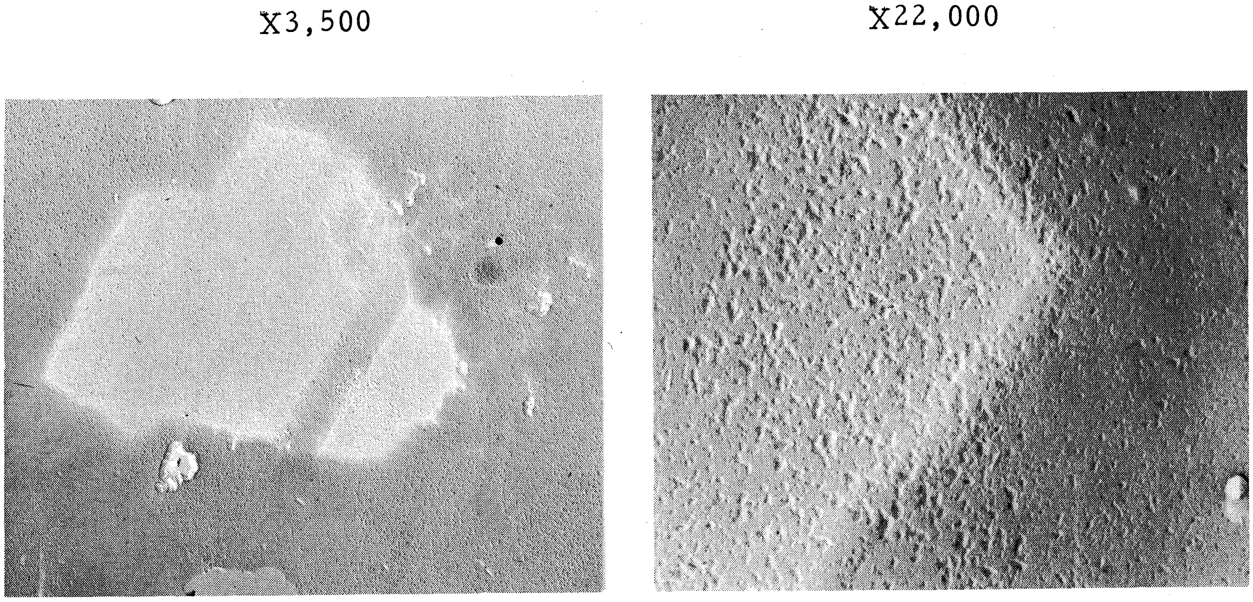


(b) 8.4-percent reduction in 2 passes.

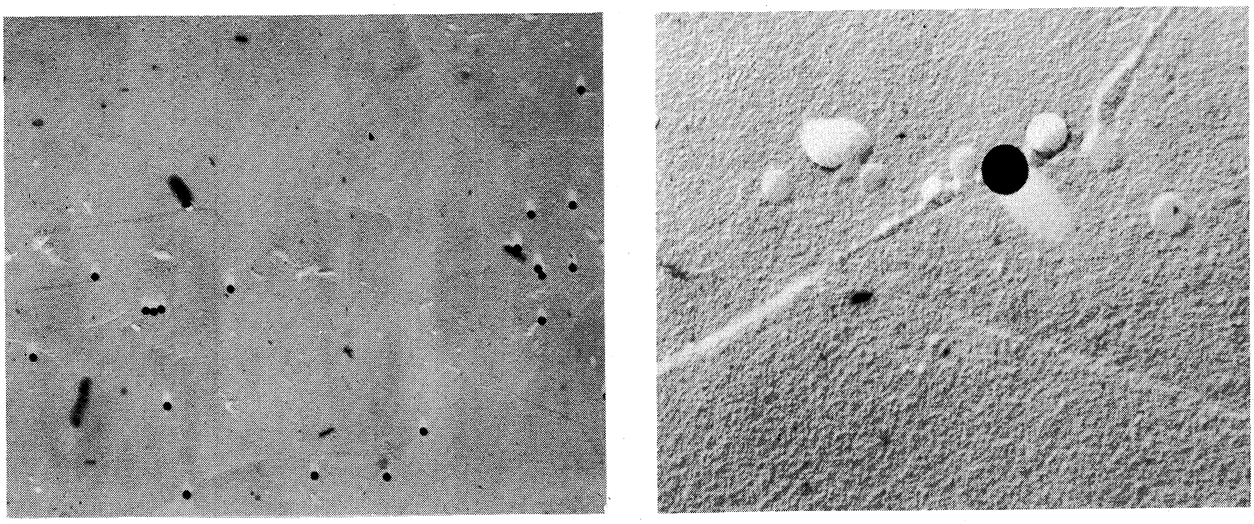


(c) 21.5-percent reduction in 2 passes.

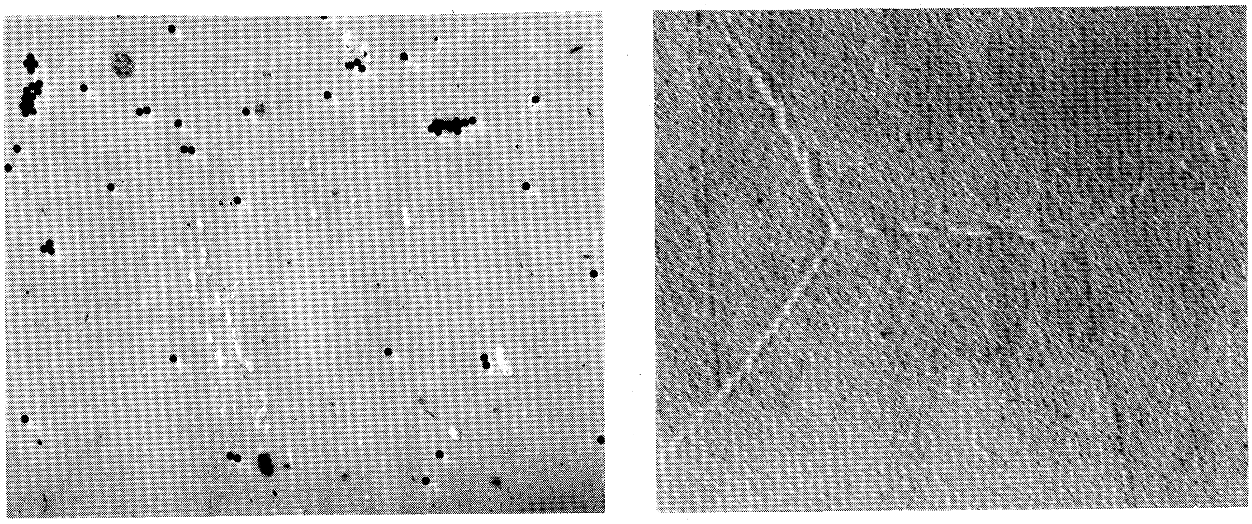
Figure 22. Electron Micrographs of A-286 Alloy Rolled at 1700°F + Solution Treated 1 Hour at 1650°F, Oil Quenched, and Aged 16 Hours at 1325°F.



(d) 31.7-percent reduction in 2 passes.



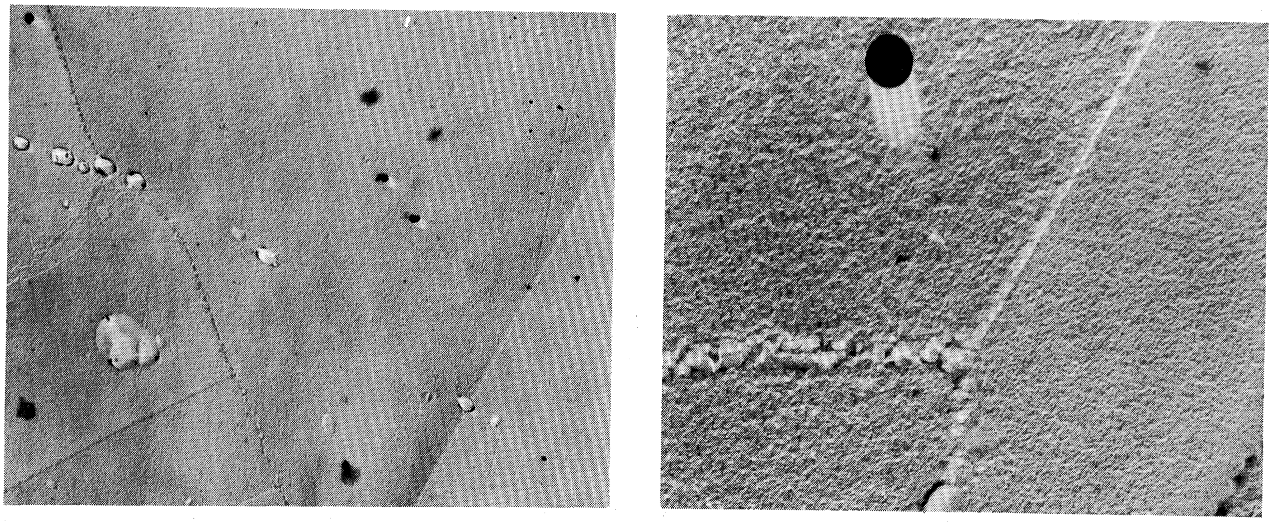
(e) 17.7-percent reduction in 3 passes.



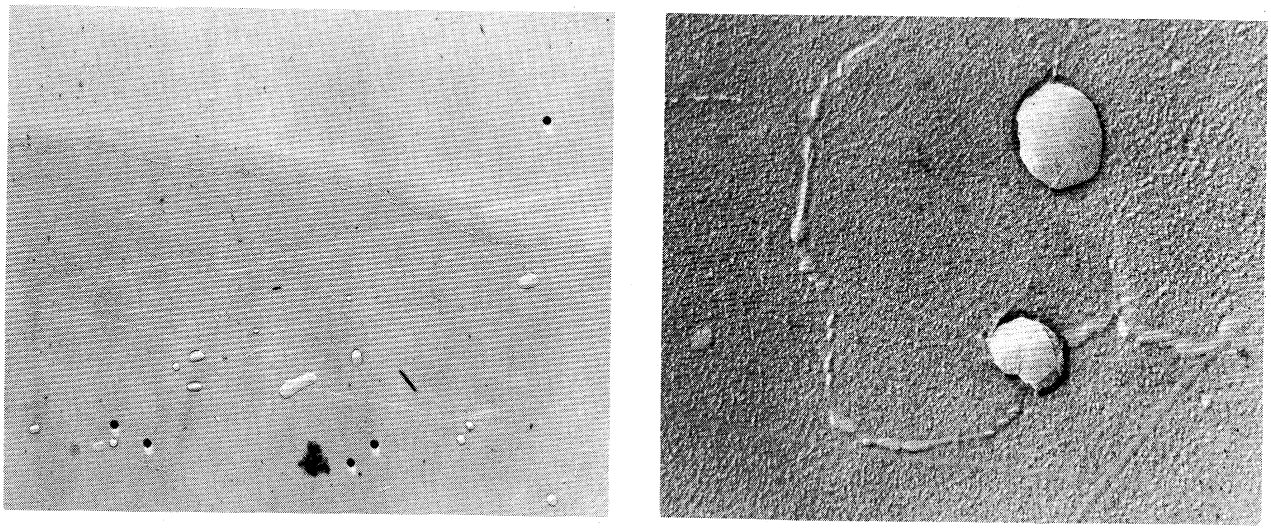
(f) 37.6-percent reduction in 5 passes.

Figure 22. Concluded.

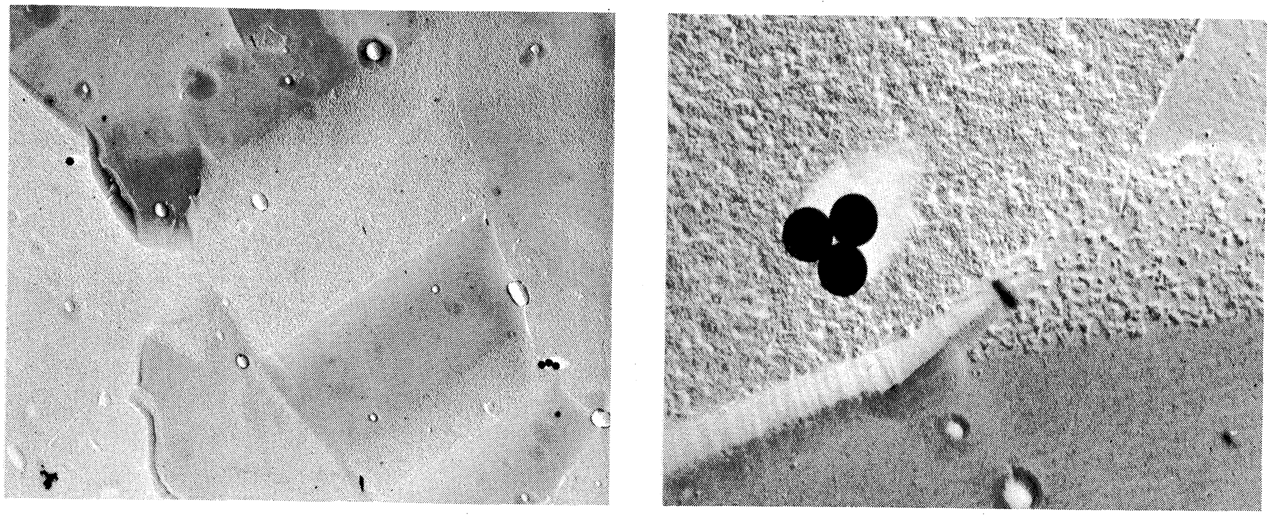
X3,500 X22,000



(a) No reduction.

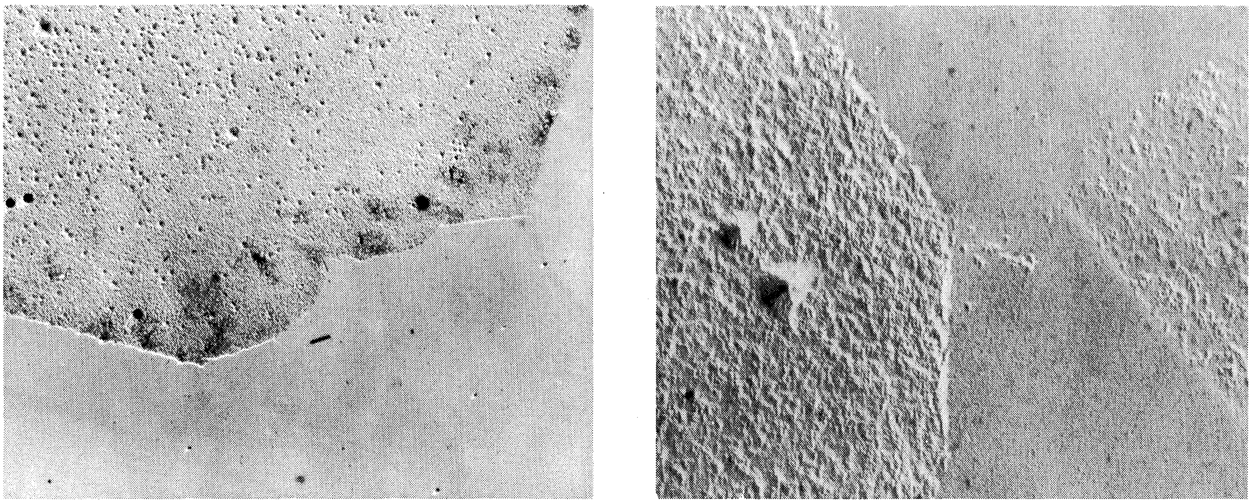


(b) 12.2-percent reduction in 2 passes.

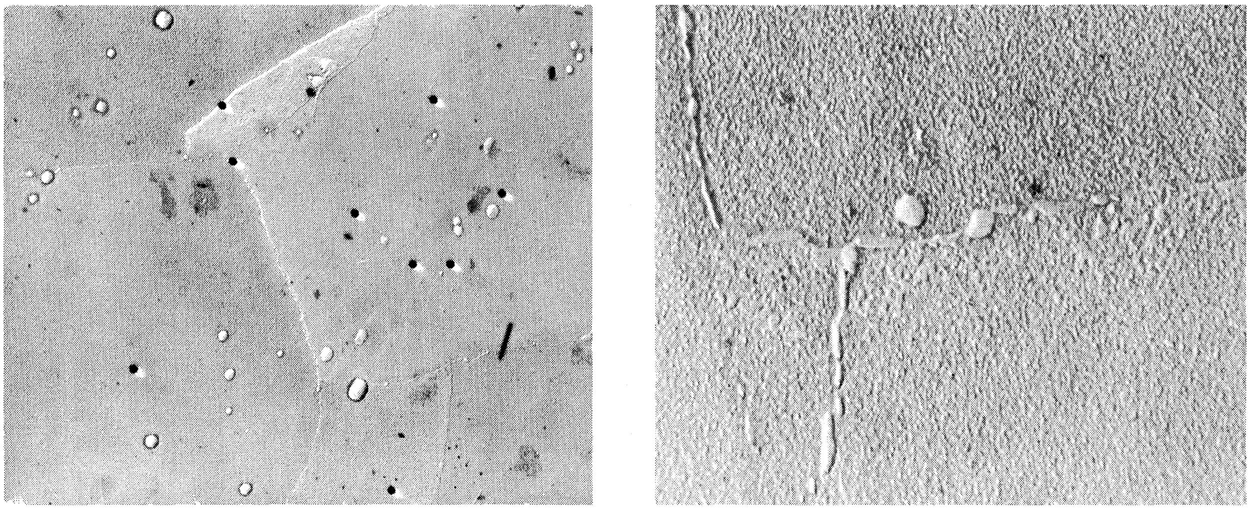


(c) 23.5-percent reduction in 2 passes.

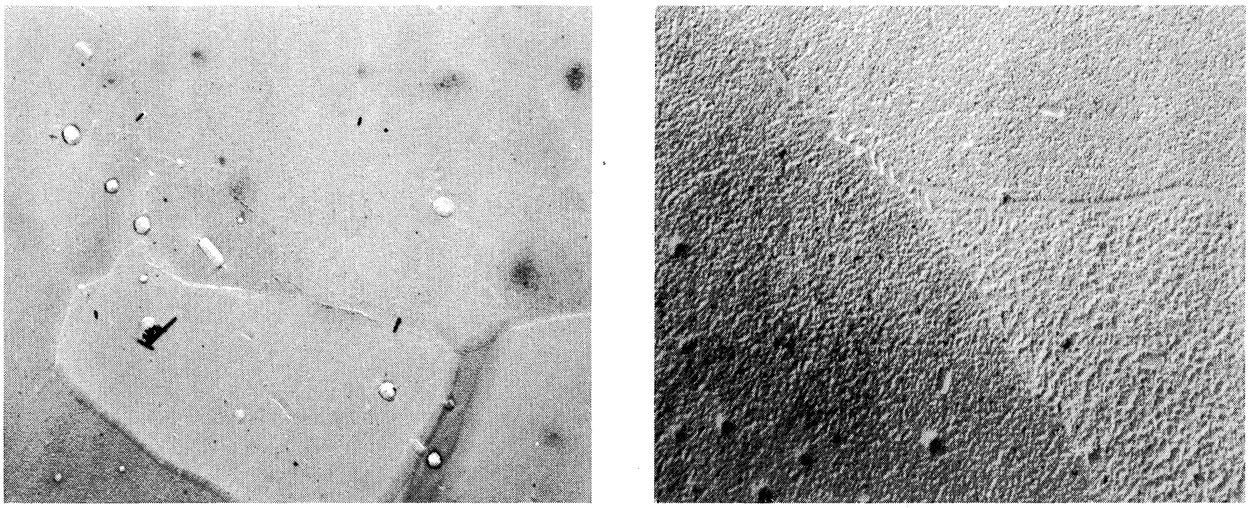
Figure 23. Electron Micrographs of A-286 Alloy Rolled at 1950°F + Solution Treated 1 Hour at 1650°F, Oil Quenched, and Aged 16 Hours at 1325°F.



(d) 34.2-percent reduction in 2 passes.



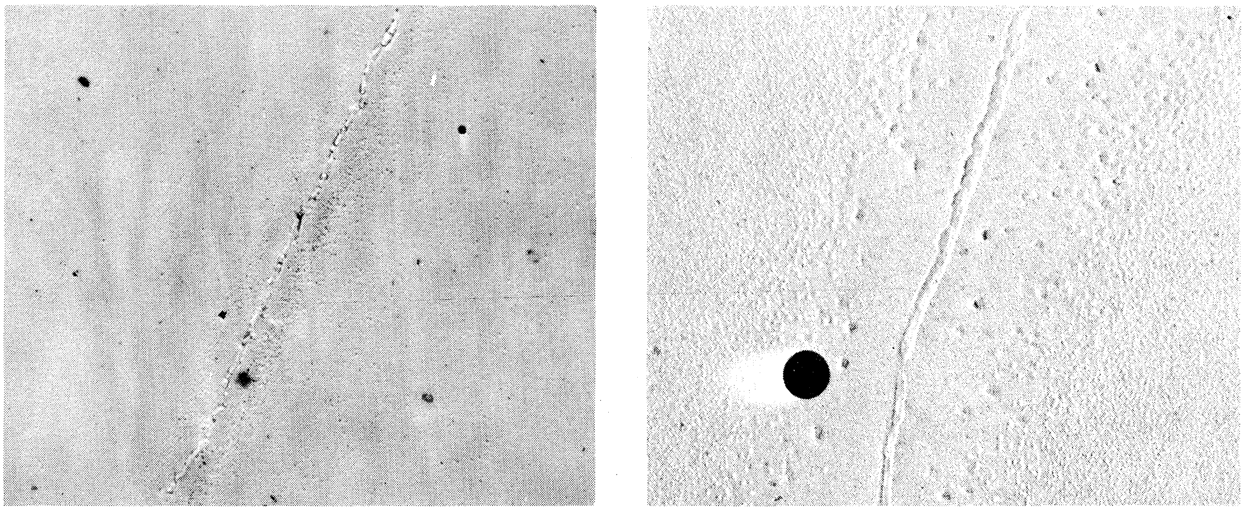
(e) 18.8-percent reduction in 3 passes.



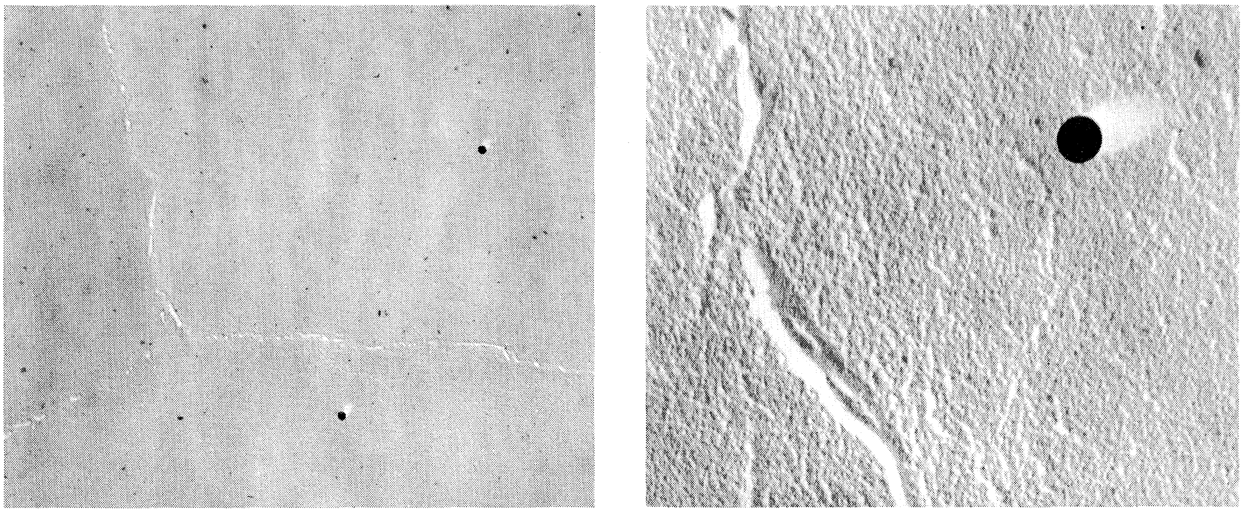
(f) 38.7-percent reduction in 5 passes.

Figure 23. Concluded.

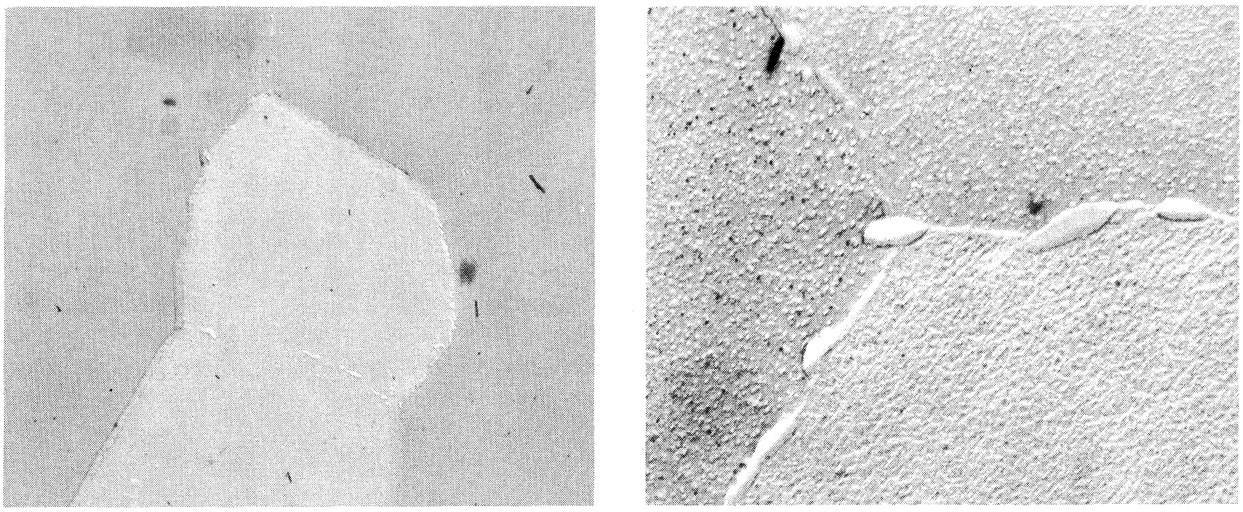
X3,500 X22,000



(a) No reduction.

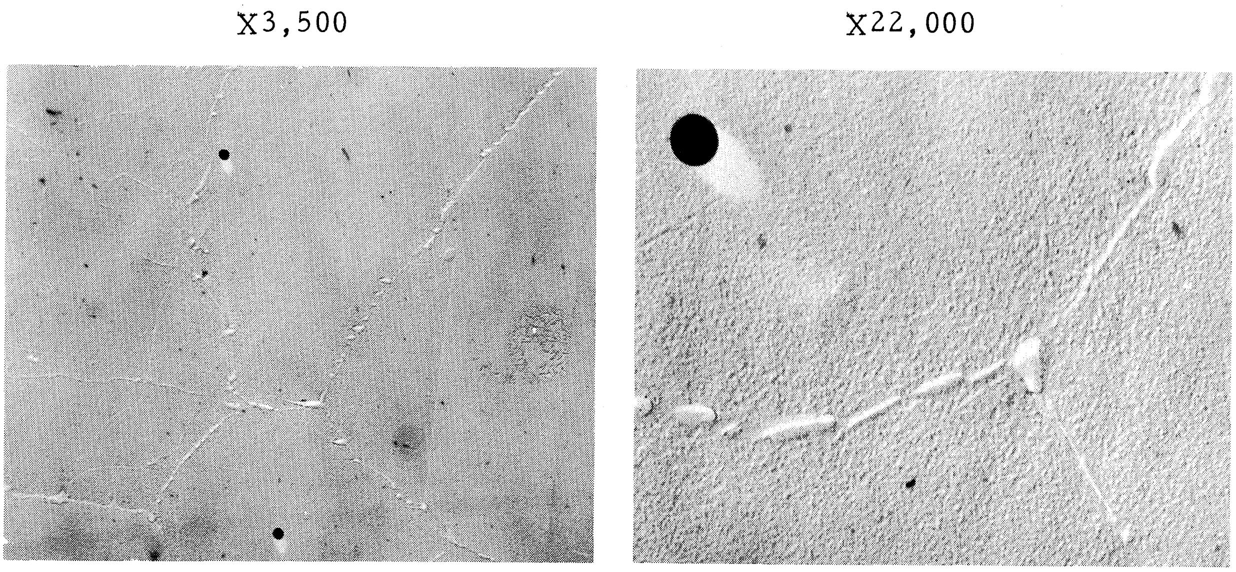


(b) 10.4-percent reduction in 2 passes.

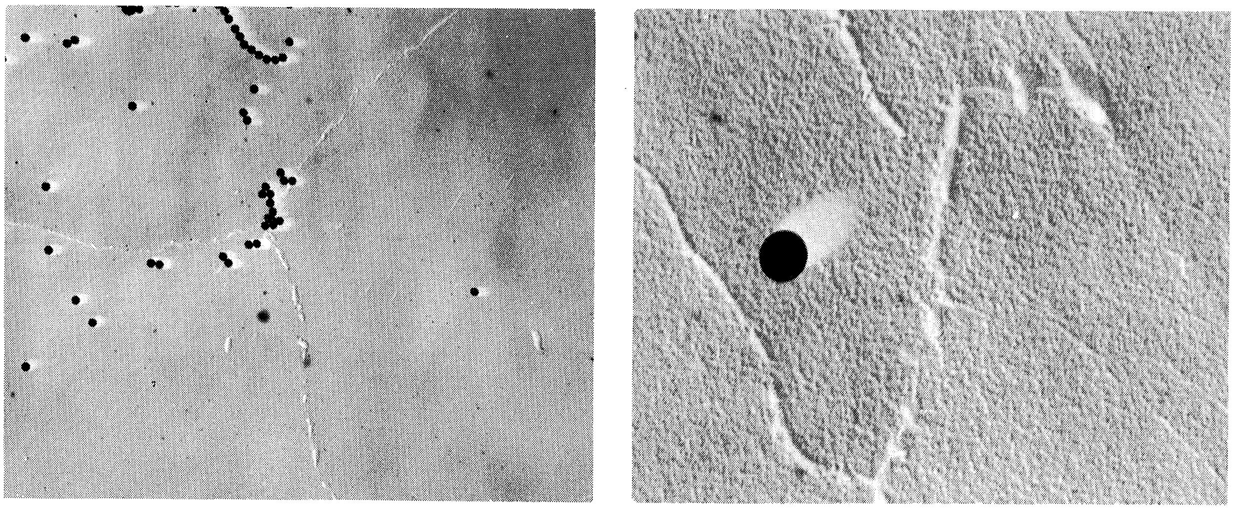


(c) 26.1-percent reduction in 2 passes.

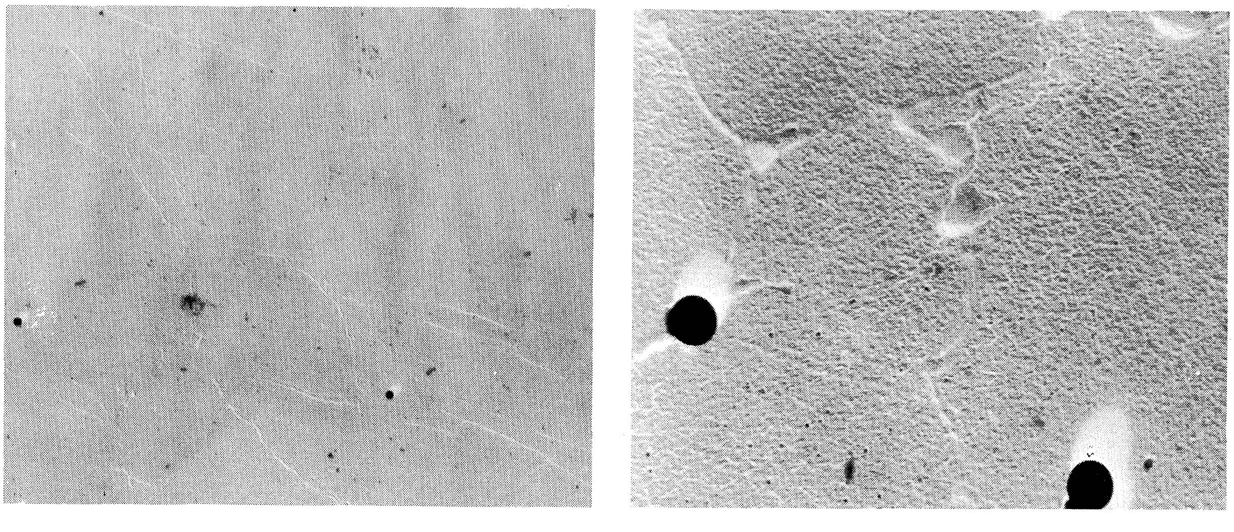
Figure 24. Electron Micrographs of A-286 Alloy Rolled at 2200°F + Solution Treated 1 Hour at 1650°F, Oil Quenched, and Aged 16 Hours at 1325°F.



(d) 35.1-percent reduction in 2 passes.



(e) 20.8-percent reduction in 3 passes.



(f) 39.8-percent reduction in 5 passes.

Figure 24. Concluded.

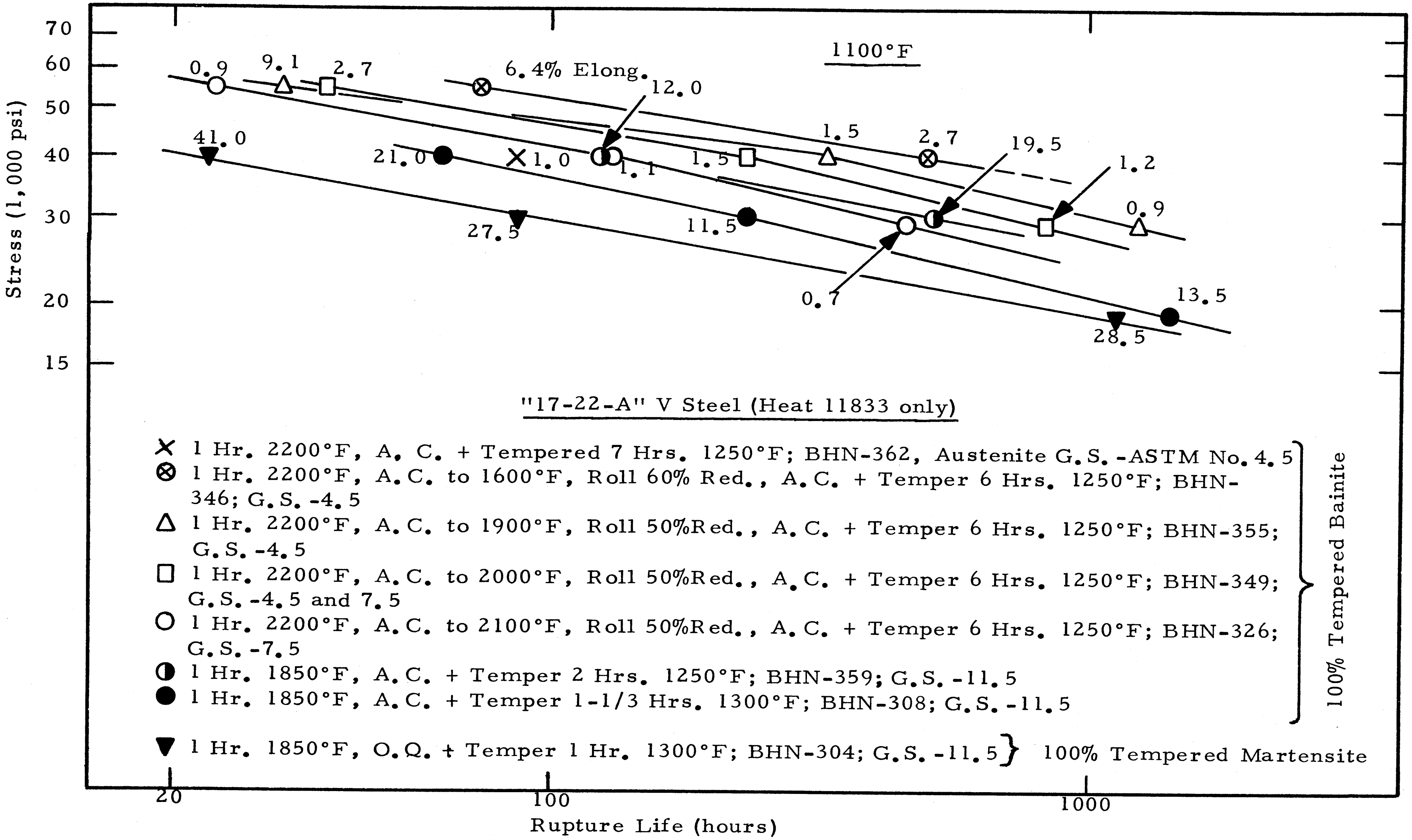
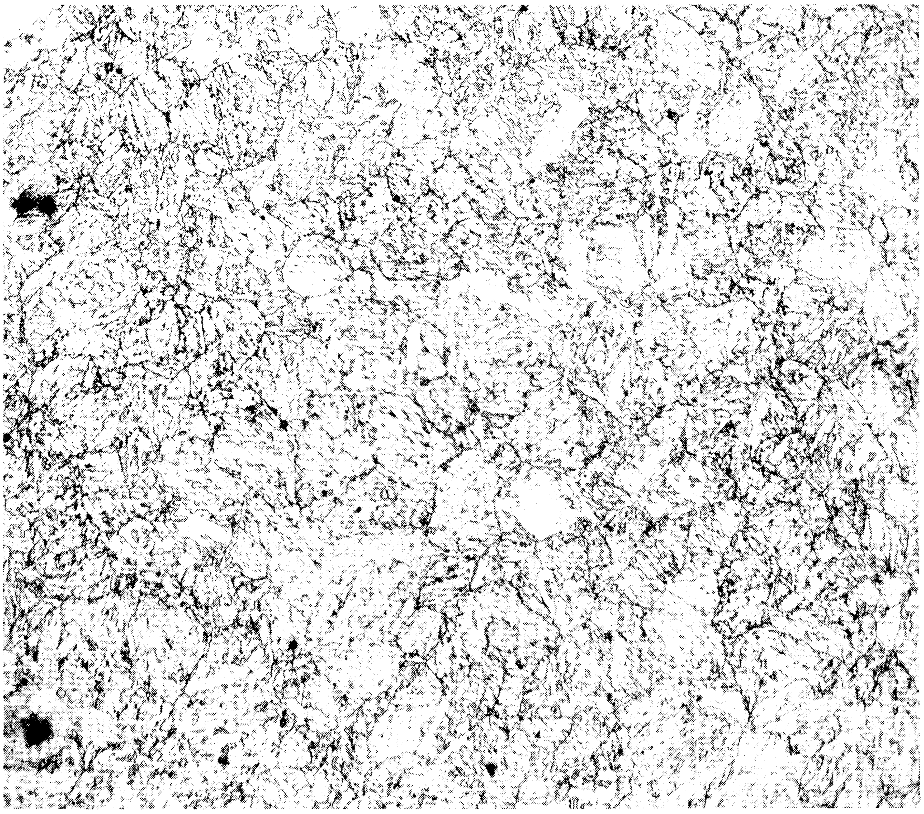
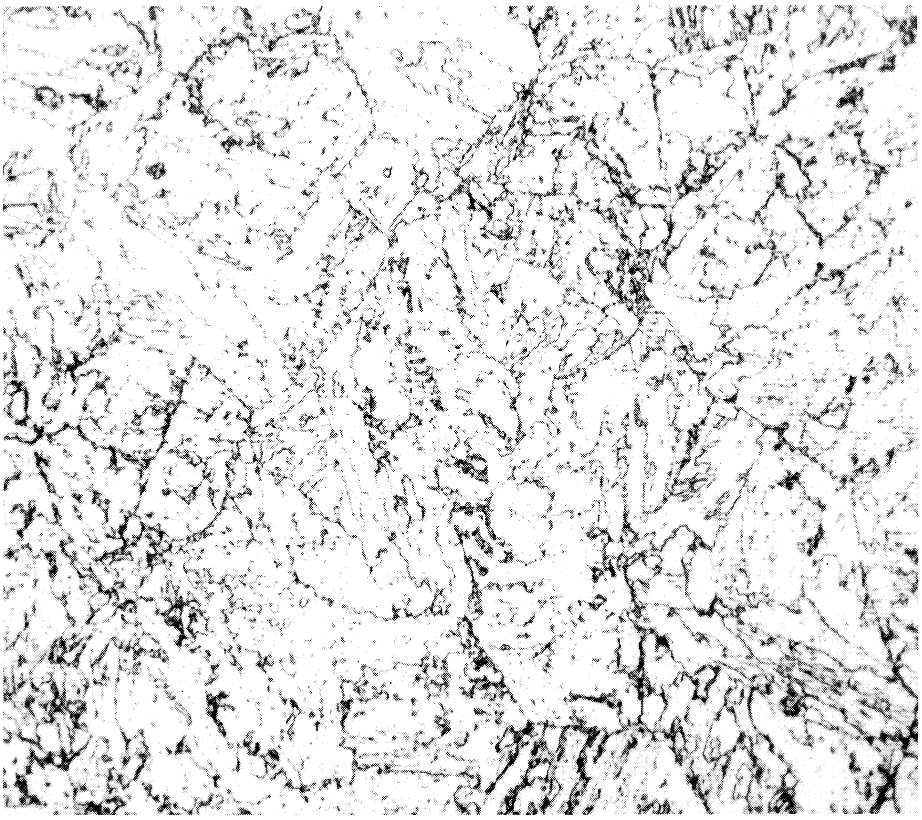


Figure 25. Variation of Stress-Rupture Properties of "17-22-A"V Steel at 1100°F with Processing Conditions.

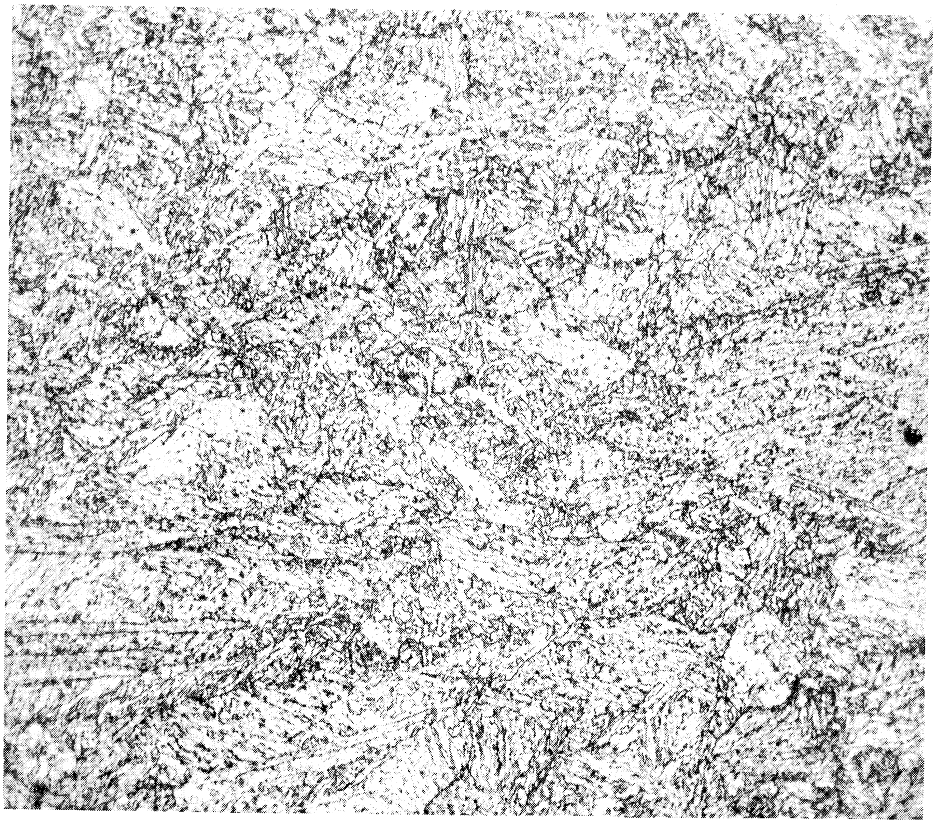


X250D

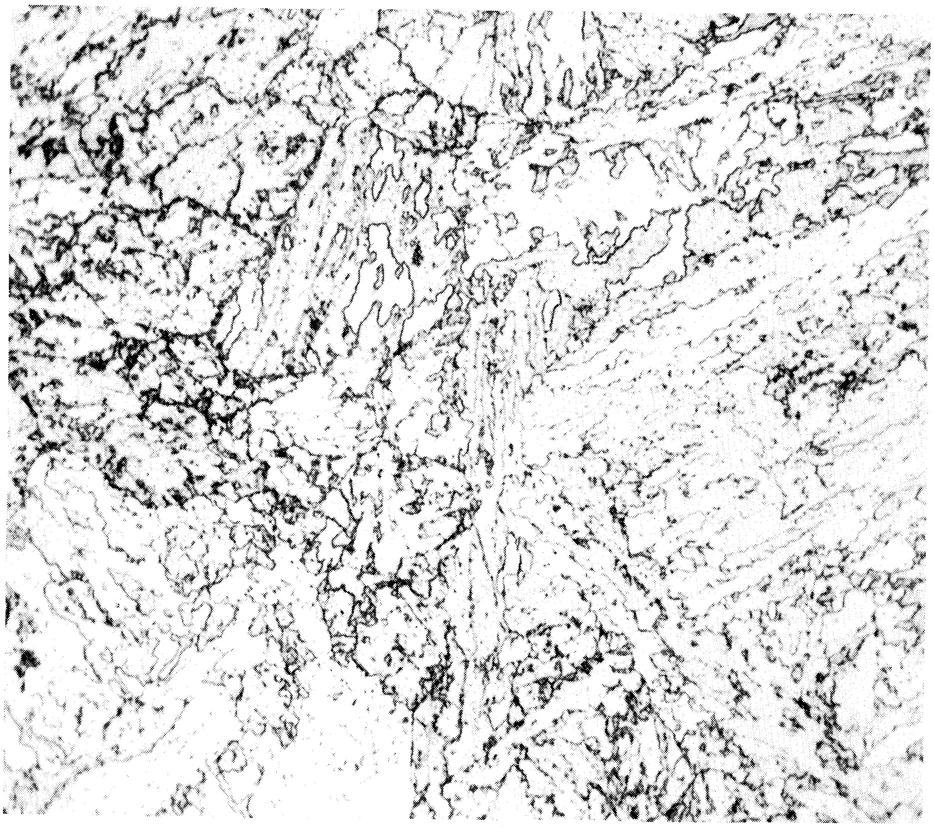


X1000D

Figure 26. "17-22-A" V Steel Heated 1 Hour at 2200°F., Air Cooled to 2100°F and Rolled 50 Percent Reduction in 2 Passes, and Air Cooled to Room Temperature + Tempered 6 Hours at 1250°F. Complete Recrystallization During Rolling. Etchant: 2-Percent Nital.

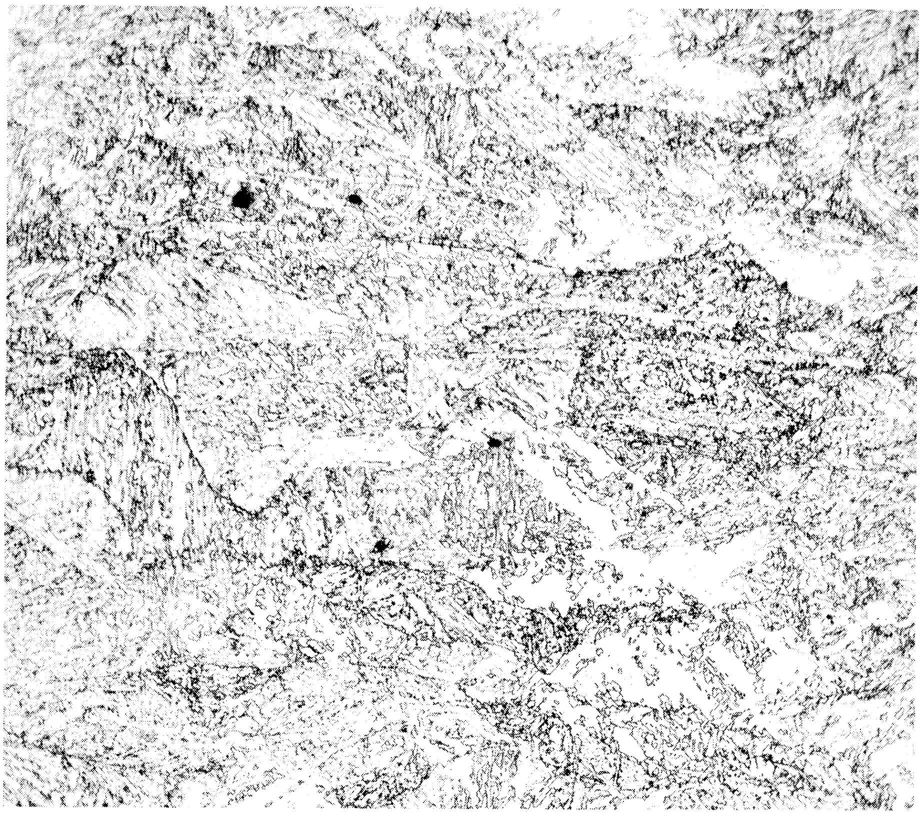


X250D

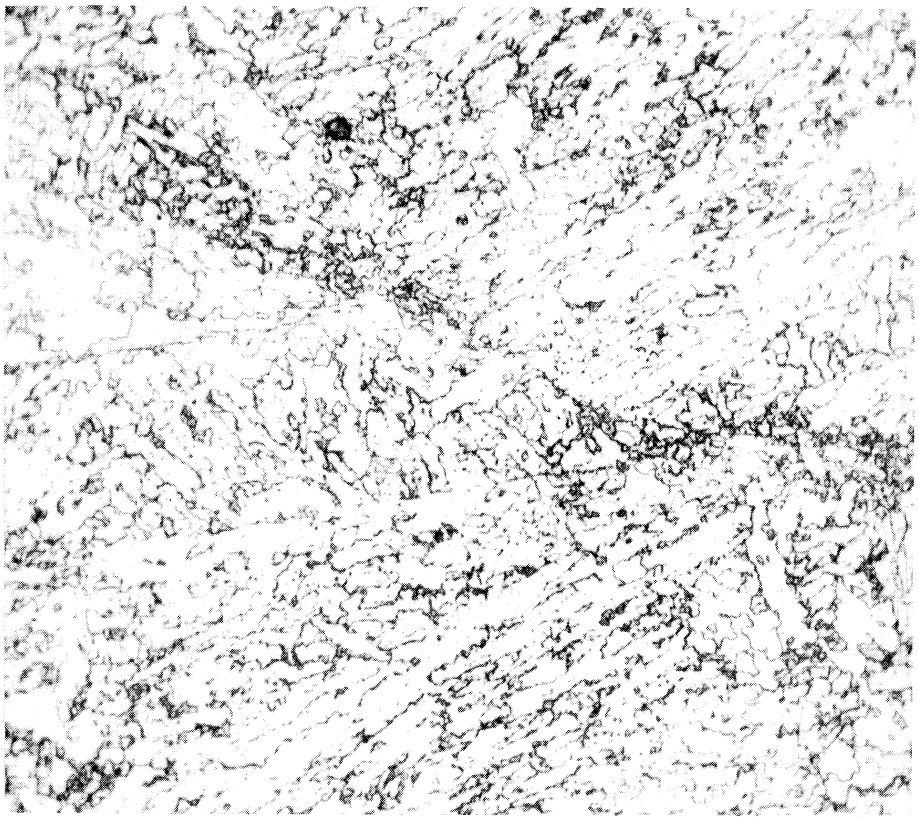


X1000D

igure 27. "17-22-A" V Steel Heated 1 Hour at 2200°F, Air Cooled to 2000°F and Rolled 50 Percent Reduction in 2 Passes, and Air Cooled to Room Temperature + Tempered 6 Hours at 1250°F. Partial Recrystallization During Rolling. Etchant: 2-Percent Nital.

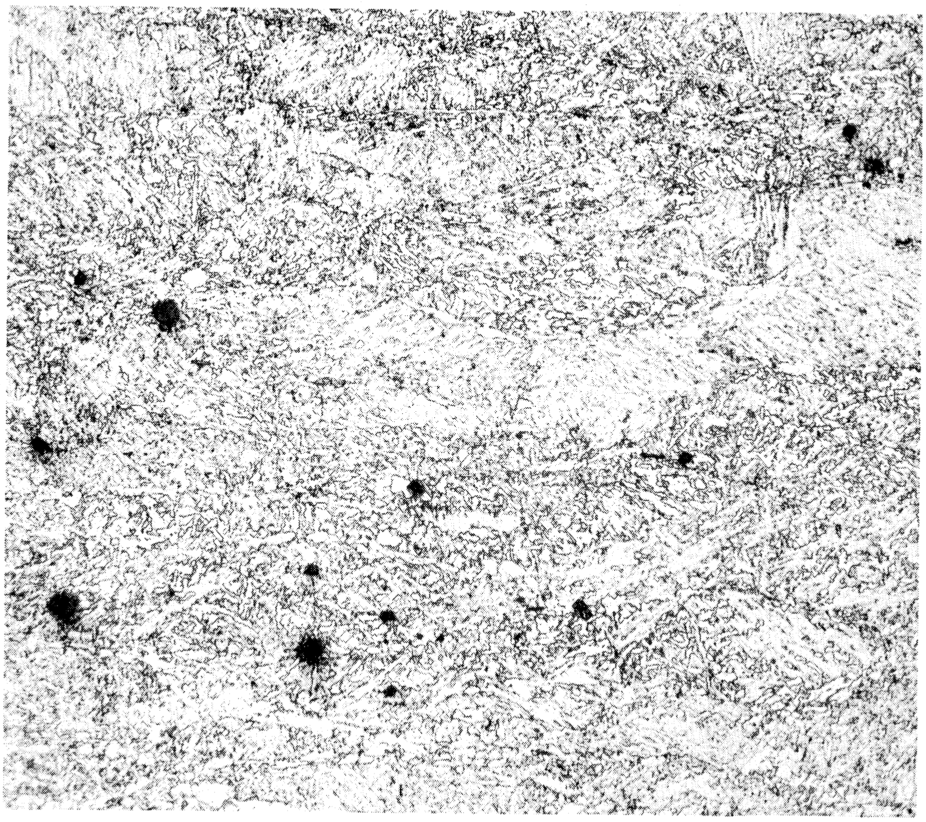


X250D



X1000D

Figure 28. "17-22-A"V Steel Heated 1 Hour at 2200°F, Air Cooled to 1900°F and Rolled 50 Percent Reduction in 2 Passes, and Air Cooled to Room Temperature + Tempered 6 Hours at 1250°F. No Recrystallization. Etchant: 2-Percent Nital.

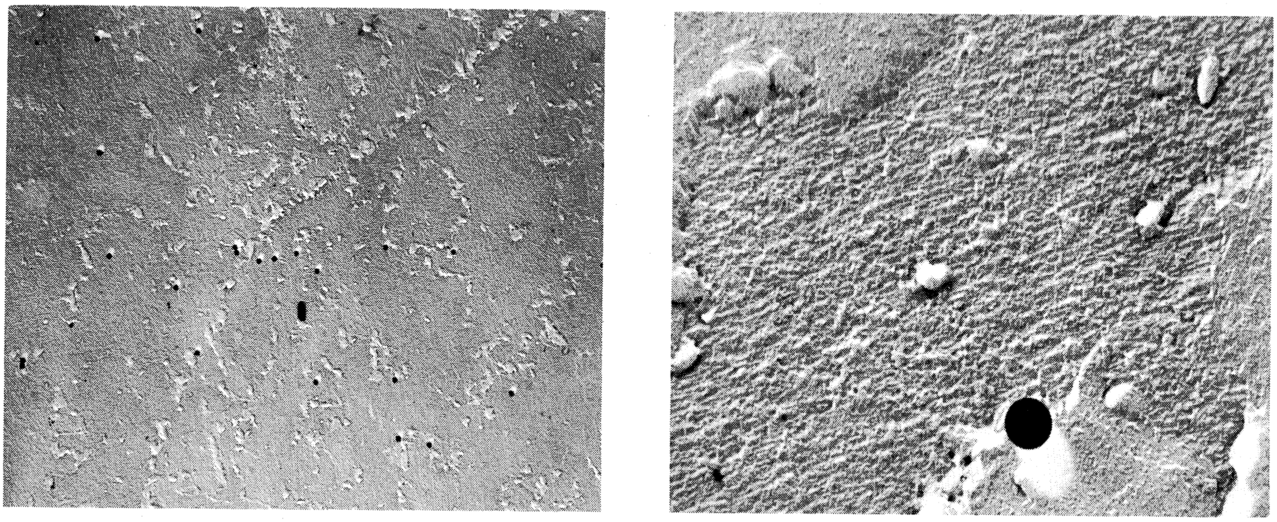


X250D

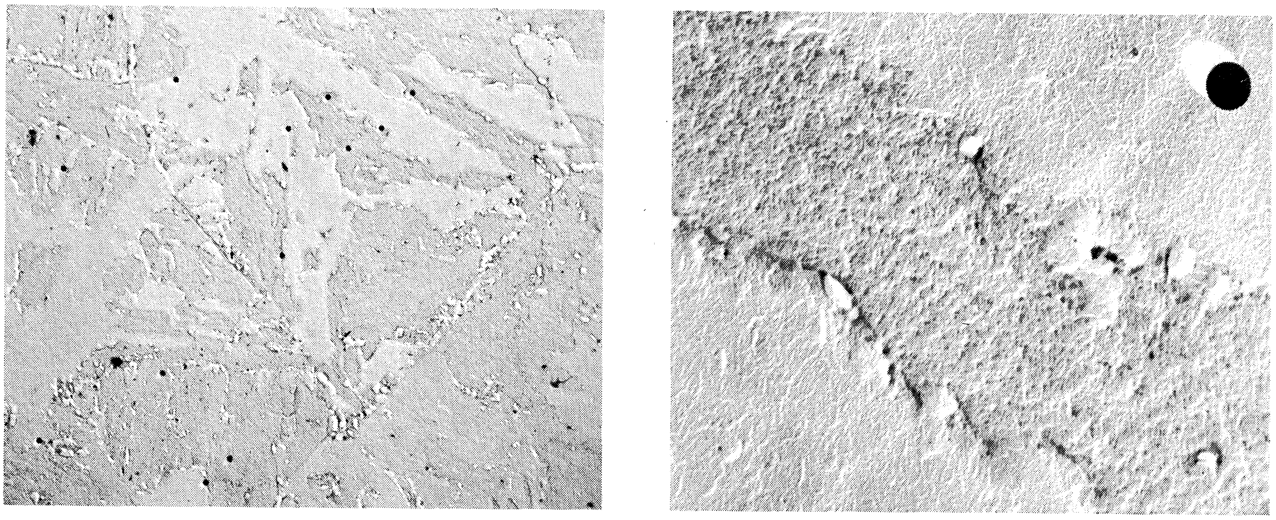
X1000D

igure 29. "17-22-A" V Steel Heated 1 Hour at 2200°F, Air Cooled to 1600°F and Rolled 60 Percent Reduction in 5 Passes, and Air Cooled to Room Temperature + Tempered 6 Hours at 1250°F. Etchant: 2-Percent Nital.

X2,200 X22,000

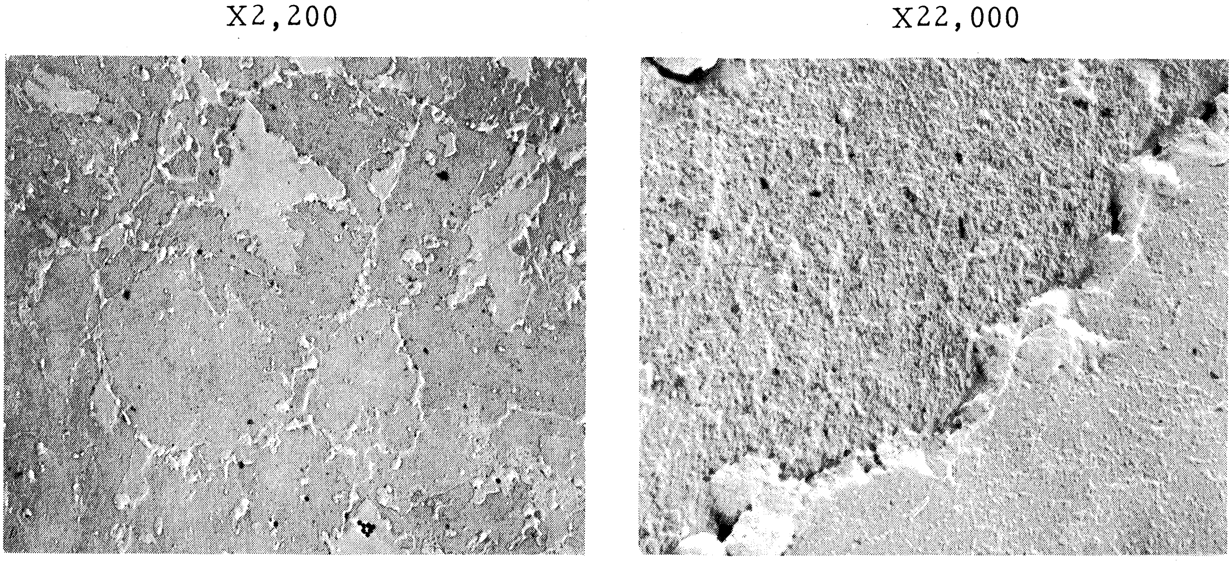


(a) 1 Hr. 2200°F., AC + Tempered 7 Hrs. at 1250°F; BHN-362; Austenite Grain Size - ASTM No. 4.5.

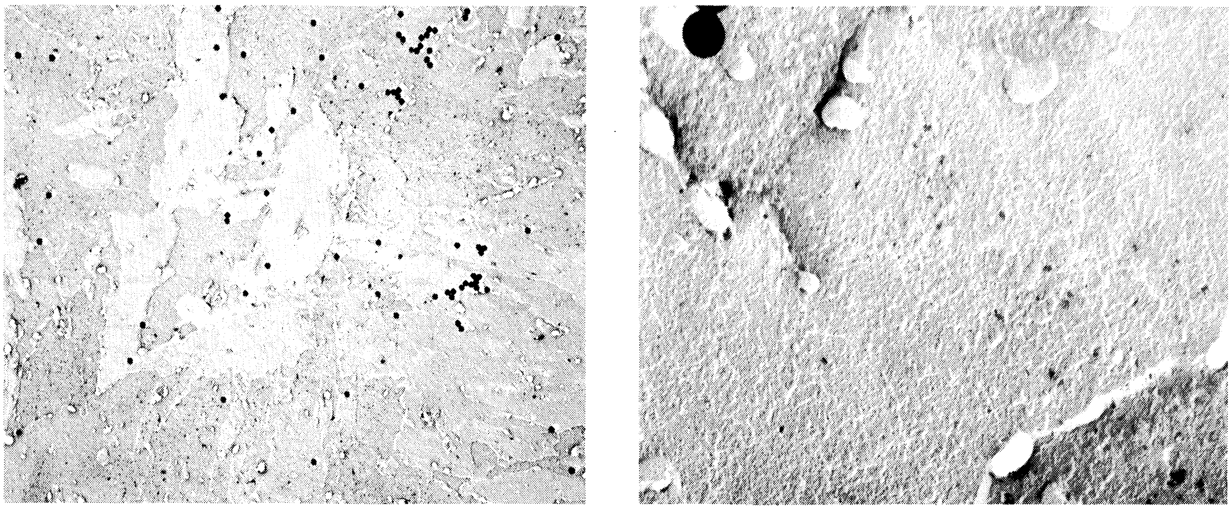


(b) 1 Hr. 2200°F, AC to 2100°F, Rolled 50% Red. in 2 Passes, AC + Tempered 6 Hrs. at 1250°F; BHN-326; G.S. -7.5.

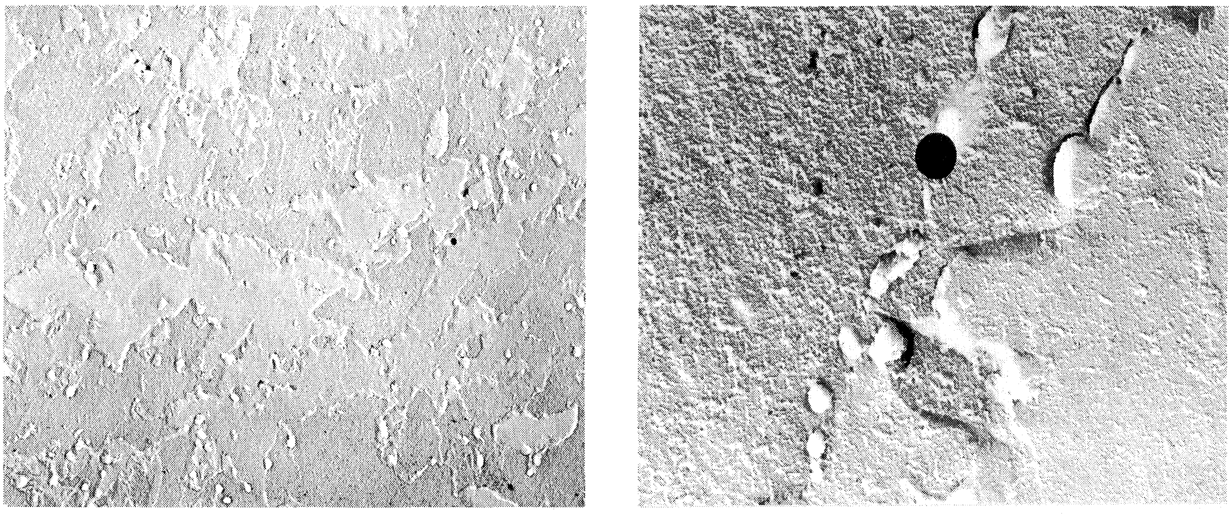
Figure 30. Electron Micrographs of "17-22-A"V Steel As Heated One Hour at 2200°F and (a) Air Cooled + Tempered, (b) Air Cooled to 2100°F, Rolled 50%, and Air Cooled + Tempered, (c) Air Cooled to 2000°F, Rolled 50%, and Air Cooled + Tempered, (d) Air Cooled to 1900°F, Rolled 50%, and Air Cooled + Tempered, and (e) Air Cooled to 1600°F, Rolled 60%, and Air Cooled + Tempered. Etched with 5% Nital. Collodion replicas were shadowed with palladium after 0.34 micron diameter polystyrene balls were placed on replicas.



(c) 1 Hr. 2200°F, AC to 2000°F, Rolled 50% Red. in 2 Passes, AC + Tempered 6 Hrs. at 1250°F; BHN-349; G.S.-Mixed 4.5 and 7.5.



(d) 1 Hr. 2200°F, AC to 1900°F, Rolled 50% Red. in 2 Passes, AC + Tempered 6 Hrs. at 1250°F; BHN-355; G.S.-4.5.



(e) 1 Hr. 2200°F, AC to 1600°F, Rolled 60% Red. in 5 Passes, AC + Tempered 6 Hrs. at 1250°F; BHN-346; G.S.-4.5.

Figure 30 Concluded

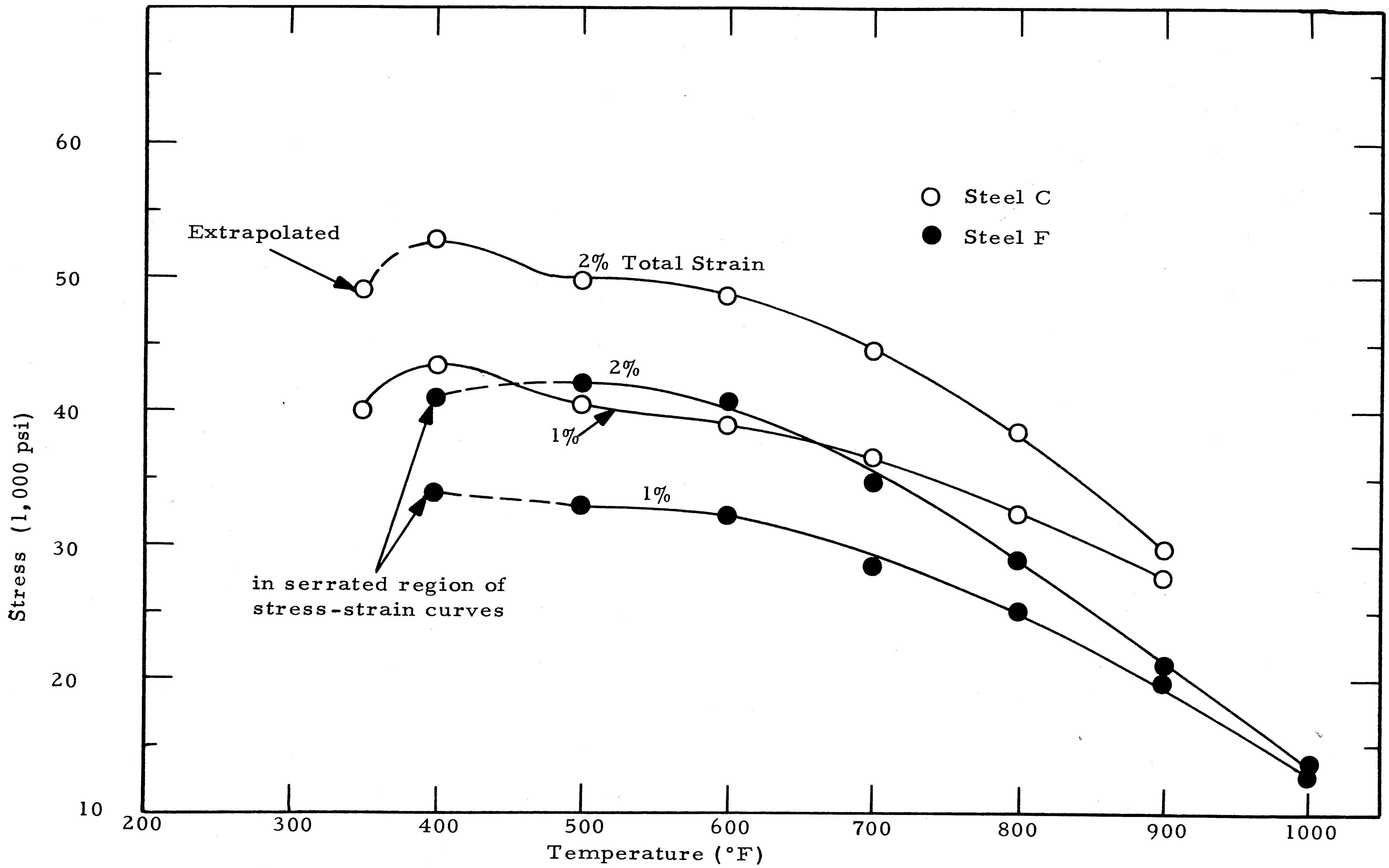


Figure 31. Stress-Temperature Curves for Steels C and F Strained at a Constant Rate of 0.25 Percent per Hour to Total Strains of 1 and 2 Percent. The Si-deoxidized Steel (C) was normalized from 1650°F, and the Si-Al-deoxidized Steel (F) was annealed from 1650°F.

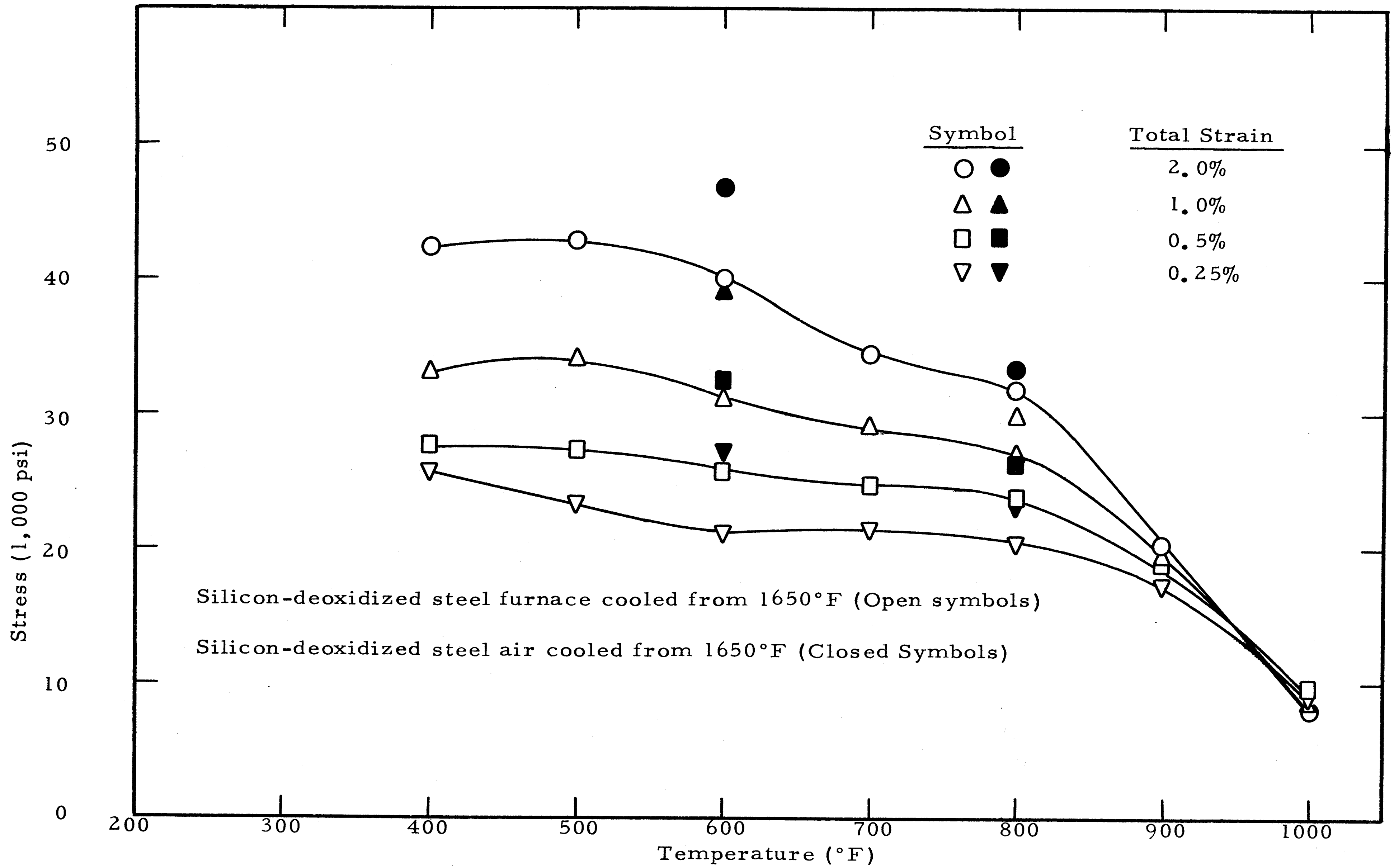


Figure 32. Stress-Temperature Curves for Steel C for a Constant Strain Rate of 0.004 Percent per Hour at Total Deformations of 0.25, 0.5, 1.0, and 2.0 percent.

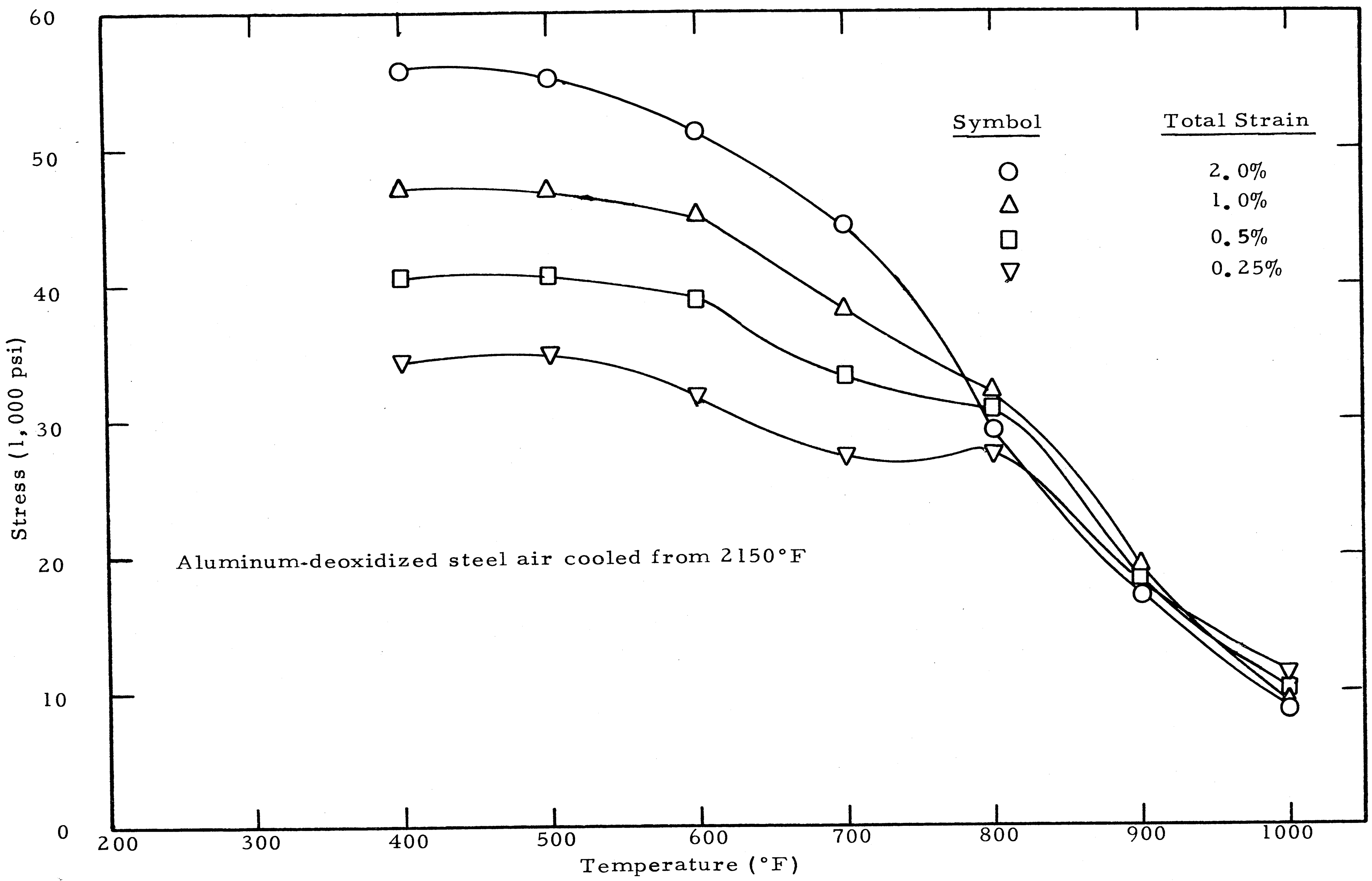


Figure 33. Stress-Temperature Curves for Steel FA at total deformation of 0.25, 0.5, 1.0, and 2.0 Percent for a Constant Strain Rate of 0.004 Percent per Hour.

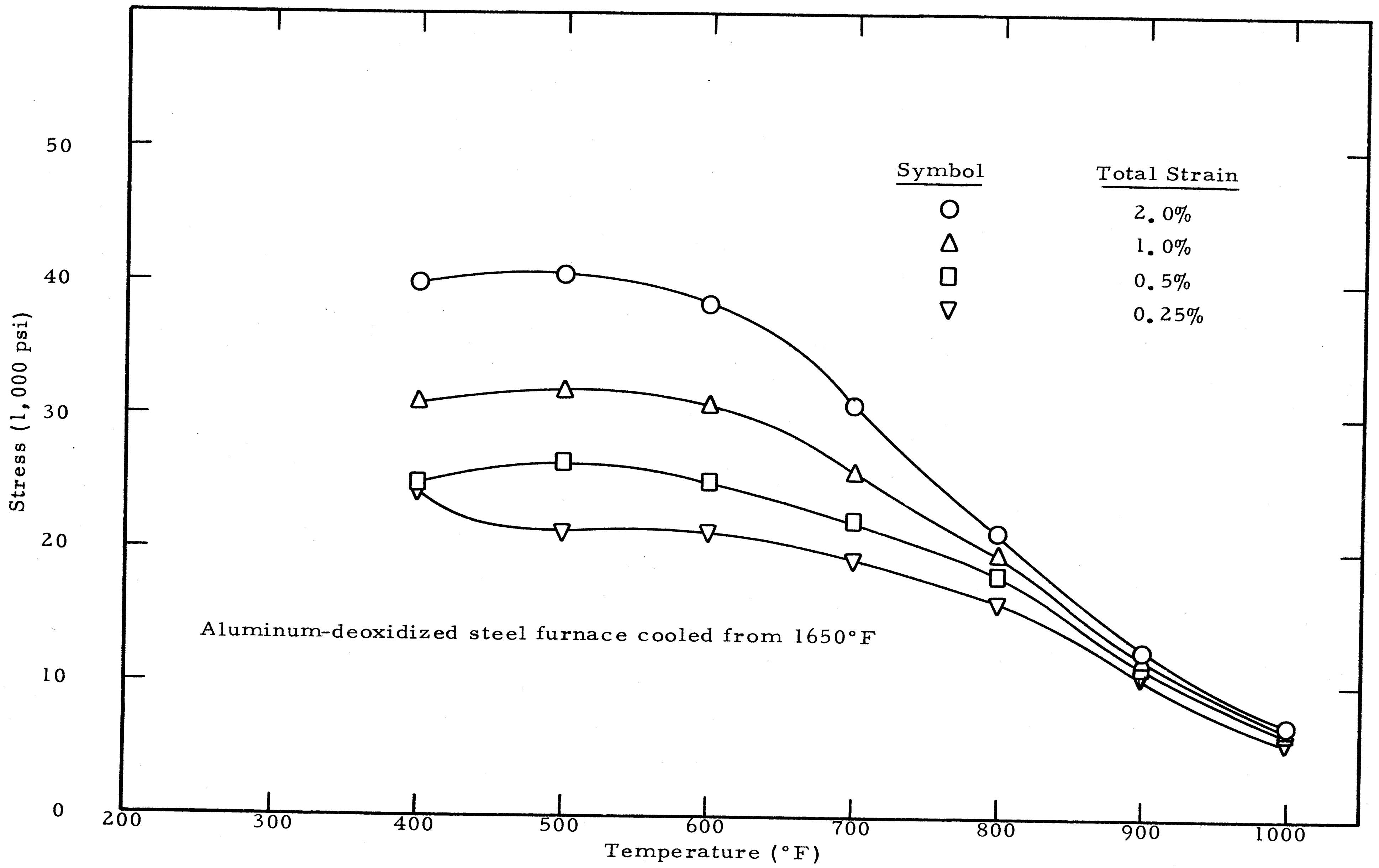


Figure 34. Stress-Temperature Curves for Steel FN at Total Deformation of 0.25, 0.5, 1.0, and 2.0 Percent for a Constant Strain Rate of 0.004 Percent per Hour.

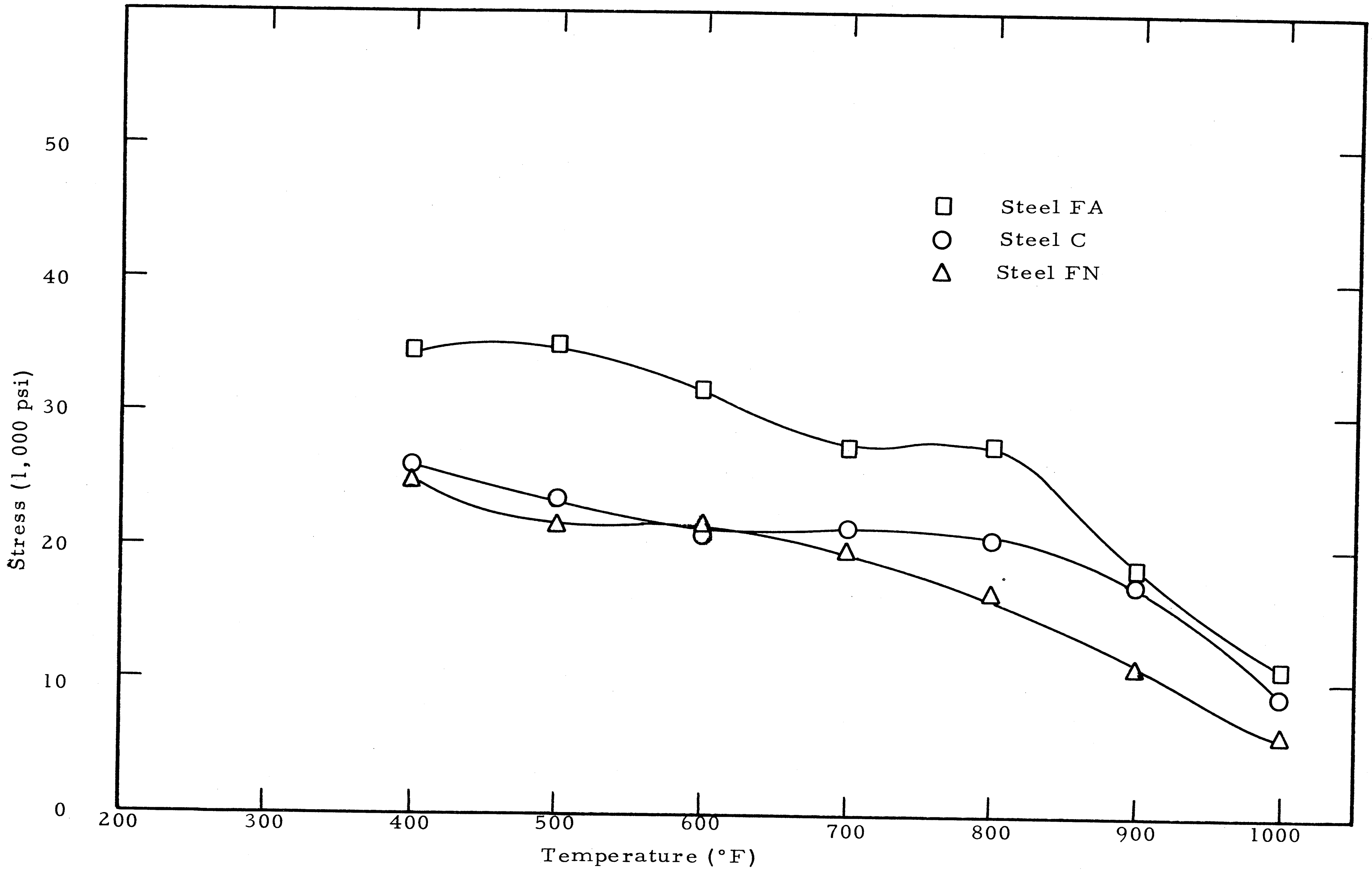


Figure 35. Stress-Temperature Curves of Steel C, FA, and FN for 0.25 Percent Total Strain at a Constant Strain Rate of 0.004 Percent per Hour.

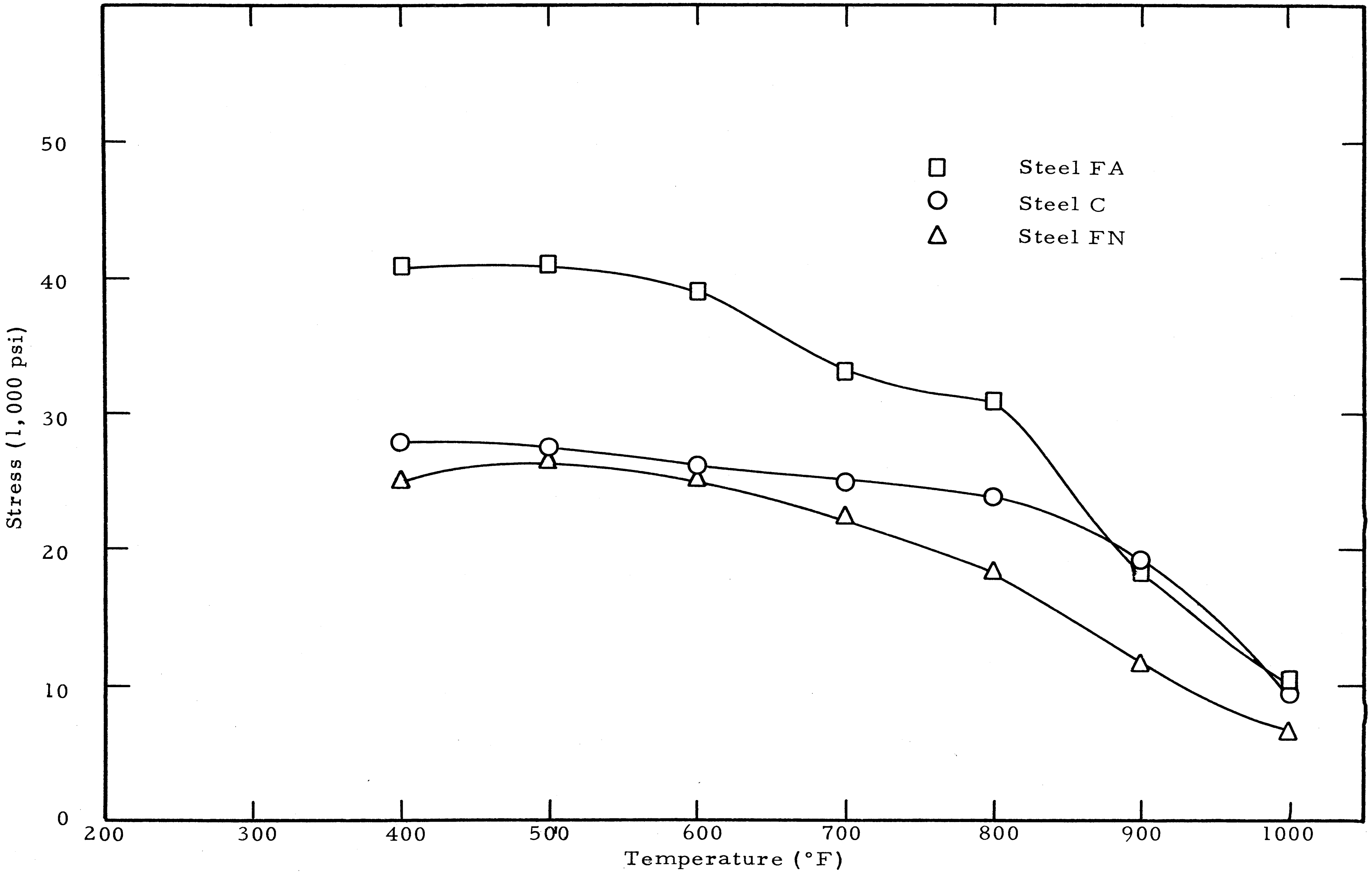


Figure 36. Stress-Temperature Curves of Steels C, FA and FN for 0.50 Percent Total Strain at a Constant Strain Rate of 0.004 Percent per Hour.

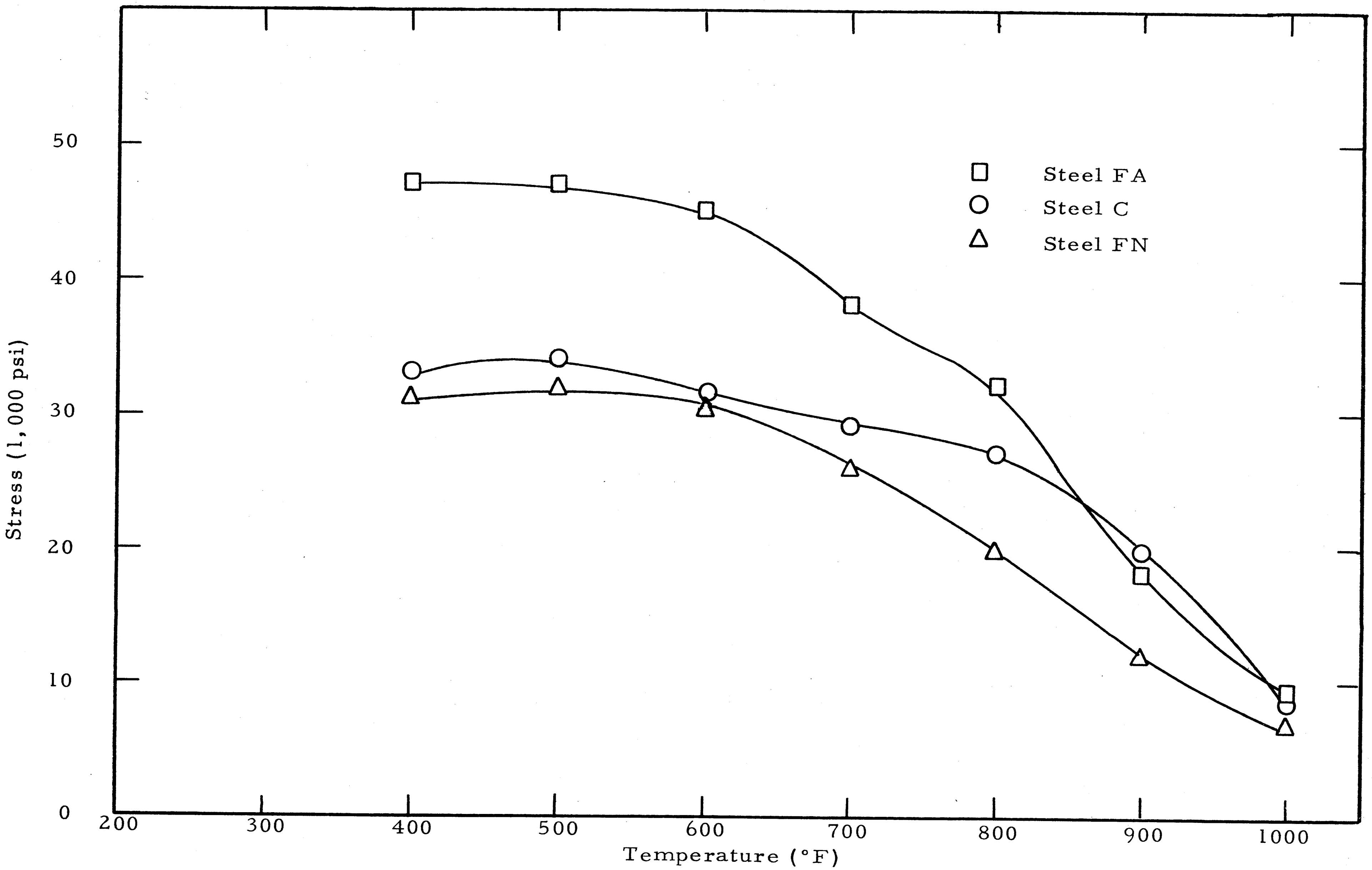


Figure 37. Stress-Temperature Curves of Steels C, FA, and FN for 1.0 Percent Total Strain at a Constant Strain Rate of 0.004 Percent per Hour.

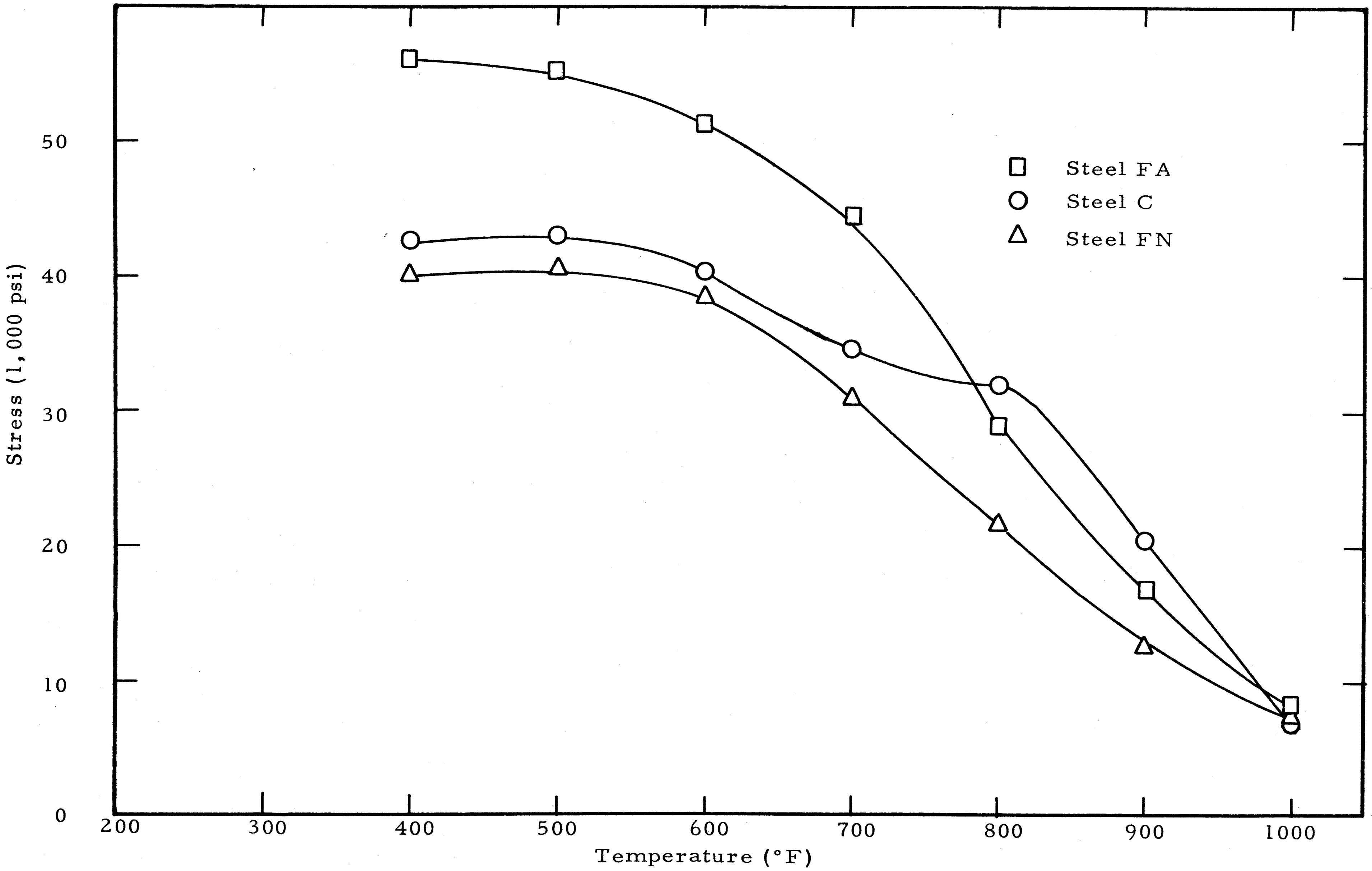
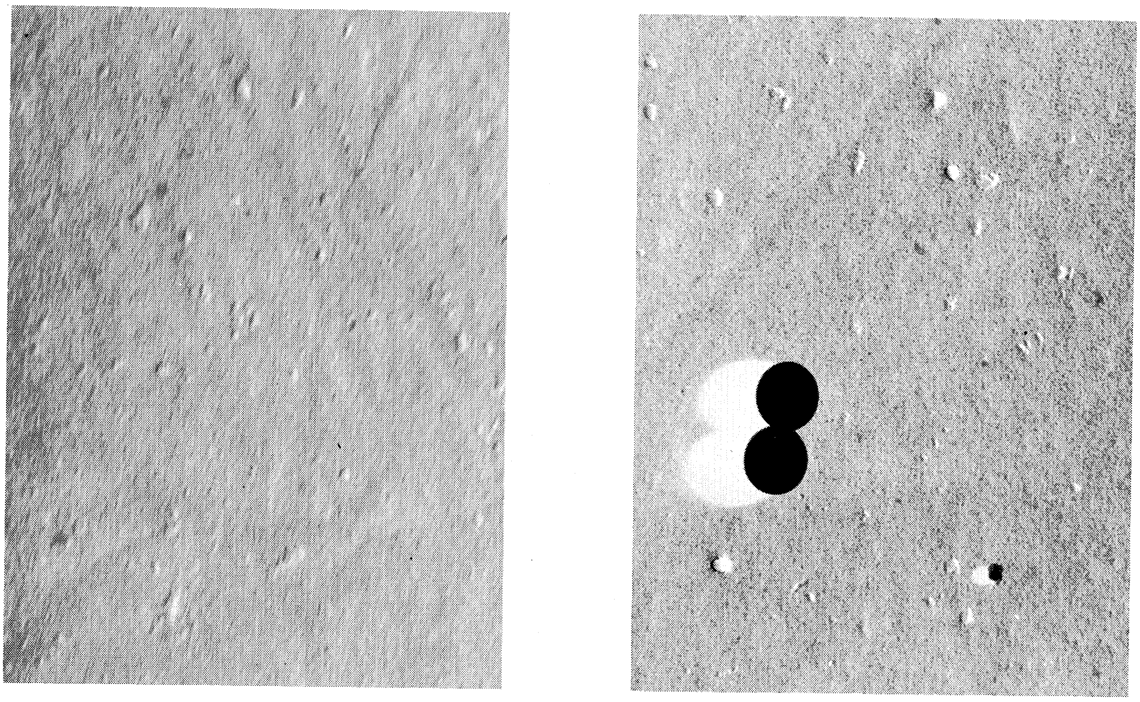


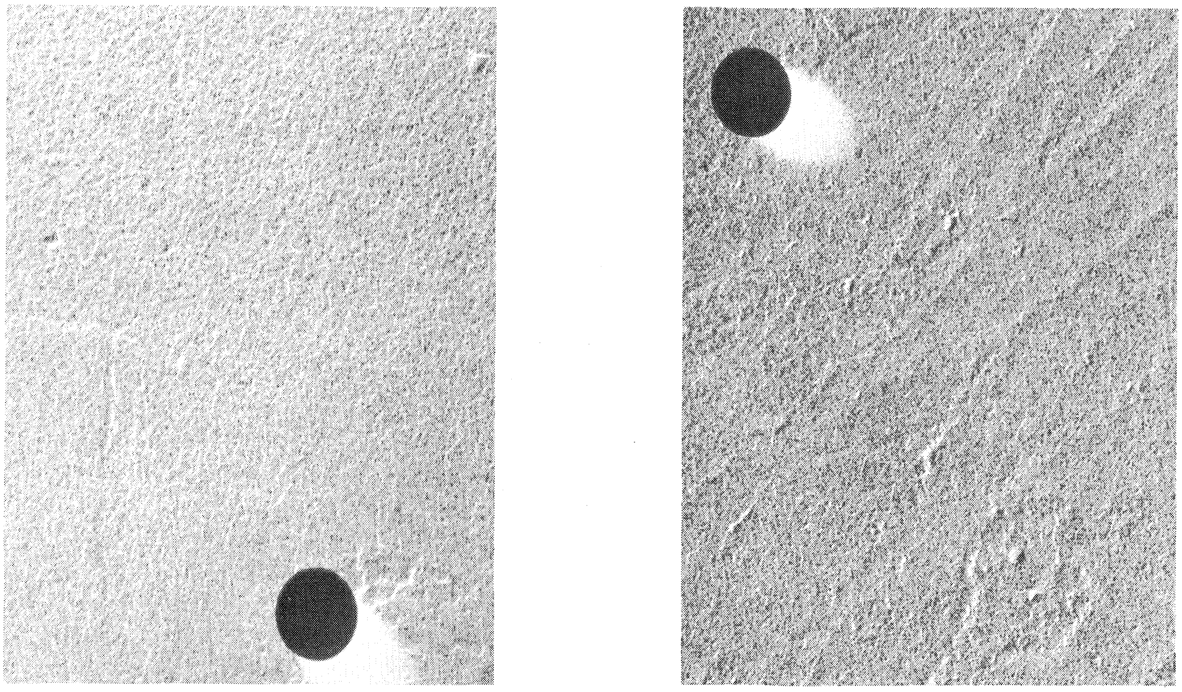
Figure 38. Stress-Temperature Curves of Steel C, FA, and FN for 2.0 Percent Total Strain at a Constant Strain Rate of 0.004 Percent per Hour.



Steel FA; 126 DPH
30,000X

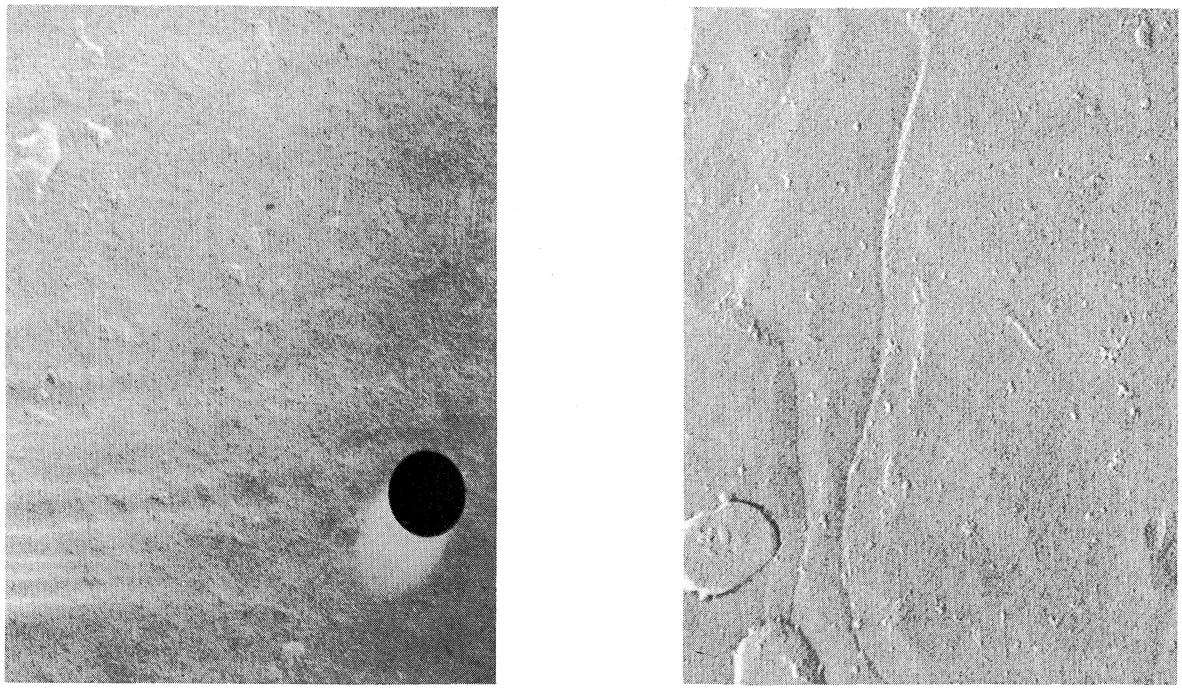
Steel C; 112 DPH
30,000X

Figure 39. Electron Micrographs of Steels FA and C after 2-Percent Deformation at a Constant Strain Rate of 0.004 Percent per Hour at 1000°F. Etchant: 2-Percent Nital. Replica: Collodion shadowed with palladium.



As Heat Treated - 143 DPH

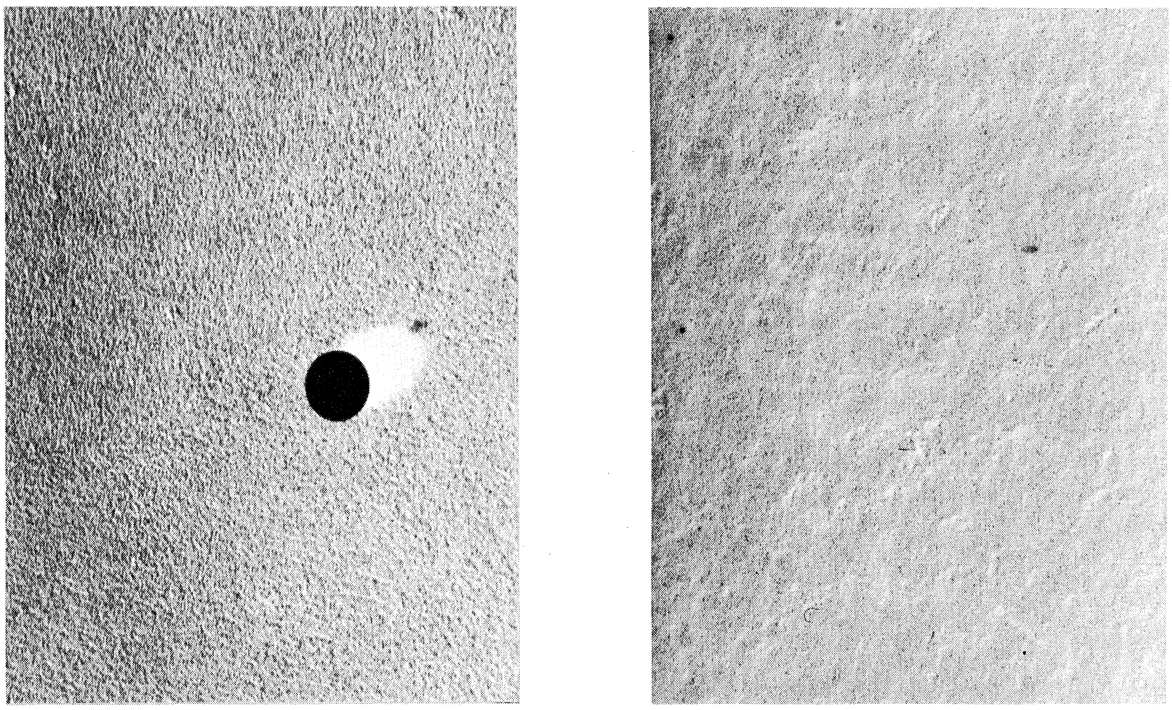
Aged 72 Hours at 1000°F-120DPH



Aged 144 Hours at 1000°F-118DPH

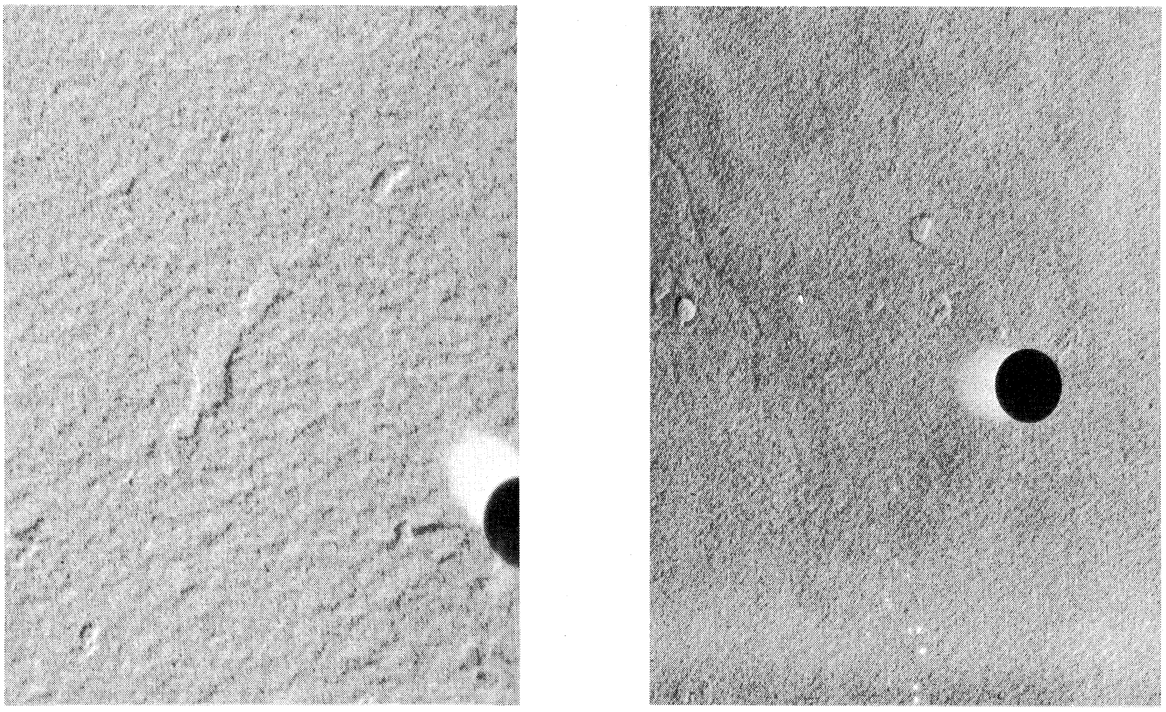
Aged 500 Hours at 1000°F-113DPH

igure 40. Electron Micrographs of Steel FA after Aging at 1000°F for Indicated Times. Magnification: 30,000X. Etchant: 2-Percent Nital. Replica: Collodion shadowed with palladium.



As Heat Treated - 118 DPH

Aged 72 Hours at 1000°F - 105 DPH



Aged 144 Hours at 1000°F - 104 DPH

Aged 500 Hours at 1000°F - 100 DPH

Figure 41. Electron Micrographs of Steel C after Aging at 1000°F for Indicated Times. Magnification: 30,000X. Etchant: 2-Percent Nital. Replica: Collodion shadowed with palladium.

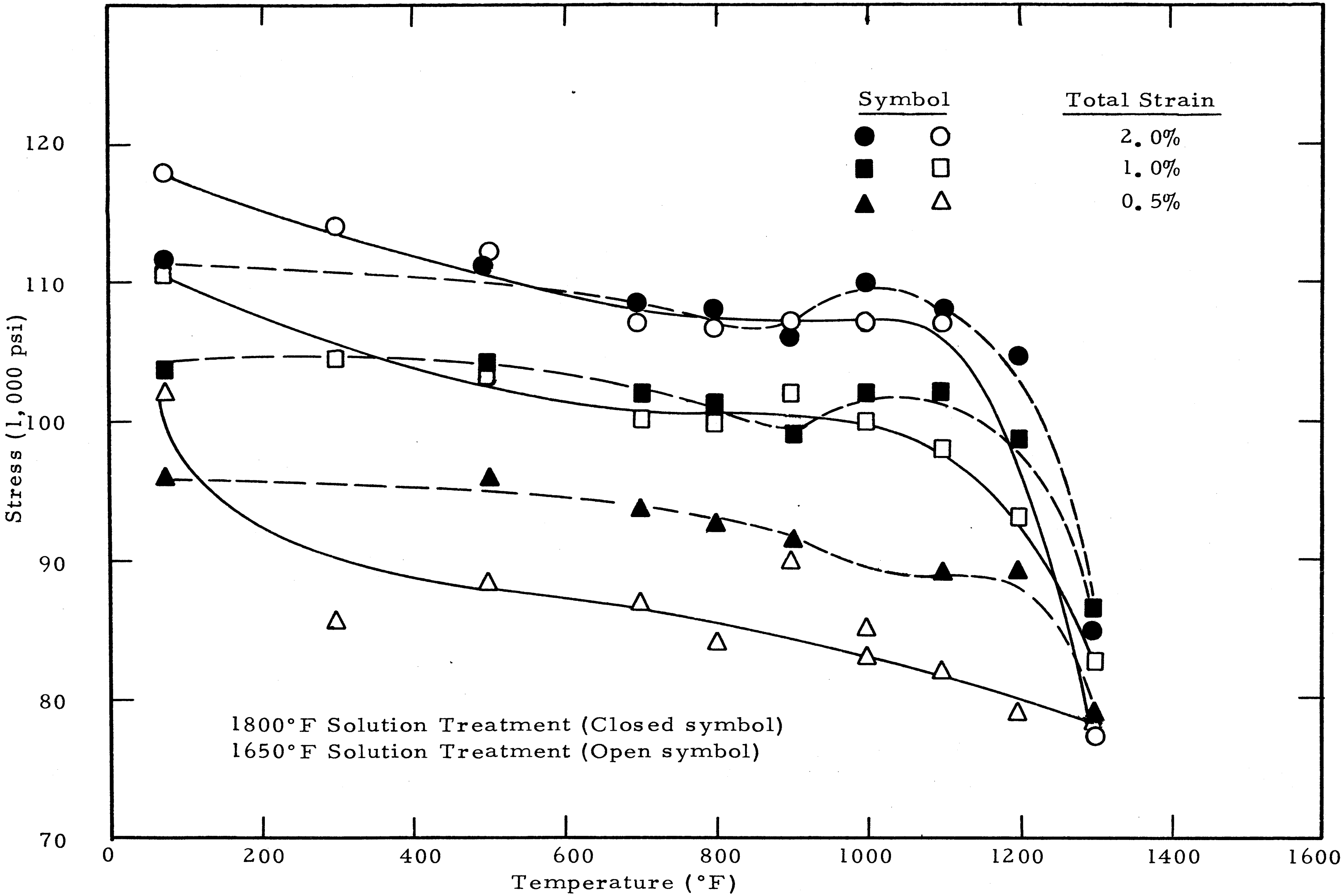


Figure 42. Stress-Temperature Curves of A-286 Alloy for 0.5, 1.0, and 2.0 Percent Total Strain on Tensile Testing.

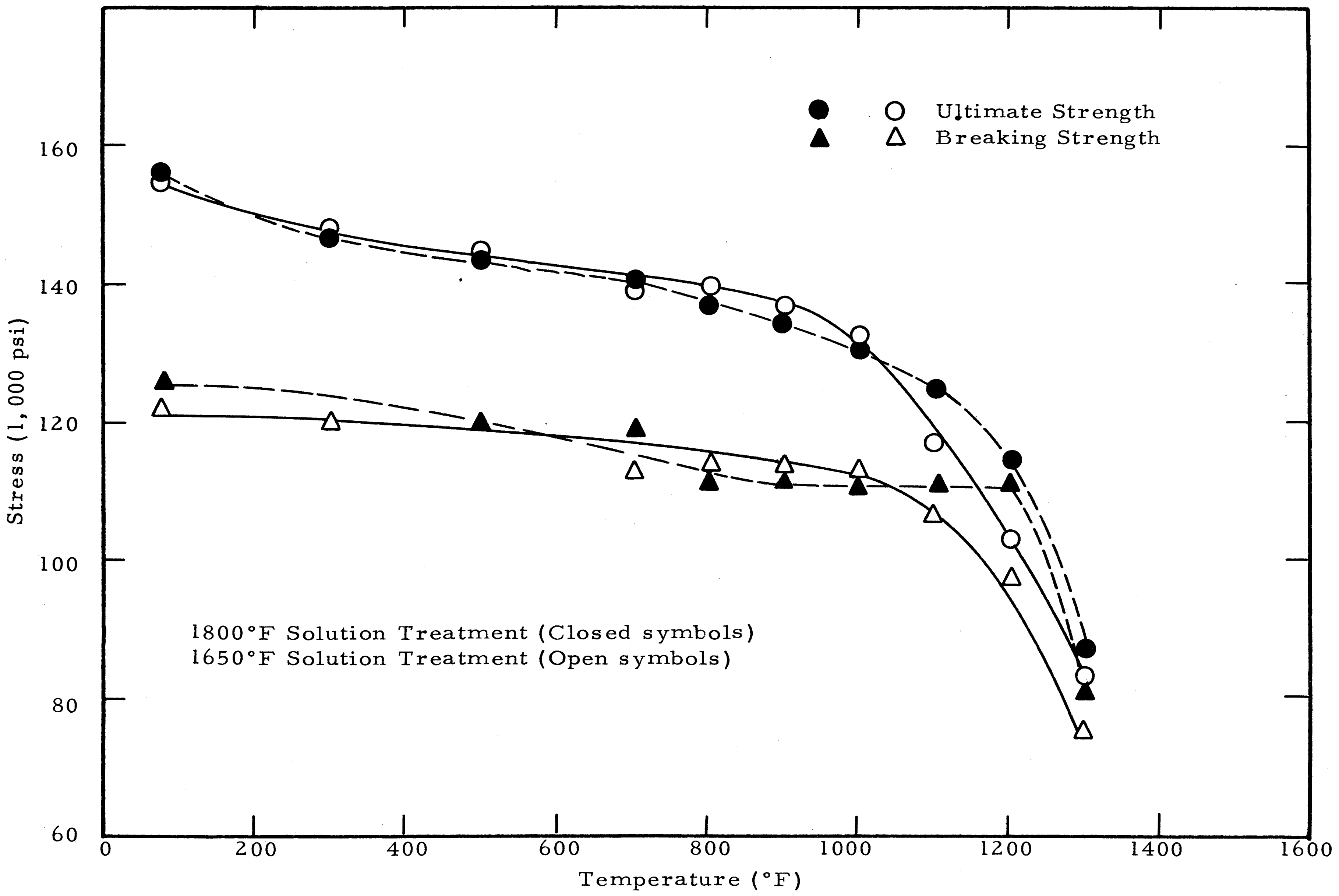


Figure 43. Ultimate and Breaking Strength versus Temperature of A-286 Alloy on Tensile Testing.

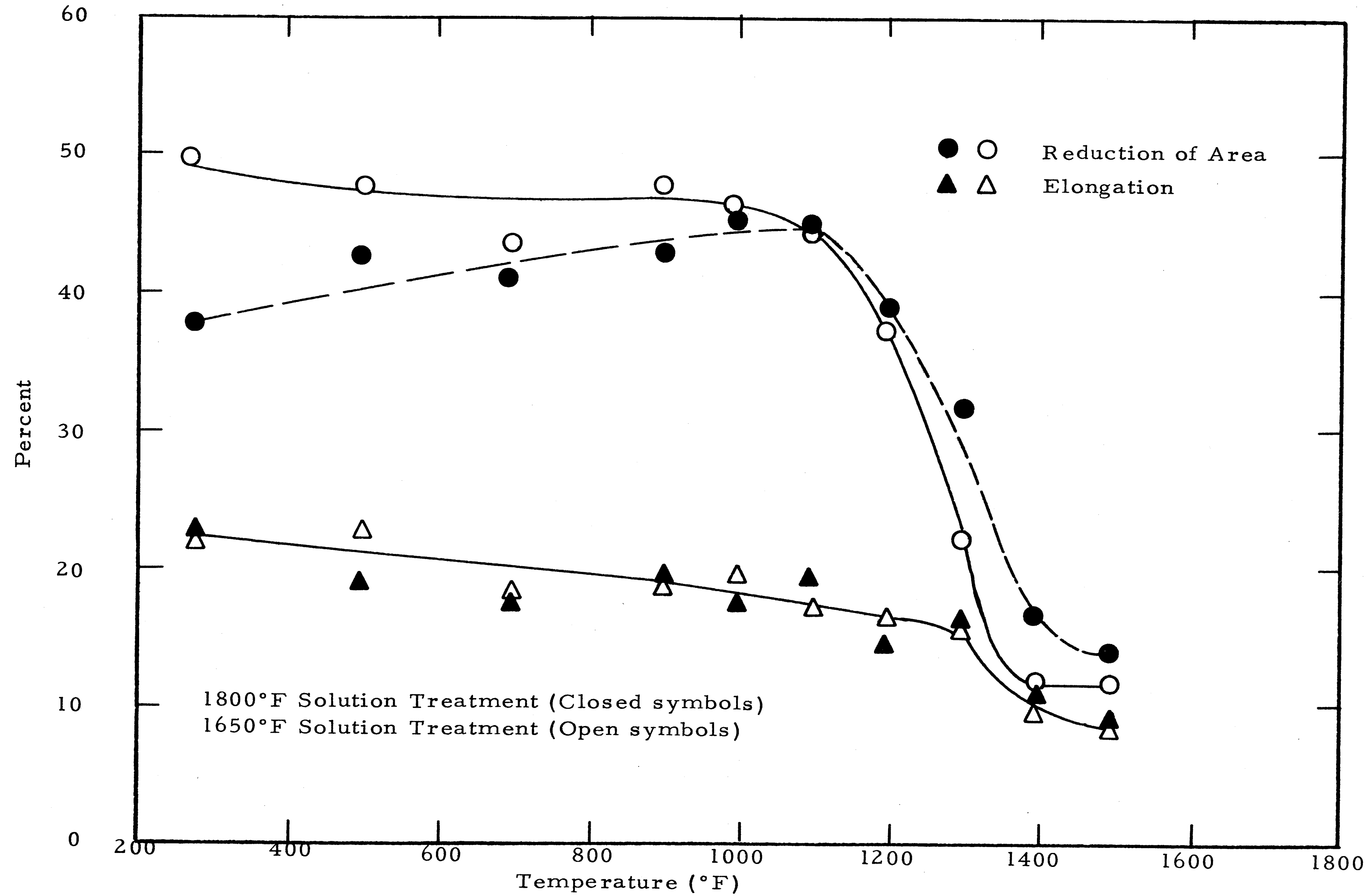


Figure 44. Percent Elongation and Reduction of Area versus Temperature for A-286 Alloy on Tensile Testing.

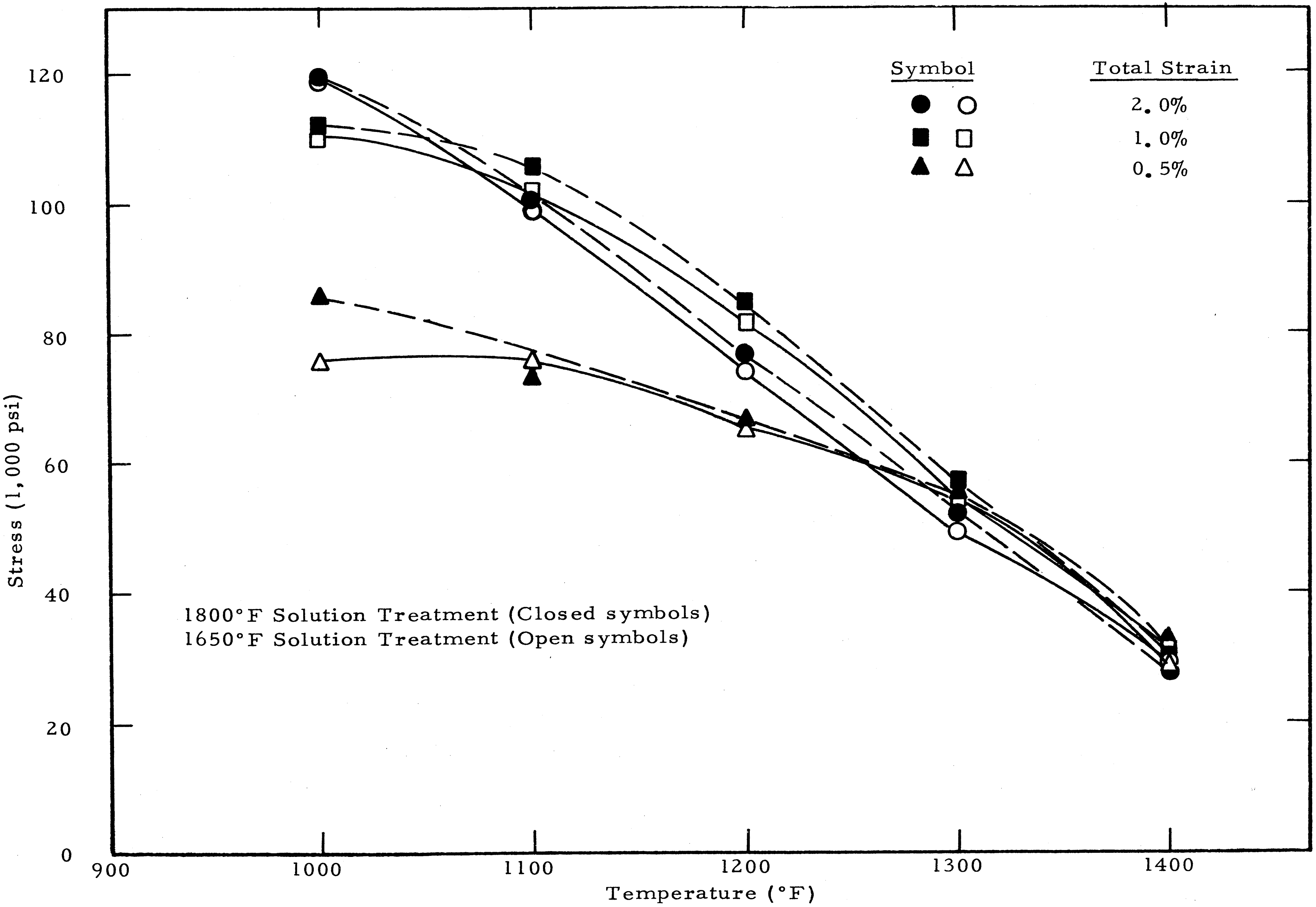


Figure 45. Stress-Temperature Curves for A-286 Alloy at Total Strains of 0.5, 1.0, and 2.0 Percent at a Constant Strain Rate of 0.1 Percent per Hour.

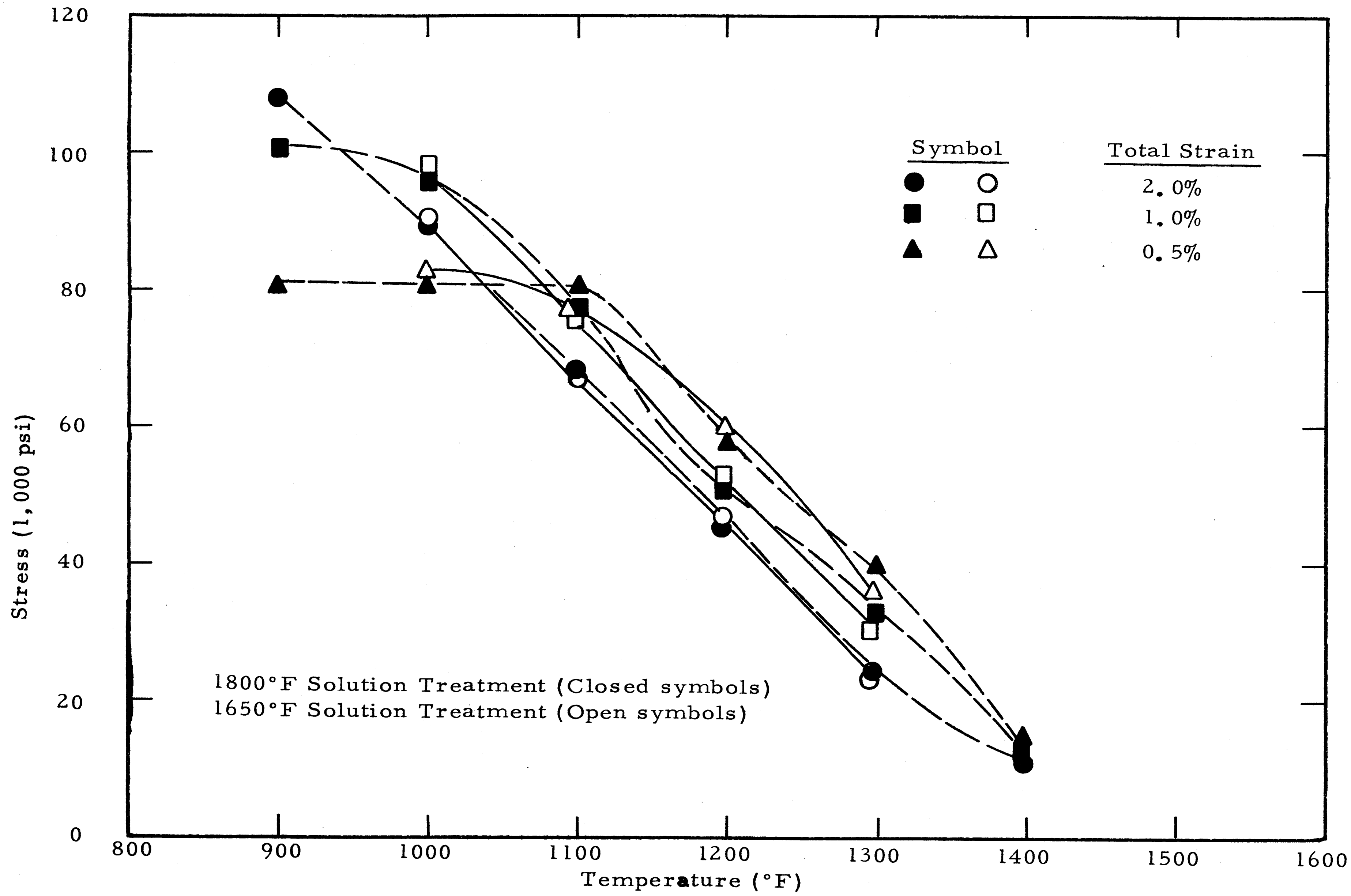


Figure 46. Stress-Temperature Curves for A-286 Alloy at Total Strains of 0.5, 1.0, 2.0 Percent for a Constant Strain Rate of 0.004 Percent per Hour.

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